

DP Barcode :D162977  
:D162897  
:D159912  
:D159805  
:D155563  
:D161966

JUN 27 1991

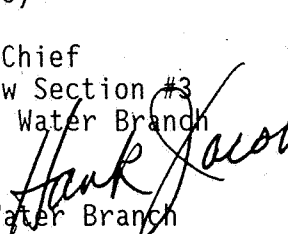
PC Code No.: 108501

Date Out of EAB: \_\_\_\_\_

TO: Lois Rossi/Terri Stowe  
Product Manager 74  
Registration Division (H7505C)

FROM: Akiva D. Abramovitch, Ph.D., Chief  
Environmental Chemistry Review Section #3  
Environmental Fate and Ground Water Branch

THRU: Hank Jacoby, Chief  
Environmental Fate & Ground Water Branch  
Environmental Fate & Effects Division (H7507C)



Attached, please find the EFGWB review of...

Reg./File # : Submission No. S393409, S388375, S388218, S381787,

Common Name : Pendimethalin.

Type Product : Herbicide.

Product Name : Prowl, Squadron, Accotab, Go-Go San, Herbadox, Sipaxol,  
Stomp, Wax-Up, AC 92,553.

Company Name : American Cyanamid Company.

Purpose : Addendum to the pendimethalin registration standard.

Date Received: 9/12/90 EFGWB #: 91-0312 Time (days): 22

90-0875  
91-0303  
91-0366  
91-0484  
91-0492  
91-0438

Deferrals to:

\_\_\_ EEB/EFED                      \_\_\_ DEB/HED                      \_\_\_ TB1/HED

\_\_\_ SIPS/EFED                      \_\_\_ OREB/HED                      \_\_\_ TB2/HED

Use this form for individual studies & to submit pesticide applications.



United States Environmental Protection Agency  
Office of Pesticide Programs  
Washington, DC 20460  
**Data Review Record**  
Confidential Business Information - Does not contain  
National Security Information (E.O. 12065)

Pack Number

Date Received

1. Product Name

Chemical Name

FENDIETHALIN (1990)

2. Identifying Number	3. Record Number	4. Action Code	5. MRID/ Accession Number	6. Study Guideline or Narrative
# 2107	228458	660	00152762	GLN 117-2
		660	00152764	GLN 161-2
		660	40185104	GLN 162-1
		660	40185105	GLN 163-2
		660	40313501	GLN 163-2
		660	00152765	GLN 162-1
		660	00152766	GLN 162-2
		660	00152767	GLN 165-4

7. Reference No.	8. Date Rec'd (EPA)	9. Prod/Review Mgr/DCI	10. PM/RM Team No.	11. Date to HED/EFED/RD/BEAD	12. Proj Return Date	13. Date Returned to RD/SRRD
1	7/15/90	T. STOWE	74	9/11/90	12/11/90	

Instructions

PLEASE REVIEW ~~THE~~ <sup>ABOVE</sup> DATA IN ORDER TO REVIEW  
THE 2 AQUATIC (SEDIMENT) DISSOLUTION STUDY PREVIOUSLY  
SUBMITTED, (SEE A-FOURED REVIEW),

This Section Applies to Review of Studies Only

14. Check Applicable Box	15. No. of Individual Studies Submitted
<input type="checkbox"/> Adverse 6(a)(2) Data (405) <input type="checkbox"/> Special Review Data (870) <input checked="" type="checkbox"/> Generic Data (Reregistration) (660) <input type="checkbox"/> Product Specific Data (Reregistration) (655)	8
16. Have any of the above studies (in whole or in part) been previously submitted for review?	17. Related Actions
<input type="checkbox"/> Yes (Please identify the study(ies)) <input checked="" type="checkbox"/> No	

18.	To	Type of Review	19. Reviews Also Sent to	20. Data Review Criteria
HED		Science Analysis & Coordination	<input type="checkbox"/> SAC <input type="checkbox"/> PC	A. Policy Note No. 31 <input type="checkbox"/> 1 = data which meet 6(a)(2) or meet 3(c)(2)(B) flagging criteria <input checked="" type="checkbox"/> 2 = data of particular concern from registration standard <input type="checkbox"/> 3 = data necessary to determine tiered testing requirements
		Toxicology/HFA	<input type="checkbox"/> TOX/HFA <input type="checkbox"/> PL	
		Toxicology/IR	<input type="checkbox"/> TOX/IR	
		Dietary Exposure	<input type="checkbox"/> DEB <input type="checkbox"/> EA	
		Nondietary Exposure	<input type="checkbox"/> NDE <input type="checkbox"/> AC	
EFED		Ecological Effects	<input type="checkbox"/> EEB <input type="checkbox"/> BA	
	<input checked="" type="checkbox"/>	Environmental Fate & Groundwater	<input type="checkbox"/> EFGWB	
SRRD		Special Review		
		Reregistration		
		Generic Chemical Support	<input type="checkbox"/> SR	
RD		Insecticide-Rodenticide	<input type="checkbox"/> RER	B. Section 18 <input type="checkbox"/> 1 = data in support of section 3 in lieu of section 18
		Fungicide-Herbicide	<input type="checkbox"/> GSC	
		Antimicrobial		
		Product Chemistry	<input type="checkbox"/> IR	
		Precautionary Labeling	<input type="checkbox"/> FH	
BEAD		Economic Analysis	<input type="checkbox"/> AM	C. Inert Ingredients <input type="checkbox"/> 1 = data in support of continued use of List 1 inert
		Analytical Chemistry		
		Biological Analysis		
<input type="checkbox"/> Confidential Statement of Formula (EPA Form 8570-4) Attached (Trade Secrets)			<input type="checkbox"/> Label Attached	

2

1. CHEMICAL: Common name:

Pendimethalin.

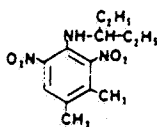
Chemical name:

N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitrobenzenamine.

Trade name(s):

Prowl, Squadron, Accotab, Go-Go San, Herbadox, Sipaxol, Stomp, Wax-Up, AC 92,553.

Structure:



Formulations:

EC.

Physical/Chemical properties:

Molecular formula: C<sub>13</sub>H<sub>19</sub>N<sub>3</sub>O<sub>4</sub>.

Molecular weight: 281.3.

Physical state: orange-yellow crystals.

Melting Point: 54-58 C.

Vapor pressure: 4.0 mPa (25 C).

Solubility: <0.5 ppm in water (20 C), 700 g/L acetone, 77 g/L propan-2-ol, 628 g/L xylene.

2. TEST MATERIAL: Studies 1-8, and 10.: Active ingredient. Studies 9-11: End-Use Product.

3. STUDY/ACTION TYPE: Addendum to the pendimethalin registration standard.

4. STUDY IDENTIFICATION: [Studies by Lee (1989) and Smyth, et al. (1990) at end of list

Barringer, D.F. 1986. Prowl herbicide (AC 92,553): Isolation and identification of residues from bluegill sunfish exposed to radiolabeled pendimethalin in a flow-through study. Project No. 0463. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (00158235)

Forbis, A.D. 1986. Uptake, depuration, and bioconcentration of <sup>14</sup>C-AC 92,553 by bluegill sunfish (Lepomis macrochirus). Unpublished study

performed by Analytical Bio-Chemistry Laboratories, Inc., Columbia, MO, and submitted by American Cyanamid Company, Princeton, NJ. (00156726)

Lavin, M. and W. Cranor. 1987a. Aerobic soil metabolism of [<sup>14</sup>C] pendimethalin. Final Report No. 33731. Unpublished study performed and submitted by Analytical Biochemistry Laboratories, Inc., Columbia, MO. (40185104)

Lavin, M. and W. Cranor. 1987b. Anaerobic soil metabolism of [<sup>14</sup>C]pendimethalin. Final Report No. 33731. Unpublished study performed by Analytical Biochemistry Laboratories, Inc., Columbia, MO, and submitted by American Cyanamid Company, Princeton, NJ. (40185105)

Mangels, G. 1985a. Prowl Herbicide, pendimethalin (AC 92,553): Adsorption/desorption studies. Report No. PD-M Volume 22-37. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (00153765)

Mangels, G. 1985b. Prowl Herbicide, pendimethalin (AC 92,553): Photodegradation in soil. Report No. PD-M Volume 22-35. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (00153764)

Manuel, A. 1980. Analysis for residues of Prowl in soil and in water from Prowl treated rice fields. Laboratory report No. CY 17. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (41245601)

Sanders, P. 1985a. Prowl Herbicide, pendimethalin (AC 92,553): Photodegradation in water. Report No. PD-M Volume 22-36. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (00153763)

Sanders, P. 1985b. Prowl Herbicide, pendimethalin (AC92,533): Volatilization from soil. Project No. 0166, Report No. PD-M Volume 22-38. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (00153766)

Sanders, P. 1988. Pendimethalin (AC 92,553): Anaerobic aquatic degradation in soil from a rice field. Laboratory Report Number PD-M 25-25. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (40813501)

Steller, W.S., and M. Smyth. 1989. Pendimethalin (AC 92,553): Residues of AC 92,553 in soil (postemergence, sandy loam); Kerman, California, 1988. Laboratory Report No. C-3280/Protocol No. PR88CA01. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (41725204)

Steller, W.S., and M. Smyth. 1990. Pendimethalin (AC 92,553): Residues of AC 92,553 in soil (postemergence, sandy); Brawley, California, 1988.

Laboratory Report No. C-3281/Protocol No. PR88CA02. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (41725205)

Lee, T.M. 1989. Pendimethalin (CL 92, 553): Confined accumulation studies of carbon-14 labeled CL 92,553 in lettuce, wheat, carrots, radishes, and snap beans as representative rotational crops (Princeton, New Jersey). Laboratory project ID: CY 38. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ. (41806801)

Smyth, M., D. Koch, and J. Smith. 1990. Pendimethalin (AC 92,553): Freezer stability in soil. Laboratory Report No: C-3467. Performed by American Cyanamid Company, Princeton, NJ, and Analytical Bio-Chemistry Laboratories, Inc. Columbia, MO. and submitted by American Cyanamid Company, Princeton, NJ. (41725206)

5. REVIEWED BY:

H. Manning  
Microbiologist  
EFGWB/EFED/OPP  
Review Section #3

Signature: H. Manning

Date: JUN 27 1991

6. APPROVED BY:

Akiva Abramovitch  
Chief  
EFGWB/EFED/OPP  
Review Section #3

Signature: Akiva Abramovitch

Date: JUN 27 1991

7. CONCLUSION:

7.1 Study No. 1- Photodegradation in Water (00153763)

1. The study is not acceptable at this time to satisfy the 161-2 data requirement, but it may provide supplementary information. The study may be made acceptable if the registrant provides detailed information about the artificial light source and data from earlier sampling intervals showing the pattern of degradation of pendimethalin.

2. Pendimethalin photodegraded with a half-life of 21 days in water at 25 C continuously irradiated with artificial light (xenon arc lamp) for 28 days. There were up to 18 uncharacterized degradates present at = or < 6% of the applied radioactivity in the samples irradiated for 28 days. Pendimethalin did not degrade in the dark controls incubated at 25 C for 28 days.

7.2 Study No. 2-Photodegradation on Soil (00153764)

1. The study is not acceptable at this time to satisfy the 161-3 data requirement, but it may provide supplementary information. The study may be made acceptable if the registrant provides detailed information about

the artificial light as compared to natural sunlight.

2. Pendimethalin did not photodegrade on sandy loam soil that was continuously irradiated with artificial light (xenon arc lamps) for 4 weeks at 25 C. Pendimethalin also did not degrade in the dark controls incubated at 25 C for 4 weeks.

#### 7.3 Study No. 3-Aerobic Soil Metabolism (40185104)

1. The study is not acceptable at this time to satisfy the 162-1 data requirement, but may provide supplementary information. The study may be made acceptable if the registrant identifies the degradates with  $R_f$  values of 0.15 present at 0.6% of the applied radioactivity (0.01 ppm) and 0.07 at 2.2% (0.04 ppm) in the soil extracts, and volatiles in the ethylene glycol trap at 1.0% (0.02 ppm).

2. Pendimethalin degraded with a half-life of 1322 days in sandy loam soil incubated in the dark at 24.8 +/- 0.8 C and 53-62% of 0.33 bar moisture capacity. The nonvolatile degradates identified were 2,6-dinitro-3,4-xylidine (CL 84, 846); 4-[(1-ethylpropyl)amino]-3,5-dinitro-o-toluic acid (CL 99,900); and 4-[(1-ethylpropyl)amino]-2-methyl-3,5-dinitro-benzyl alcohol (CL 202,347).

#### 7.4 Study No. 4-Anaerobic Soil Metabolism (40185105)

1. The study is not acceptable at this time to satisfy the 162-2 data requirement, but may provide supplementary information. The study may be made acceptable if the registrant identifies/characterizes the radioactivity in the acetonitrile extracts ("remainder") present at 0.05 ppm (2.7% of the applied radioactivity) in the "aqueous" phase present at 0.05 ppm (2.7% of applied).

However, this study may be replaced by an acceptable Anaerobic Aquatic Metabolism (162-2) study (see Study No. 5, below), since the aquatic study may be substituted for the soil study.

2. Pendimethalin was relatively stable in sandy loam soil that was anaerobically (flooding plus nitrogen atmosphere) incubated in the dark for 60 days following an aerobic incubation period of 30 days. Three nonvolatile degradates were identified during the aerobic incubation period: 2,6-dinitro-3,4-xylidine (CL 84,846), 4-[(1-ethylpropyl)amino]-2-methyl-3,5-dinitro-benzyl alcohol (CL 202,347), and 4-[(1-ethylpropyl)amino]-3,5-dinitro-o-toluic acid (CL 99,900).

#### 7.5 Study No. 5-Anaerobic Aquatic Metabolism (40813501)

1. The study is not acceptable at this time to satisfy the 162-3 data requirement, but it may provide supplementary information. The study was not done according to Subdivision N Guidelines, therefore, it is not repairable and must be repeated.

2. Pendimethalin degraded in anaerobic silt loam soil, which had been treated and incubated aerobically for 1 week prior to establishment of anaerobic (flooding and nitrogen) conditions. Parent pendimethalin was

50-58% of the nominal application at 9 weeks posttreatment. Degradates were not identified.

7.6 Study No. 6- Leaching and Adsorption/Desorption (00153765)

1. The study is not acceptable to satisfy the 163-1 data requirement because the soils were sieved through a 0.5 mm screen sieve rather than a 2.0 mm sieve, thereby reducing the apparent mobility of pendimethalin. The study must be repeated.

7.7 Study No. 7- Laboratory Volatility (00153766)

1. The study is acceptable and fulfills the 163-2 data requirement.

2. Pendimethalin slowly volatilized from dry sandy loam soil incubated at 21 C, with a maximum air concentration of 0.54 ug/m<sup>3</sup> and a volatility rate of 5.0 x 10<sup>-5</sup> ug/cm<sup>2</sup>/hour when averaged over the initial 4.7-day sampling period. The cumulative loss of pendimethalin after 8.7 days was 3.3 ug, which was 0.05% of the applied. In moist sandy loam soil, pendimethalin volatilized at a maximum air concentration of 31 ug/m<sup>3</sup> and a volatility rate of 2.1 x 10<sup>-3</sup> ug/cm<sup>2</sup>/hour when averaged over the initial 24-hour sampling period.

A Field Volatility study (163-3) is not required because the acute toxicities are in Toxicity Category III or IV.

7.8 Study No. 8- Accumulation in Fish (00158235, 00156726)

1. The study is acceptable and fulfills the 165-4 data requirement.

2. Pendimethalin residues accumulated in bluegill sunfish exposed to 3.0 ppb of pendimethalin, with maximum mean bioconcentration factors of 1400X, 5800X, and 5100X for edible, nonedible, and whole fish tissues, respectively. Pendimethalin comprised 68.2-80.8% of the recovered radioactivity, and the degradate 4-[(1-ethylpropyl)amino]-2-methyl-3,5-dinitro-benzyl alcohol (CL 202,347) was 2.0-3.1% of the recovered activity. Depuration was rapid, with 87-91% of the accumulated [<sup>14</sup>C]residues eliminated from the fish tissues by day 14 of the depuration period.

7.9 Study No. 9- Terrestrial Field Dissipation (41725204)

1. The study is not acceptable at this time to satisfy the 164-1 data requirement, but it may provide supplementary information. The study may be made acceptable for one of the two sites required for California if the registrant fully discusses the extraction procedure, isolation and quantification of degradates (at all sampling intervals).

2. Pendimethalin residues dissipated with a half-life of approximately 34 days from sandy loam soil located in an almond orchard in California that was treated with pendimethalin (Prowl, 4 lb/gal EC) at 4 lb ai/A. Pendimethalin residues did not appear to leach into soil horizons below 6 inches.

7.10 Study No. 10-Terrestrial Field Dissipation (41725205)

1. The study is unacceptable and cannot be used to satisfy the 164-1 data requirement. The data do not provide sufficient information to predict the fate of pendimethalin or its degradates. The data were too variable, the soil cores were not properly identified, and the sampling protocol was inappropriate. A new study is needed.

7.11 Study No. 11- Aquatic Field Dissipation (41245601)

1. Both the CA and LA portions of the study are unacceptable and cannot be used to satisfy the 164-2 data requirement. The data do not provide sufficient information to predict the fate of pendimethalin or its degradates. The sampling intervals were inadequate and the data too variable to assess the decline of pendimethalin. A new study is needed.

7.12 Study No. 12- Confined Rotational Crop (41806801)

1. The study is not acceptable at this time to satisfy the 165-1 data requirement, but may provide supplementary information. The study may be made acceptable if the registrant adequately identifies pendimethalin residues in the soil and crops.

2. [<sup>14</sup>C]Pendimethalin residues accumulated in rotational lettuce, snap beans, radishes, carrots, and wheat that were planted 30-365 days after small field plots of sandy loam soil were treated with 3,4-dimethyl-labeled [<sup>14</sup>C]pendimethalin at approximately 0.75 and 1.64 lb ai/acre (nominal rate 2 lb ai/A). In general, total [<sup>14</sup>C]residues were greatest in the tissue of crops planted at 30 days posttreatment and decreased as the plants matured. The majority (58.6-91.9%) of the [<sup>14</sup>C]residues in the plants were extractable, and the majority of the extractable residues were not identified.

7.13 Study No. 13- Freezer Storage Stability (Ancillary) (41725206)

1. Freezer storage stability studies are not specifically required by Subdivision N Guidelines, but are necessary when storage of samples occurs before analysis.

2. Pendimethalin (purity 99.5%) was stable when stored frozen (-15 to -20 C) in uncharacterized "composite control" soil at 0.5 ppm for up to 2 years. Recovery of pendimethalin from soil samples that were stored up to 2 years was 81-100%.

Preliminary Environmental Fate Assessment:

While only one fate study in this review was acceptable (Bioaccumulation in Fish) and many of the other studies may provide only supplemental information, a preliminary assessment of the environmental fate of pendimethalin may be made.

The major degradative pathway appears to be through photodegradation in water (t<sub>1/2</sub>= 21 days). Pendimethalin was stable to all other degradative processes: hydrolysis at pH 5, 7, and 9, photodegradation on soil, aerobic



soil metabolism ( $t_{1/2}$ = 1322 days), and anerobic soil metabolism (98.0% of parent remaining after 60 days). Pendimethalin in field dissipation studies was moderately persistent and relatively immobile ( $t_{1/2}$ = 34 days; no leaching below 6 inches). Accumulation of residues occurred in rotated lettuce, snap beans, radishes, carrots, and wheat using rotation interval of 30-365 days. Bioaccumulation in bluegill sunfish was 1400X (edible tissue), 5800X (nonedible), and 5100X (whole fish).

**NOTE:** There is a large discrepancy between the half-life in the laboratory aerobic soil study ( $T_{1/2}$ = 1322 days) and the half-life during terrestrial field dissipation ( $T_{1/2}$ = 34 days). The difference is important since hydrolysis and photodegradation on soil did not contribute significantly to the degradation of pendimethalin in laboratory studies.

#### 8.0 RECOMMENDATIONS:

8.1 Inform the registrant that the following studies are acceptable and fulfill the data requirements:

- Hydrolysis (Reviewed for Reg. Std. 3/85)
- Laboratory Volatility (00153766)
- Bioaccumulation in Fish (00158235, 00156726)
- Freezer Storage Stability (Ancillary) (41725206)

8.2 Inform the registrant that the studies shown below, while unacceptable at this time, may provide supplemental information. They may be made acceptable if the information requested in the DER is supplied:

- Photodegradation in Water (00153763)
- Photodegradation on Soil (00153764)
- Aerobic Soil Metabolism (40185104)
- Confined Rotational Crop (41806801)
- Anaerobic Soil Metabolism (40185105)
- Terrestrial Field Dissipation (41725204)

8.3 Inform the registrant that the following studies were not submitted, but are required:

- Aerobic Aquatic Metabolism (162-4)
- Accumulation in Irrigated Crops (165-3)
- Field Rotational Crop (165-2) [Confined had accumulated residues]

8.4 Inform the registrant that the following studies were reviewed and judged unacceptable and must be repeated:

- Adsorption/Desorption (00153765)
- Terrestrial Field Dissipation (41725205)
- Aquatic Field Dissipation (41245601)
- Anaerobic Aquatic Metabolism (40813501)

8.5 Inform the registrant that the following studies are reserved:

- Long Term Terrestrial Field Dissipation (164-5)

- Spray Drift data (201-1, 202-1)
- Ground Water Monitoring data (166-1, -2, -3)
- Surface Water Monitoring data (167-1, -2)

9. BACKGROUND:

A. Introduction- The studies reviewed above were submitted in response to the Registration Standard.

B. Directions for Use

Pendimethalin is a dinitroaniline herbicide registered for use on terrestrial food + feed, aquatic food, and fiber crops as well as ornamental plants (including Christmas tree plantations) and non-agricultural areas (including lawns, industrial sites, road, utility and railroad rights-of-way, etc.) to control annual grasses and some broadleaf weeds. Pendimethalin is applied as a preemergence and/or postemergence treatment for these crops, either broadcast or as a preemergence application. Single active ingredient formulations include emulsifiable concentrate. Pendimethalin is not toxic to bees or birds, but it is toxic to fish.

10. DISCUSSION OF INDIVIDUAL TESTS OR STUDIES:

Refer to attached reviews.

11. COMPLETION OF ONE-LINER:

The One-Liner was updated and is attached.

12. CBI APPENDIX:

All data reviewed here are considered "company confidential" by the registrant and must be treated as such.

*He has the data*

DP BARCODE: D161966

REREG CASE #

CASE: 819421  
SUBMISSION: S380935

DATA PACKAGE RECORD  
BEAN SHEET

DATE: 03/04/  
Page 1 of

\*\*\* CASE/SUBMISSION INFORMATION \*\*\*

CASE TYPE: REREGISTRATION ACTION: 660 GENERIC DATA REREGIS  
CHEMICAL: 108501 Pendimethalin ( N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitro  
ID#: 108501  
COMPANY:  
PRODUCT MANAGER: 50 JAY ELLENBERGER 703-308-8085 ROOM: CST 4J1  
PM TEAM REVIEWER: TERRI STOWE 703-308-8043 ROOM: CST 3D5  
RECEIVED DATE: 08/17/89 DUE OUT DATE: 12/15/89

\*\*\* DATA PACKAGE INFORMATION \*\*\*

DP BARCODE: 161966 EXPEDITE: Y DATE SENT: 09/11/90 DATE RET.: / /  
DP TYPE: 001 Submission Related Data Package LABEL: N  
ADMIN DUE DATE: ~~12/10/90~~ 03/30/91 CSF: N  
ASSIGNED TO DATE IN DATE OUT  
DIV : EFED 3 / 7 / 91 / /  
BRAN: EFGB 3 / 7 / 91 / /  
SECT: / / / /  
REVR : / / / /  
CONTR: / / / /

\*\*\* DATA PACKAGE REVIEW INSTRUCTIONS \*\*\*

Per review dated 08/22/90 (EFGWB # 900713) the following MRIDs were requested in order to review MRID 41245601 (Submission #S380935, DP Barcode #D154918 - OLTS # 267552) sent to EFGWB on 08/19/90. The following MRIDs were sent on 09/11/90:

MRID #	GLN #	MRID #	GLN #
00153763	161-2	00153764	161-3
40185104	162-1	40185105	162-2
40813501	162-3	00153765	163-1
00153766	163-2	00158235	165-4

In addition to these studies, the following MRIDs have also been submitted:

MRID #	GLN #	
41725204	164-1	Sent 01/03/91
41725205	164-1	Sent 01/03/91
41725206	164-1	Sent 01/03/91
00156726	165-4?	(EEB said to re-route to EFGWB for re-view). Sent 12/21/90.

These MRIDs should make up a complete data package for pendimethalin. Please let me know if I can be of further assistance. Please send a copy of the final review to:

DP BARCODE: D155563

REREG CASE #

CASE: 819421  
SUBMISSION: S381787

DATA PACKAGE RECORD  
BEAN SHEET

DATE: 09/11/90  
Page 1 of 1

\*\*\* CASE/SUBMISSION INFORMATION \*\*\*

CASE TYPE: REREGISTRATION ACTION: GENERIC DATA REREGISTRATION  
CHEMICAL: 108501 Pendimethalin ( N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitro  
ID#: 108501  
COMPANY: 74 LOIS ROSSI 8084 3J2  
PRODUCT MANAGER: ~~XXXXXXXXXXXXXXXXXXXX~~ 703-308-~~XXXX~~ ROOM: CST ~~XXX~~  
PM TEAM REVIEWER: ~~XXXXXXXXXXXX~~ TSTOWE 703-308-~~XXXX~~ ROOM: CST ~~XXX~~  
RECEIVED DATE: 07/18/90 DUE OUT DATE: 11/15/90 8043 3D5

\*\*\* DATA PACKAGE INFORMATION \*\*\*

DP BARCODE: 155563 EXPEDITE: N DATE SENT: 09/11/90 DATE RET.: / /  
DP TYPE: 001 Submission Related Data Package  
ADMIN DUE DATE: 12/10/90 CSF: N LABEL: N  
ASSIGNED TO DATE IN ASSIGNED TO DATE IN  
DIV : EFED 09/12/90 REVR : / /  
BRAN: EFGB / / CONTR: / /  
SECT: / /

\*\*\* DATA PACKAGE REVIEW INSTRUCTIONS \*\*\*

*OLTS Rec # 268458*

*HERB*  

12/10
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*92-0875 / DP Barcode D155563*

Pendimethalin

DP BARCODE: D159805

REREG CASE #

CASE: 819421  
SUBMISSION: S388218

DATA PACKAGE RECORD  
BEAN SHEET

DATE: 12/31/  
Page 1 of

\*\*\* CASE/SUBMISSION INFORMATION \*\*\*

CASE TYPE: REREGISTRATION ACTION: 660 GENERIC DATA REREGIS  
CHEMICAL: 108501 Pendimethalin ( N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitro  
ID#: 108501  
COMPANY: *74 LOIS ROSSI*  
PRODUCT MANAGER: ~~50~~ JAY ELLENBERGER- 703-308-~~8085~~<sup>8080</sup> ROOM: CST 4J1  
PM TEAM REVIEWER: TERRI STOWE 703-308-8043 ROOM: CST 3D5  
RECEIVED DATE: 01/30/86 DUE OUT DATE: / /

\*\*\* DATA PACKAGE INFORMATION \*\*\*

DP BARCODE: 159805 EXPEDITE: Y DATE SENT: 12/31/90 DATE RET.: / /  
DP TYPE: 001 Submission Related Data Package  
ADMIN DUE DATE: 03/31/91 CSF: N LABEL: N  
ASSIGNED TO DATE IN ASSIGNED TO DATE IN  
DIV : EFED *12/31/90* REVR : / /  
BRAN: EFGB / / CONTR: / /  
SECT: / /

\*\*\* DATA PACKAGE REVIEW INSTRUCTIONS \*\*\*

FOR PRIORITY REVIEW: PLEASE REVIEW DATA FOR GLN 165-4 -  
(MRID 00156726) PREVIOUSLY ROUTED TO EEB BY MISTAKE BY  
PM 25 TEAM. A PREVIOUS DATA PACKAGE FOR PENDIMETHALIN  
WAS SENT ON 09/11/90 FOR A COMPREHENSIVE REVIEW. PLEASE  
SEND REVIEW TO: TERRI STOWE  
SRRD/RB (H7508C)  
CRYSTAL STATION 1 - 3RD FLOOR - 33D5

THANK YOU!!

\*\*\* ADDITIONAL DATA PACKAGES FOR THIS SUBMISSION \*\*\*

DP BC BRANCH/SECTION DATE OUT DUE BACK INS CSF LABEL

*EFGB # 91-0303*

DP BARCODE: D159912

REREG CASE #

CASE: 819421  
SUBMISSION: S388375

DATA PACKAGE RECORD  
BEAN SHEET

DATE: 01/03/91  
Page 1 of 1

\*\*\* CASE/SUBMISSION INFORMATION \*\*\*

CASE TYPE: REREGISTRATION ACTION: 660 GENERIC DATA REREGIS  
CHEMICAL: 108501 Pendimethalin ( N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitro  
ID#: 108501  
COMPANY: *74 LOIS ROSSI*  
PRODUCT MANAGER: ~~50 JAY ELLENBERGER~~ *8080* 703-308-~~8085~~ ROOM: CST 4J1  
PM TEAM REVIEWER: TERRI STOWE 703-308-8043 ROOM: CST 3D5  
RECEIVED DATE: 12/07/90 DUE OUT DATE: / /

\*\*\* DATA PACKAGE INFORMATION \*\*\*

DP BARCODE: 159912 EXPEDITE: Y DATE SENT: 01/03/91 DATE RET.: / /  
DP TYPE: 001 Submission Related Data Package LABEL: N  
ADMIN DUE DATE: 04/03/91 CSF: N DATE IN  
ASSIGNED TO DATE IN ASSIGNED TO  
DIV : EFED *01/10/91* REVR : / /  
BRAN: EFGB / / CONTR: / /  
SECT: / /

\*\*\* DATA PACKAGE REVIEW INSTRUCTIONS \*\*\*

FOR IMMEDIATE REVIEW - Please add to previously submitted data packages.  
Please review pendimethalin data for GLN 164-1 (MRIDs 41725204 + 41725205 + 41725206). Please send review and a status report of EFGWB requirements for pendimethalin to:  
Terri Stowe  
SRRD/RB (H7508C)  
CS 1 - 3rd fl. - rm. 33D5

Thank you!!!

\*\*\* ADDITIONAL DATA PACKAGES FOR THIS SUBMISSION \*\*\*

DP BC BRANCH/SECTION DATE OUT DUE BACK INS CSF LABEL

EFGWB # 91-0312

14

DP BARCODE: D162897

REREG CASE #

CASE: 819421  
SUBMISSION: S393409

DATA PACKAGE RECORD  
BEAN SHEET

DATE: 03/26/  
Page 1 of

\*\*\* CASE/SUBMISSION INFORMATION \*\*\*

CASE TYPE: REREGISTRATION ACTION: 665 DATA PROT-REG STND GN DAT  
CHEMICAL: 108501 Pendimethalin ( N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitro  
ID#: 108501  
COMPANY:  
PRODUCT MANAGER: ~~50 JAY ELLENBERGER~~ 703-308-8085 ROOM: CST 4J1  
PM TEAM REVIEWER: TERRI STOWE 703-308-8043 ROOM: CST 3D5  
RECEIVED DATE: 07/18/90 DUE OUT DATE: 10/16/90

\*\*\* DATA PACKAGE INFORMATION \*\*\*

DP BARCODE: 162897 EXPEDITE: Y DATE SENT: 03/26/91 DATE RET.: / /  
DP TYPE: 001 Submission Related Data Package LABEL: N  
ADMIN DUE DATE: 06/04/91 CSF: N  
ASSIGNED TO DATE IN DATE OUT  
DIV : EFED 03/27/91 / /  
BRAN: EFGB / / / /  
SECT: / / / /  
REVR : / / / /  
CONTR: / / / /

\*\*\* DATA PACKAGE REVIEW INSTRUCTIONS \*\*\*

ATTN.: FOR IMMEDIATE REVIEW - PENDIMETHALIN PROTOCOL FOR  
GLN 164-2.

Please review the Pendimethalin protocol for GLN 164-2. I  
have attached both the original protocol and the protocol  
that was modified per EEB's review dated 02/06/90. Please  
send a copy of the review to: Terri Stowe  
SRRD/RB (H7508W)  
Crystal Station I - 3rd fl.

THANK YOU!!

\*\*\* ADDITIONAL DATA PACKAGES FOR THIS SUBMISSION \*\*\*

DP BC	BRANCH/SECTION	DATE OUT	DUE BACK	INS	CSF	LABEL
162891	EEB	03/26/91	06/04/91	Y	N	N

15

DP BARCODE: D162977

REREG CASE #

CASE: 819421  
SUBMISSION: S381787

DATA PACKAGE RECORD  
BEAN SHEET

DATE: 03/  
Page 1 0

\*\*\* CASE/SUBMISSION INFORMATION \*\*\*

CASE TYPE: REREGISTRATION ACTION: 660 GENERIC DATA REREGIS  
CHEMICAL: 108501 Pendimethalin ( N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinit  
ID#: 108501  
COMPANY: **74 LOIS ROSSI**  
PRODUCT MANAGER: ~~50 GAY EDELBERGER~~ **8080**  
PM TEAM REVIEWER: ~~DON CLAYTON~~ **Terri Stowe** 703-308-~~8005~~ ROOM: CST  
RECEIVED DATE: 07/18/90 DUE OUT DATE: / / 703-308-~~8006~~ ROOM: CST  
**5043**

\*\*\* DATA PACKAGE INFORMATION \*\*\*

DP BARCODE: 162977 EXPEDITE: Y DATE SENT: 03/28/91 DATE RET.: /  
DP TYPE: 999 Miscellaneous Data Package LABEL: N  
ADMIN DUE DATE: 06/26/91 CSF: N  
ASSIGNED TO DATE IN DATE OUT  
DIV : EFED **07/18/91** / /  
BRAN: EFGB / /  
SECT: / /  
REVR : / /  
CONTR: / /

\*\*\* DATA PACKAGE REVIEW INSTRUCTIONS \*\*\*

ATTN.: FOR PRIORITY REVIEW

PLEASE REVIEW PENDIMETHALIN DATA RECEIVED IN RESPONSE TO THE  
09/04/90 DCI FOR GLN 165-1 "CONFINED ROTATIONAL CROP STUDY"

PLEASE SEND A COPY OF THE REVIEW AND AN UPDATE STATUS REPORT  
ON PENDIMETHALIN TO: TERRI STOWE  
SRRD/RB (H7508W)  
CRYSTAL STATION I - 3RD FL.

THANK YOU!!

\*\*\* ADDITIONAL DATA PACKAGES FOR THIS SUBMISSION \*\*\*

DP BC BRANCH/SECTION DATE OUT DUE BACK INS CSF LABEL

14



## **PENDIMETHALIN ADDENDUM**

### **TASK 1: REVIEW AND EVALUATION OF INDIVIDUAL STUDIES**

June 7, 1991

Final Report

Contract No. 68D90058

Submitted to:  
Environmental Protection Agency  
Arlington, VA 22202

Submitted by:  
Dynamac Corporation  
The Dynamac Building  
2275 Research Boulevard  
Rockville, MD 20850-3262

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## INTRODUCTION

Pendimethalin is a dinitroaniline herbicide registered for use on terrestrial food + feed, aquatic food, and fiber crops as well as ornamental plants (including Christmas tree plantations) and non-agricultural areas (including lawns, industrial sites, road, utility and railroad rights-of-way etc.) to control annual grasses and some broadleaf weeds. Pendimethalin is applied as a preemergence and/or postemergence treatment for these crops, either broadcast or as a preemergence application. Single active ingredient formulations include 1-4 lb ai/gal and 21.05% EC, 0.5-4.13% G, 60% DF, and 35 and 50% WP. Pendimethalin is not toxic to bees or birds, but it is toxic to fish.

DATA EVALUATION RECORD

STUDY 1

-----  
CHEM 108501

Pendimethalin

§161-2

FORMULATION--00--ACTIVE INGREDIENT  
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00153763

Sanders, P. 1985a. Prowl Herbicide, pendimethalin (AC 92,553):  
Photodegradation in water. Report No. PD-M Volume 22-36. Unpublished study  
performed and submitted by American Cyanamid Company, Princeton, NJ.

-----  
DIRECT REVIEW TIME = 8  
-----

REVIEWED BY: L. Parsons

TITLE: Staff Scientist

EDITED BY: W. Martin  
K. Patten

TITLE: Staff Scientist  
Task Leader

APPROVED BY: W. Spangler

TITLE: Project Manager

ORG: Dynamac Corporation  
Rockville, MD  
TEL: 468-2500  
-----

APPROVED BY: H. Manning  
TITLE: Microbiologist  
ORG: EFGWB/EFED/OPP  
TEL: 557-7323

SIGNATURE:

*H. Manning*

CONCLUSIONS:

Degradation - Photodegradation in Water

1. This study cannot be used to fulfill data requirements at this time.
2. Pendimethalin photodegraded with a half-life of 21 days in water at 25 C continuously irradiated with artificial light (xenon arc lamp) for 28 days. There were up to 18 uncharacterized degradates present at  $\leq 6\%$  of the applied radioactivity in the samples irradiated for 28 days. Pendimethalin did not degrade in the dark controls incubated at 25 C for 28 days.

PENDIMETHALIN

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3. This study is scientifically sound, but does not meet Subdivision N guidelines for the following reasons:

the light source was not adequately characterized, and was not adequately compared to natural sunlight, and

insufficient data were presented to address the pattern of formation and decline of degradates.

4. In order for this study to fulfill data requirements, the registrant should provide detailed information about the artificial light source and data from earlier sampling intervals showing the pattern of degradation of pendimethalin.

#### METHODOLOGY:

[<sup>14</sup>C]Pendimethalin (3,4-dimethyl labeled, radiochemical purity >99%, specific activity 25.2 uCi/mg, American Cyanamid) dissolved in water at 0.27 ppm was aliquoted into Vycor test tubes. The tubes were sealed with foil-lined rubber stoppers. The tubes were placed at a 30 degree angle in a Mallory environmental chamber and continuously irradiated with a xenon arc lamp equipped with two borosilicate glass filters to simulate natural sunlight at 25 C. Duplicate tubes were removed for analysis at 0, 5, 9, 16, 23, and 28 days posttreatment.

Aliquots of the test solutions were analyzed by LSC. The remaining sample was passed through a C18 Bond-Elute column; compounds which adsorbed to the column were eluted with hexane and acetonitrile. The hexane eluants were analyzed for pendimethalin by GC with electron capture detection. The water, hexane, and acetonitrile eluants were analyzed by LSC and concentrated (method not reported) for analysis by TLC. The aliquots of the hexane and acetonitrile eluants were analyzed by TLC on silica gel plates developed with xylene:chloroform:methanol (280:70:7, v:v:v). Aliquots of the acetonitrile and aqueous eluants were analyzed by TLC on silica gel plates developed with chloroform:methanol (225:30, v:v). The aqueous eluant was also analyzed on reverse phase TLC (matrix not reported) developed with 0.1 N sodium chloride in water:methanol (50:50, v:v). The radioactive areas on all TLC plates were located by autoradiography. The radioactive areas were scraped from the plates and quantified by LSC. Samples were cochromatographed with unspecified standard reference compounds. The Vycor tubes were rinsed with 0.1% HCl in water:methanol (50:50, v:v) and the rinsate was analyzed by LSC.

#### DATA SUMMARY:

[<sup>14</sup>C]Pendimethalin, (3,4-dimethyl labeled, radiochemical purity >99%) photodegraded with a half-life of 21 days in water continuously irradiated with artificial light (xenon arc lamp) for 28 days at 25

C. Up to 18 uncharacterized photodegradates were isolated from the aqueous, hexane and acetonitrile fractions. At 23 and 28 days posttreatment, each TLC spot ranged from 0.5 to 6% of the applied radioactivity (Table I). Total uncharacterized degradates comprised up to 30% of the applied radioactivity. Pendimethalin did not degrade in the dark controls incubated at 25 C for 28 days.

COMMENTS:

1. The characterization of the light source was inadequate. The study author provided only the manufacturer's specifications rather than actual measurements taken at the time of the study; however, the distance that the samples were placed from the lamp and the amount of wattage used to power the lamp used in the study did not match the conditions under which the manufacturer had tested the lamp. Also, the measure of intensity vs. wavelengths was provided for ranges rather than as discrete values.
2. The amount of data present in the results section was limited, consisting of the TLC results for the three eluants of the day 23 and day 28 posttreatment test solutions. The presentation of the data for the last two sampling intervals does not permit an analysis of the pattern of formation and decline of the degradates. It is possible that some degradates were present in excess of 10% of the applied earlier in the study. These data are characterized only as radioactive areas on the TLC plates from each eluant solution. The registrant did not provide information on the mobility of the unspecified standards in the various TLC systems.
3. The concentration of parent was measured by GC at each sampling interval, but these data were not reported, which made independent verification of the half-life of pendimethalin difficult. In addition, the GC tracings were abbreviated, which precluded an independent assessment of the study author's statement that "the hexane extract quantitatively contained" pendimethalin.
4. The study author stated that some of the radioactivity was lost because of adsorption to the Vycor test tube walls. Methanol:water rinses removed some of this radioactivity. The method the study author used to estimate the amount of material remaining on the test tube walls is unclear (Table II). Material balances were approximately 89% of the applied at 28 days posttreatment (Table II, reviewer observed); this figure included the radioactivity rinsed from the test tube walls. The reviewer considered the data listed in the appendix as "water pre-C18" to be an approximate material balance. These values were 86.0-103.1% of the applied ("dose"). An additional 1-4% of the radioactive material was bound to the test tubes.
5. The study author stated that the output of lights in the environmental chamber was comparable to summer sunlight in Chicago,

Illinois, at noon on June 30. However, no evidence, such as a comparison of wavelength vs. intensity graphs for the artificial light and sunlight, was provided to support this claim.

6. The methods description was incomplete. For example, the photolysis apparatus was not fully described, and the sterility and the pH of the water were not reported.
7. The study was conducted in a closed system and volatiles were not trapped.
8. Subdivision N guidelines require that photodegradation in water studies be conducted in sterile aqueous buffered solutions adjusted to the pH at which the parent is most stable. This study was conducted in water but pendimethalin was stable in the dark controls.
9. The data presented in Table I and Table II do not agree. The radioactivity in the various aqueous fraction "spots" totals 18% of the applied in Table I, while the aqueous phase accounts for 23% of the applied in Table II. The study author did not address this discrepancy.

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Pages 24 through 28 are not included.

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The material not included contains the following type of information:

- Identity of product inert ingredients.
  - Identity of product impurities.
  - Description of the product manufacturing process.
  - Description of quality control procedures.
  - Identity of the source of product ingredients.
  - Sales or other commercial/financial information.
  - A draft product label.
  - The product confidential statement of formula.
  - Information about a pending registration action.
  - FIFRA registration data.
  - The document is a duplicate of page(s) \_\_\_\_\_.
  - The document is not responsive to the request.
- 

The information not included is generally considered confidential by product registrants. If you have any questions, please contact the individual who prepared the response to your request.

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DATA EVALUATION RECORD

STUDY 2

CHEM 108501

Pendimethalin

§161-3

FORMULATION--00--ACTIVE INGREDIENT

00153764

Mangels, G. 1985b. Prowl Herbicide, pendimethalin (AC 92,553):  
Photodegradation in soil. Report No. PD-M Volume 22-35. Unpublished study  
performed and submitted by American Cyanamid Company, Princeton, NJ.

DIRECT REVIEW TIME = 8

REVIEWED BY: L. Parsons TITLE: Staff Scientist

EDITED BY: W. Martin TITLE: Staff Scientist  
K. Patten Task Leader

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation  
Rockville, MD

TEL: 468-2500

APPROVED BY: H. Manning  
TITLE: Microbiologist  
ORG: EFGWB/EFED/OPP  
TEL: 557-7323

SIGNATURE:

*H. Manning*

CONCLUSIONS:

Degradation - Photodegradation on Soil

1. The study cannot be used to fulfill data requirements at this time.
2. Pendimethalin did not photodegrade on sandy loam soil that was continuously irradiated with artificial light (xenon arc lamps) for 4 weeks at 25 C. Pendimethalin also did not degrade in the dark controls incubated at 25 C for 4 weeks.

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The material not included contains the following type of information:

- Identity of product inert ingredients.
  - Identity of product impurities.
  - Description of the product manufacturing process.
  - Description of quality control procedures.
  - Identity of the source of product ingredients.
  - Sales or other commercial/financial information.
  - A draft product label.
  - The product confidential statement of formula.
  - Information about a pending registration action.
  - FIFRA registration data.
  - The document is a duplicate of page(s) \_\_\_\_\_.
  - The document is not responsive to the request.
- 

The information not included is generally considered confidential by product registrants. If you have any questions, please contact the individual who prepared the response to your request.

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3. This study is scientifically sound, but does not meet Subdivision N guidelines for the following reason:

the light source was not adequately characterized, and was not adequately compared to natural sunlight.

4. In order for this study to fulfill the photodegradation on soil data requirement, additional detailed information about the artificial light must be provided.

#### METHODOLOGY:

Princeton sandy loam soil (55.6% sand, 33.2% silt, 11.2% clay, 1.6% organic matter, pH 6.4, CEC 8.4 meq/100 g) was placed in petri dishes to an unspecified depth. [<sup>14</sup>C]Pendimethalin (3,4-dimethyl labeled; radiochemical purity >99%, specific activity 14.7 uCi/mg, American Cyanamid) fortified with unlabeled pendimethalin (purity not reported) dissolved in hexane were applied to the soil surface at approximately 50 ug/g soil. The hexane was allowed to evaporate and the dishes were covered with Pyrex covers. The dark control dishes were wrapped in aluminum foil. The dishes were placed in a Mallory environmental chamber and continuously irradiated with a xenon arc lamp equipped with two borosilicate filters at 25 C. [The lamp manufacturer's literature states that the constant light output at 48 cm is 114,340 mcwatts/cm<sup>2</sup> with the following spectral distribution: 1500 (<340 nm), 5750 (340-400 nm), 58,700 (400-750 nm), and 48,400 (>750 nm) mcwatts/cm<sup>2</sup> when the lamps are powered at 6.5 kilowatts and equipped with dual borosilicate filters. However, in this study, the lamps were at a distance of 80 cm and were powered at 6.0 kilowatts. Samples were removed for analysis at 0, 1, 2, 3, and 4 weeks posttreatment.

Soil samples were removed from the petri dishes, the dishes were rinsed with methanol, and the rinse was added to the soil sample. The soil was extracted twice with "2% HCl/methanol" with shaking for 1 hour; the soil was filtered after each extraction. The extracts were combined and analyzed by LSC. The soil was then extracted with water:"2% HCl/methanol" (1:3, v:v) with shaking for 1 hour and filtered. The soil extracts were combined and evaporated (method not reported), and the residues were redissolved in water. The aqueous solution was partitioned twice with hexane; aliquots of the water and hexane phases were analyzed by LSC. The remaining water and hexane solutions were concentrated under vacuum, and analyzed by TLC on silica gel plates developed with either xylene:chloroform:methanol (280:70:7, v:v:v) or chloroform:methanol (225:30, v:v). Radioactive areas, identified by autoradiography, were scraped from the plates and quantified by LSC. Samples were cochromatographed with unspecified standard reference compounds; the method of visualizing the reference compounds was not reported. The extracted soil was analyzed by LSC following combustion.

DATA SUMMARY:

[<sup>14</sup>C]Pendimethalin (3,4-dimethyl labeled; radiochemical purity >99%) did not photodegrade on a sandy loam soil continuously irradiated with artificial light (xenon arc lamps) for 4 weeks at 25 C.

[<sup>14</sup>C]Pendimethalin comprised 90.5-99.1% of the applied radioactivity at each sampling interval with no discernible pattern of decline (Table IV). Pendimethalin also did not degrade in the dark controls incubated at 25 C for 4 weeks. During the study, material balances ranged from 96.1-102.6% of the applied radioactivity (Table II).

COMMENTS:

1. The characterization of the light source was inadequate. The study author provided only the manufacturer's specifications rather than actual measurements taken at the time of the study; however, the distance that the samples were placed from the lamp and the amount of wattage used to power the lamp in the study did not match the conditions under which the manufacturer had tested the lamp. Also, the measure of intensity vs. wavelengths was provided for ranges rather than as discrete values.
2. The study author stated that the output of lights in the environmental chamber was comparable to summer sunlight in Chicago, Illinois, at noon on June 30. However, no evidence, such as a comparison of wavelength vs. intensity graphs for the artificial light and sunlight, was provided to support this claim.
3. The study author stated that the parent compound was contained in the hexane fraction. The aqueous extracts contained 0.6-9.9% of the applied radioactivity (Table III).
4. The methods description was incomplete; for example, the sieve size and moisture conditions of the soil, and the depth of soil in the petri dishes were not reported.

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Pages 33 through 38 are not included.

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The material not included contains the following type of information:

- Identity of product inert ingredients.
  - Identity of product impurities.
  - Description of the product manufacturing process.
  - Description of quality control procedures.
  - Identity of the source of product ingredients.
  - Sales or other commercial/financial information.
  - A draft product label.
  - The product confidential statement of formula.
  - Information about a pending registration action.
  - FIFRA registration data.
  - The document is a duplicate of page(s) \_\_\_\_\_.
  - The document is not responsive to the request.
- 

The information not included is generally considered confidential by product registrants. If you have any questions, please contact the individual who prepared the response to your request.

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DATA EVALUATION RECORD

STUDY 3

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CHEM 108501

Pendimethalin

\$162-1  
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FORMULATION--00--ACTIVE INGREDIENT  
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STUDY ID 40185104

Lavin, M. and W. Cranor. 1987a. Aerobic soil metabolism of [<sup>14</sup>C] pendimethalin. Final Report No. 33731. Unpublished study performed and submitted by Analytical Biochemistry Laboratories, Inc., Columbia, MO.  
-----

DIRECT REVIEW TIME = 16  
-----

REVIEWED BY: L. Parsons TITLE: Staff Scientist

EDITED BY: W. Martin TITLE: Staff Scientist  
K. Patten Task Leader

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation  
Rockville, MD

TEL: 468-2500  
-----

APPROVED BY: H. Manning  
TITLE: Microbiologist  
ORG: EFGWB/EFED/OPP  
TEL: 557-7323  
-----

SIGNATURE: 

CONCLUSIONS:

Metabolism - Aerobic Soil

1. This study cannot be used to fulfill data requirements at this time.
2. Pendimethalin degraded with a half-life of 1322 days in sandy loam soil incubated in the dark at 24.8 ± 0.8 C and 53-62% of 0.33 bar moisture capacity. The nonvolatile degradation products identified were 2,6-dinitro-3,4-xylidine (CL 84,846); 4-[(1-ethylpropyl)amino]-3,5-dinitro-o-toluic acid (CL 99,900); and 4-[(1-ethylpropyl)amino]-2-methyl-3,5-dinitro-benzyl alcohol (CL 202,347).

3. This study is scientifically sound, but does not meet Subdivision N guidelines for the following reason:

two degradates in the soil extract and residues in the ethylene glycol trap, each present at  $>0.01$  ppm (0.5% of the applied radioactivity), were not identified.

4. In order for this study to fulfill the aerobic soil metabolism data requirement, degradates with  $R_f$  values of 0.15 present at 0.6% of the applied radioactivity (0.01 ppm) and 0.07 at 2.2% (0.04 ppm) in the soil extracts, and volatiles in the ethylene glycol trap at 1.0% (0.02 ppm) must be identified.

#### METHODOLOGY:

Samples of sieved (2 mm) sandy loam soil (61% sand, 26% silt, 13% clay, 2.4% organic matter, pH 4.8, CEC 7.2 meq/100 g) were adjusted to 62% of field capacity (0.33 bar) with deionized water. [ $^{14}\text{C}$ ]Pendimethalin (radiochemical purity 98.8%, specific activity 13710 dpm/ug, American Cyanamid) was added at a nominal rate of 2 ppm and the soil was mixed by tumbling. Treated and control soil samples were added to 3 L resin pots (Figure 2) and incubated in an environmental chamber in the dark at 23-34 C. Humidified  $\text{CO}_2$ -free air was pumped (100 ml/min) through the system and into trapping solutions of ethylene glycol, 1 N sulfuric acid, and 1 N KOH (2 bottles). Subsamples of the soil were removed for analysis immediately posttreatment and at 1, 2, 3, 4, 6, 9, and 12 months posttreatment. The volatile traps were sampled and replaced with fresh solutions weekly for the first 4 months and biweekly for the remainder of the study.

Triplicate subsamples of the soil were extracted three times with acetonitrile by vortexing for 1 minute. After centrifugation, the extracts were combined, and aliquots were analyzed for total radioactivity by LSC. The pooled extracts were concentrated under a nitrogen stream and aliquots were analyzed by TLC on Kieselgel F-254 silica gel plates developed with chloroform:ethyl acetate:glacial acetic acid (95:4:1.5, v:v:v). Aliquots of the extracts from the 1- and 12-month posttreatment samples were also chromatographed on silica gel TLC plates developed with chloroform:acetone (90:10, v:v). The soil was dried under a nitrogen stream and reextracted three times with 2% HCl:methanol (1:1, v:v) by shaking. After centrifugation, aliquots of these extracts were analyzed by LSC. The extracts were diluted with deionized water, then partitioned with chloroform. The organic phase was dried over anhydrous sodium sulfate, and evaporated under reduced pressure on a rotary evaporator. The residues were transferred to a culture tube, diluted with chloroform, and aliquots were analyzed by LSC. Additional aliquots of the chloroform solutions were concentrated under a nitrogen stream and analyzed by TLC as described above. Radioactive areas, identified by autoradiography, were scraped from the plates

and quantified by LSC. Samples were cochromatographed with [<sup>14</sup>C]pendimethalin as a standard reference compound. Subsamples of the pre- and postextracted soil were analyzed by LSC following combustion.

DATA SUMMARY:

Pendimethalin (radiochemical purity 98.8%) degraded with a calculated half-life of 1322 days in a sandy loam soil that was incubated in the dark at  $24.8 \pm 0.8$  C and 53-62% of 0.33 bar moisture capacity. Pendimethalin declined from 98.7% of the applied radioactivity to 83.1% at 365 days posttreatment (Table 7). Three nonvolatile degradates were identified:

2,6-dinitro-3,4-xylidine (CL 84,846), which was a maximum of 2.3% of the applied radioactivity at 9 months posttreatment;

4-[(1-ethylpropyl)amino]-2-methyl-3,5-dinitro-benzyl alcohol (CL 202,347), which was a maximum of 1.3% at 6 months posttreatment; and

4-[(1-ethylpropyl)amino]-3,5-dinitro-o-toluic acid (CL 99,900), which was a maximum of 1.3% at 3 and 6 months posttreatment.

Two uncharacterized degradates ("unknown") were 0.6% (0.01 ppm) and 2.2% (0.04 ppm) of the applied radioactivity (Table 7). Cumulative volatile residues in the ethylene glycol traps were 1.0% (0.02 ppm) of the applied radioactivity at 365 days; CO<sub>2</sub> was 3.2% (0.06 ppm, Table 7). Material balances ranged from 94.3-103.0% of the applied radioactivity.

COMMENTS:

1. Two degradates, one with an R<sub>f</sub> value of 0.15, present at 0.6% of the applied radioactivity (0.01 ppm) at 61 days posttreatment, and the second with an R<sub>f</sub> value of 0.07, present at 2.2% (0.04 ppm) at 365 days posttreatment were isolated but not identified. In addition, volatile residues in the ethylene glycol trap present at 1.0% (0.02 ppm) at 365 days posttreatment were not identified. Subdivision N guidelines require that degradates present at  $\geq 0.01$  ppm be identified.
2. EFGWB prefers that [<sup>14</sup>C]residues in samples be separated by chromatographic methods (such as TLC, HPLC, or GC) with at least three solvent systems of different polarity, and that specific compounds isolated by chromatography be identified using a confirmatory method such as MS in addition to comparison to the R<sub>f</sub> of reference standards.

In this study, the sample extracts were analyzed using one-



dimensional TLC with two solvent systems. Radioactive areas on the TLC plates were identified only by comparison to  $R_f$  values measured in a preliminary study.

3. The statistical estimate of the half-life of pendimethalin computed by the study authors is of limited value because the calculations involve extrapolation considerably beyond the experimental time limits of the study.
4. The copy provided for review was poor. Complete identification information was not included on page 37, and many of the raw data pages in Appendix II (ie., pgs 116-121) were illegible.

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Page \_\_\_\_\_ is not included in this copy.

Pages 43 through 53 are not included.

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The material not included contains the following type of information:

- Identity of product inert ingredients.
  - Identity of product impurities.
  - Description of the product manufacturing process.
  - Description of quality control procedures.
  - Identity of the source of product ingredients.
  - Sales or other commercial/financial information.
  - A draft product label.
  - The product confidential statement of formula.
  - Information about a pending registration action.
  - FIFRA registration data.
  - The document is a duplicate of page(s) \_\_\_\_\_.
  - The document is not responsive to the request.
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The information not included is generally considered confidential by product registrants. If you have any questions, please contact the individual who prepared the response to your request.

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DATA EVALUATION RECORD

STUDY 4

CHEM 108501 Pendimethalin §162-2

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 40185105

Lavin, M. and W. Cranor. 1987b. Anaerobic soil metabolism of [<sup>14</sup>C]pendimethalin. Final Report No. 33731. Unpublished study performed by Analytical Biochemistry Laboratories, Inc., Columbia, MO, and submitted by American Cyanamid Company, Princeton, NJ.

DIRECT REVIEW TIME = 8

REVIEWED BY: L. Parsons TITLE: Staff Scientist

EDITED BY: W. Martin TITLE: Staff Scientist  
K. Patten Task Leader

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation  
Rockville, MD

TEL: 468-2500

APPROVED BY: H. Manning  
TITLE: Microbiologist  
ORG: EFGWB/EFED/OPP  
TEL: 557-7323

SIGNATURE: *H. Manning*

CONCLUSIONS:

Metabolism - Anaerobic Soil

1. This study cannot be used to fulfill data requirements at this time.
2. Pendimethalin was relatively stable (98.0% of applied at 60 days anaerobic incubation) in sandy loam soil that was anaerobically (flooding plus nitrogen atmosphere) incubated in the dark for 60 days following an aerobic incubation period of 30 days. Three nonvolatile degradates were identified during the aerobic incubation period: 2,6-dinitro-3,4-xylidine (CL 84,846), 4-[(1-ethylpropyl)amino]-2-methyl-3,5-dinitro-benzyl alcohol (CL 202,347), and 4-[(1-ethylpropyl)amino]-3,5-dinitro-o-toluic acid (CL 99,900).

*54*

3. This study is scientifically sound, but does not meet Subdivision N guidelines for the following reason:

uncharacterized residues present in the organic soil extracts ("remainder") at up to 0.05 ppm (2.7% of the applied radioactivity), and the aqueous soil extracts at up to 0.05 ppm (2.7%) were not identified.

4. In order for this study to fulfill the anaerobic soil metabolism data requirement, radioactivity in the acetonitrile extracts ("remainder") present at 0.05 ppm (2.7% of the applied radioactivity), and radioactivity in the "aqueous" phase present at 0.05 ppm (2.7%) must be characterized. However, this study (Anaerobic Soil Metabolism, 162-2) is not required if an acceptable Anaerobic Aquatic Metabolism (162-3) study is performed, since the aquatic study may be substituted for the soil study.

#### METHODOLOGY:

Samples of sieved (2 mm) sandy loam soil (61% sand, 26% silt, 13% clay, 2.4% organic matter, pH 4.8, CEC 7.2 meq/100 g) were adjusted to 62% of field capacity (0.33 bar) with deionized water. [<sup>14</sup>C]Pendimethalin (radiochemical purity 98.8%, specific activity 13710 dpm/ug, American Cyanamid) was added at a nominal rate of 2 ppm and the soil was mixed by tumbling. Treated and control soil samples were added to 3 L resin pots (Figure 2) and incubated in an environmental chamber in the dark at 23-26 C. Humidified CO<sub>2</sub>-free air was pumped (100 ml/min) through the system and into four trapping solutions: ethylene glycol, 1 N sulfuric acid, and 1 N KOH (two traps). Soil samples were removed for analysis immediately posttreatment and at 30 days posttreatment. At 30 days posttreatment, a subsample of both the treated and control soils was placed in a second 3 L resin pot and flooded with "Millipore treated" water. Humidified nitrogen was then pumped through the system as described above. Soil samples were taken from the anaerobic system at 60 and 90 days posttreatment (30 and 60 days of anaerobic incubation). Volatile traps were sampled and replaced with fresh solutions at 0, 30, 51, 60, 68, 76, 83, and 90 days posttreatment.

Triplicate subsamples of the soil were extracted three times with acetonitrile by vortexing for 1 minute. After centrifugation, the extracts were combined, and an aliquot was analyzed for total radioactivity by LSC. The extracts were then concentrated under a nitrogen stream and an aliquot was analyzed by TLC on Kieselgel F-254 silica gel plates developed with chloroform:ethyl acetate:glacial acetic acid (95:4:1.5, v:v:v). Aliquots of the extracts from the 90-day posttreatment (60 days of anaerobic incubation) samples were also chromatographed on silica gel TLC plates developed with chloroform:acetone (90:10, v:v). The extracted soil was dried under a stream of nitrogen and reextracted three times with 2% HCl:methanol (1:1, v:v) and shaking. After centrifugation, aliquots of the

55

extracts were analyzed by LSC. The extracts were then diluted with deionized water and partitioned with chloroform. The chloroform fraction was concentrated under a nitrogen stream and an aliquot was analyzed by TLC as described above. Radioactive areas, identified by autoradiography, were scraped from the plates and quantified by LSC. Samples were cochromatographed with [<sup>14</sup>C]pendimethalin as a standard reference compound. The pre- and postextracted soil was analyzed by LSC following combustion.

#### DATA SUMMARY:

[<sup>14</sup>C]Pendimethalin (radiochemical purity 98.8%) was relatively stable in a sandy loam soil incubated in the dark under anaerobic conditions (flooding plus nitrogen atmosphere) following a 30-day aerobic incubation period under similar conditions. Parent pendimethalin was 98.7% of the applied immediately posttreatment, 96.1% at 30 days and 98.0% at 90 days posttreatment (60 days of anaerobic incubation) (Table 7). Three nonvolatile degradates were identified in the aerobic incubation phase of the study:

2,6-dinitro-3,4-xylidine (CL 84,846), which was a maximum of 1.5% of applied radioactivity at 30 days posttreatment;

4-[(1-ethylpropyl)amino]-2-methyl-3,5-dinitro-benzyl alcohol, (CL 202,347), which was a maximum of 0.7% at 30 days posttreatment; and

4-[(1-ethylpropyl)amino]-3,5-dinitro-o-toluic acid (CL 99,900), which was a maximum of 0.5% at 30 days posttreatment.

Uncharacterized residues ("remainder") were present at 0.05 ppm (2.7% of the applied radioactivity) at 30 days and at 0.04 ppm (2.3%) at 90 days posttreatment (Table 7). Volatile residues in the ethanolamine trap were a maximum of 0.2% of the applied, and CO<sub>2</sub> was a maximum of 0.4% (0.01 ppm). Uncharacterized residues in the aqueous extract were a maximum of 0.05 ppm (2.7%) at 90 days posttreatment. Unextractable residues were a maximum of 2% of the applied at 90 days posttreatment.

Material balances were 85.3-105.9% of the applied radioactivity (Table 6).

#### COMMENTS

1. Uncharacterized residues present in the organic soil extract at 0.05 ppm at 30 days and at 0.04 ppm at 90 days posttreatment were not characterized. In addition, residues in the aqueous extract present at 0.05 ppm (2.7% of the applied) were not characterized. Subdivision N guidelines state that all degradates present at  $\geq 0.01$  ppm must be identified. Since this study was performed concurrently

with the aerobic soil metabolism study, the characterization of the residues in that study would correspond with the highest level of the degradates in this study. However, the registrant would need to confirm that these degradates are the ones present at the end of the anaerobic incubation period and must identify the residues in the aqueous solution at the end of the study.

2. The material balance was 85.3% of the applied at 60 days posttreatment (30 days anaerobic). The study author stated that pre-extraction combustion of the soil showed 1.73 ppm or 89% of the initial radioactivity. Since LSC analysis showed no volatile degradates, the study author attributed this low material balance to non-homogeneity of the test system. This problem was corrected by 90 days posttreatment (60 days anaerobic) when the material balance was 105.9%.
3. The label position of the [<sup>14</sup>C]pendimethalin was not cited in the study.
4. The temperature of the aerobic portion of the study was 25.1 ± 0.8 C; the anaerobic incubation temperature was not reported.
5. The study author stated that non-rounded values were used to calculate % applied radioactivity (initial dose measured). The values provided for review need more tabulated significant figures; for example, in Table 7, 0.01 ug/g is 0.05, 0.06 0.07 and 0.08% initial dose measured.
6. Water used to flood the soils was described as "Millipore treated water". Since Millipore manufactures many filtration and purification units, this is not an adequate description.
7. Subsamples of soil were measured by LSC following combustion at the initiation of the study.
8. There is an apparent typographical error in Table 6; in the summary data for day 30, the total ug/g should be 1.59 instead of 0.159.
9. EFGWB prefers that [<sup>14</sup>C]residues in samples be separated by chromatographic methods (such as TLC, HPLC, or GC) with at least three solvent systems of different polarity, and that specific compounds isolated by chromatography be identified using a confirmatory method such as MS in addition to comparison to the R<sub>f</sub> of reference standards.

In this study, the sample extracts were analyzed using one-dimensional TLC with two solvent systems. Radioactive areas on the TLC plates were identified only by comparison to R<sub>f</sub> values of known standards (Table 4) measured in a preliminary study.

with the aerobic soil metabolism study, the characterization of the residues in that study would correspond with the highest level of the degradates in this study. However, the registrant would need to confirm that these degradates are the ones present at the end of the anaerobic incubation period and must identify the residues in the aqueous solution at the end of the study.

2. The material balance was 85.3% of the applied at 60 days posttreatment (30 days anaerobic). The study author stated that pre-extraction combustion of the soil showed 1.73 ppm or 89% of the initial radioactivity. Since LSC analysis showed no volatile degradates, the study author attributed this low material balance to non-homogeneity of the test system. This problem was corrected by 90 days posttreatment (60 days anaerobic) when the material balance was 105.9%.
3. The label position of the [<sup>14</sup>C]pendimethalin was not cited in the study.
4. The temperature of the aerobic portion of the study was 25.1 ± 0.8 C; the anaerobic incubation temperature was not reported.
5. The study author stated that non-rounded values were used to calculate % applied radioactivity (initial dose measured). The values provided for review need more tabulated significant figures; for example, in Table 7, 0.01 ug/g is 0.05, 0.06 0.07 and 0.08% initial dose measured.
6. Water used to flood the soils was described as "Millipore treated water". Since Millipore manufactures many filtration and purification units, this is not an adequate description.
7. Subsamples of soil were measured by LSC following combustion at the initiation of the study.
8. There is an apparent typographical error in Table 6; in the summary data for day 30, the total ug/g should be 1.59 instead of 0.159.
9. EFGWB prefers that [<sup>14</sup>C]residues in samples be separated by chromatographic methods (such as TLC, HPLC, or GC) with at least three solvent systems of different polarity, and that specific compounds isolated by chromatography be identified using a confirmatory method such as MS in addition to comparison to the R<sub>f</sub> of reference standards.

In this study, the sample extracts were analyzed using one-dimensional TLC with two solvent systems. Radioactive areas on the TLC plates were identified only by comparison to R<sub>f</sub> values of known standards (Table 4) measured in a preliminary study.

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Page \_\_\_\_\_ is not included in this copy.

Pages 59 through 65 are not included.

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The material not included contains the following type of information:

- Identity of product inert ingredients.
  - Identity of product impurities.
  - Description of the product manufacturing process.
  - Description of quality control procedures.
  - Identity of the source of product ingredients.
  - Sales or other commercial/financial information.
  - A draft product label.
  - The product confidential statement of formula.
  - Information about a pending registration action.
  - FIFRA registration data.
  - The document is a duplicate of page(s) \_\_\_\_\_.
  - The document is not responsive to the request.
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The information not included is generally considered confidential by product registrants. If you have any questions, please contact the individual who prepared the response to your request.

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DATA EVALUATION RECORD

STUDY 5

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CHEM 108501 Pendimethalin §162-3

FORMULATION--00--ACTIVE INGREDIENT  
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40813501

Sanders, P. 1988. Pendimethalin (AC 92,553): Anaerobic aquatic degradation in soil from a rice field. Laboratory Report Number PD-M 25-25. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ.

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DIRECT REVIEW TIME = 18  
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REVIEWED BY: C. Little TITLE: Staff Scientist

EDITED BY: K. Patten TITLE: Task Leader  
W. Martin Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation  
Rockville, MD  
TEL: 468-2500

-----  
APPROVED BY: H. Manning  
TITLE: Chemist  
ORG: EFGWB/EFED/OPP  
TEL: 557-7323

SIGNATURE: *H. Manning*

CONCLUSIONS:

Metabolism - Anaerobic Aquatic

1. This study cannot be used to fulfill data requirements.
2. Pendimethalin degraded in anaerobic silt loam soil, which had been treated and incubated aerobically for 1 week prior to establishment of anaerobic (flooding and nitrogen) conditions. Parent pendimethalin was 50-58% of the nominal application at 9 weeks posttreatment. Degradates were not identified.

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Pages \_\_\_\_\_ through \_\_\_\_\_ are not included.

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The material not included contains the following type of information:

- Identity of product inert ingredients.
  - Identity of product impurities.
  - Description of the product manufacturing process.
  - Description of quality control procedures.
  - Identity of the source of product ingredients.
  - Sales or other commercial/financial information.
  - A draft product label.
  - The product confidential statement of formula.
  - Information about a pending registration action.
  - FIFRA registration data.
  - The document is a duplicate of page(s) \_\_\_\_\_.
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The information not included is generally considered confidential by product registrants. If you have any questions, please contact the individual who prepared the response to your request.

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3. This study is scientifically sound, but does not meet Subdivision N guidelines for the following reasons:

the experimental design was inappropriate to determine the rate of metabolism of pendimethalin under anaerobic aquatic conditions (the soil was treated and incubated aerobically for 1 week prior to conversion to anaerobic conditions); and

degradates were isolated but were not identified.

4. Because the study was not conducted according to Subdivision N guidelines, the problems associated with this study cannot be resolved by the submission of additional information. A new study is required.

#### METHODOLOGY:

Ring-labeled [<sup>14</sup>C]pendimethalin (radiochemical purity 97%, specific activity 6.07 uCi/mg, American Cyanamid), in methanol, was formulated with monochloro-benzene, 89% (w:w); FloMo PLA, 5.5% (w:w); and FloMo PHN, 5.5% (w:w). The formulation was applied at 1.3 lb ai/A to portions (300 g dry weight) of moist, sieved (2 mm) silt loam soil (22.4% sand, 63.2% silt, 14.4% clay, pH 5.1, 2.1% organic matter, CEC 12.1 meq/100 g) in twelve canning jars; the formulation was dripped onto the surface of the soil. Duplicate samples were removed for time 0 analysis, and all other jars were placed in a greenhouse and exposed to natural light. After one week of aerobic incubation, all jars were removed from the greenhouse and duplicate samples were taken for analysis. The remaining jars were flooded with water (uncharacterized) and incubated anaerobically in the dark at "room temperature" in a chamber which had been vacuum pumped and filled with nitrogen three times. Duplicate samples were taken at 2-week intervals.

To analyze time 0 samples, duplicate portions were extracted by shaking with acetonitrile for 1 hour. The extract was filtered and the soil was re-extracted with acetonitrile; the extracts were combined, concentrated and analyzed by TLC on normal-phase silica gel plates developed with toluene:p-dioxane:acetic acid (90:30:1) and on reverse-phase C18 plates developed with acetonitrile:water (80:20) in 0.1 N NaCl solution. Samples were cochromatographed with a non-labeled pendimethalin standard. Radiolabeled residues on the plates were visualized by autoradiography; radioactive zones were scraped from the plates and were quantified using LSC. Radioactivity in the soil was determined by LSC following combustion.

To analyze samples taken immediately following the aerobic incubation period, duplicate portions were extracted with acetonitrile as described above, followed by an additional extraction with methanol:water:concentrated hydrochloric acid (500:100:12, v:v:v). All extracts were analyzed by LSC and the acetonitrile extracts were

concentrated for TLC analysis. Extracted soil was analyzed as described above.

To analyze all flooded samples, water was filtered from the samples and analyzed for total radioactivity by LSC. The soil was then extracted twice with acetonitrile and once with methanol:water:HCl as described above; extracts were concentrated and analyzed by TLC as described above. Extracted soil was analyzed by LSC following combustion. The 6-week interval (7 weeks posttreatment) samples were also extracted with methanol:water:5 N NaOH (50:10:3, v:v:v). One of the 8-week interval (9 weeks posttreatment) samples, following the acetonitrile extractions, was extracted sequentially with methanol:water (5:1, v:v), acetone:water (5:1, v:v), methanol:water:5 N NaOH (50:10:3, v:v:v), and twice with methanol:water:concentrated HCl (500:100:12); the extracts were analyzed by LSC. The soil was then refluxed with methanol:water:HCl overnight. The solution was removed by filtering and the soil was refluxed with methanol:water:5 N NaOH. The solution was filtered and the soil was analyzed by LSC following combustion. For the second sample from the 8-week sampling interval, extraction was done with methanol:water:HCl and methanol:water:5 N NaOH, and the sample was then refluxed as above. Extracts from the 8-week samples were analyzed by normal- and reverse-phase TLC, as described above, and using normal-phase silica-gel plates developed with methylene chloride:tetrahydrofuran:acetic acid (70:30:1).

#### DATA SUMMARY:

Ring-labeled [<sup>14</sup>C]pendimethalin [radiochemical purity 97%; dissolved in methanol and formulated with monochloro-benzene (89%), FloMo PLA (5.5%) and FloMo PHN (5.5%)], at 1.3 lb ai/A, degraded in silt loam soil that was treated and incubated aerobically for 1 week prior to incubation under anaerobic (flooding plus nitrogen) conditions in the dark at room temperature. As determined by reverse-phase TLC analysis, parent pendimethalin decreased from 95% of the nominal application at time 0 to 78-79% by 1 week posttreatment (Table IV). During the anaerobic phase of the study, parent pendimethalin accounted for 73-81% of the nominal application up to 7 weeks posttreatment (6 weeks of anaerobic incubation), then decreased to 50-58% by 9 weeks posttreatment (8 weeks of anaerobic incubation). Unextracted radioactive residues increased with incubation time, from 1% of the nominal application rate at 1 week posttreatment (start of anaerobic conditions) to 4-6% at 9 weeks posttreatment (8 weeks of anaerobic incubation; Table III). Several degradates, identified only as A1, A2 and A3, were isolated at up to 2.5% of the nominal application rate. Several other degradates, at up to 1.1% of the applied, were isolated but were not identified (Table V). In addition, uncharacterized origin material in the acetonitrile extracts accounted for up to 2.3% of the nominal application.

Material balances were 91-101% of the nominal application.

COMMENTS:

1. The soil in this study was treated with pendimethalin and incubated aerobically for 1 week prior to the establishment of anaerobic conditions. Subdivision N guidelines state that for an anaerobic aquatic study, the soil must be flooded for thirty days prior to the addition of the test substance to ensure the presence of anaerobic conditions. Also, the study does not qualify as an anaerobic soil metabolism study because the aerobic incubation of pendimethalin lasted only 1 week rather than 30 days.
2. Several degradates were isolated but not identified. It could not be determined whether the degradates were present at concentrations  $\geq 0.01$  ppm, since the data were reported in percentages (of the nominal application rate) rather than in specific units, the nominal application rate was reported only as 1.3 lb ai/A, and the actual rate of application was not reported.
3. Attached is a 1982 letter from the EPA giving the company permission to modify the protocol. Since the guidelines and policies have changed considerably over the last eight years, Dynamac has reviewed these studies strictly according to Subdivision N guidelines, with the intention that the EPA reviewer will address the applicability of the previous correspondence.
4. Volatiles were neither controlled nor measured. The study author suggested that, because the sample containers were not sealed, volatiles could have accounted for the decrease in total radioactivity recovered which occurred most noticeably from time 0 to the first sampling interval (1 week). During that time, recovered radioactivity decreased from 101-102% of the nominal application to 94-95%; recovered radioactivity ranged from 91-94% of the nominal application for the remainder of the study.
5. The test water was not characterized.
6. The FloMo series (including PLA and PHN) consists of multipurpose spreading, penetrating, wetting and emulsifying agents used in agricultural formulations.

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Pages 71 through 81 are not included.

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The material not included contains the following type of information:

- Identity of product inert ingredients.
  - Identity of product impurities.
  - Description of the product manufacturing process.
  - Description of quality control procedures.
  - Identity of the source of product ingredients.
  - Sales or other commercial/financial information.
  - A draft product label.
  - The product confidential statement of formula.
  - Information about a pending registration action.
  - FIFRA registration data.
  - The document is a duplicate of page(s) \_\_\_\_\_.
  - The document is not responsive to the request.
- 

The information not included is generally considered confidential by product registrants. If you have any questions, please contact the individual who prepared the response to your request.

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Appendix B

American Cyanamid Company  
Agricultural Research Division  
P.O. Box 400  
Princeton, NJ 08540  
(609) 799-0400

November 10, 1982

Mr. Robert J. Taylor  
Product Manager (25)  
Registration Division (TS-767C)  
Office of Pesticide Programs  
U. S. Environmental Protection Agency  
Crystal Mall, Building #2  
1921 Jefferson Davis Highway  
Arlington, VA 22202

**BEST AVAILABLE COPY**

Re: PROWL<sup>®</sup> herbicide  
EPA Reg. No. 241-243  
Rice  
EPA Letter of December 16, 1981

Dear Mr. Taylor:

This letter will confirm a telephone conversation between Dr. Samuel Creeger, EPA, and Mr. William Steller, American Cyanamid, on September 7, 1982.

In their telephone conversation regarding an appropriately designed aquatic metabolism study in support of a full registration for use of PROWL<sup>®</sup> herbicide in rice, Mr. Creeger will recall that Mr. Steller expressed our concern that neither the aerobic or anaerobic aquatic metabolism study protocols as outlined in the EPA Guidelines, Subpart N, dated June 1981, are appropriate to the use pattern of PROWL<sup>®</sup> herbicide in rice. At Mr. Creeger's recommendation we are submitting a modified protocol for a study which reflects current practice in rice culture and is in accord with our label instructions for use of the herbicide.

Prior work (American Cyanamid Company Report PD-M, Vol. 11, 332-375 (1974) Princeton, N.J.), (EPA Accession Number 94475), has shown that the major route of PROWL degradation in soil occurs under anaerobic conditions. Consequently, to satisfy the requirements for aquatic metabolism work as indicated by Mr. Robert Taylor to Ms. L. A. Melville in his letter of December 16, 1981, the following is proposed:

Only an anaerobic aquatic study will be conducted according to EPA Guidelines, Subpart N, Section 163.162-3 with the following modification:

DI 82

~~CONFIDENTIAL~~

Mr. Robert S. Taylor

-2-

November 10, 1982

In Section 163.162-3(C)(2)(ii) substitute the following: Soil typical of that used for rice culture will be treated with PROWL<sup>®</sup> herbicide at a concentration consistent with label instructions and incubated aerobically for seven (7) days prior to establishment of anaerobic conditions by flooding and deaeration of the system.

For "If the test is performed using flooded soil, oxygen depletion should be established by flooding for 30 days prior to adding the test substance."

This modification best reflects conditions for the use of PROWL<sup>®</sup> herbicide in rice.

Should you have any further questions please contact me at (609)799-0400. We appreciate your further consideration of this protocol modification.

Very truly yours,

*Lynne M. Gregory*

Lynne M. Gregory  
Registrations Coordinator  
Plant Industry Registrations

LMG:fep  
Attachment

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Dr

83



DATA EVALUATION RECORD

STUDY 6

CHEM 108501

Pendimethalin

§163-1

FORMULATION--00--ACTIVE INGREDIENT

00153765

Mangels, G. 1985a. Prowl Herbicide, pendimethalin (AC 92,553):  
Adsorption/desorption studies. Report No. PD-M Volume 22-37. Unpublished  
study performed and submitted by American Cyanamid Company, Princeton, NJ.

DIRECT REVIEW TIME = 8

REVIEWED BY: L. Parsons TITLE: Staff Scientist

EDITED BY: W. Martin TITLE: Staff Scientist  
K. Patten Task Leader

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation  
Rockville, MD

TEL: 468-2500

APPROVED BY: H. Manning  
TITLE: Microbiologist  
ORG: EFGWB/EFED/OPP  
TEL: 557-7323

SIGNATURE:

*H. Manning*

CONCLUSIONS:

Mobility - Leaching and Adsorption/Desorption

1. This study cannot be used to fulfill data requirements.
2. These data are considered to be of uncertain value and should not be used to predict the environmental behavior of pendimethalin and its degradates.
3. This study is unacceptable for the following reason:

the soils were sieved through a 0.5 mm screen sieve, which may have removed a significant portion of the sand fraction and reduced the apparent mobility of pendimethalin.

PD-M Volume 25-25



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

DATE RECEIVED

DEC 17 1982

DEC 17 1982

American Cyanamid Company  
Agricultural Research Division  
P.O. Box 400  
Princeton, NJ 08540

Attention: Lynn M. Gregory

Gentlemen:

Subject: Prowl Herbicide (Protocols for Aerobic and  
Anaerobic Metabolism)  
EPA Registration No. 241-243  
Your Letter of November 10, 1982

The scientific review and evaluation of the product and letter submitted  
above have been completed. We concur with the change in protocol and that the  
aerobic aquatic metabolism study will not be needed.

Sincerely yours,

A handwritten signature in dark ink, appearing to read "Robert J. Taylor".

Robert J. Taylor  
Product Manager (E5)  
Fungicide-Herbicide Branch  
Registration Division (TS-767C)

BEST AVAILABLE COPY

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Handwritten initials or a signature in the bottom right corner of the page.

4. Since the soils were improperly sieved prior to use, the problems with this study cannot be resolved with the submission of additional data. A new study must be conducted.

#### METHODOLOGY:

Triplicate subsamples (1 g) of air-dried, sieved (0.5 mm) loamy sand, sandy loam, silt loam, loam, and silty clay loam soils (Table I) added to 0.01 N CaCl<sub>2</sub> solutions containing 0.243, 0.182, 0.1215, and 0.0608 ppm [<sup>14</sup>C]pendimethalin (labeled in the 3,4-dimethyl positions; radiochemical purity 99%, specific activity 25.2 uCi/mg, American Cyanamid) in glass centrifuge tubes. The soil:solution slurries were equilibrated for 3 days with shaking. After the equilibration period, the slurries were centrifuged and the supernatants decanted; aliquots of supernatant were analyzed by LSC.

To determine the desorption potential of pendimethalin, the decanted supernatants were replaced with 25 mL of pesticide-free 0.01 N CaCl<sub>2</sub> solution and the slurries were equilibrated by shaking for 3 days. After equilibration, the supernatants and soil were analyzed using LSC and LSC following combustion, respectively.

#### DATA SUMMARY:

Based on batch equilibrium studies, pendimethalin was mobile in loamy sand soil and slightly mobile in sandy loam, silt loam, loam, and silty clay loam soil:0.01 N CaCl<sub>2</sub> solution slurries (1 g:40 mL) containing 0.243, 0.182, 0.1215, and 0.0608 ppm of [<sup>14</sup>C]pendimethalin (labeled in the 3,4 dimethyl position; radiochemical purity 99%). Freundlich K<sub>ads</sub> values were 30 for the loamy sand soil, 110 for the sandy loam soil, 380 for the silt loam soil, 301 for the loam soil, and 854 for the silty clay loam soil; respective Koc values were 15000, 13000, 14100, 13700, and 29400 (Table II). Following adsorption, 29.3-44.38% of the adsorbed radioactivity was desorbed from the loamy sand soil, and 4.85-13.95% was desorbed from the sandy loam, silt loam, loam, silty clay loam soils.

#### COMMENTS:

1. The soils were sieved through a 0.5-mm screen rather than a 2-mm screen, so that a significant portion of the soil sand fraction (particles 0.5-2 mm in diameter) may have been removed during sieving. Since the sand fraction of a soil consists of relatively large particles and is generally inert, decreasing the relative percentage of sand in a soil may decrease the apparent mobility of the test substance.
2. Light conditions and temperatures during equilibration were not provided.

3. Freundlich  $K_{des}$  were not reported; percent desorption data were included in the appendix. There are discrepancies between the "percent desorption" reported at the bottom of the tables and the "percent dose" desorbed reported in the body of the tables. The "percent desorption" was reported in this review.
4. Material balances included in an appendix of raw data varied widely, ranging from 9.65-138.00%; the majority were >110% recovered.
5. Although the values for pendimethalin concentration in the methodology section (0.243, 0.182, 0.1215, 0.608 ppm) were not reported as "nominal", the tabulated concentrations listed in the appendix (0.191, 0.127, 0.085, 0.0425 ppm) were lower than the concentrations in the text.

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DATA EVALUATION RECORD

STUDY 7

CHEM 108501 Pendimethalin §163-2

FORMULATION--00--ACTIVE INGREDIENT

00153766

Sanders, P. 1985b. Prowl Herbicide, pendimethalin (AC92,533):  
Volatilization from soil. Project No. 0166, Report No. PD-M Volume 22-38.  
Unpublished study performed and submitted by American Cyanamid Company,  
Princeton, NJ.

DIRECT REVIEW TIME = 10

REVIEWED BY: W. Hurtt TITLE: Staff Scientist

EDITED BY: K. Patten TITLE: Task Leader  
W. Martin Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation  
Rockville, MD  
TEL: 468-2500

APPROVED BY: H. Manning  
TITLE: Microbiologist  
ORG: EFGWB/EFED/OPP  
TEL: 557-7323

SIGNATURE: *H. Manning*

CONCLUSIONS:

Mobility - Laboratory Volatility

1. This study is acceptable and fulfills EPA Data Requirements for Registering Pesticides by providing information on the volatility of pendimethalin under laboratory conditions.
2. Pendimethalin slowly volatilized from dry sandy loam soil incubated at 21 C, with a maximum air concentration of  $0.54 \text{ ug/m}^3$  and a volatility rate of  $5.0 \times 10^{-5} \text{ ug/cm}^2/\text{hour}$  when averaged over the initial 4.7-day sampling period. The cumulative loss of pendimethalin after 8.7 days was 3.3 ug, which was 0.05% of the applied. In moist sandy loam soil, pendimethalin volatilized at a maximum air

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concentration of  $31 \text{ ug/m}^3$  and a volatility rate of  $2.1 \times 10^{-3} \text{ ug/cm}^2/\text{hour}$  when averaged over the initial 24-hour sampling period.

#### METHODOLOGY:

Sandy loam soil (55.6% sand, 33.2% silt, 11.2% clay, 1.6% organic matter, pH 6.4, CEC 8.4 meq/100 g) was sieved (2-mm mesh) and dried at 68 C for 3 days. Samples (500 g dry weight) of dry or moistened soil were placed in 2.8-L Erlenmeyer flasks (surface area  $327 \text{ cm}^2$ ) and treated with 6.6 mg of pendimethalin (purity >99%, American Cyanamid) dissolved in 5 mL of hexane:acetonitrile (1:1) by dripping the solution over the soil surface; the application was equivalent to  $20 \text{ ug/cm}^2$  or 2 kg/ha. The solvent was allowed to evaporate. The Erlenmeyer flasks were then fitted with 1-cm diameter glass air inlet and outlet tubes, which terminated 1 inch above the soil surface and 1 inch below the stopper, respectively (Figure 1). The inlet tube was connected to an air pump, which forced the air through a scrubber (a trap containing 5 mL of XAD-2 resin) prior to entering the flask. The outlet tube was connected to a second resin trap to adsorb any volatilized pendimethalin and to a flow meter for regulating the air flow. The capacity of the pump was adequate to operate up to four replicate volatilization flasks simultaneously. The traps were changed at intervals varying from 1 to 7 days; the experiments were continued for cumulative volatilization periods of 9 or 18 days. The temperature was maintained at 21 C.

At each sampling interval, the resin and glass wool from the traps were extracted by shaking with 50 mL of hexane in an Erlenmeyer flask for 1 hour. The extract was filtered with hexane rinses into a 100-mL round-bottom flask for concentration under vacuum prior to GC analysis. At the conclusion of the last sampling interval, the soil in the volatilization flask was mixed, and 50-g samples were transferred to bottles containing 100 mL of acetonitrile. After shaking for 30 minutes, the acetonitrile extract was transferred with rinses and filtration to a round-bottom flask for concentration as previously described. Pendimethalin was quantified in aliquots of the extracts by GC analysis using a 10% OV-101 column, argon:methane (95:5) as the carrier gas, and electron-capture detection.

The efficiency of the XAD-2 resin traps was determined by dripping 100 ug of pendimethalin dissolved in 150 uL of hexane on duplicate traps, allowing the solvent to evaporate, and pumping 600 mL air/minute through the traps over a period of one night. The resin and glass wool from the traps were extracted and analyzed as previously described.

Dry soil experiment: For the dry soil experiment, duplicate treated soil samples were exposed to a mean air flow of 520 mL/minute (500 to 546 mL/minute range) for a total of 8.7 days. The traps were changed for analysis at 4.7 and 8.7 days posttreatment. Because the flow rate differed somewhat over the 0- to 4.7- and 4.7- to 8.7-day



intervals, it was normalized to 500 mL/minute by multiplying by an appropriate correction factor, e.g., Table VI: 5.4 times (500 divided by 543) equals the normalized volatility (flux) value of  $5.0 \times 10^{-5}$  ug/cm<sup>2</sup>/hour.

Moist soil experiment: For the moist soil experiment, two bubblers (containing 500 mL of distilled water each) were placed between the air pump and the first resin trap to moisten the air stream and reduce soil desiccation. Triplicate soil samples were each mixed with 41 mL of distilled water (equivalent to 80% of field capacity), placed in the volatilization flasks, and treated with pendimethalin as previously described. The soils were exposed to a mean air flow of 425 mL/minute (358-517 mL/minute range) for a total of 18 days. The traps were changed for analysis at 1, 2, 8, 11, and 18 days. The flow rates for the individual sampling intervals were normalized to a constant 400 mL/minute because of the variability previously indicated (Table IV).

#### DATA SUMMARY:

Pendimethalin (purity >99%), formulated in hexane:acetonitrile and surface applied to oven-dry sandy loam soil at 20 ug/cm<sup>2</sup> (equivalent to 2 kg/ha), volatilized slowly with a maximum air concentration of 0.54 ug/m<sup>3</sup> (Table V) and a volatilization rate of  $5.4 \times 10^{-5}$  ug/cm<sup>2</sup>/hour (Table VI) when averaged over the initial 4.7-day sampling period. During the 8.7-day study, the air flow through the system averaged 520 mL/minute, and the temperature was 21 C. At 8.7 days posttreatment, the volatilized pendimethalin totaled 3.3 ug, which was 0.05% of the applied (Appendix, p. 27).

In contrast, pendimethalin applied to a moistened sandy loam soil volatilized with a maximum air concentration of 31 ug/m<sup>3</sup> (Table III) and a volatilization rate of  $2.1 \times 10^{-3}$  ug/cm<sup>2</sup>/hour (Table IV) when averaged over the initial 24-hour sampling period. By the last sampling period (11-18 days), the air concentration had decreased to 15 ug/m<sup>3</sup> (approximately one-half of its initial value), and the volatilization rate had decreased to  $1.3 \times 10^{-3}$  ug/cm<sup>2</sup>/hour. Using normalized volatility data for six sampling periods from 0 to 18 days (Table IV), the computed volatility half-life was 300 hours or 12.5 days (Figure 4). During the 18-day study, the air flow through the system averaged 425 mL/minute, and the temperature was 21 C. At 18 days posttreatment, the volatilized pendimethalin totaled 251 ug (Table III), which was 3.8% of the applied.

The vapor pressure of pendimethalin was reported from the literature as  $3.0 \times 10^{-5}$  mm Hg at 25 C (Pesticide Manual, British Crop Protection Council, 1979).

At the conclusion of these studies, the material balances ranged from 96-101% of the applied (Table II).

## COMMENTS:

1. An addendum to the original document noted that although Subdivision N guidelines require the use of a typical end-use formulation, the study author used analytical (reference standard) grade material to achieve a greater treatment accuracy than would have been possible using the commercial formulation.
2. The value reported as the half-life of pendimethalin was actually the half-life of the normalized rate of volatility of pendimethalin (Figure 4). Although, in general terms, there is a relationship between the volatility of a pesticide per unit of surface area and the amount of pesticide adsorbed on the soil, it is not a linear relationship that would allow the half-life of volatility to be interchanged with the half-life of the pesticide adsorbed on the soil.
3. There was no indication that soil moisture content was monitored over the 18-day study; therefore, the possible effect of changing moisture content of the soil on the volatility of pendimethalin cannot be assessed. The study author, at one point, suggested that the slight changes in the air concentration of pendimethalin during the first several days was attributable to the soil absorbing additional moisture and, at another point, suggested that apparent discrepancies in the data were attributable to the greater air flow over the soil immediately below the inlet tube. Since the latter suggests desiccation, it appears that changing soil moisture content affects volatility of pendimethalin. As a minimum alternative to monitoring relative humidity, the moisture content of the soil should have been determined at the end of the 18-day experiment.
4. Apparently because the flow rate of air through the volatilization apparatus was somewhat variable in both studies, the study author normalized the actual flow rates in Tables IV and VI to 400 and 500 mL/minute, respectively.
5. Several numerical inconsistencies existed between tables, which may have been a function of rounding off or may have been transcriptional errors. For the moist soil study, the reported 249 ug of cumulative volatilized pendimethalin in Table II does not agree with the sum of the individual values (251 ug) found in a summary table in the Appendix (p. 25) or in Table III. For the dry soil study, the reported 4.3 ug in Table II does not agree with the sum of the individual values (3.3 ug) found in a summary table in the Appendix (p. 27) or in Table V. In Table III, the fourth sampling interval was 4.7 to 7 days but in Table IV, it was 7.7 days. Additionally, the half-life was variously reported as 300 hours and 300 days.
6. The description of the environmental conditions under which the two studies were conducted consisted only of a statement that the

temperature was 21 C (no indication was given as to whether this was the mean temperature or a one-time measurement). Furthermore, it was not specified whether the studies were conducted in the dark and relative humidity data were not provided.

7. Recovery from resin traps spiked with 100 ug pendimethalin was 98-100% (Appendix, p. 28). In determining the extraction efficiency of the resin trap, it was reported that the contents of the trap (5 mL of resin plus glass wool) were extracted with 50 mL of hexane in a 50-mL Erlenmeyer flask. Assuming that it was possible to put the glass wool and 55 mL of resin plus solvent in a 50-mL container, it is questionable how much agitation could be obtained by shaking an over-full container.

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DATA EVALUATION RECORD

STUDY 8

CHEM 108501

Pendimethalin

§165-4

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 00156726

Forbis, A.D. 1986. Uptake, depuration, and bioconcentration of <sup>14</sup>C-AC 92,553 by bluegill sunfish (Lepomis macrochirus). Unpublished study performed by Analytical Bio-Chemistry Laboratories, Inc., Columbia, MO, and submitted by American Cyanamid Company, Princeton, NJ.

STUDY ID 00158235

Barringer, D.F. 1986. Prowl herbicide (AC 92,553): Isolation and identification of residues from bluegill sunfish exposed to radiolabeled pendimethalin in a flow-through study. Project No. 0463. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ.

DIRECT REVIEW TIME = 16

REVIEWED BY: J. Harlin

TITLE: Staff Scientist

EDITED BY: K. Patten  
W. Martin

TITLE; Task Leader  
Staff Scientist

APPROVED BY: W. Spangler

TITLE: Project Manager

ORG: Dynamac Corporation  
Rockville, MD  
TEL: 468-2500

APPROVED BY: H. Manning

TITLE: Chemist

ORG: EFGWB/EFED/OPP

TEL: 557-7323

SIGNATURE:

*H. Manning*

CONCLUSIONS:

Laboratory Accumulation - Fish

1. This study is acceptable and fulfills EPA Data Requirements for Registering Pesticides by providing information on the accumulation of methyl-labeled [<sup>14</sup>C]pendimethalin in laboratory fish.

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2. Pendimethalin residues accumulated in bluegill sunfish exposed to 3.0 ppb of pendimethalin, with maximum mean bioconcentration factors of 1400x, 5800x, and 5100x for edible, nonedible, and whole fish tissues, respectively. Pendimethalin comprised 68.2-80.8% of the recovered radioactivity, and the degradate 4-[(1-ethylpropyl)amino]-2-methyl-3,5-dinitro-benzylalcohol (CL 202,347) was 2.0-3.1% of the recovered. Depuration was rapid, with 87-91% of the accumulated [<sup>14</sup>C]residues eliminated from the fish tissues by day 14 of the depuration period.

#### METHODOLOGY:

Bluegill sunfish (*Lepomis macrochirus*; mean length and weight of 45 mm and 2.7 g, respectively) were held in culture tanks on a 16-hour daylight photoperiod for  $\geq 14$  days prior to the initiation of the study. Flow-through aquatic exposure systems were prepared using two 100-L aquaria. Aerated well water (15-20 C, pH 7.8-8.3, dissolved oxygen content 9.2-10.1 ppm, total hardness 225-275 ppm as CaCO<sub>3</sub>, and alkalinity 325-375 ppm as CaCO<sub>3</sub>; Table 1) was provided to each aquarium at a rate of approximately eight turnovers per day (90% replacement/7 hours). The aquaria were immersed in a water bath and maintained at 22  $\pm$  2 C. The flow-through systems were allowed to equilibrate for 24 hours prior to the start of the study.

Then, 120 fish were transferred into each aquarium, and one aquarium was continuously treated with methyl ring-labeled [<sup>14</sup>C]pendimethalin (radiochemical purity 99.43%, specific activity 25.7 uCi/mg, American Cyanamid) at 3.0 ppb. The test concentration was confirmed by LSC prior to transferring the fish. The remaining aquarium served as an untreated control. During the exposure period, water and six fish from each of the treated and control aquaria were sampled on days 0, 0.17, 1, 3, 7, 14, 21, and 28. Additional fish were taken from the treated and control aquaria on days 28 and 35 of the exposure period and day 14 of the depuration period for metabolite identification. Following a 35-day exposure period, water from the treated and control aquaria was siphoned until a depth of approximately 3 inches remained, and the aquaria were filled with 70 L of uncontaminated well water; this procedure was repeated. The fish were then exposed to flowing uncontaminated well water for a 14-day depuration period. During the depuration period, water and six fish were sampled from each aquarium on days 1, 3, 7, 10, and 14.

At each sampling interval, 500-mL aliquots of the water samples were quantified for total [<sup>14</sup>C]residues using LSC. The detection limit was 0.0472 ppb. Additional 500-mL water samples were collected on days 0, 21, and 35 of the exposure period and day 14 of the depuration period for possible metabolite identification.

Pooled (3 fish) edible tissue (body, muscle, skin, skeleton), nonedible tissue (fins, head, internal organs) samples, and whole fish (3) taken at each sampling interval were homogenized with dry

ice and analyzed for total radioactivity by LSC following combustion. Recovery rates of the oxidizer determined prior to sample analyses were 95-100%; the data were not corrected for percentage recovery (Table 12). The detection limits for whole fish, edible tissues, and nonedible tissues were 2.57 ppb, 2.31 ppb, and 2.86 ppb, respectively.

Samples of edible and nonedible tissues from fish taken on days 28 and 35 of the exposure period were stored frozen prior to analysis. The samples were mixed with acetonitrile and centrifuged. The remaining solids were separated from the extracts by filtration. Total radioactivity in the extracts was determined by LSC. The supernatants resulting from extraction were analyzed by one- or two-dimensional TLC on silica gel plates. One of the plates was developed in benzene:dioxane:acetic acid (90:30:1, v:v:v); radioactive zones on this plate were located using a TLC scanner. The remaining samples were analyzed by two-dimensional TLC using solvent systems of methylene chloride:petroleum ether (2:1, v:v) in the first direction and benzene:dioxane:acetic acid (90:30:1, v:v:v) in the second direction. Unlabeled pendimethalin and its 4-hydroxymethyl analog, 4-[(1-ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol (CL 202,347) were cochromatographed with the samples. On separate two-dimensional TLC plates, the samples were compared with the 4-carboxylic acid analog, 4-[(1-ethylpropyl)amino]-3,5-dinitro-*o*-toluic acid (CL 99,900). In addition, the samples were compared with the 6-amino analog, N<sup>4</sup>-(1-ethylpropyl)-3-nitro-*o*-xylene-4,5-diamine (CL 94,756) using two-dimensional TLC plates developed in ethyl acetate:1-propanol:water:formic acid (30:50:15:5, v:v:v:v) in the first direction and ethyl acetate:1-propanol:water:ammonium hydroxide (30:50:15:5, v:v:v:v) in the second direction. Following development, radioactive zones on the plates were located using autoradiography. Radioactive areas were quantified by scraping the radioactive zones from the plates and analyzing the material by LSC.

#### DATA SUMMARY:

[<sup>14</sup>C]Pendimethalin residues accumulated in bluegill sunfish exposed to methyl ring-labeled [<sup>14</sup>C]pendimethalin (radiochemical purity 99.43%) at 3.0 ppb for 35 days under flow-through aquarium conditions. The maximum mean bioconcentration factors were 1400x for edible tissues, 5800x for nonedible tissues, and 5100x for whole fish (Table 5). Maximum mean concentrations of total [<sup>14</sup>C]residues occurred at 35 days and were 4200 ppb for edible tissues, 17000 ppb for nonedible tissues, and 15000 ppb for whole fish (Table 5). The mean concentration of [<sup>14</sup>C]residues in the water during the exposure period was 2.9 ppb. Based on TLC analyses of the 28- and 35-day edible, nonedible, and whole fish samples, pendimethalin comprised 68.2-80.8% of the recovered radioactivity, the degradate

4-[(1-ethylpropyl)amino]-2-methyl-3,5-dinitro-benzyl  
alcohol (CL 202,347)

was 2.0-3.1%, origin material was 1.8-6.6%, and unidentified extractable [<sup>14</sup>C]residues were 0.5-3.2% of the recovered (Table I). Unextracted [<sup>14</sup>C]residues comprised 12.0-26.0% of the recovered radioactivity in the fish tissues.

After 14 days of depuration, [<sup>14</sup>C]residues were 390 ppb in edible tissues and 1900 ppb in both nonedible tissues and whole fish; depuration rates were 91%, 89%, and 87%, respectively (Tables 5 and 6).

Throughout the study, the temperature of the treated water was 21-22 C, the pH ranged from 8.0 to 8.2, and the dissolved oxygen content ranged from 7.6 to 8.6 mg/L; values were comparable to the control aquarium. Total [<sup>14</sup>C]residues in the treated water were 2.2-4.2 ppb during the exposure period.

COMMENTS:

1. Radioactive residues in the water were not characterized; however, data from the photodegradation in water study (Study 1) indicate that it was unlikely that pendimethalin degraded significantly during the 7-hour period required to replace 90% of the aquarium water. The study author stated that additional water samples were collected on days 0, 21, and 35 of the exposure period and on day 14 of the depuration period for possible metabolite characterization; no data were provided for these analyses.
2. No mortality or abnormal behavior was observed in the control and treated fish during the entire study.
3. The detection limits varied for both the water and fish samples, and were dependent upon counting efficiency, sample size, and background levels of radiation for the liquid and combusted samples.
4. Control data from analyses of untreated water and fish were not provided.
5. Recoveries of pendimethalin from fortified fish tissue and water samples were not reported.
6. Based on the information provided regarding TLC analyses of the fish tissue extracts, the study author stated that the samples were compared with the 4-carboxylic acid analog and the 6-amino analog on separate TLC plates. It was unclear whether the samples were cochromatographed with each analog or were chromatographed on separate TLC plates.



7. A preliminary study was performed to determine the acute toxicity of pendimethalin to bluegill sunfish. The 7-day  $LC_{50}$  and 7-day  $EC_{50}$  values were determined to be <2.0 and 1.4 ppm, respectively. In view of these results, the study author chose an exposure level of 3.0 ppb (1/100 of the estimated no-effect level) for the bioaccumulation study.

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DATA EVALUATION RECORD

STUDY 9

CHEM 108501

Pendimethalin

§162-1

FORMULATION--12--Emulsifiable Concentrate (EC)

STUDY ID 41725204

Steller, W.S., and M. Smyth. 1989. Pendimethalin (AC 92,553): Residues of AC 92,553 in soil (postemergence, sandy loam); Kerman, California, 1988. Laboratory Report No. C-3280/Protocol No. PR88CA01. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ.

DIRECT REVIEW TIME = 8

REVIEWED BY: L. Binari

TITLE: Staff Scientist

EDITED BY: K. Ferguson  
W. Martin

TITLE: Task Leader  
Staff Scientist

APPROVED BY: W. Spangler

TITLE: Project Manager

ORG: Dynamac Corporation  
Rockville, MD

TEL: 301-417-9800

APPROVED BY: H. Manning

TITLE: Chemist

ORG: EFGWB/EFED/OPP

TEL: 703-557-7323

SIGNATURE: *H. Manning*

CONCLUSIONS:

Field Dissipation - Terrestrial

1. This study cannot be used to fulfill data requirements at this time.
2. Pendimethalin residues dissipated with a half-life of approximately 34 days from sandy loam soil located in an almond orchard in California that was treated with pendimethalin (Prowl, 4 lb/gallon EC) at 4 lb ai/A. Pendimethalin residues did not appear to leach into soil horizons below 6 inches. Note: There is a very large discrepancy between the laboratory aerobic soil half-life (1322 days) and the terrestrial field dissipation half-life (34 days)!

3. This study is scientifically sound, but does not meet Subdivision N Guidelines for the following reasons:

a description of the extraction procedure was not provided, and it could not be determined if only parent pendimethalin or total pendimethalin residues were extracted from the treated soil;

the pattern of formation and decline of degradates may not have been not addressed.

4. In order for this study to partially fulfill the terrestrial field dissipation data requirement, a complete discussion of the extraction procedure, and isolation and quantification of pendimethalin degradates at all sampling intervals.

#### METHODOLOGY:

Pendimethalin (Prowl, 4 lb/gallon EC, American Cyanamid) was broadcast sprayed at 4 lb ai/A as a pre-emergence weed treatment to a plot (63 x 168 feet) of sandy loam soil (Table IV) in a non-bearing almond orchard located near Kerman, California, on March 25, 1988. An untreated section (63 x 168 feet) of the almond orchard was maintained as a control. Eighteen soil cores (1-inch diameter, 0- to 36-inch depth) were collected using a Giddings soil probe from the treated plot prior to treatment, immediately posttreatment, and at 7, 14, 33, 60, 90, 120, 159, 186, 210, 239, and 270 days posttreatment. Six soil cores were collected from the control plot at 0, 33, 60, 90, and 120 days posttreatment. Soil cores were divided into six 6-inch segments; segments from similar depths were combined, subsampled, and stored frozen between 34 and 531 days prior to analysis.

The procedure used to extract pendimethalin residues from the soil samples was not described. Soil extracts were analyzed for pendimethalin residues by GC with nitrogen-phosphorus detection. The detection limit was 0.05 ppm. Recovery efficiencies from soil samples fortified with pendimethalin at 0.05 to 5.0 ppm ranged from 66 to 126% (average 104%) of the applied (Table II). Results were expressed on a dry soil basis and were not corrected for recovery efficiency.

#### DATA SUMMARY:

Pendimethalin residues dissipated with a calculated half-life of 33.6 days from the upper 6 inches of a plot (63 x 168 feet) of sandy loam soil in an almond orchard in California that was treated with pendimethalin (Prowl, 4 lb/gallon EC) at 4 lb ai/A on March 25, 1988. In the 0- to 6-inch soil layer, pendimethalin residues increased from an average 0.78 ppm immediately posttreatment to 1.04 ppm (maximum 1.12 ppm) at 7 days, then decreased to 0.51 ppm at 33 days, 0.26 ppm

at 60 days, 0.07 ppm at 120 days, and were  $\leq 0.05$  ppm at 159-210 days (Table I).

In the lower soil layers, pendimethalin residues were detected only in a few isolated samples. In the 6- to 12-, 12- to 18-, 18- to 24-, and 24- to 30-inch soil depths, average pendimethalin residues were  $< 0.05$  ppm at all sampling intervals. In the 30- to 36-inch soil depth, average pendimethalin residues were 0.06 ppm (maximum 0.10 ppm) immediately posttreatment and  $< 0.05$  ppm at all other sampling intervals.

During the study (03/25/88 to 12/20/88), rainfall plus irrigation totaled approximately 16 inches, and air temperatures ranged from 32 to 108 F; meteorological data were obtained from the Fresno NOAA station.

#### COMMENTS:

1. The extraction procedure was referenced [Cyanamid SOP M-1453 titled PROWL Herbicide, pendimethalin (CL 92,553): GC Method for the Determination of CL 92,553 Residues], but not provided for review. Without a review of the extraction procedure, it was not possible to determine if only parent pendimethalin or total pendimethalin residues were extracted from the treated soil. In either case, the pattern of formation and decline of degradates was not addressed. The results were presented as "pendimethalin residues".
2. Freezer storage stability data were not provided. It was reported that a freezer storage stability study of pendimethalin was conducted by Analytical-BioChemistry Laboratories of Columbia, MO (ABC Report #36370), and it was found that pendimethalin at 0.10 ppm was stable in soil stored frozen for up to 2 years; however, actual data were not provided for review.
3. Pendimethalin residues were detected in the lower soil depths only in a few samples and primarily at the time 0 sampling interval suggesting contamination occurred during the sampling procedure.
4. The meteorological data provided were barely legible. It was reported by the study authors that air temperature data were obtained from the NOAA station in Fresno located approximately 9 miles from the test site and that rainfall data were recorded at the test site. The rainfall data from the test site matches exactly the rainfall data from the Fresno weather station (Appendix C); therefore, it appears that the rainfall data were not recorded at the test site during the study. Soil temperature data were not provided.
5. The depth to the water table was 10 feet, and the slope of the field was 0.25%.

6. During the study, the plot received 50 units of liquid urea in 1.5 inches of flood irrigation water on May 16, 1988 and was treated with glyphosate (1.5 lb ai/A) on June 1, 1988. The site was planted to almond trees in 1986; prior to 1986, the site was an irrigated pasture. In 1987, the site received a total of 150 lbs of nitrogen (three 50 lb applications in flood water irrigations) and was treated once with glyphosate (1.5 lb ai/A).
7. The study authors calculated a dissipation half-life of 40 days using linear regression and total pendimethalin residues detected in all soil layers (0- to 36-inch soil depth) between 0.1 and 186 days posttreatment; the data from the 159-day sampling interval were not included in the regression (Appendix J). The Dynamac reviewer calculated a dissipation half-life of 33.6 days using linear regression and pendimethalin residues detected in the 0- to 6-inch soil layer between 0 and 120 days posttreatment.

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Pages ~~135~~ through ~~152~~ are not included.

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  - Description of the product manufacturing process.
  - Description of quality control procedures.
  - Identity of the source of product ingredients.
  - Sales or other commercial/financial information.
  - A draft product label.
  - The product confidential statement of formula.
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DATA EVALUATION RECORD

STUDY 10

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CHEM 108501                      Pendimethalin                      §162-1

FORMULATION--12--Emulsifiable Concentrate (EC)

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STUDY ID 41725205

Steller, W.S., and M. Smyth. 1990. Pendimethalin (AC 92,553): Residues of AC 92,553 in soil (postemergence, sandy); Brawley, California, 1988. Laboratory Report No. C-3281/Protocol No. PR88CA02. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ.

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DIRECT REVIEW TIME = 7

---

REVIEWED BY: L. Binari                      TITLE: Staff Scientist


EDITED BY: K. Ferguson                      TITLE: Task Leader  
              W. Martin                                 Staff Scientist

APPROVED BY: W. Spangler                      TITLE: Project Manager

ORG: Dynamac Corporation  
      Rockville, MD  
TEL: 301-417-9800

---

APPROVED BY: H. Manning  
TITLE: Chemist  
ORG: EFGWB/EFED/OPP  
TEL: 703-557-7323

SIGNATURE: 

CONCLUSIONS:

Field Dissipation - Terrestrial

1. This study cannot be used to fulfill data requirements.
2. These data are considered to be of uncertain value and should not be used to predict the environmental behavior of pendimethalin.
3. This study is unacceptable for the following reasons:  
  
the data were too variable to accurately assess the dissipation of pendimethalin residues from the soil;  
  
confidence in the residue data is limited because the soil samples were not accurately labeled when collected in the field



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Pages \_\_\_\_\_ through \_\_\_\_\_ are not included.

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and the soil core layer identities could not be precisely determined; and

the sampling protocol was inappropriate because continuous core samples were not collected below the 0- to 12-inch soil horizon and the sampling technique was varied throughout the study.

4. Since the data were too variable to accurately assess the dissipation of pendimethalin residues from the soil, the soil samples were not accurately labeled upon collection in the field, and the sampling protocol was inappropriate, the problems with this study cannot be resolved with the submission of additional data. A new study must be conducted.

#### METHODOLOGY:

Pendimethalin (Prowl, 4 lb/gallon EC, American Cyanamid) was broadcast-sprayed at 6 lb ai/A as a postemergence treatment to a plot (100 x 400 feet) of sand soil (Table IV) planted to alfalfa located near Brawley, California, on May 9, 1988. The test site had been planted to alfalfa in October of 1986; the alfalfa was cut to a height of 3-4 inches on May 5, 1988. An untreated plot (100 x 300 feet) of alfalfa was maintained as a control. Eighteen soil cores (1-inch diameter) were collected using a Giddings soil probe from the treated plot 6 days prior to treatment, immediately posttreatment, and at 4, 7, 14, 30, 60, 87, 116, 147, 171, 206; and 240 days posttreatment. Soil cores were taken to a 12-inch depth at all sampling intervals, then the cores were divided into 0- to 6- and 6- to 12-inch layers. After the 0- to 12-inch soil sample was collected, the probe was re-inserted 12 inches three separate times, then the upper and lower 4-inch segments were removed from each 12-inch section in an attempt to reduce contamination. The amount of soil removed from the top and bottom of the cores was not consistent between sampling intervals and the following samples were collected: 16- to 23- and 28- to 35-inch depths at days 0, 60, 87, 116, and 147; 14- to 20-, 20- to 26-, 26- to 32-, and 32- to 36-inch depths at day 4; 12- to 18-, 18- to 24-, 24- to 30-, and 30- to 36-inch depths at day 7; 14- to 22-, 24- to 30-, and 30- to 36-inch depths at day 14; and 15- to 23- and 27- to 35-inch depths at day 30. Six soil cores were collected from the control plot 5 days prior to treatment and at 30, 60, 115, and 171 days posttreatment; cores were divided into four to six segments. Soil segments from similar depths were combined, subsampled, and stored frozen from 247 to 554 days prior to analysis.

The procedure used to extract pendimethalin residues from the soil samples was not described. Soil extracts were analyzed for pendimethalin residues by GC with nitrogen phosphorus detection. The detection limit was 0.05 ppm. Recovery efficiencies from soil samples fortified with pendimethalin at 0.05-0.10 ppm ranged from 82 to 128% (average 111%) of the applied (Table II). Results were

expressed on a dry soil basis and were not corrected for recovery efficiency.

DATA SUMMARY:

Pendimethalin residues were highly variable, ranging from <0.05 ppm (detection limit) to 1.54 ppm, in the 0- to 6-inch soil depth up to 87 days after a plot (100 x 400 feet) of sand soil planted to alfalfa in California was treated with pendimethalin (Prowl, 4 lb/gallon EC) at 6 lb ai/A. Pendimethalin residues were <0.05-0.08 ppm at 116 days posttreatment and were not detected at 147 days (Table I). The presence of pendimethalin residues in the lower soil layers was erratic and may have been due to improper handling and/or labeling of the soil core samples. Below the 0- to 6-inch soil horizon, pendimethalin residues were detected at an average 0.24 ppm in the 28- to 35-inch soil depth immediately posttreatment, 0.05 ppm in the 14- to 20-inch depth at 4 days, 0.21 ppm in the 24- to 30-inch depth at 7 days, and 0.09 ppm in the 6- to 12-inch depth at 14 days; residues were <0.05 ppm at all other depths and sampling intervals.

During the study (05/09/88 to 12/31/88), rainfall plus irrigation totaled approximately 72.5 inches, and air temperatures ranged from 24 to 112 F; meteorological data were obtained from the Brawley NOAA station.

COMMENTS:

1. The data were too variable to accurately assess the dissipation of pendimethalin residues from the soil. In the 0- to 6-inch soil layer, average pendimethalin residues were 0.31 ppm immediately posttreatment, increased to 0.65 ppm at 4 days, were not detected (<0.05 ppm) at 7 days, increased to 0.41 ppm at 14 days, then ranged from 0.10 to 0.18 ppm between 30 and 87 days. The variability may have been in part due to improper handling and/or labeling of the soil core samples. It was reported by the study authors that the soil core layer identities could not be precisely determined because the samples were not accurately labeled and that soil cores may have been inverted prior to segmentation and homogenization (see footnote 2 in Table I). Significant residue levels were detected in the 28- to 35-inch soil depth immediately posttreatment and in the 24- to 30-inch depth at 7 days that could not be attributed to leaching.
2. The sampling protocol was inappropriate because continuous core samples were not collected below the 0- to 12-inch soil horizon. The study authors reported that a complete 0- to 36-inch soil core could not be taken because of the "nature of the soil". After the 0- to 12-inch soil sample was collected, the soil probe was re-inserted 12 inches three separate times, then 0-4 inches was removed from the top and bottom of the cores to reduce contamination. On any given sampling interval, there were significant gaps in the residue data;

for example, residue data for day 30 were provided for the 0- to 6-, 6- to 12-, 15- to 23-, and 27- to 35-inch soil depths, but the 12- to 15- and 23- to 27-inch depths were apparently discarded (Table I). In addition, the amount of soil removed from the top and bottom of the cores was not consistent between sampling intervals; therefore, it was not possible to make direct comparisons of residue levels in the soil depths below 12 inches from sampling interval to sampling interval.

3. The extraction procedure was referenced [Cyanamid SOP M-1453 titled PROWL Herbicide, pendimethalin (CL 92,553): GC Method for the Determination of CL 92,553 Residues], but not provided for review. Without a review of the extraction procedure, it was not possible to determine if only parent pendimethalin or total pendimethalin residues were extracted from the treated soil. In either case, the pattern of formation and decline of degradates was not addressed. The results were presented as "pendimethalin residues".
4. Freezer storage stability data were not provided. It was reported that a freezer storage stability study of pendimethalin was conducted by Analytical-BioChemistry Laboratories of Columbia, MO (ABC Report #36370), and it was found that pendimethalin at 0.10 ppm was stable in soil stored frozen for up to 2 years; however, actual data were not provided.
5. Rainfall and air temperature data were obtained from the NOAA station in Brawley located approximately 10 miles from the test site. Soil temperature data were not provided.
6. The depth to the water table was 3-5 feet, and the slope of the field was 0.2%.
7. During the study, the plot received 0-45-0 fertilizer (150 lb/A) in 6 inches of flood irrigation water on June 15, 1988. Alfalfa had been grown on the test site for a number of years prior to the study. The site was treated with 0-45-0 fertilizer (150 lb/A) twice in 1985 and once in the summer of 1986, 11-52-0 fertilizer (300 lb/A) was applied in the fall of 1986, and methomyl and chlorpyrifos in 1986 and 1987.

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DATA EVALUATION RECORD

STUDY 11

CHEM 108501 Pendimethalin §164-2

FORMULATION--12--EMULSIFIABLE CONCENTRATE (EC)

STUDY ID 41245601

Manuel, A. 1980. Analysis for residues of Prowl in soil and in water from Prowl treated rice fields. Laboratory report No. CY 17. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ.

DIRECT REVIEW TIME =16

REVIEWED BY: W. Martin TITLE: Staff Scientist

EDITED BY: K. Ferguson TITLE: Task Leader  
N. Shishkoff Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation  
Rockville, MD  
TEL: 301-417-9800

APPROVED BY: H. Manning  
TITLE: Chemist  
ORG: EFGWB/EFED/OPP  
TEL: 703-557-7323

SIGNATURE:

*H. Manning*

CONCLUSIONS:

Dissipation - Aquatic Field

1. This study cannot be used to fulfill data requirements.
2. These data are considered to be of uncertain value and should not be used to predict the environmental behavior of pendimethalin and its degradates.
3. The portion of this study conducted in Texas is unacceptable for the following reason:

the sampling intervals were inadequate to accurately establish the half-life of the test substance (>50% degraded between two sampling intervals).

164  
~~165~~

The portion of this study conducted in Louisiana is unacceptable for the following reason:

the data were too variable to accurately assess the decline of pendimethalin under field conditions.

4. Since the problems with this study preclude the calculation of an accurate half-life for pendimethalin and cannot be resolved by the submission of additional data, a new study is required.

#### METHODOLOGY:

Texas site: Two vegetated plots (each 8 x 100 feet) of loam soil (52.0% sand, 40.0% silt, 8.0% clay, 0.7% organic matter, pH 4.9) located near Katy, Texas, were treated either with pendimethalin (Prowl; 4 lb/gallon EC, American Cyanamid) at 1.5 lb ai/A or with pendimethalin tank-mixed with propanil at 1.0 and 3.0 lb ai/A, respectively. The plots contained rice (Labelle variety) at the 2-4 leaf stage when the broadcast application was made on April 30, 1980. At 14 days posttreatment, the plots were flooded (water source not specified) to a depth of 3 inches. Water samples (approximately 1 pint/sample) were collected at 15, 29, and 42 days posttreatment (1, 15, and 28 days postflooding). Soil samples (0- to 3- and 3- to 6-inch depths) were collected prior to treatment and at 0, 3, 7, 14, 30, 60, and 94 days posttreatment using a 6-inch soil probe. The soil and water samples were stored frozen prior to shipment to the laboratory for analysis.

Louisiana site: One vegetated plot (6 x 40 feet) of clay soil (4.0% sand, 10.0% silt, 85.0% clay, 1.5% organic matter) located near Crowley, Louisiana, was treated with pendimethalin (Prowl, 4 lb/gallon EC, American Cyanamid) tank-mixed with propanil at 1 and 3.0 lb ai/A, respectively. The plot contained rice (Labelle variety) at the 2-3 leaf stage when the broadcast application was made on July 1, 1980. At 2 days posttreatment, the field was flooded (water source not specified) to a depth of 6 inches. Water samples (1 pint/sample) were collected at 3, 10, 17, and 31 days posttreatment (1, 8, 15, and 29 days postflooding). Soil samples (0- to 4-inch depth) were collected at 0, 3, 7, 14, 30, 60, and 115 days posttreatment using a 6-inch soil probe.

Laboratory analyses: The water samples were analyzed using American Cyanamid Method M-631. An aliquot of each water sample was acidified with 0.1 N hydrochloric acid in aqueous sodium chloride. The acidified solution was partitioned twice with hexane; the hexane fractions were pooled and concentrated to dryness by rotary evaporation. The residues were redissolved in benzene, and an aliquot of the benzene solution was analyzed by GC with electron capture detection. For those samples which were found to contain interfering compounds, the benzene solution was concentrated to

dryness by rotary evaporation, and the residues were redissolved in hexane. The hexane solution was filtered through a Florisil column, then the column was eluted with hexane:benzene (80:20). The eluant was concentrated to dryness by rotary evaporation, and the residues were redissolved in benzene and analyzed by GC with electron capture detection. The limit of detection was 1.0 ppb.

The soil was analyzed using American Cyanamid Method M-520. A subsample of the soil was mixed with water and acidified methanol (2% hydrochloric acid in methanol) and shaken overnight; the ratio of the mixture was 1:2.5:7.5. The mixture was allowed to settle, then the supernatant was filtered. An aliquot of the filtrate was mixed (1:1) with 0.1 N hydrochloric acid and partitioned twice with hexane. The hexane fractions were pooled and concentrated to dryness by rotary evaporation; the resulting residues were dissolved in hexane and chromatographed on a Florisil column eluted with hexane:benzene (80:20). The eluant was concentrated to dryness by rotary evaporation; the residues were redissolved in benzene and analyzed by GC with electron capture detection. The limit of detection was 0.05 ppm.

#### DATA SUMMARY:

In Texas, pendimethalin (4 lb/gallon EC), applied at 1.5 lb ai/A alone or at 1.0 lb ai/A in combination with propanil, dissipated from plots of loam soil planted to rice with an observed half-life of 14-30 days (Table I). In 0- to 3- inch depth of the plots treated at 1.5 and 1.0 lb ai/A, pendimethalin was 0.69 and 0.47 ppm, respectively, immediately after treatment, 0.36 ppm at 14 days, and  $\leq 0.05$  ppm at 30 days; the rapid decrease coincided with the flooding of the plots at 15 days posttreatment (Table IA). Pendimethalin did not leach into the soil; in the 3- to 6-inch soil depths, pendimethalin was 0.24 and 0.14 ppm immediately posttreatment and  $\leq 0.08$  ppm at all other intervals in both plots. In the floodwater from the Texas plot treated with pendimethalin at 1.5 lb ai/A, pendimethalin was 0.00259 ppm and its degradate,

4-[(1-ethylpropyl)amino]-3,5-dinitro-o-toluic acid (CL 99,900), was 0.00242 ppm at 1 day postflooding (15 days posttreatment); both compounds were below the limit of detection ( $< 0.001$  ppm) at all other sampling intervals (Table IB). In the floodwater from the plot treated at 1.0 lb ai/A, pendimethalin was 0.0013 ppm 1 day after flooding (15 days posttreatment) and  $< 0.0010$  ppm at all other sampling intervals.

In Louisiana, the concentration of pendimethalin (4 lb/gallon EC) was extremely variable with no discernable pattern in a plot of clay soil that was planted to rice, treated with pendimethalin (4 lb/gallon EC) at 1.0 lb ai/A in combination with propanil, and flooded at 2 days posttreatment (Table IC). In 0- to 4- inch soil depth, pendimethalin



was 0.18 ppm immediately posttreatment, 0.06 ppm at 3 days, 0.19 ppm at 7 days,  $\leq 0.06$  ppm at 14 and 30 days, 0.14 ppm at 60 days, and 0.05 ppm at 115 days. Pendimethalin was  $<0.0010$  ppm in the floodwater at all sampling intervals (Table ID).

COMMENTS:

General

1. The study author stated that pendimethalin tank-mixed with propanil was used as well as the single active ingredient formulation because the tank mixture is the only pendimethalin product registered for use on rice.
2. Storage stability data were not provided for pendimethalin in either the water or soil substrates. Also, details of the handling of the samples prior to freezer storage and the length of frozen storage were not reported.

Texas site

1. The sampling intervals were inadequate to assess the dissipation of pendimethalin in the soil. Pendimethalin exhibited no evidence of dissipation during the 14 days prior to flooding (0.25-0.47 ppm); the soil was not sampled again until 15 days postflooding, at which time the concentration of pendimethalin had decreased to 0.05 ppm.
2. The CEC of the soil was presented only as a numeric (6), the units were not reported.
3. The meteorological data were incomplete. Temperature data were reported only for April and May. The maximum temperature (air?) was 93 F; the minimum temperature was 46 F. There is a discrepancy in the rainfall plus irrigation data: on page 28, the cumulative precipitation for May 1980 was 3.50 inches; on page 47, it appeared that May rainfall was 10.08 inches. The cumulative rainfall plus irrigation for April through July was 27.56 or 34.14 inches.
4. The year prior to this study, the field plots were a pasture; no pesticides were used.

Louisiana site

1. The data were too variable to assess the dissipation of pendimethalin in the soil. The concentration of pendimethalin in the soil varied from  $<0.05$  to 0.19 ppm with no discernable pattern throughout the course of the study.
2. The soil was not sampled deeply enough to assess the potential of pendimethalin to leach. The soil was sampled only to a depth of 4 inches and all of the soil samples contained pendimethalin.

3. The soil CEC and pH were not reported.
4. The test plot was planted to rice the year prior to the study; pesticide usage was not reported.
5. The meteorological data were incomplete; only the August weather was reported. The maximum temperature (air?) was 99 F, the minimum was 64 F; cumulative rainfall plus irrigation was 2.25 inches.
6. It appeared that no soil samples were taken prior to treatment to confirm that the site was not contaminated.

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DATA EVALUATION RECORD

STUDY 12

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CHEM 108501 Pendimethalin §165-1

FORMULATION--12--EMULSIFIABLE CONCENTRATE  
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STUDY ID 41806801

Lee, T.M. 1989. Pendimethalin (CL 92, 553): Confined accumulation studies of carbon-14 labeled CL 92,553 in lettuce, wheat, carrots, radishes, and snap beans as representative rotational crops (Princeton, New Jersey). Laboratory project ID: CY 38. Unpublished study performed and submitted by American Cyanamid Company, Princeton, NJ.

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DIRECT REVIEW TIME = 16  
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REVIEWED BY: N. Shishkoff TITLE: Staff Scientist

EDITED BY: K. Ferguson TITLE: Task Leader  
L. Mickley Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation  
Rockville, MD  
TEL: 301-417-9800  
-----

APPROVED BY: H. Manning  
TITLE: Microbiologist  
ORG: EFGWB/EFED/OPP  
TEL: 703-557-7323

SIGNATURE:

*H. Manning*

CONCLUSIONS:

Confined Accumulation - Rotational Crops

1. This study cannot be used to fulfill data requirements.
2. [<sup>14</sup>C]Pendimethalin residues accumulated in rotational lettuce, snap beans, radishes, carrots, and wheat that were planted 30-365 days after small field plots of sandy loam soil were treated with 3,4-dimethyl-labeled [<sup>14</sup>C]pendimethalin at approximately 0.75 and 1.64 lb ai/acre (nominal rate 2 lb ai/A). In general, total [<sup>14</sup>C]residues were greatest in the tissue of crops planted at 30 days posttreatment and decreased as the plants matured. The majority (58.6-91.9%) of

the [<sup>14</sup>C]residues in the plants were extractable, and the majority of the extractable residues were not identified.

3. This study is scientifically sound, but does not meet Subdivision N guidelines for the following reasons:

[<sup>14</sup>C]Residues in the crops were not adequately characterized; several [<sup>14</sup>C]compounds were isolated from the plants at up to 0.04 ppm but were not identified, and extracts containing up to 0.10 ppm of [<sup>14</sup>C]compounds and plant tissue containing up to 0.19 ppm were not further analyzed to determine the nature of the residues.

[<sup>14</sup>C]Residues in the soil were not characterized.

The confirmed application rate was only 37-82% of the nominal rate.

4. In order for this study to be used towards the fulfillment of the accumulation in rotational crops data requirement, the registrant must adequately identify pendimethalin residues in the soil and crops.

#### METHODOLOGY:

3,4-Dimethyl-labeled [<sup>14</sup>C]pendimethalin (formulated as a 4 lb/gallon EC; radiochemical purity of the unformulated material >99%, specific activity 25.7 mCi/mg, American Cyanamid) was applied at a nominal rate of 2.0 lb ai/A to small (4 feet x 3 or 6 feet) plots of sandy loam soil (57.6% sand, 40.0% silt, 8.4% clay, 3.1% organic matter, pH 5.7, CEC 13.3 meq/100 g) in Princeton, New Jersey, using a pressurized hand sprayer. Two plots were treated on April 9, 1987, a third on July 9, 1987; the surface 1 inch of soil was raked immediately posttreatment. Nearby plots served as untreated controls. The plots were divided into subplots for planting of various rotational crops (Figure 1). One set of subplots was planted to carrots and spring wheat at 30 days posttreatment, carrots at 90 days, and winter wheat at 120 days; after harvest of these rotational crops, the subplots were planted to carrots and spring wheat at 365 days posttreatment. A second set of subplots was planted to lettuce, radishes, and snap beans at 30 and 90 days posttreatment; after harvest, the subplots were planted to all three crops at 365 days posttreatment. The third plot, divided in half, was planted with winter wheat at 110 days posttreatment and spring wheat at 270 days. The crops were harvested when half-mature and at maturity; weeds were periodically removed from plots (Form 1). When sampled, the lettuce, beans, and wheat were cut 1 inch above the soil surface, and entire carrots and radishes (tops and roots) were collected. Soil cores (0- to 12-inch depth; 1-6 per sampling interval) were collected immediately posttreatment, and following each crop planting and harvest using a zero-contamination tube (Form 9).

The plant samples were washed in distilled water, then mixed in a Waring blender with dry ice until homogenous. The soil cores were divided into 3-inch segments, and corresponding cores were combined, mixed, and air-dried. Portions of the ground plant tissue and the soil were analyzed for total radioactivity using LSC following combustion. The remainder of the plant tissue was stored frozen until further analysis (length of storage not specified); the soil was not further analyzed.

Portions of the plant tissues were refluxed in methanol:water (4:1) at 60 C for 2 hours, filtered, and then refluxed with methanol:0.25 N aqueous hydrochloric acid (1:1) for 2 hours. The two extracts were separately concentrated, then diluted with methanol:water (1:1). Aliquots of the extracts and portions of the extracted plant tissue were analyzed for total radioactivity using LSC and LSC following combustion, respectively. The extract resulting from the first methanol:water reflux was partitioned three times in hexane, then three times with methylene chloride. The hexane phases were combined and evaporated to dryness under vacuum, then redissolved in hexane and analyzed using LSC and one- and two-dimensional TLC. The methylene chloride phases were combined and evaporated to dryness under vacuum, then redissolved in methylene chloride and analyzed using LSC. The extracted aqueous phase was analyzed using LSC and HPLC. The hexane solution was cochromatographed on silica gel plates with nonradiolabeled reference standards of pendimethalin, CL 99,900 (4[(1-ethylpropyl)amino]-3,5-dinitro-o-toluic acid), and CL 202,347 (4[(1-ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol). All plates were developed in one dimension using toluene:p-dioxane:acetic acid (75:25:1). For two-dimensional TLC, plates were developed in the second dimension with ethyl acetate:n-propanol:ammonium hydroxide (50:50:2). Reference standards were visualized using UV. The radioactive bands were located by autoradiography, then scraped from the silica gel plates and analyzed by LSC. The extracted aqueous solution was analyzed using reverse-phase HPLC with a mobile phase of methanol:water:0.2% formic acid. HPLC fractions were collected and quantified using LSC.

If the methanol:acid extract contained "sufficient" radioactivity to warrant further analysis, the methanol was evaporated off the sample. The pH of the resulting aqueous solution was neutralized with NaOH, and the solution was partitioned with hexane and methylene chloride as described. The aqueous solution was centrifuged after partitioning to separate a precipitate that formed; the precipitate was dried and analyzed using LSC following combustion.

The twice-refluxed plant tissue was further analyzed to quantify [<sup>14</sup>C]residues in crude cellulose and lignin. The plant tissue was refluxed under nitrogen at 70 C in dioxane:0.2 N aqueous hydrochloric acid (9:1), then filtered. The resulting filter cake was designated as crude cellulose. The filtrate was concentrated under vacuum with a rotary evaporator, and the concentrate was diluted with water; a precipitate formed which was separated by centrifugation and

designated crude lignin. The crude cellulose and lignin were analyzed using LSC following combustion.

DATA SUMMARY:

[<sup>14</sup>C]Pendimethalin residues accumulated in rotational lettuce, snap beans, radish, carrots, and wheat that were planted 30-365 days after small field plots of sandy loam soil in New Jersey were treated with 3,4-dimethyl-labeled [<sup>14</sup>C]pendimethalin (radiochemical purity >99%) at approximately 0.75 and 1.64 lb ai/acre (nominal rate 2 lb ai/A; Forms 1 and 9). In general, total [<sup>14</sup>C]residues were greatest in the tissue of crops planted at 30 days posttreatment and decreased as the plants matured. The majority (58.6-91.9%) of the [<sup>14</sup>C]residues in the plants were extractable, and the majority of the extractable residues were not identified (Forms 2-7B). Except in spring and winter wheat straw, in which unextracted [<sup>14</sup>C] residues were incorporated in the lignin (16.25 and 21.09%, respectively), the majority of the unextracted [<sup>14</sup>C]residues were incorporated into the cellulose (Form 8).

In lettuce, snap beans, and carrots that were planted 30 days posttreatment and harvested at maturity, total [<sup>14</sup>C]residues (TRR) ranged from 0.07 to 0.52 ppm (Form 1). In lettuce leaves (TRR 0.24 ppm), extractable [<sup>14</sup>C]residues totaled 0.18 ppm, of which 0.03 ppm was soluble in methanol:acid (residues not further characterized), 0.02 ppm was pendimethalin, 0.003 ppm was 4[(1-ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol (CL 202,347), one unidentified compound was 0.033 ppm, and other unidentified compounds were each ≤0.01 ppm (Forms 3 and 3A). In whole beans (TRR 0.07 ppm), extractable [<sup>14</sup>C]residues totaled 0.05 ppm, of which 0.04 and 0.01 ppm were soluble in methanol:water and methanol:acid, respectively (residues not further characterized; Form 3). In bean plants (TRR 0.52 ppm), extractable [<sup>14</sup>C]residues totaled 0.39 ppm, of which 0.09 and 0.04 ppm were soluble in methanol:acid and methylene chloride, respectively; 0.01 ppm was pendimethalin; 0.006 ppm was CL 202,347; one unidentified compound was 0.041 ppm; and other unidentified compounds were each ≤0.025 ppm (Forms 3 and 3B). In carrot tops (TRR 0.20 ppm), extractable [<sup>14</sup>C]residues totaled 0.16 ppm, of which 0.05 ppm was soluble in methanol:acid, 0.02 ppm was pendimethalin; 0.008 ppm was CL 202,347, and several unidentified compounds were each ≤0.01 ppm (Forms 3 and 3C). In carrot roots (TRR 0.29 ppm), extractable [<sup>14</sup>C]residues totaled 0.24 ppm, of which 0.03 ppm was soluble in methanol:acid, 0.13 ppm was pendimethalin, 0.01 ppm was CL 202,347, and several unidentified compounds were each ≤0.023 ppm (Forms 3 and 3D).

In lettuce, snap beans, and carrots that were planted 90 days posttreatment and harvested at maturity, total [<sup>14</sup>C]residues (TRR) ranged from 0.06 to 0.16 ppm; [<sup>14</sup>C]residues in the half-mature crops ranged from 0.15 to 0.59 ppm (Forms 1 and 4-5D). In mature lettuce leaves (TRR 0.06 ppm), extractable [<sup>14</sup>C]residues totaled 0.04 ppm, of

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which 0.03 and 0.01 ppm were soluble in methanol:water and methanol:acid, respectively (residues not further characterized; Form 4). In mature bean plants (TRR 0.16 ppm), extractable [<sup>14</sup>C]residues totaled 0.12 ppm, of which 0.03 ppm were soluble in methanol:acid; 0.02 ppm were soluble in hexane; 0.01 ppm were soluble in methylene chloride; and several unidentified compounds were each ≤0.01 ppm (Forms 4 and 4A). In mature whole beans (TRR 0.02 ppm), extractable [<sup>14</sup>C]residues totaled 0.013 ppm, of which 0.011 and 0.002 ppm were soluble in methanol:water and methanol:acid, respectively (Form 4). In mature carrot tops (TRR 0.09 ppm), extractable [<sup>14</sup>C]residues totaled 0.06 ppm, of which 0.03 and 0.03 ppm were soluble in methanol:water and methanol:acid, respectively (Form 4). In carrot roots (TRR 0.15 ppm), extractable [<sup>14</sup>C]residues totaled 0.14 ppm, of which 0.03 ppm was soluble in methanol:acid, 0.01 ppm was soluble in methylene chloride, 0.03 ppm was soluble in water, 0.07 ppm was pendimethalin, <0.001 ppm was CL 202,347, and several unidentified compounds were each ≤0.001 ppm (Forms 4 and 4B).

In winter wheat that was planted 110 days posttreatment and harvested at maturity, total [<sup>14</sup>C]residues (TRR) were 0.19 ppm in the straw, 0.10 ppm in the chaff, and 0.03 ppm in the seed (Form 1). In the wheat straw (the only plant part further analyzed), extractable [<sup>14</sup>C]residues totaled 0.11 ppm; no extractable fraction contained ≥0.013 ppm of [<sup>14</sup>C]residues (Forms 6 and 6A). In immature wheat forage, TRR were 0.08 ppm (Form 1).

In winter wheat that was planted 120 days posttreatment and harvested at maturity, total [<sup>14</sup>C]residues (TRR) were 0.07 ppm in the straw, 0.04 ppm in the chaff, and 0.01 ppm in the seed; the samples were not analyzed further (Form 1).

In spring wheat that was planted 270 days posttreatment and harvested at maturity, total [<sup>14</sup>C]residues (TRR) were 0.15 ppm in the straw, 0.09 ppm in the chaff, and 0.02 ppm in the seed (Form 1). In the wheat straw (the only plant part further analyzed), extractable [<sup>14</sup>C]residues totaled 0.10 ppm; no extractable fraction contained ≥0.015 ppm of [<sup>14</sup>C]residues (Forms 6 and 6B). In immature wheat forage, TRR were 0.10 ppm (Form 1).

In lettuce, spring wheat, snap beans, radishes, and carrots that were planted 365 days posttreatment and harvested at maturity, total [<sup>14</sup>C]residues (TRR) ranged from 0.02 to 0.15 ppm; [<sup>14</sup>C]residues in the half-mature crops ranged from 0.03 to 0.19 ppm (Form 1). Total [<sup>14</sup>C]residues (TRR) were 0.15, 0.06, and 0.02 ppm in the spring wheat straw, chaff, and seed, respectively; 0.06 ppm in whole beans; 0.02 and 0.04 ppm in radish tops and roots; and 0.02 and 0.05 ppm in carrot tops and roots (no additional characterization of these residues; Form 1). In mature lettuce leaves (TRR 0.12 ppm), extractable [<sup>14</sup>C]residues totaled 0.071 ppm, individual extractable fractions contained 0.001-0.04 ppm of [<sup>14</sup>C]residues (Form 7 and 7A). In mature bean plants (TRR 0.14 ppm), extractable [<sup>14</sup>C]residues



totalled 0.095 ppm, individual extractable fractions contained <0.001-0.012 ppm of [<sup>14</sup>C]residues (Form 7 and 7B).

Soil [<sup>14</sup>C]residues (uncharacterized) were 0.75-0.76 ppm in the 0- to 3-inch depth of the plots treated in April, 1987, and used for the 30-, 90-, 120-, and 365-day rotations, and were 1.64 in the plot treated in July 1987 and used for the 110- and 270-day (wheat) rotations (Form 9). The [<sup>14</sup>C]residues remained primarily in the 0- to 3-inch depth, where, during the 16 months following application, they varied from 0.55 ppm to 1.97 ppm with a gradual downward trend over time (Form 9). [<sup>14</sup>C]Residues in the 3- to 6-inch depth were ≤0.04 ppm, except for 0.15 ppm at 4.5 months posttreatment and 0.07 at 16 months. [<sup>14</sup>C]Residues in the 6- to 9- and 9- to 12-inch depths were determined sporadically, and were <0.01 to 0.07 ppm.

During the study, air temperatures ranged from -7 to 101 F. Rainfall totaled 53.81 inches in 1987 and 28.44 inches in 1988 (January-September only).

#### COMMENTS:

1. [<sup>14</sup>C]Residues in the crops were not adequately characterized. Subdivision N guidelines specify that all compounds present at ≥0.01 ppm be identified. However, several [<sup>14</sup>C]compounds were isolated from the plants at up to 0.04 ppm but were not identified, and extracts containing up to 0.10 ppm of [<sup>14</sup>C]compounds and plant tissue containing up to 0.19 ppm were not further analyzed to determine the nature of the residues.
2. [<sup>14</sup>C]Residues in the soil were not characterized. The uptake of [<sup>14</sup>C]residues by the plant can therefore be related only to the concentration of total residues in the soil, not to the presence of specific compounds.
3. Although the plots were treated at a nominal rate of 2 lb ai/A, the plots treated in April (30-, 90-, 110-, and 365-day rotations) contained only 0.75 lb ai/A immediately posttreatment (although 1.97 lb ai/A were detected by 2.75 months) and the plots treated in July (120- and 270-day rotations) contained only 1.65 lb ai/A.
4. Pendimethalin was labeled on methyl groups attached to the benzene ring portion of the molecule, and identification of residues was done using radiochromatography only. Therefore, it is impossible to determine the concentration of residues containing only the unlabeled aromatic portion of the parent material that accumulated in the plants.
5. Method detection limits and recovery efficiencies were not reported.
6. Freezer storage stability data are provided in Study 2 (MRID 41725206) of this report.

7. A detailed description of the test site was provided in the supplemental document. The land was described as flat and well-drained, and had been farmland prior to 1969, when it was used to house turkeys for two months. After 1970 "pest control measures were given only to specific test plots... and only as needed."
8. A 4 EC formulation of pendimethalin (CL 92,553) was made by dissolving 43.79 mg of [<sup>14</sup>C]pendimethalin (labeled in methyl groups at the 3- and 4- positions on the benzene ring, radiochemical purity >99%, specific activity 25.7 mCi/mg, American Cyanamid) and 206.35 mg of unlabeled pendimethalin (purity 97.5%, American Cyanamid) in monochlorobenzene, plus Flo-Mo-Pla, and Flo-Mo-Phn formulation reagents.

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Page \_\_\_\_\_ is not included in this copy.

Pages 190 through 223 are not included.

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The material not included contains the following type of information:

- Identity of product inert ingredients.
  - Identity of product impurities.
  - Description of the product manufacturing process.
  - Description of quality control procedures.
  - Identity of the source of product ingredients.
  - Sales or other commercial/financial information.
  - A draft product label.
  - The product confidential statement of formula.
  - Information about a pending registration action.
  - FIFRA registration data.
  - The document is a duplicate of page(s) \_\_\_\_\_.
  - The document is not responsive to the request.
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The information not included is generally considered confidential by product registrants. If you have any questions, please contact the individual who prepared the response to your request.

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DATA EVALUATION RECORD

STUDY 13

CHEM 108501

Pendimethalin

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 41725206

Smyth, M., D. Koch, and J. Smith. 1990. Pendimethalin (AC 92,553): Freezer stability in soil. Laboratory Report No: C-3467. Performed by American Cyanamid Company, Princeton, NJ, and Analytical Bio-Chemistry Laboratories, Inc. Columbia, MO. and submitted by American Cyanamid Company, Princeton, NJ.

DIRECT REVIEW TIME = 5

REVIEWED BY: N. Shishkoff TITLE: Staff Scientist

EDITED BY: K. Ferguson TITLE: Task Leader  
L. Mickley Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation  
Rockville, MD  
TEL: 301-417-9800

APPROVED BY: H. Manning  
TITLE: Microbiologist  
ORG: EFGWB/EFED/OPP  
TEL: 703-557-7323

SIGNATURE:

*H. Manning*

CONCLUSIONS:

Ancillary Study- Freezer Storage Stability

1. Pendimethalin was stable in soil stored frozen at -15 to -20 C for 2 years.
2. This study is scientifically sound. Based on the information provided in this study, soil samples containing pendimethalin may be stored frozen for up to 2 years prior to analysis.
3. No additional information on storage stability of pendimethalin is required at this time. However, if pendimethalin is stored for

longer than 2 years prior to analysis, additional storage stability information will be required.

#### METHODOLOGY:

Portions (25 g) of a "composite control soil" (soil not further characterized) were treated at 0.5 ppm (12.5 ug/25 g) with pendimethalin (purity 99.5%, American Cyanamid) dissolved in hexane. The hexane was evaporated in a stream of nitrogen, and the samples were placed in a freezer maintained at -15 to -20 C. Additional untreated soil samples were frozen with the treated samples for later use. Two treated and three untreated soil samples were removed from the freezer at 0, 1, 2, 3, 6, 12, 18, and 24 months posttreatment.

The soil samples were thawed, and the untreated soils were treated with pendimethalin at 0.5 ppm. The soils were mixed with deionized water and acidic methanol (2% HCl) on a reciprocating shaker for 1 hour. A 25-mL aliquot of each supernatant was filtered, then acidified with 0.1 HCl. The acidified solutions were adsorbed to a C18 column, and the columns were eluted twice with 1% methanol in hexane. The eluants were combined and evaporated to dryness, then resuspended in hexane for analysis using GC with thermal detection. The limit of detection for pendimethalin was 0.05 ppm.

#### DATA SUMMARY:

Pendimethalin (purity 99.5%) was stable when stored frozen (-15 to -20 C) in uncharacterized "composite control" soil at 0.5 ppm for up to 2 years (Table 1). Recovery of pendimethalin from soil samples that were stored up to 2 years was 81-100%, compared to recoveries of 59-97% for freshly fortified concurrent samples.

#### COMMENTS:

1. The soil was described only as a composite control soil. Characteristics such as texture, organic matter content, pH, and CEC were not provided.
2. The soil samples were analyzed only for pendimethalin.
3. During the first year of the study, the eluants were evaporated under a nitrogen stream prior to GC analysis. This procedure was discontinued due to possible effects on percent recovery. The remainder of the samples were rotoevaporated, resuspended in hexane, transferred to culture tubes, and evaporated with nitrogen until the correct volume for chromatographic analysis was attained.
4. In general, freezer temperatures ranged from -15 to -20 C, with deviations from -35 to 7 C.

-13.2-13.3-

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Page \_\_\_\_\_ is not included in this copy.

Pages 226 through 228 are not included.

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The material not included contains the following type of information:

- Identity of product inert ingredients.
  - Identity of product impurities.
  - Description of the product manufacturing process.
  - Description of quality control procedures.
  - Identity of the source of product ingredients.
  - Sales or other commercial/financial information.
  - A draft product label.
  - The product confidential statement of formula.
  - Information about a pending registration action.
  - FIFRA registration data.
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Environmental Fate & Effects Division  
 PESTICIDE ENVIRONMENTAL FATE ONE LINE SUMMARY  
**PENDIMETHALIN**

Last Update on June 14, 1991

[V] = Validated Study    [S] = Supplemental Study    [U] = USDA Data

LOGOUT	Reviewer: <i>HJM</i>	Section Head: <i>e</i>	Date: <i>6/29/91</i>
--------	----------------------	------------------------	----------------------

Common Name: PENDIMETHALIN

Smiles Code: c(c(c(c1N(=O)=O)C)C)c(N(=O)=O)c1NC(CC)CC

PC Code # : 108501      CAS #: 40487-42-1      Caswell #:

Chem. Name : N-(1-ETHYLPROPYL)-3,4-DIMETHYL-2,6-DINITROBENZENEAMINE

Action Type: Herbicide

Trade Names: PROWL

(Formul'tn): G, DISPERSABLE GRANULAR, EC

Physical State:

Use : WEEDS - SOYBEANS, COTTON, CORN, BEANS, PEANUTS, POTATOES,  
 Patterns : RICE, SORGHUM, SUNFLOWER, TOBACCO, ORNAMENTALS, NON-BEARING  
 (% Usage) : FRUIT AND NUT, VINEYARDS, i.e. Terr Food/Non-Food, Aquatic.  
 : Food.

Empirical Form:  $C_{13}H_{19}N_3O_4$   
 Molecular Wgt.: 281.31      Vapor Pressure: 2.90E -6 Torr  
 Melting Point : °C      Boiling Point: °C  
 Log Kow :      pKa: @ °C  
 Henry's : 2.22E -5 Atm. M3/Mol (Measured)      2.15E -6 (calc'd)

Solubility in ...					Comments
Water	0.50E	ppm	@20.0	°C	
Acetone	E	ppm	@	°C	
Acetonitrile	E	ppm	@	°C	
Benzene	E	ppm	@	°C	
Chloroform	E	ppm	@	°C	
Ethanol	E	ppm	@	°C	
Methanol	E	ppm	@	°C	
Toluene	E	ppm	@	°C	
Xylene	E	ppm	@	°C	
	E	ppm	@	°C	
	E	ppm	@	°C	

Hydrolysis (161-1)

[V] pH 5.0: STABLE	Reviewed for Reg. Std. 3/85.
[V] pH 7.0: STABLE	
[V] pH 9.0: STABLE	
[ ] pH :	
[ ] pH :	
[ ] pH :	

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Environmental Fate & Effects Division  
PESTICIDE ENVIRONMENTAL FATE ONE LINE SUMMARY  
PENDIMETHALIN

Last Update on June 14, 1991

[V] = Validated Study [S] = Supplemental Study [U] = USDA Data

Photolysis (161-2, -3, -4)

- [ ] Air :
- [S] Soil :Stable on sandy loam soil, exposed to Xenon lamp.
- [S] Water:T1/2= 21 days after exposure to Xenon lamp.
- [ ] : Reviewed 6/91.
- [ ] :
- [ ] :

Aerobic Soil Metabolism (162-1)

- [S] T1/2= 1322 days in sandy loam soil. Reviewed 6/91.
- [ ]
- [ ]
- [ ]
- [ ]
- [ ]
- [ ]

Anaerobic Soil Metabolism (162-2)

- [S] Relatively stable (parent was 98.0% of applied after 60 days anaerobic incubation. Reviewed 6/91.
- [ ]
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- [ ]
- [ ]

Anaerobic Aquatic Metabolism (162-3)

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- [ ]

Aerobic Aquatic Metabolism (162-4)

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Environmental Fate & Effects Division  
PESTICIDE ENVIRONMENTAL FATE ONE LINE SUMMARY

PENDIMETHALIN

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Soil Partition Coefficient (Kd) (163-1)

[ ]  
[ ]  
[ ]  
[ ]  
[ ]  
[ ]

Soil Rf Factors (163-1)

[S] AGED RESIDUES WERE IMMOBILE Reviewed for Reg Std 3/85.  
[ ] IN SdLm COLUMN AFTER LEACHING  
[ ] WITH 22.5" OF WATER IN 45 DAY  
[ ]  
[ ]  
[ ]

Laboratory Volatility (163-2)

[V] rate was  $2.1 \times 10^{-3}$  ug/cm sq/hour over 24 hour period.  
[ ] Reviewed 6/91.

Field Volatility (163-3)

[ ]  
[ ]

Terrestrial Field Dissipation (164-1)

[S] T<sub>1/2</sub> = 34 days in sandy loam in CA; no leaching below 6 inches.  
[ ] Reviewed 6/91.  
[ ]  
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[ ]  
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[ ]

Aquatic Dissipation (164-2)

[ ]  
[ ]  
[ ]  
[ ]  
[ ]  
[ ]

Forestry Dissipation (164-3)

[ ]  
[ ]

Environmental Fate & Effects Division  
PESTICIDE ENVIRONMENTAL FATE ONE LINE SUMMARY

PENDIMETHALIN

Last Update on June 14, 1991

[V] = Validated Study [S] = Supplemental Study [U] = USDA Data

Long-Term Soil Dissipation (164-5)

[ ]  
[ ]

Accumulation in Rotational Crops, Confined (165-1)

[S] Residues accumulated in lettuce, snap beans, radishes, carrots,  
[ ] wheat planted 30-365 days in sandy loam soil. Rev'd 6/91.

Accumulation in Rotational Crops, Field (165-2)

[ ]  
[ ]

Accumulation in Irrigated Crops (165-3)

[ ]  
[ ]

Bioaccumulation in Fish (165-4)

[V] Bioaccum Factors: 1400X (edible), 5800X (nonedible), 5100X (whole  
[ ] fish). Reviewed 6/91.

Bioaccumulation in Non-Target Organisms (165-5)

[ ]  
[ ]

Ground Water Monitoring, Prospective (166-1)

[ ]  
[ ]  
[ ]  
[ ]

Ground Water Monitoring, Small Scale Retrospective (166-2)

[ ]  
[ ]  
[ ]  
[ ]

Ground Water Monitoring, Large Scale Retrospective (166-3)

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[ ]  
[ ]  
[ ]

Ground Water Monitoring, Miscellaneous Data (158.75)

[ ]  
[ ]  
[ ]

Environmental Fate & Effects Division  
PESTICIDE ENVIRONMENTAL FATE ONE LINE SUMMARY  
PENDIMETHALIN

Last Update on June 14, 1991

[V] = Validated Study [S] = Supplemental Study [U] = USDA Data

Field Runoff (167-1)

[ ]  
[ ]  
[ ]  
[ ]

Surface Water Monitoring (167-2)

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[ ]  
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[ ]

Spray Drift, Droplet Spectrum (201-1)

[ ]  
[ ]  
[ ]  
[ ]

Spray Drift, Field Evaluation (202-1)

[ ]  
[ ]  
[ ]  
[ ]

Degradation Products

In aerobic soil: 2,6-dinitro-3,4-xylidine (CL 84,846)  
4-[Cl-ethylpropyl)amino]-3,5-dinitro-o-toluic acid (CL 99,900)  
4-[(1-ethylpropyl)amino]-2-methyl-3,5-dinitro-benzyl alcohol  
(CL 202,347)

Environmental Fate & Effects Division  
PESTICIDE ENVIRONMENTAL FATE ONE LINE SUMMARY  
**PENDIMETHALIN**

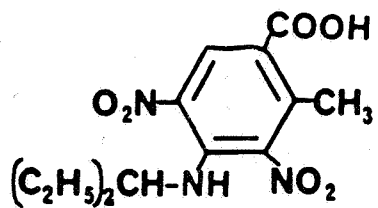
Last Update on June 14, 1991

[V] = Validated Study [S] = Supplemental Study [U] = USDA Data

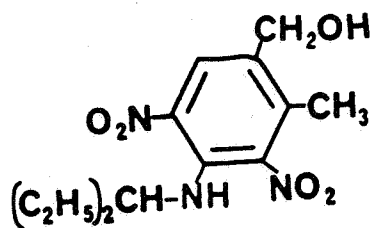
Comments

Freezer Storage Stability Study: pendimethalin was stable up to two years when frozen -15 to -20 C. Recovery was 81-100%.  
Rev'd 6/91.

References: EPA REVIEWS, Reg. Std. and 6/91.  
Writer : H. Manning

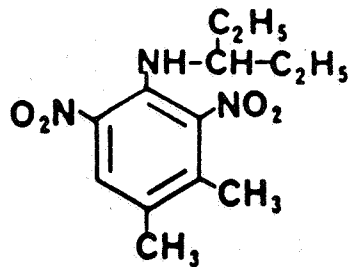


4-[(1-ethylpropyl)amino]-3,5-dinitro-*o*-toluic acid,  
CL 99,900

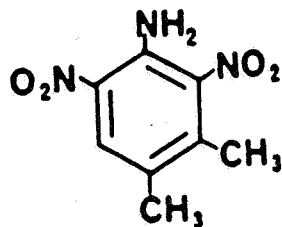


4-[(1-ethylpropyl)amino]-2-methyl-3,5-dinitrobenzylalcohol,  
CL 202,347

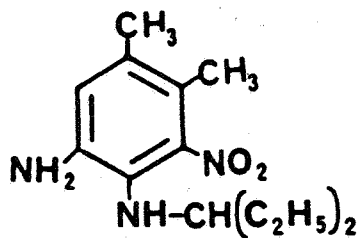




N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitrobenzenamine,  
Pendimethalin, CL 92,553



2,6-dinitro-3,4-xylidine,  
CL 84,846



N'-(1-ethylpropyl)-3-nitro-o-xylene-4,5-diamine,  
CL 94,756

APPENDIX

PENDIMETHALIN AND ITS DEGRADATES