



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
PREVENTION, PESTICIDES, AND
TOXIC SUBSTANCES

*received 8-23-05
R. Low for DAS*

PC Code: 005100
DP BARCODE: D301682

May 12, 2005

MEMORANDUM

SUBJECT: Aminopyralid: Transmittal of Data Evaluation Reports for Fate Studies

TO: Joanne Miller, Product Manager
Registration Division (7505C)

FROM: Roxolana Kashuba, Environmental Scientist
ERBIV/EFED (7507C)

*Roxolana Kashuba 5/12/05
Cheryl Sutton 5/12/05*

REVIEWED BY: Cheryl A. Sutton, Ph.D., Environmental Scientist
ERBIV/EFED (7507C)

THRU: Elizabeth Behl, Branch Chief
ERBIV/EFED (7507C)

EBehl 5/12/05

This memo is to let you know that the DERs for aminopyralid are finalized and harmonized with Canada's PMRA. The study types and MRIDs are listed in Table 1 below.

Table 1. Aminopyralid Study Types, MRIDs and the Electronic File Names.

Hydrolysis	46235726	005100 46235726 161-1.wpd 005100 46235726 161-1 Calculations .xls
Aqueous Photolysis	46235727	005100 46235727 161-2.wpd 005100 46235727 161-2 Calculations .xls
Photodegradation on Soil	46235728	005100 46235728 161-3.wpd 005100 46235728 161-3 Calculations .xls 005100 46235728 161-3 Nonlinear .jnb 005100 46235728 161-3 Nonlinear summary .doc
Aerobic Soil Metabolism	46235729	005100 46235729 162-1.wpd 005100 46235729 162-1 Calculations .xls 005100 46235729 162-1 Nonlinear .jnb 005100 46235729 162-1 Nonlinear summary .doc
Anaerobic Aquatic Metabolism	46235730	005100 46235730 162-3.wpd 005100 46235730 162-3 Calculations .xls
Aerobic Aquatic Metabolism	46235731	005100 46235731 162-4.doc 005100 46235731 162-4 Calculations .xls 005100 46235731 162-4 Nonlinear .jnb 005100 46235731 162-4 Nonlinear summary .doc
Adsorption/Desorption	46235732	005100 46235732 163-1.wpd 005100 46235732 163-1 Calculations .xls
Terrestrial Field Dissipation	46235734	005100 46235733 164-1.wpd 005100 46235733 164-1 Calculations .xls

received 8-23-05
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Data Evaluation Report on the hydrolysis of aminopyralid (XDE-750)

PMRA Submission Numbers 2004-0789 / 2004-0790

EPA MRID Number 46235726



Data Requirement: PMRA DATA CODE: 8.2.3.2
EPA DP Barcode: D306825
OECD Data Point: IIA 2.9.1, IIA 7.5
EPA Guideline: Subdivision N, §161-1

Test material:

Common name: Aminopyralid.

Chemical name:

IUPAC: 4-amino-3,6-dichloropyridine-2-carboxylic acid
CAS name: 4-amino-3,6-dichloro-2-pyridinecarboxylic acid
CAS No: 150114-71-9
synonyms: XDE-750, DE-750

Primary Reviewer: Anne Gosselin (#1615), PMRA **Date:** 2004-11-03

Secondary Reviewer(s): Hemendra Mulye, PhD (#213), PMRA **Date:** 2004-11-30

Secondary Reviewer(s): Roxolana Kashuba, EPA/EFED/ERB4 **Date:** 2005-02-21

Roxolana Kashuba 5/12/05

Company Code: DOW
Active Code: AMD

Use Site Categories: 13 (terrestrial feed crops), 14 (terrestrial food crops) and 16 (industrial and domestic vegetation control non-food sites)

EPA PC Code: 005100

CITATION: Cook W L. 2003. Hydrolysis of XDE-750 at pH 5, 7, and 9. Regulatory Laboratories—Indianapolis Lab (Indianapolis, Indiana), Dow AgroSciences LLC, Study number 020067, February 12 2003, 45 pages. Unpublished.

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Data Evaluation Report on the hydrolysis of aminopyralid (XDE-750)

PMRA Submission Numbers 2004-0789 / 2004-0790

EPA MRID Number 46235726

EXECUTIVE SUMMARY:

Hydrolysis of [2,6-¹⁴C]-labelled 4-amino-3,6-dichloropyridine-2-carboxylic acid (aminopyralid; XDE-750) at 0.4 mg a.i./L was studied in the dark at 20 °C in sterile aqueous buffered solutions at pH 5 (acetate buffer), pH 7 (tris hydroxymethyl aminomethane buffer), and pH 9 (borate buffer) for 31 days. Hydrolysis was also studied at elevated temperature (50 °C for 5 days). The experiment was conducted in accordance with the US EPA Subdivision N, Section 161-1 and Canada PMRA DACO Number 8.2.3.2, SETAC Section 9 and in compliance with the US EPA Good Laboratory Practice Standards, 40 CFR Part 160. Samples incubated at 20 °C were analyzed at 0, 3, 5, 10, 17, and 31 days and samples incubated at 50 °C were analyzed at 0, 3, and 5 days by removing aliquots, and residues of [2,6-¹⁴C]aminopyralid were analyzed by liquid scintillation counting (LSC) and reverse phase HPLC. Identification of transformation products was not attempted because no transformation products were observed at any of the pH and temperatures tested.

Material balance was $99.3 \pm 0.8\%$, $99.3 \pm 1.2\%$ and $96.9 \pm 1.2\%$ of the applied radioactivity at pH 5, pH 7 and pH 9, respectively, for the samples incubated at 20 °C ; and $98.9 \pm 1.0\%$, $98.8 \pm 1.5\%$ and $96.3 \pm 1.2\%$ of the applied at pH 5, pH 7 and pH 9, respectively, for the samples incubated at 50 °C. At test termination, the HPLC peak areas for [2,6-¹⁴C]aminopyralid remained 100% in pH 5, 7, and 9 at both temperatures. No major nor minor transformation products were detected in any of the pHs tested at both temperatures. No CO₂ or other volatile products were formed.

[2,6-¹⁴C]Aminopyralid was hydrolytically stable at pH 5, 7, and 9 at 20 and 50 °C.

RESULTS SYNOPSIS:

	Half-life (days)	Major transformation products
pH 5	stable	none
pH 7	stable	none
pH 9	stable	none

Study Acceptability: This study is classified as acceptable for a hydrolysis study.

I. MATERIALS AND METHODS

GUIDELINE FOLLOWED: US EPA Subdivision N, §161-1,
Canada PMRA DACO Number 8.2.3.2
EEC Method 7 Point 2.9.1 of Directive 91/414/EEC

COMPLIANCE: Good Laboratory Practices standards, 40 CFR Part 160

Data Evaluation Report on the hydrolysis of aminopyralid (XDE-750)

PMRA Submission Numbers 2004-0789 / 2004-0790

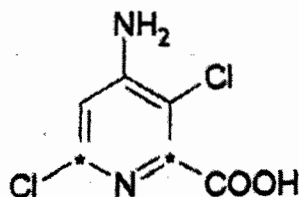
EPA MRID Number 46235726

A. MATERIALS:

1. Test Material

[2,6-¹⁴C]XDE-750 (aminopyralid; p. 16; Figure 1, p. 37).

Chemical Structure:



Description:

Technical, solid (p. 16).

Purity:

Analytical purity: N/A.

Radiochemical purity: 98.6%, #INV1590.

Specific activity: 27.4 mCi/mmole.

Locations of the label: 2, 6-¹⁴C (Figure 1, p. 37).

Storage conditions of test chemicals:

Stored in freezer in acetonitrile, in dark (p. 17).

Physico-chemical properties of aminopyralid (XDE-750):

Parameter	Values	Comments
Water solubility		
pH 5	212 g/L	at 20 °C (1)
pH 7	205 g/L	at 20 °C (1)
pH 9	203 g/L	at 20 °C (1)
unbuffered	2.48 g/L	at 20 °C (1)
Vapour pressure	7.14×10^{-11} mm Hg	at 20 °C (2)
UV absorption	N/A	N/A
pKa	2.56	at 20 °C (3)

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Kow/log Kow	0.201	at 19 °C, unbuffered water (4)
Stability of compound at room temperature, if provided	Stable at least 5 months after receipt	Confirmed by HPLC re-analysis

Data were obtained from p. 16 of the study report.

2. Buffer Solution: Water was obtained from a Millipore Milli-Q water purification unit that filters and exposes the water to UV light to kill bacteria (p. 18 and Table 1, p. 29).

Table 1: Description of buffer solutions.

pH	Type and final molarity of buffer	Composition ^a
5	0.01 M sodium acetate	1.36 g sodium acetate tri-hydrate, 1 L water ^b
7	0.01 M tris(hydroxymethyl) aminomethane (THAM)	1.21 g THAM, 1 L water
9	0.01 M sodium borate	3.81 g sodium tetraborate, 1 L water

Data were obtained from p. 18 and Table 1, p. 29 of the study report.

^a Concentrated HCl was used to adjust the buffer solutions to desired pH.

^b Milli-Q water.

B. STUDY DESIGN

1. Preliminary Study: No preliminary studies were conducted.

2. Experimental conditions

Table 2: Experimental parameters.

Parameters	Details
Duration of test	a) 31 days at 20 °C b) 5 days at 50 °C
Test concentrations (mg a.i./L) nominal: measured:	0.4 mg/l 0.4 mg/l
No. of replicates per time point	2

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EPA MRID Number 46235726

Preparation of test medium	volume used/treatment	200 mL bulk dosing solution prepared; 5-mL of dosing solution pipetted for individual samples
	method of sterilization	Autoclave; sterility of each sample verified using soy broth solution.
	co-solvent (type/concentration)	230 µL acetonitrile = 0.1% co-solvent
	volume of application solution used/treatment	230 µL
	application method	Positive displacement pipette
Test apparatus (type/material/volume)		20-mL amber vials sealed with Teflon-lined screw caps
Details of traps for volatile, if any		N/A
If no traps were used, is the test system closed/open		Test system closed
Is there any indication of XDE-750 adsorbing to the walls of the test apparatus?		No indication of adsorption based on material balances
Experimental conditions Temperature (°C): Lighting:		a) 20 ± 1°C b) 50 ± 1°C Dark
Other details, if any		None

Data were obtained from p. 18-20 and Table 2, p. 30 of the study report.

3. Supplementary Experiments: None

4. Sampling: Duplicate samples of each buffer at 20 °C were analyzed 3, 5, 10, 17, and 31 days after treatment and samples at 50 °C were analyzed 3 and 5 days after treatment. Zero-time samples, used for the 20 and 50 °C data, were analyzed immediately following dosing. Sterility and sample pH were measured at each time point. Sampling details are listed in Table 3.

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Table 3: Sampling details.

Criteria		Details
Sampling intervals for the parent product		Duplicate samples at each time point 0, 3 and 5 days at 50 °C and 0, 3, 5, 10, 17 and 31 days at 20 °C
Sampling method		Direct analysis of samples by LSC and reverse phase HPLC
Sampling methods for the volatile compounds, if any		N/A
Sampling intervals/times	pH measurement	Measured for every sample at every sampling time using pH meter
	sterility checks	Measured for every sample by adding approximately 500 µL of sample to soy broth solution. Incubate dosed soy broth solution at 25 °C for at least 3 days and observe. Clear solution indicates sterility.
Sample storage before analysis		N/A
Other observation, if any (e.g.: precipitation, color change etc.)		None

Data were obtained from p. 20 and Table 3, p. 31 of the study report.

C. ANALYTICAL METHODS:

At each sample point, an aliquot of each sample was removed for sterility check. Triplicate 0.1-mL aliquots were then assayed by LSC. Another aliquot was removed for HPLC analysis. The pH of each sample was then recorded (p. 21).

An isocratic reverse phase HPLC method was used to quantitate XDE-750 concentrations. A 4.6 mm x 25 cm Inertsil ODS column was used. The isocratic mobile phase was 90/10/0.5 water/acetonitrile/TFA. The flow-rate was 1.0 mL/min and the UV absorbance was set at 270 nm. The eluent was collected in 50-mL vials, the eluent volume was recorded and a 2-mL aliquot was counted to assess column recoveries. The stop-time was 20 minutes and typical injection volumes were 95 µL ¹⁴C sample and 5-µL non-labeled XDE-750 reference standard. A radioactive flow-through detector (RAM) was used to quantitate the amount of XDE-750 in each sample (p. 22).

Additional HPLC was conducted to confirm the presence of XDE-750 in solution. Single replicates of the 31-DAT samples (pH 5, 7, and 9) were analyzed using hydrophilic interaction

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chromatography (HILIC) using a 4.6 mm x 20 cm polyhydroxyethyl Aspartamidetm column (PolyLC, Incorporated). Samples must contain at least 70% acetonitrile for HILIC HPLC. Therefore, 1-mL aqueous aliquots were concentrated to dryness and re-dissolved in 500 μ L acetonitrile. A HP 1050 HPLC equipped with two solvent reservoirs, one containing 10 mmol ammonium acetate and the other containing acetonitrile with 1% acetic acid was used. From 0 to 10 minutes, the gradient consisted of 100% acetonitrile with 1% acetic acid. The acetonitrile solution was decreased linearly from 100% at 10 minutes to 30% at 30 minutes. Conversely, the 10 mmol ammonium acetate was increased linearly from 0% at 10 minutes to 70% at 30 minutes. The flow-rate was 1.0 mL/minute and the UV absorbance was set at 270 nm. The test was 30 minutes and the injection volume was 95 μ L sample and 5 μ L cold standard. The run for column was equilibrated at initial conditions for 15 minutes between samples (p. 22).

The LOD and LOQ were determined using the Currie (6) method. The LOD and LOQ were 13 and 57 dpm above background. Each aqueous aliquot counted contained greater than 10,000 dpm. Therefore, the quantitation limit of 14 C for the aqueous recoveries analyses was $< 0.57\%$ ($57/10,000$) of applied radiocarbon (p. 23-24).

II. RESULTS AND DISCUSSION:

A. TEST CONDITIONS:

The pH and sterility of each sample were assayed at sample sacrifice. Sample pH was ± 0.1 pH unit for pH 5, 7, and 9. All samples were sterile throughout the study (p. 24). Temperature was maintained at $20 \pm 1^\circ\text{C}$ and $50 \pm 1^\circ\text{C}$, respectively, (p. 20, Table 2, p. 30), although no supporting temperature data was provided.

B. MASS BALANCE:

At 20°C , total radiocarbon recovery ranged from 98.3 to 100.6% of the applied amount at pH 5, 96.6 to 100.7% of the applied amount at pH 7 and 95.0 to 98.9% of the applied amount at pH 9, in replicate samples. At 50°C , total radiocarbon recovery ranged from 97.6 to 100% of the applied amount at pH 5, 96.9 to 100.6% of the applied amount at pH 7 and 95 to 98.3% of the applied amount at pH 9, in replicate samples (Tables 6-8, p. 34-36).

Table 4: Hydrolysis of [2,6- 14 C]aminopyralid, expressed as percentage of the applied radioactivity (mean \pm s.d.), at pH 5 at 20°C .

Compound	Sampling times (days)					
	0	3	5	10	17	31
Parent compound	99.8 ± 0.4	98.3 ± 0.0	99.6 ± 1.4	99.4 ± 0.6	99.8 ± 0.6	98.6 ± 0.1
Transformation product 1	N/A	N/A	N/A	N/A	N/A	N/A

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Unidentified radioactivity, if any		N/A	N/A	N/A	N/A	N/A	N/A
Volatiles	CO ₂	N/A	N/A	N/A	N/A	N/A	N/A
	volatile organic 1	N/A	N/A	N/A	N/A	N/A	N/A
Total % recovery		99.8 ±0.4	98.3 ±0.0	99.6 ±1.4	99.4 ±0.6	99.8 ±0.6	98.6 ±0.1

Data were obtained from Table 6, p. 34 of the study report.

Table 4B: Hydrolysis of [2,6-¹⁴C]aminopyralid, expressed as percentage of the applied radioactivity (mean ± s.d.), at pH 5 at 50 °C.

Compound		Sampling times (days)		
		0	3	5
Parent compound		99.8 ±0.4	98.7 ±1.5	98.2 ±0.6
Transformation product 1		N/A	N/A	N/A
Unidentified radioactivity, if any		N/A	N/A	N/A
Volatiles	CO ₂	N/A	N/A	N/A
	volatile organic 1	N/A	N/A	N/A
Total % recovery		99.8 ±0.4	98.7 ±1.5	98.2 ±0.6

Data were obtained from Table 6, p. 34 of the study report.

Table 5: Hydrolysis of [2,6-¹⁴C]aminopyralid, expressed as percentage of the applied radioactivity (mean ± s.d.), at pH 7 at 20 °C.

Compound		Sampling times (days)					
		0	3	5	10	17	31
Parent compound		100.1 ±0.7	97.5 ±1.2	99.9 ±0.3	98.8 ±0.0	98.8 ±0.1	100.7 ±0.1
Transformation product 1		N/A	N/A	N/A	N/A	N/A	N/A

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Data Evaluation Report on the hydrolysis of aminopyralid (XDE-750)

PMRA Submission Numbers 2004-0789 / 2004-0790

EPA MRID Number 46235726

Unidentified radioactivity, if any		N/A	N/A	N/A	N/A	N/A	N/A
Volatiles	CO ₂	N/A	N/A	N/A	N/A	N/A	N/A
	volatile organic 1	N/A	N/A	N/A	N/A	N/A	N/A
Total % recovery		100.1 ±0.7	97.5 ±1.2	99.9 ±0.3	98.8 ±0.0	98.8 ±0.1	100.7 ±0.1

Data were obtained from Table 7, p. 35 of the study report.

Table 5B: Hydrolysis of [2,6-¹⁴C]aminopyralid, expressed as percentage of the applied radioactivity (mean ± s.d.), at pH 7 at 50 °C.

Compound		Sampling times (days)		
		0	3	5
Parent compound		100.1 ±0.7	97.9 ±0.6	98.4 ±2.1
Transformation product 1		N/A	N/A	N/A
Unidentified radioactivity, if any		N/A	N/A	N/A
Volatiles	CO ₂	N/A	N/A	N/A
	volatile organic 1	N/A	N/A	N/A
Total % recovery		100.1 ±0.7	97.9 ±0.6	98.4 ±2.1

Data were obtained from Table 7, p. 35 of the study report.

Table 6: Hydrolysis of [2,6-¹⁴C]aminopyralid, expressed as percentage of the applied radioactivity (mean ± s.d.), at pH 9 at 20 °C.

Compound		Sampling times (days)					
		0	3	5	10	17	31
Parent compound		97.5 ±1.2	96.2 ±1.1	96.5 ±0.4	96.6 ±1.0	96.2 ±1.6	98.5 ±0.6
Transformation product 1		N/A	N/A	N/A	N/A	N/A	N/A
Unidentified radioactivity, if any		N/A	N/A	N/A	N/A	N/A	N/A

Data Evaluation Report on the hydrolysis of aminopyralid (XDE-750)

PMRA Submission Numbers 2004-0789 / 2004-0790

EPA MRID Number 46235726

III. STUDY DEFICIENCIES:

- 1) The concentration of the test substance at each sampling time should have been measured in triplicates instead of duplicates.
- 2) [2,6-¹⁴C]Aminopyralid is very soluble in water. Therefore, use of a solvent was unnecessary.
- 3) The temperature records were not provided. It was only stated that the incubation temperatures were 20°C ±1°C and 50°C ±1°C during the two parts of study.

These deficiencies are considered minor and, therefore, this study is acceptable.

IV. REVIEWER'S COMMENTS:

These results indicate that [2,6-¹⁴C]aminopyralid is stable to hydrolysis at environmentally-relevant acidic, neutral and alkaline pH.

V. REFERENCES:

1. Nelson, R., "Determination of the Water Solubility of XDE-750", FOR01015, unpublished report of Dow AgroSciences LLC, 2002.
2. Griffin, K. A., "Vapor Pressure of XR-750 by Knudsen-Effusion Weight Loss Method", AS-PR 2001-002, unpublished report of The Dow Chemical Company, 2001.
3. Cathie, C., "Determination of Dissociation Constant of XR-750 using UV-Visible Spectrophotometry", 01-822-AG, unpublished report of Dow AgroSciences LLC, 2001.
4. Madsen, S., "Determination of the n-Octanol/Water Partition Coefficient (Shake Flask Method) of XDE-750", FOR01009, unpublished report of Dow AgroSciences LLC, 2003.
5. Roulin, S. P., "Determination of the Organic Solubility of XR-750", 01003/DA, unpublished report of Dow AgroSciences LLC, 2001.
6. Currie, L. A. "Limits for Qualitative Detection and Quantitative Determination-Application to Radiochemistry", Anal. Chem. 1968, 40, 586-593.

Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235726
 Guideline No: 161-1

Tables 4-6 Hydrolysis of [2,6-C14]aminopyralid in pH 5, 7, and 9 buffers, incubated at 20°C, expressed as percentage of the applied radioactivity

pH	5		7		9	
	Parent	Total	Parent	Total	Parent	Total
0	100.1	100.1	99.6	99.6	96.6	96.6
0	99.5	99.5	100.6	100.6	98.3	98.3
AVG	99.8	99.8	100.1	100.1	97.5	97.5
STDEV	0.4	0.4	0.7	0.7	1.2	1.2
3	98.3	98.3	96.6	96.6	95.4	95.4
3	98.3	98.3	98.3	98.3	97.0	97.0
AVG	98.3	98.3	97.5	97.5	96.2	96.2
STDEV	0.0	0.0	1.2	1.2	1.1	1.1
5	98.6	98.6	100.1	100.1	96.8	96.8
5	100.6	100.6	99.7	99.7	96.2	96.2
AVG	99.6	99.6	99.9	99.9	96.5	96.5
STDEV	1.4	1.4	0.3	0.3	0.4	0.4
10	99.0	99.0	98.8	98.8	97.3	97.3
10	99.8	99.8	98.8	98.8	95.9	95.9
AVG	99.4	99.4	98.8	98.8	96.6	96.6
STDEV	0.6	0.6	0.0	0.0	1.0	1.0
17	99.4	99.4	98.7	98.7	95.0	95.0
17	100.2	100.2	98.9	98.9	97.3	97.3
AVG	99.8	99.8	98.8	98.8	96.2	96.2
STDEV	0.6	0.6	0.1	0.1	1.6	1.6
31	98.5	98.5	100.6	100.6	98.0	98.0
31	98.7	98.7	100.7	100.7	98.9	98.9
AVG	98.6	98.6	100.7	100.7	98.5	98.5
STDEV	0.1	0.1	0.1	0.1	0.6	0.6
AVR		99.3		99.3		96.9
STDEV		0.8		1.2		1.2

Data were obtained from Tables 6-8, p. 34-36 of the study report.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235726
 Guideline No: 161-1

Tables 4B-6B Hydrolysis of [2,6-C14]aminopyralid in pH 5, 7, and 9 buffers, incubated at 50°C, expressed as percentage of the applied radioactivity

pH	5		7		9	
Time (days)	Parent	Total	Parent	Total	Parent	Total
0	100.1	100.1	99.6	99.6	96.6	96.6
0	99.5	99.5	100.6	100.6	98.3	98.3
AVG	99.8	99.8	100.1	100.1	97.5	97.5
STDEV	0.4	0.4	0.7	0.7	1.2	1.2
3	97.6	97.6	98.3	98.3	95.1	95.1
3	99.7	99.7	97.4	97.4	96.6	96.6
AVG	98.7	98.7	97.9	97.9	95.9	95.9
STDEV	1.5	1.5	0.6	0.6	1.1	1.1
5	98.6	98.6	96.9	96.9	95.0	95.0
5	97.8	97.8	99.8	99.8	96.4	96.4
AVG	98.2	98.2	98.4	98.4	95.7	95.7
STDEV	0.6	0.6	2.1	2.1	1.0	1.0
AVR		98.9		98.8		96.3
STDEV		1.0		1.5		1.2

Data were obtained from Tables 6-8, p. 34-36 of the study report.

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received 8/23/06
R. Loma for DAS

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Data Evaluation Report on the phototransformation of aminopyralid (XDE-750) in water

PMRA Submission Number 2004-0789 / 2004-0790

EPA MRID Number 46235727



Data Requirement:

PMRA DATA CODE:	8.2.3.3.2
US EPA DP Barcode:	Not available
OECD Data Point:	IIA 2.9.2, IIA 7.6
US EPA Guideline:	Subdivision N, §161-2

Test material:

Common name: XDE-750

Chemical name:

IUPAC: 4-amino-3,6-dichloropyridine-2-carboxylic acid

CAS name: 4-amino-3,6-dichloro-2-pyridinecarboxylic acid

CAS No: 150114-71-9

Synonyms: aminopyralid, DE-750

SMILES string: ClC(=O)C1=CC(=C(N)C1)Cl

Primary Reviewer: Anne Gosselin (#1615), PMRA

Date: 2004-11-05

Secondary Reviewer(s): Hemendra Mulye, PhD (#213), PMRA

Date: 2004-11-29

Secondary Reviewer(s): Roxolana Kashuba, EPA

Date: 2005-02-01

Roxolana Kashuba 5/12/05

Company Code: DOW

Active Code: AMD

Use Site Categories: 13 (terrestrial feed crops), 14 (terrestrial food crops) and 16 (industrial and domestic vegetation control non-food sites)

EPA PC Code: 005100

CITATION: Cook W. L. 2003. Aqueous photolysis of XDE-750 in pH 5 buffer under xenon light. Regulatory Laboratories—Indianapolis Lab (Indianapolis, Indiana), Dow AgroSciences LLC, Study number 020066, October 21 2003, 92 pages. Unpublished.

Data Evaluation Report on the phototransformation of aminopyralid (XDE-750) in water

PMRA Submission Number 2004-0789 / 2004-0790

EPA MRID Number 46235727

EXECUTIVE SUMMARY:

The aqueous phototransformation of [2,6-¹⁴C]-labelled 4-amino-3,6-dichloropyridine-2-carboxylic acid (aminopyralid; XDE-750) was studied at 20 °C in sterile aqueous acetate buffered solution at pH 5 at initial concentrations of 0.2 and 30 mg a.i./L by continuous irradiation using a xenon lamp (290-400 nm with a combination of infrared and soda lime filters) for an equivalent of 38 days of summer sunlight at 40° N latitude. Two separate sets of low dose samples were used to study kinetics, the rise and decline of degradates, and the stability of [2,6-¹⁴C]aminopyralid in the dark. The high dose samples were used to identify degradation products. The experiment was conducted in accordance with the US EPA Subdivision N Section 161-2, SETAC Part 1, Section 10.1, and to meet the US EPA Good Laboratory Practice Standards, 40 CFR Part 160. Following 2 and 6 days of sample irradiation, test vessels were removed from the lamp room and connected to traps for the collection of CO₂ and organic volatiles. The 12 and 15-day samples contained CO₂ traps within the sample container. Low concentration kinetics-determining samples were analyzed at 0, 1, 2, 4, 6, and 8 hours after treatment, low concentration degradate-determining samples were analyzed 2, 6, 12, and 15 days after treatment (DAT) by reverse phase HPLC (samples were not extracted). High concentration samples were analyzed at 3 days after treatment; identification of transformation products was done by HPLC.

A PNA/pyridine (*p*-nitroacetophenone/pyridine) chemical actinometer solution was used to quantitate the amount of light that the 1, 2, 4, 6, and 8-hour samples received. Based on the PNA/pyridine actinometry data the xenon lamp was 1.58 times as intense as summer sunlight at 40° N latitude for these samples.

A PNAP/pyridine (*p*-nitroanisole/pyridine) chemical actinometer solution was used to quantitate the amount of light that the 2, 6, 12, and 15-day samples received. Based on the PNAP/pyr actinometer data, 15 days of continuous irradiation was equivalent to 38 days of irradiation in the summer sun at 40° N latitude.

The mass balance was $97.8 \pm 2.6\%$ and $99.4 \pm 0.7\%$ of the applied amount for the kinetics sample set in the dark and irradiated samples, respectively. The mass balance was $94.0 \pm 5.9\%$ and $100.0 \pm 0.0\%$ of the applied amount for the degradate sample set in the dark and irradiated samples, respectively. At study termination, 100.0% of the applied radioactivity remained as the parent in the dark samples. No transformation occurred in the dark samples. Volatiles in the dark samples were not trapped.

In the irradiated samples, the concentration of the parent compound decreased from 96.8% at day 0 to 0.0% of the applied amount at 2 days post-treatment. The major transformation products detected in the irradiated samples were not all identified nor separately quantified. The only two potentially major transformation products identified, but not quantified, in the irradiated samples were oxamic acid (CAS #471-47-6) and malonamic acid (CAS # 2345-56-4). These two products plus at least four more acid amides (2 or 3 carbons in length) together reached a

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maximum concentration of 68.8% of applied radioactivity by 12 days post-treatment. These six or more transformation products together decreased to 65.7% of applied radioactivity at study termination. The minor transformation products were not identified, but were detected at maximums of 2.9%, 4.0 %, and 3.3% of the applied amount in replicate samples at 12 days, 8 hours, and 2 days, respectively. At study termination, in the irradiated samples, evolved CO₂ amounted to 28.4% of the applied amount. Volatile organics were not detected at study termination, despite having reached a maximum of 2.4 % of the applied amount at 6 days post-treatment. The total unidentified radioactivity was a maximum of 0.0% and 7.2% of the applied amount at 8 hours post treatment in the dark and irradiated samples, respectively. All products are formed by phototransformation only.

[2,6-¹⁴C]Aminopyralid photodegrades via de-chlorination and ring cleavage to form unidentified acid-amides, oxamic acid and malonamic acid, as well as CO₂. The acid-amide products are of 2- and 3- carbon chain lengths and contain only carbon, hydrogen, nitrogen, and oxygen.

The half-life of [2,6-¹⁴C]aminopyralid in the irradiated samples was 0.3 days. This value does not reflect adjustments for continuous irradiation, geometry effects of the test tubs or the quantity of light samples received from the xenon lamp. No degradation was observed in the dark controls.

The predicted environmental phototransformation half-life of [2,6-¹⁴C]aminopyralid, derived from the measured half-life in the laboratory in a sterile buffer solution under the xenon arc lamp, was calculated to be 0.6 days for 40° N latitude in summer sunlight.

Results Synopsis

Test medium:	0.01 M acetate buffer at pH 5
Source of irradiation:	xenon lamp
Half-life/DT50 for dark:	No degradation observed in dark controls.
Half-life/DT50 for phototransformation:	0.3 days
Predicted environmental half-life/DT50 for phototransformation:	0.6 days at 40° N latitude in the summer sun

Major transformation products: oxamic acid, malonamic acid, and four or more unidentified acid amides (maximum of 68.8% of applied radioactivity in total at 12 DAT), CO₂ (maximum of 28.4% of applied radioactivity at study termination).

Minor transformation products: three additional minor degradates characterized, but not identified, as acid-amide products containing 2 and/or 3 carbons, hydrogen, nitrogen, and oxygen (maximums of 2.9%, 4.0 %, and 3.3% of the applied amount in replicate samples at 12 days, 8 hours, and 2 days, respectively).

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Study Acceptability: This study is classified as supplemental for an aquatic phototransformation study because not all degradation products over 10% of applied radioactivity were individually identified and quantified in all sampling intervals.

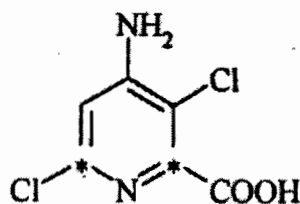
I. MATERIALS AND METHODS

GUIDELINE FOLLOWED: EPA Pesticide Registration Guidelines, Subdivision N, §161-2 and SETAC Part 1 Section 10.1

COMPLIANCE: Good Laboratory Practices standards, 40 CFR Part 160

A. MATERIALS:

1. Test Material [2,6-¹⁴C]XDE-750 (aminopyralid; p. 18; Figure 1, p. 60).



Chemical Structure:

Description: Technical, solid (p.18)

Purity: Analytical purity: N/A
Radiolabelled: Radiochemical purity: 99.0% (p. 60).

Inventory No.: INV 1590.
FA & PC Reference No.: 014001.
SPS Reference No.: F380-135a.
Specific activity: 27.4 mCi/mmole.
Locations of the label: Ring labelled at the C-2 and C-6 positions (Figure 1, p. 60).

Non-radiolabelled: Analytical purity: 99.5% (p. 19; Figure 1, p.60).
ID No.: TSN102298.

Storage conditions of test chemicals: Stored in the freezer in the dark when not in use (p. 19).

Physico-chemical properties of [2,6-¹⁴C]aminopyralid:

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Parameter	Values	Comments
Water solubility		
pH 5	212 g/L	at 20 °C (1)
pH 7	205 g/L	at 20 °C (1)
pH 9	203 g/L	at 20 °C (1)
unbuffered	2.48 g/L	at 20 °C (1)
Vapour pressure	7.14×10^{-11} mm Hg	at 20 °C (2)
UV absorption	See Figure 1	[2,6- ¹⁴ C]aminopyralid was prepared in pH 5 buffer at concentration of 1.51×10^{-5} M using 1% acetonitrile as a co-solvent.
pKa	2.56	at 20 °C (3)
Kow/log Kow	0.201	at 19 °C, unbuffered water(4)
Stability of compound at room temperature, if provided	Stable at least 5 months after receipt	Confirmed by HPLC re-analysis

Data were obtained from p. 19 of the study report.

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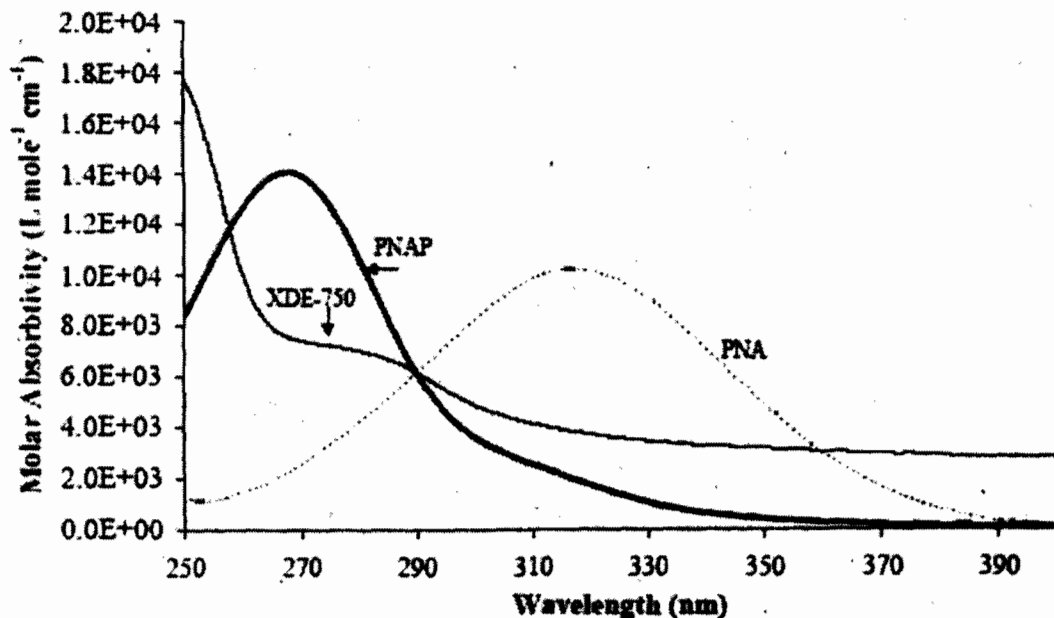


Figure 1. Molar absorptivity of [2,6-¹⁴C]aminopyralid, PNA and PNAP. Data were obtained from p. 20 and Figure 4, p. 64 of the study report.

2) Buffer solution: Buffer solutions were made with Milli-Q water as follows:

Table 1: Description of buffer solutions.

pH	Type of buffer and final molarity	Composition
5	0.01 M acetate	1.38 g sodium acetate trihydrate in 1.0 L water adjusted to pH 5 using glacial acetic acid

Data were obtained from p. 21 and Table 1, p. 48 of the study report.

3) Details of light source: A xenon lamp (Atlas xenon arc light system, Atlas Electric Devices, Company, Chicago, Illinois) was used to irradiate the [2,6-¹⁴C]aminopyralid samples and actinometers. The earth's atmosphere filters radiation below 290 nm. Therefore, the lamp was equipped with a combination of infrared and soda lime filters to help maintain temperature and effectively simulate natural sunlight (Table 2, p. 49). Samples were placed on a revolving turntable so that the light from the lamp was evenly distributed to the samples during exposure. Actinometers were arranged between [2,6-¹⁴C]aminopyralid samples on the turntable. The entire photolysis set-up was kept in a temperature-controlled room at 20 °C. A fan circulated the air in

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the room to help maintain temperature and the lamp was left on continuously (p. 22). However, there was no cooling apparatus in which to put the test vessels.

Table 2: Artificial light source.

Property	Details
Nature of light source	Xenon Lamp
Emission wavelength spectrum	Measured using a radiometer. These intensity values were similar to values from the Federal Register (6) values at 40°N latitude in the summer sun (see Figure 2).
Light intensity	429 W m ⁻²
Filters used	Inner filter: Quartz with infra-red coating Outer filter: soda lime wavelengths filtered: < 290 nm
Relationship to natural sunlight	Determined using both chemical actinometry and radiometer data.

Data were obtained from p. 22 and Table 2, p. 49 of the study report.

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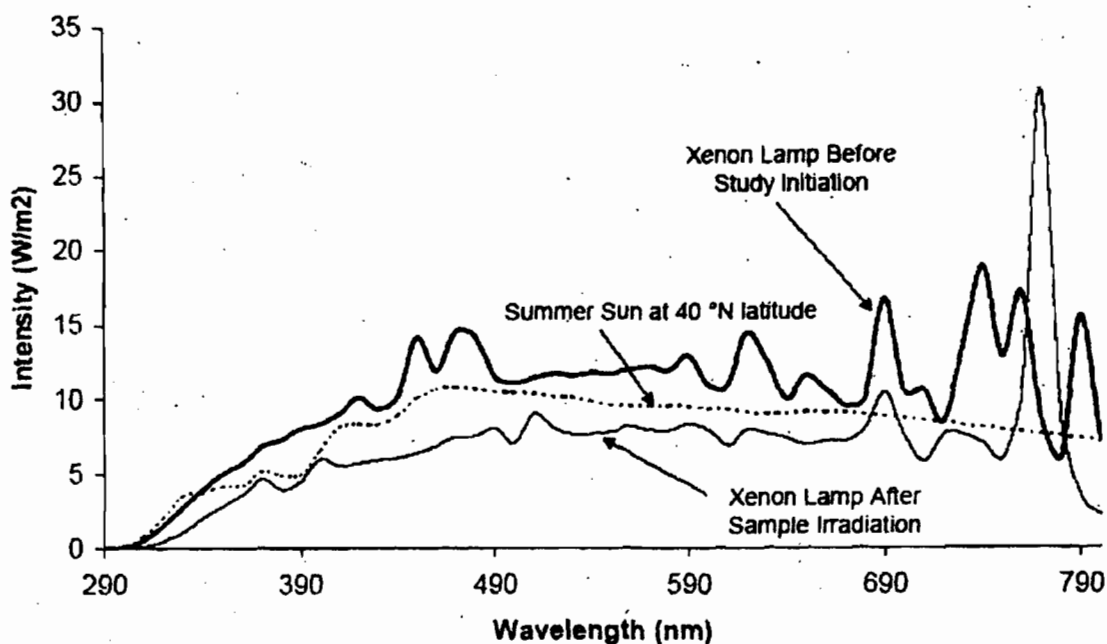
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Figure 2. Emission spectra of xenon light source vs summer sunlight. Data were obtained from Figure 6, p. 66 of the study report.

B. EXPERIMENTAL CONDITIONS:

Light Source Characterization



1) Preliminary Study: N/A

2) Experimental Conditions:

The experimental design is detailed in Table 3. To determine kinetics of irradiated samples, a bulk solution containing 0.2 mg/L [2,6-¹⁴C]aminopyralid was prepared in pH 5 buffer. Aliquots (9-mL) were added to fifteen 10-mL quartz test tubes. PNA actinometer samples were prepared by adding 9 mL of actinometer solution to five 10-mL quartz test tubes and 5 mL into one amber vial for the 0-time sample. Duplicate samples and single actinometers were irradiated for 1, 2, 4, 6 and 8 hours. No traps were used to trap volatile photoproducts (p. 23).

Additional samples were prepared to assess the rise and decline of degradates and stability of [2,6-¹⁴C]aminopyralid in the dark. A bulk solution was prepared containing 0.2 mg/L [2,6-¹⁴C]aminopyralid in pH 5 buffer. Aliquots were added to twenty 10-mL quartz test tubes and fourteen 20-mL amber vials. PNAP actinometer samples were prepared by adding 9 mL to twelve 10-mL quartz test tubes and 6 mL to fourteen 20-mL amber vials. Duplicate irradiated

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samples and dark controls for [2,6-¹⁴C]aminopyralid and actinometers were collected at 2, 6, 12, and 15 DAT (p. 23).

Additional samples were prepared to identify degradates. These samples were prepared in pH 5 buffer using ¹⁴C-radiolabeled and non-radiolabeled [2,6-¹⁴C]aminopyralid for a final concentration of 30 µg/mL [2,6-¹⁴C]aminopyralid. This solution was dispensed into two 50-mL quartz vials. The samples contained 1.0% acetonitrile co-solvent and were irradiated continuously for 3 days (p. 27).

Table 3: Experimental parameters

Parameters		Details
Duration of the study		0 to 8 hours for kinetics sample set ^a , 0 to 15 DAT for degradate sample set ^b , and 3 DAT for degradate identification
Test concentrations nominal: measured:		0.2 mg/l, 30 µg/mL 0.2 mg/l, 30 µg/mL
Dark controls used		yes, for degradate sample set
Replication	Dark:	duplicates for samples 2 to 15 DAT
	Irradiated:	duplicates for all sampling times
Preparation of the test medium:	volume used/treatment:	9 mL for kinetics and degradate sample sets; 50 mL for degradate identification
	method of sterilization:	autoclave
	co-solvent, if any:	≤ 0.05% acetonitrile in buffer for kinetics and degradate sample sets; 1.0% acetonitrile in buffer for degradate identification
Test material application	volume of application solutions used/treatment	0.1 mL in 200 mL pH 5 buffer for kinetics sample set 0.17 mL in 300 mL pH 5 buffer for degradate sample set Not reported for degradate identification
	Application method	positive displacement pipette

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Test apparatus (Type/Material/Volume)	constant temperature room equipped with xenon lamp; experimental vials were 10-mL quartz vials (irradiated samples) or 20-mL amber glass (dark controls) both with Teflon-lined screw caps or mininert valves
Details of traps for volatile compounds, if any	0.2 M NaOH traps
Is there any indication of the test material adsorbing to the walls of the test apparatus?	Not stated
Experimental Conditions Temperature: Duration of light/darkness:	20 ± 1 °C 15 DAT continuous irradiation equal to 38 days in the summer sun at 40° N latitude based on PNAP actinometry data
Other details	none

Data were obtained from p. 23, 25, 27, and Table 3, p. 50 of the study report.

^a Irradiated from September 27, 2002, 8:30 a.m. to September 27, 2002, 4:30 p.m.

^b Irradiated from August 14, 2002, 10:20 a.m. to August 29, 2002, 10:35 a.m

3) **Supplementary experiments:** N/A

4) **Sampling:**

Samples were analyzed 0, 1, 2, 4, 6, and 8 hours and 2, 6, 12, and 15 days after treatment (DAT). Duplicate [2,6-¹⁴C]aminopyralid samples were removed from the photolysis chamber and the incubator at each sampling time unless otherwise noted. Single or duplicate irradiated actinometer samples were collected at the same sampling times. Dark control [2,6-¹⁴C]aminopyralid and actinometer samples were not collected at 1, 2, 4, 6, and 8 hours. Sample pH and sterility were measured at each time point for each [2,6-¹⁴C]aminopyralid sample (p. 26). Sampling details are listed in Table 4.

Table 4: Sampling details

Observations	Details
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Sampling intervals for the parent/transformation products		0, 1, 2, 4, 6 to 8 hours for kinetics sample set and 0, 2, 6, 12 and 15 DAT for degradate sample set
Sampling method for all [2,6- ¹⁴ C]aminopyralid samples		Remove 500 uL aliquot for sterility check. Connect to purge and trap system if necessary. Count triplicate aliquots of aqueous samples to determine recoveries. Transfer sample to labeled amber vial and check pH. Sample analyzed by reverse phase HPLC.
Sampling method for all actinometer samples		Transfer sample to labeled amber vial and store in freezer. Sample was analyzed by reverse phase HPLC following study completion.
Method of sampling CO ₂ and volatile organics		Samples at 2 and 6 DAT were connected to a purge and trap system. Samples at 12 and 15 DAT contained 0.2 M NaOH traps.
Sampling intervals/times for:	sterility check:	Checked at each sample by transferring an aliquot to soy broth and checking for microbial growth.
	pH measurement:	Checked at each sample time using pH meter.
Sample storage before analysis, if any		Freezer (for less than one day, as samples analyzed the day of sacrifice)
Other observation, if any (eg: precipitation, color change etc.)		None

Data were obtained from p. 26 and Table 4, p. 51 of the study report.

C. ANALYTICAL METHODS:

Extraction/Concentration Methods

No extraction or concentration methods were required for the analysis of ¹⁴C in solution. An aliquot of each [2,6-¹⁴C]aminopyralid sample was counted and analyzed directly by reverse phase HPLC.

One of the identification samples was concentrated using a Turbovap. This concentrate was re-dissolved in water and analyzed using reverse phase HPLC Method 3. Several injections were made and each time the eluent was collected in a set of 7-mL LSC vials. Aliquots of the eluent were counted and fractions collected at 5, 6, and 7 minutes were combined. This polar peak was concentrated to dryness, re-dissolved in 1 mL of 50/50 acetonitrile/water and was further

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analyzed using reverse phase HPLC Method 4 and HILIC (hydrophilic interaction chromatography) HPLC Method 2.

The other identification sample was concentrated to dryness using a Turbovap and re-dissolved in water. This sample was analyzed using reverse phase HPLC Method 5. An aliquot of the aqueous solution was diluted using acetonitrile resulting in a 50/50 acetonitrile/water solution. This sample was analyzed using HILIC (hydrophilic interaction chromatography) HPLC Method 2 (p. 28).

Maintenance and Collection of Volatile Traps

At 2, 6, 12, and 15 DAT volatile traps were collected. Triplicate aliquots of the 0.2 M NaOH trapping solutions were counted by LSC to determine the presence of volatiles. The presence of $^{14}\text{CO}_2$ was confirmed by precipitation with BaCl_2 and CaCO_3 (p. 27).

Radiocarbon Determination Procedures

Radioactive material in solution was quantified by a liquid scintillation spectrometer. Reference ^{14}C standards obtained from the Packard Instrument Co. were used to verify the performance of the counter frequently, typically each day samples were analyzed. ScintiSafe Plus scintillation cocktail was added to each sample before counting. Samples were generally counted for 3 minutes; however samples that were expected to have low amounts of radioactivity were often counted for 10 minutes or longer (p. 28).

Chromatographic and Spectroscopic Procedures

Reverse Phase HPLC Method 1- [2,6- ^{14}C]aminopyralid Degradation

The gradient used for the reverse phase HPLC method used for sample analysis is presented in Table 5. An Aquasil 4.6 x 250 mm column by Keystone Scientific and a Hewlett Packard (HP) 1050 HPLC were used. The flow-rate was 1.0 mL/min. Fractions (0.5- or 1.0-minute) were collected for irradiated samples and the collected fractions were counted by LSC and used to generate reconstructed radiochromatograms. A direct spike of each sample analyzed by HPLC was compared to the sum of the radioactivity eluted from the column and used to determine chromatographic recovery. A UV detector set at 270 nm wavelength was used to determine the retention times of non-radiolabeled standards. A RAM flow-through detector was used in conjunction with the fraction collector to characterize the radioactivity in solution (p. 28-29).

Reverse Phase HPLC Method 2- Test Substance Purity

An isocratic reverse phase HPLC method was used to assess purity of the test material before study initiation. A 4.6 x 250 mm Inertsil ODS column and HP 1050 HPLC were used. The isocratic mobile phase was 90%/10%/0.5% water:acetonitrile:TFA. The flow-rate was 1.0 mL/min and the UV absorbance was set at 270 nm. Fractions (0.5-minute/1.0 minute) were collected and the collected fractions were counted by LSC and used to generate reconstructed radiochromatograms (p. 29).

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Reverse Phase HPLC Method 3- Isolation of transformation products

An isocratic reverse phase HPLC method was used to isolate the polar material for degradate identification work. A 4.6 x 250 mm Aquasil C18 column and HP 1050 HPLC were used. The isocratic mobile phase was 75%/25%/0.5% water:acetonitrile:TFA. The flow-rate was 1.0 mL/min and the UV absorbance was set at 270 nm. Fractions (0.5-minute/1.0 minute) were collected and the collected fractions were counted by LSC and used to generate reconstructed radiochromatograms (p. 29).

Reverse Phase HPLC Method 4- Identification of transformation products

An isocratic reverse phase HPLC method was used for co-chromatography with the HPLC reference standards. An aliquot, typically 50 µL, was analyzed using an HP 1050 HPLC equipped with a Phenomenex Synergi Hydro RP C18 column (4.6 x 250 mm). The mobile phase was 100% water containing 0.5% TFA. The UV wavelength was 225 nm and the flow rate was 0.7 mL/min (p. 29).

Reverse Phase HPLC Method 5- Identification of transformation products

An isocratic reverse phase HPLC method was used for co-chromatography with the HPLC reference standards. An aliquot, typically 100 µL, was analyzed using an HP 1050 HPLC equipped with a Phenomenex Synergi Polar RP C18 column (4.6 x 250 mm). The mobile phase was 100% water containing 0.5% TFA. The UV wavelength was 230 nm and the flow rate was 0.7 mL/min (p. 30).

HILIC HPLC Method 1. Rise and Decline of [2,6-¹⁴C]aminopyralid Photo transformation products

Reverse Phase HPLC Method 1 used for kinetics determination did not provide adequate retention or separation of [2,6-¹⁴C]aminopyralid degradates. Therefore, the 2, 6, and 15-DAT samples were further analyzed using hydrophilic interaction chromatography (HILIC). A 4.6 mm x 200 mm polyhydroxyethyl Aspartamide™ column by PolyLC inc. and HP 1050 HPLC were used. The HPLC gradient conditions are listed in Table 5. The flow-rate was 1.0 mL/minute and the UV absorbance was set at 270 nm. The injection volume was typically 100 µL. The column was re-equilibrated at initial solvent conditions for approximately 10 to 20 minutes between samples (p. 30).

HILIC HPLC Method 2- Identification of transformation products

HILIC HPLC Method 2 was used as a confirmatory co-chromatography method. A 4.6 mm x 200 mm polyhydroxyethyl Aspartamide™ column by PolyLC inc. and HP 1050 HPLC were used. The HPLC gradient conditions are listed in Table 5. The flow-rate was 1.0 mL/minute and the UV absorbance was set at 245 or 270 nm. The injection volume was typically 100 µL. The column was reequilibrated at initial solvent conditions for approximately 10 to 20 minutes between samples (p. 30).

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HPLC Analysis of PNA/pyridine Actinometer Solutions

The PNA/pyridine actinometer samples were analyzed by reverse phase HPLC. A 4.6 x 250 mm Inertsil ODS column was used. The isocratic mobile phase was 60% water/40% acetonitrile. The flow-rate was 1.5 mL/min and the UV absorbance was set at 288 nm. The stop-time was 15 minutes and injection volume was typically 150 µL. A standard curve of PNA was run before and after analysis of samples (p. 31).

HPLC Analysis of PNAP/pyridine Actinometer Solutions

The PNAP/pyridine actinometer samples were analyzed by reverse phase HPLC. A 4.6 x 250 mm Spherisorb ODS column was used. The isocratic mobile phase was 50% water/50% acetonitrile. The flow-rate was 1.5 mL/min and the UV absorbance was set at 288 nm. The stop-time was 10 minutes and injection volume was typically 100 µL. A standard curve of PNAP was run before and after analysis of samples (p. 31).

Identification and Characterization of transformation products

Identification of transformation products was performed by comparison of chromatographic retention times of commercially purchased reference standards (p. 31).

Quantitation of Components by HPLC

The percentage of applied ¹⁴C present in each peak in an HPLC chromatogram was calculated by multiplying the percentage of radioactivity present in the peak in the radiochromatogram by the total percentage of radioactivity in the aqueous phase. For example, if a peak in a chromatogram contained 91.2% [2,6-¹⁴C]aminopyralid, and the aqueous phase comprised 81% AR, then that peak would contain 73.9% AR. All HPLC peaks exceeding 2% of the HPLC peak area were monitored. However, multiple HPLC peak areas less than 2% may have been present at each sampling time. These peaks were referred to as non-identified radiocarbon. The non-identified radiocarbon was determined based on the following equation: % AR in the AQ layer - (sum of %AR in the HPLC Peak areas tracked) (p. 32-33).

LOD/LOQ for [2,6-¹⁴C]aminopyralid

Using the method of Currie (7), the limits of detection and quantitation were calculated to be 13 dpm and 57 dpm above background, respectively (pp. 39-40).

Table 5. Gradient HPLC conditions for analysis of [2,6-¹⁴C]aminopyralid photolysis samples

Time (minutes)	Water 0.5% Trifluoroacetic acid	Acetonitrile 0.5% Trifluoroacetic acid
Reverse phase HPLC Method 1		
0	100%	0%
5	100%	0%

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25	90%	10%
35	90%	10%
Time (minutes)	100 mmol Ammonium acetate in water	Acetonitrile
HILIC HPLC Method 1		
0	0%	100%
10	0%	100%
40	70%	30%
55	70%	30%
HILIC HPLC Method 2		
0	0%	100%
30	80%	20%
35	100%	0%
40	100%	0%

Data were obtained from Table 5, p. 52 of the study report.

II. RESULTS AND DISCUSSION:

A. TEST CONDITIONS: The pH, sterility, temperature and other experimental conditions were reported to be maintained throughout the study (p. 40); no supporting temperature data were provided.

Actinometer Data: PNA actinometer data for the samples irradiated 0-8 hours indicates that the xenon lamp was approximately 1.58 times more intense than the summer sun at 40° N latitude for this sample set. This data was used to calculate lamp intensity values, which were used to calculate the quantum yield of [2,6-¹⁴C]aminopyralid. Once the [2,6-¹⁴C]aminopyralid quantum yield was calculated, the predicted half-lives at various geographies and seasons was calculated (p. 42-43).

PNAP actinometer data for the samples irradiated 2 – 15 days after treatment (DAT) indicates that 1 DAT of exposure in the xenon lamp is equivalent to 2.54 days in the summer sun. This data indicates that 15 days of continuous exposure is equivalent to 38 days in the summer sun at 40° N latitude for this sample set (p. 42-43).

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B. MASS BALANCE: Material balance was between 90-110% applied radiocarbon for all samples except the 6-DAT irradiated sample (84.7%). However, the photodegradation rate of [2,6-¹⁴C]aminopyralid was based on sampling times between 0 and 8 hours. Therefore, all samples used for kinetics calculations had sufficient mass balance. The mass balance was $97.8 \pm 2.6\%$ and $99.4 \pm 0.7\%$ of the applied amount for the kinetics sample set in the dark and irradiated samples, respectively. The mass balance was $94.0 \pm 5.9\%$ and $100.0 \pm 0.0\%$ of the applied amount for the degradate sample set in the dark and irradiated samples, respectively (p. 41 and Table 12, p. 59).

Table 6: Phototransformation of [2,6-¹⁴C]aminopyralid in pH 5 buffer, expressed as percentage of the applied radioactivity (mean \pm s.d.).

Compounds		Sampling times									
		0	1 hr	2 hr	4 hr	6 hr	8 hr	2 dat ^{a,c}	6 dat ^c	12 dat ^c	15 dat
Parent compound	irradiated	N/A	89.0 ± 4.8	78.4 ± 3.4	69.5 ± 1.9	59.9 ± 2.7	43.3 ± 1.9	nd	nd	nd	nd
	dark	99.4 ± 0.7	N/A	N/A	N/A	N/A	N/A	100	100.0 ± 0.0	100.0 ± 0.0	100.0 ± 0.0
Transformation product 1 (8 min.) ^b	irradiated	N/A	7.3 ± 0.9	12.9 ± 1.1	25.4 ± 1.2	33.3 ± 0.8	42.0 ± 3.1	61.5	58.6	68.8	65.7 ± 1.9
	dark	nd	N/A	N/A	N/A	N/A	N/A	nd	nd	nd	nd
Transformation product 2 (11.5 min.)	irradiated	N/A	nd	0.9 $\pm 1.3^d$	nd	nd	nd	1.9	1.2	2.9	1.4 $\pm 1.9^d$
	dark	nd	N/A	N/A	N/A	N/A	N/A	nd	nd	nd	nd
Transformation product 3 (13 min.)	irradiated	N/A	nd	nd	nd	nd	2.0 $\pm 2.8^d$	nd	nd	nd	nd
	dark	nd	N/A	N/A	N/A	N/A	N/A	nd	nd	nd	nd
Transformation product 4 (16 min.)	irradiated	N/A	nd	0.9 $\pm 1.3^d$	2.1 ± 1.1	1.6 $\pm 2.3^d$	2.4 ± 0.3	3.3	nd	nd	nd
	dark	nd	N/A	N/A	N/A	N/A	N/A	nd	nd	nd	nd
Unidentified product(s)	irradiated	N/A	1 ± 0.0	4.1 ± 1.3	3.0 ± 0.2	3.0 ± 0.4	7.2 ± 2.1	2.9	2.6	0.9	0.8
	dark	nd	N/A	N/A	N/A	N/A	N/A	nd	nd	nd	nd
CO ₂	irradiated	N/A	N/A	N/A	N/A	N/A	N/A	21.9	19.9	27.8	28.4
	dark	nd	N/A	N/A	N/A	N/A	N/A	nd	nd	nd	nd

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VOC	irradiated	N/A	N/A	N/A	N/A	N/A	N/A	1.1	2.4	N/A	N/A
	dark	nd	N/A	N/A	N/A	N/A	N/A	nd	nd	nd	nd
Total % recovery	irradiated	N/A	97.3 ±5.7	97.2 ±3.3	99.9 ±1.8	97.7 ±0.8	96.9 ±0.2	92.6	84.7	100	96.1 ±1.6
	dark	99.4 +0.7	N/A	N/A	N/A	N/A	N/A	100	100.0 +0.0	100.0 +0.0	100.0 +0.0

Data were obtained from Table 12, p. 59 of the study report.

"N/A" stands for Not Applicable, when an interval was not sampled.

"nd" stands for not detected, i.e., below the LOD which was $\leq 0.3\%$ of applied radioactivity.

* "dat" stands for days after treatment. These are actual sampling times, which are not corrected for continuous irradiation or geometry effects.

^b Additional chromatography indicated this peak consists of multiple degradates, including oxamic acid, malonamic acid, and minor acid amides (all of which would be 2 or 3 carbons in length).

^c Only one replicate in data set for irradiated (2, 6, 12 dat) and/or dark samples (2 dat).

^d One replicate was below minimum detection limit; its value was assumed zero for the purpose of average and standard deviation calculation.

C. TRANSFORMATION OF PARENT COMPOUND: At study termination, 100.0% of the applied radioactivity remained as the parent in the dark samples. No transformation occurred in the dark samples. Volatiles in the dark samples were not trapped. In the irradiated samples, the concentration of the parent compound decreased from 96.8% at day 0 to 0.0% of the applied amount at 2 days post-treatment (Table 12, p. 59).

TRANSFORMATION PRODUCTS: The major transformation products detected in the irradiated samples were not all identified nor separately quantified. Two transformation products identified in the irradiated samples were oxamic acid (CAS #471-47-6) and malonamic acid (CAS # 2345-56-4), which, together with four or more unidentified acid amides (2 or 3 carbons in length), in total reached a maximum concentration of 68.8% of applied radioactivity by 12 days post-treatment. These six or more transformation products together decreased to 65.7% of applied radioactivity at study termination. At study termination, in the irradiated samples, evolved CO₂ amounted to 28.4% of the applied amount. Volatile organics were not detected at study termination, despite having reached a maximum of 2.4 % of the applied amount at 6 days post-treatment. The total unidentified radioactivity was a maximum of 0.0% and 7.2% of the applied amount at 8 hours post treatment in the dark and irradiated samples, respectively (Table 12, p.59).

The minor transformation products were not identified, but were detected at maximums of 2.9%, 4.0 %, and 3.3% of the applied amount in replicate samples at 12 days, 8 hours, and 2 days, respectively. All products are formed by phototransformation only (Table 12, p. 59).

PATHWAY: [2,6-¹⁴C]Aminopyralid photodegrades via de-chlorination and ring cleavage to form unidentified acid-amides, oxamic acid and malonamic acid, as well as CO₂. The acid-amide

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products are of 2- and 3- carbon chain lengths and contain only carbon, hydrogen, nitrogen, and oxygen.

Table 7: Chemical names and CAS numbers for the transformation products of [2,6-¹⁴C]aminopyralid

Applicant's Code Name	CAS Number	CAS and/or IUPAC Chemical Name(s)	Chemical formula	Molecular weight (g)
DOW	471-47-6	oxamic acid	C ₂ H ₃ NO ₃	89.05
DOW	2345-56-4	malonamic acid	C ₃ H ₃ NO ₃	104.05

Data were obtained from Figure 1, p. 60 of the study report.

HALF-LIFE: The half-life of [2,6-¹⁴C]aminopyralid in the irradiated samples, calculated using first-order linear regression, was 0.3 days. This value does not reflect adjustments for continuous irradiation, geometry effects of the test tubs or the quantity of light samples received from the xenon lamp. No degradation was observed in the dark controls.

Table 8. Half-lives/DT50

Test system	First order half-life			DT50 (days)	DT90 (days)
	half-life (days)	Regression equation	R ²		
Dark	stable	N/A	N/A	N/A	N/A
Irradiated	0.3	y = 98.486e-2.2871x	0.9476	0.3	1.0

The predicted environmental phototransformation half-life of [2,6-¹⁴C]aminopyralid, derived from the measured half-life in the laboratory in a sterile buffer solution under the xenon arc lamp, was calculated to be 0.6 days for 40° N latitude in summer sunlight. The expected half-life value calculations are based on the quantum yield of [2,6-¹⁴C]aminopyralid and the sunlight intensity values reported by Leifer (8).

D. SUPPLEMENTARY EXPERIMENT-RESULTS: N/A

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III. STUDY DEFICIENCIES:

All degradation products over 10% of applied radioactivity were not individually identified and quantified in all sampling intervals. The only two potentially major transformation products identified, but not quantified, in the irradiated samples were oxamic acid (CAS #471-47-6) and malonic acid (CAS # 2345-56-4). These two products plus at least four more acid amides (2 or 3 carbons in length) together reached a maximum concentration of 68.8% of applied radioactivity by 12 days post-treatment. But this 68.8% was never completely quantitatively separated into its components (See Reviewer's Comment 2).

IV. REVIEWER'S COMMENTS:

1. These results indicate that [2,6-¹⁴C]aminopyralid is susceptible to phototransformation at environmentally relevant wavelengths of light.
2. Despite the use of six different chromatographic methods, not all components of the major 8-minute peak were identified nor adequately quantified at all sampling intervals. Figures 11, 12, and 13 (p. 71-73) show the use of HILIC Chromatography Method 1 to attempt to identify components of the 8-minute Reverse Phase HPLC Method 1 peak at 2, 6, and 15 days post-treatment. However, HILIC HPLC results do not completely separate and quantify the 61.5%, 58.6% and 65.7% of applied radioactivity reported in each interval, respectively, for Reverse Phase HPLC Method 1. This means that the potential major degradates were not adequately characterized. The study does not report how much oxamic acid and malonic acid were present individually, nor it is evident if any other components of the 8-minute peak were present as major degradates. The study author identifies the two components which exceeded 10% of applied radioactivity at 15 days post-treatment, but does not identify all components exceeding 10% of applied radioactivity at 2 and 6 days post-treatment. All degradates over 10% should be identified at all sampling intervals, not just at study termination.
3. The study temperature was reported to be 20° (pp. 21, 26) but more detailed information was not provided. It is preferred that minimum, maximum, and average temperatures be reported. Any significant deviations from the average and their duration should be noted. Also, there was no experimental apparatus used to maintain the solution temperature, such as a temperature-controlled water bath or cooling jackets.
4. There were no [2,6-¹⁴C]aminopyralid concentrations reported for irradiated samples at time zero (only total aqueous phase at time zero).
5. Percent applied radioactivity reported in Table 10 and in Table 12 for dark controls are not consistent.

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Time (days)	Total (Table 10) (% applied radioactivity)	Total (Table 12) (% applied radioactivity)
0	99.0	100.0
0	92.2	100.0
0	94.3	99.2
0	101.6	98.5
2	95.1	100.0
6	97.8	100.0
6	98.6	100.0
12	99.3	100.0
12	99.5	100.0
15	99.5	100.0
15	99.2	100.0

6. There were no replicates for irradiated samples for 2, 6, and 12 days post-treatment.
7. HILIC chromatography method 1 degrade identification is not reported for 12 days post-treatment, the sampling time at which the maximum degrade concentration (68.8%) was recorded.
8. There were no dark controls measured concurrently with the 1, 2, 4, 6, and 8 hour samples, which were used for kinetics calculations, although there was no degradation in the dark controls of the second low dose sample set at 15 days post-treatment.
9. References 7 and 8 were incomplete.

V. REFERENCES:

1. Nelson R. 2002. Determination of the water solubility of XDE-750. FOR01015, unpublished report of Dow AgroSciences LLC.
2. Griffin K.A. 2001. Vapor pressure of XR-750 by Knudsen-Effusion weight loss method. AS-PR 2001-002, unpublished report of The Dow Chemical Company.

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3. Cathie C. 2001. Determination of dissociation constant of XR-750 using UV-visible spectrophotometry. 01-822-AG, unpublished report of Dow AgroSciences LLC.
4. Madsen S. 2003. Determination of the n-octanol/water partition coefficient (Shake Flask Method) of XDE-750", FOR01009, unpublished report of Dow AgroSciences LLC.
5. Roulin S. P. 2001. Determination of the organic solubility of XR-750", 01003/DA, unpublished report of Dow AgroSciences LLC.
6. Federal Register, Part II EPA 40 CFR Parts 796, 797, and 798 Toxic Substances Control Act Test Guidelines; Final Rules, Friday September 27, 1985, p. 39318.
7. Currie L. A. Limits for qualitative detection and quantitative determination-application to radiochemistry.
8. Leifer A. 1998. The Kinetics of Environmental Aquatic Photochemistry: American Chemical Society.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235727
 Guideline No: 161-2

Table 6 Material balance of [2,6-C14]aminopyralid in 5 pH buffer (dark samples),
 expressed as percentage of the applied radioactivity

Time (hours)	Time (days)	Aqueous	CO2	VOC	Total		
0	0	99.0	N/S	N/S	99.0		
0	0	92.2	N/S	N/S	92.2		
0	0	94.3	N/S	N/S	94.3		
0	0	101.6	N/S	N/S	101.6		
AVG	AVG	96.8	N/A	N/A	96.8	Kinetics:	
STDEV	STDEV	4.3	N/A	N/A	4.3	AVR	96.8
						STDEV	4.3
48	2	95.1	N/S	N/S	95.1		
144	6	97.8	N/S	N/S	97.8		
144	6	98.6	N/S	N/S	98.6		
AVG	AVG	98.2	N/A	N/A	98.2		
STDEV	STDEV	0.6	N/A	N/A	0.6		
288	12	99.3	N/S	N/S	99.3		
288	12	99.5	N/S	N/S	99.5		
AVG	AVG	99.4	N/A	N/A	99.4		
STDEV	STDEV	0.1	N/A	N/A	0.1		
360	15	99.5	N/S	N/S	99.5		
360	15	99.2	N/S	N/S	99.2	Degradate:	
AVG	AVG	99.4	N/A	N/A	99.4	AVR	98.4
STDEV	STDEV	0.2	N/A	N/A	0.2	STDEV	1.6

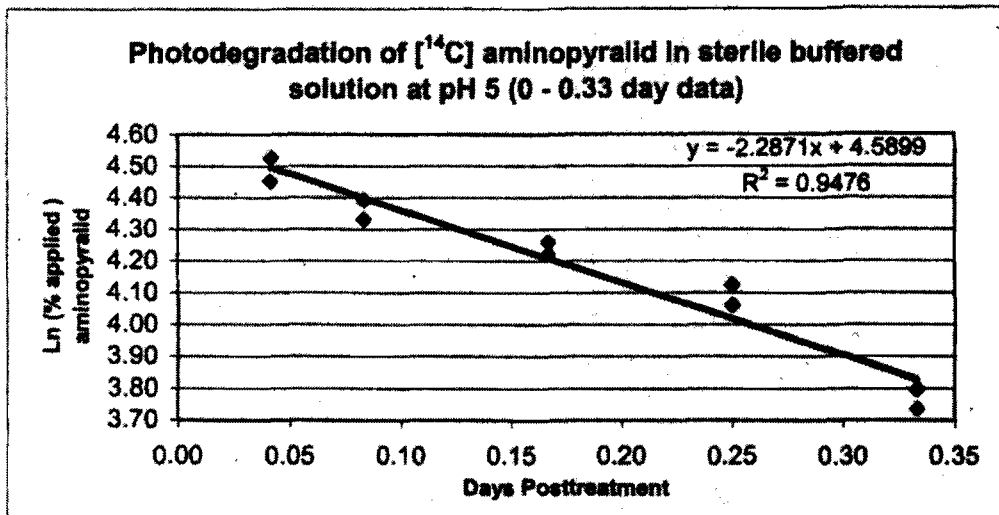
Data were obtained from Table 10, pp. 57 of the study report.
 N/A= Not Applicable
 N/S= Not Sampled

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235727
 Guideline No: 161-2

Days	Hours	% applied	Ln (% applied)
0.04	1	92.4	4.53
0.04	1	85.6	4.45
0.08	2	80.8	4.39
0.08	2	76.0	4.33
0.17	4	68.1	4.22
0.17	4	70.8	4.26
0.25	6	58.0	4.06
0.25	6	61.8	4.12
0.33	8	44.6	3.80
0.33	8	41.9	3.74
2	48	<MDL	#VALUE!
6	144	<MDL	#VALUE!
12	288	<MDL	#VALUE!
15	360	<MDL	#VALUE!
15	360	<MDL	#VALUE!

First order linear Half life = 0.3 days



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EPA MRID Number 46235728



Data Requirement: PMRA DATA CODE: 8.2.3.3.1
EPA DP Barcode: D301682
OECD Data Point: IIA 7.1.3
US EPA Guideline: Subdivision N, §161-3

Test material:

Common name: Aminopyralid.

Chemical name:

IUPAC: 4-amino-3,6-dichloropyridine-2-carboxylic acid.

CAS name: 4-amino-3,6-dichloro-2-pyridinecarboxylic acid.

CAS No: 150114-71-9.

Synonyms: XDE-750, DE-750.

SMILES string: ClC(=O)OC1=CC=C(N)C(Cl)=C1

Primary Reviewer: Anne Gosselin (#1615), PMRA

Signature:

Date: 2004-11-18

Secondary Reviewer: Hemendra Mulye, PhD (#213), PMRA

Signature:

Date: 2004-11-29

Secondary Reviewer: Roxolana Rashuba, EPA/EFED/ERB4

Signature:

Date: 2005-02-14

Company Code: DOW

Active Code: AMD

Use Site Categories: 13 (terrestrial feed crops), 14 (terrestrial food crops) and 16 (industrial and domestic vegetation control non-food sites)

EPA PC Code: 005100

CITATION: Rutherford L.A. 2004. Photodegradation of XDE-750 on Soil. Regulatory Laboratories - Indianapolis Lab (Indianapolis, Indiana), Dow AgroSciences LLC, Study number 020080, January 27 2004, 80 pages. Unpublished.



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Data Evaluation Report on the phototransformation of aminopyralid (XDE-750) on soil

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EPA MRID Number 46235728

EXECUTIVE SUMMARY:

The phototransformation of [2,6-¹⁴C]-labelled 4-amino-3,6-dichloropyridine-2- carboxylic acid (aminopyralid; XDE-750) was studied on a silt loam soil (pH 7.7, organic carbon 1.0%) from Germany at 5.24 mg a.i./kg soil for 44 days (the equivalent of 28 days of summer sunlight at 40° N latitude) at 25 °C and 75% of 1/3 bar moisture. The experiment was conducted in accordance with the EPA Pesticide Registration Guidelines, Subdivision N, §161-3, and SETAC-Europe Procedures for Assessing the Environmental Fate and Ecotoxicity of Pesticides, Section 2.0 guidelines, and in compliance with the Good Laboratory Practices standards, 40 CFR Part 160. [2,6-¹⁴C]Aminopyralid was applied in water on the soil surface by positive displacement pipette. The treated samples were irradiated by intermittent irradiation using a 6500 W xenon arc lamp, with an inner borosilicate filter and an outer Soda Lime filter. The intermittent irradiation consisted of a 12 hour irradiation/12 hour dark cycle to imitate a natural photoperiod. Irradiated test vessels were connected to traps containing ascarite for the collection of CO₂ and acidic volatiles. Dark test vessels were not connected to volatile collection traps. Samples were taken at 0, 2, 7, 14, 26, 35, and 44 days after treatment (DAT) for the determination of the parent compound and transformation products. The soil samples were extracted with 90:10 acetone:1.0 N HCl and the [2,6-¹⁴C]aminopyralid residues were analyzed by HPLC. There were no transformation products present at greater than 10% of applied, therefore, identification of transformation products was not attempted.

A PNAP/pyridine (*p*-nitroacetophenone/pyridine) chemical actinometer solution was used to quantitate the amount of light that the samples received. Based on the PNAP/pyr actinometer data, 44 DAT of irradiation was equivalent to 28 days of irradiation in the summer sun at 40° N latitude.

The mean mass balance was 100.1 ± 4.4 % and 93.8 ± 7.3 % in the dark and irradiated samples, respectively. The material balance in irradiated samples was 105.2% of the applied at day 0, decreased to 97.9-98.0% of the applied at days 2-7, and decreased to 85.2% of the applied by the end of the study. In dark samples, the material balance was also 105.2% of the applied at day 0, and varied from 96.5% to 103.7% of the applied for the remainder of the study. At test termination, 92.5% of the applied ¹⁴C remained as the parent in the dark samples. The remaining radioactivity was present as non-extractable residues (3.8% of applied at study termination), unidentified radioactivity (0.9% at study termination), and possibly untrapped CO₂ or material losses.

In the irradiated samples, concentration of the parent compound decreased from 104.2% of the applied at day 0 to 79.8-81.9% of the applied at days 14-26, and to 69.3 % of the applied amount at test termination. There were no major transformation products formed in the irradiated samples. The minor transformation product or products formed in the irradiated samples at a maximum of 4.6% of the applied amount on the 44th day of incubation. Since the transformation product or products formed in the irradiated samples at less than 5% of applied, no conclusive

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identification was attempted. In irradiated samples, at the end of the study, the evolved CO₂ and other volatiles together amounted to a total of 3.4% of the applied radioactivity.

Extractable [¹⁴C] residues decreased from 104.6% of the applied amount at day 0 to 93.4% and 73.9% of the applied amount at termination in the dark and irradiated samples, respectively. In the irradiated samples, non-extractable [¹⁴C] residues increased from 0.6% of the applied amount at day 0 to 8.0% of the applied at study termination. Non-extractable residues in the dark samples were 0.6% of the applied amount at day 0, and 3.8% of the applied amount at test termination. [2,6-¹⁴C]Aminopyralid photodegraded into non-extractable residues and volatiles.

The half-lives of [2,6-¹⁴C]aminopyralid in the dark and irradiated samples were 364.8 and 93.7 days, respectively. The half-life for phototransformation (the difference between irradiated and dark samples, calculated by subtracting rate constants) was 126.0 days. Empirical DT50 values should not be reported, as aminopyralid had not degraded 50% by the termination of the study.

The predicted environmental phototransformation half-life of [2,6-¹⁴C]aminopyralid, derived from the measured half-life in the laboratory under the xenon arc lamp, was calculated to be 72.2 days for 40° N latitude in summer sunlight.

Results Synopsis

Soil type: Parabraun Erde, silt loam

Source of irradiation: xenon lamp

Half-life for dark: 364.8 days

Half-life for irradiated: 93.7 days

Half-life for phototransformation: 126.0 days

Predicted environmental half-life for phototransformation: 72.2 days

Major transformation products: None.

Minor transformation products: Non-extractable residues and volatile material.

Study Acceptability: This study is classified supplemental for a study on phototransformation on soil because of loss in material balance of the irradiated samples and concurrent loss and variability in material balance in the dark samples.

I. MATERIALS AND METHODS

GUIDELINE FOLLOWED:

EPA Pesticide Registration Guidelines, Subdivision N, § 161-3, and SETAC-Europe Procedures for

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Assessing the Environmental Fate and Ecotoxicity of Pesticides, Section 2.0 guidelines

COMPLIANCE:

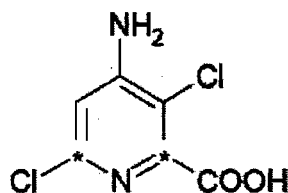
Good Laboratory Practices standards, 40 CFR Part 160

A. MATERIALS:

1. Test Material :

[2,6-¹⁴C]XDE-750 (aminopyralid; p. 19; Figure 1, p.54).

Chemical Structure:



Description:

Technical, solid (p. 19).

Purity:

Analytical purity: N/A
Radiochemical purity: 98.25% (Figure 1, p. 54).
Specific activity: 28.6 mCi/mmole
Locations of the radio label: 2,6-¹⁴C

Storage conditions of test chemicals: Stored in a freezer, maintained in the dark (p. 19).

Physico-chemical properties of XDE-750:

Parameter	Values	Comments
Water solubility		
pH 5	212 g/L	at 20 °C (1)
pH 7	205 g/L	at 20 °C (1)
pH 9	203 g/L	at 20 °C (1)
unbuffered	2.48 g/L	at 20 °C (1)

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Vapour pressure	1.94 x 10 ⁻¹⁰ mm Hg	at 25 °C (2)
UV absorption	N/A	N/A
pKa	2.56	at 20 °C (3)
Log Kow		
unbuffered	0.201	at 20 °C (4)
pH 5	-1.75	at 20 °C (4)
pH 7	-2.87	at 20 °C (4)
pH 9	-2.96	at 20 °C (4)
Stability of compound at room temperature, if provided	N/A ^a	

Data were obtained from p. 19 of the study report.

^a Samples were stored refrigerated or frozen.

2. Soil Characteristics:

Table 1: Field information and handling procedures.

Information	Details
Geographic location	Nordchein, Germany
Site description	Winter wheat, bare soil
Pesticide use history at the collection site	Not provided
Collection date	April 10, 2002
Collection procedures	Hand trowel, 10-12 sites within 50' x 50' plot, into plastic 5-gallon bucket
Sampling depth (cm)	Not provided
Storage conditions	Refrigerated until samples weighed out for pre-incubation
Storage length prior to use	18 months
Soil preparation	2 mm sieve

Data were obtained from p. 20-21 and Appendix B, p. 68 of the study report.

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Table 2: Properties of the soil.

Property	Details	
Soil texture class	silt loam	
% sand	28	
% silt	58	
% clay	14	
pH ^a	7.7	
Organic matter ^b (%)	2.0	
Organic carbon ^c (%)	1.0	
CEC (meq/100 g)	8.1	
Field moisture capacity at 1/3 bar (%)	17.9	
Bulk density (g/cm ³)	1.18	
Soil microbial biomass (µg/g)	Initial	Final
	40.6	Not provided
Soil Taxonomic classification	N/A	
Soil mapping unit (for EPA)	N/A	

Data were obtained from p. 21 and Table 1, p. 44 of the study report.

^a pH analytical procedure - 1:1 soil:water method

^b furnace method

^c Walkley Black method with heat

3) Details of light source:

Table 3: Artificial light source

Property	Details
Type of lamp used	xenon lamp
Emission wavelength spectrum	Measured using a radiometer. These intensity values were similar to values from the Federal register (6) values at 40°N latitude in the summer sun. See Figure 1.

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Light intensity	12 hours (1 day of exposure) was equivalent to 0.65 days of summer sunlight at 40° N latitude
Filters used	Inner Filter: Borosilicate Glass Filter Outer Filter: Soda Lime Filter wavelengths filtered: < 290 nm
Relationship to natural sunlight	Determined using both chemical actinometry and radiometer data.

Data were obtained from p. 21-22 and Table 3, p. 46 of the study report.

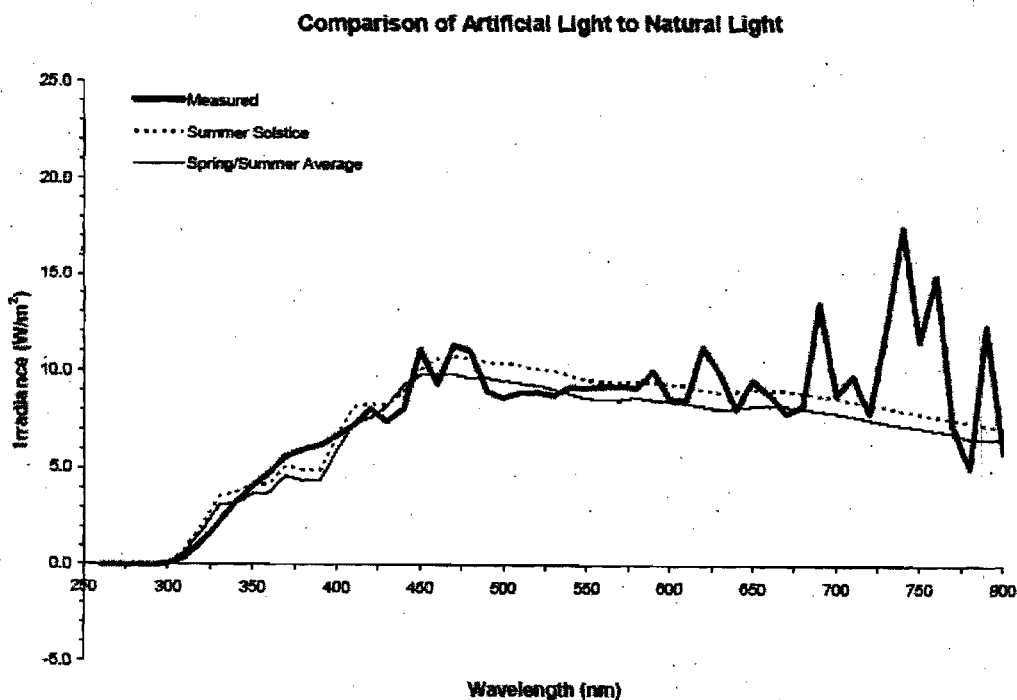


Figure 1: Measured emission spectra of the xenon light source used in experimental set-up vs. summer sunlight (Figure 4, p. 57).

B. EXPERIMENTAL DESIGN

1) Preliminary Study: No preliminary studies were conducted.

2) Experimental Design:

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The current anticipated maximum application rate of XDE-750 is 120 g a.i./ha. Assuming a 11.4 cm² soil surface area, the maximum application rate translates to approximately 14 µg/sample. A chemical actinometer solution was used to determine the irradiance received during the study. A PNAP/pyr actinometer was chosen because the concentration of pyridine can be varied to give a half-life from a few hours to 2 months. PNAP is not volatile from water and is not readily sorbed to glass. The quantum yield for a PNAP/pyr actinometer is independent of wavelength and adjusted by the concentration of pyridine, as stated in the equation: $\phi = 0.0169 [\text{pyr}]$ (7, 8). The actinometer solution used for this study contained 1.0 x 10⁻⁴ M PNAP and 0.019 M pyridine in HPLC grade water. Five-milliliter aliquots of this solution were added to the same type of quartz flasks and amber-colored Pyrex glass tubes as were used for the irradiated and dark control samples. The irradiated actinometers were fitted with glass stoppers; parafilm was wrapped around the fitting. The actinometers were then put in the waterbath in the xenon lamp room with the dosed irradiated soil samples. Because of the size of the waterbath, the number of actinometers irradiated at any given time was limited. Therefore, actinometer samples were replaced with new actinometer samples periodically during irradiation. The dark control actinometers were capped with Teflon-lined screw-caps, wrapped in aluminum foil, and placed in the incubator with the dosed dark control soil samples (p. 18, 22).

Table 4: Experimental design.

Parameter		Details
Duration of the test		44 DAT, equivalent to 28 days of summer sunlight at 40° N latitude
Condition of soil:	Air dried/fresh:	Fresh, moisture maintained at approximately 75% of 1/3 bar
	Sterile/Non-sterile:	Non-sterile
Test concentrations (mg a.i./kg soil)		
dark		5.2
irradiated		5.2
Dark controls used (Yes/No)		yes
Is it foil wrapped and kept in darkness		kept in darkness
Replications	Dark control:	duplicates
	Irradiated:	duplicates
Identity and concentration of co-solvent:		HPLC-grade water

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Pesticide application	Volume of test solution used/treatment:	0.028 mL
	Method of application (eg., spraying):	positive displacement pipette
	Is the co-solvent evaporated:	no
Test apparatus: Type/Material/Volume		quartz boiling flasks with a flat bottom for irradiated samples; amber-colored Pyrex glass vials for dark samples
Details of traps for volatile, if any		Traps contained a layer of 80:20 Silica Gel Grade 35, 12-42 mesh and a layer of glass wool, followed by Ascarite II, 20-30 mesh. Tops of traps were covered with parafilm. Entire traps were wrapped in aluminum foil.
If no traps were used, is the system closed/open		Traps were opened to atmosphere for passive trapping. Systems opened for moisture adjustment.
Any indication of the test material adsorbing to the walls of the test apparatus		none
Experimental Conditions	Temperature: Temperature maintenance method:	approximately 25 waterbath/constant temperature room
	Moisture content: Moisture maintenance method:	75% of 1/3 bar gravimetric
	Duration of light/darkness:	12 hour light/dark cycles
Other details, if any		none

Data were obtained from p. 22-26 and Table 2, p. 45.

3) Supplementary experiments: No supplementary experiments were conducted.

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4) Sampling:

Table 5: Sampling details.

Criteria	Details
Sampling intervals of soil samples	0, 2, 7, 14, 26, 35, and 44 DAT, equivalent to 0, 1, 5, 9, 17, 23, and 28 days of summer sunlight at 40° N latitude
Sampling method	Transfer soil from flask to centrifuge tube (dark controls already in tubes for extraction). Extract 3 times with 5 mL 90:10 acetone: HCl 1 N. Analyze aliquots by LSC and HPLC.
Method of sampling CO ₂ and volatile organic compounds, if any	Separate silica gel and ascarite. Extract ascarite with water. Analyze aliquots by LSC.
Sampling intervals/times for moisture content: sterility check, if any:	21, 35 DAT—irradiated samples only N/A
Sample storage before analysis	Sample extracts were analyzed by LSC on the day of sacrifice. Organic extracts analyzed by HPLC, initially, within one week of sacrifice. Actinometers, ascarite trap extracts, and purified soil extracts were stored refrigerated. Organic soil extracts were stored in freezer
Other observations, if any	none

Data were obtained from p. 25-26 and Table 4, p. 47 of the study report.

C. ANALYTICAL METHODS

At each sampling point (except time 0), the samples were removed from the water bath and the silica gel and ascarite in the traps were poured into separate glass vials. The silica gel vial was capped and stored at room temperature. As no radioactivity was expected to be trapped in the silica gel layer, no analysis of the gel was performed. The ascarite was dissolved and extracted with water. The dissolved ascarite solutions were weighed, the density determined, and aliquots were taken for LSC analysis (p. 25-26).

The dark control soil samples were extracted in their sample vials by adding approximately 10 mL of 90:10 acetone: HCl 1 N and shaking on a mechanical shaker for one hour. The samples were then centrifuged at 2000 rpm for 15 minutes. The supernatant was decanted into a

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24-mL Pyrex vial. This process was repeated twice using 5 mL of extraction solution, shaking for 30 minutes, and centrifuging at 2000 rpm for 10 minutes. The extracts were combined and weighed. Three 1.0-mL aliquots of the extract were assayed for ¹⁴C by LSC. Aliquots of sample extracts were then prepared for HPLC analysis (p. 26-27).

For the irradiated samples, 10 mL of the 90:10 acetone: HCl 1 N solution were added to the quartz flask. The samples were swirled and poured into 24-mL Pyrex vials. The samples were then extracted using the same process as for the dark control samples (p. 27). The extracted soil pellet was allowed to air dry in a hood for at least one week prior to combustion analysis. Extracted, air-dried soil samples were combusted to determine the amount of non-extractable residues (NER) (p. 27).

To characterize the NER sub-samples (about 0.5 g air-dry weight) of previously extracted, airdried soil were extracted with 2.5 mL of 0.5 M NaOH on a mechanical shaker at room temperature for approximately 24 hours. The sample was centrifuged at 2000 rpm for 15 minutes and the supernatant transferred to a centrifuge tube. The sample was briefly mixed with another 2.5-mL aliquot of 0.5 M NaOH and centrifuged as above, and the supernatant was combined with the original extract. The soil pellet was then rinsed with 2.5 mL of deionized water, centrifuged as above, and the supernatant was combined with the original extract. The soil pellet was air-dried and combusted to determine the amount of radioactivity associated with the humin (p. 28).

The above caustic solution was acidified to pH 2 and allowed to stand at room temperature for approximately 24 hours. After this period, the sample was centrifuged at 3000 rpm for 15 minutes. The supernatant was transferred to a volumetric flask and diluted to volume with deionized water. Aliquots of the acidic solution (fulvic acid) were assayed by LSC. The precipitate (humic acid) was re-dissolved in 2 mL of 0.5 M NaOH. Aliquots of the humic acid fraction were assayed by LSC (p. 28).

Organic extracts were purified prior to HPLC analysis. The extracts were analyzed by one of the two methods shown below:

Reverse phase method 1 (used to analyze 0-35 DAT soil extracts)

Aquasil, 5 µm, 4.6 x 250 mm

Solvent A: Water + 0.5% TFA

Solvent B: Acetonitrile + 0.5% TFA

1.0 mL/min flow rate

UV: 270 nm

5 minute post-equilibration time

Time (minutes)	% solvent A	% solvent B
0	100	0

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5	100	0
15	90	10
25	90	10

Data were obtained from Table 5, p. 48 of the study report.

Reverse phase method 2 (used to analyze 44 DAT soil extracts)

Aquasil, 5 μ m, 4.6 x 250 mm

Solvent A: Water + 0.5% TFA

Solvent B: Acetonitrile + 0.5% TFA

1.0 mL/min flow rate

UV: 270 nm

5 minute post-equilibration time

Time (minutes)	% solvent A	% solvent B
0	100	0
5	100	0
15	70	30
25	70	30

Data were obtained from Table 5, p. 48 of the study report.

II. RESULTS AND DISCUSSION:

A. TEST CONDITIONS: Temperature of samples was reported maintained at $25 \pm 1^\circ\text{C}$ (p. 25 and Table 2, p. 45); no supporting temperature data was provided. The soil moisture of the irradiated samples was reported adjusted to 75% of 1/3 bar several times throughout the study; soil moisture of the dark samples was not monitored nor adjusted (p. 25 and Table 2, p. 45). No supporting moisture data was provided. The actinometry data indicate that 1 DAT of exposure to the xenon lamp was equivalent to 0.65 days in the summer sun at 40°N latitude. Therefore, in order to calculate the predicted environmental photodegradation half-life, the sample exposure times of 2, 7, 14, 26, 35, and 44 DAT were converted to 1, 5, 9, 17, 23, and 28 days in the summer sun at 40°N latitude and these converted sampling times were used in the kinetics calculations to correct to natural sunlight intensity for the irradiated samples (p. 35).

B. MASS BALANCE: The mean mass balance was $100.1 \pm 4.4\%$ and $93.8 \pm 7.3\%$ in the dark and irradiated samples, respectively. Over time the material balance of the irradiated samples decreased from 105.2% at 0 DAT to 97.9-98.0% of the applied at days 2-7 to 85.2% of applied at study termination. A possible explanation for this decrease offered by the study authors is that $[2,6-^{14}\text{C}]$ aminopyralid degraded into volatile material, and since the test apparatus relied on

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passive trapping of CO₂ and other gases in the ascarite trap, volatile transformation product(s) could have been lost when the sample was opened to adjust the soil moisture (p. 36). In dark samples, the material balance was also 105.2% of the applied at day 0, and varied from 96.5% to 103.7% of the applied for the remainder of the study.

Table 6: Phototransformation of XDE-750, expressed as percentage of applied radioactivity (mean ± s.d.; n=2), on soil.

Compound		Sampling times (DAT)						
		0	2	7	14	26	35	44
Parent aminopyralid	irradiated	104.2 ±1.4	94.5 ±0.1	91.3 ±3.2	79.8 ±4.0	81.9 ±12.4	78.7 ±7.0	69.3 ±7.6
	dark	104.2 ±1.4	97.7 ±2.7	99.6 ±0.4	94.4 ±1.3	100.3 ±10.0	93.4 ±1.2	92.5 ±3.2
Unidentified extracted radioactivity	irradiated	0.4 ±0.1	0.1 ±0.1	1.2 ±1.5	0.4 ±0.1	1.3 ±1.0	3.3 ±2.1	4.6 ±3.3
	dark	0.4 ±0.1	1.1 ±1.4	0.6 ±0.6	0.0 ±0.0	0.9 ±1.3	0.0 ±0.0	0.9 ±0.1
Total extractable residues	irradiated	104.6 ±1.3	94.6 ±0.2	92.4 ±1.7	80.2 ±3.9	83.2 ±11.5	81.9 ±4.9	73.9 ±4.3
	dark	104.6 ±1.3	98.8 ±1.3	100.2 ±0.2	94.4 ±1.3	101.2 ±8.7	93.5 ±1.2	93.4 ±3.3
Non-extractable residue	irradiated	0.6 ±0.0	2.3 ±0.8	3.6 ±1.0	5.3 ±1.0	6.0 ±1.6	5.9 ±0.2	8.0 ±1.5
	dark	0.6 ±0.0	1.1 ±0.1	1.7 ±0.0	2.1 ±0.1	2.5 ±0.1	3.2 ±0.2	3.8 ±0.1
Ascarite trap (CO ₂)	irradiated	N/A	1.0 ±0.3	2.0 ±0.4	3.8 ±1.7	1.3 ±1.1	3.0 ±0.7	3.4 ±1.3
	dark	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Total % recovery	irradiated	105.2 ±1.3	97.9 ±1.3	98.0 ±0.3	89.3 ±1.2	90.5 ±8.7	90.8 ±5.4	85.2 ±4.2
	dark	105.2 ±1.3	99.9 ±1.3	101.9 ±0.2	96.5 ±1.2	103.7 ±8.8	96.6 ±1.4	97.1 ±3.3

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C. TRANSFORMATION OF PARENT COMPOUND:

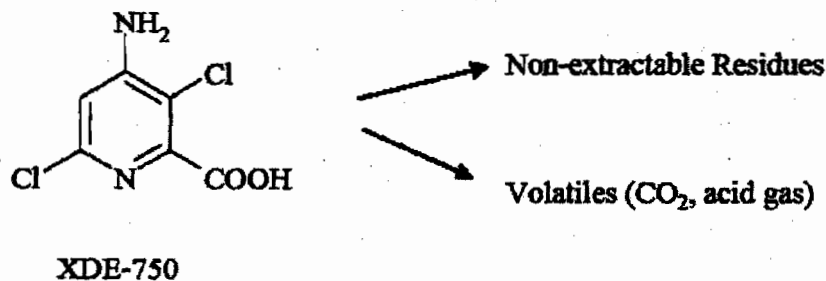
At test termination, 92.5% of the applied ^{14}C remained as the parent in the dark samples. The remaining radioactivity was present as non-extractable residues (3.8% of applied at study termination), unidentified radioactivity (0.9% at study termination), and possibly untrapped CO_2 .

In the irradiated samples, concentration of the parent compound decreased from 104.2% of the applied at day 0 to 79.8-81.9% of the applied at days 14-26, and to 69.3 % of the applied amount at the end of the study. No major transformation products were detected in the irradiated samples. The minor transformation product or products formed in the irradiated samples at a maximum of 4.6% of the applied amount on the 44th day of incubation. Since the transformation product or products formed in the irradiated samples at less than 5% of applied, no conclusive identification was attempted. In irradiated samples, at the end of the study, the evolved CO_2 and volatiles together amounted to a total of 3.4% of the applied radioactivity. At the end of study, total unidentified radioactivity was 0.9% and 4.6% of the applied amount in the dark and irradiated samples, respectively.

NON-EXTRACTABLE AND EXTRACTABLE RESIDUES:

Extractable [^{14}C] residues decreased from 104.6% of the applied amount at day 0 to 93.4% and 73.9% of the applied at the end of the incubation period in the dark and irradiated samples, respectively. Non-extractable [^{14}C] residues in the irradiated samples increased from 0.6% of the applied amount at day 0 to 8.0% of the applied at study termination. In the dark samples, non-extractable radioactivity was 0.6% of the applied amount at day 0, and 3.8% of the applied at study termination.

PATHWAY: [2,6- ^{14}C]Aminopyralid photodegraded slowly to form non-extractable residues (8.0% of applied at 44 DAT) and CO_2 and volatiles (3.4% of applied at 44 DAT). At study termination, 69.3% of the applied radioactivity was still present as parent.



HALF-LIFE: The half-lives of [2,6- ^{14}C]aminopyralid in the dark and irradiated samples calculated via first-order, linear regression on log-transformed data were 364.8 and 93.7 days, respectively. Empirical DT50 values should not be reported, as aminopyralid had not degraded 50% by the termination of the study.

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Table 7. Half-lives of [2,6-¹⁴C]aminopyralid.

Test system	First order half-life		
	Half-life (days)	Regression equation	R ²
Irradiated	Linear: 93.7	$y = -0.0074x + 4.5766$	0.7065
	Nonlinear: 91.2	$y = 97.6932e^{(0.0076t)}$	0.7161
Dark	Linear: 364.8	$y = -0.0019x + 4.6118$	0.3448
	Nonlinear: 385.1	$y = 100.7439e^{(0.0018t)}$	0.3306

Data were reviewer-calculated from Appendix D, p. 77 of the study report and Attachment 1.

The half-life for phototransformation (the difference between irradiated and dark samples, calculated by subtracting rate constants) was 126.0 days.

The predicted environmental phototransformation half-life of [2,6-¹⁴C]aminopyralid, derived from the measured half-life in the laboratory under the xenon arc lamp, was calculated to be 72.2 days for 40°N latitude in summer sunlight.

D. SUPPLEMENTARY EXPERIMENT-RESULTS: No supplementary experiments were conducted.

III. STUDY DEFICIENCIES:

There was a large loss in material balance in the irradiated samples and concurrent loss and variability in material balance in the dark samples. Also, dark samples were not moistened at any time during the study.

IV. REVIEWER'S COMMENTS:

1) These results indicate that photodegradation of [2,6-¹⁴C]aminopyralid on soil is slow, and is not likely to be a significant process of pathway in the overall environmental degradation of aminopyralid.

2) More than 20% and 8% of the applied radioactivity was unaccounted for in the irradiated and dark samples, respectively, by the end of the study (irradiated: 105.3% and 85.2% of applied, at beginning and end of study; dark: 105.2% and 97.1% of applied, at beginning and end of study). A possible explanation given by the study author is that the [2,6-¹⁴C]aminopyralid degraded into a volatile material that was lost. Indeed, as the test apparatus relied on passive trapping of CO₂ and acid gases in the ascarite traps, a volatile could have been lost when the sample was opened

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to adjust the soil moisture. And volatiles in dark samples were not trapped by design. More precautions should have been taken to minimize losses of volatiles from test apparatus. Also, it is stated in the study that no analysis of the silica gel layer was performed because no activity was expected to be trapped there; the author should have provided a brief rationale to support this statement.

3) The material balance in the dark samples varied from 96.6% to 103.7% of applied from days 2 through the end of the study. This unpredictable temporal variability in material balance calls into question measures of parent radioactivity for these intervals.

4) In contrast with irradiated samples, dark controls were not moistened at any time during the experiment. All conditions except irradiation status should have been similar in both dark and irradiated soils so that differences between dark and irradiated samples can be attributed only to photodegradation. Moisture content optimal for microbial degradation should have been maintained in both dark and irradiated samples. Lack of moisture control and, hence, lack of consistency in microbial degradation processes may be a reason why there is variability in mass balance over time in the dark samples.

5) The correlation coefficient obtained for first order linear regression on log-transformed data for kinetics in dark controls was low ($r^2 = 0.3448$).

6) It was not stated whether there was a pesticide use history at the site of soil collection. Also, the sampling depth was not specified (soil should be sampled from the surface). Finally, there was no information provided to support reported study temperature. It is preferred that the study author reports either the temperature of each sampling interval, or the maximum, minimum and average study temperature, as well as any significant deviations from the average and their duration.

V. REFERENCES:

- 1) Nelson R. 2002. Determination of the water solubility of XDE-750. FOR01015, unpublished report of Dow AgroSciences LLC.
- 2) Griffin K. A. 2001. Vapor pressure of XR-750 by Knudsen-Effusion weight loss method. AS-PR 2001-002, unpublished report of The Dow Chemical Company.
- 3) Cathie C. 2001. Determination of dissociation constant of XR-750 using UV-visible spectrophotometry. 01-822-AG, unpublished report of Dow AgroSciences LLC, 2001.
- 4) Madsen S. 2003. Determination of the n-octanol/water partition coefficient (Shake flask method) of XDE-750. FOR01009, unpublished report of Dow AgroSciences LLC.

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- 5) Roulin S. P. 2001. Determination of the organic solubility of XR-750. 01003/DA, unpublished report of Dow AgroSciences LLC.
- 6) Federal Register, Part II EPA 40 CFR Parts 796, 797, and 798 Toxic Substances Control Act Test Guidelines; Final Rules, Friday September 27, 1985, p. 39318.
- 7) Dulin D. and T. Mill. 1982. Development and evaluation of sunlight actinometer. Environ. Sci. Technol. 16, pp. 815-820.
- 8) Leifer A. 1988. The kinetics of environmental aquatic photochemistry. American Chemical Society.
- 9) Yoder R.N. and K.P. Smith. 2003. Aerobic soil degradation of XDE-750 in four european soils. Study number 020054, Dow AgroSciences LLC. Unpublished report.

Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235728
 Guideline No: 161-3

Table 6 Phototransformation of [2,6-C¹⁴]aminopyralid on Parabraun Erde silt loam (irradiated samples), expressed as percentage of the applied radioactivity

Time (days)	Parent	Unidentified	Total extractable	Non-extractable	Ascarite Trap?		Total
					CO2	VOC?	
0	105.2	0.3	105.5	0.6	N/A		106.1
0	103.2	0.5	103.7	0.6	N/A		104.3
AVG	104.2	0.4	104.6	0.6	N/A		105.2
STDEV	1.4	0.1	1.3	0.0	N/A		1.3
2	94.4	0.0	94.4	1.8	0.8		97.0
2	94.6	0.1	94.7	2.9	1.2		98.8
AVG	94.5	0.1	94.6	2.4	1.0		97.9
STDEV	0.1	0.1	0.2	0.8	0.3		1.3
7	93.5	0.1	93.6	2.9	1.7		98.2
7	89.0	2.2	91.2	4.3	2.3		97.8
AVG	91.3	1.2	92.4	3.6	2.0		98.0
STDEV	3.2	1.5	1.7	1.0	0.4		0.3
14	77.0	0.4	77.4	6.0	5.0		88.4
14	82.6	0.3	82.9	4.6	2.6		90.1
AVG	79.8	0.4	80.2	5.3	3.8		89.3
STDEV	4.0	0.1	3.9	1.0	1.7		1.2
26	73.1	2.0	75.1	7.1	2.1		84.3
26	90.7	0.6	91.3	4.8	0.5		96.6
AVG	81.9	1.3	83.2	6.0	1.3		90.5
STDEV	12.4	1.0	11.5	1.6	1.1		8.7
35	73.7	4.7	78.4	6.0	2.5		86.9
35	83.6	1.8	85.4	5.7	3.5		94.6
AVG	78.7	3.3	81.9	5.9	3.0		90.8
STDEV	7.0	2.1	4.9	0.2	0.7		5.4
44	63.9	6.9	70.8	9.0	2.4		82.2
44	74.6	2.3	76.9	6.9	4.3		88.1
AVG	69.3	4.6	73.9	8.0	3.4		85.2
STDEV	7.6	3.3	4.3	1.5	1.3		4.2
						AVR	93.8
						STDEV	7.3

Data were obtained from Appendix D, pp. 77 of the study report.
 N/A= Not Applicable because not sampled

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235728
 Guideline No: 161-3

Table 6 Phototransformation of [2,6-C¹⁴]aminopyralid on Parabraun Erde silt loam (dark samples), expressed as percentage of the applied radioactivity

Time (days)	Parent	Unidentified	Total extractable	Non-extractable	Ascarite Trap?		Total
					CO2	VOC?	
0	105.2	0.3	105.5	0.6	N/A		106.1
0	103.2	0.5	103.7	0.6	N/A		104.3
AVG	104.2	0.4	104.6	0.6	N/A		105.2
STDEV	1.4	0.1	1.3	0.0	N/A		1.3
2	95.8	2.1	97.9	1.0	N/A		98.9
2	99.6	0.1	99.7	1.1	N/A		100.8
AVG	97.7	1.1	98.8	1.1	N/A		99.9
STDEV	2.7	1.4	1.3	0.1	N/A		1.3
7	99.9	0.1	100.0	1.7	N/A		101.7
7	99.3	1.0	100.3	1.7	N/A		102.0
AVG	99.6	0.6	100.2	1.7	N/A		101.9
STDEV	0.4	0.6	0.2	0.0	N/A		0.2
14	93.5	0.0	93.5	2.1	N/A		95.6
14	95.3	0.0	95.3	2.0	N/A		97.3
AVG	94.4	0.0	94.4	2.1	N/A		96.5
STDEV	1.3	0.0	1.3	0.1	N/A		1.2
26	93.2	1.8	95.0	2.4	N/A		97.4
26	107.3	0.0	107.3	2.6	N/A		109.9
AVG	100.3	0.9	101.2	2.5	N/A		103.7
STDEV	10.0	1.3	8.7	0.1	N/A		8.8
35	94.3	0.0	94.3	3.3	N/A		97.6
35	92.6	0.0	92.6	3.0	N/A		95.6
AVG	93.5	0.0	93.5	3.2	N/A		96.6
STDEV	1.2	0.0	1.2	0.2	N/A		1.4
44	94.7	1.0	95.7	3.7	N/A		99.4
44	90.2	0.8	91.0	3.8	N/A		94.8
AVG	92.5	0.9	93.4	3.8	N/A		97.1
STDEV	3.2	0.1	3.3	0.1	N/A		3.3
						AVR	100.1
						STDEV	4.4

Data were obtained from Appendix D, pp. 78 of the study report.
 N/A= Not Applicable because not sampled

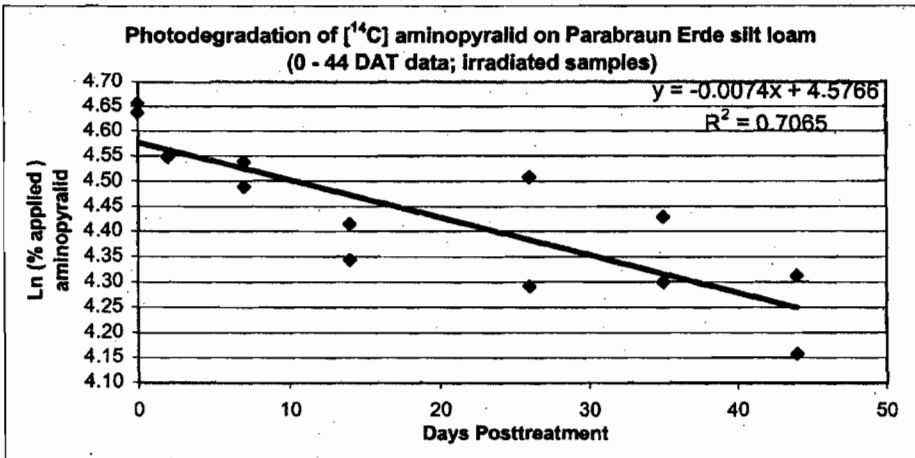
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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235728
 Guideline No: 161-3

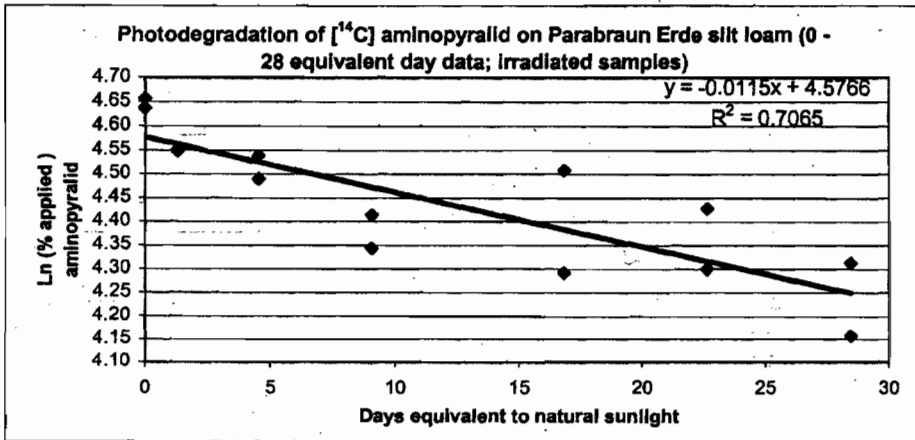
Days posttreatment	Equivalent days in summer sun	% applied	Ln (% applied)
0	0	105.2	4.66
0	0	103.2	4.64
2	1	94.4	4.55
2	1	94.6	4.55
7	5	93.5	4.54
7	5	89.0	4.49
14	9	77.0	4.34
14	9	82.6	4.41
26	17	73.1	4.29
26	17	90.7	4.51
35	23	73.7	4.30
35	23	83.6	4.43
44	28	63.9	4.16
44	28	74.6	4.31

Data were obtained from Appendix D, pp. 77 of the study report.

First order linear half-life = 93.7 days
 First order nonlinear half-life = 91.2 days



(Predicted) First order linear half-life = 60.3 days

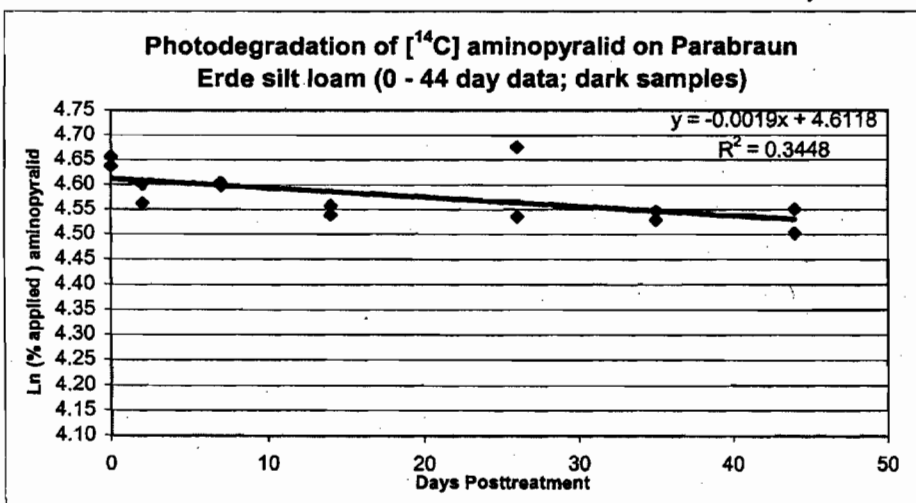


Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235728
 Guideline No: 161-3

Days	% applied	Ln (% applied)
0	105.2	4.66
0	103.2	4.64
2	95.8	4.56
2	99.6	4.60
7	99.9	4.60
7	99.3	4.60
14	93.5	4.54
14	95.3	4.56
26	93.2	4.53
26	107.3	4.68
35	94.3	4.55
35	92.6	4.53
44	94.7	4.55
44	90.2	4.50

Data were obtained from Appendix D, pp. 78 of the study report.

First order linear half-life = 364.8 days
 First order nonlinear half-life = 385.1 days



k (irradiated)= 0.0074 DAT⁻¹
 k (dark)= 0.0019 DAT⁻¹
 Difference between dark and irradiated k values= 0.0055 DAT⁻¹
 Half-life for photodegradation only= 126.0 DAT

Corrected for natural sunlight:
 k (irradiated, corrected)= 0.0115 days⁻¹
 Difference between dark and irradiated, corrected k values= 0.0096 days⁻¹
 Predicted half-life for photodegradation only= 72.2 days

Irradiated samples

Nonlinear Regression

Equation: Single, 2 Parameter

$$y=ae^{-bx}$$

R = 0.84619869 Rsqr = 0.71605222 Adj Rsqr = 0.69238990

Standard Error of Estimate = 6.7561

	Coefficient	Std. Error	t	P
a	97.6932	2.8844	33.8699	<0.0001
b	0.0076	0.0014	5.3631	0.0002

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	1381.2755	1381.2755	30.2613	0.0001
Residual	12	547.7395	45.6450		
Total	13	1929.0150	148.3858		

PRESS = 728.6378

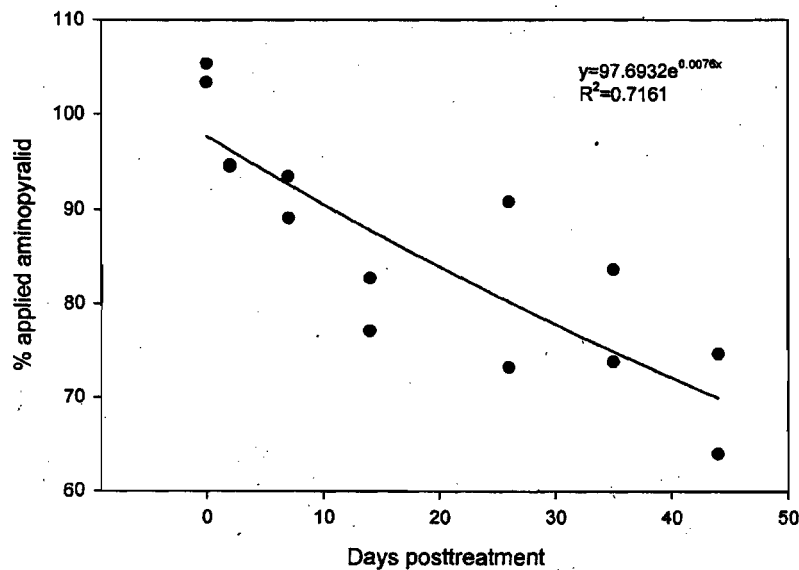
Durbin-Watson Statistic = 1.9133

Normality Test: Passed (P = 0.5248)

Constant Variance Test: Passed (P = 0.5729)

Power of performed test with alpha = 0.0500: 0.9847

Phototransformation of [2,6-¹⁴C]aminopyralid on Parabraun Erde silt loam: Irradiated samples



Dark samples

Nonlinear Regression

Equation: Single, 2 Parameter

$$y = ae^{-bx}$$

R = 0.57497899 Rsqr = 0.33060084 Adj Rsqr = 0.27481758

Standard Error of Estimate = 4.3472

	Coefficient	Std. Error	t	P
a	100.7439	1.7972	56.0568	<0.0001
b	0.0018	0.0008	2.4206	0.0323

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	111.9984	111.9984	5.9265	0.0315
Residual	12	226.7738	18.8978		
Total	13	338.7721	26.0594		

PRESS = 289.8561

Durbin-Watson Statistic = 2.0241

Normality Test: Passed (P = 0.3083)

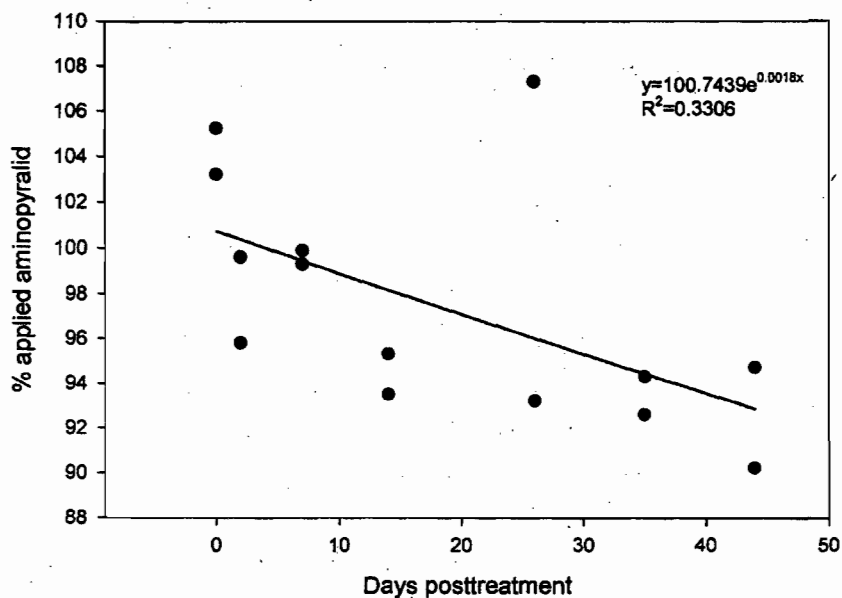
Constant Variance Test: Passed (P = 0.6263)

Power of performed test with alpha = 0.0500: 0.5840

The power of the performed test (0.5840) is below the desired power of 0.8000.

You should interpret the negative findings cautiously.

Phototransformation of [2,6-¹⁴C]aminopyralid on Parabraun Erde silt loam: Dark samples



Data Evaluation Report on the aerobic biotransformation of aminopyralid (XDE-750) in five North American soils

PMRA Submission Number 2004-0789

EPA MRID Number 46235729



Data Requirement:

PMRA DATA CODE: 8.2.3.4.2
US EPA DP Barcode: Not available
OECD Data Point: IIA 7.1.1, IIA 7.2.1, IIA 7.2.2,
IIA 7.2.3, IIIA 9.1.1
US EPA Guideline: Subdivision N, §162-1

Test material:

Common name: XDE-750

Chemical name:

IUPAC: 4-amino-3,6-dichloropyridine-2-carboxylic acid

CAS name: 4-amino-3,6-dichloro-2-pyridinecarboxylic acid

CAS No.: 150114-71-9

Synonyms: aminopyralid, DE-750

SMILES string: ClC(=O)Oc1nc(c(c1N)Cl)Cl

Primary Reviewer: 1046
{EPA/OECD/PMRA}

Signature:
Date: Dec. 6/2004

Secondary Reviewer(s): Roxolana Kashuba
(EPA/EFED/ERB4)

Signature:
Date: May 9/2005

Company Code: [for PMRA]
Active Code: [for PMRA]
Use Site Category: 13, 14 and 16
EPA PC Code:

CITATION: R. N. Yoder, K. P. Smith, 2002, Aerobic Soil Degradation of XDE-750 in Five North American Soils, Regulatory Laboratories—Indianapolis Lab, 010091, Dow AgroSciences LLC, December 20, 2002



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EXECUTIVE SUMMARY:

The biotransformation of radiolabeled aminopyralid (XDE-750) [2,6-¹⁴C]-labeled 4-amino-3,6-dichloropyridine-2-carboxylic acid (aminopyralid; XDE-750) was studied in a Holdrege silt loam (pH 4.6, 1.5% organic carbon) from Kansas (USA), a Regent loam (pH 7.5, 3.4% organic carbon) from Manitoba (Canada), a Manning sandy loam (pH 7.3, 1.2% organic carbon) from North Dakota (USA), a Barnes clay loam (pH 4.8, 3.6% organic carbon) from North Dakota (USA), and a Houston Black clay (pH 7.5, 3.4% organic carbon) from Texas (USA). Fifty-gram soil samples were incubated for up to 1 year under aerobic conditions in the dark at 25 °C and 75% of 1/3 bar % moisture. [2,6-¹⁴C]aminopyralid was applied at the rate of 0.06 mg/kg soil, equivalent to 120 g a.i./ha or 0.11 lb. a.i. per acre. This rate is equivalent to the anticipated maximum use rate. Other samples were dosed at 0.03 mg/kg, equivalent to 60 g a.i./ha or 0.05 lb. per acre, the expected typical seasonal application rate. The experiment was conducted in accordance with the US EPA Subdivision N. Section §162-1 and Canada PMRA DACO Number 8.2.3.4.2, and to meet the GLP standards, the US EPA Good Laboratory Practice Standards, 40 CFR Part 160. The test system consisted of two-chambered biometer flasks with traps for the collection of CO₂. Samples were analyzed at 0, 1, 4, 8, 14, and 22 days and 1, 2, 3, 4, 6, 9 and 12 months of incubation. Samples of each soil type and application rate were not analyzed at all the listed time points. The soil samples were extracted three times with 90:10 acetone: 0.1 N HCl on a horizontal shaker at low speed. The [2,6-¹⁴C]aminopyralid residues were separated by reverse phase HPLC and were subsequently analyzed by LSC. Fractions were collected for all radioactive samples. The fraction counts were used to reconstruct radiochromatograms.

Summary of material balance for the five soils tested (% of applied radioactivity).¹

	Holdrege 60 g/ha	Holdrege 120 g/ha	Regent 60 g/ha	Regent 120 g/ha	Manning 60 g/ha	Manning 120 g/ha	Barnes 60 g/ha	Barnes 120 g/ha	Houston 60 g/ha	Houston 120 g/ha
Mean	94.7	94.2	91.1	86.9	83.7	87.4	90.1	90.0	91.1	91.1
Std. Dev.	22.0	7.5	5.8	8.9	17.5	8.0	4.6	8.7	3.4	9.0
Min.	78.4	77.5	78.3	67.8	74.4	77.9	84.3	74.9	84.4	72.8
Max.	109.0	104.8	100.3	102.7	102.6	108.4	100.8	108.8	95.8	110.6

Data were obtained from Tables 9-18 and Appendix C, pp. 57-66, 94-95, 97-98, 100-101, and 103-106 of the study report.

¹ Statistics calculated using all replicates.

Material balance for the Holdrege silt loam in each replicate was 94.7 ± 22.0% (range = 78.4 to 109.0%) and 94.2 ± 7.5% (range = 77.5 to 104.8%) of the applied amount at the 60- and

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120-g/ha rates, respectively. No pattern of decline in material balances was observed during the first 190 days of the study, and then a 15% decline was observed between day 190 and day 365 for both rates. At 60 g/ha, the concentration of the parent compound decreased from 99.7% of the applied amount at Day 0 to 55.8% of the applied at 33 DAT and 3.8% of the applied at the end of the study period (365 DAT). At 120 g/ha, the concentration of the parent compound decreased from 102.2% of the applied amount at Day 0 to 56.9% of the applied at 33 DAT and 3.7% of the applied at the end of the study period (365 DAT). The first order nonlinear half-life of [2,6-¹⁴C]aminopyralid in aerobic soil was 42.0 days (60 g/ha) and 38.7 days (120 g/ha). Nonlinear first order half-lives were consistent with empirical DT50s (45.0 days for both 60 g/ha and 120 g/ha), while linear first order half-lives overestimated empirical ones (73.0 days for both 60 g/ha and 120 g/ha). When half-lives were calculated assuming non-extractible residues were composed of non-extracted parent (at 120 g/ha), the first order linear half-life was 103.5 days, the first order nonlinear half-life was 58.2 days, and the empirical DT50 was 70.0 days.

Material balance for the Regent loam in each replicate was $91.1 \pm 5.8\%$ (range = 78.3 to 100.3%) and $86.9 \pm 8.9\%$ (range = 67.8 to 102.7%) of the applied amount at the 60- and 120-g/ha rates, respectively. Substantial variation in material balances was observed during the first 124 days (60 g/ha) and 91 days (120 g/ha), followed by a pattern of decline through the end of the study for both rates. There is a 10% loss in material balance between day 0 and day 8 (the first sampling interval) at 120 g/ha, adding uncertainty to the concurrent reported 25% loss in parent radioactivity. At 60 g/ha, the concentration of the parent compound decreased from 99.4% of the applied amount at Day 0 to 55.6% of the applied at 14 DAT and 1.5% of the applied at the end of the study period (365 DAT). At 120 g/ha, the concentration of the parent compound decreased from 100.3% of the applied amount at Day 0 to 60.8% of the applied at 14 DAT, then 24.6% of the applied at 33 DAT and 1.6% of the applied at the end of the study period (365 DAT). The first order nonlinear half-life of [2,6-¹⁴C]aminopyralid in aerobic soil was 19.8 days (60 g/ha) and 17.0 days (120 g/ha). Nonlinear first order half-lives were slightly less than empirical DT50s (15.0 and 20.0 days for 60 g/ha and 120 g/ha, respectively), while linear first order half-lives overestimated empirical ones (61.9 and 62.4 days for 60 g/ha and 120 g/ha, respectively). When half-lives were calculated assuming non-extractible residues were composed of non-extracted parent (at 120 g/ha), the first order linear half-life was 67.3 days, the first order nonlinear half-life was 30.4 days, and the empirical DT50 was 30.0 days. However, all DT50s and half-lives are of questionable value due to material balance problems.

Material balance for the Manning sandy loam in each replicate was $83.7 \pm 17.5\%$ (range = 74.4 to 102.6%) and $87.4 \pm 8.0\%$ (range = 77.9 to 108.4%) of the applied amount at the 60- and 120-g/ha rates, respectively. Material balances for both rates drop sharply during the first sampling interval (8 DAT) and remain lower than 90% of applied radioactivity for the remainder of the study. At 60 g/ha, the concentration of the parent compound decreased from 101.5% of the applied amount at Day 0 to 62.8% of the applied radioactivity at 14 DAT, then 31.5% of the

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applied radioactivity at 33 DAT, and 2.6% of the applied at the end of the study period (190 DAT). At 120 g/ha, the concentration of the parent compound decreased from 103.2% of the applied amount at Day 0 to 73.2% of the applied radioactivity at 14 DAT, then 30.9% of the applied radioactivity at 33 DAT and 2.3% of the applied at the end of the study period (190 DAT). The first order nonlinear half-life of [2,6-¹⁴C]aminopyralid in aerobic soil was 18.8 days (60 g/ha) and 19.4 days (120 g/ha). Nonlinear first order half-lives were slightly less than empirical DT50s (20.0 and 22.0 days for 60 g/ha and 120 g/ha, respectively), while linear first order half-lives overestimated empirical ones (32.4 and 31.9 days for 60 g/ha and 120 g/ha, respectively). When half-lives were calculated assuming non-extractible residues were composed of non-extracted parent (at 120 g/ha), the first order linear half-life was 55.9 days, the first order nonlinear half-life was 25.8 days, and the empirical DT50 was 27.0 days. However, all DT50s and half-lives are of questionable value due to low material balances.

Material balance for the Barnes clay loam in each replicate was $90.1 \pm 4.6\%$ (range = 84.3 to 100.8%) and $90.0 \pm 8.7\%$ (range = 74.9 to 108.8%) of the applied amount at the 60- and 120-g/ha rate, respectively, with a variable pattern dropping below 90% of the applied radioactivity for five of the ten 60 g/ha sampling intervals and three of the eight 120 g/ha sampling intervals. At 60 g/ha, the concentration of the parent compound was 91.9% of the applied amount at Day 0, ranged from 81.3 to 92.1% of the applied from 8 to 63 DAT, and decreased to 42.5% of the applied at the end of the study period (365 DAT). At 120 g/ha, the concentration of the parent compound decreased from 94.6% of the applied amount at Day 0 to 91.4% of the applied at 8 DAT, ranged from 69.0 to 83.5% of the applied from 14 to 91 DAT, and decreased to 41.1% of the applied at the end of the study period (365 DAT). The first order linear half-life of [2,6-¹⁴C]aminopyralid in aerobic soil was 330.1 days (both 60 g/ha and 120 g/ha). Nonlinear first order half-lives were identical to linear ones for both rates, and slightly greater than DT50s (290 and 300 days for 60 g/ha and 120 g/ha, respectively). When half-lives were calculated assuming non-extractible residues were composed of non-extracted parent (at 120 g/ha), the both first order linear and nonlinear half-lives were 533.2 days, and there was no empirical DT50, as parent did not degrade to less than 50% of applied by study completion. However, all DT50s and half-lives are of questionable value due to variability and fluctuation of material balances.

Material balance for the Houston Black clay in each replicate was $91.1 \pm 3.4\%$ (range = 84.4 to 95.8%) and $91.1 \pm 9.0\%$ (range = 72.8 to 110.6%) of the applied amount at the 60- and 120-g/ha rates, respectively. For 60 g/ha, material balances showed a pattern of decline from Day 0 until 8 DAT and a pattern of incline from 8 DAT to the end of the study (65 DAT). For 120 g/ha, material balances were variable, with decreases of almost 10% of applied radioactivity from Day 0 to Day 1, and from Day 14 to Day 22. At 60 g/ha, the concentration of the parent compound decreased from 86.2% of the applied amount at Day 0 to 46.3% of the applied at 4 DAT, and 1.4% of the applied at the end of the study period (65 DAT). At 120 g/ha, the concentration of the parent compound decreased from 90.1% of the applied amount at Day 0 to

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53.1% of the applied at 4 DAT, and 1.1% of the applied at the end of the study period (65 DAT). The first order nonlinear half-life of [2,6-¹⁴C]aminopyralid in aerobic soil was 4.0 days (60 g/ha) and 6.1 days (120 g/ha). Nonlinear first order half-lives were more representative of empirical DT50s (3.5 and 4.5 days for 60 g/ha and 120 g/ha, respectively) than linear first order half-lives (10.4 and 9.4 days for 60 g/ha and 120 g/ha, respectively). When half-lives were calculated assuming non-extractable residues were composed of non-extracted parent (at 120 g/ha), the first order linear half-life was 31.5 days, the first order nonlinear half-life was 17.9 days, and the empirical DT50 was 13.0 days. However, all DT50s and half-lives are of questionable value due to variable material balances.

No major transformation products other than CO₂ and NER (non-extractable residues) were detected. No minor transformation products were detected. None of the radioactivity detected was unidentified; the entire extractable radioactivity was characterized as [2,6-¹⁴C]aminopyralid. For Holdrege silt loam, Regent loam, Manning sandy loam, and Houston Black clay treated at 60 g/ha, extractable [¹⁴C]residues decreased from 99.7, 99.4, 101.5, and 86.2% of the applied amount at Day 0 to 3.8, 1.5, 2.6, and 1.4% of applied at the end of the study period (365, 365, 190, and 65 DAT), respectively. Similarly, for Holdrege silt loam, Regent loam, Manning sandy loam, and Houston Black clay treated at 120 g/ha, extractable [¹⁴C]residues decreased from 102.2, 100.3, 103.2, and 90.1% of the applied amount at Day 0 to 3.7, 1.6, 2.3, and 1.1% of applied at the end of the study period (365, 365, 190, and 65 DAT), respectively. For Barnes clay loam, however, extractable [¹⁴C]residues decreased from 91.9 and 94.6% of the applied amount at Day 0 to 42.5 and 41.1% of applied at the end of the study period (365 DAT) for 60 g/ha and 120 g/ha, respectively. Non-extractable [¹⁴C]-residues increased from about 0% of the applied at Day 0 to 4.9, 15.7, 9.0, and 13.7% of the applied (60 g/ha) and to 5.4, 0.0, 8.3, and 14.0% of the applied (120 g/ha) at the end of the incubation period in Holdrege silt loam, Regent loam, Manning sandy loam, and Barnes clay loam, respectively. Non-extractable [¹⁴C]-residues in Houston Black clay increased from 8.1 and 9.6 % of the applied at Day 0 to 23.1 and 24.3% of the applied at the end of the incubation period (65 DAT) for 60 g/ha and 120 g/ha, respectively.

Organic matter from non-extractable residues in Holdrege silt loam, Regent loam, Manning sandy loam, and Houston Black clay dosed at 60 g/ha at 91, 91, 124, and 8 days, respectively, consisted of fulvic acid fractions of 7.5, 7.5, 7.6, and 23.0 % of the applied, humic fractions of 2.5, 3.8, 0.2, and 1.0 % of the applied, and humin fractions of 3.0, 7.3, 3.3, and 11.4 %. Organic matter from non-extractable residues in Holdrege silt loam, Regent loam, Manning sandy loam, and Houston Black clay dosed at 120 g/ha at 190, 63, 190, and 14 days, respectively, consisted of fulvic acid fractions of 5.2, 8.0, 10.2, and 11.7 % of the applied, humic fractions of 2.2, 4.2, 0.8, and 0.9 % of the applied, and humin fractions of 4.1, 4.5, 1.1, and 12.8 %

At study termination, evolved ¹⁴CO₂ were 70.3, 65.7, 73.2, and 67.5% of the applied at 60 g/ha and 69.3, 66.9, 71.5, and 67.4% of the applied at 120 g/ha, in Holdrege silt loam, Regent loam, Manning sandy loam, and Houston Black clay, respectively. In the Barnes clay loam, 30.0 and

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27.0% of the applied radioactivity was CO₂ at study termination at 60 g/ha and 120 g/ha, respectively. Volatile organics were not measured.

Results Synopsis:

Half-lives/DT50:

Soil type	DT50 (days)	First order half-life, linear regression ¹		First order half-life, non-linear regression ¹	
		half-life (days)	R ²	half-life (days)	R ²
Holdrege 60 g/ha	50.0	73.0	0.9003	42.0	0.9866
Holdrege 120g/ha	60.0	73.0	0.8809	38.7	0.9839
Regent 60 g/ha	15.0	61.9	0.7565	19.8	0.8799
Regent 120 g/ha	20.0	62.4	0.6505	17.0	0.9951
Manning 60 g/ha	20.0	32.4	0.7914	18.8	0.9850
Manning 120 g/ha	22.0	31.9	0.7675	19.4	0.9829
Barnes 60 g/ha	290.0	330.1	0.9635	330.1	0.9481
Barnes 120 g/ha	300.0	330.1	0.8615	330.1	0.7952
Houston 60 g/ha	3.5	10.4	0.6787	4.0	0.9903
Houston 120 g/ha	4.5	9.4	0.7707	6.1	0.9481

Data were obtained from Tables 9-18 and Appendix C, pp. 57-66, 94-95, 97-98, 100-101, and 103-106 of the study report.
¹See Appendix A of this DER for plots of linear and nonlinear regression and for equations of the models used.

Half-lives/DT50 treating non-extractable residues as parent:

Soil type	DT50 (days)	First order half-life, linear regression ¹		First order half-life, non-linear regression ¹	
		half-life (days)	R ²	half-life (days)	R ²
Holdrege 120g/ha	70	103.5	0.9240	58.2	0.9721
Regent 120 g/ha	30	63.7	0.9282	30.4	0.9543
Manning 120 g/ha	27	55.9	0.7654	25.8	0.9747
Barnes 120 g/ha	N/A	533.2	0.7419	533.2	0.6727
Houston 120 g/ha	13	31.5	0.7543	17.9	0.8989

Data were obtained from Tables 9-18 and Appendix C, pp. 57-66, 94-95, 97-98, 100-101, and 103-106 of the study report.
¹See Appendix A of this DER for plots of linear and nonlinear regression and for equations of the models used.

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Major transformation products: CO₂ and non-extractable residues (NER).

Minor transformation products: None.

Study Acceptability: This study is classified as supplemental for an aerobic biotransformation study in soil, however only the Holdrege silt loam provides a useable half-life. Regent loam, Manning sandy loam, Barnes clay loam, and Houston Black clay material balances were either low or variable and the data from these soils cannot be used quantitatively. The method used for extraction was not harsh enough to ensure with certainty that radioactivity attributed to non-extractable residue was truly not able to be extracted. Additionally, because aerobic soil metabolism in Barnes clay loam was not valid, the Subdivision N requirement that one of the soils tested in the adsorption/desorption study also be tested in the aerobic soil metabolism study was not fulfilled.

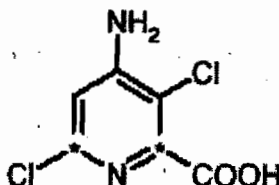
I. MATERIALS AND METHODS

GUIDELINE FOLLOWED: U.S. Environmental Protection Agency (EPA) requirements for aerobic soil metabolism as outlined in the EPA Pesticide Registration Guidelines, Subdivision N, §162-1 and Canada PMRA DACO Number 8.2.3.4.2 - Biotransformation in Soil - Aerobic.

COMPLIANCE: Good Laboratory Practices standards, 40 CFR Part 160

A. MATERIALS:

1. **Test Material:** [2,6-¹⁴C]XDE-750 (aminopyralid; p.20; Figure 1., p.70)



Chemical Structure:

Description: Radiolabeled active substance

Purity: Analytical purity: N/A Radiochemical purity: 99.6% INV1590 Specific activity: 27.4 mCi/mmol Locations of the radiolabel: 2,6-¹⁴C (Figure 1; p. 70)

Storage conditions of test chemicals: frozen, maintained in the dark

Physico-chemical properties of aminopyralid (XDE-750):

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Parameter	Values	Comments
Water solubility		
pH 5	212 g/L	at 20 °C (1)
pH 7	205 g/L	at 20 °C (1)
pH 9	203 g/L	at 20 °C (1)
unbuffered	2.48 g/L	at 20 °C (1)
Vapor Pressure	7.14 x 10 ⁻¹¹ mm Hg	at 20 °C (2)
pKa	2.56	at 20 °C (3)
Log Kow		
Unbuffered	0.2	
pH 5	-1.76	
pH 7	-2.87	
pH 9	-2.96	
Stability of Compound at Room Temperature	Stable at least 5 months after receipt	Confirmed by HPLC re-analysis
Organic Solvent Solubilities		
Acetone	30 g/L	re-analysis
Ethyl Acetate	4 g/L	at 20 °C (4)
Methanol	50 g/L	at 20 °C (4)
1,2-dichloroethane	2 g/L	at 20 °C (4)
Xylene	43 mg/L	at 20 °C (4)
Heptane	less than 10 mg/L	at 20 °C (4)

Data were obtained from p. 21 of the study report.

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2. Soil Characteristics

Table 1: Description of soil collection and storage.

Description	Holdrege	Regent	Manning	Barnes	Houston Black
Geographic location	Decatur Co KS, USA	Whitewater Rural, MB, Canada	Ward Co, ND, USA	Grand Forks, ND, USA	Travis Co, TX, USA
Pesticide use history at the collection site	None for previous year	None for previous year	None for previous year	Pursuit Plus, Double play previous year	Nitrogen fertilizer previous year
Collection procedure	N/A	N/A	N/A	N/A	N/A
Sampling depth (cm)	N/A	N/A	N/A	N/A	N/A
Storage conditions	Stored ambient	Stored ambient	Stored ambient	Stored ambient	Stored ambient
Storage length	2 months	2 months	2 months	2 months	3 months
Soil preparation	Sieved, 2 mm	Sieved, 2 mm air-dried slightly	Sieved, 2 mm air-dried slightly	Sieved, 2 mm air-dried slightly	Sieved, 2 mm air-dried slightly

Table 2: Properties of the soils.

Property	Holdrege	Regent	Manning	Barnes	Houston Black
Soil texture	Silt Loam	Loam	Sandy Loam	Clay Loam	Clay
% sand	21	45	74	34	17
% silt	60	40	16	34	32
% clay	19	15	10	32	51
pH	4.6	7.5	7.3	4.8	7.5
Organic carbon (%)	1.5	3.4	1.2	3.6	3.4
CEC (meq/100 g)	14.5	25.6	15.5	23.5	45.0
Moisture at 1/3 (%)	25.9	30.3	14.3	33.1	40.8
Bulk density (g/cm ³)	1.06	1.03	1.24	0.96	0.99
Soil Taxonomic classification	(1)	(2)	(3)	(4)	(5)
Initial & Final Soil Biomass µg/g	81.5 61.3	313.5 220.4	N/A 105.3	N/A 93.6	672.8 759.9
Soil Mapping Unit	N/A	N/A	N/A	N/A	N/A

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- (1) Fine-silty, mixed, mesic, Typic Argiustolls
- (2) Fine, smectitic, frigid, Vertic Argiustolls
- (3) Coarse-loamy over sand or sandy-skeletal, mixed, superactive frigid Typic Haplustolls
- (4) Fine-loamy, mixed, superactive, frigid Calcic Hapludolls
- (5) Fine, smectitic, thermic Udic Haplusterts

Data were obtained from Table 1, p. 48 of the study report.

B. EXPERIMENTAL CONDITIONS:

- 1. Preliminary experiments:** No preliminary experiments were conducted.

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2. Experimental conditions:

Table 3: Experimental design.

Parameter		Description
Duration of the test		Up to 365 days
Soil condition: (Air dried/fresh)		Samples brought to moisture and temperature for at least 2-7 days prior to treatment
Soil (g/replicate)		Moist soil equivalent to 50-g oven-dry
Test concentrations (mg a.i./kg soil) and equivalent g a.i./ha		0.030 and 0.060 mg a.i./kg soil 58 and 117 g a.i./ha
Control conditions		Same conditions as treated
No. of Replication	Controls	8 controls per soil type
	Treatments	25 samples at 60-g/ha rate, 19 samples at 120-g/ha rate per soil type
Test apparatus (Type/material/volume)		Two-chambered, closed system w/ moist soil in one chamber and NaOH solution in other, slight positive O ₂ pressure to maintain aerobicity
Details of traps for CO ₂		0.2 N NaOH
Identity and concentration of co-solvent		water
Test material application	Volume of test solution	0.8-1.0 mL
	Application method	Syringe to soil surface
	co-solvent evaporated:	N/A
Test material sorption to apparatus?		N/A
Experimental	Temperature (°C)	25 ± 1 °C
	Moisture content	75% of 1/3 bar moisture
	Moisture maintenance method:	Moisture adjusted pre-treatment and maintained in a closed system
	Continuous darkness:	Yes
Other details, if any		Additional samples dosed at 5- and 20-g/ha to establish correlation between concentration and degradation rate. Samples treated, incubated and analyzed in same fashion as above for up to 2 months

Data were obtained from Table 2, p. 50 of the study report.

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3. Aerobic conditions: Biometers were connected via an expansion bulb in the caustic trap to an oxygen manifold to sustain aerobic conditions during incubation. The soil side of each flask was closed with a ground glass stopper, using vacuum grease to create an airtight seal (p.24).

4. Supplementary experiments: Additional samples of four soil types were treated at rates equivalent to 5 and 20 g a.i./ha (less than required maximum rate, 120 g a.i./ha) to determine the impact of concentration on [2,6-¹⁴C]aminopyralid aerobic soil metabolism (p.26).

Samples dosed at 60 and 120 g a.i./ha were subjected to a two-step extraction procedure at 0, 14 and 33 DAT (Houston Black samples at 0 and 14 DAT). This two-step extraction included a brief (5-minute) extraction with 0.01 M CaCl₂ followed by an exhaustive extraction with 90:10 acetone 1.0 N HCl. The extraction with aqueous followed by organic allowed for the determination of [2,6-¹⁴C]aminopyralid sorption potential with soil contact time (p.29).

Control samples of each soil type were dosed with ¹⁴C-dichlorobenzoic acid (DCBA) to characterize soil microbial activity with incubation time (p.27).

5. Sampling:

Table 4: Sampling details.

Parameters	Details
Sampling intervals	Sample intervals dependent on soil type and concentration. See Table 5 -14 for actual time points.
Sampling method for soil samples	All samples extracted 3x with 90:10 acetone: 1.0 N HCl. Several time points extracted first with 0.01 M CaCl ₂ to determine apparent K _d values
Method of collection of CO ₂ and volatile organic compounds	Aspiration of NaOH trap, LSC of triplicate 1-mL aliquots. No volatiles other than CO ₂ were collected
Sampling intervals/times for: Sterility check, Moisture content: Redox potential/other:	No sterile samples were used Moisture content determined by weight at each sampling interval Redox potential was not determined
Sample storage before analysis	N/A, samples were extracted and assayed by LSC the day sacrificed
Other observations, if any	Control samples treated w/ DCBA at 0 DAT, and after 1, 6 and 12 months incubation

Data were obtained from Table 3, p. 51 of the study report.

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C. ANALYTICAL METHODS:

Extraction/clean up/concentration methods:

At each sampling point, the caustic trapping solution was removed from the biometer side arm and aliquots were assayed by LSC. The entire soil sample was transferred to a labeled, weighed glass jar. A 10-g wet weight sub-sample was transferred to a 45-mL centrifuge tube for extraction. The remaining, unextracted soil sample was stored in a freezer. The entire 1-year sample was extracted (pp.29-31).

A two-step extraction procedure was used to analyze 60- and 120-g/ha samples at 0, and 14 DAT and 1 month after treatment, with the exception of the Houston Black clay samples where the 1-month samples were not extracted with CaCl₂ solution. The two-step procedure consisted of a brief extraction with 0.01 M CaCl₂, followed by a more extensive extraction with 90:10 acetone: 1 N HCl. This two-step extraction process was used to determine the non-equilibrium desorption coefficients of [2,6-¹⁴C]aminopyralid with time. All other samples were extracted only with acidified acetone.

Additionally, samples treated at the 5- and 20-g/ha rates were extracted only with acidified acetone.

A 10-mL aliquot of each organic extract was concentrated under a stream of N₂ using a Turbovap to remove the organic solvent. The concentrate was filtered through a 0.45 μm PTFE filter before HPLC analysis. Samples dosed at the 5- and 20-g/ha rates were not analyzed by HPLC. The profile of these samples was assumed to be the same as the 60- and 120-g a.i./ha samples.

Sample extracts were analyzed by reverse phase HPLC using a C₁₈ Inertsil column. The mobile phase for this isocratic system was 90:10 water: acetonitrile, each containing 0.5% TFA. Fractions were collected for all radioactive samples. The fractions were mixed with scintillation cocktail and assayed by LSC. The fraction counts were used to reconstruct radiochromatograms.

Non-extractable residue determination:

The extracted soil pellet was allowed to air dry in a hood for at least one week prior to combustion analysis. Extracted, air-dried soil samples were combusted to determine the amount of non-extractable residues.

Non-extractable residues were characterized by partitioning into fulvic acid, humic acid, and humin pools. Sub-samples (ca. 5 g air-dry weight) of previously extracted, air-dried soil were transferred to a centrifuge tube and extracted with 0.5 M NaOH. The total non-extractable residue is equivalent to the sum of radioactivity associated with the fulvic acid, humic acid, and humin fractions (pp. 31-32).

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Total ¹⁴C measurement: Total ¹⁴C residues were determined by summing the concentration of residues measured in the soil extracts, extracted soil, and volatile trapping solutions.

Derivatization method, if used: A derivation method was not employed.

Volatile residue determination: The presence of CO₂ was confirmed by precipitate of Ba¹⁴CO₃ by the addition of BaCl₂ to several caustic traps (pp. 28-29).

Identification and quantification of parent compound: Sample extracts were analyzed by reverse phase HPLC using a C₁₈ Inertsil column. [2,6-¹⁴C]aminopyralid was identified by LSC only; HPLC fractions were collected once per minute (pp. 71-78) and were analyzed in a LSC.

Table 5: Limits of Detection and Quantitation.

Sub-sample Identification	60 g/ha rate		120 g/ha rate	
	LOD	LOQ	LOD	LOQ
Caustic Trap	0.2	0.9	0.1	0.5
CaCl ₂ Extracts	0.3	1.1	0.1	0.6
Organic Extracts	0.6	2.2	0.3	1.1
Soil Combustions	0.2	0.9	0.1	0.5
HPLC-Aqueous Extract	0.3	1.1	0.1	0.6
HPLC-Organic Extract	0.05	0.8	0.03	0.4

As purity control samples and soil extract samples showed a very similar radiochromatogram, i.e. only one peak with similar retention times, the applicant assumed that the only product eluted from the soil extracts was the parent chemical. Therefore all extractable radioactivity was assumed to be the parent chemical and quantity of [2,6-¹⁴C]aminopyralid was as the percent radiocarbon remaining as extractable radioactivity.

Identification and quantification of transformation products: No transformation products were detected by HPLC-LSC. ¹⁴CO₂ and non-extractable residues (NER) were the only major transformation products detected (p. 42; Figure 6-9, pp. 75-78).

Detection limits (LOD, LOQ) for the parent compound:

The table below presents the limits of detection (LOD) and limits of quantitation (LOQ) as percent of applied radioactivity in the various phases for this study. The LOD and LOQ are presented for the 60-g/ha rate samples and the 120-g/ha samples (p. 37-38).

II. RESULTS AND DISCUSSION:

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A. TEST CONDITIONS: Aerobicity, moisture, temperature and other experimental conditions were maintained throughout the study (p. 38).

B. MATERIAL BALANCE:

Material balance was seen to decrease in some of the soils over time. The loss was most likely from the caustic traps, as very little radioactivity was extractable or recovered by combustion from these later time points, and in several samples, the amount of CO₂ was seen to drop over time, an indication of loss of activity from the traps.

Material balance was recalculated by the reviewer. The applicant reported values did not include those from sampling days with a total recovery range outside of 85-110% radioactivity. The reviewer reports the actual material balance, (which was also used in determining the half-life of the test material), including sampling events reported as MB (from Appendix C, pp. 94-106).

Summary of material balance for the five soils tested (% of applied radioactivity).¹

	Holdrege 60 g/ha	Holdrege 120 g/ha	Regent 60 g/ha	Regent 120 g/ha	Manning 60 g/ha	Manning 120 g/ha	Barnes 60 g/ha	Barnes 120 g/ha	Houston 60 g/ha	Houston 120 g/ha
Mean	94.6	94.2	91.1	86.9	87.4	87.38	90.1	90	91.6	91.1
Std. Dev.	22	7.6	5.6	8.9	7.2	7.9	4.3	5.3	3.4	9
Min.	78.9	77.5	82.7	67.8	74.4	77.9	84.3	74.9	84.4	72.8
Max.	103.6	104.8	99.4	101	102.6	108.4	100.8	108.8	95.8	110.6

Data were obtained from Tables 9-16 and Appendix C, pp. 57-65, 94-95, 97-98, 100-101, and 103-105 of the study report.

¹ Statistics calculated and reported using all replicates.

Material balance for the Holdrege silt loam in each replicate was $94.7 \pm 22.0\%$ (range = 78.4 to 109.0%) and $94.2 \pm 7.5\%$ (range = 77.5 to 104.8%) of the applied amount at the 60- and 120-g/ha rates, respectively. No pattern of decline in material balances was observed during the first 190 days of the study, and then a 15% decline was observed between day 190 and day 365 for both rates (Tables 9-10 and Appendix C, pp. 57-58, 94-95).

Material balance for the Regent loam in each replicate was $91.1 \pm 5.8\%$ (range = 78.3 to 100.3%) and $86.9 \pm 8.9\%$ (range = 67.8 to 102.7%) of the applied amount at the 60- and 120-g/ha rates, respectively. Substantial variation in material balances was observed during the first 124 days (60 g/ha) and 91 days (120 g/ha), followed by a pattern of decline through the end of the study for both rates. There is a 10% loss in material balance between day 0 and day 8 (the first sampling interval) at 120 g/ha, adding uncertainty to the concurrent reported 25% loss in parent radioactivity (Tables 11-12 and Appendix C, pp. 59-60, 97-98).

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Material balance for the Manning sandy loam in each replicate was $83.7 \pm 17.5\%$ (range = 74.4 to 102.6%) and $87.4 \pm 8.0\%$ (range = 77.9 to 108.4%) of the applied amount at the 60- and 120-g/ha rates, respectively. Material balances for both rates drop sharply during the first sampling interval (8 DAT) and remain lower than 90% of applied radioactivity for the remainder of the study (Tables 13-14 and Appendix C, pp. 61-62, 100-101).

Material balance for the Barnes clay loam in each replicate was $90.1 \pm 4.6\%$ (range = 84.3 to 100.8%) and $90.0 \pm 8.7\%$ (range = 74.9 to 108.8%) of the applied amount at the 60- and 120-g/ha rate, respectively, with a variable pattern dropping below 90% of the applied radioactivity for five of the ten 60 g/ha sampling intervals and three of the eight 120 g/ha sampling intervals (Tables 15-16 and Appendix C, pp. 63-64, 103-104).

Material balance for the Houston Black clay in each replicate was $91.1 \pm 3.4\%$ (range = 84.4 to 95.8%) and $91.1 \pm 9.0\%$ (range = 72.8 to 110.6%) of the applied amount at the 60- and 120-g/ha rates, respectively. For 60 g/ha, material balances showed a pattern of decline from Day 0 until 8 DAT and a pattern of incline from 8 DAT to the end of the study (65 DAT). For 120 g/ha, material balances were variable, with decreases of almost 10% of applied radioactivity from Day 0 to Day 1, and from Day 14 to Day 22 (Tables 17-18 and Appendix C, pp. 65-66, 105-106).

Table 5: Biotransformation of [2,6-¹⁴C]aminopyralid, expressed as percentage of applied radioactivity (n=2; mean \pm s.d.), in Holdrege silt loam, 60 g/ha rate, under aerobic conditions.

Compound	Sampling times (days)									
	0	8	14	33	63	91	124	190	274	365
Parent compound	99.7 ± 5.9	87.1 ± 8.7	77.6 ± 2.4	55.8 ± 2.0	36.7	22.1 ± 3.7	11.0 ± 0.4	8.9 ± 3.7	4.5 ± 0.0	3.8 ± 0.1
Total extractable residues	Total extractables are assumed to be the parent chemical									
CO ₂	N/A	7.8 ± 0.3	13.7 ± 0.8	31.3 ± 1.1	55.0 ± 1.1	61.9 ± 0.1	71.7 ± 3.6	74.1 ± 5.1	74.8 ± 0.8	70.3 ± 1.3
Total volatile organics	Not monitored									
Non-extractable residues	0.1 ± 0.1	4.4 ± 0.3	6.9 ± 1.3	7.6 ± 1.8	13.0 ± 0.1	13.3 ± 0.4	12.6 ± 1.0	10.6 ± 1.6	6.9 ± 0.1	4.9 ± 0.4
Total % recovery	99.8 ± 5.9	99.3 ± 8.7	98.2 ± 2.9	94.7 ± 0.9	103.6 ± 7.6	97.3 ± 3.2	95.3 ± 4.2	93.6 ± 0.2	86.2 ± 0.7	79.0 ± 0.8

N/A is not applicable because not sampled.

Data were obtained from Table 9 and Appendix C, pp. 57 and 94 of the study report.

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Table 6: Biotransformation of [2,6-¹⁴C]aminopyralid, expressed as percentage of applied radioactivity (n=2; mean ± s.d.), in Holdrege silt loam, 120 g/ha rate, under aerobic conditions.

Compound	Sampling times (days)							
	0	8	14	33	63	91	190	365
Parent compound	102.2 ±3.5	89.1 ±3.0	78.5 ±1.1	56.9 ±4.5	27.7 ±0.4	24.6 ±12.4	5.9 ±0.6	3.7 ±0.4
Total extractable residues	Total extractables are assumed to be the parent chemical							
CO ₂	N/A	8.1 ±0.3	12.9 ±0.1	30.0 ±5.0	55.1 ±1.4	52.9 ±11.2	76.2 ±0.6	69.3 ±1.0
Total volatile organics	Not monitored							
Non-extractable residues	0.1 ±0.0	4.9 ±0.2	5.1 ±0.5	8.4 ±0.4	13.3 ±0.4	12.3 ±2.1	11.3 ±0.2	5.4 ±0.6
Total % recovery	102.3 ±3.5	102.0 ±3.5	96.4 ±0.6	95.3 ±0.9	96.1 ±1.5	89.7 ±0.9	93.3 ±1.0	78.4 ±1.2

N/A is not applicable because not sampled.

Data were obtained from Table 10 and Appendix C, pp. 58 and 95 of the study report.

Note that for the remaining transformation studies, summary tables are presented below as the original tables from the applicant. Material balance in each case was adjusted as in the above table for the parent chemical and the total % recovery to include all sampling dates, some of which were listed as "MB" according to the raw data presented in Appendix C, pg. 93-106. The adjusted material balance was used in all half-life and DT50 calculations as well as in the reporting of material balance in this document.

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Table 8: Biotransformation of XDE-750, expressed as percentage of applied radioactivity (average 92.4± 4.4), in Regent (Loam), 60 g a.i./ha rate, under aerobic conditions.

	Rep. No.	Sampling Times (DAT)									
		0	8	14	33	63	91	124	190	274	365
Volatiles											
CO ₂	1	N/A	13.9	23.2	56.0	MB(1)	68.4	71.3	72.3	71.1	66.1
	2	N/A	13.4	24.4	55.1	67.4	66.9	71.1	72.1	72.6	MB
	Mean	N/A	13.7	23.8	55.6	67.4	67.7	71.2	72.3	71.9	66.1
Extractable Radioactivity											
0.01 M CaCl ₂	1	74.0	N/A	27.8	4.7	N/A	N/A	N/A	N/A	N/A	N/A
	2	72.3	N/A	26.6	5.3	N/A	N/A	N/A	N/A	N/A	N/A
	Mean	73.2	N/A	27.2	5.0	N/A	N/A	N/A	N/A	N/A	N/A
90:10 Acetone: 1.0 N HCl	1	26.3	75.6	28.5	9.9	MB	7.9	5.1	2.3	1.7	1.6
	2	26.1	77.6	28.2	12.4	5.7	5.7	4.9	2.3	2.7	MB
	Mean	26.2	76.6	28.4	11.2	5.7	6.8	5.0	2.3	2.2	1.6
Total (2)	1	100.4	75.6	56.3	14.5	MB	7.9	5.1	2.3	1.7	1.6
	2	98.3	77.6	54.9	17.7	5.7	5.7	4.9	2.3	2.7	MB
	Mean	99.4	76.6	55.6	16.1	5.7	6.8	5.0	2.3	2.2	1.6
Bound Residue	1	0.0	8.1	10.9	21.0	MB	18.5	19.8	15.7	15.4	17.9
	2	0.0	8.2	9.9	17.0	15.8	18.7	18.7	15.7	12.3	MB
	Mean	0.0	8.2	10.4	19.0	15.8	18.6	19.3	15.7	13.9	17.9
Material Balance	1	100.4	97.5	90.4	91.5	MB	94.8	96.2	90.3	88.2	85.5
	2	98.3	99.3	89.2	89.8	88.9	91.3	94.7	90.1	87.5	MB
	Mean	99.4	98.4	89.8	90.7	88.9	93.1	95.5	90.2	87.9	85.5

- (1) MB = Material Balance less than 85% or greater than 110%
- (2) Since XDE-750 was the only peak detected in all HPLC chromatograms, percent of applied radioactivity in the Total extracted pool is equivalent to percent of applied as XDE-750

Data were obtained from Table 11, p. 59 of the study report.

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Table 9: Biotransformation of XDE-750, expressed as percentage of applied radioactivity (average 90.7 ± 5.3), in Regent (Loam), 120 g a.i./ha rate, under aerobic conditions.

	Rep. No.	Sampling Times (DAT)							
		0	8	14	33	63	91	190	365
Volatiles									
CO ₂	1	N/A	9.5	MB(1)	47.1	69.1	64.2	70.3	MB
	2	N/A	9.5	18.0	46.9	68.3	MB	71.5	MB
	Mean	N/A	9.5	18.0	47.0	65.1	64.2	70.9	MB
Extractable Radioactivity									
0.01 M CaCl ₂	1	80.4	N/A	MB	10.5	N/A	N/A	N/A	N/A
	2	84.6	N/A	32.9	9.6	N/A	N/A	N/A	N/A
	Mean	82.5	N/A	32.9	10.1	N/A	N/A	N/A	N/A
90:10 Acetone: 1.0 N HCl	1	18.1	77.3	MB	14.7	5.0	3.4	2.1	MB
	2	17.4	72.3	30.3	14.3	4.2	MB	2.2	MB
	Mean	17.8	74.8	30.3	14.5	4.6	3.4	2.2	MB
Total (2)	1	98.5	77.3	MB	25.1	5.0	3.4	2.1	MB
	2	102.1	72.3	63.2	23.9	4.2	MB	2.2	MB
	Mean	100.3	74.8	63.2	14.5	4.6	3.4	2.2	MB
Bound Residue	1	0.8	6.6	MB	14.8	16.8	18.2	14.9	MB
	2	0.7	5.9	8.3	15.8	16.8	MB	14.6	MB
	Mean	0.8	6.3	8.3	15.3	16.8	18.2	14.8	MB
Material Balance	1	99.3	93.4	MB	87.1	90.9	85.8	87.4	MB
	2	102.7	87.7	89.6	86.6	89.3	MB	88.3	MB
	Mean	101.0	90.6	89.6	86.9	90.1	85.8	87.9	MB

- (1) MB = Material Balance less than 85% or greater than 110%
- (2) Since XDE-750 was the only peak detected in all HPLC chromatograms, percent of applied radioactivity in the Total extracted pool is equivalent to percent of applied as XDE-750

Data were obtained from Table 12, p. 60 of the study report.

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Table 10: Biotransformation of XDE-750, expressed as percentage of applied radioactivity (average 91.8 ± 6.3), in Manning (Sandy Loam), 60 g a.i./ha rate, under aerobic conditions.

	Rep. No.	Sampling Times (DAT)							
		0	8	14	33	63	91	124	190
Volatiles									
CO ₂	1	N/A	11.0	20.0	MB(1)	69.9	MB	MB	MB
	2	N/A	10.6	19.7	MB	69.0	MB	70.6	MB
	Mean	N/A	10.8	19.9	MB	69.5	MB	70.6	MB
Extractable Radioactivity									
0.01 M CaCl ₂	1	87.5	N/A	46.0	N/A	N/A	N/A	N/A	MB
	2	86.9	N/A	45.6	N/A	N/A	N/A	N/A	MB
	Mean	87.2	N/A	45.8	N/A	N/A	N/A	N/A	MB
90:10 Acetone: 1.0 N HCl	1	15.0	79.2	16.6	MB	5.0	MB	MB	MB
	2	13.5	81.4	17.3	MB	4.9	MB	3.2	MB
	Mean	14.3	80.3	33.9	MB	5.0	MB	3.2	MB
Total (2)	1	102.5	79.2	62.7	MB	5.0	MB	MB	MB
	2	100.5	81.4	62.9	MB	4.9	MB	MB	MB
	Mean	101.5	80.3	62.8	MB	5.0	MB	MB	MB
Bound Residue	1	0.1	2.9	5.1	MB	11.8	MB	MB	MB
	2	0.1	2.7	4.8	MB	11.8	MB	14.1	MB
	Mean	0.1	2.8	5.0	MB	11.8	MB	14.1	MB
Material Balance	1	102.6	93.1	87.7	MB	86.7	MB	MB	MB
	2	100.5	94.7	87.4	MB	85.6	MB	87.9	MB
	Mean	101.6	93.9	87.6	MB	86.2	MB	87.9	MB

(1) MB = Material Balance less than 85% or greater than 110%

(2) Since XDE-750 was the only peak detected in all HPLC chromatograms, percent of applied radioactivity in the Total extracted pool is equivalent to percent of applied as XDE-750

Data were obtained from Table 13, p. 61 of the study report.

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Table 11: Biotransformation of XDE-750, expressed as percentage of applied radioactivity (average 91.8 ± 7.9), in Manning (Sandy Loam), 120 g a.i./ha rate, under aerobic conditions.

	Rep. No.	Sampling Times (DAT)						
		0	8	14	33	63	91	190
Volatiles								
CO ₂	1	N/A	8.1	14.8	MB(1)	70.1	MB	MB
	2	N/A	8.5	14.0	MB	69.3	MB	MB
	Mean	N/A	8.3	14.4	MB	69.7	MB	MB
Extractable Radioactivity								
0.01 M CaCl ₂	1	102.5	N/A	54.2	N/A	N/A	N/A	N/A
	2	93.8	N/A	55.5	N/A	N/A	N/A	N/A
	Mean	98.2	N/A	54.9	N/A	N/A	N/A	N/A
90:10 Acetone: 1.0 N HCl	1	5.7	76.3	17.6	N/A	4.2	MB	MB
	2	4.3	76.1	19.1	N/A	5.0	MB	MB
	Mean	5.0	76.2	18.4	N/A	4.6	MB	MB
Total (2)	1	108.2	76.3	71.8	N/A	4.2	MB	MB
	2	98.0	76.1	74.6	N/A	5.0	MB	MB
	Mean	103.1	76.2	73.2	N/A	4.6	MB	MB
Bound Residue								
	1	0.2	2.8	2.7	N/A	10.3	MB	MB
	2	0.2	3.1	3.1	N/A	13.2	MB	MB
	Mean	0.2	3.0	2.9	N/A	11.8	MB	MB
Material Balance								
	1	108.4	87.1	89.3	N/A	84.7	MB	MB
	2	98.2	87.6	91.7	N/A	87.5	MB	MB
	Mean	103.3	87.4	90.5	N/A	86.1	MB	MB

- (1) MB = Material Balance less than 85% or greater than 110%
- (2) Since XDE-750 was the only peak detected in all HPLC chromatograms, percent of applied radioactivity in the Total extracted pool is equivalent to percent of applied as XDE-750

Data were obtained from Table 14, p. 62 of the study report.

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Table 12: Biotransformation of XDE-750, expressed as percentage of applied radioactivity (average 91.1 ± 4.3), in Barnes (Clay Loam), 60 g a.i./ha rate, under aerobic conditions.

	Rep. No.	Sampling Times (DAT)									
		0	8	14	33	63	91	124	190	274	365
Volatiles											
CO ₂	1	N/A	0.9	1.2	2.6	4.8	8.2	11.0	16.6	23.3	28.1
	2	N/A	MB(1)	1.2	2.5	5.7	8.3	11.5	MB	21.9	MB
	Mean	N/A	0.9	1.2	2.6	5.3	8.3	11.3	16.6	22.6	28.1
Extractable Radioactivity											
0.01 M CaCl ₂	1	60.6	N/A	33.6	38.2	N/A	N/A	N/A	N/A	N/A	N/A
	2	66.5	N/A	35.0	36.9	N/A	N/A	N/A	N/A	N/A	N/A
	Mean	63.6	N/A	34.3	37.6	N/A	N/A	N/A	N/A	N/A	N/A
90:10 Acetone: 1.0 M HCl	1	30.7	83.2	52.6	52.7	82.7	71.8	68.7	62.1	51.1	45.2
	2	25.9	MB	56.7	56.4	79.8	75.3	69.6	MB	52.6	MB
	Mean	28.3	83.2	54.7	54.6	81.25	73.6	69.2	62.1	51.9	45.2
Total (2)	1	91.3	83.2	86.2	90.9	82.7	71.8	68.7	62.1	51.1	45.2
	2	92.4	MB	91.7	93.2	79.8	75.3	69.6	N/A	52.6	MB
	Mean	28.3	83.2	54.7	54.6	81.25	73.6	69.2	62.1	51.9	45.2
Bound Residue											
	1	0.1	2.2	3.0	4.8	6.3	6.9	8.6	10.9	11.3	14.6
	2	0.0	MB	2.8	5.0	6.0	8.1	11.1	MB	11.3	MB
	Mean	.01	2.2	2.9	4.9	6.2	7.5	9.9	10.9	11.3	14.6
Material Balance											
	1	91.4	86.3	90.4	98.3	93.8	86.9	88.4	89.6	85.7	87.9
	2	92.4	MB	95.7	100.7	91.5	91.7	92.2	N/A	85.8	MB
	Mean	91.9	86.3	91.05	99.5	92.7	89.3	90.3	89.6	85.8	87.9

- (1) MB = Material Balance less than 85% or greater than 110%
- (2) Since XDE-750 was the only peak detected in all HPLC chromatograms, percent of applied radioactivity in the Total extracted pool is equivalent to percent of applied as XDE-750

Data were obtained from Table 15, p. 63 of the study report.

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Table 13: Biotransformation of XDE-750, expressed as percentage of applied radioactivity (average 96.4 ± 5.34), in Barnes (Clay Loam), 120 g a.i./ha rate, under aerobic conditions.

	Rep. No.	Sampling Times (DAT)							
		0	8	14	33	63	91	190	365
Volatiles									
CO ₂	1	N/A	0.8	1.3	3.6	6.1	8.0	18.1	MB
	2	N/A	MB(1)	MB	2.9	MB	9.4	18.1	MB
	Mean	N/A	0.8	1.3	3.3	6.1	8.7	18.1	MB
Extractable Radioactivity									
0.01 M CaCl ₂	1	66.5	N/A	33.7	34.5	N/A	N/A	N/A	N/A
	2	75.9	N/A	MB	35.2	N/A	N/A	N/A	N/A
	Mean	71.2	N/A	33.7	34.9	N/A	N/A	N/A	N/A
90:10 Acetone: 1.0 M HCl	1	19.6	104.4	51.0	48.6	76.9	73.1	63.9	MB
	2	27.2	MB	MB	48.6	MB	79.2	64.9	MB
	Mean	23.4	104.4	51.0	48.6	76.9	76.2	64.4	MB
Total (2)	1	86.1	104.4	84.7	83.2	76.9	73.1	63.9	MB
	2	103.1	MB	MB	83.8	MB	79.2	64.9	MB
	Mean	94.6	104.4	84.7	83.5	76.9	76.2	64.4	MB
Bound Residue	1	0.7	3.6	2.6	4.6	7.1	7.7	12.2	MB
	2	0.8	MB	MB	4.8	MB	9.7	11.0	MB
	Mean	0.8	3.6	2.6	4.7	7.1	8.7	11.6	MB
Material Balance	1	86.9	108.7	88.6	91.4	90.2	88.9	94.2	MB
	2	104.0	MB	MB	91.4	MB	98.4	94.0	MB
	Mean	94.5	108.7	88.6	91.4	90.2	93.7	94.1	MB

- (1) MB = Material Balance less than 85% or greater than 110%
 (2) Since XDE-750 was the only peak detected in all HPLC chromatograms, percent of applied radioactivity in the Total extracted pool is equivalent to percent of applied as XDE-750

Data were obtained from Table 16, p. 64 of the study report.

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Table 14: Biotransformation of XDE-750, expressed as percentage of applied radioactivity (average 91.5 ± 3.0), in Houston Black (Clay), 60 g a.i./ha rate, under aerobic conditions.

	Rep. No.	Sampling Times (DAT)							
		0	1	4	8	14	22	31	65
Volatiles									
CO ₂	1	N/A	1.6	13.6	30.4	55.6	55.5	67.5	67.9
	2	N/A	1.8	13.9	MB(1)	57.0	57.3	65.5	67.1
	Mean	N/A	1.7	13.8	30.4	56.3	56.4	66.5	67.5
Extractable Radioactivity									
0.01 M CaCl ₂	1	85.1	N/A	N/A	N/A	4.6	N/A	N/A	N/A
	2	87.1	N/A	N/A	N/A	2.9	N/A	N/A	N/A
	Mean	86.1	N/A	N/A	N/A	3.8	N/A	N/A	N/A
90:10 Acetone: 1.0 M HCl	1	0.0	65.9	47.2	20.7	3.1	1.9	2.2	1.3
	2	0.2	63.6	45.4	MB	2.7	1.4	1.2	1.4
	Mean	0.1	64.8	46.3	20.7	2.9	1.7	1.7	1.4
Total (2)	1	85.1	65.9	47.2	20.7	7.7	1.9	2.2	1.3
	2	87.3	63.6	45.4	MB	5.6	1.4	1.2	1.4
	Mean	86.2	64.8	46.3	20.7	6.7	1.7	1.7	1.4
Bound Residue	1	7.8	28.3	28.4	35.3	27.3	37.6	24.8	22.5
	2	8.4	26.6	28.9	MB	25.9	29.3	25.1	23.7
	Mean	8.1	27.5	28.7	35.3	26.6	33.5	25.0	23.1
Material Balance	1	92.9	95.9	89.2	86.5	90.6	95.0	94.6	91.7
	2	95.7	92.1	88.1	MB	88.5	88.1	91.8	92.1
	Mean	94.3	94.0	88.7	86.5	90.0	95.6	93.2	91.9

(1) MB = Material Balance less than 85% or greater than 110%

(2) Since XDE-750 was the only peak detected in all HPLC chromatograms, percent of applied radioactivity in the Total extracted pool is equivalent to percent of applied as XDE-750

Data obtained from Table 17, p. 65 of the study report.

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Table 15: Biotransformation of XDE-750, expressed as percentage of applied radioactivity (average 93.4 ± 6.9), in Houston Black (Clay), 120 g a.i./ha rate, under aerobic conditions.

	Rep. No.	Sampling Times (DAT)							
		0	1	4	8	14	22	31	65
Volatiles									
CO ₂	1	N/A	1.2	9.2	41.2	43.3	56.8	63.2	67.5
	2	N/A	1.3	10.1	MB(1)	39.2	MB	63.2	67.2
	Mean	N/A	1.3	9.7	41.2	41.3	56.38	63.2	67.4
Extractable Radioactivity									
0.01 M CaCl ₂	1	81.4	N/A	N/A	N/A	9.6	N/A	N/A	N/A
	2	89.7	N/A	N/A	N/A	19.8	N/A	N/A	N/A
	Mean	85.6	N/A	N/A	N/A	14.7	N/A	N/A	N/A
90:10 Acetone: 1.0 M HCl	1	2.6	63.2	52.8	32.3	6.3	1.7	1.2	1.1
	2	6.4	63.4	53.3	MB	9.9	MB	1.2	1.0
	Mean	4.5	63.3	53.1	32.3	8.1	1.7	1.2	1.1
Total (2)	1	84.1	63.2	52.8	32.3	16.0	1.7	1.2	1.1
	2	96.1	63.4	53.3	MB	29.6	MB	1.2	1.0
	Mean	90.1	63.3	53.1	32.3	22.8	1.7	1.2	1.1
Bound Residue	1	8.6	26.0	29.9	37.1	25.7	31.2	24.5	25.2
	2	10.5	25.3	29.8	MB	25.3	MB	24.2	23.4
	Mean	9.6	25.7	29.9	37.1	25.5	31.2	24.4	24.3
Material Balance	1	92.6	90.4	91.9	110.5	85.0	89.7	89.0	93.9
	2	106.6	90.1	93.2	MB	94.2	MB	88.6	91.6
	Mean	99.6	90.3	92.6	110.5	89.6	98.7	88.8	92.8

(1) MB = Material Balance less than 85% or greater than 110%

(2) Since XDE-750 was the only peak detected in all HPLC chromatograms, percent of applied radioactivity in the Total extracted pool is equivalent to percent of applied as XDE-750

Data obtained from Table 18, p. 66 of the study report.

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C. TRANSFORMATION OF PARENT COMPOUND:

For Holdrege silt loam at 60 g/ha, the concentration of the parent compound decreased from 99.7% of the applied amount at Day 0 to 55.8% of the applied at 33 DAT and 3.8% of the applied at the end of the study period (365 DAT). At 120 g/ha, the concentration of the parent compound decreased from 102.2% of the applied amount at Day 0 to 56.9% of the applied at 33 DAT and 3.7% of the applied at the end of the study period (Tables 9-10 and Appendix C, pp. 57-58, 94-95).

For Regent loam at 60 g/ha, the concentration of the parent compound decreased from 99.4% of the applied amount at Day 0 to 55.6% of the applied at 14 DAT and 1.5% of the applied at the end of the study period (365 DAT). At 120 g/ha, the concentration of the parent compound decreased from 100.3% of the applied amount at Day 0 to 60.8% of the applied at 14 DAT, then 24.6% of the applied at 33 DAT and 1.6% of the applied at the end of the study period (Tables 11-12 and Appendix C, pp. 59-60, 97-98).

For Manning sandy loam at 60 g/ha, the concentration of the parent compound decreased from 101.5% of the applied amount at Day 0 to 62.8% of the applied radioactivity at 14 DAT, then 31.5% of the applied radioactivity at 33 DAT, and 2.6% of the applied at the end of the study period (190 DAT). At 120 g/ha, the concentration of the parent compound decreased from 103.2% of the applied amount at Day 0 to 73.2% of the applied radioactivity at 14 DAT, then 30.9% of the applied radioactivity at 33 DAT and 2.3% of the applied at the end of the study period (Tables 13-14 and Appendix C, pp. 61-62, 100-101).

For Barnes clay loam at 60 g/ha, the concentration of the parent compound was 91.9% of the applied amount at Day 0, ranged from 81.3 to 92.1% of the applied from 8 to 63 DAT, and decreased to 42.5% of the applied at the end of the study period (365 DAT). At 120 g/ha, the concentration of the parent compound decreased from 94.6% of the applied amount at Day 0 to 91.4% of the applied at 8 DAT, ranged from 69.0 to 83.5% of the applied from 14 to 91 DAT, and decreased to 41.1% of the applied at the end of the study period (Tables 15-16 and Appendix C, pp. 63-64, 103-104).

For Houston Black clay at 60 g/ha, the concentration of the parent compound decreased from 86.2% of the applied amount at Day 0 to 46.3% of the applied at 4 DAT, and 1.4% of the applied at the end of the study period (65 DAT). At 120 g/ha, the concentration of the parent compound decreased from 90.1% of the applied amount at Day 0 to 53.1% of the applied at 4 DAT, and 1.1% of the applied at the end of the study period (Tables 17-18 and Appendix C, pp. 65-66, 105-106).

Fifty percent transformation occurred in all soils within 60 days, with the exception of the Barnes clay, where [2,6-¹⁴C]aminopyralid was persistent. Also, in all soils except for the Barnes clay, an initial rapid transformation phase was followed by a second slow transformation phase, with

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detectable levels of the parent material after 365 days. However, due to non-harsh extraction procedures, it is not clear whether study data accounted for all parent. It is possible that some amount of parent remained not extracted and was instead counted as non-extractable material.

HALF-LIFE: First-order, log-transformed half-lives of aminopyralid and aminopyralid plus non-extractables were calculated, using all data points and a first-order linear regression analysis in Excel. First-order, non-transformed half-lives of aminopyralid and aminopyralid plus non-extractables were also calculated, using all data points and a first-order nonlinear regression analysis in Sigma Plot (Tables 9-16 and Appendix C, pp. 57-65, 94-95, 97-98, 100-101, and 103-105; Attachment 1). In all cases, with the exception of the Barnes clay loam, the non-linear first-order regressions produced a better fit to the initial degradation data than the linear first order regressions, which tended to overestimate the DT50 (empirical time required for 50% transformation). However, nonlinear regression underestimated chronic concentrations. Therefore, while the nonlinear half-life values represent a better estimate of the initial transformation rate of [2,6-¹⁴C]aminopyralid in aerobic soil, linear half-life values ensure that chronic concentrations are not underestimated. Due to material balance problems, the only half-life estimates not of questionable value are those calculated for Holdrege silt loam..

For Holdrege silt loam, the first order nonlinear half-life of [2,6-¹⁴C]aminopyralid in aerobic soil was 42.0 days (60 g/ha) and 38.7 days (120 g/ha). Nonlinear first order half-lives were consistent with empirical DT50s (45.0 days for both 60 g/ha and 120 g/ha), while linear first order half-lives overestimated empirical ones (73.0 days for both 60 g/ha and 120 g/ha). When half-lives were calculated assuming non-extractible residues were composed of non-extracted parent (at 120 g/ha), the first order linear half-life was 103.5 days, the first order nonlinear half-life was 58.2 days, and the empirical DT50 was 70.0 days.

For Regent loam, the first order nonlinear half-life of [2,6-¹⁴C]aminopyralid in aerobic soil was 19.8 days (60 g/ha) and 17.0 days (120 g/ha). Nonlinear first order half-lives were slightly less than empirical DT50s (15.0 and 20.0 days for 60 g/ha and 120 g/ha, respectively), while linear first order half-lives overestimated empirical ones (61.9 and 62.4 days for 60 g/ha and 120 g/ha, respectively). When half-lives were calculated assuming non-extractible residues were composed of non-extracted parent (at 120 g/ha), the first order linear half-life was 67.3 days, the first order nonlinear half-life was 30.4 days, and the empirical DT50 was 30.0 days. However, all DT50s and half-lives are of questionable value due to material balance problems.

For Manning sandy loam, the first order nonlinear half-life of [2,6-¹⁴C]aminopyralid in aerobic soil was 18.8 days (60 g/ha) and 19.4 days (120 g/ha). Nonlinear first order half-lives were slightly less than empirical DT50s (20.0 and 22.0 days for 60 g/ha and 120 g/ha, respectively), while linear first order half-lives overestimated empirical ones (32.4 and 31.9 days for 60 g/ha and 120 g/ha, respectively). When half-lives were calculated assuming non-extractible residues were composed of non-extracted parent (at 120 g/ha), the first order linear half-life was 55.9

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days, the first order nonlinear half-life was 25.8 days, and the empirical DT50 was 27.0 days. However, all DT50s and half-lives are of questionable value due to low material balances.

For Barnes clay loam, the first order linear half-life of [2,6-¹⁴C]aminopyralid in aerobic soil was 330.1 days (both 60 g/ha and 120 g/ha). Nonlinear first order half-lives were identical to linear ones for both rates, and slightly greater than DT50s (290 and 300 days for 60 g/ha and 120 g/ha, respectively). When half-lives were calculated assuming non-extractible residues were composed of non-extracted parent (at 120 g/ha), the both first order linear and nonlinear half-lives were 533.2 days, and there was no empirical DT50, as parent did not degrade to less than 50% of applied by study completion. However, all DT50s and half-lives are of questionable value due to variability and fluctuation of material balances.

For Houston Black clay, the first order nonlinear half-life of [2,6-¹⁴C]aminopyralid in aerobic soil was 4.0 days (60 g/ha) and 6.1 days (120 g/ha). Nonlinear first order half-lives were more representative of empirical DT50s (3.5 and 4.5 days for 60 g/ha and 120 g/ha, respectively) than linear first order half-lives (10.4 and 9.4 days for 60 g/ha and 120 g/ha, respectively). When half-lives were calculated assuming non-extractible residues were composed of non-extracted parent (at 120 g/ha), the first order linear half-life was 31.5 days, the first order nonlinear half-life was 17.9 days, and the empirical DT50 was 13.0 days. However, all DT50s and half-lives are of questionable value due to variable material balances.

Half-lives/DT50:

Soil type	DT50 (days)	First order half-life, linear regression ¹		First order half-life, non-linear regression ¹	
		half-life (days)	R ²	half-life (days)	R ²
Holdrege 60 g/ha	50	73	0.9003	42	0.9866
Holdrege 120g/ha	60	73	0.8809	38.7	0.9839
Regent 60 g/ha	15	61.9	0.7565	19.8	0.8799
Regent 120 g/ha	20	62.4	0.6505	17	0.9951
Manning 60 g/ha	20	32.4	0.7914	18.8	0.985
Manning 120 g/ha	22	31.9	0.7675	19.4	0.9829
Barnes 60 g/ha	290	330.1	0.9635	330.1	0.9481
Barnes 120 g/ha	300	330.1	0.8615	330.1	0.7952
Houston 60 g/ha	3.5	10.4	0.6787	4	0.9903
Houston 120 g/ha	4.5	9.4	0.7707	6.1	0.9481

Data were obtained from Tables 9-18 and Appendix C, pp. 57-66, 94-95, 97-98, 100-101, and 103-106 of the study report.

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¹See Appendix A of this DER for plots of linear and nonlinear regression and for equations of the models used.

Half-lives/DT50 treating non-extractable residues as parent:

Soil type	DT50 (days)	First order half-life, linear regression ¹		First order half-life, non-linear regression ¹	
		half-life (days)	R ²	half-life (days)	R ²
Holdrege 120g/ha	70	103.5	0.9240	58.2	0.9721
Regent 120 g/ha	30	63.7	0.9282	30.4	0.9543
Manning 120 g/ha	27	55.9	0.7654	25.8	0.9747
Barnes 120 g/ha	N/A	533.2	0.7419	533.2	0.6727
Houston 120 g/ha	13	31.5	0.7543	17.9	0.8989

Data were obtained from Tables 9-18 and Appendix C, pp. 57-66, 94-95, 97-98, 100-101, and 103-106 of the study report.
¹See Appendix A of this DER for plots of linear and nonlinear regression and for equations of the models used.

TRANSFORMATION PRODUCTS: The major transformation products detected were CO₂ and non-extractable residues. No other degradation products were observed. All the extractable radioactivity was quantified as [2,6-¹⁴C]aminopyralid (p. 42; Figure 6-9, pp. 75-78).

NON-EXTRACTABLE AND EXTRACTABLE RESIDUES: For Holdrege silt loam, Regent loam, Manning sandy loam, and Houston Black clay treated at 60 g/ha, extractable [¹⁴C]residues decreased from 99.7, 99.4, 101.5, and 86.2% of the applied amount at Day 0 to 3.8, 1.5, 2.6, and 1.4% of applied at the end of the study period (365, 365, 190, and 65 DAT), respectively. Similarly, for Holdrege silt loam, Regent loam, Manning sandy loam, and Houston Black clay treated at 120 g/ha, extractable [¹⁴C]residues decreased from 102.2, 100.3, 103.2, and 90.1% of the applied amount at Day 0 to 3.7, 1.6, 2.3, and 1.1% of applied at the end of the study period (365, 365, 190, and 65 DAT), respectively. For Barnes clay loam, however, extractable [¹⁴C]residues decreased from 91.9 and 94.6% of the applied amount at Day 0 to 42.5 and 41.1% of applied at the end of the study period (365 DAT) for 60 g/ha and 120 g/ha, respectively. Non-extractable [¹⁴C]-residues increased from about 0% of the applied at Day 0 to 4.9, 15.7, 9.0, and 13.7% of the applied for 60 g/ha and to 5.4, 0.0, 8.3, and 14.0% of the applied for 120 g/ha at the end of the incubation period in Holdrege silt loam, Regent loam, Manning sandy loam, and Barnes clay loam, respectively. Non-extractable [¹⁴C]-residues in Houston Black clay increased from 8.1 and 9.6 % of the applied at Day 0 to 23.1 and 24.3% of the applied at the end of the incubation period (65 DAT) for 60 g/ha and 120 g/ha, respectively.

Organic matter from non-extractable residues in Holdrege silt loam, Regent loam, Manning sandy loam, and Houston Black clay dosed at 60 g/ha at 91, 91, 124, and 8 days, respectively, consisted of fulvic acid fractions of 7.5, 7.5, 7.6, and 23.0 % of the applied, humic fractions of 2.5, 3.8, 0.2, and 1.0 % of the applied, and humin fractions of 3.0, 7.3, 3.3, and 11.4 %. Organic

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matter from non-extractable residues in Holdrege silt loam, Regent loam, Manning sandy loam, and Houston Black clay dosed at 120 g/ha at 190, 63, 190, and 14 days, respectively, consisted of fulvic acid fractions of 5.2, 8.0, 10.2, and 11.7 % of the applied, humic fractions of 2.2, 4.2, 0.8, and 0.9 % of the applied, and humin fractions of 4.1, 4.5, 1.1, and 12.8 % (Table 4, p. 52).

VOLATILIZATION: At study termination, evolved $^{14}\text{CO}_2$ were 70.3, 65.7, 73.2, and 67.5% of the applied at 60 g/ha and 69.3, 66.9, 71.5, and 67.4% of the applied at 120 g/ha, in Holdrege silt loam, Regent loam, Manning sandy loam, and Houston Black clay, respectively. In the Barnes clay loam, 30.0 and 27.0% of the applied radioactivity was CO_2 at study termination at 60 g/ha and 120 g/ha, respectively (Tables 9-18 and Appendix C, pp. 57-66, 94-95, 97-98, 100-101, and 103-106). Volatile organics were not measured.

TRANSFORMATION PATHWAY: The primary route of [2,6- ^{14}C]aminopyralid aerobic soil metabolism is mineralization to CO_2 . Incorporation to non-extractable soil residues is also a critical component of [2,6- ^{14}C]aminopyralid degradation. No degradates, other than CO_2 and bound residues, were detected in this study.

D. SUPPLEMENTARY EXPERIMENT-RESULTS: Application rates between 5- and 120-g a.i./ha had no impact on the half-life of [2,6- ^{14}C]aminopyralid. Non-equilibrium sorption coefficients at 0 DAT ranged from 0.1 to 1.2 L/kg. In all five of the soils tested, the apparent K_d values showed a 3 to 10-fold increase from 0 DAT to 14 DAT, indicating that contact-time with soil increases [2,6- ^{14}C]aminopyralid sorption potential.

III. STUDY DEFICIENCIES: Material balance was poor at several sampling dates during the study. Specifically, for soils dosed at the required maximum application rate (120 g/ha), there was a material balance loss greater than 10% of the applied between sampling intervals 0 and 8 DAT for Regent loam and Manning sandy loam, and a material balance gain greater than 10% of the applied between the same intervals for Barnes clay loam. For Houston Black clay, there were losses of 9.4% and 8.3% between intervals 0 and 1 DAT, and 14 and 22 DAT, respectively. These large fluctuations in material balance seriously put into question the validity of measurements of parent radioactivity across these intervals and throughout the study for the Regent loam, Manning sandy loam, Barnes clay loam and Houston Black clay. In addition, 41 of the 78 reported material balances across all soils for all replicates were outside the range of 90-110% of the applied.

The method used for extraction (90:10 acetone:1N HCl) was not harsh enough to ensure with certainty that radioactivity attributed to non-extractable residue was truly not able to be extracted. It is possible that the radioactivity attributed to non-extractable residue was actually parent which just had not been extracted from the soil. Additionally, because aerobic soil metabolism in Barnes clay loam was not valid, the Subdivision N requirement that one of the soils tested in the adsorption/desorption study also be tested in the aerobic soil metabolism study was not fulfilled.

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Also, samples were not collected at days 1 and 4 as outlined in USEPA Subdivision N Guidelines §162-1, except in one of the five soils (Houston Black clay). A coupled detector was not used with the HPLC for the analysis of the soil extracts. Samples were not analyzed with more sophisticated analytical equipment such as an LC-MS to confirm HPLC findings that extracted radioactivity was present as parent and no transformation products were formed. Volatile organics were not monitored. Redox potential was not determined.

IV. REVIEWER'S COMMENTS:

1.) Although soil samples were not collected on days 1 and 4, at least 3 data points were available for each soil study within the initial rapid transformation phase, therefore this should not affect the calculated half-life values.

2.) HPLC and radiometric detection were the only methods used in the studies to identify and quantitate the parent chemical. HPLC fractions were collected only once per minute and subsequently analyzed by an LSC. This resulted in broad peaks, with 3-5 minute elution times on representative radiochromatograms. The applicant could have used a coupled detector such as UV at 270 nm in this study, as was done in the hydrolysis and other studies. These result in much higher resolution and thus higher certainty of identification of the parent.

3.) The authors did not use in calculations intervals which had material balance values outside of the 85-110% range. For the purpose of this DER, as per USEPA policy, all data points (reported in Appendix C, pp. 91-106) irrespective of material balance were used in calculating interval averages and transformation rates.

4.) The authors incorrectly calculated and reported average total extractable % applied radioactivity for the following intervals:

Soil	Rate	Interval	Replicate 1	Replicate 2	Reported average	Actual average
Holdrege	120 g/ha	33 DAT	53.6	60.1	33.4	56.9
Regent	120 g/ha	33 DAT	25.1	23.9	14.5	24.5
Barnes	60 g/ha	0 DAT	91.3	92.4	28.3	91.9
Barnes	60 g/ha	14 DAT	86.2	91.7	54.7	89
Barnes	60 g/ha	33 DAT	90.9	93.2	54.6	92.1

(Data were obtained in Tables 10, 12, and 15; pp. 58, 60, and 63)

5.) The authors used first order kinetics with linear regression on log-transformed data to calculate half-lives, which overestimated the true 50% transformation time (DT50) of

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aminopyralid in aerobic soil. Therefore, a first-order, two-parameter nonlinear regression on non-transformed data was used to better approximate the transformation process of [2,6-¹⁴C]aminopyralid. Under USEPA guidelines, only half-lives calculated at the maximum use rate (in this case, 120 g/ha) are to be used as modeling input.

6.) Although the authors state that CO₂ loss from the NaOH traps most likely resulted in the low recovery of radioactivity, it is not clear how the experimental apparatus allowed for this to occur. Also, it is not clear how oxygen was supplied to the system, based on the information provided. Although the loss of radioactivity from the NaOH traps does not have an impact on the transformation of the parent material, lower than optimal recovery of unproven cause calls into question the transformation estimate results.

V. REFERENCES:

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EPA MRID Number 46235729

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Appendix A:

Plots of the aerobic biotransformation of [2,6-¹⁴C]aminopyralid in five North American soils. Each set of plots shows the measured percent parent material, the first-order log-transformed dissipation curve with linear regression and the first-order nonlinear models fitted to the measured untransformed data.

Chemical: Aminopyralid
 PC Code: 005100
 MRID: 48235729
 Guideline No: 161-2

Table 5 Biotransformation of [2,6-C14]aminopyralid, expressed as percentage of applied radioactivity in Holdrege silt loam, 60 g/ha, under aerobic conditions

Time (days)	CaCl ₂ extract	Organic extract	Total extract ¹	Bound residues	CO ₂	Total radioactivity
0	71.4	24.1	95.5	0.1	N/A	95.6
0	79.6	24.3	103.9	0.0	N/A	103.9
AVG	75.5	24.2	99.7	0.1	N/A	99.8
STDEV	5.8	0.1	5.9	0.1	N/A	5.9
8	N/A	93.2	93.2	4.6	7.6	105.4
8	N/A	80.9	80.9	4.2	8.0	93.1
AVG	N/A	87.1	87.1	4.4	7.8	99.3
STDEV	N/A	8.7	8.7	0.3	0.3	8.7
14	35.7	43.6	79.3	7.8	13.1	100.2
14	34.2	41.7	75.9	6.0	14.2	96.1
AVG	35.0	42.7	77.6	6.9	13.7	98.2
STDEV	1.1	1.3	2.4	1.3	0.8	2.9
33	21.2	33.2	54.4	8.8	32.1	95.3
33	23.3	33.9	57.2	6.3	30.5	94.0
AVG	22.3	33.6	55.8	7.6	31.3	94.7
STDEV	1.5	0.5	2.0	1.8	1.1	0.9
63	N/A	41.8	41.8	13.0	54.2	109.0
63	N/A	29.6	29.6	12.9	55.7	98.2
AVG	N/A	35.7	35.7	13.0	55.0	103.6
STDEV	N/A	8.6	8.6	0.1	1.1	7.6
91	N/A	24.7	24.7	13.0	61.8	99.5
91	N/A	19.5	19.5	13.6	61.9	95.0
AVG	N/A	22.1	22.1	13.3	61.9	97.3
STDEV	N/A	3.7	3.7	0.4	0.1	3.2
124	N/A	10.7	10.7	13.3	74.2	98.2
124	N/A	11.3	11.3	11.9	69.1	92.3
AVG	N/A	11.0	11.0	12.6	71.7	95.3
STDEV	N/A	0.4	0.4	1.0	3.6	4.2
190	N/A	6.2	6.2	9.5	77.7	93.4
190	N/A	11.5	11.5	11.7	70.5	93.7
AVG	N/A	8.9	8.9	10.6	74.1	93.6
STDEV	N/A	3.7	3.7	1.6	5.1	0.2
274	N/A	4.5	4.5	7.0	74.2	85.7
274	N/A	4.5	4.5	6.8	75.4	86.7
AVG	N/A	4.5	4.5	6.9	74.8	86.2
STDEV	N/A	0.0	0.0	0.1	0.8	0.7
365	N/A	3.9	3.9	5.2	69.3	78.4
365	N/A	3.7	3.7	4.6	71.2	79.5
AVG	N/A	3.8	3.8	4.9	70.3	79.0
STDEV	N/A	0.1	0.1	0.4	1.3	0.8
						94.7
						22.0
						AVR
						STDEV

¹Total extractables are assumed to be composed of all parent compound.
 Data were obtained from Table 9 and Appendix C, pp. 57 and 94 of the study report.
 N/A= Not Applicable because not sampled.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235729
 Guideline No: 161-2

Table 6 Biotransformation of [2,6-C14]aminopyralid, expressed as percentage of applied radioactivity in Holdrege silt loam, 120 g/ha, under aerobic conditions

Time (days)	CaCl ₂ extract	Organic extract	Total extract ¹	Bound residues	CO ₂	Total radioactivity	
0	83.3	21.4	104.7	0.1	N/A	104.8	
0	78.9	20.8	99.7	0.1	N/A	99.8	
AVG	81.1	21.1	102.2	0.1	N/A	102.3	
STDEV	3.1	0.4	3.5	0.0	N/A	3.5	
8	N/A	91.2	91.2	5.0	8.3	104.5	
8	N/A	86.9	86.9	4.7	7.9	99.5	
AVG	N/A	89.1	89.1	4.9	8.1	102.0	
STDEV	N/A	3.0	3.0	0.2	0.3	3.5	
14	34.9	44.4	79.3	4.7	12.8	96.8	
14	33.8	43.9	77.7	5.4	12.9	96.0	
AVG	34.4	44.2	78.5	5.1	12.9	96.4	
STDEV	0.8	0.4	1.1	0.5	0.1	0.6	
33	21.1	32.6	53.7	8.7	33.5	95.9	
33	25.9	34.2	60.1	8.1	26.4	94.6	
AVG	23.5	33.4	56.9	8.4	30.0	95.3	
STDEV	3.4	1.1	4.5	0.4	5.0	0.9	
63	N/A	27.9	27.9	13.0	54.1	95.0	
63	N/A	27.4	27.4	13.6	56.1	97.1	
AVG	N/A	27.7	27.7	13.3	55.1	96.1	
STDEV	N/A	0.4	0.4	0.4	1.4	1.5	
91	N/A	15.8	15.8	13.7	60.8	90.3	
91	N/A	33.3	33.3	10.8	44.9	89.0	
AVG	N/A	24.6	24.6	12.3	52.9	89.7	
STDEV	N/A	12.4	12.4	2.1	11.2	0.9	
190	N/A	5.4	5.4	11.4	75.8	92.6	
190	N/A	6.3	6.3	11.1	76.6	94.0	
AVG	N/A	5.9	5.9	11.3	76.2	93.3	
STDEV	N/A	0.6	0.6	0.2	0.6	1.0	
365	N/A	3.9	3.9	5.0	68.6	77.5	
365	N/A	3.4	3.4	5.8	70.0	79.2	
AVG	N/A	3.7	3.7	5.4	69.3	78.4	
STDEV	N/A	0.4	0.4	0.6	1.0	1.2	
						94.2	AVR
						7.5	STDEV

¹Total extractables are assumed to be composed of all parent compound.
 Data were obtained from Table 10 and Appendix C, pp. 58 and 95 of the study report.
 N/A= Not Applicable because not sampled.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235729
 Guideline No: 161-2

Table 7 Biotransformation of [2,6-C14]aminopyralid, expressed as percentage of applied radioactivity in Regent loam, 60 g/ha, under aerobic conditions

Time (days)	CaCl ₂ extract	Organic extract	Total extract ¹	Bound residues	CO ₂	Total radioactivity	
0	74.0	26.3	100.3	0.0	N/A	100.3	
0	72.3	26.1	98.4	0.0	N/A	98.4	
AVG	73.2	26.2	99.4	0.0	N/A	99.4	
STDEV	1.2	0.1	1.3	0.0	N/A	1.3	
8	N/A	75.6	75.6	8.1	13.9	97.6	
8	N/A	77.6	77.6	8.2	13.4	99.2	
AVG	N/A	76.6	76.6	8.2	13.7	98.4	
STDEV	N/A	1.4	1.4	0.1	0.4	1.1	
14	27.8	28.5	56.3	10.9	23.2	90.4	
14	26.6	28.2	54.8	9.9	24.4	89.1	
AVG	27.2	28.4	55.6	10.4	23.8	89.8	
STDEV	0.8	0.2	1.1	0.7	0.8	0.9	
33	4.7	9.9	14.6	21.0	56.0	91.6	
33	5.3	12.4	17.7	17.0	55.1	89.8	
AVG	5.0	11.2	16.2	19.0	55.6	90.7	
STDEV	0.4	1.8	2.2	2.8	0.6	1.3	
63	N/A	58.8	58.8	9.1	10.4	78.3	
63	N/A	5.7	5.7	15.8	67.4	88.9	
AVG	N/A	32.3	32.3	12.5	38.9	83.6	
STDEV	N/A	37.5	37.5	4.7	40.3	7.5	
91	N/A	7.9	7.9	18.5	68.4	94.8	
91	N/A	5.7	5.7	18.7	66.9	91.3	
AVG	N/A	6.8	6.8	18.6	67.7	93.1	
STDEV	N/A	1.6	1.6	0.1	1.1	2.5	
124	N/A	5.1	5.1	19.8	71.3	96.2	
124	N/A	4.9	4.9	18.7	71.1	94.7	
AVG	N/A	5.0	5.0	19.3	71.2	95.5	
STDEV	N/A	0.1	0.1	0.8	0.1	1.1	
190	N/A	2.3	2.3	15.7	72.3	90.3	
190	N/A	2.3	2.3	15.7	72.1	90.1	
AVG	N/A	2.3	2.3	15.7	72.2	90.2	
STDEV	N/A	0.0	0.0	0.0	0.1	0.1	
274	N/A	1.7	1.7	15.4	71.1	88.2	
274	N/A	2.7	2.7	12.3	72.6	87.6	
AVG	N/A	2.2	2.2	13.9	71.9	87.9	
STDEV	N/A	0.7	0.7	2.2	1.1	0.4	
365	N/A	1.6	1.6	17.9	66.1	85.6	
365	N/A	1.4	1.4	13.4	65.3	80.1	
AVG	N/A	1.5	1.5	15.7	65.7	82.9	
STDEV	N/A	0.1	0.1	3.2	0.6	3.9	
						91.1	AVR
						5.8	STDEV

¹Total extractables are assumed to be composed of all parent compound.
 Data were obtained from Table 11 and Appendix C, pp. 59 and 97 of the study report.
 N/A= Not Applicable because not sampled.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235729
 Guideline No: 161-2

Table 8 Biotransformation of [2,6-C14]aminopyralid, expressed as percentage of applied radioactivity in Regent loam, 120 g/ha, under aerobic conditions

Time (days)	CaCl ₂ extract	Organic extract	Total extract ¹	Bound residues	CO ₂	Total radioactivity	
0	80.4	18.1	98.5	0.8	N/A	99.3	
0	84.6	17.4	102.0	0.7	N/A	102.7	
AVG	82.5	17.8	100.3	0.8	N/A	101.0	
STDEV	3.0	0.5	2.5	0.1	N/A	2.4	
8	N/A	77.3	77.3	6.6	9.5	93.4	
8	N/A	72.3	72.3	5.9	9.5	87.7	
AVG	N/A	74.8	74.8	6.3	9.5	90.6	
STDEV	N/A	3.5	3.5	0.5	0.0	4.0	
14	30.0	28.4	58.4	7.4	18.3	84.1	
14	32.9	30.3	63.2	8.3	18.0	89.5	
AVG	31.5	29.4	60.8	7.9	18.2	86.8	
STDEV	2.1	1.3	3.4	0.6	0.2	3.8	
33	10.5	14.7	25.2	14.8	47.1	87.1	
33	9.6	14.3	23.9	15.8	46.9	86.6	
AVG	10.1	14.5	24.6	15.3	47.0	86.9	
STDEV	0.6	0.3	0.9	0.7	0.1	0.4	
63	N/A	5.0	5.0	16.8	69.1	90.9	
63	N/A	4.2	4.2	16.8	68.3	89.3	
AVG	N/A	4.6	4.6	16.8	68.7	90.1	
STDEV	N/A	0.6	0.6	0.0	0.6	1.1	
91	N/A	3.4	3.4	18.2	64.2	85.8	
91	N/A	3.3	3.3	17.5	61.2	82.0	
AVG	N/A	3.4	3.4	17.9	62.7	83.9	
STDEV	N/A	0.1	0.1	0.5	2.1	2.7	
190	N/A	2.1	2.1	14.9	70.3	87.3	
190	N/A	2.2	2.2	14.6	71.5	88.3	
AVG	N/A	2.2	2.2	14.8	70.9	87.8	
STDEV	N/A	0.1	0.1	0.2	0.8	0.7	
365	N/A	1.5	1.5	0.0	66.3	67.8	
365	N/A	1.6	1.6	0.0	67.4	69.0	
AVG	N/A	1.6	1.6	0.0	66.9	68.4	
STDEV	N/A	0.1	0.1	0.0	0.8	0.8	
						86.9	AVR
						8.9	STDEV

¹Total extractables are assumed to be composed of all parent compound.
 Data were obtained from Table 12 and Appendix C, pp. 60 and 98 of the study report.
 N/A= Not Applicable because not sampled.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235729
 Guideline No: 161-2

Table 9 Biotransformation of [2,6-C14]aminopyralid, expressed as percentage of applied radioactivity in Manning sandy loam, 60 g/ha, under aerobic conditions

Time (days)	CaCl ₂ extract	Organic extract	Total extract ¹	Bound residues	CO ₂	Total radioactivity	
0	87.5	15.0	102.5	0.1	N/A	102.6	
0	86.9	13.5	100.4	0.1	N/A	100.5	
AVG	87.2	14.3	101.5	0.1	N/A	101.6	
STDEV	0.4	1.1	1.5	0.0	N/A	1.5	
8	N/A	79.2	79.2	2.9	11.0	93.1	
8	N/A	81.4	81.4	2.7	10.6	94.7	
AVG	N/A	80.3	80.3	2.8	10.8	93.9	
STDEV	N/A	1.6	1.6	0.1	0.3	1.1	
14	46.0	16.6	62.6	5.1	20.0	87.7	
14	45.6	17.3	62.9	4.8	19.7	87.4	
AVG	45.8	17.0	62.8	5.0	19.9	87.6	
STDEV	0.3	0.5	0.2	0.2	0.2	0.2	
33	29.8	12.3	42.1	6.2	26.1	74.4	
33	11.7	9.1	20.8	8.6	53.4	82.8	
AVG	20.8	10.7	31.5	7.4	39.8	78.6	
STDEV	12.8	2.3	15.1	1.7	19.3	5.9	
63	N/A	5.0	5.0	11.8	69.9	86.7	
63	N/A	4.9	4.9	11.8	69.0	85.7	
AVG	N/A	5.0	5.0	11.8	69.5	86.2	
STDEV	N/A	0.1	0.1	0.0	0.6	0.7	
91	N/A	3.7	3.7	9.9	67.4	81.0	
91	N/A	3.6	3.6	9.9	67.4	80.9	
AVG	N/A	3.7	3.7	9.9	67.4	81.0	
STDEV	N/A	0.1	0.1	0.0	0.0	0.1	
124	N/A	3.1	3.1	10.0	70.9	84.0	
124	N/A	3.2	3.2	14.1	70.6	87.9	
AVG	N/A	3.2	3.2	12.1	70.8	86.0	
STDEV	N/A	0.1	0.1	2.9	0.2	2.8	
190	N/A	2.5	2.5	9.1	73.3	84.9	
190	N/A	2.7	2.7	8.9	73.1	84.7	
AVG	N/A	2.6	2.6	9.0	73.2	84.8	
STDEV	N/A	0.1	0.1	0.1	0.1	0.1	
						87.4	AVR
						7.3	STDEV

¹Total extractables are assumed to be composed of all parent compound.
 Data were obtained from Table 13 and Appendix C, pp. 61 and 100 of the study report.
 N/A= Not Applicable because not sampled.

Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235729
 Guideline No: 161-2

Table 10 Biotransformation of [2,6-C14]aminopyralid, expressed as percentage of applied radioactivity in Manning sandy loam, 120 g/ha, under aerobic conditions

Time (days)	CaCl ₂ extract	Organic extract	Total extract ¹	Bound residues	CO ₂	Total radioactivity
0	102.5	5.7	108.2	0.2	N/A	108.4
0	93.8	4.3	98.1	0.2	N/A	98.3
AVG	98.2	5.0	103.2	0.2	N/A	103.4
STDEV	6.2	1.0	7.1	0.0	N/A	7.1
8	N/A	76.3	76.3	2.8	8.1	87.2
8	N/A	76.1	76.1	3.1	8.5	87.7
AVG	N/A	76.2	76.2	3.0	8.3	87.5
STDEV	N/A	0.1	0.1	0.2	0.3	0.4
14	54.2	17.6	71.8	2.7	14.8	89.3
14	55.5	19.1	74.6	3.1	14.0	91.7
AVG	54.9	18.4	73.2	2.9	14.4	90.5
STDEV	0.9	1.1	2.0	0.3	0.6	1.7
33	21.6	10.6	32.2	8.1	43.8	84.1
33	19.4	10.2	29.6	7.9	45.4	82.9
AVG	20.5	10.4	30.9	8.0	44.6	83.5
STDEV	1.6	0.3	1.8	0.1	1.1	0.8
63	N/A	4.2	4.2	10.3	70.1	84.6
63	N/A	5.0	5.0	13.2	69.3	87.5
AVG	N/A	4.6	4.6	11.8	69.7	86.1
STDEV	N/A	0.6	0.6	2.1	0.6	2.1
91	N/A	3.3	3.3	10.8	65.8	79.9
91	N/A	3.1	3.1	9.6	65.2	77.9
AVG	N/A	3.2	3.2	10.2	65.5	78.9
STDEV	N/A	0.1	0.1	0.8	0.4	1.4
190	N/A	2.2	2.2	8.6	72.2	83.0
190	N/A	2.4	2.4	7.9	70.8	81.1
AVG	N/A	2.3	2.3	8.3	71.5	82.1
STDEV	N/A	0.1	0.1	0.5	1.0	1.3
						87.4
						8.0
						AVR
						STDEV

¹Total extractables are assumed to be composed of all parent compound.
 Data were obtained from Table 14 and Appendix C, pp. 62 and 101 of the study report.
 N/A= Not Applicable because not sampled.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235729
 Guideline No: 161-2

Table 11 Biotransformation of [2,6-C14]aminopyralid, expressed as percentage of applied radioactivity in Barnes clay loam, 60 g/ha, under aerobic conditions

Time (days)	CaCl ₂ extract	Organic extract	Total extract ¹	Bound residues	CO ₂	Total radioactivity
0	60.6	30.7	91.3	0.1	N/A	91.4
0	66.5	25.9	92.4	0.0	N/A	92.4
AVG	63.6	28.3	91.9	0.1	N/A	91.9
STDEV	4.2	3.4	0.8	0.1	N/A	0.7
8	N/A	83.2	83.2	2.2	0.9	86.3
8	N/A	81.0	81.0	2.8	0.8	84.6
AVG	N/A	82.1	82.1	2.5	0.9	85.5
STDEV	N/A	1.6	1.6	0.4	0.1	1.2
14	33.6	52.6	86.2	3.0	1.2	90.4
14	35.0	56.7	91.7	2.8	1.2	95.7
AVG	34.3	54.7	89.0	2.9	1.2	93.1
STDEV	1.0	2.9	3.9	0.1	0.0	3.7
33	38.2	52.7	90.9	4.8	2.6	98.3
33	36.9	56.4	93.3	5.0	2.5	100.8
AVG	37.6	54.6	92.1	4.9	2.6	99.6
STDEV	0.9	2.6	1.7	0.1	0.1	1.8
63	N/A	82.7	82.7	6.3	4.8	93.8
63	N/A	79.8	79.8	6.0	5.7	91.5
AVG	N/A	81.3	81.3	6.2	5.3	92.7
STDEV	N/A	2.1	2.1	0.2	0.6	1.6
91	N/A	71.8	71.8	6.9	8.2	86.9
91	N/A	75.3	75.3	8.1	8.3	91.7
AVG	N/A	73.6	73.6	7.5	8.3	89.3
STDEV	N/A	2.5	2.5	0.8	0.1	3.4
124	N/A	68.7	68.7	8.6	11	88.3
124	N/A	69.6	69.6	11.1	11.5	92.2
AVG	N/A	69.2	69.2	9.9	11.3	90.3
STDEV	N/A	0.6	0.6	1.8	0.4	2.8
190	N/A	62.1	62.1	10.9	16.6	89.6
190	N/A	59.0	59.0	8.3	17.1	84.4
AVG	N/A	60.6	60.6	9.6	16.9	87.0
STDEV	N/A	2.2	2.2	1.8	0.4	3.7
274	N/A	51.1	51.1	11.3	23.3	85.7
274	N/A	52.6	52.6	11.3	21.9	85.8
AVG	N/A	51.9	51.9	11.3	22.6	85.8
STDEV	N/A	1.1	1.1	0.0	1.0	0.1
365	N/A	45.2	45.2	14.6	28.1	87.9
365	N/A	39.7	39.7	12.7	31.9	84.3
AVG	N/A	42.5	42.5	13.7	30.0	86.1
STDEV	N/A	3.9	3.9	1.3	2.7	2.5
						90.1
						4.8
						AVR
						STDEV

¹Total extractables are assumed to be composed of all parent compound.
 Data were obtained from Table 15 and Appendix C, pp. 63 and 103 of the study report.
 N/A= Not Applicable because not sampled.

Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235729
 Guideline No: 161-2

Table 12 Biotransformation of [2,6-C14]aminopyralid, expressed as percentage of applied radioactivity in Barnes clay loam, 120 g/ha, under aerobic conditions

Time (days)	CaCl ₂ extract	Organic extract	Total extract ¹	Bound residues	CO ₂	Total radioactivity	
0	66.5	19.6	86.1	0.7	N/A	86.8	
0	75.9	27.2	103.1	0.8	N/A	103.9	
AVG	71.2	23.4	94.6	0.8	N/A	95.4	
STDEV	6.6	5.4	12.0	0.1	N/A	12.1	
8	N/A	104.4	104.4	3.6	0.8	108.8	
8	N/A	78.3	78.3	2.2	0.9	81.4	
AVG	N/A	91.4	91.4	2.9	0.9	95.1	
STDEV	N/A	18.5	18.5	1.0	0.1	19.4	
14	33.7	51.0	84.7	2.6	1.3	88.6	
14	33.1	47.1	80.2	2.5	1.2	83.9	
AVG	33.4	49.1	82.5	2.6	1.3	86.3	
STDEV	0.4	2.8	3.2	0.1	0.1	3.3	
33	34.5	48.6	83.1	4.6	3.6	91.3	
33	35.2	48.6	83.8	4.8	2.9	91.5	
AVG	34.9	48.6	83.5	4.7	3.3	91.4	
STDEV	0.5	0.0	0.5	0.1	0.5	0.1	
63	N/A	76.9	76.9	7.1	6.1	90.1	
63	N/A	61.0	61.0	7.5	6.4	74.9	
AVG	N/A	69.0	69.0	7.3	6.3	82.5	
STDEV	N/A	11.2	11.2	0.3	0.2	10.7	
91	N/A	73.1	73.1	7.7	8	88.8	
91	N/A	79.2	79.2	9.7	9.4	98.3	
AVG	N/A	76.2	76.2	8.7	8.7	93.6	
STDEV	N/A	4.3	4.3	1.4	1.0	6.7	
190	N/A	63.9	63.9	12.2	18.1	94.2	
190	N/A	64.9	64.9	11.0	18.1	94.0	
AVG	N/A	64.4	64.4	11.6	18.1	94.1	
STDEV	N/A	0.7	0.7	0.8	0.0	0.1	
365	N/A	41.5	41.5	13.5	28	83.0	
365	N/A	40.7	40.7	14.5	26.0	81.2	
AVG	N/A	41.1	41.1	14.0	27.0	82.1	
STDEV	N/A	0.6	0.6	0.7	1.4	1.3	
						90.0	AVR
						8.7	STDEV

¹Total extractables are assumed to be composed of all parent compound.
 Data were obtained from Table 16 and Appendix C, pp. 64 and 104 of the study report.
 N/A= Not Applicable because not sampled.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235729
 Guideline No: 161-2

Table 13 Biotransformation of [2,6-C14]aminopyralid, expressed as percentage of applied radioactivity in Houston Black clay, 60 g/ha, under aerobic conditions

Time (days)	CaCl ₂ extract	Organic extract	Total extract ¹	Bound residues	CO ₂	Total radioactivity	
0	85.1	0.0	85.1	7.8	N/A	92.9	
0	87.1	0.2	87.3	8.4	N/A	95.7	
AVG	86.1	0.1	86.2	8.1	N/A	94.3	
STDEV	1.4	0.1	1.6	0.4	N/A	2.0	
1	N/A	65.9	65.9	28.3	1.6	95.8	
1	N/A	63.6	63.6	26.6	1.8	92.0	
AVG	N/A	64.8	64.8	27.5	1.7	93.9	
STDEV	N/A	1.6	1.6	1.2	0.1	2.7	
4	N/A	47.2	47.2	28.4	13.6	89.2	
4	N/A	45.4	45.4	28.9	13.9	88.2	
AVG	N/A	46.3	46.3	28.7	13.8	88.7	
STDEV	N/A	1.3	1.3	0.4	0.2	0.7	
8	N/A	20.7	20.7	35.3	30.4	86.4	
8	N/A	16.9	16.9	33.4	34.1	84.4	
AVG	N/A	18.8	18.8	34.4	32.3	85.4	
STDEV	N/A	2.7	2.7	1.3	2.6	1.4	
14	4.6	3.1	7.7	27.3	55.6	90.6	
14	2.9	2.7	5.6	25.9	57.0	88.5	
AVG	3.8	2.9	6.7	26.6	56.3	89.6	
STDEV	1.2	0.3	1.5	1.0	1.0	1.5	
22	N/A	1.9	1.9	37.6	55.5	95.0	
22	N/A	1.4	1.4	29.3	57.3	88.0	
AVG	N/A	1.7	1.7	33.5	56.4	91.5	
STDEV	N/A	0.4	0.4	5.9	1.3	4.9	
31	N/A	2.2	2.2	24.8	67.5	94.5	
31	N/A	1.2	1.2	25.1	65.5	91.8	
AVG	N/A	1.7	1.7	25.0	66.5	93.2	
STDEV	N/A	0.7	0.7	0.2	1.4	1.9	
65	N/A	1.3	1.3	22.5	67.9	91.7	
65	N/A	1.4	1.4	23.7	67.1	92.2	
AVG	N/A	1.4	1.4	23.1	67.5	92.0	
STDEV	N/A	0.1	0.1	0.8	0.6	0.4	
						91.1	AVR
						3.4	STDEV

¹Total extractables are assumed to be composed of all parent compound.
 Data were obtained from Table 16 and Appendix C, pp. 65 and 105 of the study report.
 N/A= Not Applicable because not sampled.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235729
 Guideline No: 161-2

Table 14 Biotransformation of [2,6-C14]aminopyralid, expressed as percentage of applied radioactivity in Houston Black clay, 120 g/ha, under aerobic conditions

Time (days)	CaCl ₂ extract	Organic extract	Total extract ¹	Bound residues	CO ₂	Total radioactivity	
0	81.4	2.6	84.0	8.6	N/A	92.6	
0	89.7	6.4	96.1	10.5	N/A	106.6	
AVG	85.6	4.5	90.1	9.6	N/A	99.6	
STDEV	5.9	2.7	8.6	1.3	N/A	9.9	
1	N/A	63.2	63.2	26.0	1.2	90.4	
1	N/A	63.4	63.4	25.3	1.3	90.0	
AVG	N/A	63.3	63.3	25.7	1.3	90.2	
STDEV	N/A	0.1	0.1	0.5	0.1	0.3	
4	N/A	52.8	52.8	29.9	9.2	91.9	
4	N/A	53.3	53.3	29.8	10.1	93.2	
AVG	N/A	53.1	53.1	29.9	9.7	92.6	
STDEV	N/A	0.4	0.4	0.1	0.6	0.9	
8	N/A	32.3	32.3	37.1	41.2	110.6	
8	N/A	23.7	23.7	34.6	19.2	77.5	
AVG	N/A	28.0	28.0	35.9	30.2	94.1	
STDEV	N/A	6.1	6.1	1.8	15.6	23.4	
14	9.6	6.3	15.9	25.7	43.3	84.9	
14	19.8	9.9	29.7	25.3	39.2	94.2	
AVG	14.7	8.1	22.8	25.5	41.3	89.6	
STDEV	7.2	2.5	9.8	0.3	2.9	6.6	
22	N/A	1.7	1.7	31.2	56.8	89.7	
22	N/A	16.1	16.1	31.7	25.0	72.8	
AVG	N/A	8.9	8.9	31.5	40.9	81.3	
STDEV	N/A	10.2	10.2	0.4	22.5	12.0	
31	N/A	1.2	1.2	24.5	63.2	88.9	
31	N/A	1.2	1.2	24.2	63.2	88.6	
AVG	N/A	1.2	1.2	24.4	63.2	88.8	
STDEV	N/A	0.0	0.0	0.2	0.0	0.2	
65	N/A	1.1	1.1	25.2	67.5	93.8	
65	N/A	1.0	1.0	23.4	67.2	91.6	
AVG	N/A	1.1	1.1	24.3	67.4	92.7	
STDEV	N/A	0.1	0.1	1.3	0.2	1.6	
						91.1	AVR
						9.0	STDEV

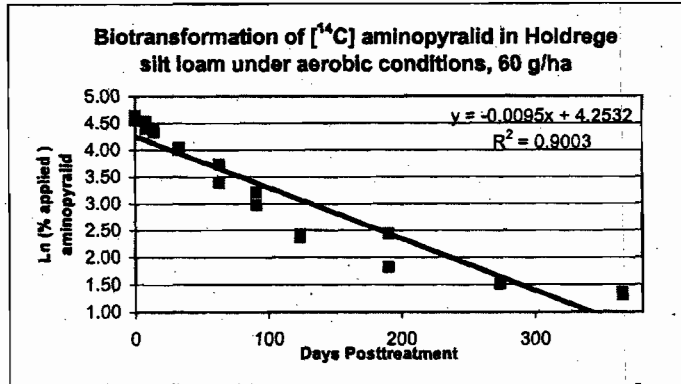
¹Total extractables are assumed to be composed of all parent compound.
 Data were obtained from Table 16 and Appendix C, pp. 65 and 105 of the study report.
 N/A= Not Applicable because not sampled.

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Chemical Name: Aminopyralid
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Days	% applied	Ln (% applied)
0	95.5	4.56
0	103.9	4.64
8	93.2	4.53
8	80.9	4.39
14	79.3	4.37
14	75.9	4.33
33	54.4	4.00
33	57.2	4.05
63	41.8	3.73
63	29.6	3.39
91	24.7	3.21
91	19.5	2.97
124	10.7	2.37
124	11.3	2.42
190	6.2	1.82
190	11.5	2.44
274	4.5	1.50
274	4.5	1.50
365	3.9	1.36
365	3.7	1.31

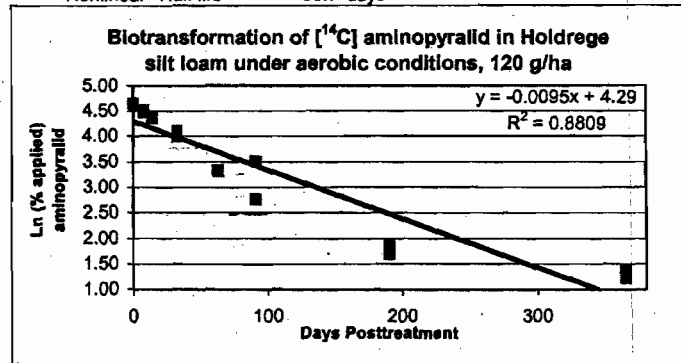
Empirical DT50= 50.0 days
 Linear Half life = 73.0 days
 Nonlinear Half life = 42.0 days



Data were obtained from Table 9 and Appendix C, pp. 57 and 94 of the study report.

Days	% applied	Ln (% applied)
0	104.7	4.65
0	99.7	4.60
8	91.2	4.51
8	86.9	4.46
14	79.3	4.37
14	77.7	4.35
33	53.7	3.98
33	60.1	4.10
63	27.9	3.33
63	27.4	3.31
91	15.8	2.76
91	33.3	3.51
190	5.4	1.69
190	6.3	1.84
365	3.9	1.36
365	3.4	1.22

Empirical DT50= 60.0 days
 Linear Half life = 73.0 days
 Nonlinear Half life = 38.7 days

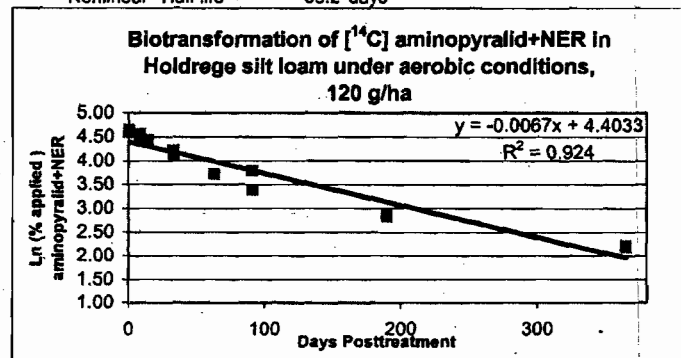


Data were obtained from Table 10 and Appendix C, pp. 58 and 95 of the study report.

Parent and non-extractible residues

Days	% applied	Ln (% applied)
0	104.8	4.65
0	99.8	4.60
8	96.2	4.57
8	91.6	4.52
14	84.0	4.43
14	83.1	4.42
33	62.4	4.13
33	68.2	4.22
63	40.9	3.71
63	41.0	3.71
91	29.5	3.38
91	44.1	3.79
190	16.8	2.82
190	17.4	2.86
365	8.9	2.19
365	9.2	2.22

Empirical DT50= 70.0 days
 Linear Half life = 103.5 days
 Nonlinear Half life = 58.2 days

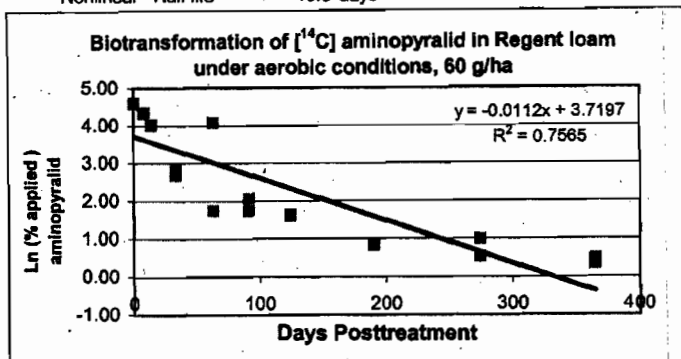


Data were obtained from Table 10 and Appendix C, pp. 58 and 95 of the study report.

Chemical Name: Aminopyralid
 MRID 46235729
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Days	% applied	Ln (% applied)
0	100.3	4.61
0	98.4	4.59
8	75.6	4.33
8	77.6	4.35
14	56.3	4.03
14	54.8	4.00
33	14.6	2.68
33	17.7	2.87
63	58.8	4.07
63	5.7	1.74
91	7.9	2.07
91	5.7	1.74
124	5.1	1.63
124	4.9	1.59
190	2.3	0.83
190	2.3	0.83
274	1.7	0.53
274	2.7	0.99
365	1.6	0.47
365	1.4	0.34

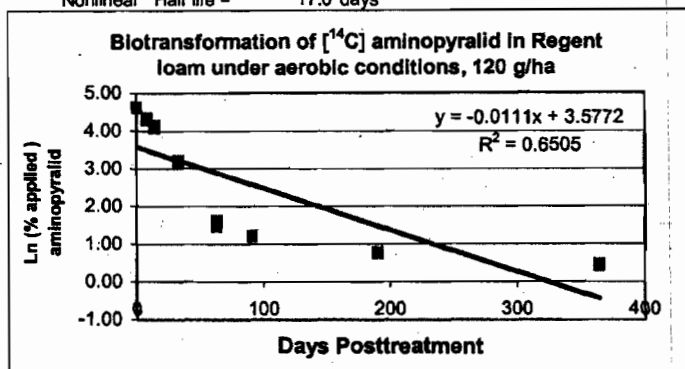
Empirical DT50= 15.0 days
 Linear Half life = 81.9 days
 Nonlinear Half life = 19.8 days



Data were obtained from Table 11 and Appendix C, pp. 59 and 97 of the study report.

Days	% applied	Ln (% applied)
0	98.5	4.59
0	102	4.62
8	77.3	4.35
8	72.3	4.28
14	58.4	4.07
14	63.2	4.15
33	25.2	3.23
33	23.9	3.17
63	5	1.61
63	4.2	1.44
91	3.4	1.22
91	3.3	1.19
190	2.1	0.74
190	2.2	0.79
365	1.5	0.41
365	1.6	0.47

Empirical DT50= 20.0 days
 Linear Half life = 62.4 days
 Nonlinear Half life = 17.0 days

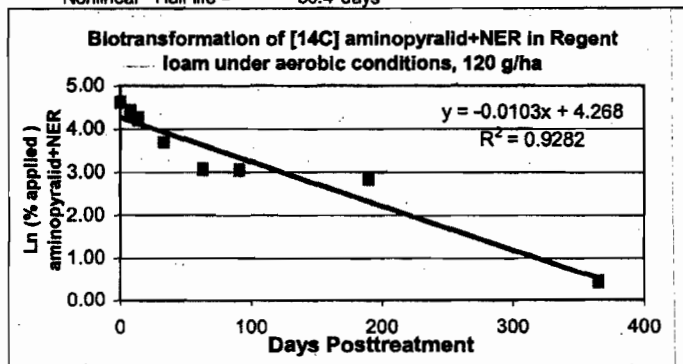


Data were obtained from Table 12 and Appendix C, pp. 60 and 98 of the study report.

Parent and non-extractible residues

Days	% applied	Ln (% applied)
0	99.3	4.60
0	102.7	4.63
8	83.9	4.43
8	78.2	4.36
14	65.8	4.19
14	71.5	4.27
33	40.0	3.69
33	39.7	3.68
63	21.8	3.08
63	21.0	3.04
91	21.6	3.07
91	20.8	3.03
190	17.0	2.83
190	16.8	2.82
365	1.5	0.41
365	1.6	0.47

Empirical DT50= 30.0 days
 Linear Half life = 67.3 days
 Nonlinear Half life = 30.4 days

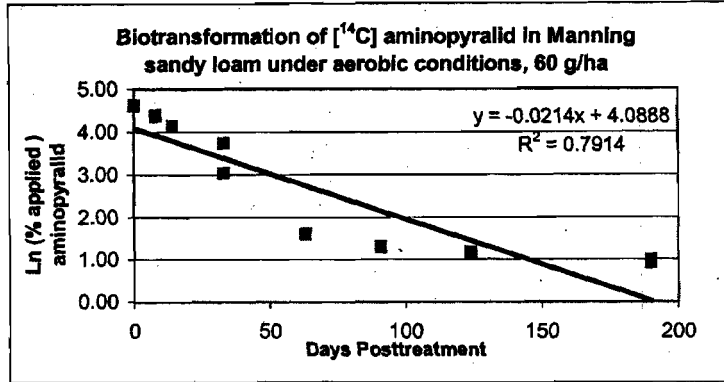


Data were obtained from Table 12 and Appendix C, pp. 60 and 98 of the study report.

Chemical Name: Aminopyralid
 MRID 46235729
 PC 005100
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Days	% applied	Ln (% applied)
0	102.5	4.63
0	100.4	4.61
8	79.2	4.37
8	81.4	4.40
14	62.6	4.14
14	62.9	4.14
33	42.1	3.74
33	20.8	3.03
63	5	1.61
63	4.9	1.59
91	3.7	1.31
91	3.6	1.28
124	3.1	1.13
124	3.2	1.16
190	2.5	0.92
190	2.7	0.99

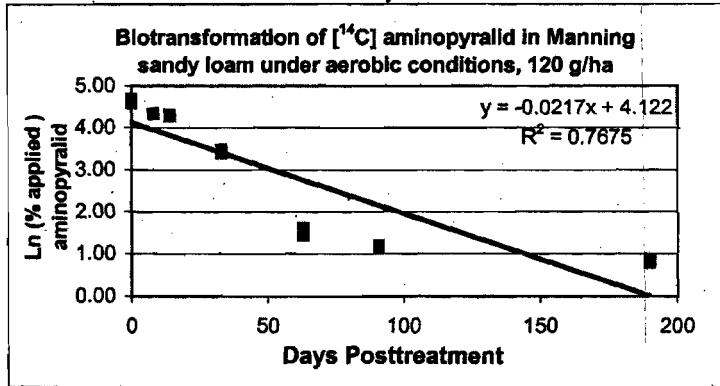
Empirical DT50= 20.0 days
 Linear Half life = 32.4 days
 Nonlinear Half life = 18.8 days



Data were obtained from Table 13 and Appendix C, pp. 61 and 100 of the study report.

Days	% applied	Ln (% applied)
0	108.2	4.68
0	98.1	4.59
8	76.3	4.33
8	76.1	4.33
14	71.8	4.27
14	74.6	4.31
33	32.2	3.47
33	29.6	3.39
63	4.2	1.44
63	5	1.61
91	3.3	1.19
91	3.1	1.13
190	2.2	0.79
190	2.4	0.88

Empirical DT50= 22.0 days
 Linear Half life = 31.9 days
 Nonlinear Half life = 19.4 days

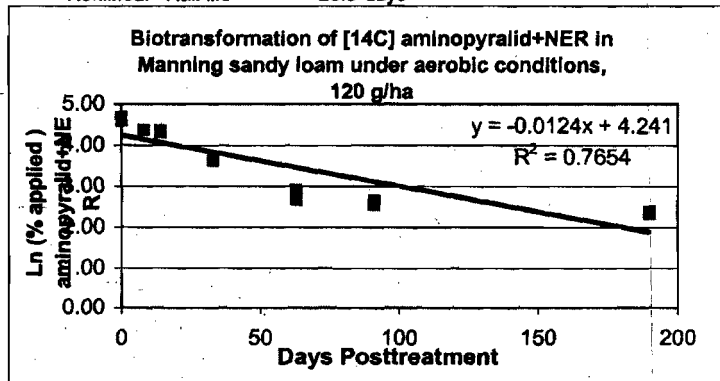


Data were obtained from Table 14 and Appendix C, pp. 62 and 101 of the study report.

Parent and non-extractible residues

Days	% applied	Ln (% applied)
0	108.4	4.69
0	98.3	4.59
8	79.1	4.37
8	79.2	4.37
14	74.5	4.31
14	77.7	4.35
33	40.3	3.70
33	37.5	3.62
63	14.5	2.67
63	18.2	2.90
91	14.1	2.65
91	12.7	2.54
190	10.8	2.38
190	10.3	2.33

Empirical DT50= 27.0 days
 Linear Half life = 55.9 days
 Nonlinear Half life = 25.8 days



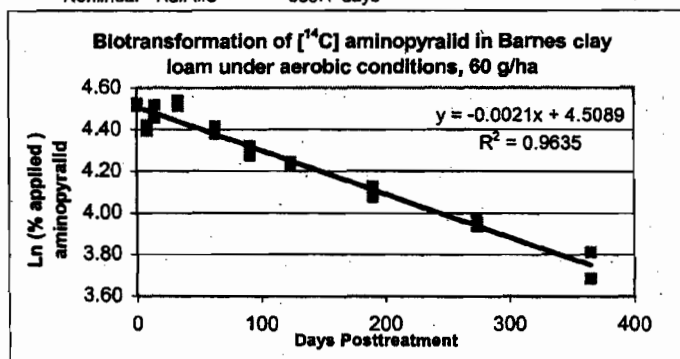
Data were obtained from Table 14 and Appendix C, pp. 62 and 101 of the study report.

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Chemical Name: Aminopyralid
 MRID 46235729
 PC 005100
 Guideline No: 161-2

Days	% applied	Ln (% applied)
0	91.3	4.51
0	92.4	4.53
8	83.2	4.42
8	81.0	4.39
14	86.2	4.46
14	91.7	4.52
33	90.9	4.51
33	93.3	4.54
63	82.7	4.42
63	79.8	4.38
91	71.8	4.27
91	75.3	4.32
124	68.7	4.23
124	69.6	4.24
190	62.1	4.13
190	59.0	4.08
274	51.1	3.93
274	52.6	3.96
365	45.2	3.81
365	39.7	3.68

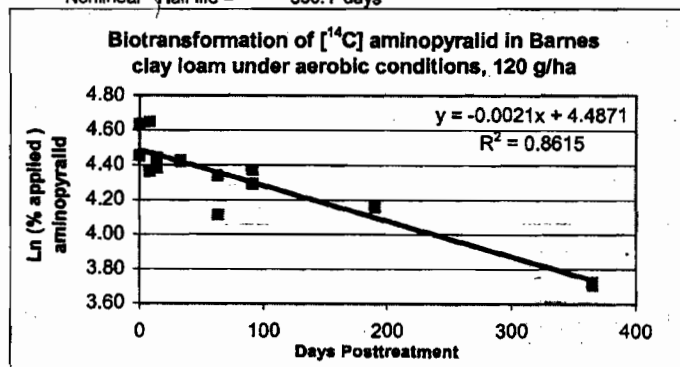
Empirical DT50= 290.0 days
 Linear Half life = 330.1 days
 Nonlinear Half life = 330.1 days



Data were obtained from Table 15 and Appendix C, pp. 63 and 103 of the study report.

Days	% applied	Ln (% applied)
0	86.1	4.46
0	103.1	4.64
8	104.4	4.65
8	78.3	4.36
14	84.7	4.44
14	80.2	4.38
33	83.1	4.42
33	83.8	4.43
63	76.9	4.34
63	61	4.11
91	73.1	4.29
91	79.2	4.37
190	63.9	4.16
190	64.9	4.17
365	41.5	3.73
365	40.7	3.71

Empirical DT50= 300.0 days
 Linear Half life = 330.1 days
 Nonlinear Half life = 330.1 days

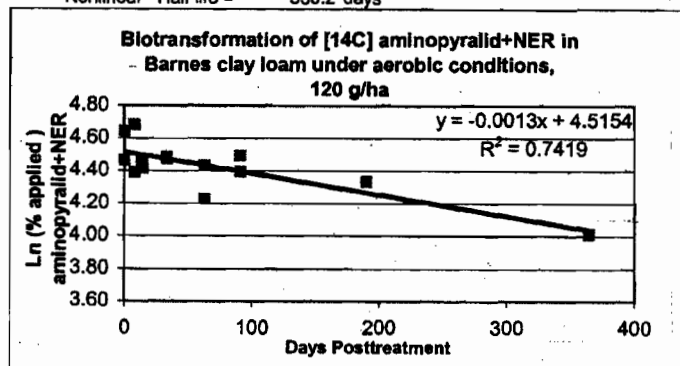


Data were obtained from Table 16 and Appendix C, pp. 64 and 104 of the study report.

Parent and non-extractible residues as parent

Days	% applied	Ln (% applied)
0	86.8	4.46
0	103.9	4.64
8	108.0	4.68
8	80.5	4.39
14	87.3	4.47
14	82.7	4.42
33	87.7	4.47
33	88.6	4.48
63	84.0	4.43
63	68.5	4.23
91	80.8	4.39
91	88.9	4.49
190	76.1	4.33
190	75.9	4.33
365	55.0	4.01
365	55.2	4.01

Empirical DT50= N/A (does not degrade below 50% in study)
 Linear Half life = 533.2 days
 Nonlinear Half life = 533.2 days



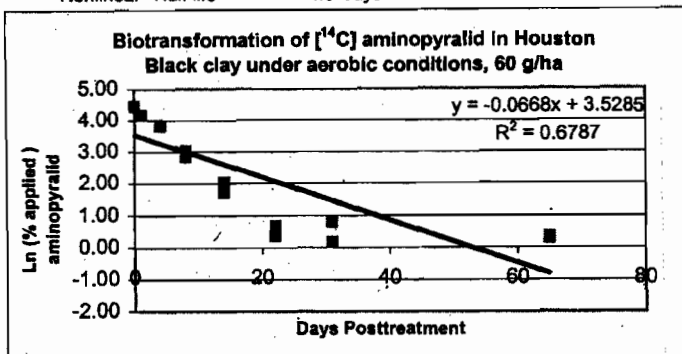
Data were obtained from Table 16 and Appendix C, pp. 64 and 104 of the study report.

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Chemical Name: Aminopyralid
 MRID 46235729
 PC 005100
 Guideline No: 161-2

Days	% applied	Ln (% applied)
0	85.1	4.44
0	87.3	4.47
1	65.9	4.19
1	63.6	4.15
4	47.2	3.85
4	45.4	3.82
8	20.7	3.03
8	16.9	2.83
14	7.7	2.04
14	5.6	1.72
22	1.9	0.64
22	1.4	0.34
31	2.2	0.79
31	1.2	0.18
65	1.3	0.26
65	1.4	0.34

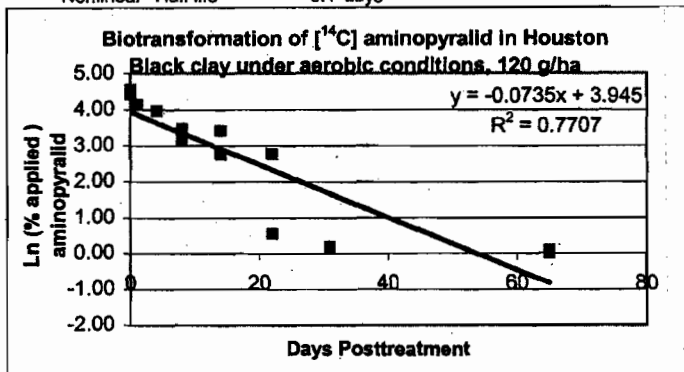
Empirical DT50= 3.5 days
 Linear Half life = 10.4 days
 Nonlinear Half life = 4.0 days



Data were obtained from Table 17 and Appendix C, pp. 65 and 105 of the study report.

Days	% applied	Ln (% applied)
0	84.0	4.43
0	96.1	4.57
1	63.2	4.15
1	63.4	4.15
4	52.8	3.97
4	53.3	3.98
8	32.3	3.48
8	23.7	3.17
14	15.9	2.77
14	29.7	3.39
22	1.7	0.53
22	16.1	2.78
31	1.2	0.18
31	1.2	0.18
65	1.1	0.10
65	1.0	0.00

Empirical DT50= 4.5 days
 Linear Half life = 9.4 days
 Nonlinear Half life = 6.1 days

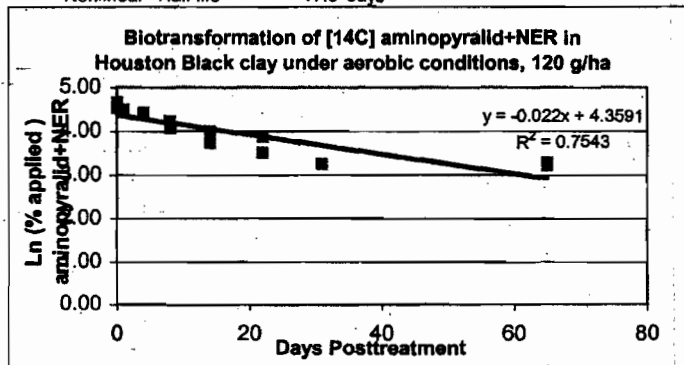


Data were obtained from Table 18 and Appendix C, pp. 66 and 106 of the study report.

Parent and non-extractible residues as parent

Days	% applied	Ln (% applied)
0	92.6	4.53
0	106.6	4.67
1	89.2	4.49
1	88.7	4.49
4	82.7	4.42
4	83.1	4.42
8	69.4	4.24
8	58.3	4.07
14	41.6	3.73
14	55.0	4.01
22	32.9	3.49
22	47.8	3.87
31	25.7	3.25
31	25.4	3.23
65	26.3	3.27
65	24.4	3.19

Empirical DT50= 13.0 days
 Linear Half life = 31.5 days
 Nonlinear Half life = 17.9 days



Data were obtained from Table 18 and Appendix C, pp. 66 and 106 of the study report.

Holdrege, 60 g/ha

Nonlinear Regression

Equation: Single, 2 Parameter

$$y=ae^{-bx}$$

R = 0.99328479 Rsqr = 0.98661468 Adj Rsqr = 0.98587105

Standard Error of Estimate = 4.2718

	Coefficient	Std. Error	t	P
a	98.7590	2.1400	46.1485	<0.0001
b	0.0165	0.0009	18.8121	<0.0001

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	24211.1078	24211.1078	1326.7564	<0.0001
Residual	18	328.4702	18.2483		
Total	19	24539.5780	1291.5567		

PRESS = 425.5189

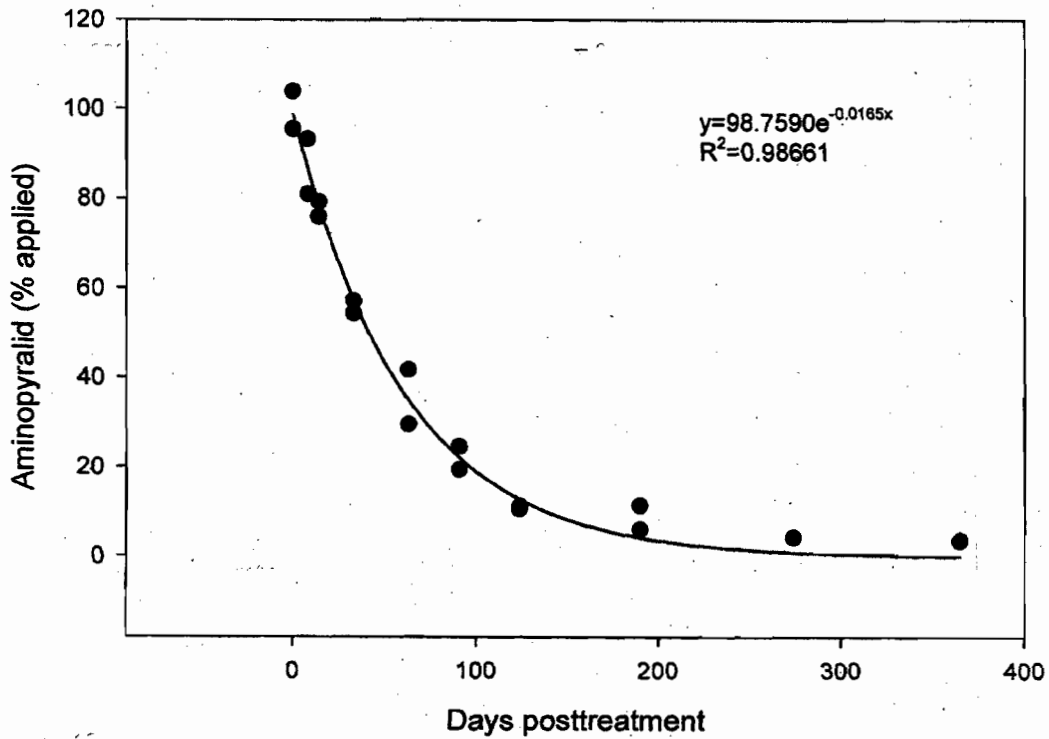
Durbin-Watson Statistic = 1.9166

Normality Test: Passed (P = 0.4653)

Constant Variance Test: Passed (P = 0.9215)

Power of performed test with alpha = 0.0500: 1.0000

Holdrege, 60 g/ha



Holdrege, 120 g/ha
 Nonlinear Regression
 Equation: Single, 2 Parameter
 $y=ae^{-bx}$

R = 0.99191277 Rsqr = 0.98389094 Adj Rsqr = 0.98274029

Standard Error of Estimate = 4.8978

	Coefficient	Std. Error	t	P
a	101.8311	2.5461	39.9948	<0.0001
b	0.0179	0.0012	15.2695	<0.0001

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	20512.0445	20512.0445	855.0759	<0.0001
Residual	14	335.8399	23.9886		
Total	15	20847.8844	1389.8590		

PRESS = 458.9942

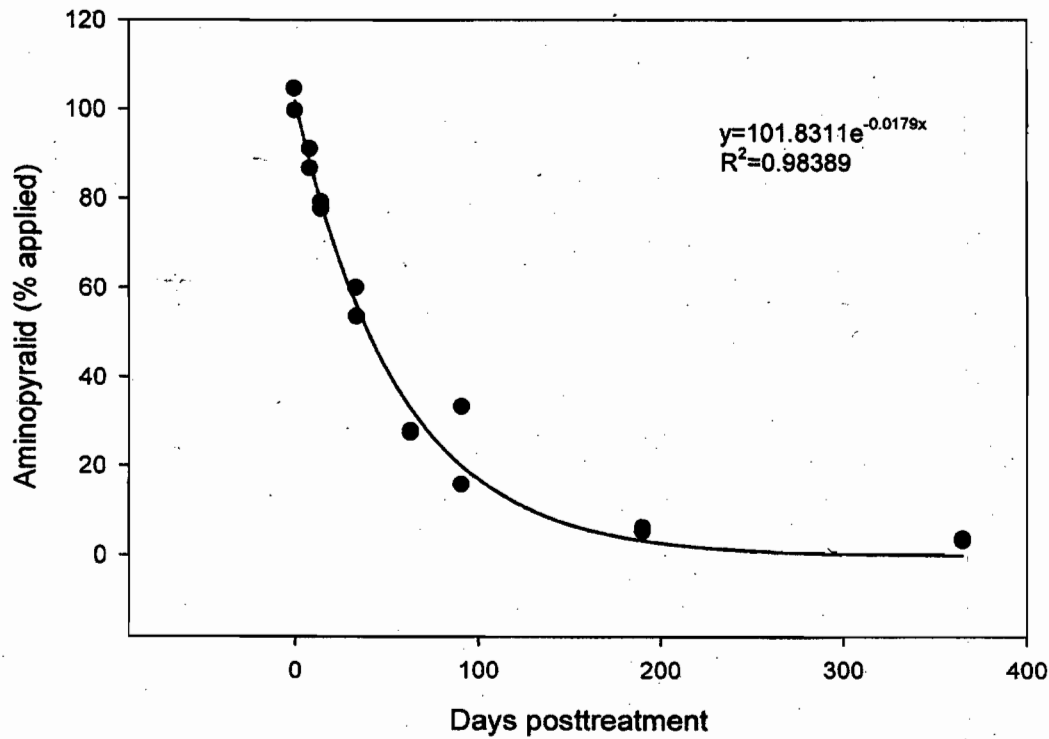
Durbin-Watson Statistic = 2.7043

Normality Test: Passed (P = 0.0922)

Constant Variance Test: Passed (P = 0.0799)

Power of performed test with alpha = 0.0500: 1.0000

Holdrege, 120 g/ha



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Holdrege, 120 g/ha, treating non-extractable residues as parent

Nonlinear Regression

Equation: Single, 2 Parameter

$$y=ae^{-bx}$$

R = 0.98592734 Rsqr = 0.97205272 Adj Rsqr = 0.97005649

Standard Error of Estimate = 5.9313

	Coefficient	Std. Error	t	P
a	100.3383	2.8306	35.4478	<0.0001
b	0.0119	0.0009	12.4952	<0.0001

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	17130.8588	17130.8588	486.9433	<0.0001
Residual	14	492.5256	35.1804		
Total	15	17623.3844	1174.8923		

PRESS = 633.9862

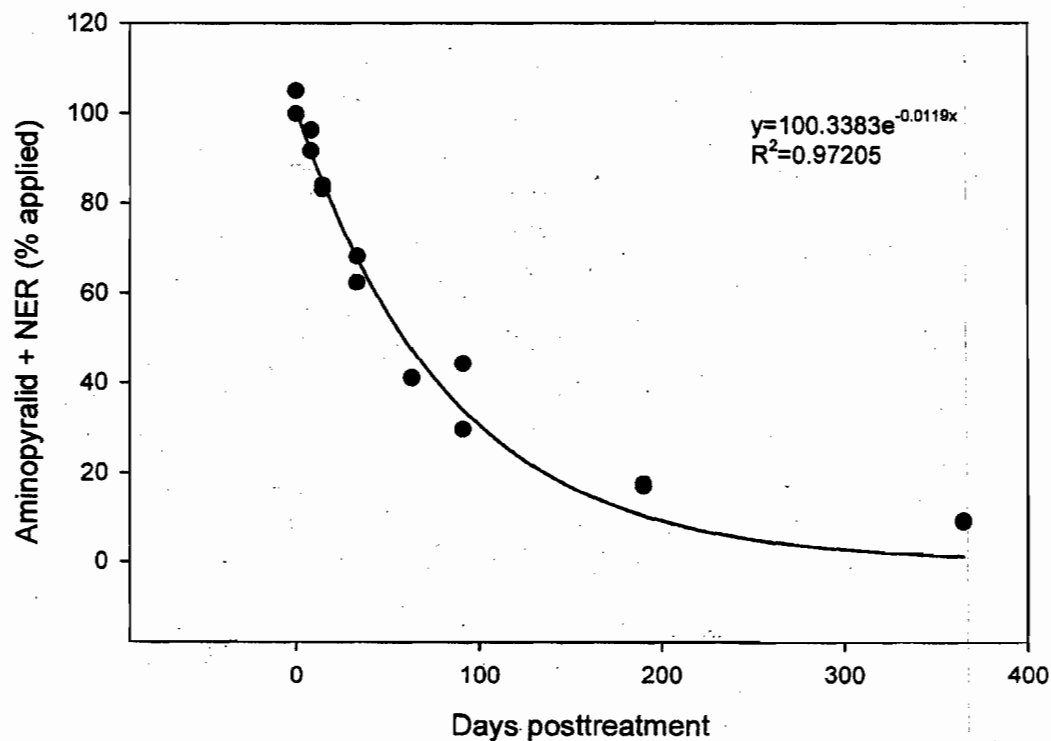
Durbin-Watson Statistic = 0.8239

Normality Test: Passed (P = 0.4219)

Constant Variance Test: Failed (P = <0.0001)

Power of performed test with alpha = 0.0500: 1.0000

Holdrege, 120 g/ha, treating non-extractables as parent



Regent, 60 g/ha

Nonlinear Regression

Equation: Single, 2 Parameter

$$y=ae^{-bx}$$

R = 0.93801774 Rsqr = 0.87987729

Adj Rsqr = 0.87320380

Standard Error of Estimate = 12.6856

	Coefficient	Std. Error	t	P
a	97.2454	7.6903	12.6452	<0.0001
b	0.0350	0.0068	5.1757	<0.0001

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	21217.3802	21217.3802	131.8468	<0.0001
Residual	18	2896.6418	160.9245		
Total	19	24114.0220	1269.1591		

PRESS = 3757.2364

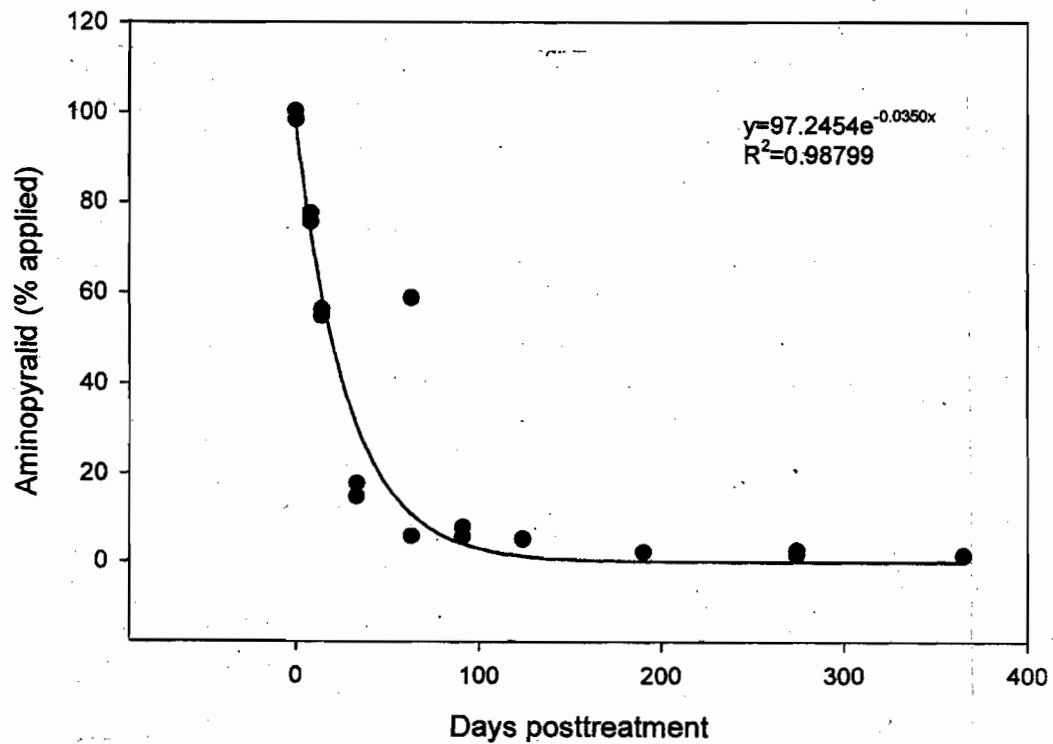
Durbin-Watson Statistic = 2.3600

Normality Test: Failed (P = <0.0001)

Constant Variance Test: Passed (P = 0.1491)

Power of performed test with alpha = 0.0500: 1.0000

Regent, 60 g/ha



Regent, 120 g/ha
 Nonlinear Regression
 Equation: Single, 2 Parameter
 $y=ae^{-bx}$

R = 0.99757345 Rsqr = 0.99515280 Adj Rsqr = 0.99480657

Standard Error of Estimate = 2.7281

	Coefficient	Std. Error	t	P
a	102.0621	1.7125	59.6000	<0.0001
b	0.0407	0.0017	24.6853	<0.0001

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	21392.0328	21392.0328	2874.2628	<0.0001
Residual	14	104.1966	7.4426		
Total	15	21496.2294	1433.0820		

PRESS = 153.8618

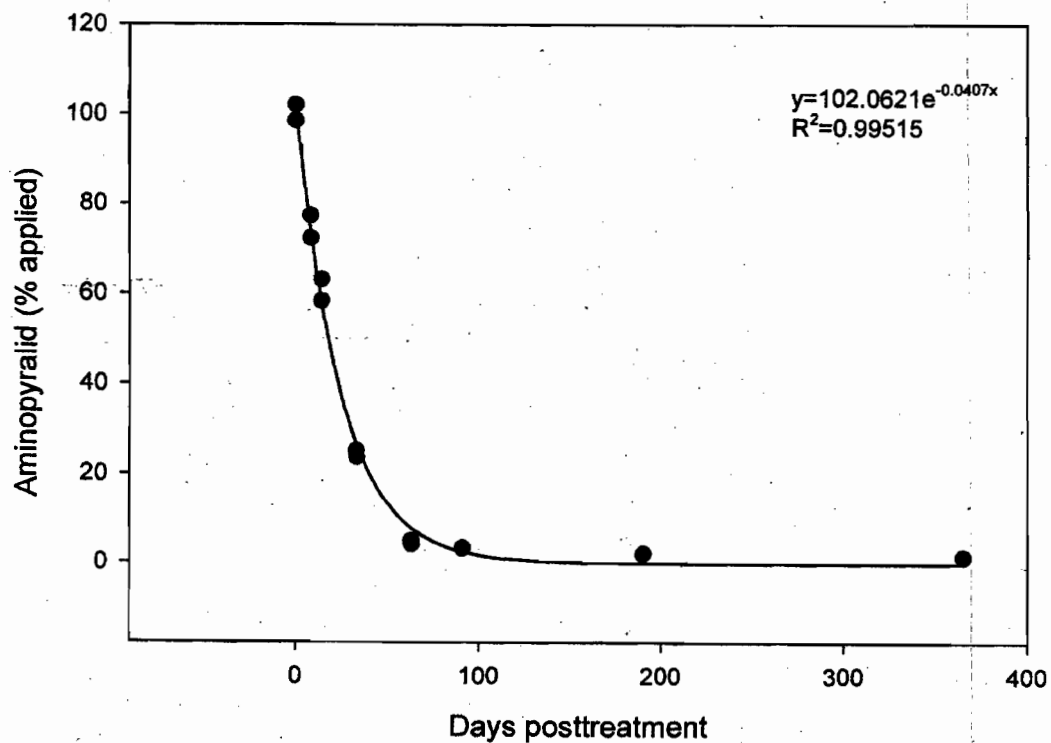
Durbin-Watson Statistic = 1.4481

Normality Test: Passed (P = 0.5185)

Constant Variance Test: Passed (P = 0.8393)

Power of performed test with alpha = 0.0500: 1.0000

Regent, 120 g/ha



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Regent, 120 g/ha, treating non-extractable residues as parent

Nonlinear Regression

Equation: Single, 2 Parameter

$$y=ae^{-bx}$$

R = 0.97689619 Rsqr = 0.95432616 Adj Rsqr = 0.95106374

Standard Error of Estimate = 7.5917

	Coefficient	Std. Error	t	P
a	97.4112	4.1566	23.4353	<0.0001
b	0.0228	0.0025	9.2597	<0.0001

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	16859.1832	16859.1832	292.5212	<0.0001
Residual	14	806.8768	57.6341		
Total	15	17666.0600	1177.7373		

PRESS = 933.3221

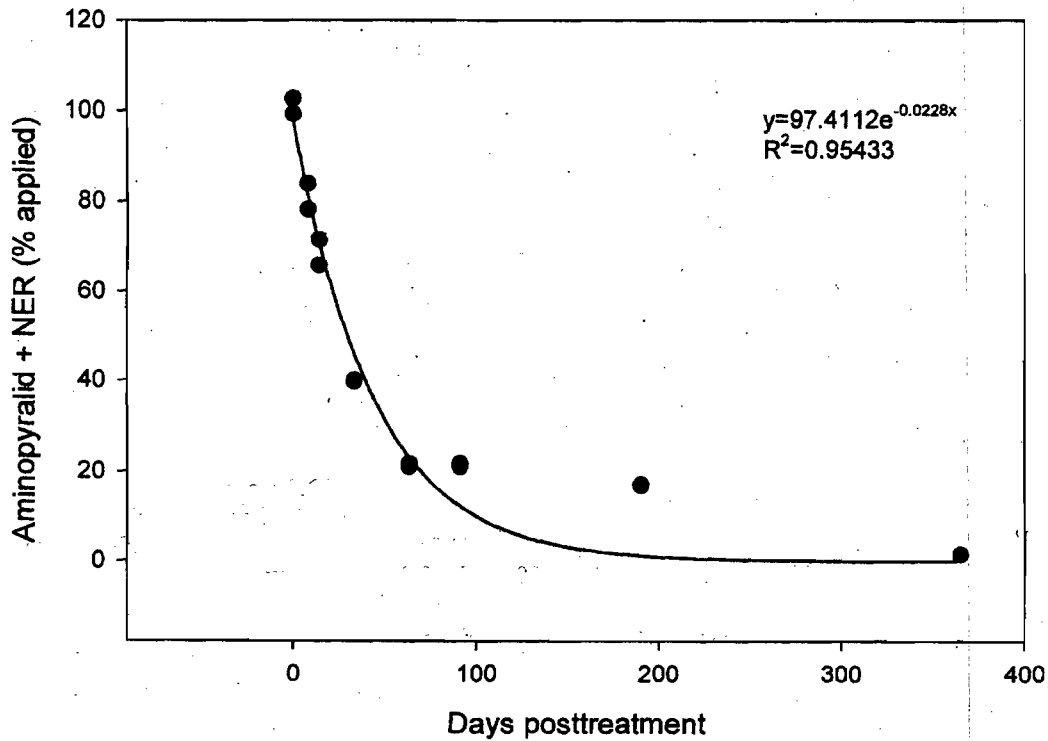
Durbin-Watson Statistic = 0.6655

Normality Test: Passed (P = 0.2314)

Constant Variance Test: Passed (P = 0.5189)

Power of performed test with alpha = 0.0500: 1.0000

Regent, 120 g/ha, treating non-extractables as parent



Manning, 60 g/ha
Nonlinear Regression
 Equation: Single, 2 Parameter
 $y=ae^{-bx}$

R = 0.99247959 Rsqr = 0.98501575 Adj Rsqr = 0.98394544

Standard Error of Estimate = 4.9188

	Coefficient	Std. Error	t	P
a	103.7819	3.0125	34.4506	<0.0001
b	0.0368	0.0026	14.2130	<0.0001

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	22267.0271	22267.0271	920.3141	<0.0001
Residual	14	338.7304	24.1950		
Total	15	22605.7575	1507.0505		

PRESS = 513.9303

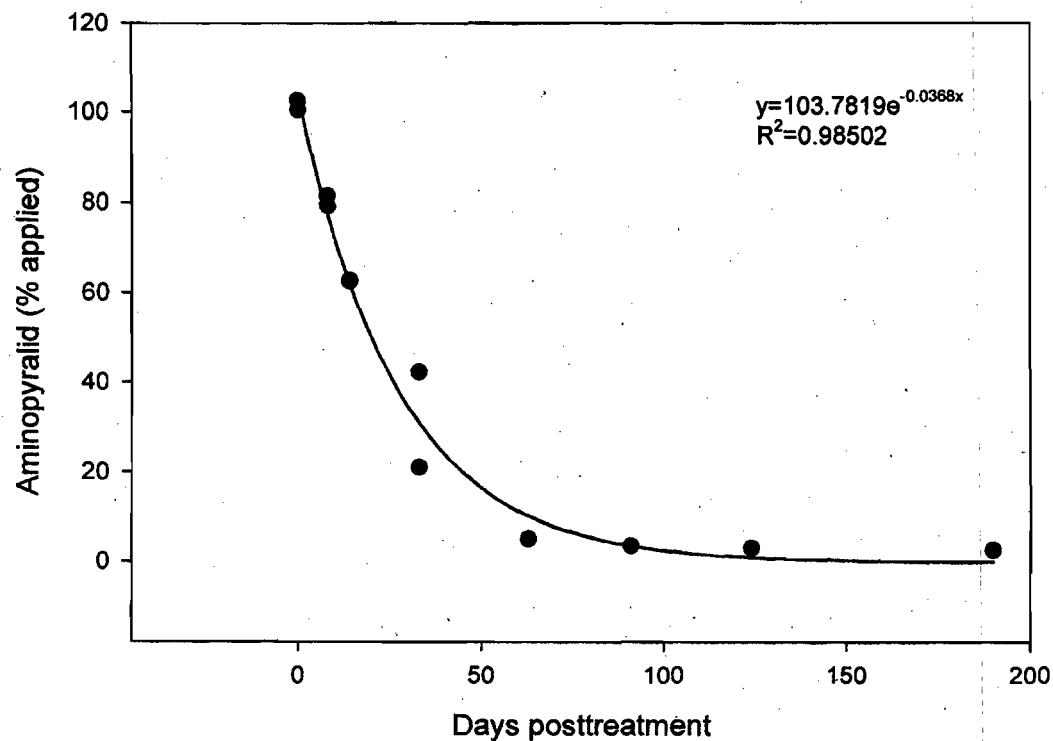
Durbin-Watson Statistic = 1.9691

Normality Test: Passed (P = 0.1872)

Constant Variance Test: Passed (P = 0.8910)

Power of performed test with alpha = 0.0500: 1.0000

Manning, 60 g/ha



Manning, 120 g/ha
Nonlinear Regression
 Equation: Single, 2 Parameter
 $y=ae^{-bx}$

R = 0.99142022 Rsqr = 0.98291405 Adj Rsqr = 0.98149023

Standard Error of Estimate = 5.4721

	Coefficient	Std. Error	t	P
a	105.3733	3.3346	31.5997	<0.0001
b	0.0357	0.0028	12.9355	<0.0001

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	20671.1073	20671.1073	690.3316	<0.0001
Residual	12	359.3248	29.9437		
Total	13	21030.4321	1617.7255		

PRESS = 549.6764

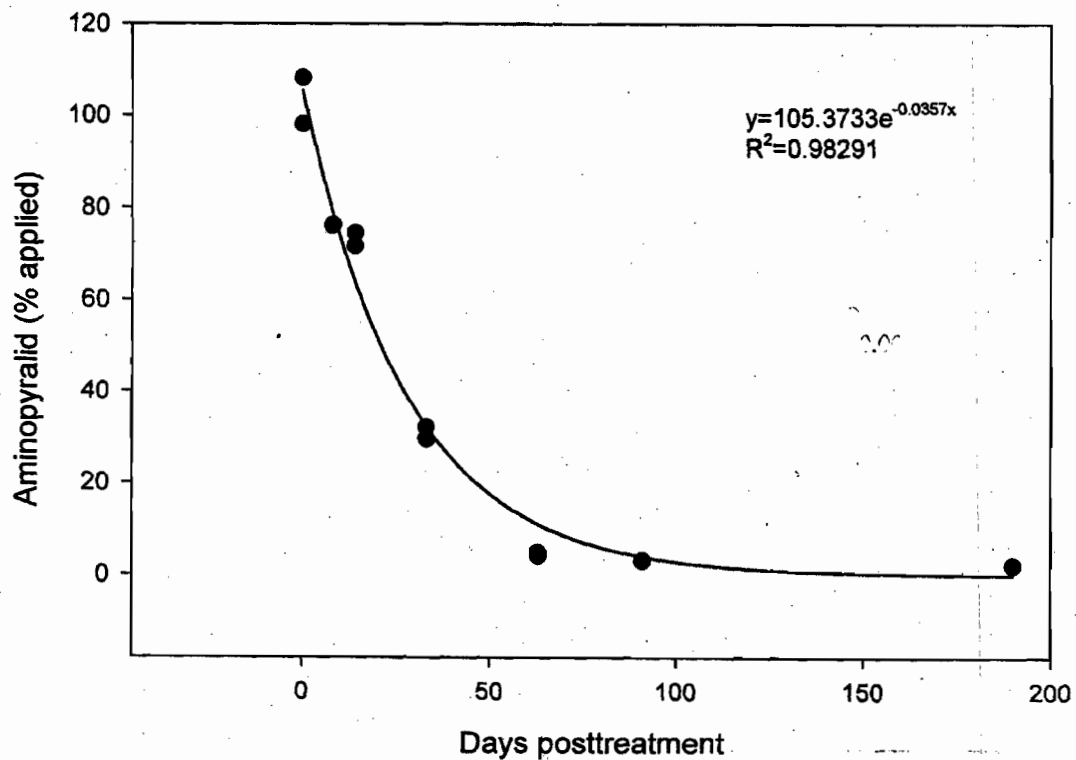
Durbin-Watson Statistic = 1.1983

Normality Test: Passed (P = 0.6595)

Constant Variance Test: Failed (P = 0.0470)

Power of performed test with alpha = 0.0500: 1.0000

Manning, 120 g/ha



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Manning, 120 g/ha, treating non-extractable residues as parent

Nonlinear Regression

Equation: Single, 2 Parameter

$$y=ae^{-bx}$$

R = 0.98726077 Rsqr = 0.97468384

Adj Rsqr = 0.97257415

Standard Error of Estimate = 6.0032

	Coefficient	Std. Error	t	P
a	102.7352	3.4184	30.0533	<0.0001
b	0.0269	0.0022	12.0248	<0.0001

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	16650.1090	16650.1090	462.0054	<0.0001
Residual	12	432.4653	36.0388		
Total	13	17082.5743	1314.0442		

PRESS = 552.9337

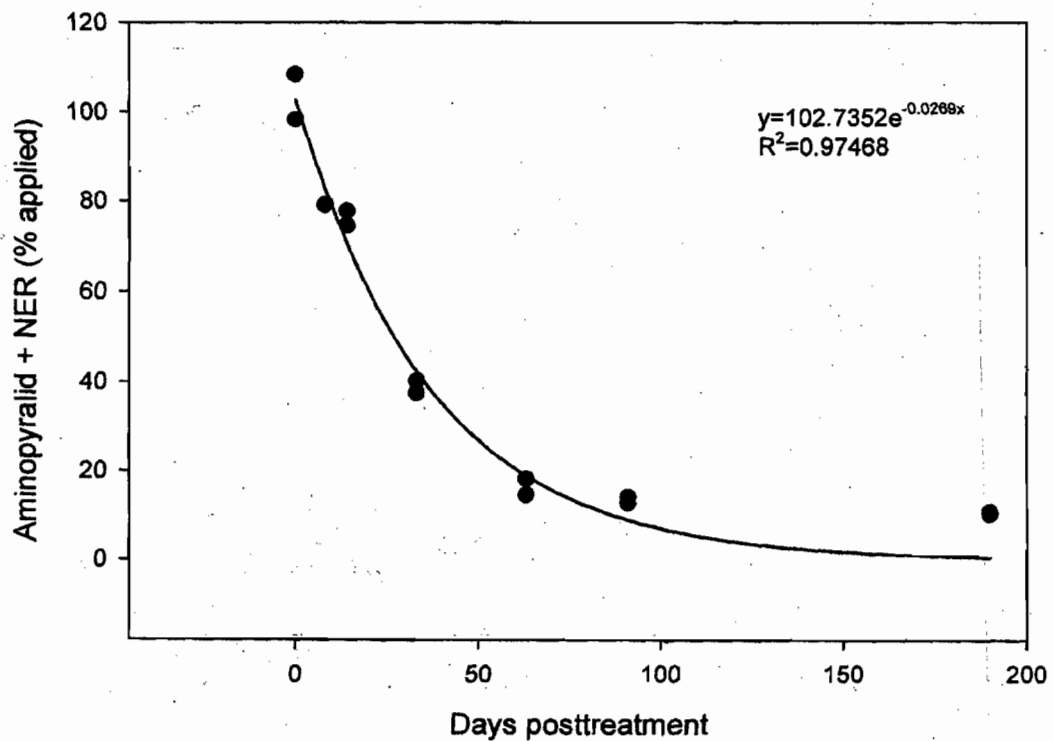
Durbin-Watson Statistic = 0.8215

Normality Test: Passed (P = 0.2166)

Constant Variance Test: Passed (P = 0.3642)

Power of performed test with alpha = 0.0500: 1.0000

Manning, 120 g/ha, treating non-extractables as parent



Barnes, 60 g/ha

Nonlinear Regression

Equation: Single, 2 Parameter

$$y=ae^{-bx}$$

R = 0.97369447 Rsqr = 0.94808093

Adj Rsqr = 0.94519654

Standard Error of Estimate = 3.9474

	Coefficient	Std. Error	t	P
a	90.9122	1.3724	66.2427	<0.0001
b	0.0021	0.0001	15.7111	<0.0001

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	5121.7721	5121.7721	328.6934	<0.0001
Residual	18	280.4799	15.5822		
Total	19	5402.2520	284.3291		

PRESS = 343.5886

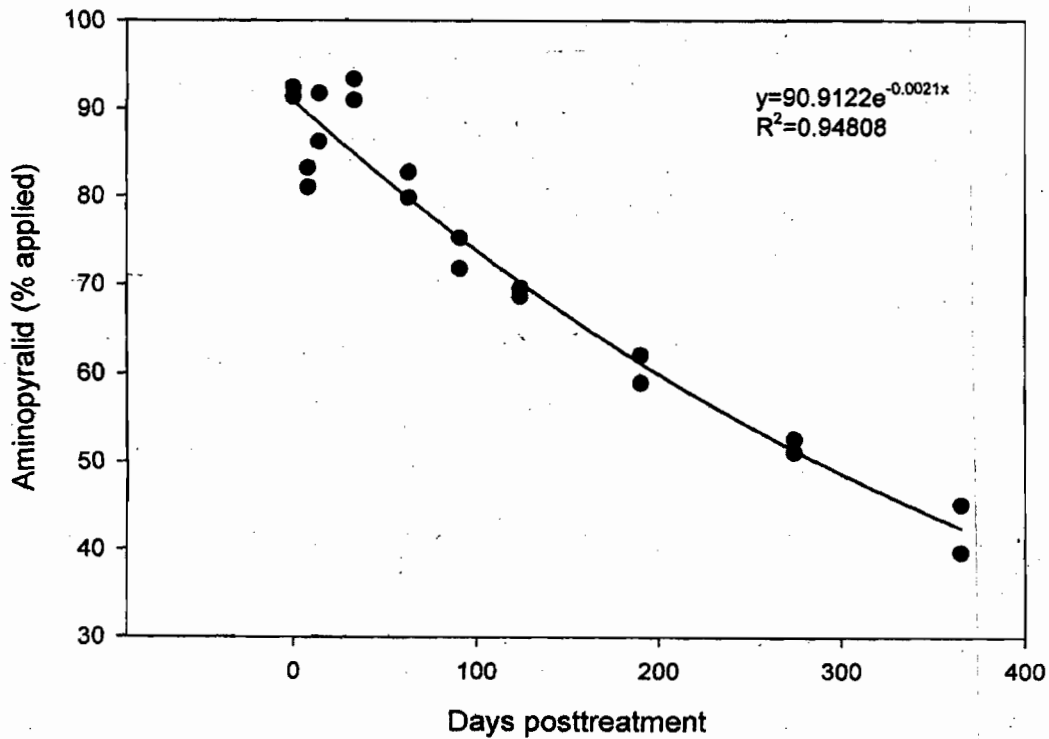
Durbin-Watson Statistic = 0.9423

Normality Test: Passed (P = 0.7756)

Constant Variance Test: Passed (P = 0.2437)

Power of performed test with alpha = 0.0500: 1.0000

Barnes, 60 g/ha



Barnes, 120 g/ha

Nonlinear Regression

Equation: Single, 2 Parameter

$y=ae^{-bx}$

R = 0.89173658 Rsqr = 0.79519414

Adj Rsqr = 0.78056515

Standard Error of Estimate = 8.3758

	Coefficient	Std. Error	t	P
a	89.4400	2.9663	30.1521	<0.0001
b	0.0021	0.0003	6.0768	<0.0001

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	3813.4086	3813.4086	54.3574	<0.0001
Residual	14	982.1607	70.1543		
Total	15	4795.5694	319.7046		

PRESS = 1214.7211

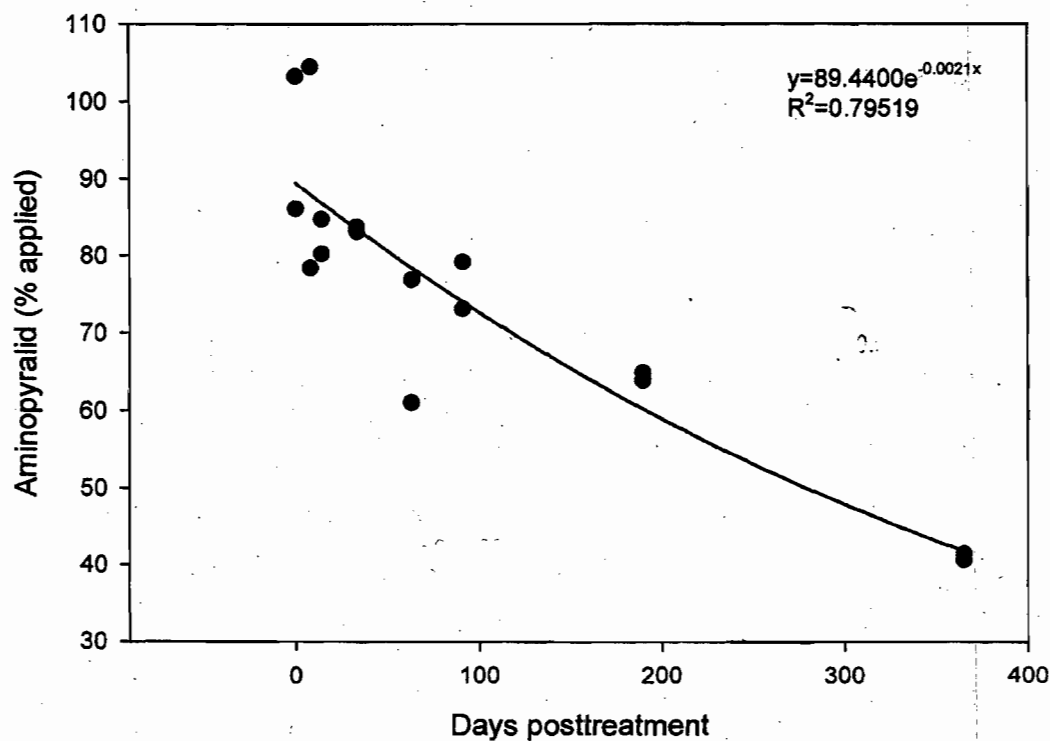
Durbin-Watson Statistic = 1.7209

Normality Test: Passed (P = 0.2170)

Constant Variance Test: Passed (P = 0.0938)

Power of performed test with alpha = 0.0500: 0.9993

Barnes, 120 g/ha



Barnes, 120 g/ha, treating non-extractable residues as parent

Nonlinear Regression

Equation: Single, 2 Parameter

$y=ae^{-bx}$

R = 0.82018301 Rsqr = 0.67270018 Adj Rsqr = 0.64932162

Standard Error of Estimate = 8.4301

	Coefficient	Std. Error	t	P
a	91.7519	2.8836	31.8189	<0.0001
b	0.0013	0.0003	4.7504	0.0003

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	2044.8837	2044.8837	28.7742	<0.0001
Residual	14	994.9307	71.0665		
Total	15	3039.8144	202.6543		

PRESS = 1225.1738

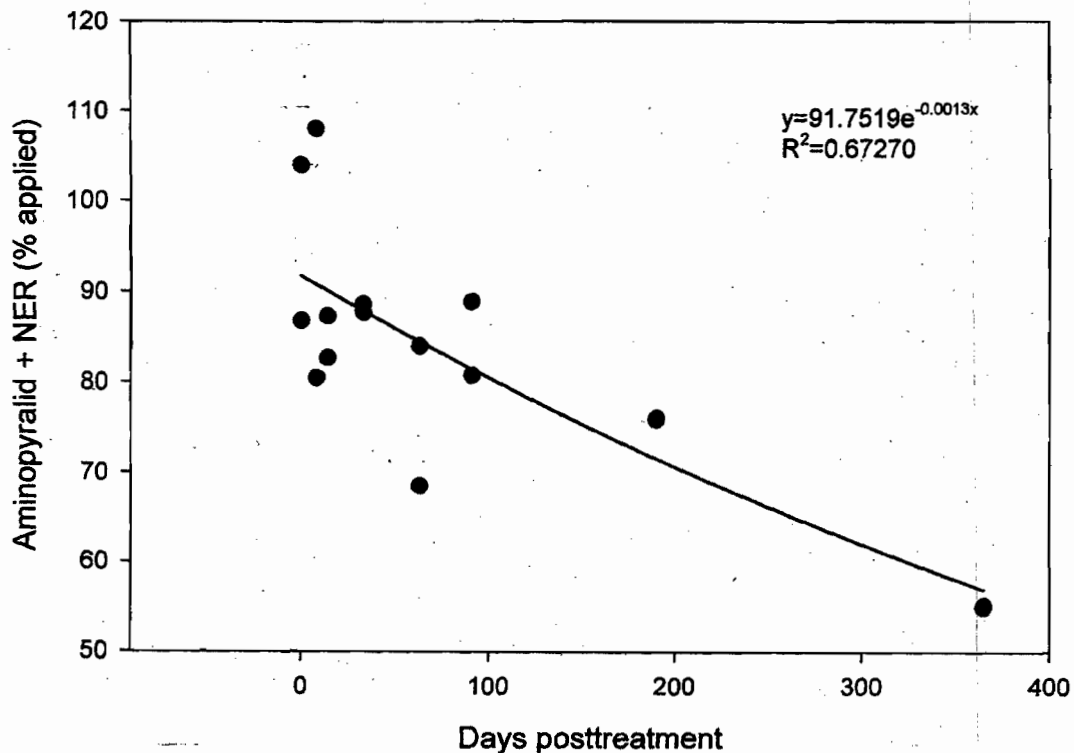
Durbin-Watson Statistic = 1.8041

Normality Test: Passed (P = 0.3900)

Constant Variance Test: Passed (P = 0.0843)

Power of performed test with alpha = 0.0500: 0.9866

Barnes, 120 g/ha, treating non-extractables as parent



Houston Black, 60 g/ha
Nonlinear Regression
 Equation: Single, 2 Parameter
 $y=ae^{-bx}$

R = 0.99515309 Rsqr = 0.99032968 Adj Rsqr = 0.98963895

Standard Error of Estimate = 3.2649

	Coefficient	Std. Error	t	P
a	83.4327	1.8784	44.4163	<0.0001
b	0.1736	0.0099	17.5069	<0.0001

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	15283.3916	15283.3916	1433.7291	<0.0001
Residual	14	149.2384	10.6599		
Total	15	15432.6300	1028.8420		

PRESS = 232.7760

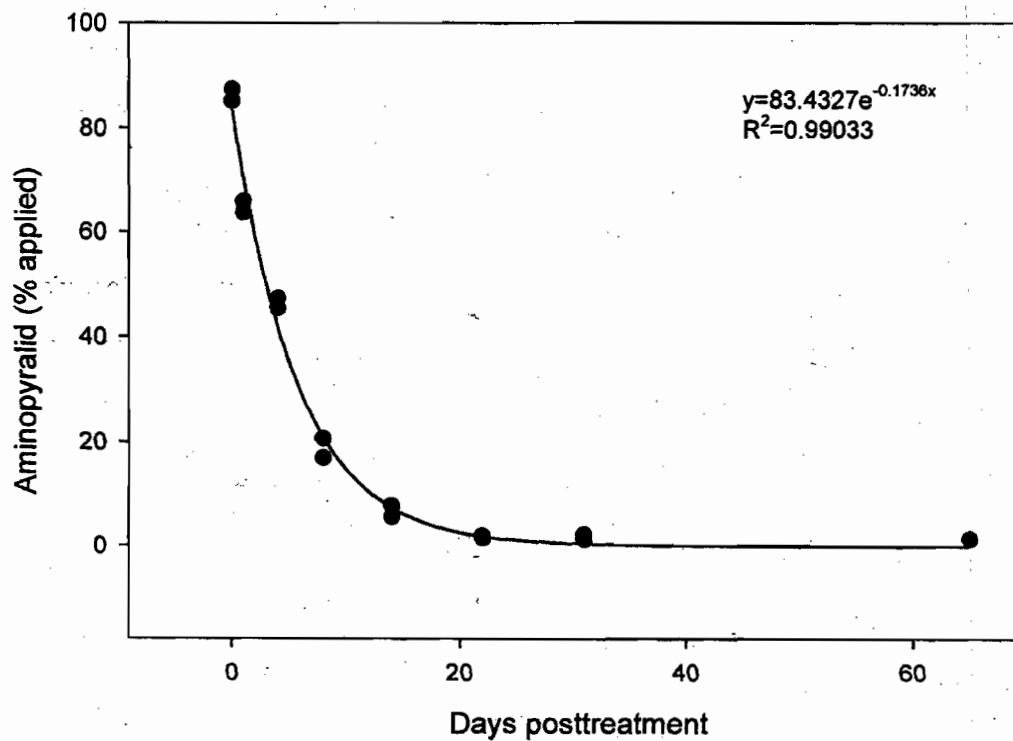
Durbin-Watson Statistic = 1.9190

Normality Test: Passed (P = 0.2616)

Constant Variance Test: Failed (P = 0.0221)

Power of performed test with alpha = 0.0500: 1.0000

Houston Black, 60 g/ha



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Houston Black, 120 g/ha
Nonlinear Regression
 Equation: Single, 2 Parameter
 $y=ae^{-bx}$

R = 0.97370220 Rsqr = 0.94809597 Adj Rsqr = 0.94438854

Standard Error of Estimate = 7.4419

	Coefficient	Std. Error	t	P
a	81.7521	3.9873	20.5030	<0.0001
b	0.1142	0.0139	8.2046	<0.0001

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	14162.5433	14162.5433	255.7286	<0.0001
Residual	14	775.3361	55.3811		
Total	15	14937.8794	995.8586		

PRESS = 1192.1991

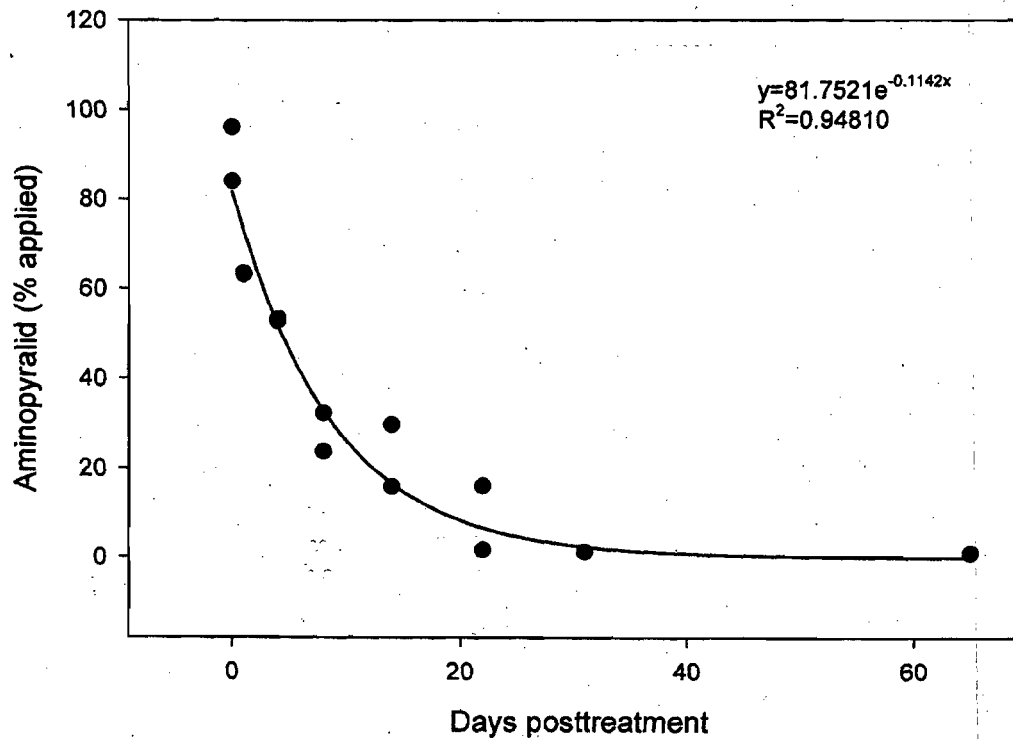
Durbin-Watson Statistic = 2.3617

Normality Test: Passed (P = 0.0509)

Constant Variance Test: Passed (P = 0.0715)

Power of performed test with alpha = 0.0500: 1.0000

Houston Black, 120 g/ha



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Houston Black, 120 g/ha, treating non-extractable residues as parent

Nonlinear Regression

Equation: Single, 2 Parameter

$$y=ae^{-bx}$$

R = 0.94809296 Rsqr = 0.89888026 Adj Rsqr = 0.89165742

Standard Error of Estimate = 9.3361

	Coefficient	Std. Error	t	P
a	93.9210	4.1480	22.6426	<0.0001
b	0.0388	0.0049	8.0056	<0.0001

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	10847.3988	10847.3988	124.4497	<0.0001
Residual	14	1220.2806	87.1629		
Total	15	12067.6794	804.5120		

PRESS = 1501.7492

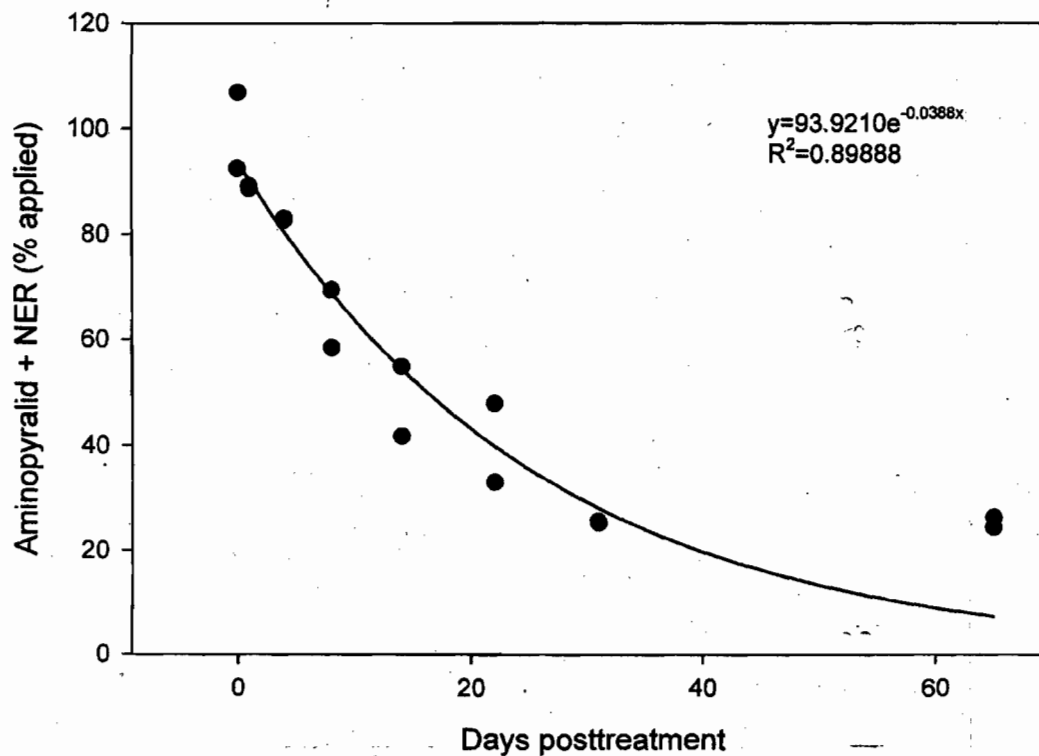
Durbin-Watson Statistic = 1.2861

Normality Test: Passed (P = 0.1158)

Constant Variance Test: Passed (P = 0.0675)

Power of performed test with alpha = 0.0500: 1.0000

Houston Black, 120 g/ha, treating non-extractables as parent



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Data Evaluation Report on the anaerobic aquatic metabolism of aminopyralid (XDE-750)

PMRA Submission Number xxxxxxxxx

EPA MRID Number 46235730



Data Requirement:	PMRA DATA CODE:	8.2.3.5.6
	EPA DP Barcode:	D306825
	OECD Data Point:	IIA 7.8.2
	EPA Guideline:	USEPA Subdivision N, Section 162-3

Test material:

Common name: Aminopyralid.

Chemical name:

IUPAC: 4-amino-3,6-dichloropyridine-2-carboxylic acid.

CAS name: 4-amino-3,6-dichloro-2-pyridinecarboxylic acid.

CAS No.: 150114-71-9.

Synonyms: XDE-750, DE-750.

Primary Reviewer :(#1615), PMRA **Date:** 2004-12-10

Secondary Reviewer :(#1032), PMRA **Date:** 2004-12-20

Secondary Reviewer: Roxolana Kashuba, **Date:** 2005-02-28
EPA/EFED/ERB4

Roxolana Kashuba 5/12/05

Company Code: DOW

Active Code: AMD

Use Site Category: 13 (terrestrial feed crops), 14 (terrestrial food crops) and 16 (industrial and domestic vegetation control non-food sites)

EPA PC Code: 005100

CITATION: Rutherford L. A. and T.J. Meitl. 2004. Anaerobic Aquatic Metabolism of XDE-750. Regulatory Laboratories—Indianapolis Lab (Indianapolis, Indiana), Dow AgroSciences LLC, Study number 020080, January 16, 2004, 753 pages. Unpublished.



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PMRA Submission Number xxxxxxxxx

EPA MRID Number 46235730

EXECUTIVE SUMMARY:

The anaerobic biotransformation of [2,6-¹⁴C]-labelled 4-amino-3,6-dichloropyridine-2-carboxylic acid (aminopyralid; XDE-750) was studied in a pond water/sediment system (water pH 7.9, dissolved organic carbon 37.2 ppm; sediment texture sandy loam, pH 8.1, organic carbon 4.9%) from North Dakota (USA) for 363 days in the dark at 25.5 °C. [2,6-¹⁴C]Aminopyralid was applied at the rate of 0.084 mg a.i./L. The sediment/water ratio used was 1:4. The anaerobic biotransformation of [2,6-¹⁴C]aminopyralid was also studied in a flooded soil system using a Cuckney soil from Bedfordshire (England) (soil texture sand, pH 6.0, organic carbon 1.3%) and HPLC grade water for 120 days in the dark at 19.5 °C. [2,6-¹⁴C]Aminopyralid was applied to water surface at the rate of 0.082 mg a.i./L. The soil/water ratio used was 1:2. The experiment was conducted in accordance with the US EPA Subdivision N, Section 162-3, SETAC Section 8.1, and Canada PMRA DACO Number 8.2.3.5.6, and to meet the GLP standards, the US EPA Good Laboratory Practice Standards, 40 CFR Part 160.

The test system consisted of two-chambered biometer flasks with traps for the collection of CO₂. Prior to treatment, flasks containing samples of sediment or soil and water were incubated in the dark at 20 °C (Cuckney soil) or 25 °C (North Dakota sediment) for at least 30 days to obtain anaerobic conditions. The samples were then dosed and purged with nitrogen to remove the oxygen that had entered the test system during dosing. Cuckney samples were analyzed at 0, 3, 10, 20, 30, 59, and 120 days of incubation. North Dakota samples were analyzed at 0, 10, 20, 30, 90, 181, 268, and 363 days of incubation. Aliquots of the water were directly analyzed by LSC and HPLC. The Cuckney soil samples were extracted on a horizontal shaker at low speed with an acetone/1.0 N HCl (90:10, v:v) solution. The North Dakota sediment samples were extracted on a horizontal shaker at low speed with a methanol/1.0 N NaOH (90:10, v:v) solution. [2,6-¹⁴C]Aminopyralid residues were analyzed by LSC and HPLC. Identification of transformation products was not attempted because there were no transformation products present at greater than 2% of applied.

The test conditions outlined in the study protocol were maintained throughout the study. More specifically, the anaerobic conditions were maintained as indicated by the redox potential values measured for both water and sediment/soil in both North Dakota and Cuckney systems (-495 to -76 mV in replicate samples).

The total material balances in the North Dakota water/sediment system and the flooded Cuckney soil system were 97.9 ± 1.6 % and 95.5 ± 2.8 % of the applied radioactivity, respectively. In the North Dakota system, the mean recovery of the radiolabelled material was 64.3 ± 2.9 % and 33.2 ± 2.8 % of the applied radioactivity in the water and sediment, respectively. In the Cuckney system, the mean recovery of the radiolabelled material was 66.1 ± 4.5 % and 29.2 ± 3.8 % of the applied radioactivity in the water and sediment, respectively. Extractable [¹⁴C] residues in the sediment increased from 27.4% at day 0 to 36.9% of the applied

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radioactivity at the end of the incubation period in the North Dakota system, and decreased from 30.0% at day 0 to 21.5% of the applied radioactivity at the end of the incubation period in the Cuckney system. Non-extractable [^{14}C] residues in the sediment ranged from 0.9% to 2.4% of the applied radioactivity and from 0.7% to 1.3% of the applied throughout the study for the North Dakota and Cuckney systems, respectively. At the end of the study 0.6% and 0.4% of the applied radioactivity were present as CO_2 in the North Dakota and Cuckney systems, respectively.

The concentration of [2,6- ^{14}C]aminopyralid in water ranged from 61.5% to 69.5% and from 59.8% to 71.7% of the applied radioactivity throughout the study in North Dakota and Cuckney water, respectively. The concentration of [2,6- ^{14}C]aminopyralid in the sediment increased from 27.3% at day 0 to 36.7% of the applied radioactivity at the end of the study period in the North Dakota system, and ranged from 21.5% to 32.3% of the applied throughout the study period in the Cuckney system. No attempt was made to measure transformation products as unidentified residues in both water and sediment were less than 1.1% and 0.7% of applied radioactivity at every interval in the North Dakota and Cuckney systems, respectively.

[2,6- ^{14}C]Aminopyralid was stable in both the North Dakota water/sediment and flooded Cuckney soil systems. A t-test showed that the slope of the degradation curve was no different than zero.

Results Synopsis:

Test system used: One sediment/pond water system; North Dakota, USA, and one flooded soil system: Bedfordshire, England.

Half-life/ DT_{50} in sediment and entire system: [2,6- ^{14}C]Aminopyralid was stable in the flooded Cuckney soil test system and the North Dakota sediment test system under anaerobic conditions.

Major transformation products: No major transformation products were observed

Minor transformation products: CO_2 and non-extractable residues.

Study Acceptability: This study is classified as acceptable for an anaerobic biotransformation study in water-sediment system.

I. MATERIALS AND METHODS

GUIDELINE FOLLOWED: EPA Pesticide Registration Guidelines, Subdivision N, §162-3, Canada PMRA DACO Number 8.2.3.5.6 - Biotransformation in Aquatic System-Anaerobic Sediment/Water and SETAC – Europe Procedures for Assessing the Environmental Fate and Ecotoxicity of Pesticides, Section 8.1 guidelines.

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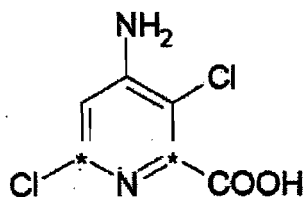
EPA MRID Number 46235730

COMPLIANCE: Good Laboratory Practices standards, 40 CFR Part 160

A. MATERIALS:

1. Test Material [2,6-¹⁴C]XDE-750 (aminopyralid; p. 18; Figure 1, p.53).

Chemical Structure:



Description: Technical, solid (p. 19).

Purity: Analytical purity: N/A.
Radiochemical purity: 99.6% , INV1590.
Specific activity: 27.4 mCi/mmmole
Locations of the radiolabel: 2,6-¹⁴C (Figure 1, p.53).

Storage conditions of test chemicals: stored in a freezer, maintained in the dark (p. 19).

Physico-chemical properties of aminopyralid (XDE-750):

Parameter	Values	Comments
Water solubility		
pH 5	212 g/L	at 20 °C (1)
pH 7	205 g/L	at 20 °C (1)
pH 9	203 g/L	at 20 °C (1)
unbuffered	2.48 g/L	at 20 °C (1)
Vapour pressure	1.94 x 10 ⁻¹⁰ mm Hg	at 25 °C (2)
UV absorption	N/A	N/A
pKa	2.56	at 20 °C (3)
Log Kow		
unbuffered	0.201	at 20 °C (4)
pH 5	- 1.75	at 20 °C (4)
pH 7	- 2.87	at 20 °C (4)
pH 9	- 2.96	at 20 °C (4)
Stability of compound at room temperature, if provided	N/A*	

Data were obtained from p. 19 of the study report.

*Samples were stored refrigerated or frozen.

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2. Water-sediment collection, storage and properties

Table 1a: Description of sediment collection and storage for North Dakota sediment and pond water.

Description	Details
Geographic location	Steele County, ND, USA
Pesticide use history at the collection site	Not available
Collection procedures for water and sediment:	12 sites approximately 9 feet apart into plastic, 5-gallon bucket; collect approximately one part sediment to two parts water, filling each container completely to exclude headspace
Sampling depth for water and sediment:	sampled to a 3-inch depth below the organic/mineral interface
Storage conditions	Ambient temperature
Storage length prior to use	19 days
Preparation of water and sediment samples (eg: water -filtered/not filtered; sediment -sieved/not sieved)	Water was filtered through glass wool and sediment was sieved (2 mm).

Data were obtained from p. 20-21 and Appendix B, p. 68.

Table 1b: Description of soil collection and storage for Cuckney soil.

Description	Details
Geographic location	Bedfordshire, England
Site description	Cropland, Oilseed rape
Pesticide use history at the collection site	Ammonium Sulphate, Ceres Promise, Poraz, Genie 25, Trifluralin, Butisan (Alpha metachlor), Roundup (time of application in relation to sampling not mentioned)
Collection procedures for soil:	Clean spade, 10-12 spots within a 50' x 50' plot, into plastic bag in plastic box
Sampling depth for water and sediment:	0-20 cm
Storage conditions	4° C
Storage length prior to use	Approximately 3 months

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Preparation of soil prior to use:	Soil was sieved (2 mm).
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Data were obtained from p. 20-21 and Appendix B, p. 69.

Table 2: Properties of the water.

Property	Details
Temperature (°C)	20°C
pH	7.9
Redox potential (mv)	Initial: - 355.4 mV Final: - 125.7 mV
Oxygen concentration (mg/L)	Initial: 0.10 ppm Final: -0.21 ppm
Dissolved organic carbon	37.2 ppm
Hardness (CaCO ₃)	669 ppm
Electrical conductivity	1.71 mmhos/cm
Biomass (mg microbial C/100 g or CFU or other)	Not stated

Data were obtained from p. 20-21 and Table 2, p. 41.

Table 3: Properties of the soil and sediment.

Property	Details	
	Cuckney Soil	North Dakota Sediment
Textural classification	sand	sandy loam
% sand	89	57
% silt	8	36
% clay	3	7
pH ^a	6.0	8.1
Organic carbon (%) ^b	2.4	6.0
CEC (meq/100 g)	1.3	4.9
Initial redox potential (mv)	- 469.2	- 430.6
Final redox potential (mv)	- 410.3	- 261.6
Bulk density (g/cm ³)	1.28	0.67
Initial biomass (mg microbial C/100 g or CFU or other)	111.4	42.7
Final biomass (mg microbial C/100 g or CFU or other)	39.5	54.3

Data were obtained from p. 20-21 and Table 1, p. 40.

^apH in 1:1 soil: water ratio

^bWalkley Black Method with Heat

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B. EXPERIMENTAL DESIGN:

1. Preliminary experiments: No preliminary experiments were conducted.

2. Experimental conditions: The current anticipated maximum application rate of aminopyralid is 120 g a.i./ha. Assuming the entire mass of aminopyralid is applied and an overspray to a 15-cm deep body of water, the expected environmental concentration in the water phase would be 0.08 µg/mL (p. 18).

Table 4: Study design.

Parameter	Details	
	Cuckney Soil	North Dakota Sediment
Duration of the test	120 days	363 days
Water	HPLC grade water	Associated pond water filtered through glass wool
Amount of sediment and water/ treatment	50 g dry wt soil, 100 mL water	30 g dry wt sediment, 120 mL water
Water/sediment ratio	1:2	1:4
Application rates (mg a.i./L)	0.082 mg a.i./L	0.084 mg a.i./L
Control conditions, if used	N/A	N/A
No. of replications		
Control, if used:	N/A	N/A
Treatments:	2	2
Test apparatus	two-chambered biometer flask	two-chambered biometer flask
Type/material/volume	250-mL Erlenmeyer	250-mL Erlenmeyer
Details of traps for CO ₂	0.2 M NaOH	0.2 M NaOH
If no traps were used, is the system closed	N/A	N/A
Identity and concentration of co-solvent	Acetonitrile, 0.08%	Acetonitrile, 0.08%

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Pesticide application method		A volume of 83 μ L of test solution was applied to water surface using a positive displacement pipet (no mixing).		A volume of 100 μ L of test solution was applied to water surface using a positive displacement pipet (no mixing).	
Volume of the test solution used/treatment: Application method (eg: mixed/not mixed)					
Any indication of the test material adsorbing to the walls of the test apparatus		No		No	
Microbial biomass/microbial population of the control (μ g/g dry wt soil)		Initial	N/A	Initial	N/A
		Final	N/A	Final	N/A
Microbial biomass/microbial population of the treated (μ g/g dry wt soil)		Initial	111.4	Initial	42.7
		Final	39.5	Final	54.3
Experimental conditions:	Temperature ($^{\circ}$ C):	19.5 \pm 1		25.5 \pm 1	
	Continuous darkness (Yes/No)	Yes		Yes	
Other details, if any		None			

Data were obtained from pp. 21-23 and Table 3, p. 42 of the study report.

3. Anaerobic conditions: Samples were incubated in two-chambered biometer flasks; one side of the biometer contained the sediment and pond water or soil and distilled water while the other chamber held 0.2 M NaOH solution for collection of CO₂. The sediment/soil side of each flask was closed with a ground glass stopper, using vacuum grease to create an airtight seal. For the Cuckney soil, each flask contained 50 g (oven dry weight) of moist soil. Enough HPLC water was added so the total amount of water in the system was 100 mL (1:2 soil:solution ratio). For the North Dakota sediment, each flask contained 30 g (oven dry weight) of moist sediment. Enough pond water was added so the total amount of water in the system was 120 mL (1:4 sediment:solution ratio) (p.21).

Duplicate biometer flasks of each sediment or soil type were prepared for each time point. Additional flasks of each test system were prepared for a biomass measurement at the end of the study. Prior to treatment, samples were purged with nitrogen and incubated in the dark at 20 $^{\circ}$ C (Cuckney soil) or 25 $^{\circ}$ C (North Dakota sediment) for at least 30 days to allow the samples to obtain anaerobic conditions. Several samples were used to determine the test system oxygen content and redox potential prior to sample dosing. The oxygen content and redox potential of each samples was monitored at each sample point as well. After treatment, the samples were purged with nitrogen and returned to the dark incubators (p.21-22).

4. Supplementary experiments: No supplemental studies were conducted.

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5. Sampling:

Table 5: Sampling details.

Criteria	Details	
	Cuckney Soil	North Dakota Sediment
Sampling intervals	0, 3, 10, 20 days, 1,2, and 4 months post application	0, 10, 20 days, 1,3, 6, 9, and 12 months post application
Sampling method	Extracted 4x with 90:10 acetone: 1.0 N HCl.	Extracted 4x with 90:10 methanol: 1.0 N NaOH.
Method of collection of CO ₂ and organic volatile compounds	Aspiration of NaOH trap, followed by LSC counting of 1 mL aliquots	Aspiration of NaOH trap, followed by LSC counting of 1 mL aliquots
Sampling intervals/times for:		
treatments:	0, 3, 10, 20 days, 1,2, and 4 months post application	0, 10, 20 days, 1,3, 6, 9, and 12 months post application
controls:	N/A	N/A
redox potential:	0, 3, 10, 20 days, 1,2, and 4 months post application	0, 10, 20 days, 1,3, 6, 9, and 12 months post application
Sample storage before analysis	Refrigeration for aqueous layer and concentrated soil extract, freezer for soil extract	Refrigeration for aqueous layer and concentrated sediment extract, freezer for sediment extract
Other observations, if any	Microbial biomass measurements at beginning and end of study	Microbial biomass measurements at beginning and end of study

Data were obtained from pp. 23-24 and Table 4, p. 43 of the study report.

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C. ANALYTICAL METHODS:

At each sampling time point (except Time 0) approximately 20 mL of the caustic trapping solution was transferred by aspirator to a glass scintillation vial (the rest was discarded as waste). Triplicate aliquots of the trapping solution were counted by LSC to determine mineralization to CO₂. Since no radioactivity was presumed present in the Time 0 traps, they were not assayed (p. 24).

The pH, dissolved oxygen content and redox potential of each aqueous sample was measured immediately upon sample sacrifice. The redox potential of the sediment was also monitored (p. 24).

The entire sample was transferred to a weighed 250-mL Nalgene bottle for extraction. The sample bottle was then centrifuged at 2500 rpm for 10 minutes. The aqueous solution was decanted into a tared container and the aqueous weight recorded. Aliquots were assayed by LSC and HPLC. Immediately after decanting the aqueous phase, the sediment/soil layer was weighed to determine the mass of water remaining in the sediment/soil (entrained water). Approximately 65 mL of 90:10 acetone: 1.0 N HCl was added to the Cuckney sand soil pellets or 65 mL of 90:10 methanol:1.0 N NaOH was added to the North Dakota sandy loam sediment pellets, and the sample was vortexed and sonicated for 15 minutes to break up the soil or sediment pellet. The sample was placed on a horizontal shaker at low speed for 1 hour and then centrifuged for approximately 15 minutes at approximately 2800 rpm. The extract was then decanted into a weighed, labeled jar and 65 mL fresh appropriate organic solvent was added to the soil or sediment pellet, vortexing, sonicating, shaking and centrifuging as before. The extracts were combined and the extraction process was repeated twice more with 65 mL of organic solvent for a total of 4 extractions. The combined extract was weighed and triplicate aliquots (0.5 mL) were assayed for ¹⁴C by LSC. The average density of the extracted sample was determined by weighing aliquots of several different sample extracts; this average density was used to determine the volume of the sample extract from the measured weight (p. 24-25).

The extracted sediment or soil pellet was allowed to air dry in a hood for at least one week prior to combustion analysis to determine the amount of non-extractable residues present (p. 24-25).

Organic extracts were concentrated and filtered prior to HPLC analysis. Aqueous and concentrated organic samples were analyzed by reverse phase HPLC (Intertsil ODS, 5- μ m) using an isocratic system of 90:10 water: acetonitrile, each containing 0.5% TFA (p. 26; Table 5, p. 44).

Transformation products were not identified nor quantified (p. 26). Using the method of Curie, the limit of detection (LOD) was 13 or 10 dpm above background and the limit of quantitation (LOQ) was 57 or 40 dpm above background, for samples and for blanks, respectively (p. 31).

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II. RESULTS AND DISCUSSION:

A. TEST CONDITIONS: Anaerobic conditions were maintained throughout the study. Samples were visibly anaerobic when caustic trapping material was pushed up into the expansion bulb. Measurements of the water D.O. content and the sediment and water redox potential confirmed anaerobic conditions. Samples were maintained in the dark at $25.5 \pm 1^\circ\text{C}$ for up to 363 days after treatment for the North Dakota sediment and $19.5 \pm 1^\circ\text{C}$ for up to 120 days after treatment for the Cuckney soil. Sediment and soil biomass determined at study initiation and termination are presented in Table 3. The microbial biomass decreased in the soil over the duration of the experiment, but slightly increased in the sediment (p. 32; Table 1, p. 40).

B. MATERIAL BALANCE: The total material balance in the North Dakota water/sediment system was $97.9 \pm 1.6\%$ of the applied radioactivity. The total material balance in the flooded Cuckney soil system was $97.9 \pm 1.6\%$ of the applied radioactivity. In the North Dakota system, the mean recovery of the radiolabelled material was $64.3 \pm 2.9\%$ and $33.2 \pm 2.8\%$ of the applied radioactivity in the water and sediment, respectively. In the Cuckney system, the mean recovery of the radiolabelled material was $66.1 \pm 4.5\%$ and $29.2 \pm 3.8\%$ of the applied radioactivity in the water and sediment, respectively (Tables 11 and 12 and Appendix C, pp. 50, 51, 71, and 72).

Table 7: Biotransformation of [2,6-¹⁴C]aminopyralid, expressed as percentage of applied radioactivity, in water-sediment system North Dakota sediment under anaerobic conditions.

Compound	Media	Sampling times (days)							
		0	10	20	30	90	181	268	363
Aminopyralid	water	69.5 ± 0.7	62.2 ± 0.6	65.5 ± 0.1	65.5 ± 2.4	62.5 ± 0.1	63.5 ± 0.6	62.0 ± 0.1	61.5 ± 0.8
	sediment	27.3 ± 0.6	29.9 ± 0.1	30.4 ± 0.1	30.5 ± 1.6	33.1 ± 1.8	31.0 ± 0.1	31.6 ± 0.1	36.7 ± 0.6
Unidentified radioactivity	water	1.1 ± 1.0	0.1 ± 0.1	0.1 ± 0.1	0.1 ± 0.1	0.0 ± 0.0	0.4 ± 0.1	0.4 ± 0.2	0.4 ± 0.0
	sediment	0.1 ± 0.0	0.5 ± 0.3	0.1 ± 0.1	0.1 ± 0.1	0.1 ± 0.1	0.1 ± 0.1	0.6 ± 0.2	0.2 ± 0.1
CO ₂	entire system	N/A	0.2 ± 0.0	0.3 ± 0.1	0.4 ± 0.1	0.6 ± 0.1	0.7 ± 0.0	0.7 ± 0.0	0.6 ± 0.0
Bound residues	sediment	0.9 ± 0.0	1.8 ± 0.3	1.7 ± 0.1	1.8 ± 0.6	2.0 ± 0.6	2.4 ± 0.6	2.0 ± 0.1	1.2 ± 0.0

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Material balance	water	70.6 +0.3	62.2 +0.6	65.6 +0.1	65.6 +2.5	62.5 +0.1	63.9 +0.5	62.4 +0.1	61.9 +0.8
	sediment	28.3 +0.6	32.2 +0.1	32.2 +0.4	32.3 +2.3	35.1 +1.3	33.4 +0.7	34.1 +0.1	38.1 +0.8
	entire system	98.9 +0.4	94.6 +0.4	98.0 +0.5	98.3 +0.1	98.2 +1.0	98.0 +0.2	97.1 +0.1	100.5 +0.0

Data were obtained from Table 12 and Appendix C, pp. 51 and 72 of the study report.

Table 8: Biotransformation of [2,6-¹⁴C]aminopyralid, expressed as percentage of applied radioactivity (mean ± s.d.), in Cuckney flooded soil system under anaerobic conditions.

Compound	Media	Sampling times (days)						
		0	3	10	20	30	59	120
Aminopyralid	water	69.2 +2.8	59.8 +3.4	61.7 +0.9	63.5 +2.9	67.0 +0.8	68.4 +0.7	71.7 +0.4
	sediment	29.9 +2.2	32.3 +1.2	31.8 +2.1	29.2 +0.7	28.3 +0.4	25.5 +1.1	21.5 +0.4
Unidentified radioactivity	water	0.0 +0.0	0.2 +0.0	0.1 +0.1	0.2 +0.1	0.2 +0.2	0.7 +0.9	0.0 +0.0
	sediment	0.1 +0.0	0.1 +0.1	0.1 +0.1	0.2 +0.0	0.0 +0.0	0.1 +0.1	0.0 +0.0
CO ₂	entire system	N/A	0.1 +0.1	0.2 +0.0	0.2 +0.0	0.3 +0.1	0.3 +0.0	0.4 +0.0
Bound residues	sediment	0.9 +0.1	0.7 +0.0	0.8 +0.1	1.0 +0.1	1.3 +0.0	0.7 +0.2	0.7 +0.1
Material balance	water	69.2 +2.8	60.0 +3.4	61.7 +0.8	63.7 +3.0	67.2 +0.6	69.1 +1.6	71.7 +0.4
	sediment	30.8 +2.3	33.0 +1.1	32.6 +2.1	30.4 +0.6	29.6 +0.4	26.2 +1.4	22.2 +0.2
	entire system	100.0 +0.6	93.1 +4.5	94.5 +1.3	94.3 +3.6	97.0 +0.4	95.6 +0.2	94.3 +0.2

Data were obtained from Table 11 and Appendix C, pp. 50 and 71 of the study report.

C. TRANSFORMATION OF PARENT COMPOUND:

In the North Dakota system, the concentration of [2,6-¹⁴C]aminopyralid in water ranged from 61.5% to 69.5% of the applied radioactivity throughout the study. The concentration of [2,6-¹⁴C]aminopyralid in the sediment increased from 27.3% at day 0 to 36.7% of the applied radioactivity at the end of the study (363 days) (Table 12 and Appendix C, pp. 51 and 72).

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EPA MRID Number 46235730

In the Cuckney system, the concentration of [2,6-¹⁴C]aminopyralid in water ranged from 59.8% to 71.7% of the applied radioactivity at study termination. The concentration of [2,6-¹⁴C]aminopyralid in the soil ranged from 21.5% to 32.3% of the applied radioactivity throughout the study period (Table 11 and Appendix C, pp. 50 and 71).

For the North Dakota sediment, the K_d values ranged from 0.44 to 0.65 L/kg, while the K_{oc} values ranged from 9.0 to 13.2 L/kg. For the Cuckney soil, the K_d values ranged from 0.27 to 0.48 L/kg, while the K_{oc} values ranged from 20.9 to 37.1 L/kg. Sorption coefficients were in range with those observed in the batch equilibrium sorption study (6) (p. 35; Table 13, p. 52).

Table 9: Non-equilibrium sorption coefficients of [2,6-¹⁴C]aminopyralid in water-sediment system North Dakota sediment and flooded soil system Cuckney soil under anaerobic conditions.

Test system	Sampling point (days)	K_d (L/kg)	K_{oc} (L/kg)
North Dakota sediment	0	0.45	9.2
	10	0.51	10.5
	20	0.56	11.4
	30	0.56	11.4
	90	0.60	12.3
	181	0.57	11.7
	270	0.55	11.2
	363	0.63	12.9
	Average:		0.55
Cuckney soil	0	0.40	30.8
	3	0.48	37.0
	10	0.44	34.0
	20	0.43	32.9
	30	0.39	30.4
	59	0.33	25.7
	120	0.27	21.0
	Average:		0.39

Data were obtained from Table 13, p. 52 of the study report.

1. Half-life: [2,6-¹⁴C]Aminopyralid was stable in anaerobic water-sediment and water-soil systems. T-tests of the slope from linear regression on log-transformed data indicated that the slopes of the degradation curves for both test systems were not statistically different from zero, signifying that [2,6-¹⁴C]aminopyralid did not degrade.

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Data Evaluation Report on the anaerobic aquatic metabolism of aminopyralid (XDE-750)

PMRA Submission Number xxxxxxxxx

EPA MRID Number 46235730

Half-lives/DT50

		Half-life (days)	Regression equation ^a	R ²	DT50	DT90
North Dakota sediment	water	stable				
	soil	stable				
	entire system	stable	Y = 0.00005X + 4.5527	0.0483	N/A	N/A
Cuckney soil	water	stable				
	sediment	stable				
	entire system	stable	Y = -0.0001X + 4.5502	0.0410	N/A	N/A

Data were obtained from Tables 11 and 12 and Appendix C, pp. 50, 51, 71, and 72.

^a A t-test was performed on the linear regression of log-transformed data and it showed that the slope of each line was no different from zero at the 99% confidence level.; therefore, [2,6-¹⁴C]aminopyralid was stable under anaerobic conditions.

2. Transformation Products: No attempt was made to measure transformation products in water or sediment/soil. Unidentified radioactivity in both water and sediment was less than 1.1% and 0.7% of applied radioactivity at every interval in the North Dakota and Cuckney systems, respectively. [2,6-¹⁴C]Aminopyralid accounted for 95% or greater of the radioactivity extractable from any sample (Tables 11,12, and Appendix C, pp. 50, 51, 71, 72).

3. Non-Extractable and Extractable Residues: Extractable [¹⁴C] residues in the sediment increased from 27.4% at day 0 to 36.9% of the applied radioactivity at the end of the incubation period in the North Dakota system, and decreased from 30.0% at day 0 to 21.5% of the applied radioactivity at the end of the incubation period in the Cuckney system. Non-extractable [¹⁴C] residues in the sediment ranged from 0.9% to 2.4% of the applied radioactivity and from 0.7% to 1.3% of the applied throughout the study for the North Dakota and Cuckney systems, respectively (Tables 11,12, and Appendix C, pp. 50, 51, 71, 72). Because the amount of non-extractable residues was below 3% of applied, the NER was not characterized.

4. Volatilization: The amount of mineralization was low, less than 1% of applied radioactivity was recovered from any sample. At the end of the study 0.6% and 0.4% of the applied radioactivity were present as CO₂, in the North Dakota and Cuckney systems, respectively (Tables 11,12, and Appendix C, pp. 50, 51, 71, 72).

5. Transformation Pathway: [2,6-¹⁴C]Aminopyralid did not degrade in either the North Dakota water/sediment or flooded Cuckney soil systems under anaerobic conditions. No transformation products were detected. Non-extractable residues and CO₂ accounted for 1.2 % and 0.6 % of applied radioactivity, respectively, in the North Dakota system and 0.7% and 0.4% of applied radioactivity, respectively, in the Cuckney system at the end of the incubation period (Tables 11,12, and Appendix C, pp. 50, 51, 71, 72).

Data Evaluation Report on the anaerobic aquatic metabolism of aminopyralid (XDE-750)

PMRA Submission Number xxxxxxxxx

EPA MRID Number 46235730

D. SUPPLEMENTARY STUDY- RESULTS: No supplementary studies were conducted.

III. STUDY DEFICIENCIES:

No major deficiencies were identified.

IV. REVIEWER'S COMMENTS:

1. These results indicate that aminopyralid is stable under anaerobic conditions in a water-sediment system and does not biotransform.
2. Control samples were not utilized.

V. REFERENCES:

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- 2) Nelson R. 2002. Determination of the water solubility of XDE-750. FOR01015, unpublished report of Dow AgroSciences LLC.
- 3) Griffin K.A. 2001. Vapor pressure of XR-750 by Knudsen-Effusion weight loss method. AS-PR 2001-002, unpublished report of The Dow Chemical Company.
- 4) Cathie C. 2001. Determination of dissociation constant of XR-750 using UV-visible spectrophotometry, 01-822-AG, unpublished report of Dow AgroSciences LLC.
- 5) Madsen S. 2003. Determination of the n-octanol/water partition coefficient (Shake flask method) of XDE-750. FOR01009, unpublished report of Dow AgroSciences LLC.
- 6) Roulin S. P. 2001. Determination of the organic solubility of XR-750. 01003/DA, unpublished report of Dow AgroSciences LLC.
- 7) Rutherford L. A. 2002. Soil batch equilibrium adsorption/desorption of XDE-750. GH-C 5552, unpublished report of Dow AgroSciences LLC.

Chemical Name: Aminopyralid
 MRID 46235730
 PC 005100
 Guideline No: 162.3
 North Dakota Sediment-Water system

Aqueous Parent				Aqueous Unidentified radioactivity			Aqueous Extractable radioactivity			Aqueous CO2			Aqueous Non-extractable radioactivity			Aqueous Total			
Days	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	Days	% applied	Ave.	St. Dev.
0	69.0	69.5	0.7	1.8	1.1	1.0	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	0	70.8	70.6	0.3
0	70.0			0.4			N/A			N/A			N/A			0	70.4		
10	61.7	62.2	0.6	0.1	0.1	0.1	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	10	61.8	62.2	0.6
10	62.6			0.0			N/A			N/A			N/A			10	62.6		
20	65.4	65.5	0.1	0.1	0.1	0.1	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	20	65.5	65.6	0.1
20	65.6			0.0			N/A			N/A			N/A			20	65.6		
30	67.2	65.5	2.4	0.1	0.1	0.1	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	30	67.3	65.6	2.5
30	63.8			0.0			N/A			N/A			N/A			30	63.8		
90	62.6	62.5	0.1	0.0	0.0	0.0	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	90	62.6	62.5	0.1
90	62.4			0.0			N/A			N/A			N/A			90	62.4		
181	63.9	63.5	0.6	0.3	0.4	0.1	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	181	64.2	63.9	0.5
181	63.1			0.4			N/A			N/A			N/A			181	63.5		
268	62.1	62.0	0.1	0.2	0.4	0.2	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	268	62.3	62.4	0.1
268	61.9			0.5			N/A			N/A			N/A			268	62.4		
363	60.9	61.5	0.8	0.4	0.4	0.0	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	363	61.3	61.9	0.8
363	62.0			0.4			N/A			N/A			N/A			363	62.4		
																AVR	64.3		
																STDEV	2.9		

Sediment Parent				Sediment Unidentified radioactivity			Sediment Extractable radioactivity			Sediment CO2			Sediment Non-extractable radioactivity			Sediment Total			
Days	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	Days	% applied	Ave.	St. Dev.
0	26.8	27.3	0.6	0.1	0.1	0.0	26.9	27.4	0.6	N/A	#DIV/0!	#DIV/0!	0.9	0.9	0.0	0	27.8	28.3	0.6
0	27.7			0.1			27.8			N/A			0.9			0	28.7		
10	30.0	29.9	0.1	0.3	0.5	0.3	30.3	30.4	0.1	N/A	#DIV/0!	#DIV/0!	2.0	1.8	0.3	10	32.3	32.2	0.1
10	29.8			0.7			30.5			N/A			1.6			10	32.1		
20	30.3	30.4	0.1	0.0	0.1	0.1	30.3	30.5	0.2	N/A	#DIV/0!	#DIV/0!	1.6	1.7	0.1	20	31.9	32.2	0.4
20	30.5			0.1			30.6			N/A			1.8			20	32.4		
30	29.3	30.5	1.6	0.0	0.1	0.1	29.3	30.5	1.7	N/A	#DIV/0!	#DIV/0!	1.4	1.8	0.6	30	30.7	32.3	2.3
30	31.6			0.1			31.7			N/A			2.2			30	33.9		
90	31.8	33.1	1.8	0.0	0.1	0.1	31.8	33.2	1.9	N/A	#DIV/0!	#DIV/0!	2.4	2.0	0.6	90	34.2	35.1	1.3
90	34.4			0.1			34.5			N/A			1.5			90	36.0		
181	31.0	31.0	0.1	0.0	0.1	0.1	31.0	31.1	0.1	N/A	#DIV/0!	#DIV/0!	1.9	2.4	0.6	181	32.9	33.4	0.7
181	30.9			0.2			31.1			N/A			2.8			181	33.9		
268	31.6	31.6	0.1	0.4	0.6	0.2	32.0	32.1	0.1	N/A	#DIV/0!	#DIV/0!	2.0	2.0	0.1	268	34.0	34.1	0.1
268	31.5			0.7			32.2			N/A			1.9			268	34.1		
363	37.1	36.7	0.6	0.3	0.2	0.1	37.4	38.9	0.8	N/A	#DIV/0!	#DIV/0!	1.2	1.2	0.0	363	38.6	38.1	0.8
363	36.2			0.1			36.3			N/A			1.2			363	37.5		
																AVR	33.2		
																STDEV	2.8		

System Parent				System Unidentified radioactivity			System Extractable radioactivity			System CO2			System Non-extractable radioactivity			System Total			
Days	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	Days	% applied	Ave.	St. Dev.
0	95.8	96.8	1.3	1.9	1.2	1.0	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	0.9	0.9	0.0	0	96.6	96.9	0.4
0	97.7			0.5			N/A			N/A			0.9			0	96.1		
10	91.7	92.1	0.5	0.4	0.6	0.2	N/A	#DIV/0!	#DIV/0!	0.2	0.2	0.0	2.0	1.8	0.3	10	94.3	94.6	0.4
10	92.4			0.7			N/A			0.2			1.6			10	94.9		
20	95.7	95.9	0.3	0.1	0.1	0.0	N/A	#DIV/0!	#DIV/0!	0.2	0.3	0.1	1.6	1.7	0.1	20	97.6	98.0	0.5
20	96.1			0.1			N/A			0.3			1.8			20	96.3		
30	96.5	96.0	0.8	0.1	0.1	0.0	N/A	#DIV/0!	#DIV/0!	0.3	0.4	0.1	1.4	1.6	0.6	30	98.3	98.3	0.1
30	95.4			0.1			N/A			0.5			2.2			30	98.2		
90	94.4	95.6	1.7	0.0	0.1	0.1	N/A	#DIV/0!	#DIV/0!	0.7	0.6	0.1	2.4	2.0	0.6	90	97.5	98.2	1.0
90	96.8			0.1			N/A			0.5			1.5			90	96.9		
181	94.9	94.5	0.6	0.3	0.5	0.2	N/A	#DIV/0!	#DIV/0!	0.7	0.7	0.0	1.9	2.4	0.6	181	97.8	98.0	0.2
181	94.0			0.6			N/A			0.7			2.8			181	96.1		
268	93.7	93.6	0.2	0.6	0.9	0.4	N/A	#DIV/0!	#DIV/0!	0.7	0.7	0.0	2.0	2.0	0.1	268	97.0	97.1	0.1
268	93.4			1.2			N/A			0.7			1.9			268	97.2		
363	98.0	98.1	0.1	0.7	0.6	0.1	N/A	#DIV/0!	#DIV/0!	0.6	0.6	0.0	1.2	1.2	0.0	363	100.5	100.5	0.0
363	98.2			0.5			N/A			0.6			1.2			363	100.5		
																AVR	97.9		
																STDEV	1.6		

Data obtained from Table 12 and Appendix C, pp. 51 and 72 of the study report.

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Chemical Name: Aminopyralid
 MRID 46235730
 PC 005100
 Guideline No: 162-3
 Cuckney Soil-Water system

Aqueous Parent				Aqueous Unidentified radioactivity			Aqueous Extractable radioactivity			Aqueous CO2			Aqueous Non-extractable radioactivity			Aqueous Total			
Days	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	Days	% applied	Ave.	St. Dev.
0	67.2	69.2	2.8	0.0	0.0	0.0	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	0	67.2	69.2	2.8
0	71.2			0.0			N/A			N/A			N/A			0	71.2		
3	57.4	59.8	3.4	0.2	0.2	0.0	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	3	57.8	60.0	3.4
3	62.2			0.2			N/A			N/A			N/A			3	62.4		
10	61.0	61.7	0.9	0.1	0.1	0.1	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	10	61.1	61.7	0.8
10	62.3			0.0			N/A			N/A			N/A			10	62.3		
20	65.5	63.5	2.9	0.3	0.2	0.1	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	20	65.8	63.7	3.0
20	61.4			0.1			N/A			N/A			N/A			20	61.5		
30	67.6	67.0	0.8	0.0	0.2	0.2	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	30	67.6	67.2	0.6
30	66.4			0.3			N/A			N/A			N/A			30	66.7		
59	67.9	68.4	0.7	0.0	0.7	0.9	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	59	67.9	69.1	1.6
59	68.9			1.3			N/A			N/A			N/A			59	70.2		
120	72.0	71.7	0.4	0.0	0.0	0.0	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	120	72.0	71.7	0.4
120	71.4			0.0			N/A			N/A			N/A			120	71.4		
																AVR	66.1		
																STDEV	4.5		

Sediment Parent				Sediment Unidentified radioactivity			Sediment Extractable radioactivity			Sediment CO2			Sediment Non-extractable radioactivity			Sediment Total			
Days	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	Days	% applied	Ave.	St. Dev.
0	31.4	29.9	2.2	0.1	0.1	0.0	31.5	30.0	2.2	N/A	#DIV/0!	#DIV/0!	0.9	0.9	0.1	0	32.4	30.8	2.3
0	28.3			0.1			28.4			N/A			0.8			0	29.2		
3	31.4	32.3	1.2	0.1	0.1	0.1	31.5	32.3	1.1	N/A	#DIV/0!	#DIV/0!	0.7	0.7	0.0	3	32.2	33.0	1.1
3	33.1			0.0			33.1			N/A			0.7			3	33.8		
10	33.2	31.8	2.1	0.0	0.1	0.1	33.2	31.8	2.0	N/A	#DIV/0!	#DIV/0!	0.9	0.8	0.1	10	34.1	32.6	2.1
10	30.3			0.1			30.4			N/A			0.7			10	31.1		
20	29.7	29.2	0.7	0.2	0.2	0.0	29.9	29.4	0.7	N/A	#DIV/0!	#DIV/0!	0.9	1.0	0.1	20	30.8	30.4	0.6
20	28.7			0.2			28.9			N/A			1.1			20	30.0		
30	28.0	28.3	0.4	0.0	0.0	0.0	28.0	28.3	0.4	N/A	#DIV/0!	#DIV/0!	1.3	1.3	0.0	30	29.3	29.6	0.4
30	28.5			0.0			28.5			N/A			1.3			30	29.8		
59	26.3	25.5	1.1	0.1	0.1	0.1	26.4	25.6	1.2	N/A	#DIV/0!	#DIV/0!	0.8	0.7	0.2	59	27.2	26.2	1.4
59	24.7			0.0			24.7			N/A			0.5			59	25.2		
120	21.2	21.5	0.4	0.0	0.0	0.0	21.2	21.5	0.4	N/A	#DIV/0!	#DIV/0!	0.8	0.7	0.1	120	22.0	22.2	0.2
120	21.7			0.0			21.7			N/A			0.6			120	22.3		
																AVR	29.2		
																STDEV	3.8		

System Parent				System Unidentified radioactivity			System Extractable radioactivity			System CO2			System Non-extractable radioactivity			System Total			
Days	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	% applied	Ave.	St. Dev.	Days	% applied	Ave.	St. Dev.
0	98.8	99.1	0.6	0.1	0.1	0.0	N/A	#DIV/0!	#DIV/0!	N/A	#DIV/0!	#DIV/0!	0.9	0.9	0.1	0	99.6	100.0	0.6
0	99.5			0.1			N/A			N/A			0.8			0	100.4		
3	88.8	92.1	4.6	0.3	0.3	0.1	N/A	#DIV/0!	#DIV/0!	0.1	0.1	0.1	0.7	0.7	0.0	3	89.9	93.1	4.5
3	95.3			0.2			N/A			0.0			0.7			3	96.2		
10	94.2	93.4	1.1	0.1	0.1	0.0	N/A	#DIV/0!	#DIV/0!	0.2	0.2	0.0	0.9	0.8	0.1	10	95.4	94.5	1.3
10	92.6			0.1			N/A			0.2			0.7			10	93.6		
20	95.2	92.7	3.6	0.5	0.4	0.1	N/A	#DIV/0!	#DIV/0!	0.2	0.2	0.0	0.9	1.0	0.1	20	96.8	94.3	3.6
20	90.1			0.3			N/A			0.2			1.1			20	91.7		
30	95.6	95.3	0.5	0.0	0.2	0.2	N/A	#DIV/0!	#DIV/0!	0.3	0.3	0.1	1.3	1.3	0.0	30	97.2	97.0	0.4
30	94.9			0.3			N/A			0.2			1.3			30	96.7		
59	94.2	93.9	0.4	0.1	0.7	0.8	N/A	#DIV/0!	#DIV/0!	0.3	0.3	0.0	0.8	0.7	0.2	59	95.4	95.6	0.2
59	93.6			1.3			N/A			0.3			0.5			59	96.7		
120	93.2	93.2	0.1	0.0	0.0	0.0	N/A	#DIV/0!	#DIV/0!	0.4	0.4	0.0	0.8	0.7	0.1	120	94.4	94.3	0.2
120	93.1			0.0			N/A			0.4			0.6			120	94.1		
																AVR	95.5		
																STDEV	2.8		

Data obtained from Table 11 and Appendix C, pp. 50 and 71 of the study report.

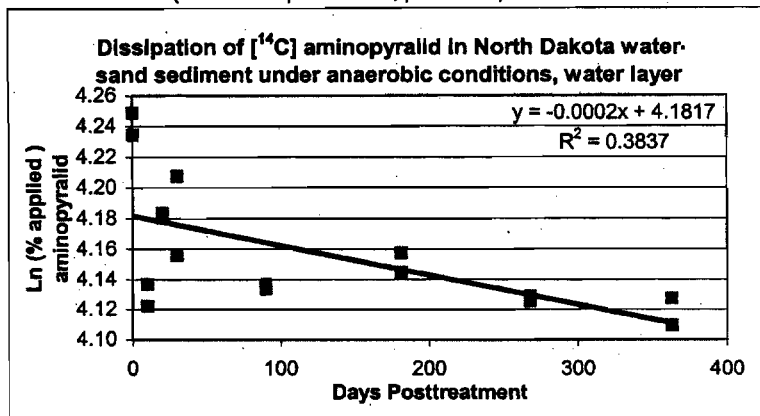
10

Chemical Name: Aminopyralid
 MRID 46235730
 PC 005100
 Guideline No: 162-3
 North Dakota water-sand sediment system

Aqueous

Days	% applied	Ln (% applied)
0	69.0	4.23
0	70.0	4.25
10	61.7	4.12
10	62.6	4.14
20	65.4	4.18
20	65.6	4.18
30	67.2	4.21
30	63.8	4.16
90	62.6	4.14
90	62.4	4.13
181	63.9	4.16
181	63.1	4.14
268	62.1	4.13
268	61.9	4.13
363	60.9	4.11
363	62.0	4.13

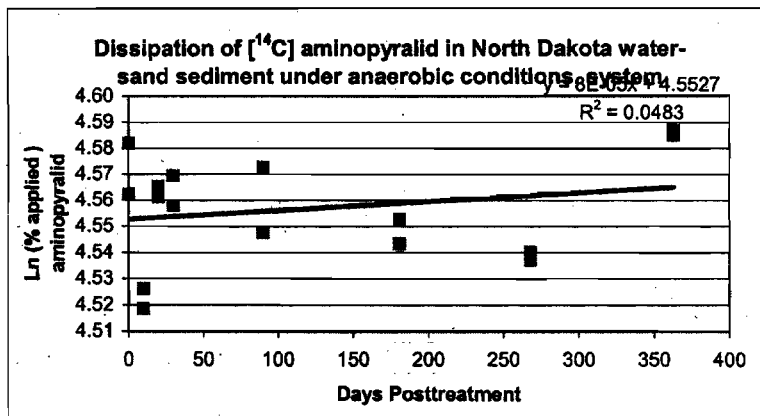
Aqueous Half life = stable
 (t-test on slope: $t = -2.95$, $p = 0.0105$)



System

Days	% applied	Ln (% applied)
0	95.8	4.56
0	97.7	4.58
10	91.7	4.52
10	92.4	4.53
20	95.7	4.56
20	96.1	4.57
30	96.5	4.57
30	95.4	4.56
90	94.4	4.55
90	96.8	4.57
181	94.9	4.55
181	94.0	4.54
268	93.7	4.54
268	93.4	4.54
363	98.0	4.58
363	98.2	4.59

System Half life = stable
 (t-test on slope: $t = 0.84$, $p = 0.4135$)



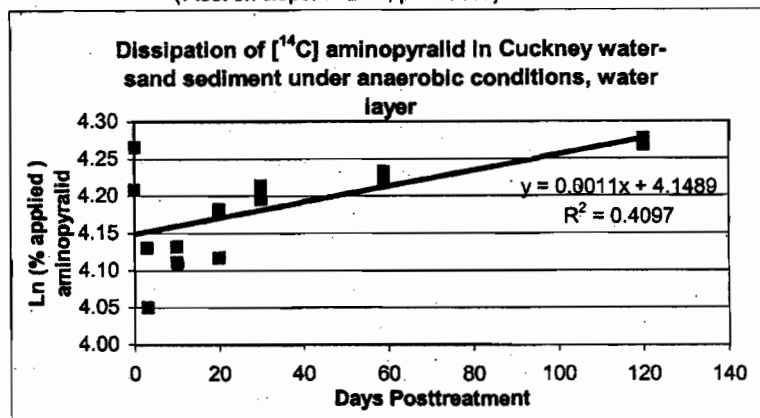
Data obtained from Table 12 and Appendix C, pp. 51 and 72 of the study report.

Chemical Name: Aminopyralid
 MRID 46235730
 PC 005100
 Guideline No: 162-3
 Cuckney Soil-Water system

Aqueous

Days	% applied	Ln (% applied)
0	67.2	4.21
0	71.2	4.27
3	57.4	4.05
3	62.2	4.13
10	61.0	4.11
10	62.3	4.13
20	65.5	4.18
20	61.4	4.12
30	67.6	4.21
30	66.4	4.20
59	67.9	4.22
59	68.9	4.23
120	72.0	4.28
120	71.4	4.27

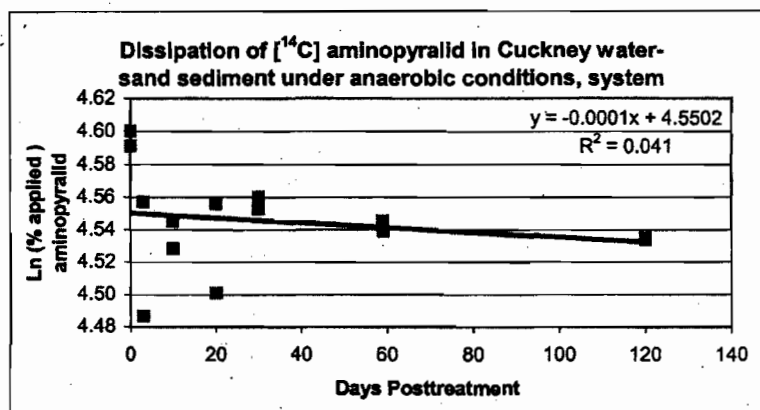
Aqueous Half life = stable
 (t-test on slope: $t = 2.89, p = 0.0137$)



System

Days	% applied	Ln (% applied)
0	98.6	4.59
0	99.5	4.60
3	88.8	4.49
3	95.3	4.56
10	94.2	4.55
10	92.6	4.53
20	95.2	4.56
20	90.1	4.50
30	95.6	4.56
30	94.9	4.55
59	94.2	4.55
59	93.6	4.54
120	93.2	4.53
120	93.1	4.53

System Half life = stable
 (t-test on slope: $t = -0.72, p = 0.4875$)



Data obtained from Table 11 and Appendix C, pp. 50 and 71 of the study report.

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EPA MRID Number 46235731

Data Requirement: PMRA DATA CODE: 8.2.3.5.4
EPA DP Barcode: D301682
OECD Data Point: IIA 7.8.1
EPA Guideline: 162-4

Test material:

Common name: Aminopyralid.
Chemical name
IUPAC: 4-Amino-3,6-dichloropyridine-2-carboxylic acid.
CAS name: 4-Amino-3,6-dichloro-2-pyridinecarboxylic acid.
CAS No.: 150114-71-9
Synonyms: XDE-750; XR-750.
SMILES string: c1c(nc(c1N)C1)C(=O)O)C1.


Primary Reviewer: Joan Gaidos, PhD.
Dynamac Corporation

Signature:
Date:

QC Reviewer: Kathleen Ferguson, PhD.
Dynamac Corporation

Signature:
Date:

Secondary Reviewer(s): Roxolana Kashuba
EPA (EFED/ERB4)

Signature: 
Date: 01/06/2005

Company Code:
Active Code:
Use Site Category:
EPA PC Code: 005100

CITATION: Yoder, R. N. and K.P. Smith. 2003. Degradation of XDE-750 in 2 European and 1 US sediment and pond water systems. Unpublished study performed by Regulatory Laboratories—Indianapolis Lab, Indianapolis, IN; sponsored and submitted by Dow AgroSciences LLC, Indianapolis, IN. Study No.: 020062. Experiment initiated June 25, 2002 and completed October 11, 2002 (p. 6). Final report issued February 21, 2003.



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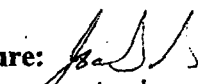
EPA MRID Number 46235731

Data Requirement: PMRA DATA CODE: 8.2.3.5.4
EPA DP Barcode: D301682
OECD Data Point: IIA 7.8.1
EPA Guideline: 162-4

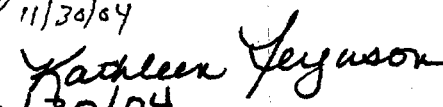
Test material:

Common name: Aminopyralid.
Chemical name
IUPAC: 4-Amino-3,6-dichloropyridine-2-carboxylic acid.
CAS name: 4-Amino-3,6-dichloro-2-pyridinecarboxylic acid.
CAS No.: 150114-71-9
Synonyms: XDE-750; XR-750.
SMILES string: c1c(nc(c1N)C1)C(=O)O)C1.

Primary Reviewer: Joan Gaidos
Dynamac Corporation

Signature: 
Date: 11/30/04

QC Reviewer: Kathleen Ferguson
Dynamac Corporation

Signature: 
Date: 11/30/04

Secondary Reviewer(s): Kevin Costello
EPA

Signature:
Date:

Company Code:
Active Code:
Use Site Category:
EPA PC Code: 005100

CITATION: Yoder, R. N. and K.P. Smith. 2003. Degradation of XDE-750 in 2 European and 1 US sediment and pond water systems. Unpublished study performed by Regulatory Laboratories—Indianapolis Lab, Indianapolis, IN; sponsored and submitted by Dow AgroSciences LLC, Indianapolis, IN. Study No.: 020062. Experiment initiated June 25, 2002 and completed October 11, 2002 (p. 6). Final report issued February 21, 2003.

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EPA MRID Number 46235731

EXECUTIVE SUMMARY:

The aerobic biotransformation of [2,6-¹⁴C]-labeled 4-amino-3,6-dichloropyridine-2-carboxylic acid (aminopyralid; XDE-750, radiochemical purity 99.3%) was studied in 3 pond water/sediment systems. One sediment/water system was collected from Haut Languedoc, France (water pH 5.9, dissolved organic carbon 2.4 ppm; sediment texture sand (USDA), pH 6.1, organic carbon 0.8%). A second sediment/water system was collected from Altogarda, Italy, (water pH 8.2, dissolved organic carbon 1.5 ppm; sediment texture silt loam (USDA), pH 7.9, organic carbon 12.0%). The third system was collected from North Dakota, USA (water pH 7.9, dissolved organic carbon 37.2 ppm; sediment texture sandy loam (USDA), pH 8.1, organic carbon 6.2%). Samples were incubated in the dark at 20 °C for up to 101 days after treatment. Aminopyralid was applied at a rate of approximately 0.04 mg a.i./L (equivalent to 120 g a.i./1-ha, 30-cm deep pond) to the aqueous phase. Approximately 6 cm of water (~25 mL) was added to 2-2.5 cm sediment (~15 g wet weight) in a centrifuge tube, resulting in a wet sediment/water ratio of approximately 3:5. The experiment was conducted in accordance with USEPA Subdivision N guidelines, and in compliance with USEPA Good Laboratory Practice Standards, 40 CFR Part 160. Each sample consisted of a 45-mL glass centrifuge tube, containing the sediment/water, attached to a second tube containing approximately 20 mL of 0.2M NaOH for the collection of ¹⁴CO₂. A vacuum pulled CO₂-scrubbed moist air through the test system to maintain aerobic conditions. Samples were analyzed at 0, 3, 7, 14, 21, 30, 62 and 101 days of incubation. The water samples were analyzed directly and the sediment samples were extracted with 90:10 acetone: 1.0 N HCl. A portion of the organic extracts were concentrated before HPLC analysis. Aminopyralid residues were assayed by LSC and analyzed by HPLC. [¹⁴C] Residues were identified by co-chromatography with unlabeled reference standards. No metabolite identification efforts were utilized.

Test conditions outlined in the study appear to have been maintained throughout the incubation. The aerobic conditions in the water layers of the French water-sand sediment system and the Italian water-silt loam sediment system were variable, but generally remained moderately reducing throughout the experiment, with redox potentials of -168.1 to 262.6 mV and oxygen contents of 0.09 to 8.58 ppm. The water layer of the US water-sediment system remained strongly to moderately reducing throughout the experiment based on redox potentials of -310.4 to 37.1 mV. Oxygen concentrations were 0.16 to 4.66 ppm, varying between 0.2 and 1.5 ppm from day zero to day 21 and increasing above 3.2 ppm for the remainder of the study. Sediment layers of all systems remained anaerobic throughout the experiment with redox potentials of -291.1 to 37.8 mV in the French sand and Italian silt loam sediments, and -438.4 to -357.9 mV in the US sandy loam sediment. The pH in all test systems remained in the environmental range throughout the experiment with pHs of 5.27 to 7.90 in the French sand, 7.06 to 8.33 in the Italian silt loam sediments, and 7.35 to 8.73 in the US sandy loam sediment.

Overall recoveries of radiolabeled material in the French water-sand sediment system ranged from 93.7 to 101.3% of the applied (average 98.9 ± 2.1%). In the Italian water-silt loam

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sediment system, recoveries ranged from 98.7 to 102.1% (average $100.4 \pm 1.3\%$). In the US water-sandy loam sediment system, recoveries ranged from 98.5 to 102.3% (average $100.5 \pm 1.2\%$). No pattern of decline in material balances were observed during the 101 day study.

Following application of [^{14}C] aminopyralid to the water layer, [^{14}C] residues associated with the sediment to varying degrees over the course of the study. In the French water-sand sediment system, the water-sediment distribution ratios were *ca.* 38:1 at time 0, 4:1 at 7 days, and 1.2:1 at 101 days (study termination). In the Italian water-silt loam sediment system, the water-sediment distribution ratios were *ca.* 42:1 at time 0, 5:1 at 7 days, and 2.3:1 at 101 days. In the US water-sandy loam sediment system, the water-sediment distribution ratios were *ca.* 37:1 at time 0, 7:1 at 7 days, and 4:1 at 101 days.

[^{14}C]Aminopyralid accounted for 95% or greater of the radioactivity extracted from any sample. Unidentified radiolabelled areas associated with column retention times of 3 minutes in the French and Italian systems, 4 minutes in the US system and 7 minutes in all systems were not detected above 1% of the applied at any sampling interval in any test system, with the exception of 3% in one replicate at 21 days in the French sediment. No major transformation products were detected in the water or the sediment of any of the test systems.

In the French water-sand sediment system, [2,6- ^{14}C]aminopyralid in the total system was 96.0% of the applied at time 0, ranged from 97.5% to 99.8% at 3-14 days, then decreased to 94.4% at 21 days, and was 90.5% at 101 days (study termination). In the water layer, [2,6- ^{14}C]aminopyralid decreased from 93.7% of the applied at time 0 to 60.9% at 21 days, and was 51.8% at 101 days (study termination). In the sediment, [^{14}C]aminopyralid increased from 2.4% of the applied at time 0 to 33.5% at 21 days, and was 38.7% at 101 days. The water-sediment distribution ratios for [^{14}C]aminopyralid were *ca.* 40:1 at time 0, 4:1 at 7 days, and 1.3:1 at 101 days. As the amount of pesticide in the sediment increased over time, extractable ^{14}C residues in the sediment increased from 2.5% at 0 DAT to 39.2% of the applied at the end of the incubation period. Similarly, non-extractable residues in the sediment increased from 0% at 0 DAT to 3.2% of the applied amount at study termination. At the end of the study less than 3% of the applied radioactivity was present as CO_2 .

In the Italian water-silt loam sediment system, [2,6- ^{14}C]aminopyralid in the total system decreased from 97.8% of the applied at time 0 to 90.9% at 14 days, 86.0% at 30 days and was 82.6% at 101 days (study termination). In the water layer, [2,6- ^{14}C]aminopyralid decreased from 96.3% of the applied at time 0 to 72.9% at 21 days, and was 67.9% at 101 days (study termination). In the sediment, [^{14}C]aminopyralid increased from 1.5% of the applied at time 0 to 12.6% at 21 days, and was 14.8% at 101 days. The water-sediment distribution ratios for [^{14}C]aminopyralid were *ca.* 67:1 at time 0, 10:1 at 7 days, and 4.6:1 at 101 days. As the amount of pesticide in the sediment increased over time, extractable ^{14}C residues in the sediment increased from 1.5% at 0 DAT to 14.8% of the applied at the end of the incubation period.

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Similarly, non-extractable residues in the sediment increased from 0.9% at 0 DAT to 14.8% of the applied amount at study termination. At the end of the study less than 2% of the applied radioactivity was present as CO₂.

In the US water-sandy loam sediment system, [2,6-¹⁴C]aminopyralid in the total system was 98.0% of the applied at time 0, ranged from 98.2% to 93.2% at 3-30 days, and was 90.3% at 101 days (study termination). In the water layer, [2,6-¹⁴C]aminopyralid decreased from 96.2% of the applied at time 0 to 82.6% at 21 days, and was 78.8% at 101 days (study termination). In the sediment, [¹⁴C]aminopyralid increased from 1.9% of the applied at time 0 to 10.8-12.0% at 21-101 days. The water-sediment distribution ratios for [¹⁴C]aminopyralid were *ca.* 52:1 at time 0, 11:1 at 7 days, and 6-8:1 at 21-101 days. As the amount of pesticide in the sediment increased over time, extractable ¹⁴C residues in the sediment increased from 1.9% at 0 DAT to 11.5% of the applied at the end of the incubation period. Similarly, non-extractable residues in the sediment increased from 0.8% at 0 DAT to 6.9% of the applied amount at study termination. At the end of the study less than 2% of the applied radioactivity was present at CO₂.

The half-lives of [¹⁴C]aminopyralid in the water and total system were calculated based on first-order linear regression analysis of log-transformed data (Excel 2000). In the French water-sandy sediment system, half-lives in the water (0-101 day data) and total system (0-101 day data) were 126.0 and 866.4 days, respectively. In the Italian water-silt loam sediment system, half-lives in the water and total system (0-101 day data) were 239.0 and 462.1 days, respectively. In the US water-sandy loam sediment system, half-lives in the water and total system (0-101 day data) were 433.2 and 990.2 days, respectively. These data were extrapolated beyond the duration of the study (study termination 101 days), and are therefore of uncertain value. However, they are useful in showing the relative stability of the parent compound in flooded sediments.

Aminopyralid degraded extremely slowly to form non-extractable residues (from 3-15% of the applied at 101 DAT) and a few minor degradates (generally <1% at any sampling interval with the exception of 3% in one replicate). Additionally, a slight amount of mineralization to CO₂ (between 1 and 2.7% at 101 DAT) was observed. From 82.6 to 90.5% of parent was present at study termination.

Results Synopsis:

Test system used: French water-sand sediment system

Half-life in water: 126.0 days ($r^2 = 0.6865$; 0-101 day data; study termination 101 days).

Half-life in system: 866.4 days ($r^2 = 0.6017$; 0-101 day data; study termination 101 days).

Test system used: Italian water-silt loam sediment system

Half-life in water: 239.0 days ($r^2 = 0.6423$; 0-101 day data; study termination 101 days).

Half-life in system: 462.1 days ($r^2 = 0.6704$; 0-101 day data; study termination 101 days).

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Test system used: US water-sandy loam sediment system

Half-life in water: 433.2 days ($r^2 = 0.5961$; 0-101 day data; study termination 101 days).

Half-life in system: 990.2 days ($r^2 = 0.6560$; 0-101 day data; study termination 101 days).

All systems:

Major transformation products: None.

Minor transformation products: CO₂.

Study Acceptability: This study is classified as supplemental because the three test systems were anaerobic (moderately to strongly reducing water and sediment phases) throughout most of the study.

I. MATERIALS AND METHODS

GUIDELINE FOLLOWED: This study was conducted in accordance with USEPA Pesticide Registration Guidelines, Subdivision N, §162-4, Canada PMRA DACO 8.2.3.5.4 - Biotransformation in Aquatic System-Aerobic Water/Sediment, and EC Directive 91/414/EEC as defined by BBA Part IV, Degradability and Persistence of Pesticides in the Water/Sediment System (pp. 1, 19). The following deviation from subdivision N guidelines were noted:

For the US water-sandy loam sediment system, the water layer was anaerobic, remaining strongly to moderately reducing throughout the experiment based on redox potentials of -310.4 to 37.1 mV, with temporal variability. Oxygen concentrations were 0.16 to 4.66 ppm, varying between 0.2 and 1.5 ppm from day zero to day 21 and increasing above 3.2 ppm for the remainder of the study.

COMPLIANCE: The study was conducted in compliance with Good Laboratory Practices standards, 40 CFR Part 160 (pp. 3, 20). Signed and dated Data Confidentiality, GLP, Quality Assurance statements and a Certification of Authenticity were provided (pp. 2-5).

A. MATERIALS:

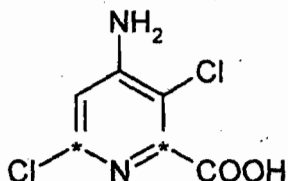
1. Test Material: [2,6-¹⁴C]-labeled aminopyralid (p. 20).
IUPAC name: 4-Amino-3,6-dichloropyridine-2-carboxylic acid.
CAS name: 4-Amino-3,6-dichloro-2-pyridinecarboxylic acid.

Chemical Structure:

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Description: Not reported.

Purity: Analytical purity: N/A
 Radiochemical purity: 99.3% (Figure 1, p. 60).
 Inventory No.: INV1590; Reference No.: F380-135a.
 Specific activity: 27.4 mCi/mmole.
 Locations of the radiolabel: 2,6-¹⁴C.

Storage conditions of test chemicals: Not reported.

Table 1: Physico-chemical properties of aminopyralid:

Parameter	Values	Comments
Molecular weight (g/mole)	207	
Molecular formula	C ₆ H ₄ Cl ₂ N ₂ O ₂	
UV adsorption:	Not reported.	
Water solubility		
pH 5	212 g/L	at 20 °C
pH 7	205 g/L	at 20 °C
pH 9	203 g/L	at 20 °C
unbuffered	2.48 g/L	at 20 °C
Vapor Pressure	7.14 x 10 ⁻¹¹ mm Hg	at 20 °C
pK _a	2.56	at 20 °C
Log K _{ow}		
Unbuffered	N/A	Definitive data for K _{ow} not available at the writing of this report
pH 5	N/A	
pH 7	N/A	
pH 9	N/A	
Stability of Compound at Room Temperature	Stable at least 5 months after receipt	Confirmed by HPLC re-analysis of ¹⁴ C test material
Organic Solvent Solubilities		
Acetone	30 g/L	at 20 °C
Ethyl Acetate	4 g/L	at 20 °C
Methanol	50 g/L	at 20 °C
1,2-dichloroethane	2 g/L	at 20 °C
Xylene	43 mg/L	at 20 °C
Heptane	less than 10 mg/L	at 20 °C

Data obtained from p. 21 of the study report.

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2. Water collection, storage and properties

Table 2a: Description of water-sediment collection and storage.

Description of Sediment Collection and Storage for French Sediment

Parameter	Description
Geographic Location	Site L Aire de Pique Nique, Monts do Somail, Haut Languedoc, France
Site Description	Small lake created by damming a stream in a forest park several decades ago. Surrounded by trees and marsh.
Latitude and Longitude	43° 32.99'N, 2° 46.56' E
Pesticide Use History	Not reported.
Collection Date	March 14, 2002
Water Collection Procedures	By immersion of 10 L container into lake
Sediment Collection Procedures	By hand at several locations over a 5.3 m area and then transferred to a 5 L container
Sampling depth (cm)	5-10 cm (not specified)
Shipping Date	March 18, 2002
Shipping Conditions	refrigerated
Storage Conditions at Facility	stored at 20 °C
Storage Length prior to use	3 months
Preparation prior to use	sediment sieved through 2 mm sieve, water filtered through glass wool

Data obtained from Appendix B, p. 79 of the study report.

Table 2b: Description of Sediment Collection and Storage for Italian Sediment

Parameter	Description
Geographic Location	Site S Alto Garda, near Turano, Provincia di Brescia, Italy
Site Description	50-30 m pond beside Toscalano River at around 590 m in the mountains of the Parco Regionale dell Alto Garda, near the village of Turano
Latitude and Longitude	45° 45.72'N 10° 35.73'E
Pesticide Use History	Not reported.
Collection Date	March 12, 2002
Water Collection Procedures	By immersion of clean 10 L container into the pond
Sediment Collection Procedures	Scooped by hand from several locations over the sample area. Then transferred into 5 L container which was topped up with retained sediment once it had settled
Sampling depth (cm)	20-30cm (not specified)
Shipping Date	March 18, 2002

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Shipping Conditions	refrigerated
Storage Conditions at Facility	stored at 20 °C
Storage Length prior to use	3 months
Preparation prior to use	sediment sieved through 2 mm sieve, water filtered through glass wool

Data obtained from Appendix B, p. 79 of the study report.

Table 2c: Description of Sediment Collection and Storage for US Sediment

Parameter	Description
Geographic Location	Steele County North Dakota, USA
Site Description	Permanent lake adjacent to grassland
Latitude and Longitude	47° 33.740' N, 97° 38.449' W
Pesticide Use History	Not reported.
Collection Date	May 16, 2002
Water Collection Procedures	Not reported.
Sediment Collection Procedures	Not reported.
Sampling depth (cm)	Not reported.
Shipping Date	May 17, 2002
Shipping Conditions	Not reported.
Storage Conditions at Facility	stored at 20 °C
Storage Length prior to use	1 month
Preparation prior to use	sediment sieved through 2 mm sieve, water filtered through glass wool

Data obtained from Appendix B, p. 80 of the study report.

Table 3: Properties of the water.

Parameter	Water			Units
	France	Italy	US	
Location				
Temperature	Not reported.	Not reported.	Not reported.	
pH	5.9	8.2	7.9	
Hardness	10 (CaCO ₃)	220 (CaCO ₃)	669 (CaCO ₃)	mg/L
Electrical Conductivity	0.11	0.36	1.71	mmhos/cm
Oxygen Conc				
Initial:	4.2	3.0	0.2	ppm
Final:	7.3	4.6	3.3	ppm
Total Suspended Solids	24	4	366	mg/L
Dissolved Organic Carbon	2.4	1.5	37.2	mg/L

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Redox Potential				
Initial:	237	19	-150	mV (Eh ₇)
Final:	269	77	-57	mV (Eh ₇)
Biomass (mg microbial C/100 g or CFU or other)	Not reported.	Not reported.	Not reported.	

Data obtained from Table 1, p. 43 of the study report.

Table 4: Properties of the sediment.

Parameter	Sediment			Units
Designation	M628	M629	M635	
Geographic Location	Haut Languedoc, France	Altogarda, Italy	North Dakota, USA	
Texture Class	Sand	Silt Loam	Sandy Loam	USDA
Sand	93	41	57	USDA%
Silt	6	56	36	USDA%
Clay	1	3	7	USDA%
pH ^a	6.1	7.9	8.1	
Organic Matter ^b	2.5	7.9	6.0	%
Organic Carbon ^c	0.8	12.0	6.2	%
Initial & Final Biomass	83.8 (initial) 207.2 (final)	166.0 (initial) 63.4 (final)	42.7 (initial) 47.2 (final)	µg/g
CEC	3.4	11.9	16.6	meq/100g
MHC	43.5	131.3	114.3	%
Bulk Density (disturbed)	1.19	0.64	0.67	g/cm ³
Redox potential:	-107.1 to -89.9 (initial)	-160.7 to -159.3 (initial)	-438.4 to -403.1 (initial)	mV

Data obtained from Table 1, p. 43, and Tables 8-10, pp. 50-52 of the study report.

^a pH in 1:1 soil: water ratio

^b Furnace method

^c LECO method

B. EXPERIMENTAL DESIGN:

1. Preliminary experiments: No preliminary experiments were conducted.

2. Experimental conditions: The current anticipated maximum application rate of aminopyralid is 120 g a.i./ha (p. 20). Assuming that the entire mass of aminopyralid is applied to a 1-ha pond, 30 cm deep, the application rate to the water phase would be 0.04 mg/L.

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Table 5: Study design.

Parameter		Description
Duration of test		Up to 101 days
Water		filtered through glass wool, stored at 20 °C
Sediment		sieved through 2 mm sieve, stored at 20 °C
Sample size water sediment		in 45-mL centrifuge tube 25 mL water (6 cm) 2-2.5 cm sediment (15 g wet weight)
Water/sediment ratio:		1.6:1 (25 mL:15 g wet weight)
Nominal application rate (mg a.i./L)		0.04 mg a.i./L (equivalent to 120 g a.i./ha)
Test concentrations (Actual)	mg a.i./kg sediment	0.069
	mg a.i./L	0.041
	total system	1.0 µg
Test concentrations (Calculated)	mg a.i./kg sediment	0.078
	mg a.i./L	0.047
	total system	1.2 µg
Control conditions (if used)		No controls were used
Number of replicates	Treatments	23 treated tubes per sediment type, 16 for duplicate samples at each sampling interval in each sediment type. Several (not specified) were used to determine the test system oxygen content and redox potential prior to dosing.
	Control	No controls were used
Test apparatus		Glass centrifuge tubes (45 mL), containing ca. 15 g wet sediment and 25 mL pond water and incubated in the dark at 20°C for 7 days to equilibrate. Vacuum pulled CO ₂ -scrubbed moist air continuously to maintain aerobicity (flow rate not reported). The test system is illustrated in Figure 3, p. 62.
Traps for CO ₂ and organic volatiles		Each tube was connected via PEEK tubing to a second centrifuge tube containing 0.2M NaOH
If no traps were used, is the system closed/open?		Traps were used.
Test material application	Identity of solvent	water
	Volume of test solution	0.1 mL (120 µL)
	Application method	Syringe to aqueous surface
	Evaporation of solvent	No
Test material sorption to walls of apparatus?		Not reported. (samples were extracted in flask)
Experimental conditions	Temperature °C	20 ± 1 °C
	Continuous darkness	Yes
Biomass (mg microbial C/100 g, CFU or other)		Sterile controls were not used.

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of controls:	
Biomass (mg microbial C/100 g, CFU or other) of treated:	Not reported.
Other details	N/A

Data obtained and reviewer-calculated from pp. 23-25, Table 2, p. 44; Figure 3, p. 62, of the study report.

3. Aerobic conditions: Kinetics samples were incubated in 45-mL glass centrifuge tubes containing 2-2.5 cm sediment and approximately 6 cm of pond water (pp. 23-24; Table 2, p. 44). Each tube was connected, via PEEK tubing, to a second centrifuge tube containing approximately 20 mL of 0.2 M NaOH. A continuous flow through each sample was maintained with a vacuum pump pulling CO₂-scrubbed moist air through the system. Flow was determined by visual inspection of the caustic traps. Air bubbles were periodically observed in each trap. However, flow rate was not reported quantitatively. Samples were weighed out 7 days before treatment to allow the water and sediment samples to equilibrate in the dark at 20 °C. Several samples were used to determine the test system oxygen content and redox potential prior to sample dosing. The oxygen content and redox potential of each samples was monitored at each sample point as well.

For the French water-sand sediment system, in the water layer at day 0, the dissolved oxygen and redox potentials in replicate samples were 4.16 and 4.80 ppm, and 178.1 and 180.0 mV, respectively; and the redox potentials in the sediment were -107.1 and -89.9 mV (Table 8, p. 50). At 101 days (study termination), the dissolved oxygen and redox potentials in the water layer were 7.27 and 7.52 ppm, and 202.6 and 204.6 mV, respectively, and in the redox potentials in the sediment were -12.2 and -4.9 mV.

For the Italian water-silt loam sediment system, in the water layer at day 0, the dissolved oxygen and redox potentials in replicate samples were 3.02 and 3.32 ppm, and 77.5 and 122.1 mV, respectively; and the redox potentials in the sediment were -160.7 and -159.3 mV (Table 9, p. 51). At 101 days (study termination), the dissolved oxygen and redox potentials in the water layer were 4.56 and 5.80 ppm, and 109.9 and 127.4 mV, respectively, and in the redox potentials in the sediment were -134.7 and -136.1 mV.

For the US water-sandy loam sediment system, in the water layer at day 0, the dissolved oxygen and redox potentials in replicate samples were 0.23 and 2.05 ppm, and -90.4 and -38.5 mV, respectively; and the redox potentials in the sediment were -438.4 and -403.1 mV (Table 10, p. 52). At 30 days, the redox potentials in the water layer were -150.6 and 34.5mV, and at 62 days they were -229.0 and 16.3 mV (see Reviewers Comments). At 101 days (study termination), the dissolved oxygen and redox potentials in the water layer were 3.30 and 3.31 ppm, and 2.2 and 37.1 mV, respectively, and in the redox potentials in the sediment were -370.4 and -361.1 mV.

4. Supplementary experiments: No supplemental studies were conducted.

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5. Sampling:

Table 6: Sampling details.

Parameter		Description
Sampling intervals		0, 3, 7, 14, 21, 30, 62, 101 DAT
Sampling methods:		Duplicate sediment samples were collected at each sampling interval. Water sampling methods were not reported.
Collection of CO ₂ and volatile organics		0.2 M NaOH trap connected to sample.
Measurement intervals	pH	Redox potential, pH and dissolved oxygen were measured at each sampling interval.
	Redox potential/Other	
	Dissolved Oxygen	
	Sterility checks	Sterile controls were not used.
Sample storage before analysis		Aqueous samples were analyzed the day of sample sacrifice. Sediments were extracted and assayed the day of sample sacrifice. Organic extracts were analyzed within one week of sample sacrifice, but stored in the freezer when not undergoing analysis.
Other observations	Contamination of the sample with back-flow of caustic trapping material	If the water sample was darkly colored and/or the aqueous pH was higher than expected for that sample type, the sample was discarded and an alternate sample was sacrificed.

Data obtained from pp. 25-26, Table 3, p. 45 of the study report.

C. ANALYTICAL METHODS: Separation of sediment and water: Samples were centrifuged at 2000 rpm for 10 minutes (p. 27). The aqueous layer was decanted into a weighed centrifuge tube. Aliquots of the aqueous layer were then analyzed by LSC and HPLC. French and Italian aqueous samples were analyzed directly, while a few drops of 1N HCl was added to ca. 1.5 mL of the US aqueous sample prior to HPLC analysis (p. 28).

Extraction/clean up/concentration methods: Approximately 20 mL of 90:10 acetone: 1.0 N HCl was added to the sediment pellet, and the sample was vortexed (p. 27). The sample was placed on a horizontal shaker at low speed for 1 hour and then centrifuged for 10 minutes at 2000 rpm. The extract was then decanted into a weighed, labeled jar and 20 mL fresh organic solvent was added to the sediment pellet, vortexing, shaking for ½ hour and centrifuging as before. The extracts were combined and the extraction process was repeated once more with another half-hour shake and 20 mL of organic solvent for a total of three extractions. Aliquots of the combined extract were assayed for ¹⁴C by LSC.

Organic extracts were concentrated and filtered prior to HPLC analysis (p. 28). Aqueous and concentrated organic samples were analyzed by reverse phase HPLC.

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Non-extractable residue determination: The extracted sediment pellet was allowed to air dry in a hood for at least one week prior to combustion analysis (p. 27). Extracted, air-dried sediment samples were combusted to determine the amount of non-extractable residues present (p. 28).

Organic matter from non-extractable 101 day Italian silt loam sediment samples were fractionated by taking sub-samples (about 1 g air-dry weight) of previously extracted, air-dried sediment, which were further extracted with 10 mL of 0.5M NaOH on a mechanical shaker at room temperature for 24 hours (p. 29). The sample was centrifuged at 3000 rpm for 15 minutes and the supernatant transferred to a centrifuge tube. The sample was briefly mixed with another 10-mL aliquot of 0.5M NaOH and centrifuged as above, and the supernatant was combined with the original extract. The sediment pellet was then rinsed with 5 mL of deionized water, centrifuged as above, and the supernatant was combined with the original extract.

The above caustic solution was acidified to pH 2 and allowed to stand at room temperature for 24 hours. After this period, the sample was centrifuged at 3000 rpm for 15 minutes. The supernatant was transferred to a centrifuge tube. Aliquots of the acidic solution (fulvic acid) were assayed by LSC. The precipitate (humic acid) was re-dissolved in 5 mL of 0.5M NaOH. Aliquots of the humic acid fraction were assayed by LSC.

Volatile residue determination: At each sampling point, the tube containing the caustic trapping solution was removed from the experimental apparatus and closed with a Teflon-lined cap (pp. 26-27). Aliquots of each trap were assayed by LSC. The tube containing the sediment and water treated with test material was also removed from the sample apparatus and capped with a Teflon-lined lid.

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

Derivatization method, if used: A derivatization method was not employed.

Identification and quantification of parent compounds: The water layer and sediment extracts were analyzed using reverse phase HPLC under the following conditions (p. 30; Table 5, p. 47): Intersil ODS column (5 μm), gradient mobile phase consisting of (A) water and 0.5% TFA, (B) acetonitrile and 0.5% TFA, isocratic 90:10 (A:B), flow rate 1 mL/min., 1-minute fractions collected for 20 minutes. Aminopyralid was identified by co-chromatography with unlabeled reference standard (Rt, 11-12 minutes).

Identification and quantification of transformation products: No transformation products were identified (p. 30).

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Detection limits (LOD, LOQ) for the parent compound and transformation products:

Table 7: Limits of Detection and Quantitation as Percent of Applied Radioactivity.

Sub-sample Identification	French Sediment		Italian Sediment		US Sediment	
	LOD	LOQ	LOD	LOD	LOQ	LOQ
Caustic Trap	0.07	0.26	0.07	0.26	0.07	0.26
Aqueous Layer	0.09	0.37	0.10	0.40	0.10	0.38
Organic Extracts	0.20	0.79	0.20	0.81	0.20	0.81
NER (Combustions)	0.08	0.31	0.04	0.15	0.03	0.14
Aqueous Layer—HPLC	0.09	0.37	0.10	0.40	0.10	0.38
Organic Extract—HPLC	0.05	0.20	0.05	0.20	0.05	0.20

HPLC LOD/LOQ Aqueous same as aqueous layer

Data obtained from Table 7, p. 49 of the study report.

II. RESULTS AND DISCUSSION:

A. TEST CONDITIONS:

Sample temperatures were maintained in the dark at 20°C for up to 101 days after treatment (pp. 35-36). A continuous flow of CO₂-scrubbed, moist air was pulled through the sample system via a vacuum pump. With the exception of a few samples, the French and Italian aquatic layers were maintained under sufficiently aerobic conditions throughout the study period (>2 ppm oxygen). The US sediment samples started out with a lower oxygen content (<2 ppm), but the amount of oxygen fluctuated and increased to 3-4 ppm by the termination of the study.

The redox potential of the aqueous layers suggested aerobic conditions with generally positive or small negative values. The redox potential of the sediment suggested mildly anaerobic to anaerobic conditions, with values in the negative range.

Test conditions outlined in the study appear to have been maintained throughout the incubation. The aerobic conditions in the water layers of the French water-sand sediment system and the Italian water-silt loam sediment system were variable, but generally remained moderately reducing throughout the experiment, with redox potentials of -168.1 to 262.6 mV and oxygen saturations of 0.09 to 8.58 ppm (Tables 8-9, pp. 50-51). The water layer of the US water-sediment system remained strongly to moderately reducing throughout the experiment with redox potentials of -310.4 to 37.1 mV and oxygen concentrations of 0.16 to 4.66 ppm. Sediment layers of all systems remained anaerobic throughout the experiment with redox potentials of -291.1 to 37.8 mV in the French sand and Italian silt loam sediments, and -438.4 to -357.9 mV in the US sandy loam sediment. The pH in all test systems ranged from 5.27 to 8.73.

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Mircobial biomass in the sediment at study initiation and termination in the French water-sand sediment system was 83.8 and 207.2 $\mu\text{g/g}$, respectively; 166.0 and 63.4 $\mu\text{g/g}$, respectively in the Italian water-silt loam sediment system; and 42.7 and 47.2 $\mu\text{g/g}$, respectively in the US water-sandy loam sediment system (Table 1, p. 43).

Final biomass values were larger than initial values in the French sediment system, were lower in the Italian system and about the same in the US system. There is no indication that the smaller final biomass activity contributed to a slower rate of degradation of aminopyralid in the Italian system.

B. MATERIAL BALANCE: Overall recoveries of radiolabeled material in the French water-sand sediment system ranged from 93.7 to 101.3% of the applied, (average $98.9 \pm 2.1\%$; Table 11, p. 53). In the Italian water-silt loam sediment system recoveries ranged from 98.7 to 102.1% (average $100.4 \pm 1.3\%$; Table 12, p. 54). In the US water-sandy loam sediment system recoveries ranged from 98.5 to 102.3% (average $100.5 \pm 1.2\%$; Table 13, p. 55). No pattern of decline in material balances was observed during the 101 day study.

Following application of [^{14}C] aminopyralid to the water layer, [^{14}C] residues appeared to associate with the sediment to varying degrees over the course of the study. In the French water-sand sediment system, the water-sediment distribution ratios were *ca.* 38:1 at time 0, 4:1 at 7 days, and 1.2:1 at 101 days (study termination). In the Italian water-silt loam sediment system, the water-sediment distribution ratios were *ca.* 42:1 at time 0, 5:1 at 7 days, and 2.3:1 at 101 days. In the US water-sandy loam sediment system, the water-sediment distribution ratios were *ca.* 37:1 at 0 days, 7:1 at 7 days, and 4:1 at 101 days (reviewer-calculated, Tables 11-13, pp. 53-55).

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Table 8: Biotransformation of [2,6-¹⁴C] aminopyralid, expressed as percentage of applied radioactivity (mean ± s.d., n = 2), in French water-sand sediment system under aerobic conditions.

Compound		Sampling Times (DAT)							
		0	3	7	14	21	30	62	101
Aminopyralid (XDE-750)	Water	93.7 ± 4.3	84.4 ± 1.1	78.2 ± 2.1	73.0 ± 1.6	60.9 ± 9.4	62.0 ± 8.6	52.0 ± 4.2	51.8 ± 2.8
	Sediment	2.4 ± 0.2	15.5 ± 1.3	19.3 ± 1.4	25.1 ± 2.2	33.5 ± 4.7	32.2 ± 8.0	39.9 ± 3.7	38.7 ± 2.0
	System	96.0 ± 4.1	99.8 ± 0.3	97.5 ± 0.6	98.0 ± 0.6	94.4 ± 4.7	94.2 ± 0.6	91.9 ± 0.5	90.5 ± 0.8
3 min.	Water	0.0	0.0	0.0	0.5 ± 0.1	0.0	0.0	0.0	0.0
	Sediment	0.0	0.0	0.0	0.6 ± 0.8	0.0	0.0	0.2 ± 0.3	0.0
	System	0.0	0.0	0.0	1.0 ± 0.7	0.0	0.0	0.2 ± 0.3	0.0
7 min.	Water	0.6 ± 0.1	<MQL	<MQL	0.0	0.2 ± 0.3	0.0	0.0	0.0
	Sediment	0.0	0.3 ± 0.4	0.0	0.0	2.8 ± 3.9	1.0 ± 0.4	0.8 ± 0.4	<0.6
	System	0.6 ± 0.1	<0.5	<MQL	0.0	3.0 ± 4.2	1.0 ± 0.4	0.8 ± 0.4	<0.6
Volatiles									
CO ₂	1	N/A	0.3	0.4	0.7	0.8	1.2	1.3	3.2
	2	N/A	0.1	0.5	0.9	1.1	1.9	2.0	2.1
	Mean	N/A	0.2	0.5	0.8	1.0	1.6	1.6	2.7
Total Extractable Sediment Residues									
90:10 Acetone: 1.0 M HCl	1	2.6	16.4	18.3	24.6	42.7	28.3	44.1	37.6
	2	2.4	15.1	21.3	26.6	30.8	38.5	37.8	40.7
	Mean	2.5	15.8	19.8	25.6	36.8	33.4	41.0	39.2
Total Non-extractable Sediment Residues	1	0.0	0.3	0.1	0.4	2.8	1.2	2.3	3.4
	2	0.0	0.2	0.4	0.8	1.3	2.0	2.7	3.0
	Mean	0.0	0.3	0.3	0.6	2.1	1.6	2.5	3.2
Total % Recovery	Water	94.2 ± 4.4	84.9 ± 1.2	78.5 ± 2.2	73.4 ± 1.6	61.3 ± 8.9	62.2 ± 8.3	52.8 ± 3.3	51.8 ± 2.8
	Sediment	2.5 ± 0.1	16.0 ± 1.0	20.1 ± 2.3	26.2 ± 1.7	38.8 ± 9.5	35.0 ± 7.8	43.5 ± 4.2	42.4 ± 1.9
	System	96.7 ± 4.2	101.0 ± 0.1	99.0 ± 0.1	100.4 ± 0.2	101.1 ± 0.4	98.8 ± 0.0	97.8 ± 0.4	96.7 ± 1.6

Data obtained from Table 11, p. 53; Table 15, p. 57 of the study report.

Duplicates samples containing only one replicate above the limit of quantitation were presented as < the detected sample.

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Table 9: Biotransformation of [2,6-¹⁴C] aminopyralid, expressed as percentage of applied radioactivity (mean ± s.d., n = 2), in Italian water-silt loam sediment system under aerobic conditions.

Compound		Sampling Times (DAT)							
		0	3	7	14	21	30	62	101
Aminopyralid (XDE-750)	Water	96.3 ± 1.0	89.8 ± 0.1	83.6 ± 0.7	79.4 ± 0.4	72.9 ± 1.1	72.7 ± 2.5	70.6 ± 0.5	67.9 ± 0.6
	Sediment	1.5 ± 0.2	6.3 ± 0.3	8.7 ± 0.6	11.5 ± 0.1	12.6 ± 0.1	13.3 ± 0.1	13.0 ± 0.8	14.8 ± 0.5
	System	97.8 ± 0.8	96.1 ± 0.2	92.3 ± 1.3	90.9 ± 0.2	85.5 ± 0.9	86.0 ± 2.5	83.6 ± 0.4	82.6 ± 0.1
3 min.	Water	0.0	0.7 ± 0.0	0.6 ± 0.1	0.6 ± 0.1	<0.5	0.6 ± 0.8	0.0	0.0
	Sediment	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	System	0.0	0.7 ± 0.0	0.6 ± 0.1	0.6 ± 0.1	<0.5	0.6 ± 0.8	0.0	0.0
7 min.	Water	<0.6	0.0	0.0	0.0	<0.7	0.0	<MQL	0.0
	Sediment	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	System	<0.6	0.0	0.0	0.0	<0.7	0.0	<MQL	0.0
Volatiles									
CO ₂	1	N/A	0.0	0.2	0.5	0.7	1.2	3.0	0.6
	2	N/A	0.1	0.3	0.7	0.4	1.4	0.3	1.3
	Mean	N/A	0.1	0.3	0.6	0.6	1.3	1.7	1.0
Total Extractable Sediment Residues									
90:10 Acetone: 1.0 M HCl	1	1.3	6.1	8.3	11.4	12.5	13.2	12.4	14.4
	2	1.6	6.5	9.1	11.6	12.7	13.3	13.6	15.1
	Mean	1.5	6.3	8.7	11.5	12.6	13.2	13.0	14.8
Total Non-extractable Sediment Residues	1	0.8	5.1	7.6	10.1	11.9	11.6	14.0	15.2
	2	0.9	5.3	8.0	10.0	12.6	14.0	14.0	14.7
	Mean	0.9	5.2	7.8	10.1	12.3	12.8	14.0	15.0
Total % Recovery	Water	97.1 ± 0.3	90.4 ± 0.2	84.2 ± 0.6	79.9 ± 0.4	74.4 ± 0.4	73.3 ± 2.8	71.1 ± 0.1	68.1 ± 0.7
	Sediment	2.3 ± 0.3	11.5 ± 0.4	16.5 ± 0.8	21.6 ± 0.1	24.9 ± 0.6	26.1 ± 1.8	27.0 ± 0.8	29.7 ± 0.1
	System	99.5 ± 0.1	101.9 ± 0.3	100.9 ± 1.6	102.0 ± 0.1	99.9 ± 0.1	100.6 ± 0.8	99.8 ± 1.2	98.8 ± 0.1

Data obtained from Table 12, p. 54; Table 16, p. 58 of the study report.

Duplicates samples containing only one replicate above the limit of quantitation were presented as < the detected sample.

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Table 10: Biotransformation of [2,6-¹⁴C] aminopyralid, expressed as percentage of applied radioactivity (mean ± s.d., n = 2), in US water-sandy loam sediment system under aerobic conditions.

Compound		Sampling Times (DAT)							
		0	3	7	14	21	30	62	101
Aminopyralid (XDE-750)	Water	96.2 ± 1.3	92.8 ± 0.1	85.7 ± 0.2	84.6 ± 0.5	82.6 ± 0.0	82.4 ± 0.6	79.8 ± 0.0	78.8 ± 0.7
	Sediment	1.9 ± 0.1	5.4 ± 0.4	7.8 ± 0.1	9.6 ± 0.6	11.3 ± 0.3	10.8 ± 0.6	12.0 ± 1.3	11.5 ± 0.2
	System	98.0 ± 1.3	98.2 ± 0.2	93.5 ± 0.1	94.1 ± 1.1	93.9 ± 0.3	93.2 ± 1.1	91.8 ± 1.3	90.3 ± 0.5
4 min.	Water	0.0	<MQL	<0.5	0.8 ± 0.1	<0.4	<MQL	0.0	0.0
	Sediment	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	System	0.0	<MQL	<0.5	0.8 ± 0.1	<0.4	<MQL	0.0	0.0
7 min.	Water	0.4 ± 0.6	0.8 ± 0.1	0.9 ± 0.1	0.7 ± 0.0	0.7 ± 0.2	<0.5	0.0	0.5 ± 0.0
	Sediment	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	System	0.4 ± 0.6	0.8 ± 0.1	0.9 ± 0.1	0.7 ± 0.0	0.7 ± 0.2	<0.5	0.0	0.5 ± 0.0
Volatiles									
CO ₂	1	N/A	0.1	0.2	0.6	0.4	0.3	1.5	1.2
	2	N/A	0.1	0.3	0.5	0.5	0.7	0.9	1.1
	Mean	N/A	0.1	0.3	0.6	0.5	0.5	1.2	1.2
Total Extractable Sediment Residues									
90:10 Acetone: 1.0 M HCl	1	1.8	5.1	7.9	10.0	11.5	11.2	13.0	11.3
	2	1.9	5.6	7.7	9.1	11.1	10.4	11.1	11.6
	Mean	1.9	5.4	7.8	9.6	11.3	10.8	12.1	11.5
Total Non-extractable Sediment Residues	1	0.8	2.5	4.1	5.4	6.2	6.6	6.4	6.7
	2	0.7	2.6	4.1	5.5	6.4	7.0	7.0	7.0
	Mean	0.8	2.6	4.1	5.4	6.3	6.8	6.7	6.8
Total % Recovery	Water	97.0 ± 0.2	93.7 ± 0.1	87.3 ± 0.5	86.2 ± 0.2	83.7 ± 0.1	82.9 ± 0.5	80.6 ± 0.5	79.5 ± 0.8
	Sediment	2.6 ± 0.0	7.9 ± 0.4	11.9 ± 0.1	14.9 ± 0.7	17.6 ± 0.1	17.6 ± 0.3	18.8 ± 0.9	18.3 ± 0.4
	System	99.6 ± 0.2	101.7 ± 0.3	99.4 ± 0.4	101.6 ± 1.0	101.8 ± 0.1	101.0 ± 0.4	100.5 ± 0.8	98.9 ± 0.5

Data obtained from Table 13, p. 55; Table 17, p. 59 of the study report.

Duplicates samples containing only one replicate above the limit of quantitation were presented as < the detected sample.

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C. TRANSFORMATION OF PARENT COMPOUND:

In the French water-sand sediment system, [2,6-¹⁴C]aminopyralid in the total system was 96.0% of the applied at time 0, ranged from 97.5% to 99.8% at 3-14 days, then decreased to 94.4% at 21 days, and was 90.5% at 101 days (study termination). In the water layer, [2,6-¹⁴C]aminopyralid decreased from 93.7% of the applied at 0 days to 60.9% at 21 days, and was 51.8% at 101 days (study termination; Table 15, p. 57). In the sediment, [¹⁴C]aminopyralid increased from 2.4% of the applied at 0 days to 33.5% at 21 days, and was 38.7% at 101 days. The water-sediment distribution ratios for [¹⁴C]aminopyralid were *ca.* 40:1 at time 0, 4:1 at 7 days, and 1.3:1 at 101 days (reviewer-calculated, Table 11, p. 53; Table 15, p. 57).

In the Italian water-silt loam sediment system, [2,6-¹⁴C]aminopyralid in the total system decreased from 97.8% of the applied at time 0 to 90.9% at 14 days, 86.0% at 30 days and was 82.6% at 101 days (study termination). In the water layer, [2,6-¹⁴C]aminopyralid decreased from 96.3% of the applied at time 0 to 72.9% at 21 days, and was 67.9% at 101 days (study termination). In the sediment, [¹⁴C]aminopyralid increased from 1.5% of the applied at time 0 to 12.6% at 21 days, and was 14.8% at 101 days. The water-sediment distribution ratios for [¹⁴C]aminopyralid were *ca.* 67:1 at time 0, 10:1 at 7 days, and 4.6:1 at 101 days (reviewer-calculated, Table 12, p. 54; Table 16, p. 58).

In the US water-sandy loam sediment system, [2,6-¹⁴C]aminopyralid in the total system was 98.0% of the applied at time 0, ranged from 98.2% to 93.2% at 3-30 days, and was 90.3% at 101 days (study termination). In the water layer, [2,6-¹⁴C]aminopyralid decreased from 96.2% of the applied at time 0 to 82.6% at 21 days, and was 78.8% at 101 days (study termination). In the sediment, [¹⁴C]aminopyralid increased from 1.9% of the applied at time 0 to 10.8-12.0% at 21-101 days. The water-sediment distribution ratios for [¹⁴C]aminopyralid were *ca.* 52:1 at time 0, 11:1 at 7 days, and 6-8:1 at 21-101 days. (reviewer-calculated, Table 13, p. 55; Table 17, p. 59).

In the French system over 90% of the applied radioactivity was present in the aqueous layer at 0 DAT, dropping to about 50% of applied at 101 DAT. The Italian and US sediment samples showed a much more moderate decline in radioactivity from the aqueous layer. At 0 DAT, over 95% of the applied radioactivity was present in the aqueous layer, before declining to 70% and 80% of applied, respectively in the Italian and US sediments.

Conversely, the amount of radioactivity extracted from the sediment increased in all test systems with time. About 2% of the applied radioactivity was extracted from the French sediment at 0 DAT, increasing to about 40% at the end of the study period. About 2% of the applied radioactivity was also extracted at 0 DAT from the Italian and US sediments. At the end of the study period, the amount of extractable radioactivity increased to 15% and 11%, respectively for the Italian and US sediments.

Data Evaluation Report on the aerobic biotransformation of aminopyralid (XDE-750) in water-sediment system

PMRA Submission Number {.....}

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HALF-LIFE/DT50: First-order, log-transformed half-lives of aminopyralid were calculated in the water and total system, using all data points and a first-order linear regression analysis in Excel. First-order, non-transformed half-lives of aminopyralid were also calculated in the water and total system, using all data points and a first-order nonlinear regression analysis in Sigma Plot (Tables 15-17, pp. 57-59; Attachment 1). A t-test performed on the data indicated that the slope of the degradation curve was statistically different from 0 for all three sediments tested, indicating that aminopyralid did indeed degrade, albeit extremely slowly under the conditions of this study. Total system half-lives were greater than 450 days. Empirical DT₅₀ values should not be reported, as aminopyralid had not degraded 50% by the termination of the study.

In the French water-sandy sediment system, half-lives in the water and total system (0-101 day data) were 126.0 and 866.4 days, respectively. In the Italian water-silt loam sediment system, half-lives in the water and total system (0-101 day data) were 239.0 and 462.1 days, respectively. In the US water-sandy loam sediment system, half-lives in the water and total system (0-101 day data) were 433.2 and 990.2 days, respectively. For each system, these data are beyond the scope of the study and are therefore of limited value. However, they are useful in showing the relative stability of the parent compound in flooded sediments.

Table 11: Half-lives*

Sediment	Half-life (days)	Regression Equation	r ²
France Total System	Linear: 866.4	y = -0.0008x + 4.5812	0.6017
	Non-linear: 770.2	y = 97.6887e [^] (-0.0009t)	0.6046
France Aqueous only	Linear: 126.0	y = -0.0055x + 4.3802	0.6865
	Non-linear: 105.0	y = 82.6246e [^] (-0.0066t)	0.7157
Italy Total System	Linear: 462.1	y = -0.0015x + 4.5350	0.6704
	Non-linear: 433.2	y = 93.4956e [^] (-0.0016t)	0.6712
Italy Aqueous only	Linear: 239.0	y = -0.0029x + 4.4491	0.6423
	Non-linear: 216.6	y = 86.5465e [^] (-0.0032t)	0.6409
US Total System	Linear: 990.2	y = -0.0007x + 4.5645	0.6560
	Non-linear: 990.2	y = 96.0647e [^] (-0.0007t)	0.6475
US Aqueous only	Linear: 433.2	y = -0.0016x + 4.4907	0.5961
	Non-linear: 407.7	y = 89.5175e [^] (-0.0017t)	0.5881

Data reviewer-calculated from Tables 15-17, pp. 57-59 of the study report and Attachment 1.

*Linear half-lives determined based on linear regression analysis on log-transformed data calculated by Excel(2000). Non-linear half-lives determined based on two-parameter exponential decay analysis on non-transformed data calculated by Sigma Plot version 6.0.

TRANSFORMATION PRODUCTS:

No major transformation products were detected in the water or the sediment.

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Data Evaluation Report on the aerobic biotransformation of aminopyralid (XDE-750) in water-sediment system

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[¹⁴C]Aminopyralid accounted for 95% or greater of the radioactivity extracted from any sample. Unidentified radiolabelled areas associated with column retention times of 3 minutes in the French and Italian systems, 4 minutes in the US system, and 7 minutes in all systems were not detected above 1% of the applied at any sampling interval in any test system, with the exception of 3% in one replicate at 21 days in the French sediment. (Tables 15-17, pp. 57-59).

NON-EXTRACTABLE AND EXTRACTABLE RESIDUES:

In the French water-sand sediment system, as the amount of pesticide in the sediment increased over time, extractable ¹⁴C residues in the sediment increased from about 2.5% at 0 DAT to 39.2% of the applied at the end of the incubation period (Table 11, p. 53). Similarly, non-extractable residues in the sediment increased from 0% at 0 DAT to 3.2% of the applied amount at study termination.

In the Italian water-silt loam sediment system, as the amount of pesticide in the sediment increased over time, extractable ¹⁴C residues in the sediment increased from 1.5% at 0 DAT to 14.8% of the applied at the end of the incubation period (Table 12, p. 54). Similarly, non-extractable residues in the sediment increased from 0.9% at 0 DAT to 14.8% of the applied amount at study termination.

In the US water-sandy loam sediment system, as the amount of pesticide in the sediment increased over time, extractable ¹⁴C residues in the sediment increased from 1.9% at 0 DAT to 11.5% of the applied at the end of the incubation period (Table 13, p. 55). Similarly, non-extractable residues in the sediment increased from 0.8% at 0 DAT to 6.9% of the applied amount at study termination.

Organic matter from non-extractable residues in the Italian silt loam sediment at 101 days consisted of fulvic acid fractions of 11.4-14.6% of the applied, humic fractions of 0.4-0.6%, and humin fractions of 0.0-2.8% (Table 4, p. 46).

VOLATILIZATION:

The amount of inorganic volatiles (composed of CO₂) was low, between 0.1 and 3% of applied radioactivity was recovered from any sample (Tables 11-13, pp. 53-55). Approximately 3%, 1% and 1% of applied reactivity were recovered from the French, Italian, and US caustic traps, respectively, at 101 DAT.

TRANSFORMATION PATHWAY:

Aminopyralid degraded extremely slowly to form non-extractable residue (from 3-15% of applied radiocarbon at 101 DAT) and a few minor degradates (generally <1% at any sampling interval with the exception of 3% in one replicate). Additionally, a slight amount of mineralization to CO₂ (between 1 and 2.7% at 101 DAT) was observed (Figure 15, p. 74). From 82.6 to 90.5% of the applied radioactivity was present as parent at study termination.

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Data Evaluation Report on the aerobic biotransformation of aminopyralid (XDE-750) in water-sediment system

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EPA MRID Number 46235731

Table 12: Chemical names for identified transformation products of aminopyralid in aerobic aquatic water-sediment.

Applicant's code	CAS Number	Chemical Name(s)	Chemical formula	Molecular weight	SMILES string
		None.			

D. SUPPLEMENTARY STUDY- RESULTS: No supplementary studies were conducted.

III. STUDY DEFICIENCIES:

According to N. Wolfe, et al., redox potentials in the range of +400 to +800 mV are considered strongly oxidizing, +200 to +400 mV moderately oxidizing, -50 to +200 mV moderately reducing, -200 to -50 mV reducing, and -400 to -200 mV strongly reducing.

In the US system, in the water layer at time 0, the dissolved oxygen and redox potentials in replicate samples were 0.23 and 2.05 ppm and -90.4 and -38.5 mV, respectively; and the redox potentials in the sediment were -438.4 and -403.1 mV (Table 10, p. 52). At 30 days, the redox potentials in the water layer were -150.6 and 34.5 mV, and at 62 days they were -229.0 and 16.3 mV. At 101 days (study termination), the dissolved oxygen and redox potentials in the water layer were 3.30 and 3.31 ppm and 2.2 and 37.1 mV, respectively, and the redox potentials in the sediment were -370.4 and -361.1 mV.

In the French system, in the water layer at time 0, the redox potentials in replicate samples were 178.1 and 180.0 mV, and the redox potentials in the sediment were -107.1 and -89.9 mV (Table 8, p. 50). At 7 days, the redox potentials in the water layer were 20.0 and 33.8 mV. At 30 days the redox potentials in the water layer were 195.5 and 167.5 mV, and the redox potentials in the sediment were -126.2 and -40.1 mV.

In the Italian system, in the water layer at time 0, redox potentials in replicate samples were 77.5 and 122.1 mV, and the redox potentials in the sediment were -160.7 and -159.3 mV (Table 9, p. 51). At 21 days, the redox potentials in the water layer were -168.1 and -97.3mV. At 101 days (study termination), redox potentials in the water layer 109.9 and 127.4 mV, and the redox potentials in the sediment were -136.1 and -134.7 mV.

Therefore, in the US water-sandy loam sediment system, the water layer was moderately to strongly reducing through much of the study and the sediment was strongly reducing through the entire study. In the French water-sand sediment and the Italian water-silt loam sediment systems, the water layers were moderately reducing through much of the study and the sediment was reducing through the entire study. For an aerobic aquatic metabolism study, USEPA Pesticide Registration Guidelines, Subdivision N, §162-4 require aerobic conditions to be present in the water layer at a minimum.

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Data Evaluation Report on the aerobic biotransformation of aminopyralid (XDE-750) in water-sediment system

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IV. REVIEWER'S COMMENTS

1. Sample sizes were small and the study was conducted in 45-mL test tubes rather than in beakers or biometer flasks.
2. The original French and Italian water and sediment samples were not surface samples and may have been anaerobic at sampling time. Aerobicity (redox potential) at time of collection and during storage was not reported. Storage length was relatively long for French and Italian samples (3 months). Insufficient information was reported regarding US sediment collection and storage.
3. The study author did not report the temperature of the system for each sampling interval, therefore it is difficult to verify if incubation conditions remained constant throughout the course of the study.

V. REFERENCES:

1. 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.
2. 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.
3. 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.

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Chemical Name: Aminopyralid
 MRID 46235731
 PC 005100
 Guideline No.: 162-4
 French Sediment

Days	Parent Aqueous			3 min Aqueous			7 min Aqueous			Total Aqueous		
	Ave.	St. Dev.		Ave.	St. Dev.		Ave.	St. Dev.		Ave.	St. Dev.	
0	90.6	93.7	4.3	0.0	0.0	0.0	0.5	0.6	0.1	91.1	94.2	4.4
0	98.7			0.0			0.6			97.3		
3	83.6	84.4	1.1	0.0	0.0	#DIV/0!	<MQL	#DIV/0!	#DIV/0!	83.6	84.4	1.1
3	85.1			<MQL			<MQL			85.1		
7	79.6	78.2	2.1	0.0	0.0	0.0	<MQL	#DIV/0!	#DIV/0!	79.6	78.2	2.1
7	76.7			0.0			<MQL			76.7		
14	74.1	73.0	1.6	0.4	0.5	0.1	0.0	0.0	0.0	74.5	73.4	1.6
14	71.8			0.5			0.0			72.3		
21	54.2	80.9	9.4	0.0	0.0	0.0	0.4	0.2	0.3	54.6	61.1	9.1
21	67.5			0.0			0.0			67.5		
30	68.1	62.0	8.8	0.0	0.0	0.0	0.0	0.0	0.0	68.1	62.0	8.8
30	55.9			0.0			0.0			55.9		
62	49.0	52.0	4.2	0.0	0.0	0.0	0.0	0.0	0.0	49.0	52.0	4.2
62	54.9			0.0			0.0			54.9		
101	53.7	51.8	2.8	0.0	0.0	0.0	0.0	0.0	0.0	53.7	51.8	2.8
101	49.8			0.0			0.0			49.8		

Days	Total Aqueous		
	Ave.	St. Dev.	
0	91.1	94.2	4.4
0	97.3		
3	83.6	84.4	1.1
3	85.1		
7	79.6	78.2	2.1
7	76.7		
14	74.5	73.4	1.6
14	72.3		
21	54.6	61.1	9.1
21	67.5		
30	68.1	62.0	8.8
30	55.9		
62	49.0	52.0	4.2
62	54.9		
101	53.7	51.8	2.8
101	49.8		

Data obtained from Table 11, p. 53 of the study report.

Days	Parent Sediment			3 min Sediment			7 min Sediment			Total Sediment		
	Ave.	St. Dev.		Ave.	St. Dev.		Ave.	St. Dev.		Ave.	St. Dev.	
0	2.5	2.4	0.2	<MQL	0.0	#DIV/0!	0.0	0.0	#DIV/0!	2.5	2.4	0.2
0	2.2			0.0			<MQL			2.2		
3	16.4	15.5	1.3	0.0	0.0	0.0	0.0	0.3	0.4	16.4	15.7	1.0
3	14.5			0.0			0.5			15.0		
7	18.3	19.3	1.4	0.0	0.0	0.0	0.0	0.0	#DIV/0!	18.3	19.3	1.4
7	20.3			0.0			<MQL			20.3		
14	23.5	25.1	2.2	1.1	0.6	0.8	0.0	0.0	0.0	24.6	25.6	1.4
14	26.6			0.0			0.0			26.6		
21	36.8	33.5	4.7	0.0	0.0	0.0	5.5	2.8	3.9	42.3	36.3	8.6
21	30.2			0.0			0.0			30.2		
30	26.5	32.2	8.0	<MQL	0.0	#DIV/0!	1.3	1.0	0.4	27.8	33.2	7.8
30	37.8			0.0			0.7			38.5		
62	42.5	39.9	3.7	0.4	0.2	0.3	1.1	0.8	0.4	44.0	40.9	4.4
62	37.3			0.0			0.5			37.8		
101	37.3	38.7	2.0	0.0	0.0	0.0	<MQL	0.8	#DIV/0!	37.3	39.0	2.4
101	40.1			0.0			0.6			40.7		

Days	Total Extractable		
	Ave.	St. Dev.	
0	2.6	2.5	0.1
0	2.4		
3	16.4	15.8	0.9
3	15.1		
7	18.3	19.8	2.1
7	21.3		
14	24.6	25.6	1.4
14	26.6		
21	42.7	36.8	8.4
21	30.8		
30	28.3	33.4	7.2
30	38.5		
62	44.1	41.0	4.5
62	37.8		
101	37.8	39.2	2.2
101	40.7		

Days	Total Unextractable		
	Ave.	St. Dev.	
0	0.0	0.0	0.0
0	0.0		
3	0.3	0.3	0.1
3	0.2		
7	0.1	0.3	0.2
7	0.4		
14	0.4	0.6	0.3
14	0.8		
21	2.8	2.1	1.1
21	1.3		
30	1.2	1.6	0.6
30	2.0		
62	2.3	2.5	0.3
62	2.7		
101	3.4	3.2	0.3
101	3.0		

Days	Total Sediment		
	Ave.	St. Dev.	
0	2.6	2.5	0.1
0	2.4		
3	16.7	16.0	1.0
3	15.3		
7	18.4	20.1	2.3
7	21.7		
14	25.0	26.2	1.7
14	27.4		
21	45.5	38.8	9.5
21	32.1		
30	29.5	35.0	7.8
30	40.5		
62	48.4	43.5	4.2
62	40.5		
101	41.0	42.4	1.9
101	43.7		

Data obtained from Table 11, p. 53 of the study report.

Days	Parent System			3 min System			7 min System			Total System		
	Ave.	St. Dev.		Ave.	St. Dev.		Ave.	St. Dev.		Ave.	St. Dev.	
0	93.1	96.0	4.1	<MQL	0.0	#DIV/0!	0.5	0.6	0.1	93.6	96.6	4.2
0	98.9			0.0			0.6			99.5		
3	100.0	99.8	0.3	0.0	0.0	#DIV/0!	<MQL	0.5	#DIV/0!	100.0	100.1	0.1
3	99.6			<MQL			0.5			100.1		
7	97.9	97.5	0.6	0.0	0.0	0.0	<MQL	#DIV/0!	#DIV/0!	97.9	97.5	0.6
7	97.0			0.0			<MQL			97.0		
14	97.6	98.0	0.6	1.5	1.0	0.7	0.0	0.0	0.0	99.1	99.0	0.1
14	98.4			0.5			0.0			98.9		
21	91.0	94.4	4.7	0.0	0.0	0.0	5.9	3.0	4.2	98.9	97.3	0.6
21	97.7			0.0			0.0			97.7		
30	94.6	94.2	0.6	<MQL	0.0	#DIV/0!	1.3	1.0	0.4	95.9	95.2	1.1
30	93.7			0.0			0.7			94.4		
62	91.5	91.8	0.5	0.4	0.2	0.3	1.1	0.8	0.4	93.0	92.9	0.2
62	92.2			0.0			0.5			92.7		
101	91.0	90.5	0.8	0.0	0.0	0.0	<MQL	0.6	#DIV/0!	91.0	90.8	0.4
101	89.9			0.0			0.6			90.5		

Days	Total System		
	Ave.	St. Dev.	
0	93.7	96.7	4.2
0	99.7		
3	100.9	101.0	0.1
3	101.1		
7	98.9	99.0	0.1
7	99.1		
14	100.2	100.4	0.2
14	100.5		
21	101.3	101.1	0.4
21	100.8		
30	98.8	98.8	0.0
30	98.8		
62	98.1	97.8	0.4
62	97.5		
101	97.8	96.7	1.6
101	95.5		

Overall	
Ave	St.Dev.
98.9	2.1

Data obtained from Table 11, p. 53 of the study report.

Data obtained from Table 15, p. 57 of the study report.

1/19/96

Chemical Name: Aminopyralid
 MRID 46235731
 PC 005100
 Guideline No.: 162-4
 Italian Sediment

Days	Parent Aqueous			3 min Aqueous			7 min Aqueous		
	Ave.	St. Dev.		Ave.	St. Dev.		Ave.	St. Dev.	
0	97.0	96.3	1.0	0.0	0.0	0.0	<MQL	0.6	#DIV/0!
0	95.6			0.0			0.6		
3	89.8	89.8	0.1	0.7	0.7	0.0	0.0	0.0	0.0
3	89.7			0.7			0.0		
7	83.1	83.6	0.7	0.5	0.6	0.1	0.0	0.0	0.0
7	84.1			0.7			0.0		
14	79.6	79.4	0.4	0.5	0.6	0.1	0.0	0.0	0.0
14	79.1			0.6			0.0		
21	73.6	72.9	1.1	0.5	0.5	#DIV/0!	<MQL	0.7	#DIV/0!
21	72.1			<MQL			0.7		
30	74.5	72.7	2.5	1.2	0.6	0.8	<MQL	0.0	#DIV/0!
30	70.9			0.0			0.0		
62	70.9	70.6	0.5	0.0	0.0	#DIV/0!	<MQL	#DIV/0!	#DIV/0!
62	70.2			<MQL			<MQL		
101	68.3	67.9	0.6	0.0	0.0	0.0	0.0	0.0	0.0
101	67.4			0.0			0.0		

Days	Total Aqueous		
	Ave.	St. Dev.	
0	97.0	96.6	0.6
0	95.2		
3	90.5	90.5	0.1
3	90.4		
7	83.6	84.2	0.8
7	84.8		
14	80.1	79.9	0.3
14	79.7		
21	74.1	73.5	0.9
21	72.8		
30	75.7	73.3	3.4
30	70.9		
62	70.9	70.6	0.5
62	70.2		
101	68.3	67.9	0.6
101	67.4		

Days	Total Aqueous		
	Ave.	St. Dev.	
0	97.3	97.1	0.3
0	96.9		
3	90.5	90.4	0.2
3	90.2		
7	83.7	84.2	0.6
7	84.6		
14	80.1	79.9	0.4
14	79.6		
21	74.6	74.4	0.4
21	74.1		
30	75.2	73.3	2.8
30	71.3		
62	71.2	71.1	0.1
62	71.0		
101	68.6	68.1	0.7
101	67.6		

Data obtained from Table 12, p. 54 of the study report.

Days	Parent Sediment			3 min Sediment			7 min Sediment		
	Ave.	St. Dev.		Ave.	St. Dev.		Ave.	St. Dev.	
0	1.3	1.5	0.2	0.0	0.0	0.0	0.0	0.0	0.0
0	1.6			0.0			0.0		
3	6.1	6.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0
3	6.5			0.0			0.0		
7	8.3	8.7	0.6	0.0	0.0	0.0	0.0	0.0	0.0
7	9.1			0.0			0.0		
14	11.4	11.5	0.1	0.0	0.0	0.0	0.0	0.0	0.0
14	11.6			0.0			0.0		
21	12.5	12.6	0.1	0.0	0.0	0.0	0.0	0.0	0.0
21	12.7			0.0			0.0		
30	13.2	13.3	0.1	0.0	0.0	0.0	0.0	0.0	0.0
30	13.3			0.0			0.0		
62	12.4	13.0	0.8	0.0	0.0	0.0	0.0	0.0	0.0
62	13.6			0.0			0.0		
101	14.4	14.8	0.5	0.0	0.0	0.0	0.0	0.0	0.0
101	15.1			0.0			0.0		

Days	Total Sediment		
	Ave.	St. Dev.	
0	1.3	1.5	0.2
0	1.6		
3	6.1	6.3	0.3
3	6.5		
7	8.3	8.7	0.6
7	9.1		
14	11.4	11.5	0.1
14	11.6		
21	12.5	12.6	0.1
21	12.7		
30	13.2	13.3	0.1
30	13.3		
62	12.4	13.0	0.8
62	13.6		
101	14.4	14.8	0.5
101	15.1		

Days	Total Extractable		
	Ave.	St. Dev.	
0	1.3	1.5	0.2
0	1.6		
3	6.1	6.3	0.3
3	6.5		
7	8.3	8.7	0.6
7	9.1		
14	11.4	11.5	0.1
14	11.6		
21	12.5	12.6	0.1
21	12.7		
30	13.2	13.3	0.1
30	13.3		
62	12.4	13.0	0.8
62	13.6		
101	14.4	14.8	0.5
101	15.1		

Days	Total Unextractable		
	Ave.	St. Dev.	
0	0.8	0.9	0.1
0	0.9		
3	5.1	5.2	0.1
3	5.3		
7	7.6	7.8	0.3
7	8.0		
14	10.1	10.1	0.1
14	10.0		
21	11.9	12.3	0.5
21	12.6		
30	11.6	12.8	1.7
30	14.0		
62	14.0	14.0	0.0
62	14.0		
101	15.2	15.0	0.4
101	14.7		

Days	Total Sediment		
	Ave.	St. Dev.	
0	2.1	2.3	0.3
0	2.5		
3	11.2	11.5	0.4
3	11.8		
7	15.9	16.5	0.8
7	17.1		
14	21.5	21.6	0.1
14	21.6		
21	24.4	24.9	0.6
21	25.3		
30	24.8	26.1	1.8
30	27.3		
62	26.4	27.0	0.8
62	27.6		
101	29.8	29.7	0.1
101	29.8		

Data obtained from Table 12, p. 54 of the study report.

Days	Parent System			3 min System			7 min System		
	Ave.	St. Dev.		Ave.	St. Dev.		Ave.	St. Dev.	
0	98.3	97.8	0.8	0.0	0.0	0.0	<MQL	0.6	#DIV/0!
0	97.2			0.0			0.6		
3	95.9	96.1	0.2	0.7	0.7	0.0	0.0	0.0	0.0
3	96.2			0.7			0.0		
7	91.4	92.3	1.3	0.5	0.6	0.1	0.0	0.0	0.0
7	93.2			0.7			0.0		
14	91.0	90.9	0.2	0.5	0.6	0.1	0.0	0.0	0.0
14	90.7			0.6			0.0		
21	86.1	85.5	0.9	0.5	0.5	#DIV/0!	<MQL	0.7	#DIV/0!
21	84.8			<MQL			0.7		
30	87.7	86.0	2.5	1.2	0.6	0.8	<MQL	0.0	#DIV/0!
30	84.2			0.0			0.0		
62	83.3	83.6	0.4	0.0	0.0	#DIV/0!	<MQL	#DIV/0!	#DIV/0!
62	83.8			<MQL			<MQL		
101	82.7	82.6	0.1	0.0	0.0	0.0	0.0	0.0	0.0
101	82.5			0.0			0.0		

Days	Total System		
	Ave.	St. Dev.	
0	98.3	98.1	0.4
0	97.8		
3	96.6	96.8	0.2
3	96.9		
7	91.9	92.9	1.4
7	93.9		
14	91.5	91.4	0.1
14	91.3		
21	86.6	86.1	0.8
21	85.5		
30	88.9	86.6	3.3
30	84.2		
62	83.3	83.6	0.4
62	83.8		
101	82.7	82.6	0.1
101	82.5		

Days	Total System		
	Ave.	St. Dev.	
0	99.5	99.5	0.1
0	99.4		
3	101.7	101.9	0.3
3	102.1		
7	99.8	100.9	1.6
7	102.0		
14	102.1	102.0	0.1
14	101.9		
21	99.8	99.9	0.1
21	99.9		
30	101.2	100.6	0.8
30	100.0		
62	100.6	99.8	1.2
62	99.9		
101	98.8	98.8	0.1
101	98.7		

Overall	
Ave	St.Dev.
100.4	1.3

Data obtained from Table 12, p. 54 of the study report.

Data obtained from Table 16, p. 58 of the study report.

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Chemical Name: Aminopyralid
 MRID 48235731
 PC 006100
 Guideline No.: 162-4
 US Sediment

Days	Parent Aqueous			4 min Aqueous			7 min Aqueous		
	Ave.	St. Dev.		Ave.	St. Dev.		Ave.	St. Dev.	
0	97.1	96.2	1.3	0.0	0.0	0.0	0.0	0.4	0.6
0	95.2			0.0			0.8		
3	92.9	92.8	0.1	<MQL	#DIV/0!	#DIV/0!	0.8	0.8	0.1
3	92.7			<MQL			0.7		
7	85.5	85.7	0.2	<MQL	0.5	#DIV/0!	0.8	0.9	0.1
7	85.8			0.5			1.0		
14	84.9	84.6	0.5	0.8	0.8	0.1	0.7	0.7	0.0
14	84.2			0.7			0.7		
21	82.6	82.6	0.0	0.4	0.4	#DIV/0!	0.5	0.7	0.2
21	82.6			<MQL			0.8		
30	82.8	82.4	0.6	<MQL	#DIV/0!	#DIV/0!	<MQL	0.5	#DIV/0!
30	82.0			<MQL			0.5		
62	79.8	79.8	0.0	0.0	0.0	0.0	<MQL	0.0	#DIV/0!
62	79.8			0.0			0.0		
101	79.3	78.8	0.7	0.0	0.0	0.0	0.5	0.5	0.0
101	78.3			0.0			0.5		

Days	Total Aqueous		
	Ave.	St. Dev.	
0	97.1	96.6	0.8
0	96.0		
3	93.7	93.6	0.2
3	93.4		
7	86.3	86.8	0.7
7	87.3		
14	86.4	86.0	0.8
14	85.6		
21	83.5	83.5	0.1
21	83.4		
30	82.8	82.7	0.2
30	82.5		
62	79.8	79.8	0.0
62	79.8		
101	79.8	79.3	0.7
101	78.8		

Days	Total Aqueous		
	Ave.	St. Dev.	
0	97.1	97.0	0.2
0	96.8		
3	93.8	93.7	0.1
3	93.6		
7	86.9	87.3	0.5
7	87.8		
14	86.3	86.2	0.2
14	86.0		
21	83.6	83.7	0.1
21	83.8		
30	83.2	82.9	0.5
30	82.5		
62	80.2	80.6	0.5
62	80.9		
101	80.0	79.5	0.8
101	78.9		

Data obtained from Table 13, p. 55 of the study report.

Days	Parent Sediment			4 min Sediment			7 min Sediment		
	Ave.	St. Dev.		Ave.	St. Dev.		Ave.	St. Dev.	
0	1.8	1.9	0.1	0.0	0.0	0.0	0.0	0.0	0.0
0	1.9			0.0			0.0		
3	5.1	5.4	0.4	0.0	0.0	0.0	0.0	0.0	0.0
3	5.6			0.0			0.0		
7	7.9	7.8	0.1	0.0	0.0	0.0	0.0	0.0	0.0
7	7.7			0.0			0.0		
14	10.0	9.6	0.6	0.0	0.0	0.0	0.0	0.0	0.0
14	9.1			0.0			0.0		
21	11.5	11.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0
21	11.1			0.0			0.0		
30	11.2	10.8	0.6	0.0	0.0	0.0	0.0	0.0	0.0
30	10.4			0.0			0.0		
62	12.9	12.0	1.3	<MQL	0.0	#DIV/0!	0.0	0.0	0.0
62	11.1			0.0			0.0		
101	11.3	11.5	0.2	0.0	0.0	0.0	0.0	0.0	0.0
101	11.6			0.0			0.0		

Days	Total Sediment		
	Ave.	St. Dev.	
0	1.8	1.9	0.1
0	1.9		
3	5.1	5.4	0.4
3	5.6		
7	7.9	7.8	0.1
7	7.7		
14	10.0	9.6	0.6
14	9.1		
21	11.5	11.3	0.3
21	11.1		
30	11.2	10.8	0.6
30	10.4		
62	12.9	12.0	1.3
62	11.1		
101	11.3	11.5	0.2
101	11.6		

Days	Total Extractable		
	Ave.	St. Dev.	
0	1.8	1.9	0.1
0	1.9		
3	5.1	5.4	0.4
3	5.6		
7	7.9	7.8	0.1
7	7.7		
14	10	9.6	0.6
14	9.1		
21	11.5	11.3	0.3
21	11.1		
30	11.2	10.8	0.6
30	10.4		
62	13	12.1	1.3
62	11.1		
101	11.3	11.5	0.2
101	11.6		

Days	Total Unextractable		
	Ave.	St. Dev.	
0	0.8	0.8	0.1
0	0.7		
3	2.5	2.6	0.1
3	2.6		
7	4.1	4.1	0.0
7	4.1		
14	5.4	5.4	0.1
14	5.3		
21	6.2	6.3	0.1
21	6.4		
30	6.6	6.8	0.3
30	7.0		
62	6.4	6.7	0.4
62	7.0		
101	6.7	6.9	0.2
101	7.0		

Days	Total Sediment		
	Ave.	St. Dev.	
0	2.6	2.6	0.0
0	2.6		
3	7.6	7.9	0.4
3	8.2		
7	12.0	11.9	0.1
7	11.8		
14	15.4	14.9	0.7
14	14.4		
21	17.7	17.6	0.1
21	17.5		
30	17.8	17.6	0.3
30	17.4		
62	19.4	18.8	0.9
62	18.1		
101	18.0	18.3	0.4
101	18.6		

Data obtained from Table 13, p. 55 of the study report.

Days	Parent System			4 min System			7 min System		
	Ave.	St. Dev.		Ave.	St. Dev.		Ave.	St. Dev.	
0	98.9	98.0	1.3	0.0	0.0	0.0	0.0	0.4	0.6
0	97.1			0.0			0.8		
3	98.0	98.2	0.2	<MQL	#DIV/0!	#DIV/0!	0.8	0.8	0.1
3	98.3			<MQL			0.7		
7	93.4	93.5	0.1	<MQL	#DIV/0!	#DIV/0!	0.8	0.9	0.1
7	93.5			<MQL			1.0		
14	94.9	94.1	1.1	0.8	0.8	0.1	0.7	0.7	0.0
14	93.3			0.7			0.7		
21	94.1	93.9	0.3	0.4	0.4	#DIV/0!	0.5	0.7	0.2
21	93.7			<MQL			0.8		
30	94.0	93.2	1.1	<MQL	#DIV/0!	#DIV/0!	<MQL	0.5	#DIV/0!
30	92.4			<MQL			0.6		
62	92.7	91.8	1.3	<MQL	0.0	#DIV/0!	<MQL	0.0	#DIV/0!
62	90.9			0.0			0.0		
101	90.8	90.3	0.5	0.0	0.0	0.0	0.5	0.5	0.0
101	88.9			0.0			0.5		

Days	Total System		
	Ave.	St. Dev.	
0	98.9	98.4	0.7
0	97.9		
3	98.8	98.9	0.1
3	99.0		
7	94.2	94.4	0.2
7	94.5		
14	96.4	95.6	1.2
14	94.7		
21	95.0	94.8	0.4
21	94.5		
30	94.0	93.5	0.8
30	92.9		
62	92.7	91.8	1.3
62	90.9		
101	91.1	90.8	0.5
101	90.4		

Days	Total System		
	Ave.	St. Dev.	
0	99.7	99.6	0.2
0	99.4		
3	101.5	101.7	0.3
3	101.9		
7	99.1	99.4	0.4
7	99.7		
14	102.3	101.8	1.0
14	100.9		
21	101.7	101.8	0.1
21	101.9		
30	101.3	101.0	0.4
30	100.7		
62	101.0	100.6	0.8
62	99.9		
101	99.2	98.9	0.5
101	98.5		

Overall	
Ave.	St. Dev.
100.5	1.2

Data obtained from Table 13, p. 55 of the study report.

Data obtained from Table 17, p. 59 of the study report.

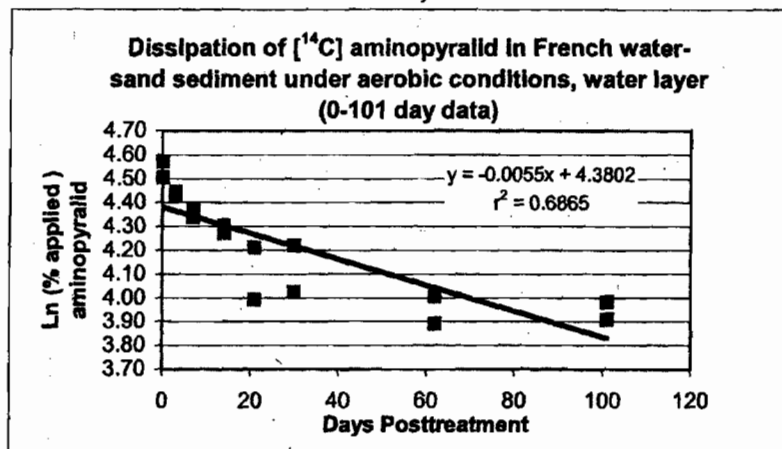
1/8

Chemical Name: Aminopyralid
 MRID 46235731
 PC 005100
 Guideline No.: 162-4

French water-sand sediment system
 Aqueous

Days	% applied	Ln (% applied)
0	90.6	4.51
0	96.7	4.57
3	83.6	4.43
3	85.1	4.44
7	79.6	4.38
7	76.7	4.34
14	74.1	4.31
14	71.8	4.27
21	54.2	3.99
21	67.5	4.21
30	68.1	4.22
30	55.9	4.02
62	49.0	3.89
62	54.9	4.01
101	53.7	3.98
101	49.8	3.91

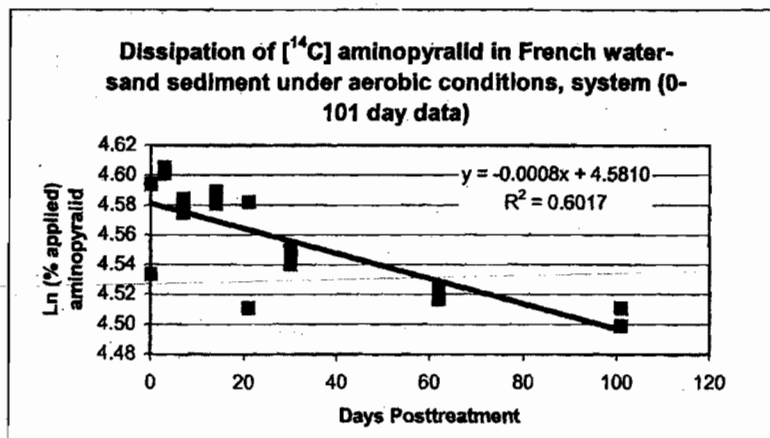
Aqueous Half life = 126.0 days
 Nonlinear 105.0 days



System

Days	% applied	Ln (% applied)
0	93.1	4.53
0	98.9	4.59
3	100.0	4.61
3	99.6	4.60
7	97.9	4.58
7	97.0	4.57
14	97.6	4.58
14	98.4	4.59
21	91.0	4.51
21	97.7	4.58
30	94.6	4.55
30	93.7	4.54
62	91.5	4.52
62	92.2	4.52
101	91.0	4.51
101	89.9	4.50

System Half life = 866.4 days
 Nonlinear 770.2 days



Data obtained from Table 15, p. 57 of the study report.

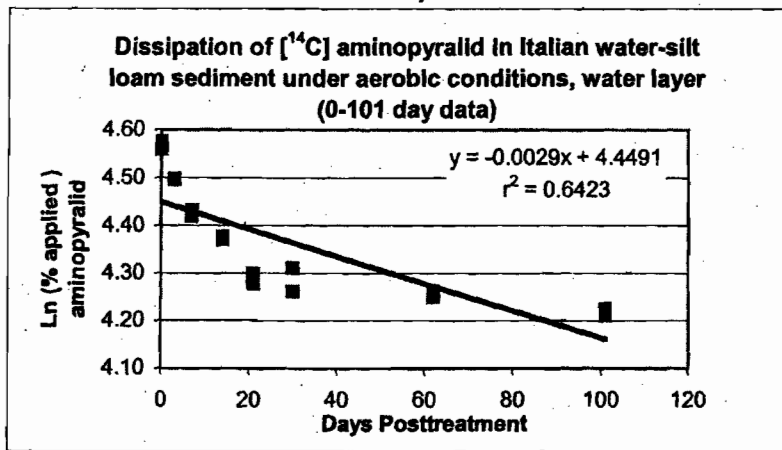
169

Chemical Name: Aminopyralid
 MRID 46235731
 PC 005100
 Guideline No.: 162-4

Italian water-silt loam sediment system
 Aqueous

Days	% applied	Ln (% applied)
0	97.0	4.57
0	95.6	4.56
3	89.8	4.50
3	89.7	4.50
7	83.1	4.42
7	84.1	4.43
14	79.6	4.38
14	79.1	4.37
21	73.6	4.30
21	72.1	4.28
30	74.5	4.31
30	70.9	4.26
62	70.9	4.26
62	70.2	4.25
101	68.3	4.22
101	67.4	4.21

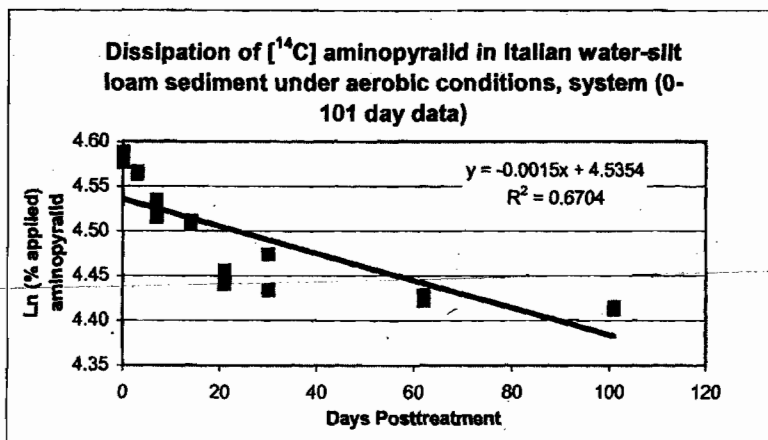
Aqueous Half life = 239.0 days
 Nonlinear 216.6 days



System

Days	% applied	Ln (% applied)
0	98.3	4.59
0	97.2	4.58
3	95.9	4.56
3	96.2	4.57
7	91.4	4.52
7	93.2	4.53
14	91.0	4.51
14	90.7	4.51
21	86.1	4.46
21	84.8	4.44
30	87.7	4.47
30	84.2	4.43
62	83.3	4.42
62	83.8	4.43
101	82.7	4.42
101	82.5	4.41

System Half life = 462.1 days
 Nonlinear 433.2 days



Data obtained from Table 16, p. 58 of the study report.

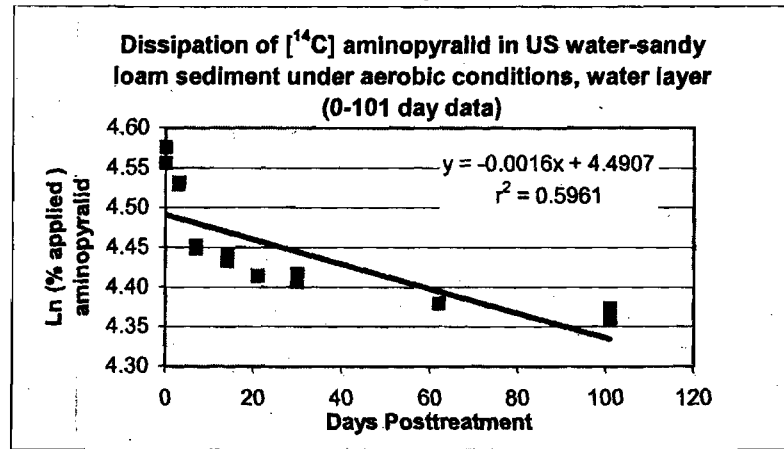
170

Chemical Name: Aminopyralid
 MRID 46235731
 PC 005100
 Guideline No.: 162-4

US water-sandy loam sediment system
 Aqueous

Days	% applied	Ln (% applied)
0	97.1	4.58
0	95.2	4.56
3	92.9	4.53
3	92.7	4.53
7	85.5	4.45
7	85.8	4.45
14	84.9	4.44
14	84.2	4.43
21	82.6	4.41
21	82.6	4.41
30	82.8	4.42
30	82.0	4.41
62	79.8	4.38
62	79.8	4.38
101	79.3	4.37
101	78.3	4.36

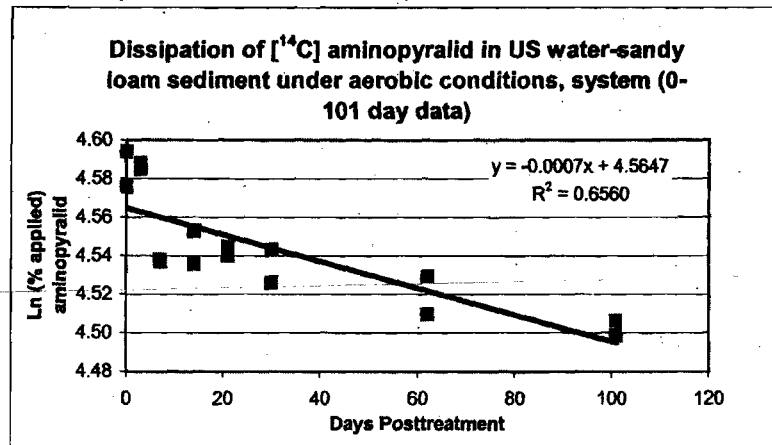
Aqueous Half life = 433.2 days
 Nonlinear 407.7 days



System

Days	% applied	Ln (% applied)
0	98.9	4.59
0	97.1	4.58
3	98.0	4.58
3	98.3	4.59
7	93.4	4.54
7	93.5	4.54
14	94.9	4.55
14	93.3	4.54
21	94.1	4.54
21	93.7	4.54
30	94.0	4.54
30	92.4	4.53
62	92.7	4.53
62	90.9	4.51
101	90.6	4.51
101	89.9	4.50

System Half life = 990.2 days
 Nonlinear 990.2 days



Data obtained from Table 16, p. 58 of the study report.

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French Aqueous
Nonlinear Regression
 Equation: Single, 2 Parameter
 $y=ae^{-bx}$

R = 0.84601281 Rsqr = 0.71573768 Adj Rsqr = 0.69543322

Standard Error of Estimate = 8.4344

	Coefficient	Std. Error	t	P
a	82.6246	3.1345	26.3601	<0.0001
b	0.0066	0.0012	5.3752	<0.0001

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	2507.6867	2507.6867	35.2503	<0.0001
Residual	14	995.9527	71.1395		
Total	15	3503.6394	233.5760		

PRESS = 1352.4535

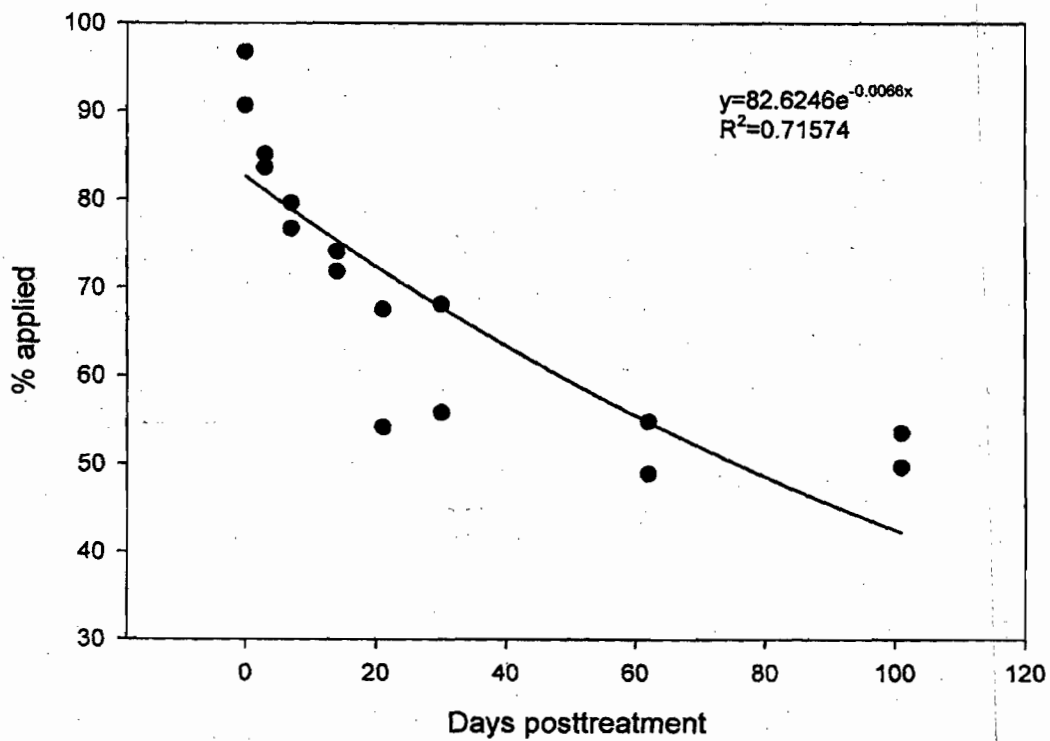
Durbin-Watson Statistic = 0.9642

Normality Test: Passed (P = 0.7808)

Constant Variance Test: Passed (P = 0.9956)

Power of performed test with alpha = 0.0500: 0.9941

French Aqueous



French Total System
Nonlinear Regression
 Equation: Single, 2 Parameter
 $y=ae^{-bx}$

R = 0.77753583 Rsqr = 0.60456196 Adj Rsqr = 0.57631639

Standard Error of Estimate = 2.2629

	Coefficient	Std. Error.	t	P
a	97.6887	0.7744	126.1416	<0.0001
b	0.0009	0.0002	4.5593	0.0004

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	109.6037	109.6037	21.4038	0.0004
Residual	14	71.6907	5.1208		
Total	15	181.2944	12.0863		

PRESS = 89.4555

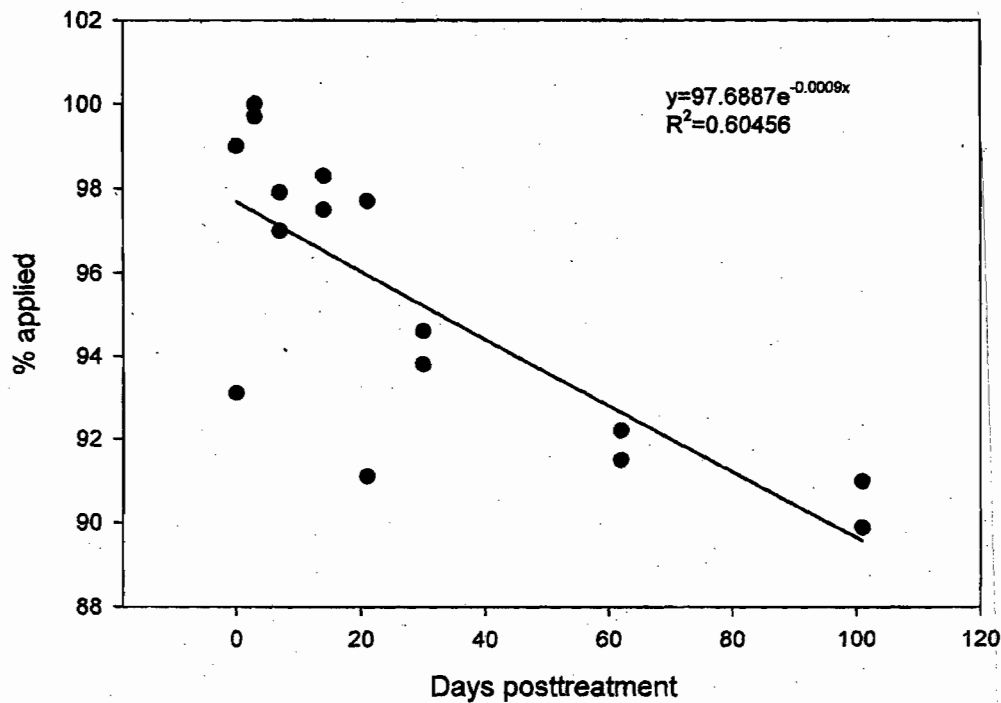
Durbin-Watson Statistic = 1.9536

Normality Test: Passed (P = 0.4844)

Constant Variance Test: Passed (P = 0.1149)

Power of performed test with alpha = 0.0500: 0.9630

French Total System



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Italian Aqueous

Nonlinear Regression

Equation: Single, 2 Parameter

$y=ae^{-bx}$

R = 0.80054976 Rsqr = 0.64087991

Adj Rsqr = 0.61522848

Standard Error of Estimate = 6.0531

	Coefficient	Std. Error	t	P
a	86.5465	2.1435	40.3764	<0.0001
b	0.0032	0.0007	4.7979	0.0003

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	915.4229	915.4229	24.9842	0.0002
Residual	14	512.9615	36.6401		
Total	15	1428.3844	95.2256		

PRESS = 680.0100

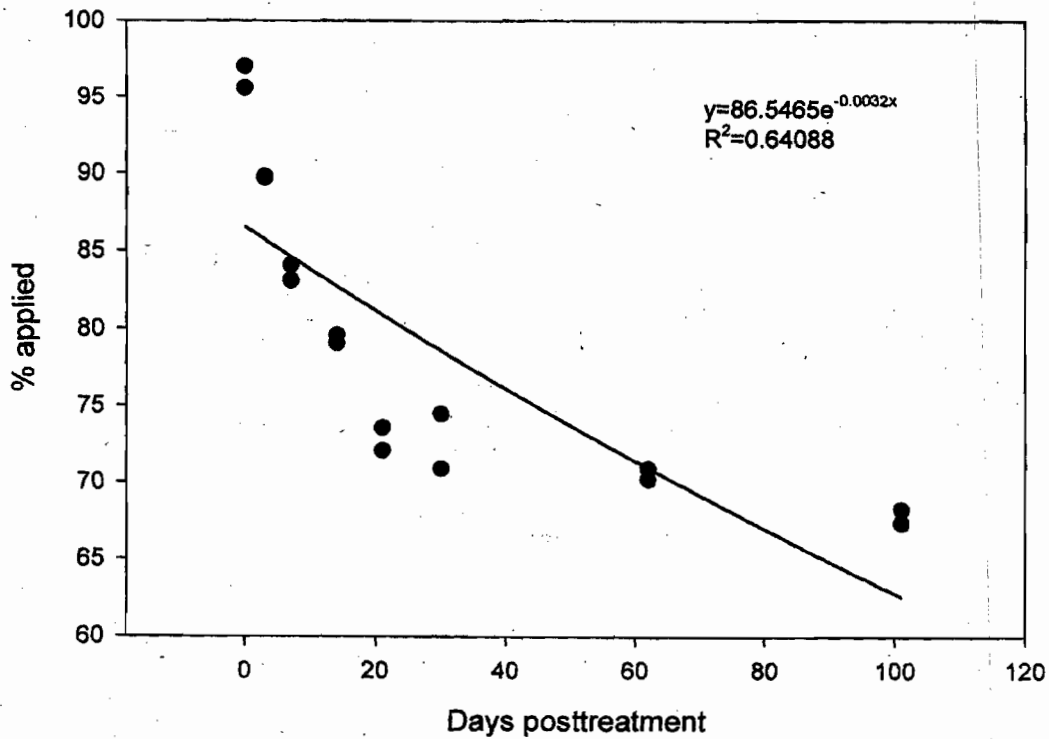
Durbin-Watson Statistic = 0.4234

Normality Test: Passed (P = 0.5915)

Constant Variance Test: Passed (P = 0.4763)

Power of performed test with alpha = 0.0500: 0.9776

Italian Aqueous



**Italian Total System
Nonlinear Regression**
Equation: Single, 2 Parameter
 $y=ae^{-bx}$

R = 0.81924736 Rsqr = 0.67116624 Adj Rsqr = 0.64767811

Standard Error of Estimate = 3.3331

	Coefficient	Std. Error	t	P
a	93.4956	1.1529	81.0944	<0.0001
b	0.0016	0.0003	5.2421	0.0001

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	317.4600	317.4600	28.5747	0.0001
Residual	14	155.5375	11.1098		
Total	15	472.9975	31.5332		

PRESS = 207.0709

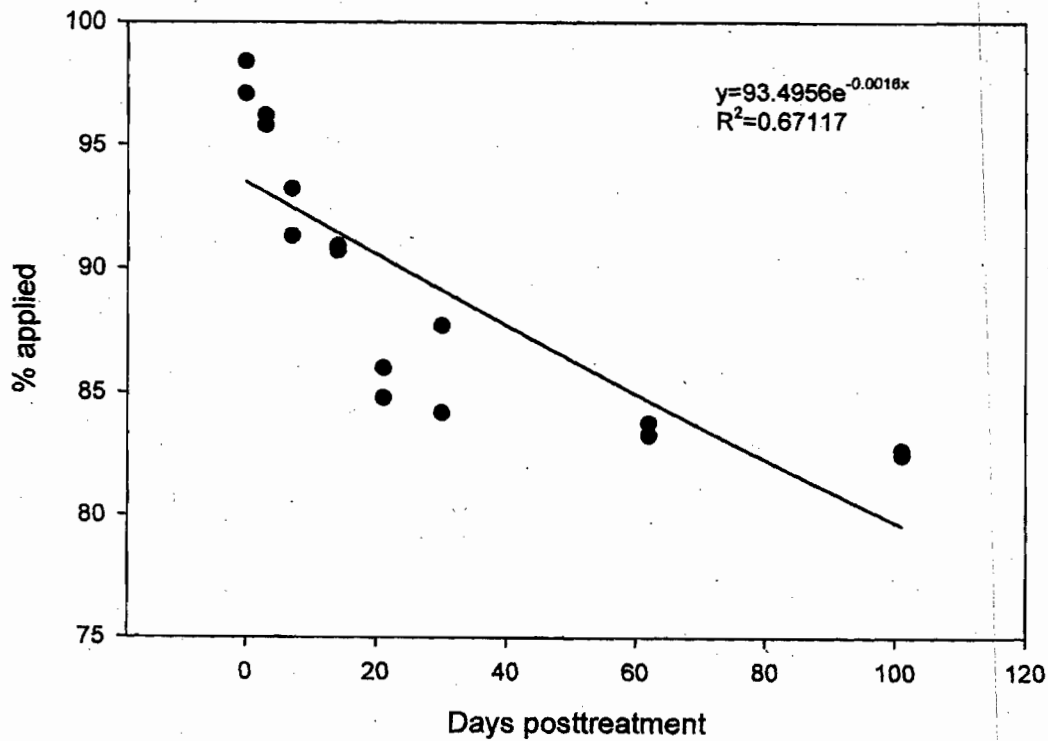
Durbin-Watson Statistic = 0.6431

Normality Test: Passed (P = 0.1870)

Constant Variance Test: Passed (P = 0.7545)

Power of performed test with alpha = 0.0500: 0.9862

Italian Total System



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US Aqueous

Nonlinear Regression

Equation: Single, 2 Parameter

$y=ae^{-bx}$

R = 0.76684711 Rsqr = 0.58805449 Adj Rsqr = 0.55862981

Standard Error of Estimate = 3.9439

	Coefficient	Std. Error	t	P
a	89.5175	1.3653	65.5673	<0.0001
b	0.0017	0.0004	4.3858	0.0006

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	310.8570	310.8570	19.9851	0.0005
Residual	14	217.7624	15.5545		
Total	15	528.6194	35.2413		

PRESS = 289.7737

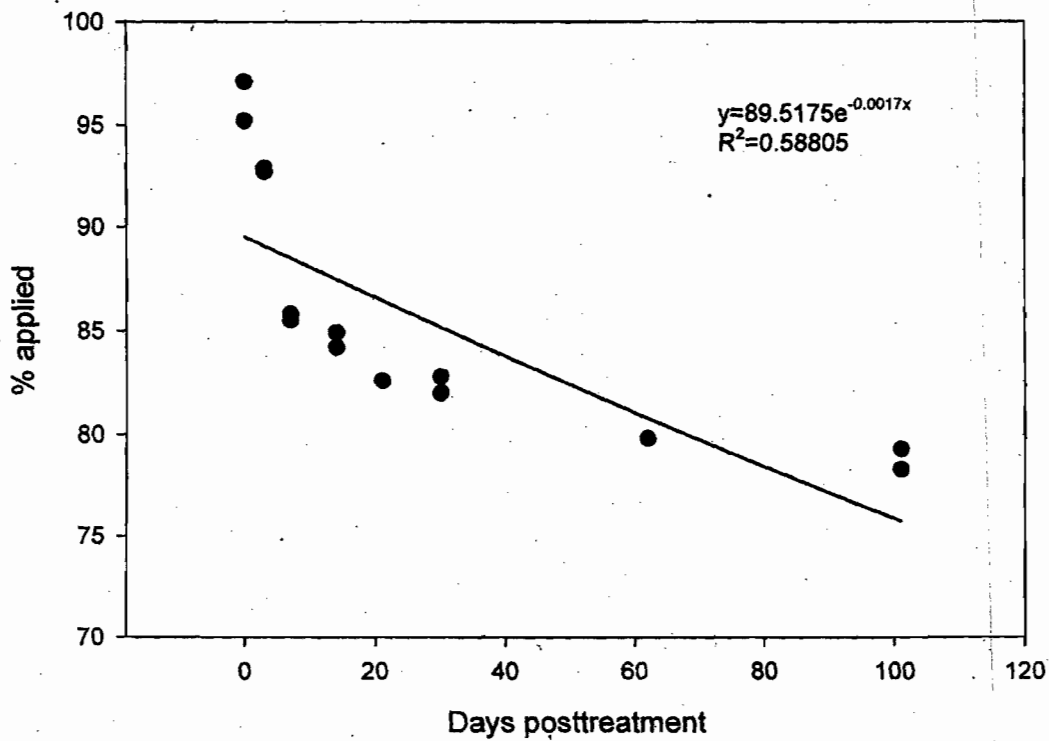
Durbin-Watson Statistic = 0.3724

Normality Test: Failed (P = 0.0189)

Constant Variance Test: Failed (P = 0.0087)

Power of performed test with alpha = 0.0500: 0.9546

US Aqueous



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US Total System
 Nonlinear Regression
 Equation: Single, 2 Parameter
 $y=ae^{-bx}$

R = 0.80465055 Rsqr = 0.64746250 Adj Rsqr = 0.62228125

Standard Error of Estimate = 1.6736

	Coefficient	Std. Error	t	P
a	96.0647	0.5714	168.1081	<0.0001
b	0.0007	0.0001	5.0178	0.0002

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	72.0168	72.0168	25.7121	0.0002
Residual	14	39.2125	2.8009		
Total	15	111.2294	7.4153		

PRESS = 49.9619

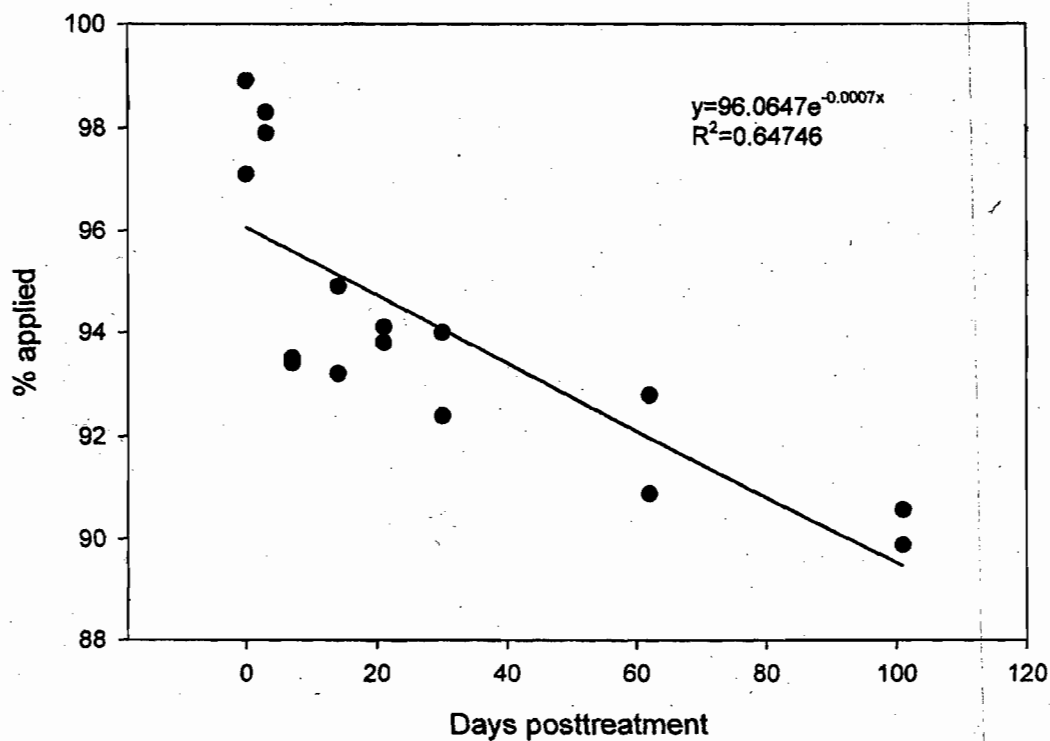
Durbin-Watson Statistic = 1.3372

Normality Test: Passed (P = 0.8270)

Constant Variance Test: Failed (P = 0.0162)

Power of performed test with alpha = 0.0500: 0.9797

US Total System



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Data Evaluation Report on the adsorption-desorption of aminopyralid (XDE-750) in soil

PMRA Submission Number {.....}

EPA MRID Number 46235732



Data Requirement: PMRA DATA CODE: 8.2.4.2
US EPA DP Barcode: D301682
OECD Data Point: IIA 7.4.1, IIA 7.4.2
US EPA Guideline: 163-1

Test material:

Common name: Aminopyralid.
Chemical name:
IUPAC: 4-Amino-3,6-dichloropyridine-2-carboxylic acid.
CAS name: 2-Pyridinecarboxylic acid, 4-amino-3,6-dichloro.
CAS No.: 150114-71-9.
Synonyms: XDE-750.
SMILES string: clc(nc(c1N)Cl)C(=O)O)Cl

Primary Reviewer: Kindra Bozicevich
Dynamac Corporation

Signature:
Date:

QC Reviewer: Joan Harlin
Dynamac Corporation

Signature:
Date:

Secondary Reviewer(s): Roxolana Kashuba
EPA (EFED/ERB4)

Signature: *Roxolana Kashuba*
Date: 01/18/2005

Company Code:
Active Code:
Use Site Category:
EPA PC Code: 005100

CITATION: Rutherford, L.A. 2002. Soil batch equilibrium adsorption/desorption of XDE-750. Unpublished study performed, sponsored and submitted by Regulatory Laboratories- Indianapolis Lab, Dow AgroSciences LLC, Indianapolis, IN. Laboratory Study ID: 010064. Experiment initiation August 27, 2001 and completion January 24, 2002 (p. 6). Final report issued December 20, 2002.



Data Evaluation Report on the adsorption-desorption of aminopyralid (XDE-750) in soil

PMRA Submission Number {.....}

EPA MRID Number 46235732



Data Requirement: PMRA DATA CODE: 8.2.4.2
US EPA DP Barcode: D301682
OECD Data Point: IIA 7.4.1, IIA 7.4.2
US EPA Guideline: 163-1

Test material:

Common name: Aminopyralid.
Chemical name:
IUPAC: 4-Amino-3,6-dichloropyridine-2-carboxylic acid.
CAS name: 2-Pyridinecarboxylic acid, 4-amino-3,6-dichloro.
CAS No.: 150114-71-9.
Synonyms: XDE-750.
SMILES string: ClC(=O)OClc1nc(c(c1N)Cl)C(=O)OCl

Primary Reviewer: Kindra Bozicevich
Dynamac Corporation

Signature: *Kindra Bozicevich*
Date: 12/02/04

QC Reviewer: Joan Harlin
Dynamac Corporation

Signature: *Joan Harlin*
Date: 12/2/04

Secondary Reviewer(s): Kevin Costello
EPA

Signature:
Date:

Company Code:
Active Code:
Use Site Category:
EPA PC Code: 005100

CITATION: Rutherford, L.A. 2002. Soil batch equilibrium adsorption/desorption of XDE-750. Unpublished study performed, sponsored and submitted by Regulatory Laboratories- Indianapolis Lab, Dow AgroSciences LLC, Indianapolis, IN. Laboratory Study ID: 010064. Experiment initiation August 27, 2001 and completion January 24, 2002 (p. 6). Final report issued December 20, 2002.

Data Evaluation Report on the adsorption-desorption of aminopyralid (XDE-750) in soil

PMRA Submission Number {.....}

EPA MRID Number 46235732

EXECUTIVE SUMMARY:

The adsorption/desorption characteristics of [2,6-¹⁴C]-labeled 4-amino-3,6-dichloropyridine-2-carboxylic acid (aminopyralid; XDE-750) were studied in eight soil types: a silt loam (M546) [pH 7.8, o.c. 1.0%] from Thessaloniki (Greece), a clay (M549) [pH 7.5, o.c. 3.2%] from Faringdon (United Kingdom), a silty clay loam (M568) [pH 7.8, o.c. 3.9%] from Manitoba (Canada), a sand (M579) [pH 6.6, o.c. 1.6%] from Bedfordshire (England), a loam (M584) [pH 6.1, o.c. 1.0%] from Charentilly (France), a clay (M599) [pH 6.9, o.c. 1.5%] from Mississippi (USA), a clay loam (M616) [pH 4.8, o.c. 3.6%, also used in the aerobic soil metabolism study] from North Dakota (USA), and a loamy sand (M617) [pH 4.5, o.c. 0.6%] from North Carolina (USA) in a batch equilibrium experiment.

The study was conducted in accordance with the OECD 106, FIFRA N-163-1, SETAC Part 1 Section 4, and OPPTS 835.1220 guidelines, and in compliance with the USEPA Good Laboratory Practice standards 40 CFR Part 160. The adsorption phase of the study was carried out by adding pesticide-free 0.01 M CaCl₂ solution and equilibrating soils at 25°C for approximately 16 hours, then equilibrating with [2,6-¹⁴C]aminopyralid at nominal test concentrations of 0.1, 0.2, 1.0, 2.0 and 10 mg a.i./kg soil in the dark at 25°C for 48 hours. The equilibrating solution used was 0.01 M CaCl₂, with a soil:solution ratio of 1:2 (w:v). The desorption phase of the study was carried out by adding approximately the amount of 0.01 M CaCl₂ removed following adsorption and equilibrating in the dark at 25°C for 2 hours. The samples were desorbed once.

Following adsorption and desorption, the supernatant solutions were separated by centrifugation and aliquots were analyzed for total radioactivity using LSC. Following desorption, all soils were extracted three times with 90:10 acetone:1.0 N HCl. The extracts were pooled and aliquots were analyzed for total radioactivity using LSC. The extracted soils were air-dried and triplicate aliquots were analyzed for total radioactivity using LSC following combustion. Aliquots of the high-dose (10.0 mg a.i./kg soil) extracts were then concentrated using a turbo evaporator and analyzed by HPLC with fraction collection. In addition, high-dose (10.0 mg a.i./kg soil) adsorption and desorption supernatants and were analyzed using HPLC.

The stability of aminopyralid during the adsorption phase averaged 99.1 ± 0.5% in the adsorption solution (10.0 mg a.i./kg soil concentration only). In the concentrated soil extracts, [2,6-¹⁴C]aminopyralid accounted for 97.1-99.6% of the radioactivity recovered. The mass balance at the end of the adsorption phase of the study was not reported. Mass balances at the end of the desorption phase in replicate samples were 98.3-101.0%, 101.6-103.8%, 96.2-100.0%, 98.4-101.6%, 95.5-99.8%, 101.0-104.1%, 94.5-98.1%, and 96.7-101.4% for the M546, M549, M568, M579, M584, M599, M616, and M617 test soils, respectively.

After 48 hours of equilibration, an average of 2.3%, 1.3%, 12.2%, 3.2%, 4.1%, 0.6%, 26.7%, and 7.2% of the applied [2,6-¹⁴C]aminopyralid was adsorbed in soils M546, M549, M568, M579, M584, M599, M616, and M617, respectively. The adsorption

Data Evaluation Report on the adsorption-desorption of aminopyralid (XDE-750) in soil

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average K_d values were 0.04, 0.03, 0.29, 0.07, 0.08, 0.03, 0.72, and 0.15 mL/g in soils M546, M549, M568, M579, M584, M599, M616, and M617, respectively. The corresponding average adsorption K_{oc} values ranged from 1.05 to 24.30 mL/g. The Freundlich adsorption K_f values ranged from 0.01 to 0.73.

At the end of the desorption phase, an average of 27.3%, 0.0%, 42.1%, 47.6%, 8.0%, 0.0%, 39.4% and 42.3% of the adsorbed amount was desorbed from soils M546, M549, M568, M579, M584, M599, M616, and M617, respectively. The desorption average K_d values were 5.98, 0.00, 3.59, 2.60, 19.14, 0.0, 3.80 and 2.18 mL/g in soils M546, M549, M568, M579, M584, M599, M616, and M617, respectively. The corresponding average desorption K_{oc} values ranged from 0.0 to 1914.5 mL/g. The desorption K_d and K_{oc} values were higher than those obtained for adsorption. The Freundlich desorption K_f values ranged from 1.24 to 3.09.

In addition, two supplementary, non-guideline experiments were conducted to study the adsorption characteristics of [2,6- 14 C]aminopyralid in M568 silty clam loam and M599 clay soils, incubated at 5 °C, and in clay loam, loam, sandy loam, and clay soils used in an aerobic soil metabolism study. The soils in both supplementary experiments were treated only at one concentration (1.0 mg a.i./kg soil). In the first experiment, two of the main study soils were incubated at 5 °C to test the effect of temperature on adsorption and desorption. In the second experiment, soils were incubated at 25 °C and desorption was not studied.

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Results Synopsis:

Soil type:	M546 Silt loam (Greece)	M549 Clay (UK)	M568 Silty clay loam (Canada)	M579 Sand (UK)	M584 Loam (France)	M599 Clay (MS)	M616 Clay loam (ND)	M617 Loamy sand (NC)
Average Amount adsorbed ^a :	2.3% +1.1%	1.3% +1.2%	12.2% +4.4%	3.2% +1.8%	4.1% +2.2%	0.6% +0.7%	26.7% +8.8%	7.2% +3.0%
Adsorption Freundlich K_f :	0.04	0.01	0.26	0.05	0.07	0.04	0.73	0.13
Average Adsorption K_d (mL/g):	0.04 +0.02	0.03 +0.02	0.29 +0.07	0.07 +0.03	0.08 +0.03	0.03 +0.01	0.72 +0.15	0.15 +0.05
Average Adsorption K_{oc} (mL/g):	4.49 +1.95	1.05 +0.73	7.39 +1.83	4.59 +2.10	7.54 +2.93	2.33 +0.58	19.95 +4.06	24.30 +8.33
Average Amount desorbed ^b :	26.2% +12.0%	0.0%	37.3% +11.6%	54.4% +30.8%	5.9% +6.1%	0.0%	34.5% +11.4%	43.6% +15.5%
Desorption Freundlich K_f :	1.97	--	3.09	1.72	1.24	--	2.88	2.12
Average Desorption K_d (mL/g):	5.98 +2.90	--	3.59 +0.43	2.60 +2.08	19.14 +4.64	--	3.80 +0.55	2.18 +1.07
Average Desorption K_{oc} (mL/g):	598.44 +289.57	--	92.16 +11.04	162.27 +130.14	1914.50 +464.44	--	105.66 +15.20	362.78 +178.19

^a Amount adsorbed to the soil calculated by subtracting the amount radioactivity remaining in solution from the amount radioactivity applied to the solution.

^b Expressed as a percentage of the adsorbed.

Study Acceptability: This study is classified supplemental for an adsorption/desorption study in soil because none of the test soils had an organic matter content of $\leq 1\%$, as required by Subdivision N guidelines.

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Data Evaluation Report on the adsorption-desorption of aminopyralid (XDE-750) in soil

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I. MATERIALS AND METHODS

GUIDELINE FOLLOWED:

This study was conducted in accordance with US Environmental Protection Agency Pesticide Assessment Guidelines, Subdivision N-Chemistry: Environmental Fate § 163-1 Leaching/Adsorption/Desorption; Society of Environmental Toxicology and Chemistry (SETAC) Part 1, Section 4 guidelines; Organization for Economic Cooperation and Development (OECD) Method 106 guidelines; the US Environmental Protection Agency Fate, Transport and Transformation Test Guidelines; and OPPTS 835.1220 (p. 19). No significant deviations from Subdivision N guidelines were noted.

COMPLIANCE:

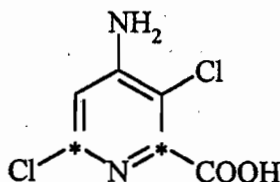
The study was conducted in compliance with the USEPA Good Laboratory Practice Standards, 40 CFR 160 (1989) and OECD GLP (1982; p. 3). Signed and dated GLP, Quality Assurance, Data Confidentiality, and Certificate of Authenticity statements were provided (pp. 2-5).

A. MATERIALS:

1. Test Material

[2,6-¹⁴C]XDE-750 (aminopyralid; p. 20; Figure 1, p. 79).

Chemical Structure:



Description:

Technical, solid (p. 20).

Purity:

Radiolabeled:

Analytical purity: Not reported.
Radiochemical purity: 99.6% (p. 20).
Inventory No.: INV 1590.
FA & PC Reference No.: 014001.
SPS Reference No.: F380-135a.
Specific activity: 27.4 mCi/mmole.
Locations of the label: Ring labeled at the C-2 and C-6 positions (Figure 1, p. 79).

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Non-radiolabeled:

Analytical purity: 99.5% (p. 21; Figure 1, p. 79).
ID No.: TSN102298.

Storage conditions of test chemicals:

Both radiolabeled and non-radiolabeled test materials were stored in a refrigerator (pp. 20-21).

Physico-chemical properties of aminopyralid (XDE-750):

Parameter	Values	Comments
Water solubility at 20°C pH 5 pH 7 pH 9 unbuffered	18.85 g cmpd/100 g H ₂ O 17.7 g cmpd/100 g H ₂ O 18.5 g cmpd/100 g H ₂ O N/A ^a	Preliminary data Preliminary data Preliminary data
Vapor Pressure at 25°C	1.94 x 10 ⁻¹⁰	
UV absorption	270 nm	
pK _a	2.56	
Log K _{ow}	N/A ^a	
Stability of Compound at room temperature	N/A ^a	

Data were obtained from p. 20 of the study report.

^a N/A = Data not currently available.

2. Soil Characteristics

Table 1a: Description of soil collection and storage for definitive study.

Description	M599	M568	M546	M584
Geographic location	Washington County, MS, USA	Whitewater Rural Municipality, Manitoba, Canada	Thessaloniki, Greece	Charentilly, Loire Valley, France
Pesticide use history at the collection site	Release, May 2000; Urea, Jun & Jul 2000	See footnote a	None	Glyphosate, May 99; Roundup BioForce, Jul & Sep 99
Collection procedures	SOP ^c	SOP ^c	SOP ^c	SOP ^c
Sampling depth (cm)	SOP ^c	SOP ^c	0-20	0-20
Storage conditions	4°C	4°C	4°C	4°C
Storage length	13 months ^d	27 months ^d	31 months ^d	19 months ^d
Soil preparation	SOP ^c	SOP ^c	SOP ^c	SOP ^c

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Table 1a (cont): Description of soil collection and storage for definitive study.

Description	M617	M549	M579	M616
Geographic location	Wake County, NC, USA	Faringdon, UK	Bedfordshire, England	Grand Forks, ND, USA
Pesticide use history at the collection site	Unknown	Unknown	See footnote b	Pursuit Plus; Double Play
Collection procedures	SOP ^c	SOP ^c	SOP ^c	SOP ^c
Sampling depth (cm)	SOP ^c	SOP ^c	SOP ^c	SOP ^c
Storage conditions	4 C	4 C	4 C	4 C
Storage length	4 months ^d	30 months ^d	22 months ^d	4 months ^d
Soil preparation	SOP ^c	SOP ^c	SOP ^c	SOP ^c

Table 1b: Description of soil collection and storage for supplementary study.

Description	M610	M611	M615	M623
Geographic location	Decatur County, KS, USA	Whitewater Rural Municipality, Manitoba, Canada	Ward County, ND, USA	Travis County, TX, USA
Pesticide use history at the collection site	None	None	None	Fertilizer (N)
Collection procedures	SOP ^c	SOP ^c	SOP ^c	SOP ^c
Sampling depth (cm)	SOP ^c	SOP ^c	0-20	0-20
Storage conditions	4 C	4 C	4 C	4 C
Storage length	7 months ^d	7 months ^d	6 months ^d	4 months ^d
Soil preparation	SOP ^c	SOP ^c	SOP ^c	SOP ^c

Data were obtained from pp. 21-22 and Appendix A, pp. 95-97 of the study report.

^a The following chemicals were used on M568 soil in the two years prior to collection: Bromoxynil, 97; MCPA ester, 97; Sethoxydim, 97; Propiconazole, 98; Dichlorprop, 98; 2,4-D, 98; Clodinafor-p-propargyl, 98; Chlorpyrifos, 98.

^b The following chemicals were used on M579 soil in the two years prior to collection: StaraneTM, Mar97; Asset, Mar 97; Tilt, April 97; Sulphurcut, May 97; Ammonium nitrate, Feb and March 98; Nkslge 24, May 98; Touchdown, Aug 98; Hallmark, Dec 98; Isotop portman, Dec 98; Javelin Gold, Dec 98; Ammonium nitrate, Mar 99; Bitersalz, Apr 99; Chlormequat, Apr 99; Fastmix mangane, Apr 99; Holdup, Apr 99; Mantra, Apr 99; Ammonium nitrate, Apr 99; Banshee, May 99; Mantra, May 99; Bavistin, Jun 99; Folicur, June 99; Mavrik, Jun 99; Clinic, Oct 99.

^c As per SOP ECL-5a: In a 15m x 15m area (50ft x 50ft) select approximately 10-12 sites. Remove any plant material growing in the area. Dig 15cm (6 inch) holes with a common garden spade and remove 15.2 inch deep slices of the soil. Make a composite of all samples taken, approximately 27 kg (60 lb). If the soil is too wet, dry it at ambient temperature to the point that it is moist, not air-dry, and will pass through a 2-mm sieve when crushed by hand or a wooden roller. Place the crushed and sieved moist soil in a labeled polyethylene bag and store in a labeled fiber pack (allow the exchange of oxygen and carbon dioxide and prevent excessive drying).

^d Approximate storage length from time of collection until date of soil moisture determinations.

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PMRA Submission Number {.....}

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Table 2a: Properties of the soils for definitive study.

Property	M568	M546	M549
Soil Texture ^a	Silty Clay Loam	Loam	Clay
% sand	16.5	37.0	25.4
% silt	46.3	45.8	28.6
% clay	37.2	17.2	46.0
pH	7.8	7.8	7.5
Organic carbon (%)	3.9	1.0	3.2
Organic matter (%)	6.7	1.7	5.5
CEC (meq/100 g)	24.6	9.9	32.7
Moisture at 1/3 atm (%)	37.5	24.1	40.3
Bulk density (g/cm ³)	1.2	1.1	1.2
Biomass (mg microbial C/100 g or CFU or other)	N/A	N/A	N/A
Soil taxonomic classification	N/A (Canada)	N/A (Europe)	N/A (Europe)
Soil mapping unit (for EPA)	N/A	N/A	N/A

Property	M599	M584	M617	M579	M616
Soil Texture ^b	Clay	Loam	Loamy Sand	Sand	Clay Loam
% sand	8	27	86	90	34
% silt	24	46	10	6	34
% clay	68	27	4	4	32
pH	6.9	6.1	4.5	6.6	4.8
Organic carbon (%)	1.5	1.0	0.6	1.6	3.6
Organic matter (%)	2.2	1.7	1.1	2.5	7.1
CEC (meq/100 g)	36.9	13.5	3.6	7.1	32.3
Moisture at 1/3 atm (%)	44.2	24.0	5.5	6.9	33.1
Bulk density (g/cm ³)	1.2	1.2	1.5	1.3	0.96
Biomass (mg microbial C/100 g or CFU or other)	N/A	N/A	N/A	N/A	N/A
Soil taxonomic classification	Very fine, smectitic, nonacid, thermic Vertic Endoaquepts	N/A (Europe)	Fine-loamy, kaolinitic, thermic Typic Kandiuults	N/A (Europe)	Fine-loamy, mixed, superactive, frigid, Calcic Hapludolls
Soil mapping unit (for EPA)	N/A	N/A	N/A	N/A	N/A

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Data Evaluation Report on the adsorption-desorption of aminopyralid (XDE-750) in soil

PMRA Submission Number {.....}

EPA MRID Number 46235732

Table 2b: Properties of the soils for supplementary study.

Property	M610	M611	M615	M623
Soil Texture ^b	Clay Loam	Loam	Sandy Loam	Heavy Clay
% sand	55	61	80	31
% silt	26	24	10	18
% clay	19	15	10	51
pH	4.6	7.5	7.3	7.5
Organic carbon (%)	1.5	3.4	1.2	3.4
Organic matter (%)	2.6	5.9	2.4	5.9
CEC (meq/100 g)	17.2	30.2	14.7	45.0
Moisture at 1/3 atm (%)	25.9	30.3	14.3	40.8
Bulk density (g/cm ³)	1.06	1.03	1.24	0.99
Biomass (mg microbial C/100 g or CFU or other)	N/A	N/A	N/A	N/A
Soil taxonomic classification	Fine-silty, mixed, mesic Typic Argiustolls	N/A (Canada)	Coarse-loamy over sandy or sandy-skeletal, mixed, superactive, frigid Typic Haplustolls	Fine, smectitic, thermic Udic Haplusterts
Soil mapping unit (for EPA)	N/A	N/A	N/A	N/A

Data were obtained from pp. 21-22 and Tables 1-3, pp. 50-54 of the study report.

^a These soils were classified according to the USDA Texture Classification; however, textural classes could not be confirmed because soil particle sizes were not reported.

^b The soils were classified according to the International Texture Classification.

C. STUDY DESIGN:

1. Preliminary study:

Several preliminary studies were conducted. Prior to the initiation of the preliminary studies, a stock solution was prepared by adding [2,6-¹⁴C]aminopyralid, dissolved in acetonitrile, to a 200-mL flask containing 0.01M CaCl₂ solution to establish a test concentration of 10 µg/mL (p. 23). The acetonitrile was evaporated under nitrogen and the stock solution was brought to volume with 0.01M CaCl₂ solution. Additional dosing solutions were also prepared prior to the initiation of the preliminary studies. A 60 µg/mL dosing solution was prepared by adding the remaining test material, dissolved in acetonitrile, to 1.45 mL of the [¹⁴C]aminopyralid stock solution, dissolved in acetone, that was used in the aerobic metabolism study. The acetonitrile was evaporated under nitrogen and the solution was brought to volume with 0.01M CaCl₂ solution. A 20 µg/mL dosing solution was prepared by diluting 5 mL of the 60 µg/mL dosing solution to 15 mL with 0.01M CaCl₂ solution. To prepare the 1.0 and 2.0 µg/mL dosing solutions, 2- and 4-mL aliquots of the 10 µg/mL stock solution, respectively, were brought to 20 mL with 0.01M CaCl₂ solution (pp. 23-24). All dosing solutions were analyzed for total radioactivity using LSC prior to dosing to ensure correct test concentration.

To determine filter sorption, 20 mL of a 0.01M CaCl₂ solution containing [2,6-¹⁴C]aminopyralid at a nominal test concentration of 0.5 µg/mL were added to a graduated

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cylinder and pushed through a Whatman 13-mm filter (0.45- μ m pore size) using a 10-mL syringe (pp. 24-25). The solution was captured in a 24 mL glass tube and triplicate aliquots were analyzed for total radioactivity using LSC. To determine container adsorption, 20 mL of a 0.01M CaCl₂ solution containing [2,6-¹⁴C]aminopyralid at a nominal test concentration of 0.5 μ g/mL were added to a 24-mL glass vial and capped with a Teflon-lined cap (p. 25). The samples were shaken for *ca.* 17 hours and 0.1-mL aliquots were analyzed for total radioactivity using LSC. Filter and container sorption tests showed that aminopyralid did not sorb to the filters or containers used during the definitive tests (pp. 25, 38).

Matrix interference was also investigated. Using soils M599 clay and M568 silty clay loam and 0.01M CaCl₂ solution, samples were prepared at a 1:1 soil:solution ratio and then shaken for approximately 4.5 hours. The 0.01 M CaCl₂ was decanted and fortified with [2,6-¹⁴C]aminopyralid to a nominal concentration of 0.5 μ g/mL. Duplicate control samples and a single blank sample per soil type were also prepared. Triplicate 0.1-mL aliquots of the fortified samples were analyzed for total radioactivity using LSC. The solution was then filtered using Whatman 13-mm filters (0.45- μ m pore size) and analyzed by HPLC. Comparison of the sample results to reference substance results showed no matrix interference (p. 39).

The Tier 1 preliminary study evaluated the 1:1, 1:5, and 1:10 soil:solution ratios using the M599 clay and M568 silty clay loam soils and 0.01 M CaCl₂ solution (pp. 25-26). The samples were pre-equilibrated by shaking on a horizontal shaker for *ca.* 17 hours in the dark at 25°C. Duplicate control samples and duplicate blanks per soil type were also prepared. The samples were then fortified at a nominal concentration of 0.5 μ g/mL and placed on a horizontal shaker in the dark at 25°C. Duplicate samples were analyzed at 2, 4, 8, 24, and 48 hours after initiation of shaking. Control samples were analyzed only at 24 hours. The samples were centrifuged, decanted, and triplicate 100- μ L aliquots were analyzed for total radioactivity using LSC. It was determined that [2,6-¹⁴C]aminopyralid did not sufficiently sorb to the test soil at the soil:solution ratios tested. Therefore, the experiment was repeated using a soil:solution ratio of 1:2 (w:v). Following adsorption, the samples were extracted three times with 90:10 acetone:1.0 N HCl by shaking for 60 minutes (30 minutes each for second and third extraction steps), centrifuging, and decanting into a separate glass vial. The extracts were pooled and weighed to determine the final volume. Aliquots of the extracts were analyzed for total radioactivity using LSC. To concentrate, 10-mL aliquots of the extracts were brought to a pH between 6 and 8 with 1.0 N NaOH using pH paper (p. 27). The samples were centrifuged and the solutions were pipetted into 15-mL centrifuge tubes. The extracts were concentrated to less than 1 mL in a TurboVap with the waterbath set at 30°C. The concentrated extracts were filtered through a 0.2- μ m filter into a 1-mL volumetric flask and brought to volume with HPLC-mobile phase (90:10 water with 0.5% TFA:acetonitrile with 0.5% TFA). The 48-hour concentrated extracts were analyzed using LSC and HPLC. The 48-hour soils were then air-dried, weighed, and analyzed for total radioactivity using LSC following combustion. Results indicated that the 1:2 soil:solution ratio was the most suitable soil:solution ratio (p. 39; Table 7, p. 58). Based on HPLC analysis, [2,6-

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¹⁴C]aminopyralid accounted for 98-100% of the radioactivity in the adsorption and extraction solutions.

The Tier 2 preliminary study evaluated the remaining soil types at the 1:2 soil:solution ratio to determine adsorption kinetics (pp. 27-28). The samples were pre-equilibrated by shaking *ca.* 5-g aliquots of each test soil with 9.49-mL aliquots of 0.01M CaCl₂ solution on a horizontal shaker for *ca.* 18 hours in the dark at 25°C. Duplicate control samples and duplicate blanks per soil type were also prepared. The samples were then fortified with [2,6-¹⁴C]aminopyralid at a nominal concentration of 0.5 µg/mL and placed on a horizontal shaker in the dark at 25°C. Duplicate samples were removed from the shaker and analyzed at 2, 4, 8, 24, and 48 hours after initiation of shaking. Control samples were analyzed only at 24 hours. The samples were centrifuged, decanted, and triplicate 500-µL aliquots were analyzed for total radioactivity using LSC. Following adsorption, the samples were extracted as previously described for the Tier 1 preliminary study. The 48-hour extracts were analyzed for total radioactivity using LSC. In addition, the 48-hour soils were combusted and analyzed as previously described. Results indicated that adsorption equilibrium was achieved in the preliminary Tiers 1 and 2 tests within 48 hours (p. 40; Table 8, p. 59). Based on HPLC analysis, [2,6-¹⁴C]aminopyralid accounted for at least 98% of the radioactivity in the 48-hour adsorption and extraction solutions.

The Tier 3 desorption kinetics tests employed a nominal 1:2 soil:solution ratio and an adsorption equilibration time of 48 hours. The samples were pre-equilibrated by shaking *ca.* 5-g aliquots of each test soil with 9.49-mL aliquots of 0.01M CaCl₂ solution on a horizontal shaker for *ca.* 65 hours in the dark at 25°C. Duplicate control samples and a single blank sample per soil type were also prepared. The samples were then fortified with [2,6-¹⁴C]aminopyralid at a nominal concentration of 0.5 µg/mL and placed on a horizontal shaker in the dark at 25°C for *ca.* 48 hours. The adsorption solution was decanted and an equivalent amount of fresh 0.01 M CaCl₂ was added back to the soil pellet. Triplicate 500-µL aliquots of the adsorption supernatant solutions were analyzed for total radioactivity using LSC. The samples were placed on a horizontal shaker in the dark at 25°C. Duplicate samples were analyzed at 2, 4, 8, 24, and 48 hours after initiation of shaking. Control samples were analyzed only at 24 hours. The samples were centrifuged, decanted, and triplicate 500-µL aliquots were analyzed for total radioactivity using LSC. Following desorption, the 2-hour samples were extracted and analyzed as previously described for the Tier 1 preliminary study. In addition, the soils were combusted as previously described. Results indicated that desorption equilibrium was achieved within 2 hours (p. 40; Table 9, p. 60). Based on HPLC analysis, [2,6-¹⁴C]aminopyralid accounted for >98% of the radioactivity in the 2-hour adsorption and desorption solutions. HPLC analysis of the concentrated extracts showed that >90% of the radioactivity was recovered as parent, except for the M584 loam soil, which was 82.2% (p. 41).

The soils from the aerobic soil metabolism study were added by protocol amendment and only analyzed for adsorption kinetics at one concentration (p. 30).

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Based on the study results, a soil:solution ratio of 1:2 (w:v), an adsorption equilibrium time of 48 hours, and a desorption equilibrium time of 2 hours were chosen to be used in the definitive study (pp. 39-41).

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2. Definitive study experimental conditions:

Table 3: Study design for the adsorption phase.

Parameters		All Soils ^a
Condition of soil (air dried/fresh) ^{b,c,d}		Fresh
Have these soils been used for other laboratory studies? (specify which)		Yes ^e
Soil (g/replicate)		5.0 g dry weight
Equilibrium solution used (name and concentration; e.g.: 0.01 N CaCl ₂)		0.01 M CaCl ₂
Control used (with salt solution only) (Yes/No)		Yes
Test material concentrations	Nominal application rates (mg a.i./kg soil)	0.1, 0.2, 1.0, 2.0, 10.0
	Analytically measured concentrations (mg a.i./kg soil)	0.096, 0.194, 1.004, 2.024, 9.914
Identity and concentration of co-solvent, if any		Acetonitrile; concentration not reported.
Soil:solution ratio		1:2
Initial pH of the equilibration solution, if provided		N/A
No. of replications	Controls	0
	Treatments	Duplicates per concentration
Equilibration	Time	48 hours
	Temperature (°C)	25; 5 ^{c,d}
	Darkness (Yes/No)	Yes
	Shaking method	Horizontal shaker
	Shaking time	48 hours
Method of separation of supernatant (e.g., centrifugation)		Centrifugation
Centrifugation	Speed (rpm)	3000
	Duration (min.)	30
	Method of separation of soil and solution	Decant

Data were obtained from pp. 22-24, 29-30 and Table 4, p. 55 of the study report.

^a All soils includes: eight test soils (M546 loam, M549 clay, M568 silty clay loam, M579 sand, M584 loam, M599 clay, M616 clay loam, and M617 loamy sand) that were equilibrated at 25°C, two test soils (M568 silty clay loam and M599 clay) that were equilibrated at 5°C, and four test soils (M610 clay loam, M611 loam, M615 sandy loam, and M623 heavy clay) that were used in the aerobic metabolism study and were equilibrated at 25°C.

^b The M546 loam, M549 clay, M568 silty clay loam, M579 sand, M584 loam, M599 clay, M616 clay loam, and M617 loamy sand test soils were pre-equilibrated by shaking overnight for *ca.* 16 hours with 0.01M CaCl₂ solution in an incubator set at 25°C.

^c Additional samples of the M568 silty clay loam and M599 clay test soils were pre-equilibrated by shaking overnight for *ca.* 16 hours with 0.01M CaCl₂ solution in an incubator set at 5°C. These samples were also treated with [2,6-¹⁴C]aminopyralid at a nominal test concentration of 1.0 mg a.i./kg soil and incubated at 5°C during equilibration.

^d The M610 clay loam, M611 loam, M615 sandy loam, and M623 heavy clay test soils were pre-equilibrated by shaking overnight for *ca.* 18 hours with 0.01M CaCl₂ solution in an incubator set at 25°C. These samples were also treated with [2,6-¹⁴C]aminopyralid at a nominal test concentration of 1.0 mg a.i./kg soil and incubated at 25°C during equilibration.

^e The M616 clay loam soil was used for the aminopyralid soil metabolism study.

Table 4: Study design for the desorption phase.

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Parameters		All Soils ^a
Were the soil residues from the adsorption phase used? If not, describe the method for adsorption using a separate adsorption Table		Yes
Amount of test material present in the adsorbed state/adsorbed amount (mg a.i./kg soil)	Concentration 1 (0.10 µg a.i./g soil)	0.00-0.03
	Concentration 2 (0.19 µg a.i./g soil)	0.00-0.06
	Concentration 3 (1.00 µg a.i./g soil)	0.01-0.28
	Concentration 4 (2.02 µg a.i./g soil)	0.00-0.50
	Concentration 5 (10.25 µg a.i./g soil)	0.00-2.12
No. of desorption cycles		1
Equilibration solution and quantity used per treatment for desorption (e.g., 0.01 M CaCl ₂)		0.01 M CaCl ₂
Soil:solution ratio		1:2
Replications	Controls	0
	Treatments	Duplicates per concentration
Desorption equilibration	Time	2 hours
	Temperature (°C)	25; 5 ^b
	Darkness	Yes
	Shaking method	Horizontal shaker
	Shaking time	2 hours
Centrifugation	Speed (rpm)	3500
	Duration (min)	30
	Method of separation of soil and solution	Decant

Data were obtained from pp. 29-30 and Table 5, p. 56 of the study report.

^a All soils includes: eight test soils (M546 loam, M549 clay, M568 silty clay loam, M579 sand, M584 loam, M599 clay, M616 clay loam, and M617 loamy sand) that were equilibrated at 25°C and two test soils (M568 silty clay loam and M599 clay) that were equilibrated at 5°C.

^b Additional samples of the M568 silty clay loam and M599 clay test soils were treated with [2,6-¹⁴C]aminopyralid at a nominal test concentration of 1.0 mg a.i./kg soil and incubated at 5°C during equilibration.

Supplemental study experimental conditions:

Two supplementary, non-guideline experiments were conducted to study the adsorption characteristics of [2,6-¹⁴C]aminopyralid in M568 silty clay loam and M599 clay test soils, in addition to four soil types used in an aerobic soil metabolism study designed for US guidelines: a clay loam (M610) [pH 4.6, o.c. 1.5%] from Kansas (USA), a loam (M611) [pH 7.5, o.c. 3.4%] from Manitoba (Canada), a sandy loam (M615) [pH 7.3, o.c. 1.2%] from North Dakota (USA), and a heavy clay (M623) [pH 7.5, o.c. 3.4%] from Texas (USA). The experimental methodology was identical to that used in the isotherm test, except samples of the M568, M599, M610, M611, M615, and M623 soils were treated with [2,6-¹⁴C]aminopyralid at a nominal test concentration of 1.0 mg a.i./kg soil only and incubated in the dark at 5°C (M568 and M599) or at 25°C (M610, M611, M615, and M623) for 48 hours. The M610, M611, M615, and M623 test soils were not desorbed.

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3. Description of definitive and supplementary study analytical procedures:

Extraction/clean up/concentration methods: Following desorption, all soils (except for the M610 clay loam, M611 loam, M615 sandy loam, and M623 heavy clay test soils) were extracted three times with 90:10 acetone:1.0 N HCl by shaking for 60 minutes (30 minutes second and third extraction steps), centrifuging, and decanting into a separate glass vial (pp. 26, 30; Figure 4, p. 82). The extracts were pooled and weighed to determine final volume (based on solution density).

To concentrate, ten-milliliter aliquots of the high-dose (10.0 mg a.i./kg soil) extracts and a single replicate for the M568 silty clay loam and M599 clay test soils equilibrated at 5°C were brought to a pH between 6 and 8 with 1.0 N NaOH using pH paper (pp. 27, 30). The samples were centrifuged and the solutions were pipetted into 15-mL centrifuge tubes. Next, the extracts were concentrated to less than 1 mL in a TurboVap with the waterbath set at 30°C. The concentrated extracts were then filtered through a 0.2-µm filter into a 1-mL volumetric flask and brought to volume with HPLC-mobile phase (90:10 water with 0.5% TFA:acetonitrile with 0.5% TFA).

Total ¹⁴C measurement: Following adsorption, desorption, and extraction, aliquots of the supernatants were analyzed for total radioactivity using LSC (pp. 26-27, 30). The extracted soils were air-dried and triplicate aliquots were analyzed for total radioactivity using LSC following combustion. The total ¹⁴C measurement was based on the amount of dpm in the adsorption and desorption supernatants, the soil extracts, and the extracted, air-dried soil pellets (as determined by combustion analysis; p. 38).

Non-extractable residues, if any: NA.

Derivatization method, if used: NA.

Identification and quantification of parent compound: The isocratic HPLC method used to characterize the high-dose (10.0 mg a.i./kg soil) adsorption and desorption supernatants, the concentrated soil extracts, and the adsorption supernatants for the M610 clay loam, M611 loam, M615 sandy loam, and M623 heavy clay test soils is shown below (pp. 29-30; 32):

Time	%A	%B
15 min	90	10

Where: A = water + 0.5% TFA and B = acetonitrile + 0.5% TFA.

The flow rate was 1 mL/min. The column was an Inertsil ODS-2 (5 µm particle size). Fractions were collected every 1.0 min for radioactive sample analysis and counted by LSC. Reconstructed radiochromatograms were generated using ChromaWiz version 1.0, an Excel macro developed at Dow AgroSciences. The initial analysis time was 15 minutes, but was extended to 20 minutes after a new Inertsil ODS-2 column was obtained. [2,6-¹⁴C]XDE-750 was identified by comparison of unlabeled reference standards that were identified using UV (270 nm; p. 33; Figure 3, p. 81).

Identification and quantification of transformation products: NA.

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Detection limits (LOD, LOQ) for the parent compound (indicate the criteria/reference, if provided): Using the method of Currie, the limits of detection and quantitation were calculated to be 13 and 57 dpm above background, respectively (pp. 34-36; Table 6, p. 57).

Detection limits (LOD, LOQ) for the transformation products, if appropriate: NA.

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II. RESULTS AND DISCUSSION

A. TEST CONDITIONS: The incubation temperatures were reported to be 25°C during the definitive study and 5°C during one supplementary study; no supporting data were provided (pp. 29-30). The pH of the test solutions during the definitive study was not reported. [2,6-¹⁴C]Aminopyralid remained stable throughout adsorption and desorption equilibrations; the high-dose (10.0 mg a.i./kg soil) adsorption and desorption solutions were composed of 98.3-100.0% [2,6-¹⁴C]aminopyralid (p. 42; Figures 5-6, pp. 83-84). Recovery of [2,6-¹⁴C]aminopyralid was low in the concentrated M579, M584, and M599 soil extracts. However, when combined with the radioactivity recovered from the precipitate formed after neutralization, material balances for the concentrated extracts were acceptable; [2,6-¹⁴C]aminopyralid comprised 97.1-99.6% of the radioactivity recovered in the concentrated extracts (Figure 7, p. 85). The test conditions outlined in the study protocol were maintained throughout the study.

B. MASS BALANCE: The mass balance at the end of the adsorption phase of the study was not reported. Mass balances at the end of the desorption phase in replicate samples were 98.3-101.0%, 101.6-103.8%, 96.2-100.0%, 98.4-101.6%, 95.5-99.8%, 101.0-104.1%, 94.6-98.1%, and 96.7-101.4% for the M546, M549, M568, M579, M584, M599, M616, and M617 test soils, respectively (pp. 38, 41-42; Table 10, pp. 61-63).

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Table 5a. Recovery of [2,6-¹⁴C]aminopyralid in the definitive isotherm test (expressed as % of applied radioactivity).

Phase ^a	M546	M549	M568	M579	M584	M599	M616	M617
Concentration : 0.048 µg/mL								
Adsorption	97.0	97.9	84.3	95.7	91.9	99.7	70.4	90.3
Desorption	0.9	0.0	5.7	2.2	0.5	0.0	9.1	4.2
Soil extract	0.2	0.0	2.2	1.4	2.0	2.2	14.5	5.8
Soil pellet ^b	1.6	4.5	6.7	0.6	1.1	2.2	1.7	0.6
Recovery	99.7	102.4	98.9	99.9	95.5	104.1	95.8	100.9
Adsorption	97.1	N/A ^c	85.0	95.5	93.5	100.0	69.3	91.1
Desorption	0.4	N/A ^c	5.2	1.6	0.0	0.0	9.5	4.0
Soil extract	0.3	N/A ^c	2.3	1.3	1.8	1.2	14.7	5.1
Soil pellet ^b	1.5	N/A ^c	3.7	0.6	1.5	2.3	1.6	0.8
Recovery	99.5	N/A ^c	96.2	99.0	96.9	103.5	95.1	101.0
all 0.048 µg/mL samples average mass balance								99.2
Concentration : 0.097 µg/mL								
Adsorption	97.0	97.9	85.9	94.8	96.9	99.5	69.4	92.3
Desorption	0.6	0.0	5.0	2.1	0.0	0.0	9.5	3.2
Soil extract	0.0	0.0	2.8	1.8	1.5	1.5	14.2	3.9
Soil pellet ^b	1.7	4.5	6.2	0.6	1.1	1.9	2.1	0.5
Recovery	99.4	102.3	100.0	99.2	99.4	102.8	95.2	99.8
Adsorption	98.1	97.1	85.8	95.4	96.7	98.2	70.1	92.1
Desorption	0.7	0.0	5.2	1.2	0.3	0.0	9.7	3.6
Soil extract	0.6	0.0	2.1	1.2	1.7	1.4	14.0	4.7
Soil pellet ^b	1.6	4.6	3.5	0.6	1.1	1.6	1.8	0.5
Recovery	101.0	101.6	96.6	98.4	99.8	101.3	95.6	101.0
all 0.097 µg/mL samples average mass balance								99.6

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Table 5a. (cont). Recovery of [2,6-¹⁴C]aminopyralid in the definitive isotherm test (expressed as % of applied radioactivity).

Phase ^a	M546	M549	M568	M579	M584	M599	M616	M617
Concentration : 0.502 µg/mL								
Adsorption	96.3	98.3	87.4	95.8	96.2	98.5	72.5	89.6
Desorption	0.6	0.0	4.3	1.5	0.0	0.0	9.1	2.8
Soil extract	0.0	0.0	1.7	1.4	1.2	0.8	12.0	3.9
Soil pellet ^b	1.4	3.9	4.3	0.6	0.9	1.7	1.6	0.4
Recovery	98.3	102.2	97.7	99.2	98.3	101.0	95.2	96.7
Adsorption	96.9	97.7	87.6	96.6	95.1	98.5	72.0	92.8
Desorption	0.7	0.0	4.3	1.4	0.3	0.0	8.6	2.3
Soil extract	0.0	0.0	1.7	1.3	1.3	0.5	12.2	3.7
Soil pellet ^b	1.5	4.0	5.2	0.5	1.0	2.1	1.7	0.4
Recovery	99.0	101.8	98.7	99.8	97.6	101.1	94.6	99.1
all 0.502 µg/mL samples average mass balance								98.8
Concentration : 1.012 µg/mL								
Adsorption	98.1	99.8	89.4	98.5	96.4	100.5	75.6	94.1
Desorption	0.3	0.0	4.0	1.5	0.6	0.0	8.4	3.3
Soil extract	0.0	0.0	1.6	1.4	1.7	0.0	11.4	3.9
Soil pellet ^b	1.1	3.4	4.7	0.2	0.6	1.7	0.9	0.2
Recovery	99.5	103.2	99.6	101.6	99.3	102.2	96.3	101.4
Adsorption	98.5	99.7	89.3	98.7	97.0	100.4	75.1	94.5
Desorption	0.5	0.0	4.3	1.4	0.2	0.0	8.9	2.9
Soil extract	0.0	0.0	1.5	1.1	1.1	0.3	11.6	3.1
Soil pellet ^b	1.1	3.7	2.9	0.3	0.7	1.7	1.2	0.2
Recovery	100.2	103.5	98.0	101.5	99.1	102.4	96.8	100.7
all 1.012 µg/mL samples average mass balance								100.3

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Table 5a. (cont). Recovery of [2,6-¹⁴C]aminopyralid in the definitive isotherm test (expressed as % of applied radioactivity).

Phase ^a	M546	M549	M568	M579	M584	M599	M616	M617	
Concentration : 4.957 µg/mL									
Adsorption	98.6	100.3	91.6	98.5	98.3	100.5	78.9	95.3	
Desorption	0.5	0.0	3.3	1.1	0.0	0.0	7.5	2.8	
Soil extract	0.0	0.0	0.7	0.8	0.8	0.0	9.2	3.0	
Soil pellet ^b	1.0	3.1	4.0	0.2	0.5	1.3	1.1	0.1	
Recovery	100.2	103.3	99.6	100.6	99.6	101.8	96.7	101.3	
Adsorption	99.2	100.4	91.3	98.1	97.2	101.2	79.6	95.8	
Desorption	0.3	0.0	3.3	1.3	0.2	0.0	8.1	2.5	
Soil extract	0.0	0.0	0.9	0.9	0.7	0.5	9.4	2.8	
Soil pellet ^b	1.0	3.4	3.6	0.2	0.5	1.2	1.0	0.2	
Recovery	100.5	103.8	99.2	100.6	98.6	102.9	98.1	101.3	
	all 4.957 µg/mL samples average mass balance								100.5
	all samples average mass balance								99.7

Data were obtained from Table 10, pp. 61-63 of the study report.

^a Adsorption = Adsorption solution; Desorption = Desorption solution.

^b The amount of radioactivity remaining on the soil was measured by oxidative combustion.

^c Sample lost during desorption phase. Not included in average, Freundlich, K_d, etc. calculations.

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Table 6a: Concentration of [2,6-¹⁴C]aminopyralid in the solid and liquid phases at the end of adsorption equilibration period in the definitive isotherm test (n = 2; mean ± s.d.).

Nominal Conc. ^d (µg a.i./mL)	M546			M549			M568		
	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% adsorbed ^b	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% adsorbed ^b	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% adsorbed ^b
Control	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
0.048	0.003 ± 0.0	0.042 ± 0.0	2.9 ± 0.1	0.002 ^c	0.044 ^c	2.1 ^c	0.015 ± 0.0	0.037 ± 0.0	15.4 ± 0.5
0.097	0.005 ± 0.0	0.085 ± 0.0	2.4 ± 0.8	0.005 ± 0.0	0.088 ± 0.0	2.5 ± 0.6	0.027 ± 0.0	0.076 ± 0.0	14.1 ± 0.1
0.502	0.035 ± 0.0	0.436 ± 0.0	3.4 ± 0.4	0.020 ± 0.0	0.464 ± 0.0	2.0 ± 0.4	0.126 ± 0.0	0.402 ± 0.0	12.5 ± 0.1
1.012	0.034 ± 0.0	0.897 ± 0.0	1.7 ± 0.3	0.004 ± 0.0	0.949 ± 0.0	0.2 ± 0.3	0.216 ± 0.0	0.828 ± 0.0	10.7 ± 0.1
4.957	0.112 ± 0.0	4.434 ± 0.0	1.1 ± 0.4	0.000 ± 0.0	4.687 ± 0.0	0.0 ± 0.0	0.878 ± 0.0	4.163 ± 0.0	8.6 ± 0.2

Nominal Conc. ^d (µg a.i./mL)	M579			M584			M599		
	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% adsorbed ^b	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% adsorbed ^b	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% adsorbed ^b
Control	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
0.048	0.004 ± 0.0	0.043 ± 0.0	4.4 ± 0.1	0.007 ± 0.0	0.043 ± 0.0	7.3 ± 1.1	0.000 ± 0.0	0.056 ± 0.0	0.1 ± 0.2
0.097	0.010 ± 0.0	0.086 ± 0.0	4.9 ± 0.4	0.006 ± 0.0	0.089 ± 0.0	3.2 ± 0.1	0.002 ± 0.0	0.090 ± 0.0	1.2 ± 0.9
0.502	0.38 ± 0.0	0.453 ± 0.0	3.8 ± 0.6	0.043 ± 0.0	0.461 ± 0.0	4.3 ± 0.8	0.015 ± 0.0	0.467 ± 0.0	1.5 ± 0.0
1.012	0.028 ± 0.0	0.934 ± 0.0	1.4 ± 0.1	0.067 ± 0.0	0.932 ± 0.0	3.3 ± 0.4	0.000 ± 0.0	0.972 ± 0.0	0.0 ± 0.0
4.957	0.174 ± 0.0	4.574 ± 0.0	1.7 ± 0.3	0.232 ± 0.1	4.660 ± 0.0	2.3 ± 0.8	0.000 ± 0.0	4.733 ± 0.0	0.0 ± 0.0

Nominal Conc. ^d (µg a.i./mL)	M616			M617		
	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% adsorbed ^b	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% adsorbed ^b
Control	N/A	N/A	N/A	N/A	N/A	N/A
0.048	0.029 ± 0.0	0.029 ± 0.0	30.1 ± 0.8	0.009 ± 0.0	0.042 ± 0.0	9.3 ± 0.6
0.097	0.058 ± 0.0	0.058 ± 0.0	30.2 ± 0.5	0.015 ± 0.0	0.086 ± 0.0	7.8 ± 0.1
0.502	0.279 ± 0.0	0.315 ± 0.0	27.8 ± 0.4	0.088 ± 0.0	0.448 ± 0.0	8.8 ± 2.3
1.012	0.498 ± 0.0	0.660 ± 0.0	24.7 ± 0.4	0.116 ± 0.0	0.925 ± 0.0	5.7 ± 0.3
4.957	2.122 ± 0.1	3.403 ± 0.0	20.7 ± 0.5	0.456 ± 0.0	4.571 ± 0.0	4.4 ± 0.4

Data were obtained from Table 11, pp. 64-67 of the study report.

^a Concentration on soil calculated by difference (total applied minus concentration in solution).

^b % adsorbed as the % of the applied radioactivity.

^c Single replicate data due to spillage (Table 11, p. 67).

^d Actual concentrations applied were 3.7-14.6% less than labeled nominal rate.

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Table 7a: Concentration of [2,6-¹⁴C]aminopyralid in the solid and liquid phases at the end of desorption in the definitive isotherm test (n = 2; mean ± s.d.).

Nominal Conc. ^d (µg a.i./mL)	M546			M549			M568		
	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% desorbed as % of adsorbed	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% desorbed as % of adsorbed	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% desorbed as % of adsorbed
Control	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
0.048	0.002 ± 0.0	0.0003 ± 0.0	22.5 ± 10.5	0.002 ^b	0.0 ^b	ND ^b	0.007 ± 0.0	0.0024 ± 0.0	35.5 ± 1.5
0.097	0.004 ± 0.0	0.0005 ± 0.0	27.6 ± 11.0	0.005 ± 0.0	0.0 ± 0.0	ND	0.014 ± 0.0	0.0045 ± 0.0	36.1 ± 0.9
0.502	0.011 ± 0.0	0.0030 ± 0.0	19.9 ± 3.8	0.021 ± 0.0	0.0 ± 0.0	ND	0.064 ± 0.0	0.0196 ± 0.0	34.2 ± 0.4
1.012	0.011 ± 0.0	0.0036 ± 0.0	25.2 ± 16.1	0.025 ± 0.0	0.0 ± 0.0	ND	0.109 ± 0.0	0.0384 ± 0.0	38.8 ± 2.0
4.957	0.091 ± 0.0	0.0186 ± 0.0	36.6 ± 1.6	0.106 ± 0.0	0.0 ± 0.0	ND	0.476 ± 0.0	0.1534 ± 0.0	38.4 ± 0.5

Nominal Conc. ^d (µg a.i./mL)	M579			M584			M599		
	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% desorbed as % of adsorbed	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% desorbed as % of adsorbed	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% desorbed as % of adsorbed
Control	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
0.048	0.002 ± 0.0	0.0009 ± 0.0	44.1 ± 10.8	0.003 ± 0.0	0.0001 ± 0.0	3.4 ± 4.1	0.004 ± 0.0	0.0 ± 0.0	ND
0.097	0.004 ± 0.0	0.0015 ± 0.0	33.1 ± 9.4	0.005 ± 0.0	0.0003 ± 0.0	8.6 ^c	0.006 ± 0.0	0.0 ± 0.0	ND
0.502	0.019 ± 0.0	0.0069 ± 0.0	38.7 ± 4.7	0.022 ± 0.0	0.0014 ± 0.0	5.9 ^c	0.026 ± 0.0	0.0 ± 0.0	ND
1.012	0.030 ± 0.0	0.0139 ± 0.0	105.9 ± 6.8	0.042 ± 0.0	0.0040 ± 0.0	12.1 ± 5.7	0.037 ± 0.0	0.0 ± 0.0	ND
4.957	0.110 ± 0.0	0.0580 ± 0.0	71.4 ± 0.6	0.130 ± 0.0	0.0083 ± 0.0	6.0 ^c	0.135 ± 0.1	0.0 ± 0.0	ND

Nominal Conc. ^d (µg a.i./mL)	M616			M617		
	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% desorbed as % of adsorbed	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% desorbed as % of adsorbed
Control	N/A	N/A	N/A	N/A	N/A	N/A
0.048	0.015 ± 0.0	0.0039 ± 0.0	31.0 ± 0.1	0.006 ± 0.0	0.0019 ± 0.0	44.4 ± 1.6
0.097	0.031 ± 0.0	0.0081 ± 0.0	31.8 ± 1.0	0.009 ± 0.0	0.0032 ± 0.0	43.5 ± 4.0
0.502	0.139 ± 0.0	0.0386 ± 0.0	31.9 ± 1.6	0.041 ± 0.0	0.0127 ± 0.0	29.6 ± 3.3
1.012	0.253 ± 0.0	0.0757 ± 0.0	35.0 ± 0.8	0.075 ± 0.0	0.0305 ± 0.0	54.3 ± 2.1
4.957	1.056 ± 0.0	0.3458 ± 0.0	37.7 ± 2.9	0.315 ± 0.0	0.1331 ± 0.0	59.7 ± 0.3

Data were obtained from Table 12, pp. 68-71 of the study report.

^a Concentration on soil calculated by addition of total µg in soil extract and in soil (determined by oxidative combustion), and dividing by dry soil weight.

^b Single replicate data due to spillage (Table 12, p. 71).

^c Single replicate data due to no desorption.

^d Actual concentrations applied were 3.7-14.6% less than labeled nominal rate.

ND = No desorption.

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Table 8a: Adsorption and desorption constants of [2,6-¹⁴C]aminopyralid in the definitive isotherm test.

Soil	Adsorption					Desorption				
	K _F ^a	1/N	R ²	K _d ^b	K _{oc}	K _F ^a	1/N	R ²	K _d ^b	K _{oc}
M546	0.04	0.79	0.951	0.04 +0.02	4.49 +1.95	1.97	0.87	0.888	5.98 +2.90	598.44 +289.57
M549	0.01	0.32	0.215	0.03 +0.02	1.05 +0.73	No Desorption				
M568	0.26	0.87	0.999	0.29 +0.07	7.39 +1.83	3.09	1.00	0.986	3.59 +0.43	92.16 +11.04
M579	0.05	0.75	0.944	0.07 +0.03	4.59 +2.10	1.72	0.94	0.991	2.60 +2.08	162.27 +130.14
M584	0.07	0.82	0.956	0.08 +0.03	7.54 +2.93	1.24	0.61	0.833	19.14 +4.64	1914.50 +464.44
M599	0.04	1.31	0.879	0.03 +0.01	2.33 +0.58	No Desorption				
M616	0.73	0.90	0.999	0.72 +0.15	19.95 +4.06	2.88	0.94	1.000	3.80 +0.55	105.66 +15.20
M617	0.13	0.86	0.987	0.15 +0.05	24.30 +8.33	2.12	0.94	0.996	2.18 +1.07	362.78 +178.19

Reviewer-calculated from data obtained from pp. 42-43; Table 13, p. 72; Figures 8-15, pp. 86-93; and Appendix D, pp. 113-115 of the study report.

K_d – Adsorption and desorption coefficients; K – Freundlich adsorption and desorption coefficients; 1/N – Slope of Freundlich adsorption/desorption isotherms

K_{oc} – Coefficient adsorption per organic carbon (K_d or K x 100/% organic carbon)

R² – Regression coefficient of Freundlich equation

^a K_F units: unitless

^b K_d units: mL/g

C. ADSORPTION: Adsorption decreased with increasing concentration. After 48 hours of equilibration, an average of an average of 2.3%, 1.3%, 12.2%, 3.2%, 4.1%, 0.6%, 26.7%, and 7.2% of the applied [2,6-¹⁴C]aminopyralid was adsorbed in soils M546, M549, M568, M579, M584, M599, M616, and M617, respectively (Table 11, pp. 64-67). The adsorption average K_d values were 0.04, 0.03, 0.29, 0.07, 0.08, 0.03, 0.72, and 0.15 mL/g in soils M546, M549, M568, M579, M584, M599, M616, and M617, respectively (reviewer-calculated from Table 11 and Appendix D, pp. 64-67, 113-115). The corresponding average adsorption K_{oc} values ranged from 1.05 to 24.30 mL/g. The Freundlich adsorption K_F values ranged from 0.01 to 0.73.

D. DESORPTION: At the end of the desorption phase, an average of 27.3%, 0.0%, 42.1%, 47.6%, 8.0%, 0.0%, 39.4% and 42.3% of the adsorbed amount was desorbed from soils M546, M549, M568, M579, M584, M599, M616, and M617, respectively. The desorption average K_d values were 5.98, 0.00, 3.59, 2.60, 19.14, 0.0, 3.80 and 2.18 mL/g in soils M546, M549, M568, M579, M584, M599, M616, and M617, respectively. (reviewer-calculated from Table 11, 12 and Appendix D, pp. 64-71, 113-115). The corresponding average desorption K_{oc} values ranged from 0.0 to 1914.5 mL/g. The desorption K_d and K_{oc} values were higher than those obtained for adsorption. The Freundlich desorption K_F values ranged from 1.24 to 3.09.

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E. SUPPLEMENTAL STUDY- RESULTS:

For soils M568 and M599 equilibrated at 5°C, mass balances at the end of the desorption phase in replicate samples were 98.7-98.8% and 99.0-99.7%, respectively (p. 43; Table 14, p. 73).

For soils M610, M611, M615, and M623, mass balances at the end of the adsorption phase in replicate samples were 95.5%, 94.1-95.1%, 97.6-97.9% and 95.5-95.8%, respectively. (p. 44; Table 17, p. 76).

Table 5b. Recovery of [2,6-¹⁴C]aminopyralid in the first supplementary study (expressed as % of applied radioactivity) for soils incubated at 5°C.

Phase ^a	M546	M549
Concentration : 0.502 µg/mL		
Adsorption	87.8	98.4
Desorption	5.0	0.0
Soil extract	1.6	0.4
Soil pellet ^b	4.3	0.2
Recovery	98.7	99.0
Adsorption	88.0	98.9
Desorption	5.2	0.0
Soil extract	1.5	0.6
Soil pellet ^b	4.1	0.2
Recovery	98.8	99.7
	average mass balance	99.1%

Data were obtained from Table 14, p. 73 of the study report.

^a Adsorption = Adsorption solution; Desorption = Desorption solution.

^b The amount of radioactivity remaining on the soil was measured by oxidative combustion.

Table 5c. Recovery of [2,6-¹⁴C]aminopyralid in the second supplementary study (expressed as % of applied radioactivity) for aerobic metabolism soils incubated at 25°C.

Phase ^a	M610	M611	M615	M623
Concentration : 0.507 µg/mL				
Adsorption	61.4	59.2	72.2	58.3
Soil extract	32.8	32.5	24.4	25.1
Soil pellet ^b	1.4	2.3	1.3	12.4
Recovery	95.5	94.1	97.9	95.8
Adsorption	61.5	60.0	71.8	58.0
Soil extract	32.6	32.7	24.4	25.0
Soil pellet ^b	1.5	2.4	1.3	12.6
Recovery	95.5	95.1	97.6	95.5
	average mass balance			95.9%

Data were obtained from Table 17, p. 76 of the study report.

^a Adsorption = Adsorption solution.

^b The amount of radioactivity remaining on the soil was measured by oxidative combustion.

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Table 6b: Concentration of [2,6-¹⁴C]aminopyralid in the solid and liquid phases at the end of adsorption equilibration period in the first supplementary study for soils incubated at 5°C (n = 2; mean ± s.d.).

Nominal Conc. ^c (µg a.i./mL)	M568			M599		
	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% adsorbed ^b	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% adsorbed ^b
Control						
0.502	0.12 ± 0.0	0.407 ± 0.0	12.1 ± 0.1	0.01 ± 0.0	0.467 ± 0.0	1.4 ± 0.4

Data were obtained from Table 15, p. 74 of the study report.

^a Concentration on soil calculated by difference (total applied minus concentration in solution).

^b % adsorbed as the % of the applied radioactivity.

^c Actual concentrations applied were not reported.

Table 7b: Concentration of [2,6-¹⁴C]aminopyralid in the solid and liquid phases at the end of desorption in the first supplementary study for soils incubated at 5°C (n = 2; mean ± s.d.).

Nominal Conc. ^b (µg a.i./mL)	M568			M599		
	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% desorbed as % of the adsorbed	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% desorbed as % of the adsorbed
Control						
0.502	0.06 ± 0.0	0.02 ± 0.0	42.2 ± 1.4	0.03 ± 0.0	0.0 ± 0.0	ND

Data were obtained from Table 15, p. 74 of the study report.

^a Concentration on soil calculated by addition of total µg in soil extract and in soil (determined by oxidative combustion), and dividing by dry soil weight.

^b Actual concentrations applied were not reported.

ND = No desorption.

Table 6c: Concentration of [2,6-¹⁴C]aminopyralid in the solid and liquid phases at the end of adsorption equilibration period in the second supplementary study for aerobic metabolism soils incubated at 25°C (n = 2; mean ± s.d.).

Nominal Conc. ^c (µg a.i./mL)	M610			M611		
	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% adsorbed ^b	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% adsorbed ^b
Control						
0.507	0.17 ± 0.0	0.407 ± 0.0	3.3 ± 0.0	0.16 ± 0.0	0.400 ± 0.0	3.1 ± 0.1

Nominal Conc. ^c (µg a.i./mL)	M615			M623		
	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% adsorbed ^b	on soil ^a (mg a.i./kg)	In solution (µg a.i./mL)	% adsorbed ^b
Control						
0.507	0.03 ± 0.0	0.478 ± 0.0	0.6 ± 0.0	0.09 ± 0.0	0.439 ± 0.0	1.8 ± 0.0

Data were obtained from Table 18, p. 77 of the study report.

^a Concentration on soil calculated by difference (total applied minus concentration in solution).

^b % adsorbed as the % of the applied radioactivity.

^c Actual concentrations applied were not reported.

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Table 8b: Adsorption and desorption constants of [2,6-¹⁴C]aminopyralid in the first supplementary study for soils incubated at 5°C.

Soil	Adsorption		Desorption	
	K _d ^a	K _{oc} ^a	K _d ^a	K _{oc} ^a
M568	0.47	12.05	6.13	157.19
M599	0.15	10.15	No Desorption	

Reviewer-calculated from data were obtained from p. 44 and Tables 15-16, p. 74-75 of the study report.

^a K_d and K_{oc} units: mL/g.

Table 8c: Adsorption constants of [2,6-¹⁴C]aminopyralid in the second supplementary study for aerobic metabolism soils incubated at 25°C.

Soil	Adsorption	
	K _d ^a	K _{oc} ^a
M610	0.49	32.96
M611	0.53	15.64
M615	0.12	9.93
M623	0.31	9.04

Reviewer-calculated from data obtained from p. 44 and Table 18-19, p. 77-78 of the study report.

^a K_d and K_{oc} units: mL/g.

For soils M568 and M599 incubated at 5°C, 12.1% and 1.4% of the applied [2,6-¹⁴C]aminopyralid was adsorbed, respectively, after 48 hours of equilibration (Table 15, p. 74). The adsorption average K_d values were 0.47 and 0.15 mL/g in M568 and M599, respectively (reviewer-calculated from Tables 15-16 and Appendix D, pp. 74-75, 113-115). The corresponding average adsorption K_{oc} values were 12.0 and 10.2 mL/g.

For soils M568 and M599, incubated at 5°C, 42.2% and 0.0% of the adsorbed amount, respectively, was desorbed at the end of the desorption phase (Table 15, p. 74). The desorption average K_d values were 6.13 and 0.0 mL/g, respectively (reviewer-calculated from Tables 15-16 and Appendix D, pp. 74-75, 113-115); corresponding desorption K_{oc} values were 157.2 and 0.0 mL/g. It does not appear that incubation temperature affects adsorption.

For soils M610, M611, M615, and M623, 3.3%, 3.1%, 0.6%, and 1.8% of the applied [2,6-¹⁴C]aminopyralid was reported adsorbed, respectively, after 48 hours of equilibration (Table 18, p. 77). However, when calculated by subtracting percent applied radioactivity in adsorption supernatant from 100% applied radioactivity, 38.6%, 40.4%, 28.0%, 41.9% of the applied aminopyralid was adsorbed, respectively (see Reviewer's Comments). The adsorption average K_d values were 0.49, 0.53, 0.12, and 0.31 mL/g in M610, M611, M615 and M623, respectively (reviewer-calculated from Tables 18-19 and Appendix D, pp. 77-78, 113-115). The corresponding average adsorption K_{oc} values were 32.96, 15.64, 9.93, and 9.04 mL/g, respectively.

III. STUDY DEFICIENCIES: This study is classified supplemental for an adsorption/desorption study in soil because none of the test soils had an organic matter content of ≤1%, as required by Subdivision N guidelines.

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IV. REVIEWER'S COMMENTS:

1. None of the test soils had an organic matter content of $\leq 1\%$, as required by Subdivision N guidelines.
2. Six of the twelve test soils used in this study were foreign in origin. The M568 silty clay loam, M546 loam, and M549 clay soils were collected from Canada, Greece, and the UK, respectively. It was reported that these three test soils were classified according to the USDA Texture Classification, with reported classifications corresponding to reported sand, silt, and clay percentages. However, the remaining nine soils, both foreign and US, were classified according to the International Texture Classification. Neither USDA nor International textural classification could be confirmed because soil particle sizes were not reported.
3. The definitive and supplementary study temperatures were reported to be 25° and 5°C (pp. 29-30). More detailed information was not provided. It is preferred that minimum, maximum, and average temperatures be reported. Any significant deviations from the average and their duration should be noted.
4. The K_d values reported in the study report were calculated using the equation:
 $K_d = S/C_{eq}$ where
 S = the sorbed phase concentration with units of mass of sorbate per solid sorbent mass; and
 C_{eq} = the aqueous-phase equilibrium concentration;
 To confirm adsorption constant data reported in the study report, the reviewer calculated adsorption K_d values using the following equation:
 $K_d = [(C_0V_0 - C_{eq}V_0)/M] / C_{eq}$ where
 C_0 = the concentration in the water before sorption;
 V_0 = the total water volume in the batch system;
 C_{eq} = the aqueous-phase equilibrium concentration; and
 M = the dry mass of sorbent.

The adsorption K_d values obtained by the reviewer were similar to those reported by the registrant. A comparison of these results are reported below:

Table 9: Comparison of reviewer-calculated and registrant-reported adsorption constants of aminopyralid in the soils.

Soil	Adsorption K_d (reviewer-calculated) (mL/g)	Adsorption K_d (registrant-reported) (mL/g)
M546	0.04	0.053
M549	0.03	0.036
M568 (25°C)	0.29	0.308
M568 (5°C)(supplementary)	0.47	0.298
M579	0.07	0.072
M584	0.08	0.089

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M599 (25°C)	0.03	0.024
M599 (5°C)(supplementary)	0.15	0.029
M616	0.72	0.853
M617	0.15	0.162
M610 (supplementary)	0.49	0.409
M611 (supplementary)	0.53	0.395
M615 (supplementary)	0.12	0.066
M623 (supplementary)	0.31	0.211

Adsorption values were reviewer-calculated using data obtained from Table 11, 15, 18, and Appendix D, pp. 64-67, 74, 77, and 113-115 of the study report.

The reviewer-calculated r^2 value for the relationship of K_d vs. % organic carbon was 0.3656, for K_d vs. % clay was 0.0003, for K_d vs. pH was 0.1725 and for K_d vs. cation exchange capacity was 0.0998.

- Control samples were not prepared for the definitive adsorption or desorption tests.
- The $1/n$ values associated with the Freundlich adsorption K value were not within the range of 0.9 to 1.1 for the M546, M549, M568, M579, M584, and M617 test soils ($1/n = 0.32-1.31$; reviewer-calculated from Table 11, p. 64-67). The $1/n$ values associated with the Freundlich desorption K value were not within the range of 0.9 to 1.1 for the M546 and M584 test soils ($1/n = 0.61-0.87$). If the $1/n$ value is not within the range of 0.9 to 1.1, then the Freundlich isotherm may not adequately or accurately represent adsorption/desorption of the compound across all test concentrations.
- Based on the results of the study, the study author concluded that aminopyralid is potentially mobile in all soils types tested (p. 45). Under the McCall Classification (Swann et al., 1983), based on K_{OC} values ranging from 1.05 to 24.30, aminopyralid is expected to be highly mobile in all soils types tested.
- Percent adsorbed reported for the supplementary study on aerobic metabolism soils (Table 18, p. 77) ranged from 0.6-3.3% of applied radioactivity. Percent adsorbed calculated by subtracting % applied radioactivity in adsorption supernatant from 100% (Table 17, p. 76) for the same soils ranged from 28.0-41.9% of applied radioactivity. The reason for this discrepancy is unclear.
- Actual applied initial concentration rate (reported in Appendix D, pp. 113-115) ranged from 3.7-14.6% less than the labeled rate (reported in Tables 11 and 12, pp. 64-71).

V. REFERENCES:

Data Evaluation Report on the adsorption-desorption of aminopyralid (XDE-750) in soil

PMRA Submission Number {.....}

EPA MRID Number 46235732

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Data Evaluation Report on the adsorption-desorption of aminopyralid (XDE-750) in soil

PMRA Submission Number {.....}

EPA MRID Number 46235732

- 9 U.S. Environmental Protection Agency. 2003. Guidance for Calculating Sorption Coefficients in Batch Equilibrium Studies.

Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

Table 4/6a Adsorption soil (ug a.i./g)

Rate (ug/mL)	M546	M549	M568	M579	M584	M599	M616	M617
0.048	0.003	0.002	0.015	0.004	0.008	0	0.028	0.009
0.048	0.003	-	0.014	0.004	0.006	0	0.029	0.008
AVG	0.003	0.002	0.015	0.004	0.007	0.000	0.029	0.009
STDEV	0.000		0.001	0.000	0.001	0.000	0.001	0.001
0.097	0.006	0.004	0.027	0.01	0.006	0.001	0.059	0.015
0.097	0.004	0.006	0.027	0.009	0.006	0.003	0.058	0.015
AVG	0.005	0.005	0.027	0.010	0.006	0.002	0.059	0.015
STDEV	0.001	0.001	0.000	0.001	0.000	0.001	0.001	0.000
0.502	0.038	0.017	0.127	0.042	0.038	0.015	0.277	0.105
0.502	0.031	0.023	0.124	0.034	0.049	0.015	0.281	0.072
AVG	0.035	0.020	0.126	0.038	0.044	0.015	0.279	0.089
STDEV	0.005	0.004	0.002	0.006	0.008	0.000	0.003	0.023
1.012	0.039	0.004	0.216	0.031	0.074	0	0.495	0.12
1.012	0.03	0.005	0.216	0.026	0.06	0	0.502	0.112
AVG	0.035	0.005	0.216	0.029	0.067	0.000	0.499	0.116
STDEV	0.006	0.001	0.000	0.004	0.010	0.000	0.005	0.006
4.957	0.139	0.000	0.861	0.156	0.175	0.000	2.162	0.484
4.957	0.085	0.000	0.896	0.192	0.289	0.000	2.083	0.428
AVG	0.112	0.000	0.879	0.174	0.232	0.000	2.123	0.456
STDEV	0.038	0.000	0.025	0.025	0.081	0.000	0.056	0.040

Data were obtained from Table 11, pp. 64-67 of the study report.

Table 5a Adsorption supernatants (% applied)

Rate (ug/mL)	M546	M549	M568	M579	M584	M599	M616	M617
0.048	97	97.9	84.3	95.7	91.9	99.7	70.4	90.3
0.048	97.1	-	85	95.5	93.5	100	69.3	91.1
0.097	97	97.9	85.9	94.8	96.9	99.5	69.4	92.3
0.097	98.1	97.1	85.8	95.4	96.7	98.2	70.1	92.1
0.502	96.3	98.3	87.4	95.8	96.2	98.5	72.5	89.6
0.502	96.9	97.7	87.6	96.6	95.1	98.5	72	92.8
1.012	98.1	99.8	89.4	98.5	96.4	100.5	75.6	94.1
1.012	98.5	99.7	89.3	98.7	97	100.4	75.1	94.5
4.957	98.6	100.3	91.6	98.5	98.3	100.5	78.9	95.3
4.957	99.2	100.4	91.3	98.1	97.2	101.2	79.6	95.8
AVG	97.68	98.79	87.76	96.76	95.92	99.70	73.29	92.79
STDEV	0.94	1.25	2.57	1.53	1.92	1.02	3.83	2.11

Data were obtained from Table 10, pp. 61-63 of the study report.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

Table 5a Desorption supernatants (% applied)

Rate (ug/mL)	M546	M549	M568	M579	M584	M599	M616	M617
0.048	0.9	0	5.7	2.2	0.5	0	9.1	4.2
0.048	0.4	-	5.2	1.6	0	0	9.5	4
0.097	0.6	0	5	2.1	0	0	9.5	3.2
0.097	0.7	0	5.2	1.2	0.3	0	9.7	3.6
0.502	0.6	0	4.3	1.5	0	0	9.1	2.8
0.502	0.7	0	4.3	1.4	0.3	0	8.6	2.3
1.012	0.3	0	4	1.5	0.6	0	8.4	3.3
1.012	0.5	0	4.3	1.4	0.2	0	8.9	2.9
4.957	0.5	0	3.3	1.1	0	0	7.5	2.8
4.957	0.3	0	3.3	1.3	0.2	0	8.1	2.5
AVG	0.55	0.00	4.46	1.53	0.21	0.00	8.84	3.16
STDEV	0.19	0.00	0.81	0.36	0.22	0.00	0.69	0.62

Table 5a Extracted (% applied)

Rate (ug/mL)	M546	M549	M568	M579	M584	M599	M616	M617
0.048	0.2	0	2.2	1.4	2	2.2	14.5	5.8
0.048	0.3	-	2.3	1.3	1.8	1.2	14.7	5.1
0.097	0	0	2.8	1.8	1.5	1.5	14.2	3.9
0.097	0.6	0	2.1	1.2	1.7	1.4	14	4.7
0.502	0	0	1.7	1.4	1.2	0.8	12	3.9
0.502	0	0	1.7	1.3	1.3	0.5	12.2	3.7
1.012	0	0	1.6	1.4	1.7	0	11.4	3.9
1.012	0	0	1.5	1.1	1.1	0.3	11.6	3.1
4.957	0	0	0.7	0.8	0.8	0	9.2	3
4.957	0	0	0.9	0.9	0.7	0.5	9.4	2.8
AVG	0.11	0.00	1.75	1.26	1.38	0.84	12.32	3.99
STDEV	0.20	0.00	0.64	0.28	0.43	0.72	2.01	0.96

Table 5a Combusted (% applied)

Rate (ug/mL)	M546	M549	M568	M579	M584	M599	M616	M617
0.048	1.6	4.5	6.7	0.6	1.1	2.2	1.7	0.6
0.048	1.5	-	3.7	0.6	1.5	2.3	1.6	0.8
0.097	1.7	4.5	6.2	0.6	1.1	1.9	2.1	0.5
0.097	1.6	4.6	3.5	0.6	1.1	1.6	1.8	0.5
0.502	1.4	3.9	4.3	0.6	0.9	1.7	1.6	0.4
0.502	1.5	4	5.2	0.5	1	2.1	1.7	0.4
1.012	1.1	3.4	4.7	0.2	0.6	1.7	0.9	0.2
1.012	1.1	3.7	2.9	0.3	0.7	1.7	1.2	0.2
4.957	1	3.1	4	0.2	0.5	1.3	1.1	0.1
4.957	1	3.4	3.6	0.2	0.5	1.2	1	0.2
AVG	1.35	3.90	4.48	0.44	0.90	1.77	1.47	0.39
STDEV	0.27	0.55	1.23	0.19	0.32	0.36	0.39	0.22

Data were obtained from Table 10, pp. 61-63 of the study report.

Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

Table 5a Recovery Mass Balance (% applied)

Rate (ug/mL)	M546	M549	M568	M579	M584	M599	M616	M617
0.048	99.7	102.4	98.9	99.9	95.5	104.1	95.7	100.9
0.048	99.3	-	96.2	99.0	96.8	103.5	95.1	101.0
0.097	99.3	102.4	99.9	99.3	99.5	102.9	95.2	99.9
0.097	101.0	101.7	96.6	98.4	99.8	101.2	95.6	100.9
0.502	98.3	102.2	97.7	99.3	98.3	101.0	95.2	96.7
0.502	99.1	101.7	98.8	99.8	97.7	101.1	94.5	99.2
1.012	99.5	103.2	99.7	101.6	99.3	102.2	96.3	101.5
1.012	100.1	103.4	98.0	101.5	99.0	102.4	96.8	100.7
4.957	100.1	103.4	99.6	100.6	99.6	101.8	96.7	101.2
4.957	100.5	103.8	99.1	100.5	98.6	102.9	98.1	101.3
AVG	99.69	102.69	98.45	99.99	98.41	102.31	95.92	100.33
STDEV	0.77	0.78	1.29	1.05	1.39	1.05	1.06	1.45

Data were obtained from Table 10, pp. 61-63 of the study report.

Table 6a Adsorption supernatant (ug a.i./mL)

Rate (ug/mL)	M546	M549	M568	M579	M584	M599	M616	M617
0.048	0.042	0.044	0.037	0.043	0.044	0.046	0.029	0.041
0.048	0.042	-	0.037	0.043	0.043	0.045	0.028	0.042
AVG	0.042	0.044	0.037	0.043	0.044	0.046	0.029	0.042
STDEV	0.000		0.000	0.000	0.001	0.001	0.001	0.001
0.097	0.085	0.089	0.076	0.086	0.089	0.091	0.058	0.086
0.097	0.085	0.088	0.076	0.086	0.089	0.089	0.059	0.086
AVG	0.085	0.089	0.076	0.086	0.089	0.090	0.059	0.086
STDEV	0.000	0.001	0.000	0.000	0.000	0.001	0.001	0.000
0.502	0.436	0.463	0.403	0.451	0.46	0.467	0.314	0.449
0.502	0.437	0.465	0.402	0.455	0.463	0.467	0.315	0.448
AVG	0.437	0.464	0.403	0.453	0.462	0.467	0.315	0.449
STDEV	0.001	0.001	0.001	0.003	0.002	0.000	0.001	0.001
1.012	0.897	0.949	0.827	0.933	0.928	0.967	0.659	0.918
1.012	0.897	0.949	0.829	0.937	0.936	0.976	0.662	0.932
AVG	0.897	0.949	0.828	0.935	0.932	0.972	0.661	0.925
STDEV	0.000	0.000	0.001	0.003	0.006	0.006	0.002	0.010
4.957	4.423	4.688	4.162	4.583	4.660	4.707	3.410	4.561
4.957	4.445	4.685	4.164	4.566	4.660	4.760	3.397	4.582
AVG	4.434	4.687	4.163	4.575	4.660	4.734	3.404	4.572
STDEV	0.016	0.002	0.001	0.012	0.000	0.037	0.009	0.015

Data were obtained from Table 11, pp. 64-67 of the study report.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

Table 6a % Adsorbed

Rate (ug/mL)	M546	M549	M568	M579	M584	M599	M616	M617
0.048	3	2.1	15.7	4.3	8.1	0.3	29.6	9.7
0.048	2.9	-	15	4.5	6.5	0	30.7	8.9
AVG	2.95	2.10	15.35	4.40	7.30	0.15	30.15	9.30
STDEV	0.07		0.49	0.14	1.13	0.21	0.78	0.57
0.097	3	2.1	14.1	5.2	3.1	0.5	30.6	7.7
0.097	1.9	2.9	14.2	4.6	3.3	1.8	29.9	7.9
AVG	2.45	2.50	14.15	4.90	3.20	1.15	30.25	7.80
STDEV	0.78	0.57	0.07	0.42	0.14	0.92	0.49	0.14
0.502	3.7	1.7	12.6	4.2	3.8	1.5	27.5	10.4
0.502	3.1	2.3	12.4	3.4	4.9	1.5	28	7.2
AVG	3.40	2.00	12.50	3.80	4.35	1.50	27.75	8.80
STDEV	0.42	0.42	0.14	0.57	0.78	0.00	0.35	2.26
1.012	1.9	0.2	10.6	1.5	3.6	0	24.4	5.9
1.012	1.5	0.3	10.7	1.3	3	0	24.9	5.5
AVG	1.70	0.25	10.65	1.40	3.30	0.00	24.65	5.70
STDEV	0.28	0.07	0.07	0.14	0.42	0.00	0.35	0.28
4.957	1.4	0	8.4	1.5	1.7	0	21.1	4.7
4.957	0.8	0	8.7	1.9	2.8	0	20.4	4.2
AVG	1.10	0.00	8.55	1.70	2.25	0.00	20.75	4.45
STDEV	0.42	0.00	0.21	0.28	0.78	0.00	0.49	0.35
AVG (all)	2.32	1.29	12.24	3.24	4.08	0.56	26.71	7.21
STDEV (all)	1.13	1.16	4.42	1.75	2.19	0.72	8.83	2.95

Data were obtained from Table 11, pp. 64-67 of the study report.

Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

Table 7a Desorption soil (ug a.i./g)

Rate (ug/mL)	M546	M549	M568	M579	M584	M599	M616	M617
0.048	0.002	0.002	0.008	0.002	0.003	0.004	0.015	0.006
0.048	0.002	-	0.006	0.002	0.003	0.003	0.016	0.006
AVG	0.002	0.002	0.007	0.002	0.003	0.004	0.016	0.006
STDEV	0.000		0.001	0.000	0.000	0.001	0.001	0.000
0.097	0.003	0.004	0.018	0.005	0.005	0.006	0.031	0.008
0.097	0.004	0.005	0.011	0.003	0.005	0.006	0.03	0.01
AVG	0.004	0.005	0.015	0.004	0.005	0.006	0.031	0.009
STDEV	0.001	0.001	0.005	0.001	0.000	0.000	0.001	0.001
0.502	0.01	0.019	0.06	0.019	0.021	0.025	0.137	0.043
0.502	0.011	0.023	0.069	0.018	0.022	0.026	0.14	0.04
AVG	0.011	0.021	0.065	0.019	0.022	0.026	0.139	0.042
STDEV	0.001	0.003	0.006	0.001	0.001	0.001	0.002	0.002
1.012	0.003	0.028	0.128	0.032	0.048	0.033	0.248	0.082
1.012	0.02	0.023	0.09	0.027	0.037	0.041	0.258	0.067
AVG	0.012	0.026	0.109	0.030	0.043	0.037	0.253	0.075
STDEV	0.012	0.004	0.027	0.004	0.008	0.006	0.007	0.011
4.957	0.094	0.098	0.483	0.105	0.135	0.095	1.049	0.325
4.957	0.088	0.113	0.471	0.114	0.125	0.175	1.062	0.305
AVG	0.091	0.106	0.477	0.110	0.130	0.135	1.056	0.315
STDEV	0.004	0.011	0.008	0.006	0.007	0.057	0.009	0.014

Data were obtained from Table 12, pp. 68-71 of the study report.

Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

Table 7a Desorption supernatant (ug a.l./mL)

Rate (ug/mL)	M546	M549	M568	M579	M584	M599	M616	M617
0.048	0.0004	0	0.0025	0.001	0.0002	0	0.0038	0.0019
0.048	0.0002	-	0.0023	0.0007	0	0	0.004	0.0019
AVG	0.0003	0	0.0024	0.0009	0.0001	0	0.0039	0.0019
STDEV	0.0001		0.0001	0.0002	0.0001	0	0.0001	0.0000
0.097	0.0005	0	0.0044	0.0019	0	0	0.008	0.003
0.097	0.0006	0	0.0046	0.0011	0.0003	0	0.0081	0.0034
AVG	0.0006	0	0.0045	0.0015	0.0002	0	0.0081	0.0032
STDEV	0.0001	0	0.0001	0.0006	0.0002	0	0.0001	0.0003
0.502	0.0029	0	0.0196	0.0071	0	0	0.0396	0.014
0.502	0.0032	0	0.0196	0.0067	0.0014	0	0.0376	0.0114
AVG	0.0031	0	0.0196	0.0069	0.0007	0	0.0386	0.0127
STDEV	0.0002	0	0.0000	0.0003	0.0010	0	0.0014	0.0018
1.012	0.0024	0	0.0369	0.0146	0.0057	0	0.0738	0.0325
1.012	0.0049	0	0.0398	0.0132	0.0023	0	0.0775	0.0285
AVG	0.0037	0	0.0384	0.0139	0.0040	0	0.0757	0.0305
STDEV	0.0018	0	0.0021	0.0010	0.0024	0	0.0026	0.0028
4.957	0.0236	0	0.1521	0.052	0	0	0.3351	0.1405
4.957	0.0136	0	0.1546	0.064	0.0083	0	0.3566	0.1256
AVG	0.0186	0	0.1534	0.0580	0.0042	0	0.3459	0.1331
STDEV	0.0071	0	0.0018	0.0085	0.0059	0	0.0152	0.0105

Data were obtained from Table 12, pp. 68-71 of the study report.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

Table 7a Percent desorbed as % of the adsorbed

Rate (ug/mL)	M546	M549	M568	M579	M584	M599	M616	M617
0.048	30.00	0.00	36.31	51.16	6.17	0.00	30.74	43.30
0.048	13.79	-	34.67	35.56	0.00	0.00	30.94	44.94
AVG	21.90	0.00	35.49	43.36	3.09	0.00	30.84	44.12
STDEV	11.46		1.16	11.04	4.36	0.00	0.14	1.16
0.097	20.00	0.00	35.46	40.38	0.00	0.00	31.05	41.56
0.097	36.84	0.00	36.62	26.09	9.09	0.00	32.44	45.57
AVG	28.42	0.00	36.04	33.24	4.55	0.00	31.74	43.56
STDEV	11.91	0.00	0.82	10.11	6.43	0.00	0.99	2.84
0.502	16.22	0.00	34.13	35.71	0.00	0.00	33.09	26.92
0.502	22.58	0.00	34.68	41.18	6.12	0.00	30.71	31.94
AVG	19.40	0.00	34.40	38.45	3.06	0.00	31.90	29.43
STDEV	4.50	0.00	0.39	3.86	4.33	0.00	1.68	3.55
1.012	15.79	0.00	37.74	100.00	16.67	0.00	34.43	55.93
1.012	33.33	0.00	40.19	107.69	6.67	0.00	35.74	52.73
AVG	24.56	0.00	38.96	103.85	11.67	0.00	35.08	54.33
STDEV	12.41	0.00	1.73	5.44	7.07	0.00	0.93	2.27
4.957	35.71	0.00	41.25	52.38	0.00	0.00	42.13	47.46
4.957	37.50	0.00	42.31	54.17	14.29	0.00	43.78	45.45
AVG	36.61	0.00	41.78	53.27	7.14	0.00	42.96	46.46
STDEV	1.26	0.00	0.75	1.26	10.10	0.00	1.17	1.42
AVG (all)	26.18	0.00	37.33	54.43	5.90	0.00	34.51	43.58
STDEV (all)	11.97	0.00	11.60	30.83	6.07	0.00	11.35	15.51

Reviewer-calculated using data obtained from Table 10, pp. 61-63 of the study report.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

Table 7a Percent desorbed as % of the adsorbed

Rate (ug/mL)	M546	M549	M568	M579	M584	M599	M616	M617
0.048	29.90	0.00	36.60	51.70	6.30	0.00	30.90	43.20
0.048	15.10	0.00	34.50	36.50	0.50	0.00	31.00	45.50
AVG	22.50	0.00	35.55	44.10	3.40	0.00	30.95	44.35
STDEV	10.47	0.00	1.48	10.75	4.10	0.00	0.07	1.63
0.097	19.80	0.00	35.50	39.70	-	0.00	31.10	40.70
0.097	35.40	0.00	36.70	26.40	8.60	0.00	32.50	46.30
AVG	27.60	0.00	36.10	33.05	8.60	0.00	31.80	43.50
STDEV	11.03	0.00	0.85	9.40		0.00	0.99	3.96
0.502	17.20	0.00	33.90	35.40	-	0.00	33.10	27.30
0.502	22.50	0.00	34.50	42.00	5.90	0.00	30.80	31.90
AVG	19.85	0.00	34.20	38.70	5.90	0.00	31.95	29.60
STDEV	3.75	0.00	0.42	4.67		0.00	1.63	3.25
1.012	13.80	0.00	37.40	101.10	16.10	0.00	34.50	55.80
1.012	36.50	0.00	40.20	110.70	8.00	0.00	35.60	52.80
AVG	25.15	0.00	38.80	105.90	12.05	0.00	35.05	54.30
STDEV	16.05	0.00	1.98	6.79	5.73	0.00	0.78	2.12
4.957	37.70	0.00	38.80	71.80	-	0.00	35.70	59.90
4.957	35.40	0.00	38.10	71.00	6.00	0.00	39.80	59.50
AVG	36.55	0.00	38.45	71.40	6.00	0.00	37.75	59.70
STDEV	1.63	0.00	0.49	0.57		0.00	2.90	0.28
AVG (all)	26.33	0.00	36.62	58.63	7.34	0.00	33.50	46.29
STDEV (all)	12.10	0.00	11.21	32.74	5.04	0.00	10.48	17.46

Data were obtained from Table 12, pp. 68-71 of the study report.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

Table 8a	Adsorption K_f (unitless)	Table 8a	Adsorption K_d (mL/g)	Table 8a	Adsorption K_{oc} (mL/g)
M546	0.04	M546	0.04	M546	4.49
M549	0.01	M549	0.03	M549	1.05
M568	0.26	M568	0.29	M568	7.39
M579	0.05	M579	0.07	M579	4.59
M584	0.07	M584	0.08	M584	7.54
M599	0.04	M599	0.03	M599	2.33
M616	0.73	M616	0.72	M616	19.95
M617	0.13	M617	0.15	M617	24.30
AVG	0.17	AVG	0.18	AVG	8.96
STDEV	0.24	STDEV	0.24	STDEV	8.50

Reviewer-calculated from data obtained in Tables 1-2, 11 and Appendix D, pp. 50-54, 64-67, 113-115 of the study report.

Table 8a	Desorption K_f (unitless)	Table 8a	Desorption K_d (mL/g)	Table 8a	Desorption K_{oc} (mL/g)
M546	1.97	M546	5.98	M546	598.44
M549	-	M549	-	M549	-
M568	3.09	M568	3.59	M568	92.16
M579	1.72	M579	2.60	M579	162.27
M584	1.24	M584	19.14	M584	1914.50
M599	-	M599	-	M599	-
M616	2.88	M616	3.80	M616	105.66
M617	2.12	M617	2.18	M617	362.78
AVG	2.17	AVG	6.22	AVG	539.30
STDEV	0.70	STDEV	6.47	STDEV	700.85

Reviewer-calculated from data obtained in Tables 1-2, 12 and Appendix D, pp. 50-54, 68-71, 113-115 of the study report.

Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

Table 5b Adsorption supernatant at 5 °C (% applied)

Rate (ug/mL)	M568	M599
0.502	87.8	98.4
0.502	88	98.9
AVG	87.90	98.65
STDEV	0.14	0.35

Table 5b Desorption supernatant at 5 °C (% applied)

Rate (ug/mL)	M568	M599
0.502	5	0
0.502	5.2	0
AVG	5.10	0.00
STDEV	0.14	0.00

Table 5b Extraction at 5 °C (% applied)

Rate (ug/mL)	M568	M599
0.502	1.6	0.4
0.502	1.5	0.6
AVG	1.55	0.50
STDEV	0.07	0.14

Table 5b Combustion at 5 °C (% applied)

Rate (ug/mL)	M568	M599
0.502	4.3	0.2
0.502	4.1	0.2
AVG	4.20	0.20
STDEV	0.14	0.00

Table 5b Recovery at 5 °C (% applied)

Rate (ug/mL)	M568	M599
0.502	98.7	99
0.502	98.8	99.7
AVG	98.75	99.35
STDEV	0.07	0.49

Data were obtained from Table 14, p. 73 of the study report.

Table 6b Adsorption soil at 5 °C (ug a.i./g)

Rate (ug/mL)	M568	M599
0.502	0.122	0.016
0.502	0.12	0.011
AVG	0.12	0.01
STDEV	0.00	0.00

Table 6b Adsorption supernatant at 5 °C (ug/mL)

Rate (ug/mL)	M568	M599
0.502	0.407	0.464
0.502	0.406	0.469
AVG	0.41	0.47
STDEV	0.00	0.00

Table 6b % Adsorbed at 5 °C

Rate (ug/mL)	M568	M599
0.502	12.2	1.6
0.502	12	1.1
AVG	12.10	1.35
STDEV	0.14	0.35

Table 7b Desorption soil at 5 °C (ug a.i./g)

Rate (ug/mL)	M568	M599
0.502	0.059	0.025
0.502	0.056	0.026
AVG	0.06	0.03
STDEV	0.00	0.00

Table 7b Desorption supernatant at 5 °C (ug/mL)

Rate (ug/mL)	M568	M599
0.502	0.023	0
0.502	0.024	0
AVG	0.02	0.00
STDEV	0.00	0.00

Table 7b Percent desorbed as % of adsorbed at 5 °C

Rate (ug/mL)	M568	M599
0.502	41.2	-
0.502	43.2	-
AVG	42.20	-
STDEV	1.41	-

Data were obtained from Table 15, p. 74 of the study report.

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Table 5c Adsorption supernatant; metabolism soils (% applied)

Rate (ug/mL)	M610	M611	M615	M623
0.507	61.4	59.2	72.2	58.3
0.507	61.5	60	71.8	58
AVG	61.45	59.60	72.00	58.15
STDEV	0.07	0.57	0.28	0.21

Table 5c Extraction; metabolism soils (% applied)

M610	M611	M615	M623
32.8	32.5	24.4	25.1
32.6	32.7	24.4	25
32.70	32.60	24.40	25.05
0.14	0.14	0.00	0.07

Table 5c Combustion; metabolism soils (% applied)

Rate (ug/mL)	M610	M611	M615	M623
0.507	1.4	2.3	1.3	12.4
0.507	1.5	2.4	1.3	12.6
AVG	1.45	2.35	1.30	12.50
STDEV	0.07	0.07	0.00	0.14

Table 5c Recovery; metabolism soils (% applied)

M610	M611	M615	M623
95.5	94.1	97.9	95.8
95.5	95.1	97.6	95.5
95.50	94.60	97.75	95.65
0.00	0.71	0.21	0.21

Data were obtained from Table 17, p. 76 of the study report.

Table 6c Adsorption soil; metabolism soils (ug a.i./g)

Rate (ug/mL)	M610	M611	M615	M623
0.507	0.17	0.16	0.03	0.09
0.507	0.17	0.15	0.03	0.09
AVG	0.17	0.16	0.03	0.09
STDEV	0.00	0.01	0.00	0.00

Table 6c Adsorption supernatant; metabolism soils (ug/mL)

M610	M611	M615	M623
0.407	0.401	0.479	0.441
0.406	0.4	0.478	0.438
0.41	0.40	0.48	0.44
0.00	0.00	0.00	0.00

Table 6c % Adsorbed; metabolism soils

Rate (ug/mL)	M610	M611	M615	M623
0.507	3.3	3.2	0.6	1.8
0.507	3.3	3	0.6	1.8
AVG	3.30	3.10	0.60	1.80
STDEV	0.00	0.14	0.00	0.00

Table 6c % Adsorbed; metabolism soils

M610	M611	M615	M623
38.6	40.8	27.8	41.7
38.5	40	28.2	42
38.55	40.40	28.00	41.85
0.07	0.57	0.28	0.21

Data were obtained from Table 18, p. 77 of the study report.

Reviewer-calculated from Table 17, p. 76 of the study report.

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Chemical: Aminopyralid
PC Code: 005100
MRID: 46235732
Guideline No: 163-1

Table 8b	Adsorption K_d at 5 °C	Table 8b	Desorption K_d at 5 °C
M568	0.47	M568	6.13
M599	0.15	M599	-
AVG	0.31	AVG	6.13
STDEV	0.23	STDEV	-

Table 8b	Adsorption K_{OC} at 5 °C	Table 8b	Desorption K_{OC} at 5 °C
M568	12.05	M568	157.19
M599	10.15	M599	-
AVG	11.10	AVG	157.19
STDEV	1.34	STDEV	-

Reviewer-calculated from data obtained in Tables 15-16, p. 74-75 of the study report.

Table 8c	Adsorption K_d ; metabolism soils	Table 8c	Adsorption K_{OC} ; metabolism soils
M610	0.49	M610	32.96
M611	0.53	M611	15.64
M615	0.12	M615	9.93
M623	0.31	M623	9.04
AVG	0.36	AVG	16.89
STDEV	0.19	STDEV	11.11

Reviewer-calculated from data obtained in Tables 18-19, p. 77-78 of the study report.

Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

M546- Adsorption

Labelled initial soln concn (C _o) (ug/mL)	Actual initial soln concn (C _o) (ug/mL)	Volume of soln (V _o) (mL)	Concen in soln after equil (C _{eq}) (ug/mL)	Dry mass of sorbent (M) (g)	[(C _o V _o)-(C _{eq} V _o)]/M (ug/g)	Kd (mL/g)	
0.048	0.043	10	0.042	5	0.0020	0.05	
0.048	0.043	10	0.042	5	0.0020	0.05	
0.097	0.087	10	0.085	5	0.0040	0.05	
0.097	0.087	10	0.085	5	0.0040	0.05	
0.502	0.453	10	0.436	5	0.0340	0.08	
0.502	0.453	10	0.437	5	0.0320	0.07	
1.012	0.912	10	0.897	5	0.0300	0.03	
1.012	0.912	10	0.897	5	0.0300	0.03	
4.957	4.480	10	4.423	5	0.1140	0.03	
4.957	4.479	10	4.445	5	0.0680	0.02	
						0.04	AVG
						0.02	STDEV

M549- Adsorption

Labelled initial soln concn (C _o) (ug/mL)	Actual initial soln concn (C _o) (ug/mL)	Volume of soln (V _o) (mL)	Concen in soln after equil (C _{eq}) (ug/mL)	Dry mass of sorbent (M) (g)	[(C _o V _o)-(C _{eq} V _o)]/M (ug/g)	Kd (mL/g)	
0.048	0.045	10	0.044	5	0.0020	0.05	
0.048	0.045	10	-	5	-	-	
0.097	0.091	10	0.089	5	0.0040	0.04	
0.097	0.091	10	0.088	5	0.0060	0.07	
0.502	0.472	10	0.463	5	0.0180	0.04	
0.502	0.472	10	0.465	5	0.0140	0.03	
1.012	0.951	10	0.949	5	0.0040	0.00	
1.012	0.951	10	0.949	5	0.0040	0.00	
4.957	4.666	10	4.688	5	-0.0440	-	
4.957	4.664	10	4.685	5	-0.0420	-	
						0.03	AVG
						0.02	STDEV

M568- Adsorption

Labelled initial soln concn (C _o) (ug/mL)	Actual initial soln concn (C _o) (ug/mL)	Volume of soln (V _o) (mL)	Concen in soln after equil (C _{eq}) (ug/mL)	Dry mass of sorbent (M) (g)	[(C _o V _o)-(C _{eq} V _o)]/M (ug/g)	Kd (mL/g)	
0.048	0.044	10	0.037	5	0.0140	0.38	
0.048	0.044	10	0.037	5	0.0140	0.38	
0.097	0.089	10	0.076	5	0.0260	0.34	
0.097	0.089	10	0.076	5	0.0260	0.34	
0.502	0.461	10	0.403	5	0.1160	0.29	
0.502	0.461	10	0.402	5	0.1180	0.29	
1.012	0.930	10	0.827	5	0.2060	0.25	
1.012	0.923	10	0.829	5	0.1880	0.23	
4.957	4.562	10	4.162	5	0.8000	0.19	
4.957	4.565	10	4.164	5	0.8020	0.19	
						0.29	AVG
						0.07	STDEV

Reviewer-calculated from data obtained in Table 11 and Appendix D, pp. 64-67, 113-115 of the study report.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

M579- Adsorption

Labelled initial soln concn (C _o) (ug/mL)	Actual initial soln concn (C _o) (ug/mL)	Volume of soln (V _o) (mL)	Concen in soln after equil (C _{eq}) (ug/mL)	Dry mass of sorbent (M) (g)	[(C _o V _o)-(C _{eq} V _o)]/M (ug/g)	Kd (mL/g)	
0.048	0.045	10	0.043	5	0.0040	0.09	
0.048	0.045	10	0.043	5	0.0040	0.09	
0.097	0.091	10	0.086	5	0.0100	0.12	
0.097	0.091	10	0.086	5	0.0100	0.12	
0.502	0.472	10	0.451	5	0.0420	0.09	
0.502	0.472	10	0.455	5	0.0340	0.07	
1.012	0.951	10	0.933	5	0.0360	0.04	
1.012	0.951	10	0.937	5	0.0280	0.03	
4.957	4.666	10	4.583	5	0.1660	0.04	
4.957	4.666	10	4.566	5	0.2000	0.04	
						0.07	AVG
						0.03	STDEV

M584- Adsorption

Labelled initial soln concn (C _o) (ug/mL)	Actual initial soln concn (C _o) (ug/mL)	Volume of soln (V _o) (mL)	Concen in soln after equil (C _{eq}) (ug/mL)	Dry mass of sorbent (M) (g)	[(C _o V _o)-(C _{eq} V _o)]/M (ug/g)	Kd (mL/g)	
0.048	0.046	10	0.044	5	0.0040	0.09	
0.048	0.046	10	0.043	5	0.0060	0.14	
0.097	0.092	10	0.089	5	0.0060	0.07	
0.097	0.092	10	0.089	5	0.0060	0.07	
0.502	0.480	10	0.460	5	0.0400	0.09	
0.502	0.480	10	0.463	5	0.0340	0.07	
1.012	0.968	10	0.928	5	0.0800	0.09	
1.012	0.968	10	0.936	5	0.0640	0.07	
4.957	4.746	10	4.660	5	0.1720	0.04	
4.957	4.746	10	4.660	5	0.1720	0.04	
						0.08	AVG
						0.03	STDEV

M599- Adsorption

Labelled initial soln concn (C _o) (ug/mL)	Actual initial soln concn (C _o) (ug/mL)	Volume of soln (V _o) (mL)	Concen in soln after equil (C _{eq}) (ug/mL)	Dry mass of sorbent (M) (g)	[(C _o V _o)-(C _{eq} V _o)]/M (ug/g)	Kd (mL/g)	
0.048	0.045	10	0.046	5	-0.0020	-	
0.048	0.045	10	0.045	5	0.0000	-	
0.097	0.091	10	0.091	5	0.0000	-	
0.097	0.091	10	0.089	5	0.0040	0.04	
0.502	0.474	10	0.467	5	0.0140	0.03	
0.502	0.474	10	0.467	5	0.0140	0.03	
1.012	0.956	10	0.967	5	-0.0220	-	
1.012	0.956	10	0.976	5	-0.0400	-	
4.957	4.687	10	4.707	5	-0.0400	-	
4.957	4.687	10	4.760	5	-0.1460	-	
						0.03	AVG
						0.01	STDEV

Reviewer-calculated from data obtained in Table 11 and Appendix D, pp. 64-67, 113-115 of the study report.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

M616- Adsorption

Labelled initial soln concn (C _o) (ug/mL)	Actual initial soln concn (C _o) (ug/mL)	Volume of soln (V _o) (mL)	Concen in soln after equil (C _{eq}) (ug/mL)	Dry mass of sorbent (M) (g)	[(C _o V _o)-(C _{eq} V _o)]/M (ug/g)	Kd (mL/g)	
0.048	0.041	10	0.029	5	0.0240	0.83	
0.048	0.041	10	0.028	5	0.0260	0.93	
0.097	0.083	10	0.058	5	0.0500	0.86	
0.097	0.083	10	0.059	5	0.0480	0.81	
0.502	0.431	10	0.314	5	0.2340	0.75	
0.502	0.431	10	0.315	5	0.2320	0.74	
1.012	0.868	10	0.659	5	0.4180	0.63	
1.012	0.867	10	0.662	5	0.4100	0.62	
4.957	4.270	10	3.41	5	1.7200	0.50	
4.957	4.267	10	3.397	5	1.7400	0.51	
						0.72	AVG
						0.15	STDEV

M617- Adsorption

Labelled initial soln concn (C _o) (ug/mL)	Actual initial soln concn (C _o) (ug/mL)	Volume of soln (V _o) (mL)	Concen in soln after equil (C _{eq}) (ug/mL)	Dry mass of sorbent (M) (g)	[(C _o V _o)-(C _{eq} V _o)]/M (ug/g)	Kd (mL/g)	
0.048	0.046	10	0.041	5	0.0100	0.24	
0.048	0.046	10	0.042	5	0.0080	0.19	
0.097	0.093	10	0.086	5	0.0140	0.16	
0.097	0.093	10	0.086	5	0.0140	0.16	
0.502	0.483	10	0.449	5	0.0680	0.15	
0.502	0.483	10	0.448	5	0.0700	0.16	
1.012	0.974	10	0.918	5	0.1120	0.12	
1.012	0.974	10	0.932	5	0.0840	0.09	
4.957	4.776	10	4.561	5	0.4300	0.09	
4.957	4.774	10	4.582	5	0.3840	0.08	
						0.15	AVG
						0.05	STDEV

Reviewer-calculated from data obtained in Table 11 and Appendix D, pp. 64-67, 113-115 of the study report.

M568- Adsorption at 5 °C

Labelled initial soln concn (C _o) (ug/mL)	Actual initial soln concn (C _o) (ug/mL)	Volume of soln (V _o) (mL)	Concen in soln after equil (C _{eq}) (ug/mL)	Dry mass of sorbent (M) (g)	[(C _o V _o)-(C _{eq} V _o)]/M (ug/g)	Kd (mL/g)	
0.502	not reported	10	0.407	5	0.1900	0.47	
0.502	not reported	10	0.406	5	0.1920	0.47	
						0.47	AVG
						0.00	STDEV

Reviewer-calculated from data obtained in Table 15, p. 74 of the study report.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

M599- Adsorption at 5 °C

Labelled initial soln concn (C _o) (ug/mL)	Actual initial soln concn (C _o) (ug/mL)	Volume of soln (V _o) (mL)	Concen in soln after equil (C _{eq}) (ug/mL)	Dry mass of sorbent (M) (g)	$[(C_oV_o) - (C_{eq}V_o)]/M$ (ug/g)	Kd (mL/g)	
0.502	not reported	10	0.464	5	0.0760	0.16	
0.502	not reported	10	0.469	5	0.0660	0.14	
						0.15	AVG
						0.02	STDEV

Reviewer-calculated from data obtained in Table 15, p. 74 of the study report.

M610- Adsorption

Labelled initial soln concn (C _o) (ug/mL)	Actual initial soln concn (C _o) (ug/mL)	Volume of soln (V _o) (mL)	Concen in soln after equil (C _{eq}) (ug/mL)	Dry mass of sorbent (M) (g)	$[(C_oV_o) - (C_{eq}V_o)]/M$ (ug/g)	Kd (mL/g)	
0.507	not reported	10	0.407	5	0.2000	0.49	
0.507	not reported	10	0.406	5	0.2020	0.50	
						0.49	AVG
						0.00	STDEV

M611- Adsorption

Labelled initial soln concn (C _o) (ug/mL)	Actual initial soln concn (C _o) (ug/mL)	Volume of soln (V _o) (mL)	Concen in soln after equil (C _{eq}) (ug/mL)	Dry mass of sorbent (M) (g)	$[(C_oV_o) - (C_{eq}V_o)]/M$ (ug/g)	Kd (mL/g)	
0.507	not reported	10	0.401	5	0.2120	0.53	
0.507	not reported	10	0.4	5	0.2140	0.54	
						0.53	AVG
						0.00	STDEV

M615- Adsorption

Labelled initial soln concn (C _o) (ug/mL)	Actual initial soln concn (C _o) (ug/mL)	Volume of soln (V _o) (mL)	Concen in soln after equil (C _{eq}) (ug/mL)	Dry mass of sorbent (M) (g)	$[(C_oV_o) - (C_{eq}V_o)]/M$ (ug/g)	Kd (mL/g)	
0.507	not reported	10	0.479	5	0.0560	0.12	
0.507	not reported	10	0.478	5	0.0580	0.12	
						0.12	AVG
						0.00	STDEV

M623- Adsorption

Labelled initial soln concn (C _o) (ug/mL)	Actual initial soln concn (C _o) (ug/mL)	Volume of soln (V _o) (mL)	Concen in soln after equil (C _{eq}) (ug/mL)	Dry mass of sorbent (M) (g)	$[(C_oV_o) - (C_{eq}V_o)]/M$ (ug/g)	Kd (mL/g)	
0.507	not reported	10	0.441	5	0.1320	0.30	
0.507	not reported	10	0.438	5	0.1380	0.32	
						0.31	AVG
						0.01	STDEV

Reviewer-calculated from data obtained in Table 18, p. 77 of the study report.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

M546- Desorption

Labeled Rate (ug/mL)	Initial soil concn (C _o) (ug/g)	Dry mass of sorbent (M) (g)	Concn in soln after equil (C _{eq}) (ug/mL)	Volume of soln (V) (mL)	[(C _o M)- (C _{eq} V)]/M (ug/g)	Kd (mL/g)	
0.048	0.002	5	0.0004	10	0.0012	3.00	
0.048	0.002	5	0.0002	10	0.0016	8.00	
0.097	0.004	5	0.0005	10	0.0030	6.00	
0.097	0.004	5	0.0006	10	0.0028	4.67	
0.502	0.034	5	0.0029	10	0.0282	9.72	
0.502	0.032	5	0.0032	10	0.0256	8.00	
1.012	0.030	5	0.0024	10	0.0252	10.50	
1.012	0.030	5	0.0049	10	0.0202	4.12	
4.957	0.114	5	0.0236	10	0.0668	2.83	
4.957	0.068	5	0.0136	10	0.0408	3.00	
						5.98	AVG
						2.90	STDEV

M549- Desorption

Labeled Rate (ug/mL)	Initial soil concn (C _o) (ug/g)	Dry mass of sorbent (M) (g)	Concn in soln after equil (C _{eq}) (ug/mL)	Volume of soln (V) (mL)	[(C _o M)- (C _{eq} V)]/M (ug/g)	Kd (mL/g)	
0.048	0.0020	5	0	10	0.0020	0.00	
0.048	-	5	-	10	-	-	
0.097	0.0040	5	0	10	0.0040	0.00	
0.097	0.0060	5	0	10	0.0060	0.00	
0.502	0.0180	5	0	10	0.0180	0.00	
0.502	0.0140	5	0	10	0.0140	0.00	
1.012	0.0040	5	0	10	0.0040	0.00	
1.012	0.0040	5	0	10	0.0040	0.00	
4.957	-	5	0	10	-	-	
4.957	-	5	0	10	-	-	
						0.00	AVG
						0.00	STDEV

M568- Desorption

Labeled Rate (ug/mL)	Initial soil concn (C _o) (ug/g)	Dry mass of sorbent (M) (g)	Concn in soln after equil (C _{eq}) (ug/mL)	Volume of soln (V) (mL)	[(C _o M)- (C _{eq} V)]/M (ug/g)	Kd (mL/g)	
0.048	0.0140	5	0.0025	10	0.0090	3.60	
0.048	0.0140	5	0.0023	10	0.0094	4.09	
0.097	0.0260	5	0.0044	10	0.0172	3.91	
0.097	0.0260	5	0.0046	10	0.0168	3.65	
0.502	0.1160	5	0.0196	10	0.0768	3.92	
0.502	0.1180	5	0.0196	10	0.0788	4.02	
1.012	0.2060	5	0.0369	10	0.1322	3.58	
1.012	0.1880	5	0.0398	10	0.1084	2.72	
4.957	0.8000	5	0.1521	10	0.4958	3.26	
4.957	0.8020	5	0.1546	10	0.4928	3.19	
						3.59	AVG
						0.43	STDEV

Reviewer-calculated from data obtained in Table 11, 12 and Appendix D, pp. 64-71, 113-115 of the study report.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

M579- Desorption

Labeled Rate (ug/mL)	Initial soil concn (C ₀) (ug/g)	Dry mass of sorbent (M) (g)	Concen in soln after equil (C _{eq}) (ug/mL)	Volume of soln (V) (mL)	[(C ₀ M)-(C _{eq} V)]/M (ug/g)	Kd (mL/g)	
0.048	0.0040	5	0.001	10	0.0020	2.00	
0.048	0.0040	5	0.0007	10	0.0026	3.71	
0.097	0.0100	5	0.0019	10	0.0062	3.26	
0.097	0.0100	5	0.0011	10	0.0078	7.09	
0.502	0.0420	5	0.0071	10	0.0278	3.92	
0.502	0.0340	5	0.0067	10	0.0206	3.07	
1.012	0.0360	5	0.0146	10	0.0068	0.47	
1.012	0.0280	5	0.0132	10	0.0016	0.12	
4.957	0.1660	5	0.052	10	0.0620	1.19	
4.957	0.2000	5	0.064	10	0.0720	1.13	
						2.60	AVG
						2.08	STDEV

M584- Desorption

Labeled Rate (ug/mL)	Initial soil concn (C ₀) (ug/g)	Dry mass of sorbent (M) (g)	Concen in soln after equil (C _{eq}) (ug/mL)	Volume of soln (V) (mL)	[(C ₀ M)-(C _{eq} V)]/M (ug/g)	Kd (mL/g)	
0.048	0.0040	5	0.0002	10	0.0036	18.00	
0.048	0.0060	5	0	10	0.0060	-	
0.097	0.0060	5	0	10	0.0060	-	
0.097	0.0060	5	0.0003	10	0.0054	18.00	
0.502	0.0400	5	0	10	0.0400	-	
0.502	0.0340	5	0.0014	10	0.0312	22.29	
1.012	0.0800	5	0.0057	10	0.0686	12.04	
1.012	0.0640	5	0.0023	10	0.0594	25.83	
4.957	0.1720	5	0	10	0.1720	-	
4.957	0.1720	5	0.0083	10	0.1554	18.72	
						19.14	AVG
						4.64	STDEV

M599- Desorption

Labeled Rate (ug/mL)	Initial soil concn (C ₀) (ug/g)	Dry mass of sorbent (M) (g)	Concen in soln after equil (C _{eq}) (ug/mL)	Volume of soln (V) (mL)	[(C ₀ M)-(C _{eq} V)]/M (ug/g)	Kd (mL/g)	
0.048	-	5	0	10	-	-	
0.048	0.0000	5	0	10	-	-	
0.097	0.0000	5	0	10	-	-	
0.097	0.0040	5	0	10	-	-	
0.502	0.0140	5	0	10	-	-	
0.502	0.0140	5	0	10	-	-	
1.012	-	5	0	10	-	-	
1.012	-	5	0	10	-	-	
4.957	-	5	0	10	-	-	
4.957	-	5	0	10	-	-	
						0.00	AVG
						-	STDEV

Reviewer-calculated from data obtained in Table 11, 12 and Appendix D, pp. 64-71, 113-115 of the study report.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

M616- Desorption

Labeled Rate (ug/mL)	Initial soil concn (C _o) (ug/g)	Dry mass of sorbent (M) (g)	Concn in soln after equil (C _{eq}) (ug/mL)	Volume of soln (V) (mL)	[(C _o M)- (C _{eq} V)]/M (ug/g)	Kd (mL/g)	
0.048	0.0240	5	0.0038	10	0.0164	4.32	
0.048	0.0260	5	0.004	10	0.0180	4.50	
0.097	0.0500	5	0.008	10	0.0340	4.25	
0.097	0.0480	5	0.0081	10	0.0318	3.93	
0.502	0.2340	5	0.0396	10	0.1548	3.91	
0.502	0.2320	5	0.0376	10	0.1568	4.17	
1.012	0.4180	5	0.0738	10	0.2704	3.66	
1.012	0.4100	5	0.0775	10	0.2550	3.29	
4.957	1.7200	5	0.3351	10	1.0498	3.13	
4.957	1.7400	5	0.3566	10	1.0268	2.88	
						3.80	AVG
						0.55	STDEV

M617- Desorption

Labeled Rate (ug/mL)	Initial soil concn (C _o) (ug/g)	Dry mass of sorbent (M) (g)	Concn in soln after equil (C _{eq}) (ug/mL)	Volume of soln (V) (mL)	[(C _o M)- (C _{eq} V)]/M (ug/g)	Kd (mL/g)	
0.048	0.0100	5	0.0019	10	0.0062	3.26	
0.048	0.0080	5	0.0019	10	0.0042	2.21	
0.097	0.0140	5	0.003	10	0.0080	2.67	
0.097	0.0140	5	0.0034	10	0.0072	2.12	
0.502	0.0680	5	0.014	10	0.0400	2.86	
0.502	0.0700	5	0.0114	10	0.0472	4.14	
1.012	0.1120	5	0.0325	10	0.0470	1.45	
1.012	0.0840	5	0.0285	10	0.0270	0.95	
4.957	0.4300	5	0.1405	10	0.1490	1.06	
4.957	0.3840	5	0.1256	10	0.1328	1.06	
						2.18	AVG
						1.07	STDEV

Reviewer-calculated from data obtained in Table 11, 12 and Appendix D, pp. 64-71, 113-115 of the study report.

Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

M568- Desorption at 5 °C

Labeled Rate (ug/mL)	Initial soil concn (C ₀) (ug/g)	Dry mass of sorbent (M) (g)	Concn in soln after equil (C _{eq}) (ug/mL)	Volume of soln (V ₀) (mL)	[(C ₀ M)- (C _{eq} V)]/M (ug/g)	Kd (mL/g)	
0.502	0.1900	5	0.023	10	0.1440	6.26	
0.502	0.1920	5	0.024	10	0.1440	6.00	
						6.13	AVG
						0.18	STDEV

M599- Desorption at 5 °C

Labeled Rate (ug/mL)	Initial soil concn (C ₀) (ug/g)	Dry mass of sorbent (M) (g)	Concn in soln after equil (C _{eq}) (ug/mL)	Volume of soln (V ₀) (mL)	[(C ₀ M)- (C _{eq} V)]/M (ug/g)	Kd (mL/g)	
0.502	0.076	5	0	10	0.0760	-	
0.502	0.066	5	0	10	0.0660	-	
						-	AVG
						-	STDEV

Reviewer-calculated from data obtained in Table 15, p. 74 of the study report.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

Adsorption

Soil	Kd (avg)	Kd (stdev)	% o. c.	Koc (avg)	Koc (stdev)
M546	0.04	0.02	1.0	4.49	1.95
M549	0.03	0.02	3.2	1.05	0.73
M568	0.29	0.07	3.9	7.39	1.83
M579	0.07	0.03	1.6	4.59	2.10
M584	0.08	0.03	1.0	7.54	2.93
M599	0.03	0.01	1.5	2.33	0.58
M616	0.72	0.15	3.6	19.95	4.06
M617	0.15	0.05	0.6	24.30	8.33

M546- Adsorption

Initial soln concn (C ₀) (ug/mL)	Kd (mL/g)	% organic carbon	Koc (mL/g)
0.048	0.05	1.0	4.76
0.048	0.05	1.0	4.76
0.097	0.05	1.0	4.71
0.097	0.05	1.0	4.71
0.502	0.08	1.0	7.80
0.502	0.07	1.0	7.32
1.012	0.03	1.0	3.34
1.012	0.03	1.0	3.34
4.957	0.03	1.0	2.58
4.957	0.02	1.0	1.53
			4.49 AVG
			1.95 STDEV

M549- Adsorption

Initial soln concn (C ₀) (ug/mL)	Kd (mL/g)	% organic carbon	Koc (mL/g)
0.048	0.05	3.2	1.42
0.048	-	3.2	-
0.097	0.04	3.2	1.40
0.097	0.07	3.2	2.13
0.502	0.04	3.2	1.21
0.502	0.03	3.2	0.94
1.012	0.00	3.2	0.13
1.012	0.00	3.2	0.13
4.957	-	3.2	-
4.957	-	3.2	-
			1.05 AVG
			0.73 STDEV

Reviewer-calculated from data obtained in Tables 1-2, 11 and Appendix D, pp. 50-54, 64-67, 113-115 of the study report.

Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

M568- Adsorption

Initial soln concn (C ₀) (ug/mL)	Kd (mL/g)	% organic carbon	Koc (mL/g)
0.048	0.38	3.9	9.70
0.048	0.38	3.9	9.70
0.097	0.34	3.9	8.77
0.097	0.34	3.9	8.77
0.502	0.29	3.9	7.38
0.502	0.29	3.9	7.53
1.012	0.25	3.9	6.39
1.012	0.23	3.9	5.81
4.957	0.19	3.9	4.93
4.957	0.19	3.9	4.94
			7.39 AVG
			1.83 STDEV

M579- Adsorption

Initial soln concn (C ₀) (ug/mL)	Kd (mL/g)	% organic carbon	Koc (mL/g)
0.048	0.09	1.6	5.81
0.048	0.09	1.6	5.81
0.097	0.12	1.6	7.27
0.097	0.12	1.6	7.27
0.502	0.09	1.6	5.82
0.502	0.07	1.6	4.67
1.012	0.04	1.6	2.41
1.012	0.03	1.6	1.87
4.957	0.04	1.6	2.26
4.957	0.04	1.6	2.74
			4.59 AVG
			2.10 STDEV

M584- Adsorption

Initial soln concn (C ₀) (ug/mL)	Kd (mL/g)	% organic carbon	Koc (mL/g)
0.048	0.09	1.0	9.09
0.048	0.14	1.0	13.95
0.097	0.07	1.0	6.74
0.097	0.07	1.0	6.74
0.502	0.09	1.0	8.70
0.502	0.07	1.0	7.34
1.012	0.09	1.0	8.62
1.012	0.07	1.0	6.84
4.957	0.04	1.0	3.69
4.957	0.04	1.0	3.69
			7.54 AVG
			2.93 STDEV

Reviewer-calculated from data obtained in Tables 1-2, 11 and Appendix D, pp. 50-54, 64-67, 113-115 of the study report.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

M599- Adsorption

Initial soln concen (C ₀) (ug/mL)	Kd (mL/g)	% organic carbon	Koc (mL/g)
0.048	-	1.5	-
0.048	-	1.5	-
0.097	-	1.5	-
0.097	0.04	1.5	3.00
0.502	0.03	1.5	2.00
0.502	0.03	1.5	2.00
1.012	-	1.5	-
1.012	-	1.5	-
4.957	-	1.5	-
4.957	-	1.5	-
			2.33 AVG
			0.58 STDEV

M616- Adsorption

Initial soln concen (C ₀) (ug/mL)	Kd (mL/g)	% organic carbon	Koc (mL/g)
0.048	0.83	3.6	22.99
0.048	0.93	3.6	25.79
0.097	0.86	3.6	23.95
0.097	0.81	3.6	22.60
0.502	0.75	3.6	20.70
0.502	0.74	3.6	20.46
1.012	0.63	3.6	17.62
1.012	0.62	3.6	17.20
4.957	0.50	3.6	14.01
4.957	0.51	3.6	14.23
			19.95 AVG
			4.06 STDEV

M617- Adsorption

Initial soln concen (C ₀) (ug/mL)	Kd (mL/g)	% organic carbon	Koc (mL/g)
0.048	0.24	0.6	40.65
0.048	0.19	0.6	31.75
0.097	0.16	0.6	27.13
0.097	0.16	0.6	27.13
0.502	0.15	0.6	25.24
0.502	0.16	0.6	26.04
1.012	0.12	0.6	20.33
1.012	0.09	0.6	15.02
4.957	0.09	0.6	15.71
4.957	0.08	0.6	13.97
			24.30 AVG
			8.33 STDEV

Reviewer-calculated from data obtained in Tables 1-2, 11 and Appendix D, pp. 50-54, 64-67, 113-115 of the study report.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

Adsorption

Soil	Kd (avg)	Kd (stdev)	% o. c.	Koc (avg)	Koc (stdev)
M568(5°C)	0.47	0.00	3.9	12.05	0.11
M599(5°C)	0.15	0.02	1.5	10.15	1.09
M610	0.49	0.00	1.5	32.96	0.29
M611	0.53	0.00	3.4	15.64	0.13
M615	0.12	0.00	1.2	9.93	0.26
M623	0.31	0.01	3.4	9.04	0.33

M568- Adsorption at 5 °C

Initial soln concn (C ₀) (ug/mL)	Kd (mL/g)	% organic carbon	Koc (mL/g)
0.502	0.47	3.9	11.97
0.502	0.47	3.9	12.13
			12.05 AVG
			0.11 STDEV

M599- Adsorption at 5 °C

Initial soln concn (C ₀) (ug/mL)	Kd (mL/g)	% organic carbon	Koc (mL/g)
0.502	0.16	1.5	10.92
0.502	0.14	1.5	9.38
			10.15 AVG
			1.09 STDEV

Reviewer-calculated from data obtained in Table 15, p. 74 of the study report.

Chemical: Aminopyralid
PC Code: 005100
MRID: 46235732
Guideline No: 163-1

M610- Adsorption; metabolism soils

Initial soln concen (C ₀) (ug/mL)	Kd (mL/g)	% organic carbon	Koc (mL/g)
0.502	0.49	1.5	32.76
0.502	0.50	1.5	33.17
			32.96 AVG
			0.29 STDEV

M611- Adsorption; metabolism soils

Initial soln concen (C ₀) (ug/mL)	Kd (mL/g)	% organic carbon	Koc (mL/g)
0.502	0.53	3.4	15.55
0.502	0.54	3.4	15.74
			15.64 AVG
			0.13 STDEV

M615- Adsorption; metabolism soils

Initial soln concen (C ₀) (ug/mL)	Kd (mL/g)	% organic carbon	Koc (mL/g)
0.502	0.12	1.2	9.74
0.502	0.12	1.2	10.11
			9.93 AVG
			0.26 STDEV

M623- Adsorption; metabolism soils

Initial soln concen (C ₀) (ug/mL)	Kd (mL/g)	% organic carbon	Koc (mL/g)
0.502	0.30	3.4	8.80
0.502	0.32	3.4	9.27
			9.04 AVG
			0.33 STDEV

Reviewer-calculated from data obtained in Table 18, p. 77 of the study report.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

M568- Desorption

Initial soln concn (C ₀) (ug/mL)	Kd (mL/g)	% organic carbon	Koc (mL/g)
0.048	3.60	3.9	92.31
0.048	4.09	3.9	104.79
0.097	3.91	3.9	100.23
0.097	3.65	3.9	93.65
0.502	3.92	3.9	100.47
0.502	4.02	3.9	103.09
1.012	3.58	3.9	91.86
1.012	2.72	3.9	69.84
4.957	3.26	3.9	83.58
4.957	3.19	3.9	81.73
			92.16 AVG
			11.04 STDEV

M579- Desorption

Initial soln concn (C ₀) (ug/mL)	Kd (mL/g)	% organic carbon	Koc (mL/g)
0.048	2.00	1.6	125.00
0.048	3.71	1.6	232.14
0.097	3.26	1.6	203.95
0.097	7.09	1.6	443.18
0.502	3.92	1.6	244.72
0.502	3.07	1.6	192.16
1.012	0.47	1.6	29.11
1.012	0.12	1.6	7.58
4.957	1.19	1.6	74.52
4.957	1.13	1.6	70.31
			162.27 AVG
			130.14 STDEV

M584- Desorption

Initial soln concn (C ₀) (ug/mL)	Kd (mL/g)	% organic carbon	Koc (mL/g)
0.048	18.00	1.0	1800.00
0.048	-	1.0	-
0.097	-	1.0	-
0.097	18.00	1.0	1800.00
0.502	-	1.0	-
0.502	22.29	1.0	2228.57
1.012	12.04	1.0	1203.51
1.012	25.83	1.0	2582.61
4.957	-	1.0	-
4.957	18.72	1.0	1872.29
			1914.50 AVG
			464.44 STDEV

Reviewer-calculated from data obtained in Tables 1-2, 11-12 and Appendix D, pp. 50-54, 64-71, 113-115 of the study report.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

M599- Desorption

Initial soln concn (C ₀) (ug/mL)	Kd (mL/g)	% organic carbon	Koc (mL/g)
0.048	-	1.5	-
0.048	-	1.5	-
0.097	-	1.5	-
0.097	-	1.5	-
0.502	-	1.5	-
0.502	-	1.5	-
1.012	-	1.5	-
1.012	-	1.5	-
4.957	-	1.5	-
4.957	-	1.5	-
			AVG
			STDEV

M616- Desorption

Initial soln concn (C ₀) (ug/mL)	Kd (mL/g)	% organic carbon	Koc (mL/g)
0.048	4.32	3.6	119.88
0.048	4.50	3.6	125.00
0.097	4.25	3.6	118.06
0.097	3.93	3.6	109.05
0.502	3.91	3.6	108.59
0.502	4.17	3.6	115.84
1.012	3.66	3.6	101.78
1.012	3.29	3.6	91.40
4.957	3.13	3.6	87.02
4.957	2.88	3.6	79.98
			105.66 AVG
			15.20 STDEV

M617- Desorption

Initial soln concn (C ₀) (ug/mL)	Kd (mL/g)	% organic carbon	Koc (mL/g)
0.048	3.26	0.6	543.86
0.048	2.21	0.6	368.42
0.097	2.67	0.6	444.44
0.097	2.12	0.6	352.94
0.502	2.86	0.6	476.19
0.502	4.14	0.6	690.06
1.012	1.45	0.6	241.03
1.012	0.95	0.6	157.89
4.957	1.06	0.6	176.75
4.957	1.06	0.6	176.22
			362.78 AVG
			178.19 STDEV

Reviewer-calculated from data obtained in Tables 1-2, 11-12 and Appendix D, pp. 50-54, 64-71, 113-115 of the study report.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

Desorption

Soil	Kd (avg)	Kd (stdev)	% o. c.	Koc (avg)	Koc (stdev)
M568(5°C)	6.13	0.18	3.9	157.19	4.73
M599(5°C)	-	-	1.5	-	-

M568- Desorption at 5 °C

Initial soln concn (C ₀) (ug/mL)	Kd (mL/g)	% organic carbon	Koc (mL/g)
0.502	6.26	3.9	160.54
0.502	6.00	3.9	153.85
			157.19 AVG
			4.73 STDEV

M599- Desorption at 5 °C

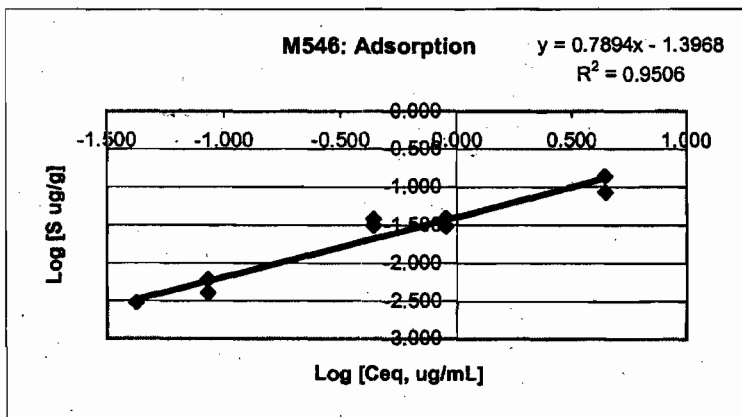
Initial soln concn (C ₀) (ug/mL)	Kd (mL/g)	% organic carbon	Koc (mL/g)
0.502	-	1.5	-
0.502	-	1.5	-
			- AVG
			- STDEV

Reviewer-calculated from data obtained in Table 15, p. 74 of the study report.

Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

M546- Adsorption

Labelled initial soln concn (C _o) (ug/mL)	Concen in soln after equil (C _{eq}) (ug/mL)	Concen in soil after equil (S) (ug/g)	Log (C _{eq}) (log ug/mL)	Log (S) (log ug/g)
0.048	0.042	0.003	-1.377	-2.523
0.048	0.042	0.003	-1.377	-2.523
0.097	0.085	0.006	-1.071	-2.222
0.097	0.085	0.004	-1.071	-2.398
0.502	0.436	0.038	-0.361	-1.420
0.502	0.437	0.031	-0.360	-1.509
1.012	0.897	0.039	-0.047	-1.409
1.012	0.897	0.030	-0.047	-1.523
4.957	4.423	0.139	0.646	-0.857
4.957	4.445	0.085	0.648	-1.071



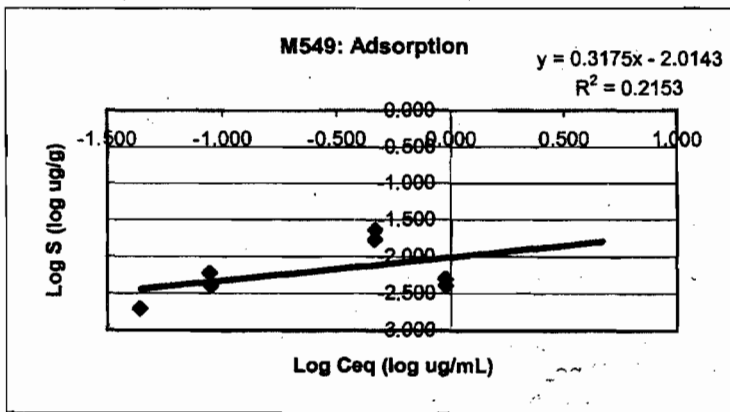
M546- Adsorption

1/n	0.789
R ²	0.951
K _F	0.040

Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

M549- Adsorption

Labelled initial soln concn (C _o) (ug/mL)	Concen in soln after equil (C _{eq}) (ug/mL)	Concen in soil after equil (S) (ug/g)	Log (C _{eq}) (log ug/mL)	Log (S) (log ug/g)
0.048	0.044	0.002	-1.357	-2.699
0.097	0.089	0.004	-1.051	-2.398
0.097	0.088	0.006	-1.056	-2.222
0.502	0.463	0.017	-0.334	-1.770
0.502	0.465	0.023	-0.333	-1.638
1.012	0.949	0.004	-0.023	-2.398
1.012	0.949	0.005	-0.023	-2.301
4.957	4.688	0.000	0.671	
4.957	4.685	0	0.671	



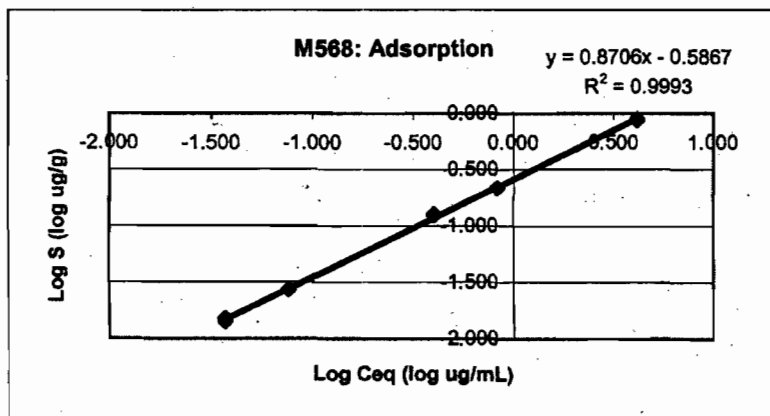
M549- Adsorption

1/n	0.318
R2	0.215
K _F	0.010

Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

M568- Adsorption

Labelled initial soln concn (C _o) (ug/mL)	Concen in soln after equil (C _{eq}) (ug/mL)	Concen in soil after equil (S) (ug/g)	Log (C _{eq}) (log ug/mL)	Log (S) (log ug/g)
0.048	0.037	0.015	-1.432	-1.824
0.048	0.037	0.014	-1.432	-1.854
0.097	0.076	0.027	-1.119	-1.569
0.097	0.076	0.027	-1.119	-1.569
0.502	0.403	0.127	-0.395	-0.896
0.502	0.402	0.124	-0.396	-0.907
1.012	0.827	0.216	-0.082	-0.666
1.012	0.829	0.216	-0.081	-0.666
4.957	4.162	0.861	0.619	-0.065
4.957	4.164	0.896	0.620	-0.048



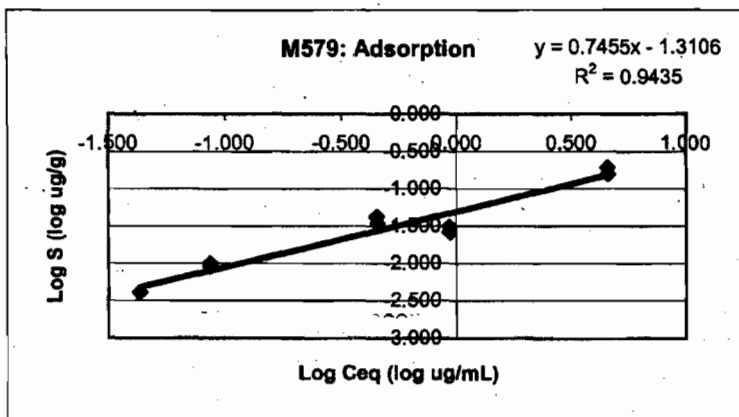
M568- Adsorption

1/n	0.871
R ²	0.999
K _F	0.259

Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

M579- Adsorption

Labelled initial soln concen (C ₀) (ug/mL)	Concen in soln after equil (C _{eq}) (ug/mL)	Concen in soil after equil (S) (ug/g)	Log (C _{eq}) (log ug/mL)	Log (S) (log ug/g)
0.048	0.043	0.004	-1.367	-2.398
0.048	0.043	0.004	-1.367	-2.398
0.097	0.086	0.01	-1.066	-2.000
0.097	0.086	0.009	-1.066	-2.046
0.502	0.451	0.042	-0.346	-1.377
0.502	0.455	0.034	-0.342	-1.469
1.012	0.933	0.031	-0.030	-1.509
1.012	0.937	0.026	-0.028	-1.585
4.957	4.583	0.156	0.661	-0.807
4.957	4.566	0.192	0.660	-0.717



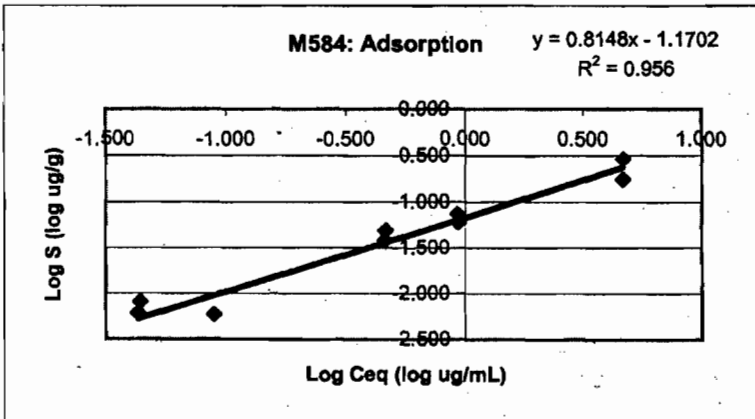
M579- Adsorption

1/n	0.746
R ²	0.944
K _F	0.049

Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

M584- Adsorption

Labelled initial soln concn (C ₀) (ug/mL)	Concen in soln after equil (C _{eq}) (ug/mL)	Concen in soil after equil (S) (ug/g)	Log (C _{eq}) (log ug/mL)	Log (S) (log ug/g)
0.048	0.044	0.008	-1.357	-2.097
0.048	0.043	0.006	-1.367	-2.222
0.097	0.089	0.006	-1.051	-2.222
0.097	0.089	0.006	-1.051	-2.222
0.502	0.46	0.038	-0.337	-1.420
0.502	0.463	0.049	-0.334	-1.310
1.012	0.928	0.074	-0.032	-1.131
1.012	0.936	0.06	-0.029	-1.222
4.957	4.66	0.175	0.668	-0.757
4.957	4.66	0.289	0.668	-0.539



M584- Adsorption

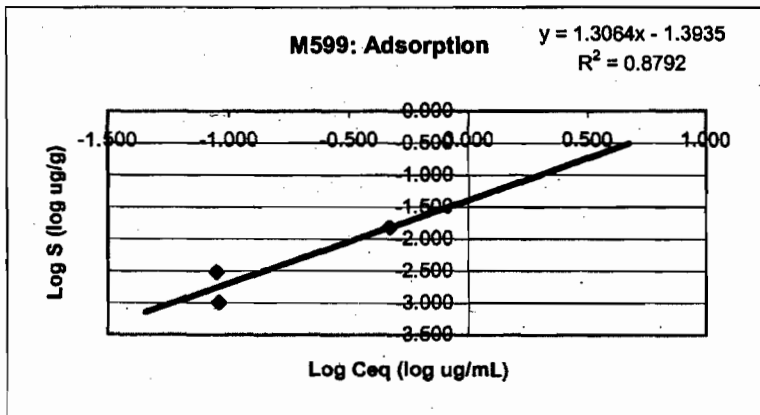
1/n	0.815
R2	0.956
K _F	0.068

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

M599- Adsorption

Labelled initial soln concn (C ₀) (ug/mL)	Concen in soln after equil (C _{eq}) (ug/mL)	Concen in soil after equil (S) (ug/g)	Log (C _{eq}) (log ug/mL)	Log (S) (log ug/g)
0.048	0.046	0	-1.337	
0.048	0.045	0	-1.347	
0.097	0.091	0.001	-1.041	-3.000
0.097	0.089	0.003	-1.051	-2.523
0.502	0.467	0.015	-0.331	-1.824
0.502	0.467	0.015	-0.331	-1.824
1.012	0.967	0	-0.015	
1.012	0.976	0	-0.011	
4.957	4.707	0	0.673	
4.957	4.760	0	0.678	



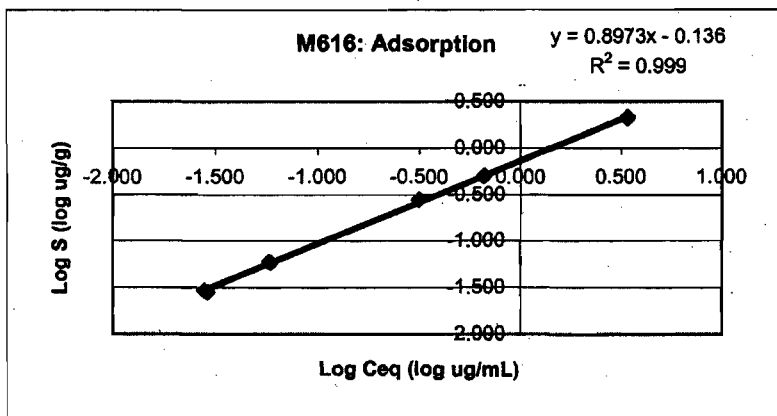
M599- Adsorption

1/n	1.306
R2	0.879
K _F	0.040

Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

M616-Adsorption

Labelled initial soln concn (C ₀) (ug/mL)	Concen in soln after equil (C _{eq}) (ug/mL)	Concen in soil after equil (S) (ug/g)	Log (C _{eq}) (log ug/mL)	Log (S) (log ug/g)
0.048	0.029	0.028	-1.538	-1.553
0.048	0.028	0.029	-1.553	-1.538
0.097	0.058	0.059	-1.237	-1.229
0.097	0.059	0.058	-1.229	-1.237
0.502	0.314	0.277	-0.503	-0.558
0.502	0.315	0.281	-0.502	-0.551
1.012	0.659	0.495	-0.181	-0.305
1.012	0.662	0.502	-0.179	-0.299
4.957	3.41	2.162	0.533	0.335
4.957	3.397	2.083	0.531	0.319



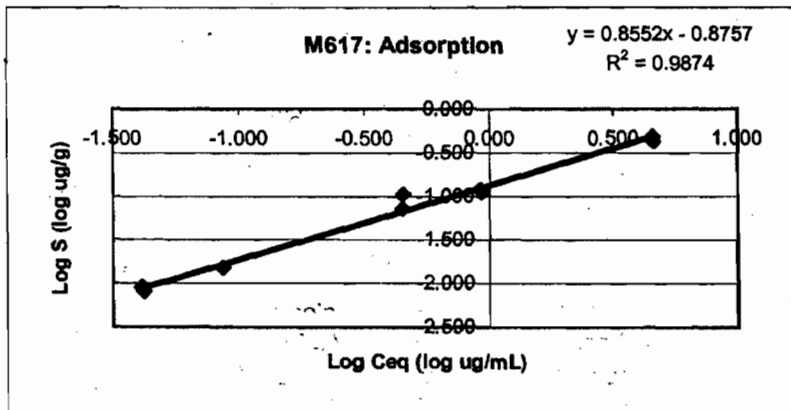
M616- Adsorption

1/n	0.897
R2	0.999
K _F	0.731

Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

M617- Adsorption

Labelled initial soln concn (C ₀) (ug/mL)	Concen in soln after equil (C _{eq}) (ug/mL)	Concen in soil after equil (S) (ug/g)	Log (C _{eq}) (log ug/mL)	Log (S) (log ug/g)
0.048	0.041	0.009	-1.387	-2.046
0.048	0.042	0.008	-1.377	-2.097
0.097	0.086	0.015	-1.066	-1.824
0.097	0.086	0.015	-1.066	-1.824
0.502	0.449	0.105	-0.348	-0.979
0.502	0.448	0.072	-0.349	-1.143
1.012	0.918	0.12	-0.037	-0.921
1.012	0.932	0.112	-0.031	-0.951
4.957	4.561	0.484	0.659	-0.315
4.957	4.582	0.428	0.661	-0.369

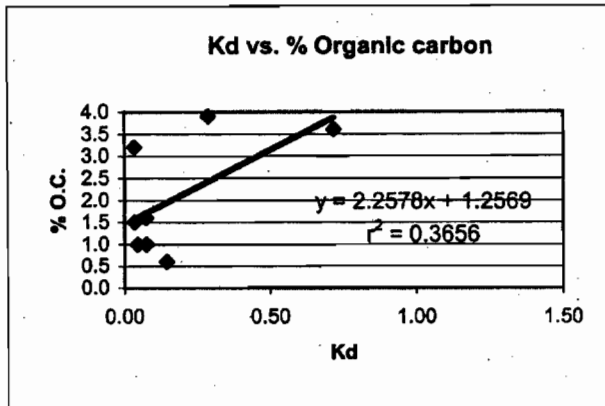


M617- Adsorption

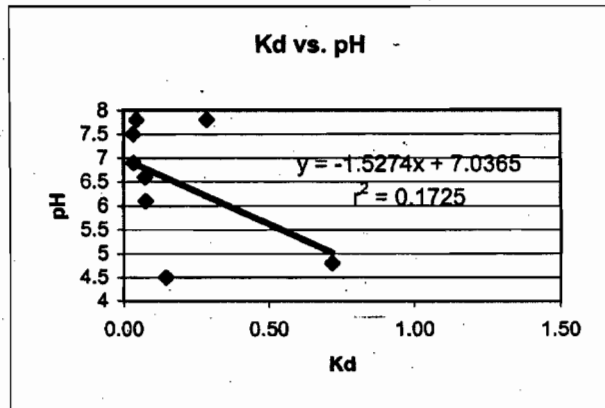
1/n	0.855
R ²	0.987
K _F	0.133

Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

Soil	Kd	% organic carbon
M546	0.04	1.0
M549	0.03	3.2
M568	0.29	3.9
M579	0.07	1.6
M584	0.08	1.0
M599	0.03	1.5
M616	0.72	3.6
M617	0.15	0.6



Soil	Kd	pH
M546	0.04	7.8
M549	0.03	7.5
M568	0.29	7.8
M579	0.07	6.6
M584	0.08	6.1
M599	0.03	6.9
M616	0.72	4.8
M617	0.15	4.5

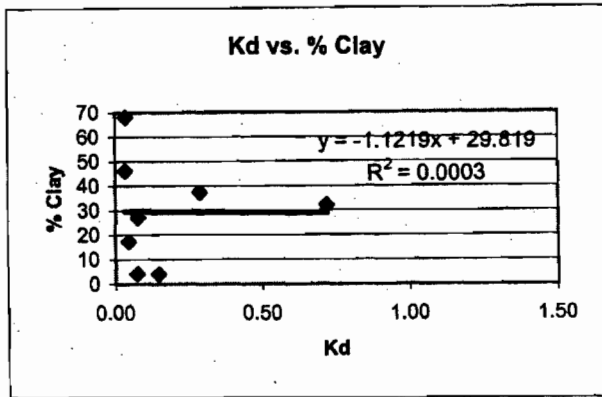


Data were obtained from Tables 1-2, pp. 50-54. Kd values were reviewer-calculated using data obtained from Table 11 and Appendix D, pp. 64-67, 113-115.

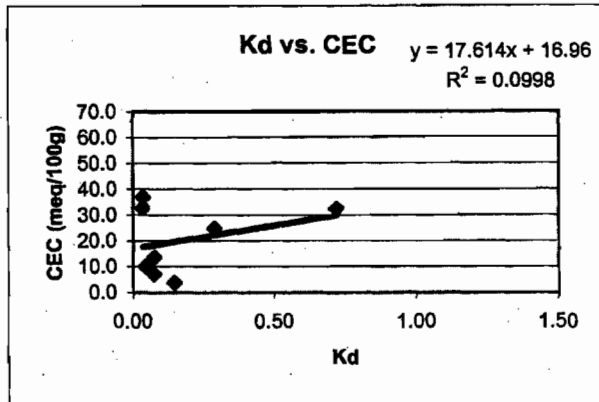
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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

Soil	Kd	% clay
M546	0.04	17.2
M549	0.03	46
M568	0.29	37.2
M579	0.07	4
M584	0.08	27
M599	0.03	68
M616	0.72	32
M617	0.15	4



Soil	Kd	CEC
M546	0.04	9.9
M549	0.03	32.7
M568	0.29	24.6
M579	0.07	7.1
M584	0.08	13.5
M599	0.03	36.9
M616	0.72	32.3
M617	0.15	3.6



Data were obtained from Tables 1-2, pp. 50-54. Kd values were reviewer-calculated using data obtained from Table 11 and Appendix D, pp. 64-67, 113-115.

Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

M546- Adsorption

Labelled initial soln concn (C ₀) (ug/mL)	Actual initial soln concn (C ₀) (ug/mL)	Difference b/w labelled and actual	% Difference
0.048	0.043	0.005	10.42
0.048	0.043	0.005	10.42
0.097	0.087	0.010	10.31
0.097	0.087	0.010	10.31
0.502	0.453	0.049	9.76
0.502	0.453	0.049	9.76
1.012	0.912	0.100	9.88
1.012	0.912	0.100	9.88
4.957	4.480	0.477	9.62
4.957	4.479	0.478	9.64

M549- Adsorption

Labelled initial soln concn (C ₀) (ug/mL)	Actual initial soln concn (C ₀) (ug/mL)	Difference b/w labelled and actual	% Difference
0.048	0.045	0.003	6.25
0.048	0.045	0.003	6.25
0.097	0.091	0.006	6.19
0.097	0.091	0.006	6.19
0.502	0.472	0.030	5.98
0.502	0.472	0.030	5.98
1.012	0.951	0.061	6.03
1.012	0.951	0.061	6.03
4.957	4.666	0.291	5.87
4.957	4.664	0.293	5.91

M568- Adsorption

Labelled initial soln concn (C ₀) (ug/mL)	Actual initial soln concn (C ₀) (ug/mL)	Difference b/w labelled and actual	% Difference
0.048	0.044	0.004	8.33
0.048	0.044	0.004	8.33
0.097	0.089	0.008	8.25
0.097	0.089	0.008	8.25
0.502	0.461	0.041	8.17
0.502	0.461	0.041	8.17
1.012	0.930	0.082	8.10
1.012	0.923	0.089	8.79
4.957	4.562	0.395	7.97
4.957	4.565	0.392	7.91

Reviewer-calculated from data obtained in Table 11 and Appendix D, pp. 64-67, 113-115 of the study report.

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Chemical: Aminopyralid
 PC Code: 005100
 MRID: 46235732
 Guideline No: 163-1

M579- Adsorption

Labelled initial soln concn (C _o) (ug/mL)	Actual initial soln concn (C _o) (ug/mL)	Difference b/w labelled and actual	% Difference
0.048	0.045	0.003	6.25
0.048	0.045	0.003	6.25
0.097	0.091	0.006	6.19
0.097	0.091	0.006	6.19
0.502	0.472	0.030	5.98
0.502	0.472	0.030	5.98
1.012	0.951	0.061	6.03
1.012	0.951	0.061	6.03
4.957	4.666	0.291	5.87
4.957	4.666	0.291	5.87

M584- Adsorption

Labelled initial soln concn (C _o) (ug/mL)	Actual initial soln concn (C _o) (ug/mL)	Difference b/w labelled and actual	% Difference
0.048	0.046	0.002	4.167
0.048	0.046	0.002	4.167
0.097	0.092	0.005	5.155
0.097	0.092	0.005	5.155
0.502	0.480	0.022	4.382
0.502	0.480	0.022	4.382
1.012	0.968	0.044	4.348
1.012	0.968	0.044	4.348
4.957	4.746	0.211	4.257
4.957	4.746	0.211	4.257

M599- Adsorption

Labelled initial soln concn (C _o) (ug/mL)	Actual initial soln concn (C _o) (ug/mL)	Difference b/w labelled and actual	% Difference
0.048	0.045	0.003	6.25
0.048	0.045	0.003	6.25
0.097	0.091	0.006	6.19
0.097	0.091	0.006	6.19
0.502	0.474	0.028	5.58
0.502	0.474	0.028	5.58
1.012	0.956	0.056	5.53
1.012	0.956	0.056	5.53
4.957	4.687	0.270	5.45
4.957	4.687	0.270	5.45

Reviewer-calculated from data obtained in Table 11 and Appendix D, pp. 64-67, 113-115 of the study report.

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Chemical: Chemical:
 PC Code: PC Code:
 MRID: MRID:
 Guideline No: Guideline No:

M616- Adsorption

Labelled initial soln concn (C _o) (ug/mL)	Actual initial soln concn (C _o) (ug/mL)	Difference b/w labelled and actual	% Difference
0.048	0.041	0.007	14.58
0.048	0.041	0.007	14.58
0.097	0.083	0.014	14.43
0.097	0.083	0.014	14.43
0.502	0.431	0.071	14.14
0.502	0.431	0.071	14.14
1.012	0.868	0.144	14.23
1.012	0.867	0.145	14.33
4.957	4.270	0.687	13.86
4.957	4.267	0.690	13.92

M617- Adsorption

Labelled initial soln concn (C _o) (ug/mL)	Actual initial soln concn (C _o) (ug/mL)	Difference b/w labelled and actual	% Difference
0.048	0.046	0.002	4.17
0.048	0.046	0.002	4.17
0.097	0.093	0.004	4.12
0.097	0.093	0.004	4.12
0.502	0.483	0.019	3.78
0.502	0.483	0.019	3.78
1.012	0.974	0.038	3.75
1.012	0.974	0.038	3.75
4.957	4.776	0.181	3.65
4.957	4.774	0.183	3.69

Reviewer-calculated from data obtained in Table 11 and Appendix D, pp. 64-67, 113-115 of the study report.

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Data Evaluation Report on the Terrestrial Field Dissipation of XDE-750 (Aminopyralid)

PMRA Submission Number {.....}

EPA MRID Number {46235734}

Page 1

Data Requirement: _____ PMRA Data Code: _____

EPA DP Barcode: D301682

OECD Data Point:

EPA Guideline: Subdivision N, § 164-1

Test Material: Aminopyralid

End Use Product name: GF-871

Concentration of a.i.: 40.6%

Concentration of a.e.: 21.1%

Formulation type: Soluble liquid concentrate (SL)

Active ingredient

Common name: Aminopyralid

Chemical name:

IUPAC: 4-amino-3,6-dichloropyridine-2-carboxylic acid

CAS: 2-pyridinecarboxylic acid, 4-amino-3,6-dichloro-

CAS No: 150114-71-9

Synonyms: XR-750; XDE-750

SMILES string: c1c(nc(c(c1N)Cl)C(=O)O)Cl

Primary Reviewer: Dan Hunt

Signature:

Dynamac Corporation

Date:

QC Reviewer: Joan Harlin

Signature:

Dynamac Corporation

Date:

Secondary Reviewer(s): Cheryl Sutton, Ph.D.

Signature: *Cheryl Sutton*

EPA

Date: *5/11/05*

Company Code: [for PMRA]

Active Code: [for PMRA]

Use Site Category: [for PMRA]

EPA PC Code: 005100

CITATION: Roberts, D.W. and Schelle, G.E. Terrestrial Field Dissipation of XDE-750 in the USA, Unpublished report of Dow AgroSciences LLC, Study ID 020032, 16-January-2004. Experiment initiation 4-August-2003 and completion 20-February-2003 (p. 6).



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EXECUTIVE SUMMARY:

Soil dissipation of XDE-750 (aminopyralid) under U.S.A. field conditions was conducted in bare plots at 2 sites in Mississippi (MS) and California (CA). The sites at MS and CA were in Ecoregions 4 and 10, respectively. The experiment was carried out in accordance with the US EPA, Subdivision N, 164-1 guidelines, and in compliance with the OECD-GLP standard. Aminopyralid was surface broadcast applied at 152 g a.e./ha at MS and 155 g a.e./ha at CA on three replicated 4.6 m x 21.3 m plots using a single application. The reported application rate was based on the theoretical spray solution concentration and the spray discharge rate; the target application rate was 150 g a.e./ha for both test sites. The target application rate corresponds to 125% (an exaggerated rate) of the proposed maximum label rate. Rainfall was supplemented with irrigation to reach targets of 120% of the 30-year average rainfall at MS, and 75% of evapotranspiration (far exceeding normal average rainfall) at CA. The treated plots were approximately 1m apart, and the control plot was approximately 16 m away from the treated plot.

The application rate was verified using application monitors (shallow soil pans). There was 82-119% recovery at MS (mean of 99.5%), and 72-117% recovery at CA (mean of 94.0%) from these monitors based on the field application calculations. Field spiking of the samples was done by fortifying control soil with aminopyralid at 15 µg a.e./kg soil (~30 g a.e./ha). There was 101% recovery of the applied aminopyralid in the field spiked samples.

Soil samples were taken at targets of 0, 7, 14, 30, 60, 90, 120 and 180 days post-application to a depth of 0 - 90 cm. The soil samples were extracted twice with a 90% acetonitrile/10% 1N HCL solution, and aminopyralid residues were analyzed by high performance liquid chromatography with positive ion Turbo Ion Spray tandem mass spectrometry (LC/MS/MS). The LOD and LOQ in soil were 0.300 µg a.e./kg and 1.500 µg a.e./kg, respectively. Although transformation products were not monitored, there were no major degradates detected in the laboratory metabolism studies.

In surface (0-15 cm) soil at MS, aminopyralid dissipated from a mean of 77.9 ng/g (range of 32.3-144.2 ng/g) at day 0 to 47.4 ng/g by day 8, increased to a mean of 92.6 ng/g (range of 65.8-119.7 ng/g) by day 15, then decreased to 35.8 ng/g by day 29 and decreased, with variability, to <LOQ by day 183 (Appendix G, Table 1). In surface (0-15 cm) soil at CA, aminopyralid was a mean of 64.8 ng/g at day 0, increased to 86.2 ng/g (range of 70.8-104.2 ng/g) by day 9, then decreased to 48.3 ng/g by day 22 and was below the LOQ by day 91. (Appendix G, Table 2). The residues of aminopyralid were primarily detected in the top 0- to 15-cm soil layer at MS; and in the top 0- to 30-cm soil layers, with detections through the 75- to 90-cm layer, at CA. Because this field study was conducted using non-radio-labeled material, total mass accounting was not calculated.

Aminopyralid dissipated in surface soil (0- to 15-cm depth only) under terrestrial field conditions with first-order half-lives (log linear) of 32.1 days ($r^2 = 0.81$) for the MS site and 20.0 days ($r^2 = 0.87$) for the CA site. However, both temporal and inter-replicate data variability make the half-lives of questionable value. The respective registrant-calculated first-order field half-lives and DT_{90} 's of aminopyralid (for residues in the total soil profile) under terrestrial field conditions were 34 days and 114 days in MS, and 26 days and 85 days in CA. At the end of the 183-day period at MS, the total carryover of aminopyralid residues was 1.6% of the applied amount. At the end of the 182-day period at CA, the total carryover of aminopyralid residues was 0.6% of the applied amount.

The major route of dissipation could not be determined because leaching was not observed (MS only), and runoff and volatilization were not measured. However, the study authors stated that no significant soil metabolites have been observed in any environmental compartment, and that degradation appears to be predominantly a microbial process leading to complete mineralization of the molecule and the formation of bound residues.

RESULTS SYNOPSIS

Site: MS
Location/soil type: Bosket silty loam
Half-life (in surface soil): 32.1 days
Half-life (in total soil profile): 34 days
DT₉₀ (in total soil profile): 114 days
Major transformation products detected: not applicable
Dissipation routes: microbial degradation

Site: CA
Location/soil type: Hisperia sand loam
Half-life (in surface soil): 20.0 days
Half-life (in total soil profile): 26 days
DT₉₀ (in total soil profile): 85 days
Major transformation products detected: not applicable
Dissipation routes: microbial degradation

Study Acceptability: This study is classified as supplemental. Both temporal and inter-replicate data variability (especially at times 0, 15 and 57 days at the MS site and 9 days at the CA site) make the half-lives of questionable value. Also, the storage stability study was in progress at the time of this report, and those samples were not stored for a length of time equal to or exceeding the length of storage for test samples.

I. MATERIALS AND METHODS

GUIDELINE FOLLOWED: Subdivision N, § 164-1. The following deviations from Subdivision N Guideline § 164-1 were noted:

The laboratory storage stability study was not conducted for a period of time equal to or exceeding the length of time that test samples were stored frozen. This does not affect the validity of the study.

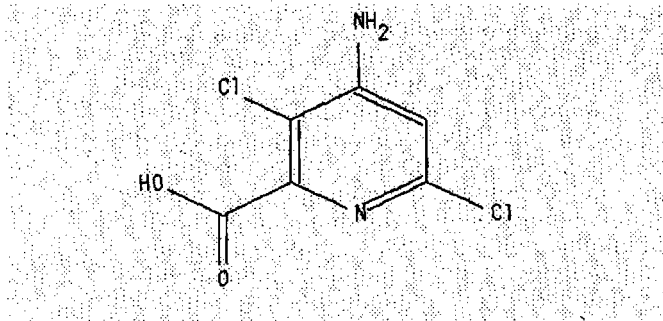
COMPLIANCE: US EPA GLPS and OECD-GLP; signed and dated GLP; Quality Assurance, Data Confidentiality, and Certification statements were provided (pp. 2-5).

A. MATERIALS:

Test Material: XDE-750 (aminopyralid; p. 17)

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Chemical structure of the active ingredient:



Description: Soluble concentrate liquid (SL; p. 17)

Storage conditions of test chemicals: Test material (GF-871) was stored in normal ambient condition in mixed light and dark conditions.

Physico-chemical Properties of the Active Ingredient(s):

Parameter	Values	Comments
Molecular formula	C ₆ H ₄ Cl ₂ N ₂ O ₂	
Molecular weight	207.026 g/mol	
Water solubility	2480 mg/L (ppm)	At 20°C
Vapor pressure/volatility	7.14e-11 mmHg or 9.52e-9 Pa	At 20°C
UV absorption	217 nm	
pKa	2.56	
K _{ow} /log K _{ow}	log K _{ow} 0.201	
Stability of compound at room temperature	99.9% (2003)	At 20°C; 99.5% (2000)

Appendix A, Tables 1-2, pp. 72-73.

Data Evaluation Report on the Terrestrial Field Dissipation of XDE-750 (Aminopyralid)

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Test site: The MS test site was located in Washington County and the CA test site was located in Fresno County (pp. 17-18). Site descriptions and pesticide use histories are reported in Tables 1 and 2 below.

Table 1: Geographic Location, Site Description and Climatic Data at the Study Site(s).

Details		Site 1 (MS)	Site 2 (CA)
Geographic coordinates	Latitude	N 33° 20'	N 36° 45'
	Longitude	W 90° 55'	W 119° 50'
	Province/State	MS	CA
	Country	USA	USA
	Ecoregion	4	10
Slope gradient		0-2%	≤1%
Depth to ground water (m)		>6 ft (1.8 m)	ca. 60 ft (18.2 m)
Distance from weather station used for climatic measurements		1,000 ft	600 ft
Indicate whether the meteorological conditions before starting or during the study were within 30 year normal levels (Yes/No). If no, provide details.		yes (136% Normal)	yes (75% of Et ₀ ; >1000% Normal)
Other details, if any			

pp. 17-18, 21-22 and Tables 4-5, pp. 41-42.

Table 2: Site Usage and Management History for the Previous Three Years.

Use	Year	Site 1 (MS)	Site 2 (CA)
Crops grown	Previous year	corn	grapes
	2 years	cotton	grapes
	3 years	cotton	grapes
Pesticides used	Previous year	Surpass EC	Super Six Sulfer, Rally, Elite, Flint, Gramaxone, Roundup, Gallery, Goal, Provado, Kelthane, Success
	2 years	Treflan HFP, Zorial Rapid 80, Temik 15G, Ridomil Gold, Cotoran, Orthene 90S, Pix Plus	Super Six Sulfer, Elite, Quintec, Rally, Surflan, Roundup, Gramaxone, Goal, Provado, Kelthane, Success
	3 years	Treflan HFP, Zorial Rapid 80, Cotoran, Ridomil Gold, Terrachlor Super X, Temik 15G, Pix Plus	Super Six Sulfer, Quintec, Rally, Elite, Surflan, Goal, Roundup, Gallery, Provado, Kelthane
Fertilizers used	Previous year	none	none
	2 years	none	none
	3 years	none	none
Cultivation methods, if provided.	Previous year	not provided	not provided
	2 years	not provided	not provided
	3 years	not provided	not provided
Other details, if any	Previous year	none	none
	2 years	none	none
	3 years	none	none

Appendix E, pp. 95-96.

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Soils:

Table 3: Properties of the Soil from Site 1 (MS)

Property	Depth (cm)					
	0-15	15-30	30-45	45-60	60-75	75-90
Textural classification	SiL	SiL	SiL	SiL	SiC	SiCL
% sand	29	29	29	21	11	9
% silt	60	56	56	56	46	56
% clay	11	15	15	23	43	35
pH (1:1 soil:water or other)	6.0	6.5	6.7	6.7	6.6	6.6
Total organic carbon (%)	0.6	0.5	0.3	0.3	0.5	0.5
CEC (meq/100 g)	10.3	13.7	15.1	19.4	29.9	24.4
Bulk density (g/cm ³)	1.18	1.18	1.17	1.20	1.17	1.17
Moisture at 1/3 atm (%)	19.3	23.7	25.0	28.6	39.0	35.8
Taxonomic classification (e.g., ferro-humic podzol)	Fine-loamy, mixed, active, thermic Mollic Hapludalfs					
Soil mapping unit	- -	- -	- -	- -	- -	- -
Others	Bosket soil series					

pp. 17-18 and Table 1, p. 38.

Table 4: Properties of the Soil from Site 2 (CA)

Property	Depth (cm)					
	0-15	15-30	30-45	45-60	60-75	75-90
Textural classification	SL	SL	SL	SL	SL	SL
% sand	52	54	62	62	62	66
% silt	30	30	26	28	30	26
% clay	18	16	12	10	8	8
pH (1:1 soil:water or other)	7.2	7.2	7.5	7.6	7.7	7.7
Total organic carbon (%)	0.8	0.3	0.2	0.1	0.1	0.1
CEC (meq/100 g)	16.4	17.4	17.7	17.2	16.6	16.0
Bulk density (g/cm ³)	1.15	1.17	1.21	1.21	1.24	1.27
Moisture at 1/3 atm (%)	19.2	16.0	15.8	15.5	15.5	15.3
Taxonomic classification (e.g., ferro-humic podzol)	Coarse-loamy, mixed, superactive, nonacid, thermic Xeric Torriorthents					
Soil mapping unit	- -	- -	- -	- -	- -	- -
Others	Hesperia soil series					

pp. 18-19 and Table 2, p. 39.

B. EXPERIMENTAL DESIGN:**Experimental design:**

Table 5: Experimental Design.

Details		Site 1 (MS)	Site 2 (CA)
Duration of study		183 days (6 mo.s)	182 days (6 mo.s)
Uncropped (bare) or cropped		bare	bare
Control used (Yes/No)		yes	yes
No. of replications	Controls	1	1
	Treatments	3	3
Plot size (L x W m)	Control	4 m x 2 m	4 m x 2 m
	Treatment	21 m x 5 m	21 m x 5 m
Distance between control plot and treated plot		15 m	15 m
Distance between treated plots		1 m	2-3 m
Application rate(s) used (g a.e./ha) (note: reported rates are based on monitoring plates rather than on analyzed soil samples)		152 g ae/ha (actual) 150 g a.e./ha (target)	155 g ae/ha (actual) 150 g a.e./ha (target)
Was the maximum label rate per ha used in study? (Yes/No)		No, 125% of maximum (exaggerated rate)	No, 125% of maximum (exaggerated rate)
Number of applications		1	1
Application date(s) (dd-mmm-yyyy)		15-April-2002	21-May-2002
For multiple applications, application rate at Day 0 and at each application time (mg a.i./kg soil)		n/a	n/a
Application method (e.g., spraying, broadcast, etc.)		ground broadcast	ground broadcast
Type of spray equipment, if used		tractor mount; compressed air	tractor mount; compressed air
Total volume of spray solution applied/plot or total amount broadcasted/plot		297.72 L/ha; 2.97 L/plot	286.98 L/ha; 2.87 L/plot
Identification and volume of carrier (e.g., water), if used		5.987 L water/plot	11.973 L water/plot

Table 5 (Cont.): Experimental Design.

Details		Site 1 (MS)	Site 2 (CA)
Name and concentration of co-solvents, adjuvants, and/or surfactants, if used		n/a	n/a
Indicate whether the following monthly reports were submitted: Average minimum and maximum precipitation Average minimum and maximum air temperature Average minimum and maximum soil temperature Average annual frost-free periods		total daily precip. yes no no	total daily precip. yes no no
Indicate whether the pan evaporation data were submitted		no	reference (Penman) evapotranspiration (ET ₀)
Meteorological conditions during application	Cloud cover	50%	- -
	Temperature (°C)	21.7 °C	25.5 °C
	Humidity	82%	44%
	Sunlight (hr)	9:05 am	1:47 pm
Pesticides used during study: name of product/a.i concentration: amount applied: application method:	Gramoxone Max: 43.8% a.i.; 1 qt/A; Glyphomax Plus: 41.0% a.i.; 1 qt/A; Oust: 75% a.i.; 2 oz/A; ground broadcast	Glyphomax Plus: 41.0% a.i.; 1 qt/A; ground broadcast	
Supplemental irrigation used (Yes/No) If yes, provide the following details: No. of irrigations: Interval between irrigation: Amount of water added each time: Method of irrigation:	Yes 22 2 wks. as needed. Avg: 0.6 in. sprinklers	Yes 63 2-8 days as needed. Avg: 0.7 in. sprinklers	
Indicate whether water received through rainfall + irrigation equals the 30-year average rainfall (Yes/No)		yes	yes
Were the application concentrations verified? (Briefly describe in Section 2', if used)		yes	yes
Were field spikes used? (Briefly describe in Section 3', if used)		yes	yes
Were good agricultural practices followed (Yes or No)		yes	yes
Indicate if any abnormal climatic events occurred during the study (e.g., drought, heavy rainfall, flooding, storm, etc.)		none	none
If cropped plots are used, provide the following details: Plant - Common name/variety: Details of planting: Crop maintenance (e.g., fertilizers used):		n/a	n/a
Was volatilization included in the study? (Yes/No) (if included, describe in Section 4')		no	no
Was leaching included in the study? (Yes/No) (if included, describe in Section 5')		Yes	Yes
Was runoff included in the study? (Yes/No) (if included, describe in Section 6')		no	no

pp. 15, 19-27, 32-33; Figure 3, p. 69; Appendix B, Tables 1-2, pp. 75-76; Appendix H, Tables 1-2, pp. 108-118.

Application Verification: For each replicate plot, used five 12-in. diameter disposable aluminium pans with shallow layer of soil (p. 24).

Field Spiking: Three 5-g control soil samples from each field site were spiked at 15 ppb, shipped frozen overnight to the test site; stored frozen at test site; removed 12-hrs prior to sampling event; exposed to same field collection and storage conditions as routine soil cores prior to shipment back to DAS lab via freezer truck (p. 27; Appendix I, p. 120). One spike and 1 control analyzed.

Volatilization: Volatilization was not measured.

Leaching: Soil samples were collected to a depth of 90 cm at targets of 7, 14, 30, 60, 90, 120 and 180 days posttreatment to determine leaching (p. 25; Table 3, p. 40).

Run off: Runoff was not measured.

Supplementary Study: Long-term frozen storage stability study is in progress (as of 9/22/03) (p. 27). Preliminary results show aminopyralid to be stable (111%) in soil stored frozen for 6 mo.s (180 days; p. 30). The longest period samples were stored in this study is 316 days (Appendix I, Table 1, p. 130).

Sampling:

Table 6: Soil Sampling

Details	Site 1 (MS)	Site 2 (CA)
Method of sampling (random or systematic)	systematic	systematic
Sampling intervals (days)	0,8,15,29,57,93,122,183	0,9,15,22,65,91,126,182
Method of soil collection (e.g., cores)	cores	cores
Sampling depth	15 cm (Day 0 samples) or 90 cm	15 cm (Day 0 samples) or 90 cm
Number of cores collected per plot	5	5
Number of segments per core	6	6
Length of soil segments	15 cm	15 cm
Core diameter (Provide details if more than one width)	0-15 cm: 11.10 cm 15-90 cm: 4.19 cm	0-15 cm: 11.05 cm 15-90 cm: 3.38 cm
Method of sample processing, if any	cut into 15-cm segments; combined by replicate plot; mixed and ground with dry-ice.	cut into 15-cm segments; combined by replicate plot; mixed and ground with dry-ice.
Storage conditions	freezing (ca. -20 °C)	freezing (ca. -20 °C)
Storage length (days)	122-316	90-272

pp. 25-27; Appendix I, Table 1, p. 130.

Analytical Procedures: Soil samples were analyzed for aminopyralid by a validated method using high performance liquid chromatography with positive ion Turbo Ion spray tandem mass spectrometry (LC/MS/MS) (pp. 27-28; Appendix I, p. 121).

Number of soil samples analyzed per treatment or composite sample:

300 samples total (1 analysis/composite sample).

Number of plant samples analyzed per treatment or composite sample: n/a**Extraction, clean up and concentration of soil samples:**

Soil samples were extracted twice with 90% acetonitrile/10% 1N HCl (Appendix I, p. 121). An aliquot of the combined extract from each sample was evaporated to dryness under a stream of nitrogen. The samples were reconstituted with 1N HCl. An aliquot of the concentrated extract was purified using a polymeric 96-well solid phase extraction (SPE) plate. Aminopyralid was eluted with acetonitrile into a vial containing the aminopyralid stable isotope internal standard solution. The eluate was then evaporated to dryness under a stream of nitrogen. Two hundred microliter of the 22:2:1 acetonitrile:pyridine:-butanol coupling reagent was then added to each sample. The samples were then derivitized by adding 10 μ L of butyl chloroformate to each sample. Next, 790 μ L of the 50:50:0.1 methanol:water:acetic acid mobile phase was added to each sample.

Identification and quantification of parent compound:

The samples were then analyzed by high performance liquid chromatography with positive ion Turbo Ion Spray tandem mass spectrometry (LC/MS/MS) (Appendix I, p. 121). Sample analysis was performed using a Diazem 3000, C18 column installed in a PE/Sciex API 3000 system with an Agilent HPLC and a PE/Sciex tandem mass selective detector. During analysis the following MS/MS transitions were monitored:

Name	Masses (amu)
aminopyralid butyl ester	263/134
aminopyralid Stable Isotope	268/139

Chromatographic data was collected using the PE/Sciex LC/MS/MS data system. The chromatograms were integrated using PE/Sciex Analyst Software, version 1.1.

The purity of the analytical standard used as the reference material was 99.5% (Appendix I, p. 121).

Identification and quantification of transformation products: n/a**Detection limits (LOD, LOQ) for the parent compound in soil:**LOD=0.3 ng g⁻¹ (ppb); LOQ=1.5 ng g⁻¹ (ppb) (Appendix I, p. 122).**Detection limits (LOD, LOQ) for the transformation products in soil:** n/a**II. RESULTS AND DISCUSSION**

APPLICATION MONITORS: The recoveries in the field application monitors were 99.5% and 94.0% of the theoretical amount based on the field application calculations at Site 1 (MS) and Site 2 (CA), respectively (Tables 6-7, pp. 43-44). Corresponding recoveries as a percent of the label rate were 125.7% and 121.7%.

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RECOVERY FROM FIELD SPIKES: The recovery from the field spiked samples was 101% of the applied (both field sites, corrected recoveries; p. 30; Appendix I, Table 1, p. 130).

MASS ACCOUNTING: Because this field study was conducted using non-radio-labeled material, total mass accounting was not calculated.

Table 7. Concentration of Aminopyralid Residues (ng/g) at Site 1 (MS).

Com- pound	Soil depth (cm)	Sampling times (days)							
		0	8	15	29	57	93	122	183
XDE- 750	0-15	144.2	52.8	119.7	27.3	17.4	7.6	17.9	(0.5)
	reps	57.21	43.4	65.8	44.2	5.5	10.3	12.4	2.4
		32.3	46.0	92.4	36.0	37.6	6.2	8.4	(0.7)
	mean	77.9	47.4	92.6	35.8	20.2	8.0	12.9	1.2
	15-30	—	ND	ND	ND	ND	ND	ND	ND
	30-45	—	ND	ND	ND	ND	ND	ND	ND
	45-60	—	—	—	—	—	—	—	—
	60-75	—	—	—	—	—	—	—	—
	75-90	—	—	—	—	—	—	—	—
Total XDE- 750	Total Pro- file	77.9	47.4	92.6	35.8	20.2	8.0	12.9	1.2

Reviewer-calculated means based on replicate data from Appendix G, pp. 100-103. Values in parens are below the LOQ.

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Table 8. Concentration of Aminopyralid Residues (ng/g) at Site 2 (CA).

Com- pound	Soil depth (cm)	Sampling times (days)							
		0	9	15	22	65	91	126	182
XDE- 750	0-15	64.4	104.2	54.1	52.8	5.9	(1.0)	(0.4)	ND
	reps	61.9	70.8	44.3	38.6	12.0	(0.5)	ND	(0.4)
		68.1	83.7	60.2	53.5	11.2	(0.8)	(0.7)	(0.7)
	mean	64.8	86.2	52.9	48.3	9.7	(0.8)	(0.4)	(0.3)
	15-30		1.5	2.3	9.3	(0.5)	ND		
	reps	—	1.6	2.9	5.6	ND	(0.5)	ND	ND
			1.7	1.8	(0.4)	ND	ND		
	mean		1.6	2.4	5.1	<LOQ	<LOQ		
	30-45			3.7	1.6	ND			
	reps	—	ND	ND	ND	(1.4)	ND	ND	ND
				ND	ND	ND			
	mean			<LOQ	<LOQ	<LOQ			
	45-60			2.5	(0.9)	(0.7)		(1.4)	
	reps	—	—	ND	ND	ND	ND	ND	ND
				ND	ND	ND	ND	ND	ND
	mean			<LOQ	<LOQ	<LOQ		<LOQ	
	60-75				(0.9)	1.9			
	reps	—	—	ND	ND	ND	ND	—	ND
					ND	ND	ND		
	mean				<LOQ	0.6			
	75-90			(0.5)		(0.6)	(0.7)		
	reps	—	—	ND	ND	ND	ND	—	ND
				ND	ND	ND	ND		
	mean			<LOQ		<LOQ	<LOQ		
Total XDE- 750	Total Pro- file	64.8	87.8	57.3	54.5	11.4	1.2	0.9	0.3

Reviewer-calculated means based on replicate data from Appendix G, pp. 104-107. Values in parens are below the LOQ.

PARENT COMPOUND:

In surface (0-15 cm) soil at MS, aminopyralid dissipated from a mean of 77.9 ng/g (range of 32.3-144.2 ng/g) at day 0 to 47.4 ng/g by day 8, increased to a mean of 92.6 ng/g (range of 65.8-119.7 ng/g) by day 15, then decreased to 35.8 ng/g by day 29 and decreased, with variability, to <LOQ by day 183 (Appendix G, Table 1). In surface (0-15 cm) soil at CA, aminopyralid was a mean of 64.8 ng/g at day 0, increased to 86.2 ng/g (range of 70.8-104.2 ng/g) by day 9, then decreased to 48.3 ng/g by day 22 and was below the LOQ by day 91. (Appendix G, Table 2). The residues of aminopyralid were primarily detected in the top 0- to 15-cm soil layer at MS; and in the top 0- to 30-cm soil layers, with detections through the 75- to 90-cm layer, at CA.

Aminopyralid dissipated in surface soil (0- to 15-cm depth only) under terrestrial field conditions with half-lives of 32.1 days ($r^2 = 0.81$) for the MS site and 20.0 days ($r^2 = 0.87$) for the CA site. However, both temporal and inter-replicate data variability makes the half-lives of questionable value. Half-lives were calculated in Excel (2000) using linear regression analysis performed on a plot of ln-transformed aminopyralid concentrations (0-15 cm soil depth) vs. time and the equation $t_{1/2} = -\ln 2 / k$, where k is the rate constant.

The registrant-calculated first-order field half-life and dissipation time for 90% of the initial concentration (DT_{90}) of aminopyralid (for residues in the total soil profile) under terrestrial field conditions were (Figures 4-5, pp. 70-71):

Site 1 (MS)

Half-life = 34 days

 $DT_{90} = 114$ days

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Site 2 (CA) Half-life = 26 days DT₉₀ = 85 days

The dissipation pattern was rapid and complete by 6 months at MS and 4 months at CA.

TRANSFORMATION PRODUCTS: Although transformation products were not monitored, there were no major degradates detected in the laboratory metabolism studies.

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

Table 9: Dissipation Routes of Aminopyralid under Field Conditions.

Route of dissipation	MS Site: % of applied amount (at the end of study)	CA Site: % of applied amount (at the end of study)
Accumulation (residues) in soil/ carry over	1.6%	0.6%
Transformation (% of transformation products)	n/a	n/a
Leaching, if measured	n/a	n/a
Volatilization, if measured	n/a	n/a
Plant uptake, if measured	n/a	n/a
Run off, if measured	n/a	n/a
Total	1.6%	0.6%

VOLATILIZATION: The concentration of applied aminopyralid lost through volatilization was not determined.

PLANT UPTAKE: N/A.

LEACHING: At Site 1 (MS), aminopyralid was not detected below the 0-15 cm segment. At Site 2 (CA), aminopyralid was not detected above the LOQ below the 60-75 cm segment.

RUN OFF: The concentration of aminopyralid in runoff was not determined.

RESIDUE CARRYOVER:

The DT₉₀ value was 114 days at Site-1 (MS) (Figure 4, p. 70). After 183 days, 1.6 % of the applied aminopyralid was detected at MS, and has no potential to carryover into the following season.

The DT₉₀ value was 85 days at Site 2 (CA) (Figure 5, p. 71). After 182 days, 0.6 % of the applied aminopyralid was detected at CA, and has no potential to carryover into the following season.

SUPPLEMENTARY STUDY RESULTS:

Long-term frozen storage stability study (1) results show aminopyralid to be stable (99.9%) in soil stored frozen for 6.5 mo.s (194 days). The longest period that samples were stored for this field dissipation study was 316 days for the MS site and 272 days for the CA site. Preliminary results. Stability data were not reported in the study report.

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III. STUDY DEFICIENCIES:

This study is classified as supplemental. Both temporal and inter-replicate data variability (especially at times 0, 15 and 57 days at the MS site and 9 days at the CA site) make the half-lives of questionable value. In addition, the laboratory storage stability study was not conducted for a period of time equal to or exceeding the length of time that test samples were stored frozen. The laboratory storage stability study was in progress and preliminary results were not available in the study report. Storage stability data should be provided for a length of time equal to or exceeding the maximum storage interval for the test samples (MS: 316 days, CA: 272 days; Appendix I, Table 1, p. 130). The study authors stated in the supplementary study results section of this DER that aminopyralid was stable in soil samples stored frozen for up to at least 194 days, with a recovery of 99.9%.

IV. REVIEWER'S COMMENTS:

1. The study authors used the highest observed mean starting soil rate as the true time 0 value. Therefore, for the MS site, the starting rate was designated as 152.4 g a.e./ha, based on the mean Day 0 soil concentration (Table 8, p. 45) and for the CA site, the starting rate was designated as 146.0 g a.e./ha, based on the mean value obtained from application verification monitors (Table 7, p. 44). For comparison purposes, the reviewer notes that for the MS site, the application rate was calculated to be 151.6 g a.e./ha based on the spray solution concentration and the discharge rate (Appendix B, Table 1, p. 75) and was determined to be 150.8 g a.e./ha based on application verification monitors (Table 6, p. 43) and for the CA site, the application rate was calculated to be 155.3 g a.e./ha based on the spray solution concentration and the discharge rate (Appendix B, Table 2, p. 76) and was determined to be 132.4 g a.e./ha based on Day 0 soil concentrations (Table 11, p. 56). However, based on concentrations found in soil samples from day 0, the application was likely not uniform at the MS site. In day 0 soil samples, aminopyralid was detected at 144.2, 57.21 and 32.2 ng/g in the replicate plots. While the rate appeared to be more uniform at the CA site based on time 0 concentrations in soil (61.9-68.1 ng/g), at that site, higher concentrations were detected for all three replicates at the next sampling interval (104.2, 70.8, 83.7 ng/g; 9 days). Thus, both temporal and inter-replicate data variability were observed and impacted the half-lives determined at each site.
2. The target application rate corresponds to 125% (an exaggerated rate) of the proposed maximum label rate. Based on application monitors (shallow soil pans) used to verify the application rates, the achieved rate was 82-119% of the target at MS (mean of 99.5%), and 72-117% at CA (mean of 94.0%). Field studies should not be conducted at exaggerated rates, but at the maximum label rate. Based on concentrations in soil samples, as discussed above in comment 1, the application was likely not uniform across all plots.
3. Mean recovery of aminopyralid from fortified samples was 87 +/- 11% from soil samples fortified over the range of 1.5-1500 ng/g (Appendix I, Table 3, p. 132).
4. Random soil sampling was not utilized; a systematic sampling was conducted.

V. REFERENCES:

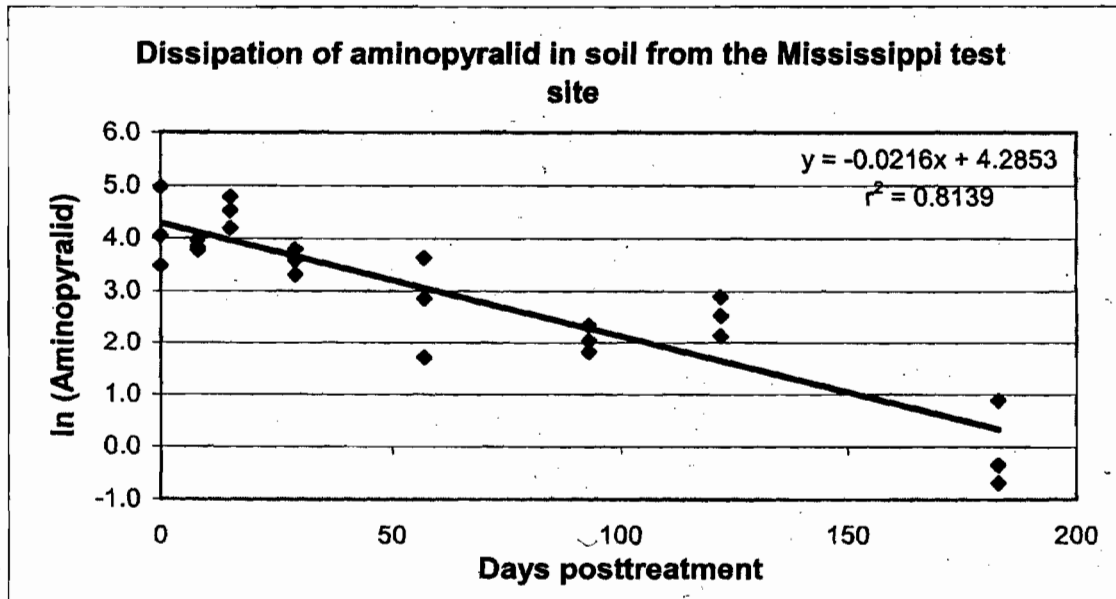
1. Lindsay, D.A. 2004. Frozen Storage Stability of XDE-750 in Soil - Interim Report. 030002. Unpublished report of Dow AgroSciences LLC.

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Chemical Name Aminopyralid MS site
 PC Code 005100 Half-life (days) = 32.1
 MRID 46235734
 Guideline No. 164-1

Days posttreatment	Aminopyralid (ng/g)	Ln (Aminopyralid)
0	144.236	4.971
0	57.214	4.047
0	32.263	3.474
8	52.784	3.966
8	43.367	3.770
8	45.985	3.828
15	119.665	4.785
15	65.849	4.187
15	92.406	4.526
29	27.319	3.308
29	44.223	3.789
29	35.998	3.583
57	17.359	2.854
57	5.531	1.710
57	37.560	3.626
93	7.613	2.030
93	10.314	2.334
93	6.158	1.818
122	17.936	2.887
122	12.449	2.522
122	8.421	2.131
183	0.503	-0.687
183	2.424	0.885
183	0.713	-0.338

Data obtained from Appendix G, Table 1, pp. 100-102 of the study report.



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Chemical Name Aminopyralid
 PC Code 005100
 MRID 46235734
 Guideline No. 164-1

Calculation of means: MS site
 Aminopyralid (ng/g)

Days posttreatment	0-15 cm depth	
	Replicates	Mean
0	144.236	
0	57.214	
0	32.263	77.9
8	52.784	
8	43.367	
8	45.985	47.4
15	119.665	
15	65.849	
15	92.406	92.6
29	27.319	
29	44.223	
29	35.998	35.8
57	17.359	
57	5.531	
57	37.560	20.2
93	7.613	
93	10.314	
93	6.158	8.0
122	17.936	
122	12.449	
122	8.421	12.9
183	0.503	
183	2.424	
183	0.713	1.2

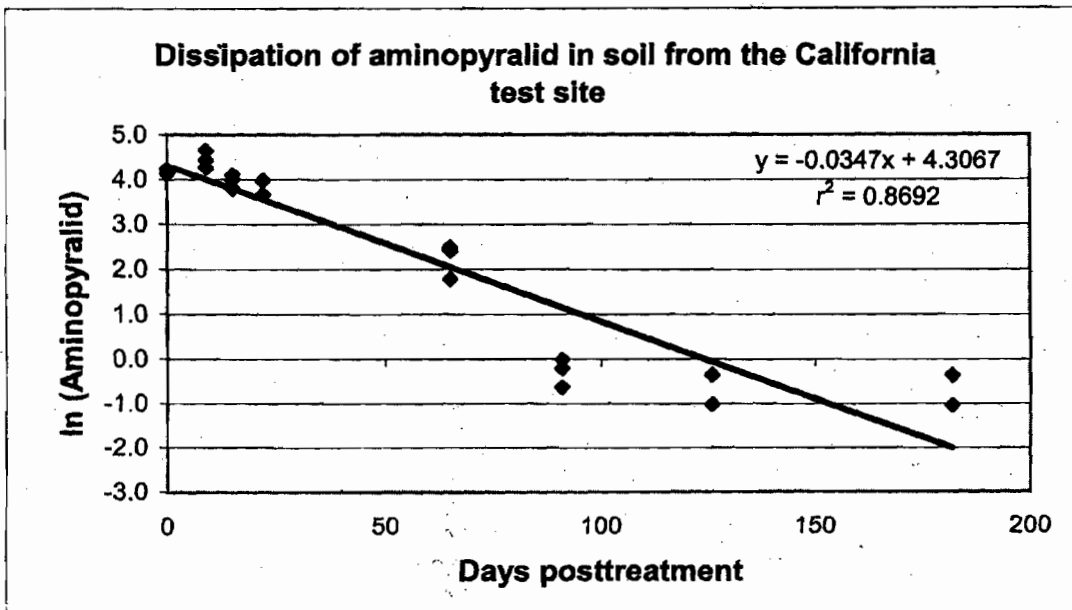
Data obtained from Appendix G, Table 1, pp. 100-103 in the study report.

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Chemical Name Aminopyralid CA site
 PC Code 005100 Half-life (days) = 20.0
 MRID 46235734
 Guideline No. 164-1

Days posttreatment	Aminopyralid (ng/g)	Ln (Aminopyralid)
0	64.354	4.164
0	61.887	4.125
0	68.094	4.221
9	104.164	4.646
9	70.776	4.260
9	83.674	4.427
15	54.068	3.990
15	44.349	3.792
15	60.238	4.098
22	52.837	3.967
22	38.566	3.652
22	53.490	3.979
65	5.909	1.776
65	12.047	2.489
65	11.154	2.412
91	0.983	-0.017
91	0.532	-0.631
91	0.811	-0.209
126	0.359	-1.024
126	ND	
126	0.699	-0.358
182	ND	
182	0.352	-1.044
182	0.696	-0.362

Data obtained from Appendix G, Table 2, pp. 104-106 of the study report.



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Chemical Name Aminopyralid
 PC Code 005100
 MRID 46235734
 Guideline No. 164-1

Calculation of means: CA site
 Aminopyralid (ng/g)

Days posttreatment	0-15 cm depth		15-30 cm depth		30-45 cm depth		45-60 cm depth		60-75 cm depth		75-90 cm depth	
	Replicates	Mean	Replicates	Mean	Replicates	Mean	Replicates	Mean	Replicates	Mean	Replicates	Mean
0	64.354											
0	61.887											
0	68.094	64.8										
9	104.164		1.519									
9	70.776		1.566									
9	83.674	86.2	1.724	1.6								
15	54.068		2.32		3.75		2.513					
15	44.349		2.902		0.00		0.000					
15	60.238	52.9	1.848	2.4	0.00	1.2	0.000	0.8				
22	52.837		9.31		1.63		0.882		0.913			
22	38.566		5.597		0.00		0.000		0.000			
22	53.490	48.3	0.425	5.1	0.00	0.5	0.000	0.3	0.000	0.304		
65	5.909		0.464		0.00		0.703		1.889			0.627
65	12.047		0.000		1.44		0.000		0.000			0.000
65	11.154	9.7	0.000	0.2	0.00	0.5	0.000	0.2	0.000	0.630		0.209
91	0.983		0.000									0.702
91	0.532		0.485									0.000
91	0.811	0.8	0.000	0.2								0.000 0.234
126	0.359						1.439					
126	0.000						0.000					
126	0.699	0.4					0.000	0.5				
182	0.000											
182	0.352											
182	0.696	0.3										

Data obtained from Appendix G, Table 2, pp. 104-106 in the study report.

.000 = Not detected

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