Flumetsulam
129016

Shaughnessy No.: _____

Date out of EFGWB: FEB 2 1990

TO: J. Miller/R. Ikeda
Product Manager #23
Registration Division (H7507C)

FROM: Emil Regelman, Supervisory Chemist
Chemistry Review Section #2
Environmental Fate and Ground Water Branch

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

Hank Jacoby 2/26/90

Attached, please find the EFGWB review of ...

Reg./File #: 464-EUP-RNG

Chemical Name: N-(2,6-difluorophenyl)-5-methyl-1,2,4-triazolo[1,5a] pyrimidine-2-sulfonamide

Type Product: Herbicide

Common Name: XRD-498

Company Name: Dow Chemical USA

Purpose: Application for an Experimental Use Permit

Date Received: 28 Nov. 1989 Date Completed: 23 Jan. 1990

Action Code: 700

EFGWB #(s): 90-0116

Total Reviewing Time: 1.1 days

Deferrals to: ___ Ecological Effects Branch, EFED

___ Science Integration and Policy Staff, EFED

___ Non-Dietary Exposure Branch, HED

___ Dietary Exposure Branch, HED

___ Toxicology Branch

1. CHEMICAL:

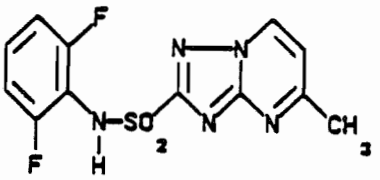
Chemical name: N-(2,6-difluorophenyl)-5-methyl-1,2,4-triazolo[1,5a] pyrimidine-2-sulfonamide

CAS no.: 98967-40-9

Common name: XRD-498

Trade name: DR-0238-5651/K-170,711

Chemical structure:



Molecular formula: $C_{12}H_9F_2N_5O_2S$

Molecular weight: 325.3

Formulation: N-(2,6-difluorophenyl)-5-methyl(1,2,4)triazolo[1,5a] pyrimidine-2-sulfonamide.....74.9%
Inert Ingredients.....25.1%

Physical/Chemical properties of active ingredient:

Physical characteristics: Light tan power

Melting point: 253°C

Vapor Pressure: 0.8 X 10^{-15} mmHg at 20°C

Solubility: 49.1 mg/l at pH 2.5 (25°C)
5.65 g/l at pH 7.0 (25°C)

Octanol/water partition coefficient: Kow = 1.62

2. TEST MATERIAL:

Active ingredient

3. STUDY/ACTION TYPE:

Application for an Experimental USe Permit.

2

4. STUDY IDENTIFICATION:

Lehmann, R.and Miller J. AQUEOUS HYDROLYSIS OF XRD-498. Sponsored and Submitted by Dow Chemical Company USA under Laboratory Project No. GH-C 2092; Received by EPA 28 September 1989; MRID No. 41263229.

Laskowshi, D., Lehmann, R., Goodwin, P., Stanley, D., French, B., and Miller, J. AEROBIC SOIL DEGRADATION OF XRD-498. Sponsored and Submitted by Dow Chemical Company USA, Midland, MI under Laboratory Project ID GH-C 2160; Received by EPA 28 September 1989; MRID No. 41263230.

Lehmann, R., Laskowski, A., Miller, J., Stanley, D., and Fontaine, D. EFFECT OF SOIL PROPERTIES ON THE DEGRADATION AND SORPTION OF XDR-498. Sponsored and Submitted by Dow Chemical Company USA under Laboratory Project ID GH-C 2243 and Protocol No. 87062; Received by EPA 28 September 1989; MRID No. 41290403.

Goodwin, P., Lehmann, R. and Miller, J. SOIL ADSORPTION/DESROPTION OF ^{14}C-XRD-498. Sponsored and Submitted by Dow Chemical Company USA, Midland, MI under Laboratory Project ID GH-C 2159; Received by EPA 28 Sept. 1989; MRID No. 41263231.

Hamburg, A., Miller, J.H., Lardie, T.S., and Baldwin, W.S. ^{14}C-XRD-498: CONFINED ACCUMULATION STUDY ON ROTATIONAL CROPS PLANTED AT 30 AND 120 DAYS AFTER SOIL TREATMENT. Sponsored and Submitted by Dow Chemical Company USA, Midland, MI under Laboratory Project ID GH-C 2170; Received by EPA 28 September 1989; MRID No. 41263232.

5. REVIEWED BY:

Gail Maske
Chemist, Review section #2
OPP/EFED/EFGWB

Signature: Gail Maske
Date: 2/9/90

6. APPROVED BY:

Emil Regelman
Supervisory Chemist
Review section #2
OPP/EFED/EFGWB

Signature:
Date: 2/2/90

7. CONCLUSIONS:

a. Hydrolysis:

The hydrolysis study is acceptable to meet Subdivision N data requirement. No further hydrolysis data is required at this time.

[^{14}C]XRD-498 was stable in sterile aqueous pH 5, 7, and 9 buffered solutions at 25°C for 66 days.

b. Aerobic Soil Metabolism:

The aerobic soil metabolism studies only partially fulfill Subdivision N data requirement. To fulfill the aerobic soil metabolism data requirement, a study of the aerobic soil metabolism of the phenyl-labelled [^{14}C]XRD-498 is required. No further data on the aerobic soil metabolism of 5-triazolopyrimidine-labelled [^{14}C]XRD-498 required at this time.

5-triazolopyrimidine-labelled [^{14}C]XRD-498 degraded with half-lives of 23-130 days under aerobic conditions in sandy loam, clay, silt loam, and loam soils. The study was carried out in the dark at 25°C with 75% of 1/3 bar moisture maintained.

c. Leaching, Adsorption/Desorption:

The leaching, adsorption/desorption mobility studies are not acceptable to meet Subdivision N data requirement. Study (MRID 4129040) was conducted using only one concentration of XRD-498, and the desorption data was not furnished. In the study (MRID 41263231) the experimental design was inadequate to accurately determine the actual desorption of XRD-498 and its degradates from the soil. A new leaching, adsorption/desorption mobility study is required.

The batch equilibrium studies did demonstrate [^{14}C]XRD-498 to be very mobile in twenty-three soils.

d. Confined Accumulation - Rotational Crops

The confined accumulation-rotational crops study is not acceptable to meet Subdivision N data requirement. There was no storage stability data for the soil and plant substrates furnished. Most samples were only analyzed for total ^{14}C-residue and not for parent and/or degradate(s). There was no data furnished to confirm the stated application rate. A new confined accumulation-rotational crops study is required.

The study demonstrated accumulation of [^{14}C]XRD-498 and its degradates at low levels in lettuce, carrot roots and tops, soybeans, green bean beans and their pods. However, larger levels were shown in soybean plant trash, wheat straw/chaff, and green bean whole plants.

e. Leaching and Groundwater contamination characteristics

A comparison of XRD-498's properties to pesticide properties which have been found characteristic of some pesticides known to leach to ground water are given below:

Property	Pesticides known to leach	Isoxaben
Water solubility	>30 ppm	5.65 g/l
K_d	<5.0 usually <1.0 or 2.0	0.05 - 2.4
K_{oc}	<300 - 500	7 - 25
Speciation	Negatively charge	slightly negative
Henry's Constant	$<10^{-2}$ atm-m^3/mol	1.9 x 10^{-16} Lxatm/mmol
Hydrolysis	> 25 weeks	stable @ 66 days
Photolysis	> 1 week	no data
Soil	> 2 to 3 weeks	25 - 130 days in aerobic soil study

Pesticides that are generally persistent and mobile are capable of leaching to ground water. The persistence characteristics and the the mobility characteristics are typical of pesticides found in ground water. The physical persistence and mobility characteristics do appear to make XRD-498 a concern for leaching and ground water contamination. However, there is not enough data available to fully assess the leaching ability of XRD-498 to ground water under environmental conditions.

f. The environmental fate data is inadequate to support the proposed Experimental Use Permit (EUP). New studies are still needed to support the mobility, accumulation in rotational crops, and accumulation in fish data requirements.

8. RECOMMENDATIONS:

The registrant should be informed of the following:

a. Environmental fate data are inadequate to support the proposed EUP. New studies are still needed to support the mobility, accumulation in rotational crops, and accumulation in fish data requirements.

b. The hydrolysis study is acceptable to meet Subdivision N data Requirement.

c. The aerobic soil metabolism studies only partially fulfill Subdivision N data requirement. To fulfill the aerobic soil data requirement, a study of the aerobic soil metabolism of phenyl-labelled [^{14}C]XRD-498 is required. However, no further data on the aerobic metabolism of 5-triazolopyrimidine-labelled [^{14}C]XRD-498 is required at present.

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d. The leaching, adsorption/desorption mobility studies are not acceptable to meet Subdivision N data requirement. A new leaching, adsorption/desorption study is required.

The study (MRID 41290403) does not meet the guidelines for the following reasons:
1. Only one concentration of XRD-498 was used; therefore, the Freudlich K_{ads} values could not be accurately determined.
2. The study did not address the desorption phase of the test.
3. The temperature monitoring data was not furnished.
4. The soils were not analyzed to confirm adsorption.
5. A material balance was not furnished.

In the study (MRID 41263231), the experimental design was not adequate to accurately determine the actual desorption of XRD-498 and its degradates.

e. The confined accumulation-rotational crops study is not acceptable to meet Subdivision N data requirement. A new study is required for the following reasons:
1. No storage stability data was furnished with the study. The soil and plant samples were stored frozen for 118 and 347 days, respectively.
2. Most samples were analyzed only for total ^{14}C-residue and not for parent and/or degradate(s).
3. The application rate was not confirmed.

f. The status of the Environmental Fate Data Requirements for an experimental use (terrestrial food crop) permit is as follows:

Environmental Fate Data Requirement	Status of Data Requirement	MRID No.
Degradation Studies-Lab		
161-1 Hydrolysis	Satisfied	41263229
Metabolism Studies-Lab		
162-1 Aerobic soil	Satisfied (for EUP)	41263230
Mobility Studies		
163-1 Leaching, Adsorption/ Desorption	New study needed	41263231 41290403
Accumulation Studies		
165-1 Rotational crops-confined 165-4 in Fish	New study needed Not Submitted	41263232

9. BACKGROUND:

XRD-498 is a selective experimental herbicide proposed for use to control broadleaf weeds in soybeans and field corn. At this time, it is for use only at an application site of a cooperator and in accordance with the terms and conditions of the experimental use permit. Single active ingredient formulations include 75% G. XRD-498 may be applied using preplant incorporation, preemergence, or postemergence treatment. Proposed application rates are Ø.Ø3-Ø.13 lb ai/A for preplant incorporation and preemergence treatment; postemergence rates on field corn are Ø.Ø15-Ø.Ø62 lb ai/A, and postemergence rates on soybeans are Ø.ØØ78-Ø.Ø15 lb ai/A. Application is by ground spray; sufficient agitation should be maintained during mixing and spraying to ensure a uniform spray mixture. When applied by preplant incorporation, XRD-498 should be incorporated into the top 2 to 3 inches of the final seedbed. Preemergence and postemergence applications are made by broadcast spraying. Livestock should not be allowed to graze in treated areas, and harvest-treated silage or grain should not be feed to meat or dairy animals.

1Ø. DISCUSSION:

See attached DERs.

11: COMPLETION OF ONE-LINER:

See attached one-liner.

12: CBI APPENDIX:

The information is considered to be CBI by the registrant, and should be treated as such.

ENVIRONMENTAL FATE & GROUND WATER BRANCH
PESTICIDE ENVIRONMENTAL FATE ONE LINE SUMMARY

Common Name: **XRD-498** Date: 01/23/90
Chem. Name : N-(2,6-difluorophenyl)-5-methyl-1,2,4-triazolo[1,5a]
: pyrimidine-2-sulfonamide
Shaugh. # : CAS Number: 98967-40-9
Type Pest. : Herbicide
Formulation: 75G
Uses : used to control broadleaf weeds on soybeans and field corn
:
:

Empir. Form: $C_{12}H_9F_2N_5O_2S$ VP (Torr): 10-15
Mol. Weight: 325.3 Log Kow : 0.209
Solub.(ppm): 5650 at pH 7 @ 25 ^{o}C Henry's :

Hydrolysis (161-1)	Photolysis (161-2, -3, -4)
pH 5:[#] Stable	Air :[ ]
pH 7:[#] Stable	Soil :[ ]
pH 9:[#] Stable	Water:[ ]
pH :[ ]	:[ ]
pH :[ ]	:[ ]
pH :[ ]	:[ ]

MOBILITY STUDIES (163-1)

Soil Partition (Kd)	Rf Factors
1.[ ]	1.[ ]
2.[ ]	2.[ ]
3.[ ]	3.[ ]
4.[ ]	4.[ ]
5.[ ]	5.[ ]
6.[ ]	6.[ ]

METABOLISM STUDIES (162-1,2,3,4)

Aerobic Soil (162-1)	Anaerobic Soil (162-2)
1.[ ]	1.[ ]
2.[ ]	2.[ ]
3.[ ]	3.[ ]
4.[ ]	4.[ ]
5.[ ]	5.[ ]
6.[ ]	6.[ ]
7.[ ]	7.[ ]

Aerobic Aquatic (162-4)	Anaerobic Aquatic (162-3)
1.[ ]	1.[ ]
2.[ ]	2.[ ]
3.[ ]	3.[ ]
4.[ ]	4.[ ]

[*] - Acceptable Study. [#] = Supplemental Study

8

Common Name: **XRD-498** Date: 01/23/90

VOLATILITY STUDIES (163-2,3)

[ ] Laboratory:
[ ] Field:

DISSIPATION STUDIES (164-1,2,3,5)

Terrestrial Field (164-1)
1.[ ]
2.[ ]
3.[ ]
4.[ ]
5.[ ]
6.[ ]

Aquatic (164-2)
1.[ ]
2.[ ]
3.[ ]
4.[ ]
5.[ ]
6.[ ]

Forestry (164-3)
1.[ ]
2.[ ]

Other (164-5)
1.[ ]
2.[ ]

ACCUMULATION STUDIES (165-1,2,3,4,5)

Confined Rotational Crops (165-1)
1.[ ]
2.[ ]

Field Rotational Crops (165-2)
1.[ ]
2.[ ]

Irrigated Crops (165-3)
1.[ ]
2.[ ]

Fish (165-4)
1.[ ]
2.[ ]

Non-Target Organisms (165-5)
1.[ ]
2.[ ]

Common Name: **XRD-498** Date: 01/23/90

GROUND WATER STUDIES (158.75)

1.[ ]
2.[ ]
3.[ ]

DEGRADATION PRODUCTS

1.
2.
3.
4.
5.
6.
7.
8.
9.
10.

COMMENTS

To be updated 1 Feb. 1990

References: EPA reviews of studies
Writer : g. maske

[*] - Acceptable Study. [#] = Supplemental Study

DYNAMAC
CORPORATION

XRD-498 EUP

TASK 1: REVIEW AND EVALUATION OF INDIVIDUAL STUDIES

TASK 2: ENVIRONMENTAL FATE ASSESSMENT

January 17, 1990

Initial Draft Report

Contract No. 68D90058

Submitted to:
Environmental Protection Agency
Arlington, VA 22202

Submitted by:
Dynamac Corporation
The Dynamac Building
11140 Rockville Pike
Rockville, MD 20852

XRD-498 EUP

Table of Contents

INTRODUCTION

XRD-498 is a selective experimental herbicide proposed for use to control broadleaf weeds in soybeans and field corn. At this time, it is for use only at an application site of a cooperator and in accordance with the terms and conditions of the experimental use permit. Single active ingredient formulations include 75% G. XRD-498 may be applied using preplant incorporation, preemergence, or postemergence treatment. Proposed application rates are 0.03-0.13 lb ai/A for preplant incorporation and preemergence treatments; postemergence rates on field corn are 0.015-0.062 lb ai/A, and postemergence rates on soybeans are 0.0078-0.015 lb ai/A. Application is by ground spray; sufficient agitation should be maintained during mixing and spraying to ensure a uniform spray mixture. When applied by preplant incorporation, XRD-498 should be incorporated into the top 2 to 3 inches of the final seedbed. Preemergence and postemergence applications are made by broadcast spraying. Livestock should not be allowed to graze in treated areas, and harvest-treated silage or grain should not be feed to meat or dairy animals.

DATA EVALUATION RECORD

STUDY 1

CHEM XRD-498 §161-1

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 41263229
Lehmann, R. and J. Miller. 1988. Aqueous hydrolysis of XRD-498. Laboratory Project ID GH-C 2092. Unpublished study prepared and submitted by Dow Chemical Company USA, Midland, MI.

DIRECT REVIEW TIME = 8

REVIEWED BY: C. Little TITLE: Staff Scientist

EDITED BY: T. Colvin-Snyder TITLE: Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

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

APPROVED BY: G. Maske
TITLE: Chemist
ORG: EFGWB/EFED/OPP
TEL: 557-9733

SIGNATURE:

CONCLUSIONS:

Degradation - Hydrolysis

1. This study can be used to fulfill data requirements.

2. XRD-498 did not hydrolyze in sterile aqueous pH 5, 7, and 9 buffered solutions incubated in the dark at 25 C for 66 days.

3. This study is acceptable and fulfills EPA Data Requirements for Registering Pesticides by providing information on the hydrolysis of XRD-498 in sterile aqueous solutions buffered to pH 5, 7, and 9.

4. No additional information on the hydrolysis of XRD-498 is required at this time.

METHODOLOGY:

[^{14}C]XRD-498 [N-2,6-difluorophenyl-5-methyl-1,2,4-triazole(1,5a)pyrimidine-2-sulfonamide] (radiochemical purity 100%, specific activity 10.9 mCi/mmol, Dow), dissolved in methanol, was added at 0.6 mg/L to sterile aqueous 0.01 M buffer solutions adjusted to pH 5 (potassium biphthalate), 7 (potassium phosphate monobasic), and 9 (boric acid, potassium chloride). The test solutions were incubated in sterile glass centrifuge tubes fitted with Teflon-backed caps in the dark at 25 ± 1 C. Samples of each test solution were removed for analysis at 0, 5, 10, 19, 38, 40, and 66 days posttreatment.

Duplicate aliquots of each sample were analyzed for total radioactivity using LSC. Additional aliquots were analyzed for XRD-498 and its degradates by HPLC using the solvent system water:acetic acid:DMOA (99:1:0.05) plus acetonitrile:acetic acid:DMOA (99:1:0.05) (Appendix B); fractions were collected and analyzed for radioactivity using LSC. To confirm the results obtained using HPLC, samples of each solution for day 66 were analyzed using TLC. Aliquots of the samples were mixed with 0.05 mL of 1 N hydrochloric acid to reduce the pH and 0.25 g of sodium chloride to increase the ionic strength. Samples were partitioned eight times with ethyl acetate. The ethyl acetate extracts were dehydrated for one hour with magnesium sulfate and evaporated to dryness under a nitrogen stream. The residues were dissolved in acetone and then cochromatographed with a XRD-498 reference standard using TLC on silica gel plates developed in ethyl acetate:acetic acid (95:5). Following development, the plates were air-dried and the reference standard was visualized under UV light. Radioactive areas on the plates were located using a RTLC scanner.

DATA SUMMARY:

[^{14}C]XRD-498 (radiochemical purity 100%), at 0.6 ppm, did not hydrolyze in sterile aqueous pH 5, 7, and 9 buffered solutions incubated in the dark at 25 ± 1 C for 66 days. [^{14}C]XRD-498, present in all solutions at ≥99%, was the sole compound identified in the buffer solutions at all sampling intervals (Table IV). Recoveries of total radioactivity from all solutions ranged from 101 to 110% of the applied (Table III).

COMMENTS:

1. Adsorption of [^{14}C]XRD-498 to glass was tested by rinsing test tubes and caps containing day 0 samples with 1 mL methanol and analyzing the rinses for radioactivity. Radioactivity was not detected in the rinses, indicating that [^{14}C]XRD-498 was not adsorbed to glass.

2. The study authors did not specify the site of the radiolabel in the [^{14}C]XRD-498 test substance.

3. TLC R_f values for XRD-498 in the 66-day samples (pH 5, 7, and 9) and the XRD-498 reference standard differed by 0.6 to 2.8% (Table V), confirming the identity of the radioactive areas on the TLC plates as XRD-498.

Fluometuron EFGWB Review

Page _____ is not included in this copy.

Pages 17 through 24 are not included in this copy.

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.

DATA EVALUATION RECORD

STUDY 2

CHEM XRD-498 §162-1

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 41263230
Laskowski, D., R. Lehmann, P. Goodwin, D. Stanley, B. French, and J. Miller. 1989. Aerobic soil degradation of XRD-498. Laboratory Project ID GH-C 2160. Unpublished study performed and submitted by Dow Chemical USA, Midland, MI.

DIRECT REVIEW TIME = 12

REVIEWED BY: E. Hirsh TITLE: Staff Scientist

EDITED BY: T. Colvin-Snyder TITLE: Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

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

APPROVED BY: G. Maske
TITLE: Chemist
ORG: EFGWB/EFED/OPP
TEL: 557-9733

SIGNATURE:

CONCLUSIONS:

Metabolism - Aerobic Soil

1. This study can be used to fulfill data requirements.

2. 5-Triazolopyrimidine-labeled [^{14}C]XRD-498 degraded with half-lives of 23-102 days in sandy loam, clay, silt loam, and loam soils that were incubated in the dark at 25 C and 75% of 1/3 bar moisture.

3. This study is acceptable and partially fulfills EPA Data Requirements for Registering Pesticides by providing information on the degradation of 5-triazolopyrimidine-labeled [^{14}C]XRD-498 in four soils.

4. No additional data on the aerobic degradation of 5-triazolopyrimidine-labeled [^{14}C]XRD-498 are required at this time. However, in

order to fulfill the aerobic soil data requirement, a study of the aerobic soil metabolism of phenyl-labeled [^{14}C]XRD-498 is required.

METHODOLOGY:

Silt loam, sandy loam, loam, and clay soils (Table 1) were sieved through a 10-mesh (2 mm) screen; 50 g portions were weighed into incubation flasks and treated with 0.2 ppm of 5-triazolopyrimidine-labeled [^{14}C]XRD-498 [N-2,6-difluorophenyl-5-methyl-1,2,4-triazole-(1,5a)pyrimidine-2-sulfonamide] (radiochemical purity ≥98%, specific activity 10.9 uCi/umole, Dow) dissolved in 0.005 M aqueous ammonium hydroxide. The treated soil samples were stirred and adjusted to 75% of 1/3 bar moisture. In order to trap volatiles, each flask containing a soil sample was sealed and connected, via a side arm, to a smaller flask containing 0.2 N sodium hydroxide trapping solution (Figure 1); the flasks containing the trapping solutions were connected to an oxygen manifold and maintained under slight positive pressure. The samples were incubated in the dark at 25 ± 1 C. Soil samples and trapping solutions were collected at 0, 7, 14, 28-34, 56-59, 100-101, 200, and 371-382 days posttreatment. Trapping solutions were also collected at 28-34 day intervals between 56 and 382 days posttreatment. The soil samples were stored frozen (1 to 4 C) prior to extraction.

The trapping solutions were assayed by LSC at the time of collection. The soil samples were extracted three times by vortexing with acetone:acetic acid:water (90:5:5) for 5 minutes. The acetone:acetic acid:water extracts were combined, assayed for total radioactivity by LSC, and stored frozen until analysis. The acetone:acetic acid:water extracts were concentrated by vacuum centrifugation and then analyzed by reverse phase HPLC with UV detection. Two solvent systems, water:acetonitrile:acetic acid (85:15:1 and 59:40:1), and water:acetic acid:DMOA (99:1:0.05) plus acetonitrile:acetic acid:DMOA (99:1:0.05), were used. Confirmation of HPLC results of the acetone:acetic acid:water extracts was accomplished by normal phase TLC on silica gel plates developed in ethylacetate:acetonitrile (50:50) and methanol:water (70:30). Analytical grade XRD-498 was co-chromatographed for identification. Radioactive spots were located and quantified using a radio scanner, and non-labeled standards were located using UV light.

In order to determine the amount of XRD-498 present in the fulvic acid plus humic acid and humin organic matter fractions, the extracted soils were further extracted with 0.5 N sodium hydroxide. The extracts were analyzed for total radioactivity by LSC and analyzed for specific compounds by reverse phase HPLC as previously described, following acidification to pH 3.0-3.5. The soil was air-dried and then analyzed for total radioactivity by LSC following combustion.

DATA SUMMARY:

5-Triazolopyrimidine-labeled [^{14}C]XRD-498 (radiochemical purity ≥98%), at 0.2 ppm, degraded with calculated first half-lives of 23, 60, 93, and 102 days in sandy loam, clay, silt loam, and loam soils, respectively, that were incubated in the dark at 25 ± 1 C and 75% of 1/3 bar moisture (Figures 16-19). Six unidentified degradates were each isolated at up to 3.4% of the applied (0.007 ppm) (Tables V and VI). At 371-382 days posttreatment, $^{14}CO_2$ comprised 34.5-53.3% of the applied radioactivity, residues in the fulvic acid plus humic acid soil organic matter fractions comprised 12.4-18.2%, and residues in the humin organic matter fraction comprised 10.9-20.1% (Tables III and V). Throughout the study, the material balances ranged from 80.4 to 104.4% (Table III).

COMMENTS:

1. Assuming first order degradation kinetics, the registrant calculated two half-lives for each soil: a half-life using data for all sampling intervals, and a first half-life using data for sampling intervals only up to first interval following the disappearance of 50% of the applied XRD-498. Data for all of the sampling intervals did not readily conform to first order kinetics (Figures 16-19). Since data used to calculate the first half-life conformed more closely to first order kinetics, these degradation half-lives were presented in the study.

2. The two HPLC units used to analyze soil extracts were identified as system #1 and system #6 in the text of the methods section of the original study. However, it is stated in Table VI that the data presented were obtained using HPLC system #2. It is not clear which HPLC system was used to obtain the data presented in Table VI.

3. A storage stability experiment indicated that XRD-498 was stable for up to 8 months in frozen acetone:acetic acid:water extracts (Table VII). Soil samples were extracted by "hand" by shaking with acetone:acetic acid:water for 1 hour. Then, after 6-8 months of frozen storage, the same soil samples were extracted by vortexing for 5 minutes using a robot (the extraction procedure used in the actual study).

4. The registrant attempted to characterize radioactivity in the sodium hydroxide extracts (fulvic/humic acid fractions); however, this radioactivity could not be characterized since XRD-498 was found to hydrolyze to a polar product in 0.5 N sodium hydroxide.

5. Although this study is acceptable, this study does not fulfill the aerobic soil metabolism data requirement because only the degradation of 5-triazolopyrimidine-labeled [^{14}C]XRD-498 was studied. A study of the degradation of phenyl-labeled [^{14}C]XRD-498 is required.

Fluroxypyr EFGWB review

Page _____ is not included in this copy.

Pages 28 through 51 are not included in this copy.

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.

DATA EVALUATION RECORD

STUDY 3

CHEM XRD-498 §162-1, §163-1

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 41290403
Lehmann, R., A. Laskowski, J. Miller, D. Stanley, and D. Fontaine. 1989. Effect of soil properties on the degradation and sorption of XRD-498. Laboratory Project ID GH-C 2243. Protocol No. 87062. Unpublished study performed and submitted by Dow Chemical USA, Midland, MI.

DIRECT REVIEW TIME = 30

REVIEWED BY: C. Little, E. Hirsh TITLE: Staff Scientists

EDITED BY: T. Colvin-Snyder TITLE: Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation
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SIGNATURE:

CONCLUSIONS:

Metabolism - Aerobic Soil

1. This portion of the study can be used to fulfill data requirements.

2. 5-Triazolopyrimidine-labeled [^{14}C]XRD-498 degraded with half-lives of 13-130 days in twenty-four soils ranging in texture from sandy loam to clay that were incubated in the dark at 25 C and 75% of 1/3 bar moisture.

3. This portion of the study is acceptable and partially fulfills EPA Data Requirements for Registering Pesticides by providing information on the degradation of 5-triazolopyrimidine-labeled [^{14}C]XRD-498 in twenty-four soils.

4. No additional data on the aerobic degradation of 5-triazolopyrimidine-labeled [^{14}C]XRD-498 are required at this time. However, in order to fulfill the aerobic soil data requirement, a study of the aerodic soil metabolism of phenyl-labeled [^{14}C]XRD-498 is required.

Mobility - Leaching and Adsorption/Desorption

1. This portion of the study cannot be used to fulfill data requirements.

2. XRD-498 was determined to be very mobile in twenty-three soils ranging in texture from sandy loam to clay. In general, it appeared that adsorption of XRD-498 increased with decreasing soil pH, increasing degradation half-life, and increasing soil organic matter content.

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

   this experiment was conducted using only one concentration of XRD-498 (accurate determination of Freundlich K_{ads} values requires data for at least four concentrations);

   desorption of XRD-498 was not addressed;

   the temperature at which the experiment was conducted was not specified; and

   the study authors failed to analyze the soils to confirm adsorption and to provide complete material balances.

4. Because this experiment was conducted using only one concentration of XRD-498, and desorption of XRD-498 was not addressed, the problems with this study cannot be resolved with the submission of additional data. A new study is required.

METHODOLOGY:

Metabolism - Aerobic Soil

Eight silt loam, four sandy loam, four clay, three silty clay loam, two clay loam, two loam, and one sandy clay loam soil (Table 1) were sieved through a 10-mesh (2 mm) screen; 50 g portions were weighed into incubation flasks and treated with approximately 0.2 ppm of 5-triazolopyrimidine-labeled [^{14}C]XRD-498 [N-2,6-difluorophenyl-5-methyl-1,2,4-triazole(1,5a)pyrimidine-2-sulfonamide] (radiochemical purity $\geq$98%, specific activity 10.9 uCi/umole, Dow) dissolved in 0.005 M aqueous ammonium hydroxide. The treated soil samples were stirred and adjusted to 75% of 1/3 bar moisture. In order to trap volatiles, each flask containing a soil sample was sealed and connected, via a side arm, to a smaller flask containing 0.2 N sodium

hydroxide trapping solution (Figure 1); the flasks containing the trapping solutions were connected to an oxygen manifold and maintained under slight positive pressure. The samples were incubated in the dark at 25 ± 1 C. Soil samples and trapping solutions were collected at intervals up to 200-382 days posttreatment. The soil samples were stored frozen (1 to 4 C) prior to extraction.

The trapping solutions were assayed by LSC at the time of collection. The soil samples were extracted three times by vortexing with acetone:acetic acid:water (90:5:5) for 5 minutes. The acetone:acetic acid:water extracts were combined, assayed for total radioactivity by LSC, and stored frozen until analysis. The acetone:acetic acid:water extracts were concentrated by vacuum centrifugation and then analyzed by reverse phase HPLC with UV detection. The solvent system water:acetic acid:DMOA (99:1:0.05) plus acetonitrile:acetic acid:DMOA (99:1:0.05) was used (Appendix B). Selected eluate fractions were further separated by HPLC using the same solvent system with a different gradient scheme. The extracted soils were air-dried and then analyzed for total radioactivity by LSC following combustion.

Four of the soils (M172, M176, M198, M203) were analyzed by a similar procedure as described in Study 2 (41263230).

Mobility - Leaching and Adsorption/Desorption

The soils described above (Table 1), with the exception of soil M192, were sieved (2-mm) and then treated with 0.01 M calcium chloride solutions (5 g soil:15 mL solution) containing 5-triazolopyrimidine ring-labeled [^{14}C]XRD-498 (radiochemical purity >99%, specific activity 10.9 uCi/umol, Dow) at 0.2 ug/mL. The test tubes containing the soil:solution slurries were then wrapped in aluminum foil and shaken for 24 hours at an unspecified temperature. Following equilibration, the soil:solution slurries were centrifuged for 10 minutes, and the supernatants were removed and analyzed for total radioactivity by LSC.

DATA SUMMARY:

Metabolism - Aerobic Soil

5-Triazolopyrimidine-labeled [^{14}C]XRD-498 (radiochemical purity ≥98%), at 0.2 ppm, degraded with half-lives of 13-130 days in twenty-four soils ranging in texture from sandy loam to clay (Table 1) incubated in the dark at 25 ± 1 C and 75% of 1/3 bar moisture (Figures 6-19). Six unidentified degradates were isolated, each at up to 4.9% of the applied (0.01 ppm) (refer to comment 1). At 200-382 days posttreatment, $^{14}CO_2$ comprised 28.6-81.2% of the applied radioactivity, and unextractables comprised 18.1-50.9% (refer to comment 1). Throughout the study, the material balances ranged from 66.7 to 129.2%.

Mobility - Leaching and Adsorption/Desorption

Based on batch equilibrium experiments, [^{14}C]XRD-498 (radiochemical purity >99%), at 0.2 ug/mL, was determined to be very mobile in twenty-three soils ranging in texture from sandy loam to clay (Table 1) equilibrated for 24 hours at an unspecified temperature; adsorption coefficients (K_d, concentration in soil/concentration in solution) were 0.05-2.42, and K_{oc} (K_d/% soil organic matter) values were 5-182 (Table XIII). In general, it appeared that adsorption increased with decreasing pH (Figures 16 and 17), increasing degradation half-life (Figure 18), and increasing soil organic matter content.

COMMENTS:

Metabolism - Aerobic Soil

1. Four of the twenty-four soils discussed in the registrant's report (M172, M176, M198, M203) were discussed previously in Study 2 (41263230). Although data for the degradation rate of XRD-498 in all twenty-four soils were presented in the registrant's report, data for degradates, unextractable radioactivity, and material balances were not provided for the four soils discussed in Study 2. For completeness, these data, obtained from Study 2, were also presented in the data summary section of this review.

   Data for XRD-498 degradates were obtained from Appendix E of this study and Tables V and VI of Study 2. Data for unextractable radioactivity and material balances were obtained from Tables II-IX of this study and Tables III and V of Study 2.

2. Although this study is acceptable, this study does not completely fulfill the aerobic soil metabolism data requirement because only the degradation of 5-triazolopyrimidine-labeled [^{14}C]XRD-498 was studied. A study of the degradation of phenyl-labeled [^{14}C]XRD-498 is required.

Mobility - Leaching and Adsorption/Desorption

1. This experiment was conducted using only one concentration of XRD-498. Since data for at least four pesticide concentrations are required to accurately determine Freundlich K_{ads} values, these values could not be calculated.

2. Desorption of XRD-498 was not addressed.

3. The temperature at which the study was conducted was not specified.

4. The study authors failed to analyze the soils to confirm adsorption and to provide complete material balances.

5. Adsorption appeared to be related to soil pH, organic matter content, and degradation half-lives; however, no statistical correlations were performed. The conclusions drawn by the study authors would be considered more valid if these conclusions had been supported by appropriate statistical analyses.

Aluminum EFGWB Review

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Pages 57 through 97 are not included in this copy.

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_____ Identity of product inert ingredients.

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DATA EVALUATION RECORD

STUDY 4

CHEM XRD-498 §163-1

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 41263231
Goodwin, P., R. Lehmann, and J. Miller. 1989. Soil adsorption/desorption of ^{14}C-XRD-498. Laboratory Project ID GH-C 2159. Unpublished study performed and submitted by Dow Chemical Company USA, Midland, MI.

DIRECT REVIEW TIME = 30

REVIEWED BY: C. Little TITLE: Staff Scientist

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SIGNATURE:

CONCLUSIONS:

1. This study cannot be used to fulfill data requirements.

2. XRD-498 was very mobile in silt loam, sandy loam, clay, and loam soils.

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

   the experimental design of the desorption experiments was inadequate to accurately assess desorption of XRD-498 and XRD-498 residues - it cannot be determined what portion of the XRD-498 or XRD-498 residues in solution following the "desorption" step was actually desorbed from the soil.

4. Because the experimental design of the desorption experiments was inadequate to accurately assess desorption of XRD-498, and the data from the adsorption phase of an adsorption/desorption experiment are necessary to adequately assess desorption, the problems with this study cannot be resolved with the submission of additional data. A new study is required.

METHODOLOGY:

Based on preliminary tests, an equilibration time of 24 hours was selected.

5-Triazolopyrimidine ring-labeled [^{14}C]XRD-498 [N-2,6-difluorophenyl-5-methyl-1,2,4-triazole(1,5a)pyrimidine-2-sulfonamide] (radiochemical purity >99%, specific activity 10.9 uCi/umol, Dow), dissolved in acetone and dimethyl formamide, was added to 0.01 M calcium chloride solutions at 0.1, 0.6, 1.2, and 3.5 ug/mL. The treated calcium chloride solutions were mixed with sieved (2-mm) silt loam, sandy loam, clay, and loam soils (Table 1) (5 g soil:15 mL solution). The test tubes containing the soil:solution slurries were then wrapped in aluminum foil and shaken for 24 hours at 24-26 C. Following equilibration, the soil:solution slurries were centrifuged for 15 minutes, and the supernatants were removed and analyzed for total radioactivity by LSC. Then, the soils were extracted three times with acidified acetone (5% acetic acid):water (15:1), and the combined extracts were analyzed by LSC. The extracted soil was then air-dried for 24 hours and analyzed by LSC following combustion. In order to determine if degradation of XRD-498 occurred during the study, aliquots of selected calcium chloride solutions and acetone extracts were analyzed for XRD-498 and possible degradates by reverse-phase HPLC with radioactivity detection using the solvent systems 1% acetic acid in water:acetonitrile (84:15) followed by 1% acetic acid in water:acetonitrile (59:40).

In order to study desorption, samples of the same soils described above were treated with [^{14}C]XRD-498 dissolved in a water:0.005 M ammonium hydroxide solution at 0.2, 1.2, 3.5, and 10.4 ug/g. The soils were wrapped in aluminum foil and incubated for 24 hours. The treated soils were then mixed with 0.01 M calcium chloride solution (5 g soil:15 mL solution), and the soil:solution slurries were shaken for 24 hours. Following equilibration, the soil:solution slurries were centrifuged for 15 minutes, and the supernatants were removed and analyzed for total radioactivity by LSC. Then, the soils were extracted three times with acetone:acetic acid:water (90:5:5), and the combined extracts were analyzed by LSC. Aliquots of the calcium chloride solutions and acetone extracts were analyzed by reverse-phase HPLC as described above.

An additional experiment was conducted in which the desorption of aged XRD-498 residues in soil was studied. Samples of the same soils described above were treated with [^{14}C]XRD-498 at 0.2 ppm and incu-

bated in the dark at 75% of 1/3 bar moisture for up to 59 days at 25 ± 2 C. The soils were sampled at 0, 7, 14, 28 or 34, and 56 or 59 days posttreatment. At each sampling interval, a portion of each treated soil was extracted three times with acetone:acetic acid:water (90:5:5), and the combined extracts were analyzed by LSC and HPLC as described in the aerobic metabolism experiment (Study 2, 41263230). Additional portions of each treated soil at each sampling interval were mixed with 0.01 M calcium chloride solution. The soil:solution ratio was 5 g: 15 mL for the silt loam, clay, and loam soils; and was 5 g:5 mL for the sandy loam soil. The soil:solution slurries were shaken for 24 hours. Following equilibration, the soil:solution slurries were centrifuged for 15 minutes, and the supernatants were removed and analyzed for total radioactivity by LSC and by reverse-phase HPLC as described for the adsorption experiment. The amount of XRD-498 remaining on each soil after equilibration was estimated by subtracting the amount of XRD-498 in the calcium chloride solution following equilibration from the amount of XRD-498 in the acetone extracts.

DATA SUMMARY:

Based on batch equilibrium experiments, [^{14}C]XRD-498 (radiochemical purity >99%), at 0.1, 0.6, 1.2, and 3.5 ug/mL, was very mobile in silt loam, sandy loam, clay, and loam soils equilibrated for 24 hours at 24-26 C; Freundlich K_{ads} values were 0.05-0.45, and K_{oc} values were 7-20 (Table X). In the same soils treated with [^{14}C]XRD-498 at 0.2, 1.2, 3.5, and 10.4 ug/g, incubated for 24 hours, and then equilibrated with 0.01 M calcium chloride solution, Freundlich K_{des} values were determined to be 0.15-0.57, and corresponding K_{oc} values were 14-25. In the same soils treated with [^{14}C]XRD-498 at 0.2 ug/g, incubated for intervals up to 59 days, and then equilibrated with 0.01 M calcium chloride solution, desorption coefficients (ug/g estimated soil concentration /ug/mL in solution after equilibration) ranged from 0.07 to 3.19, and corresponding K_{oc} values ranged from 12 to 143 (Table IX).

COMMENTS:

1. The experimental design of the desorption experiments was inadequate to accurately assess desorption of XRD-498 and XRD-498 residues. In these experiments, the soils were treated with XRD-498 and then equilibrated with pesticide-free calcium chloride solution. Since XRD-498 was added directly to the soil instead of being allowed to adsorb to the soil by equilibration with a pesticide-treated solution, some of the XRD-498 and its residues present in the soils may not have been adsorbed to the soil. Therefore, it cannot be determined what portion of the XRD-498 or XRD-498 residues in solution following the "desorption" step was actually desorbed from the soil.

2. The experiment studying desorption of aged XRD-498 residues was conducted using only one concentration of the pesticide. Therefore, Freundlich K_{des} values could not be not calculated.

3. [^{14}C]XRD-498 was dissolved in 1 mL each of acetone and dimethyl formamide prior to being added to the calcium chloride solutions. The final concentrations of acetone and dimethyl formamide in the calcium chloride solutions were not reported; this information is important since the presence of these solvents could affect the adsorption of XRD-498.

4. Two samples were not used to calculate Freundlich K_{ads} values because total recoveries of radioactivity in these samples were too high (112.8 and 119.5%) (Table IV). The study authors attributed these high recovery values to sample contamination.

5. For the experiments studying adsorption and desorption of unaged XRD-498, in addition to calculating Freundlich K_{ads} and K_{des} values, the study authors calculated adsorption and desorption coefficients (concentration in soil/concentration in solution) for each sample.

6. Although the study authors stated that the soil:solution slurries were shaken for periods of 5 minutes to 120 hours in the desorption study of aged XRD-498 residues, only data for soil:solution slurries shaken for 24 hours were reported and used in determining desorption coefficients.

Fluometuron EFGWB Review

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_____ Identity of product inert ingredients.

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DATA EVALUATION RECORD

STUDY 5

CHEM XRD-498 165-1

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 41263232
Hamburg, A., J.H. Miller, T.S. Lardie, and W.S. Baldwin. 1989. ^{14}C-XRD-498: Confined accumulation study on rotational crops planted at 30 and 120 days after soil treatment. Project ID GH-C 2170. Unpublished study performed and submitted by Dow Chemical Co., Midland, MI.

DIRECT REVIEW TIME = 24

REVIEWED BY: E. Hirsh TITLE: Staff Scientist

EDITED BY: K. Patten TITLE: Task Leader

APPROVED BY: W. Spangler TITLE: Project Manager

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APPROVED BY: G. Maske
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ORG: EFGWB/EFED/OPP
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SIGNATURE:

CONCLUSIONS:

Confined Accumulation - Rotational Crops

1. This study cannot be used to fulfill data requirements.

2. Most samples were analyzed only for total [^{14}C]XRD-498 residues were found to be present at low levels (<0.01 ppm XRD-498 equivalents) in lettuce, carrot roots and tops, and soybean beans planted 30-52 or 120 days after plots of sandy loam soil were treated with [^{14}C]XRD-498 at 134 g ai/ha (0.12 lb ai/A). Total [^{14}C]XRD-498 residues were 0.010-0.047 ppm in green bean plants, wheat grain, and wheat straw/chaff planted with the 30-day rotation, and in soybean plant trash and wheat straw/chaff planted with the 120-day rotation.

In the 0- to 15-cm field soil depth, total [^{14}C]residues were 0.066 ppm at 30 days posttreatment, 0.019 ppm at 105 days, and 0.014 ppm at 208 days. Extractable [^{14}C]residues with retention times comparable to XRD-498 accounted for 0.035 and 0.003 ppm at 30 and 118 days posttreatment.

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

   storage stability data were not provided, although the soil and plant samples were stored frozen for up to 118 and 347 days, respectively;

   all [^{14}C]residues comprising >0.01 ppm were not identified (0.0209 ppm were present in the green bean plant from day 30, and 0.022 ppm in soybean trash and 0.0288 ppm in wheat straw/-chaff from day 120); and

   the stated application rate of 0.12 lb ai/A was not confirmed, soil samples were not taken until 30 days posttreatment.

4. In order for this study to fulfill the accumulation in confined rotational crops data requirement, the registrant must submit freezer storage stability data for the soil and all plant substrates, must characterize [^{14}C]residues in the green bean whole plants and soybean trash, and must provide data to confirm the stated application rate.

METHODOLOGY:

Two field plots (Plot A - 0.61 x 5.5 m; Plot B - 0.61 x 3.7 m) of sandy loam soil (68% sand, 20% silt, 12% clay, 2.4% organic matter, pH 7.8, CEC 9.4 meq/100 g) located in Midland, Michigan, were divided into 0.2-m^2 subsections. Each subsection was treated with a 10-mL aliquot of an acetone solution containing a mixture of 5-triazolo-pyrimidine-[^{14}C]XRD-498 [N-2,6-difluorophenyl-5-methyl-1,2,4-triazole(1,5a)pyrimidine-2-sulfonamide] (radiochemical purity 99.1%, specific activity 7.24 Ci/Mol, Dow Chemical) and unlabeled XRD-498 (purity 96.6%) on May 5, 1987. The specific activity of the acetone solution was 4.04 Ci/Mol; the application rate was 134 g ai/ha (0.12 lb ai/A).

30/52-day rotation: At 30 days posttreatment, Plot A was planted with lettuce, turnips, green beans, and wheat. At 52 days posttreatment, spinach and carrots were planted into Plot A between the lettuce and turnips, because the lettuce and turnips were exhibiting signs of phytotoxicity. The spinach and turnips died before maturity. The lettuce, green beans, wheat, and carrots were harvested at maturity: lettuce at 77 and 97 days postplanting (leaves, and leaves and stems, respectively); carrots at 108 days (roots and tops); green beans at 63, 74, and 88 days (bean/pods and whole plants); and wheat at 104 days (grain and straw/chaff). Plant samples were stored

frozen for up to 66 days until analysis. Four soil cores (0- to 15-cm depth, 3.8-cm diameter) were collected after the soil was spaded to a depth of 15- to 20-cm and immediately prior to the 30-day planting. The soil cores were combined at the test site, homogenized at the laboratory, and stored frozen for up to 118 days until analysis.

120-day rotation: From Plot B, soil cores (0- to 38-cm depth, 2.5-cm diameter) were collected at approximately 105 days posttreatment. The cores were divided into 7.5-cm segments, and corresponding segments were combined and homogenized. At 118 days posttreatment, the top 23 cm of soil from Plot B was removed in blocks from the field and transferred to the greenhouse. The soils were mixed thoroughly, then used to fill nine 0.02-m^2 fiber packs; after filling, 100-g soil samples were collected from each fiber pack, combined, and homogenized. At 120 days posttreatment, wheat, lettuce, carrots, and soybeans were planted into the fiber packs. In addition, four fiber packs were filled with untreated soil for growth of identical control crops. Plants were top- or bottom-watered as necessary. Lettuce was harvested at 34, 62, and 79 days postplanting; carrots (roots and tops) at 84 days, soybeans (beans and trash) at 105 days, and wheat (grain and straw/chaff) at 112 days. After all crops were harvested from the 120-day rotation (232 days posttreatment), a soil core (0- to 15-cm depth, 3.8-cm diameter) was collected from each fiber pack. The cores were composited and homogenized. Soil samples were stored frozen for up to 30 days until analysis; plant samples were stored frozen for up to 100 days (347 days for straw/chaff) until analysis.

Plant samples were rinsed with water to remove any soil particles. Lettuce, green beans, carrot tops and roots were weighed, diced into small pieces, frozen at -10 to -20 C, and lyophilized to a constant weight. The three green bean bean/pod harvests were combined and analyzed as one sample. The water condensate was collected from the lyophilization unit and assayed for total radioactivity by LSC. Whole green bean plants, wheat plants (grain and straw/chaff), and soybean plants (stalks, beans, and pods) were weighed prior to being air-dried for 11 to 20 days in a greenhouse. All freeze- or air-dried plant samples were weighed, milled to a uniform powder, and stored at -10 to -20 C for 10 to 22 days prior to analysis for total radioactivity by LSC following combustion.

The wheat straw/chaff from the 30-day rotation was analyzed further because it contained the highest concentration of [^{14}C]residues of the various plant tissues; the plant material was analyzed 347 days after harvest, during which time the sample had been stored frozen. The analysis procedure is detailed in Figure 4. Duplicate 5-g milled straw/chaff samples were extracted sequentially twice with water and once with acetone:water (1:1). The extracts were combined, concentrated, and extracted twice with acetone. The acetone extracts were combined, concentrated, centrifuged to remove particulates, and analyzed by reversed-phase HPLC using an acetonitrile:water mobile phase and UV and radioactivity detection. The extracts were cochromatographed with reference standards for comparison. The acetone-

extracted "neutral" residue was mixed with water, centrifuged to remove particulates, and analyzed by HPLC. All particulates were analyzed for total radioactivity by LSC following combustion. The extracted straw/chaff was further extracted with sodium hydroxide; the extracts were analyzed for total radioactivity by direct LSC and the solids by LSC following combustion. An additional 10-g sample of straw/chaff was analyzed as described except that the initial set of extractions (water and acetone:water) was replaced with one acetonitrile:water (3:1) extraction and three acetone:water (1:1) extractions.

Portions of each soil sample were analyzed for total radioactivity by LSC following combustion. Soil samples collected from Plot A at 30 days posttreatment and Plot B at 118 days posttreatment were extracted using various schemes detailed in Tables VI and VII (soil samples collected after the 120-day rotation harvest were not further analyzed). In general, the soils were extracted by shaking with acetonitrile, water, or acetone, followed by extraction with acidic solvents. Aliquots of the extracts collected at each stage of the extraction procedure were analyzed by LSC. Also, the concentrated extracts were analyzed by reversed-phase HPLC and the extracted soil was analyzed for total radioactivity by LSC following combustion.

Minimum detectable amounts (MDA) and minimum quantifiable amounts (MQA) were calculated for XRD-498 equivalents in each plant sample (values are presented in the data tables).

DATA SUMMARY:

[^{14}C]XRD-498 residues were <0.006 ppm (XRD-498 equivalents) in mature lettuce, carrot roots and tops, green bean bean/pods (30 days only) and soybean beans collected from crops planted 30-52 and 120 days after two plots of sandy loam soil located in Midland, Michigan, were treated with 5-triazolopyrimidine-[^{14}C]XRD-498 (radiochemical purity 99.1%) at 134 g ai/ha (0.12 lb ai/A) (Tables IV and V). [^{14}C]XRD-498 residues were 0.0209 ppm in green bean whole plants and 0.0109 and 0.047 ppm in wheat grain and straw/chaff, respectively, in crops planted 30 days after treatment (Table IV). [^{14}C]XRD-498 residues were 0.022 ppm in soybean trash and <0.01 and 0.0288 ppm in wheat grain and straw/chaff, respectively, in crops planted 120 days after treatment (Table V). Accumulation of [^{14}C]residues in the crops decreased by approximately 50% between the 30/52- and 120-day rotations. Plants grown in untreated soil contained nondetectable or nonquantifiable concentrations (<0.0003 to 0.01 ppm XRD-498 equivalents) of [^{14}C]residues.

[^{14}C]Residues in the wheat straw/chaff sample from the 30-day rotation (0.047 ppm XRD-498 equivalents by LSC) were analyzed further: 0.013-0.017 ppm were recovered in the neutral solvent extracts, 0.011-0.018 ppm in the basic extracts, and 0.011-0.014 ppm in the unextractable fraction (Table XI). The neutral extracts contained

three major [^{14}C]compounds; two of the compounds were present at <0.001 ppm, the third was present at 0.002 ppm (Figures 11 and 12). Retention times for two components were similar to XRD-498 and its hydroxy metabolite; however, the compounds could not be reliably identified because of the low concentrations. The basic extracts were not analyzed further.

In the 0- to 15-cm field soil depth, total [^{14}C]residues were 0.066 and 0.019 ppm at 30 and 105 days posttreatment, respectively (Tables VIII, IX, and X). At 118 days posttreatment, following the mixing and removal of the surface 23-cm of treated field soil to greenhouse fiber-packs, total [^{14}C]residues were 0.018 ppm in soil subsamples (Tables VIII and IX). At 208 days posttreatment, total [^{14}C]residues were 0.014 ppm in the greenhouse soil (Table VIII). Total extractable [^{14}C]residues in the soil decreased from 0.051 ppm at 30 days to 0.004 ppm at 118 days; extractable [^{14}C]residues with retention times comparable to XRD-498 accounted for 53% (0.035 ppm) and 17% (0.003 ppm) of the recovered radioactivity at these intervals (Table IX). Unextractable [^{14}C]residues accounted for 18% of the recovered at 30 days posttreatment and 80% at 118 days.

For the 30/52-day rotation, precipitation totaled 1.9 inches between 0 and 30 days, 1.5 inches between 30 and 52 days, and approximately 113-17 inches between 52 days and the harvest of each crop. Air temperature ranged from 41 to 98 F during the study; soil temperature (4 inch) ranged from 65 to 91 F.

For the 120-day rotation, precipitation totaled 16.3 inches prior to the transfer to the greenhouse on day 118. In the greenhouse, relative humidity was 30-40% winter, 40-70% spring/fall, and the temperature ranged from 17-29 C. Natural sunlight was supplemented with artificial light (metal halide) for a 14 hour:10 hour light:dark photoperiod.

COMMENTS:

1. Storage stability data were not provided for XRD-498 in either the soil or plant substrates. The soil samples were stored frozen for up to 118 days and the plant samples for up to 347 days before analysis.

2. Only total [^{14}C]Residues were analyzed in most samples. Those samples comprising >0.01 ppm parent and/or degradates were not characterized.

3. The registrant failed to confirm the theoretical 0.12 lb ai/A application of XRD-498 with an immediate posttreatment soil sample. The first soil sample was not collected until 30 days posttreatment, at which time the concentration of XRD-498 in the 0- to 15-cm depth was 0.035 ppm; an application of 0.12 lb ai/A should result in an initial concentration of 0.06 ppm of XRD-498 in the upper 6 inches (15 cm) of soil (assuming that 1 acre of soil, 6 inches deep, weighs

2 million pounds). The total [^{14}C]residue concentration recovered from the soil at 30 days, 0.066 ppm, is close to the predicted concentration. It is possible (although not definite) that the application was correct because the half-life of XRD-498 reported in the aerobic soil metabolism study (Study 2, 41263230) was 17-48 days in sandy loam soil; the registrant should submit additional information to confirm that degradation of XRD-498 in the stock solution before use did not occur.

4. Extractable [^{14}C]residues in the soil and wheat straw/chaff were analyzed only by HPLC and identified only by comparison to reference standards.

   HPLC analysis of soil extracts from extraction scheme E identified XRD-498 (Figure 5). Analysis of extracted samples using schemes F and G (Figures 6 and 7) showed differing retention times for the major component than did extraction scheme E. The study authors assumed, but did not prove, that either the acidity of the concentrated extracts or the complex nature of the concentrated extracts altered the retention time of XRD-498.

5. Immature plant tissue was not analyzed. Although 2-3 harvests of lettuce and green bean beans were performed, the early samples were not considered harvests of immature crops.

6. Although soil samples collected from Plot B at 118 days posttreatment were extracted using all methods, only schemes F and G were included in Table VIII.

7. The field plots had been left fallow and were not treated with pesticides in the two years prior to the XRD-498 study. The field plots were treated with 168 kg N/ha on May 1, 4 days prior to the XRD-498 treatment.

8. The position of the radiolabel of the [^{14}C]XRD-498 test substance was not specified. The structure of XRD-498 is complex, and, therefore, data for accumulation of XRD-498 residues labeled in both the 5-triazolopyrimidine and phenyl positions may be required if data from the soil metabolism studies indicate that degradates of concern are formed by cleavage of XRD-498 between these two moieties.

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