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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, DC 20460

OCT 13 1988

OFFICE OF
PESTICIDES AND
TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: Ignite - EFGWB Science Chapter

FROM: Padma R. Datta, Ph.D., Chemist P.R. Datta
Environmental Fate & Ground Water Branch
Environmental Fate & Effects Division (TS-769C)

TO: Richard Mountfort, PM #23
Fungicide & Herbicide Branch
Registration Division (TS-767C)

and

Amy S. Rispin, Chief
Science Analysis and Coordination Staff
Environmental Fate & Effects Division (TS-769C)

THRU: Henry Jacoby, Acting Chief
Environmental Fate & Ground Water Branch
Environmental Fate & Effects Division (TS-769C)

THRU: Emil Regelman, Supervisory Chemist
Review Section #2
Environmental Fate & Ground Water Branch
Environmental Fate & Effects Division (TS-769C)

Attached is the Environmental Fate & Ground Water Branch (EFGWB) Science Chapter for glufosinate ammonium (Ignite). It includes Tasks I and II, a Table A, an executive summary and recommendation.

EFGWB brings to the attention of the Science Analysis and Coordination Staff (SACS) of Environmental Fate & Effects Division (EFED) the following important issues:

1. With the exception of the hydrolysis study (§161-1), all required studies submitted either failed to satisfy or to partially satisfy the environmental fate data requirements. Ignite (glufosinate ammonium) does not hydrolyze in sterile aqueous buffer solutions at pH 5, 7, and 9.

2. Although the aerobic aquatic metabolism study (§162-4) submitted by the registrant is an acceptable study, it is not required to fulfill environmental fate data requirements for the present use pattern. Aquatic use is not on the submitted labels.

TABLE A

GENERIC DATA REQUIREMENTS FOR IGNITE

Data Requirement	Test Substance[1]	Use Pattern[2]	Does EPA Have satisfactory Data? (Yes, No, Partially)	Bibliographic Citation MRID #	Must Additional Data be Submitted?	Time Frame For Submission[3]
§158.290 Environmental Fate						
DEGRADATION STUDIES-LAB:						
161-1 - Hydrolysis	TGAI or PAIRA	A,B,E,F,H	Yes	40345656	No	
Photodegradation						
161-2 - In Water	TGAI or PAIRA	A,B	No	_____	Yes[4]	
161-3 - On Soil	TGAI or PAIRA	A	No	_____	Yes[4]	
161-4 - In Air	TGAI or PAIRA	A	No	_____	No	
METABOLISM STUDIES-LAB:						
162-1 - Aerobic Soil	TGAI or PAIRA	A,B,E,,H	Partially	40501018 40345659A/B/C/D	Yes[5]	
162-2 - Anaerobic Soil	TGAI or PAIRA	A	No	_____	Yes[4]	
162-3 - Anaerobic Aquatic	TGAI or PAIRA	N/A	No	_____	No	
162-4 - Aerobic Aquatic	TGAI or PAIRA	N/A	Yes	40345660	No	
MOBILITY STUDIES:						
163-1 - Leaching and Adsorption/Desorption	TGAI or PAIRA	A,B,E,F,H	Partially	40345661/62	Yes[6]	
163-2 - Volatility (Lab)	TEP	A,E,F	No	_____	Yes[7]	
163-3 - Volatility (Field)	TEP	A,E,F	No	_____	Reserved[8]	

GENERIC DATA REQUIREMENTS FOR IGNITE

Data Requirement	Test Substance[1]	Use Pattern[2]	Does EPA Have satisfactory Data? (Yes,No,Partially)	Bibliographic Citation MRID #	Must Additional Data be Submitted?	Time Frame for Submission[3]
158.290 Environmental Fate (continued)						
DISSIPATION STUDIES-FIELD:						
164-1 - Soil	TEP	A,B,H	No		Yes[4]	
164-2 - Aquatic (Sediment)	TEP	N/A	No		No	
164-3 - Forestry	TEP	N/A	No		No	
164-4 - Combination and Tank Mixes	TEP	N/A	No		No[9]	
164-5 - Soil, Long-term	TEP	A	No		Reserved[10]	
ACCUMULATION STUDIES:						
165-1 - Rotational Crops (Confined)	PAIRA	A	No		Yes[4]	
165-2 - Rotational Crops (Field)	TEP	A	No		Reserved[11]	
165-3 - Irrigated Crops	TEP	N/A	No		No	
165-4 - In Fish	TGAI or PAIRA	A,B	No		Yes[4]	
165-5 - In Aquatic Nontarget Organisms	TEP	A,B	No		No	
158.440 Spray Drift						
201-1 - Droplet Size Spectrum	TEP	A,B	No		No	
201-1 - Drift Field Evaluation	TEP	A,B	No		No	

TABLE A

GENERIC DATA REQUIREMENTS FOR IGNITE

FOOTNOTES:

1/ Composition: TGAI = Technical grade of the active ingredient; PAIRA = Pure active ingredient, radiolabelled; TEP = Typical end-use product.

2/ The use patterns are coded as follows: A = Terrestrial, Food Crop; B = Terrestrial, Non-Food; C = Aquatic, Food Crop; D = Aquatic, Non-Food; E = Greenhouse, Food Crop; F = Greenhouse, Non-Food; G = Forestry; H = Domestic Outdoor; I = Indoor.

3/ Data must be submitted within the indicated timeframes, which begin on the date of the Guidance Document (see front cover for this date).

4/ MRID # of the reviewed study does not appear in the Table A because the study is found to be unacceptable. If the registrant can provide a satisfactory response to the comments in the individual DER of the study then, at the discretion of EFGWB the data may be acceptable after reevaluation. Otherwise, a new study must be conducted in accordance with Subdivision N Guidelines.

5/ An additional study is needed to establish the pattern of formation and decline of 2-methylphosphinico-acetic acid.

6/ An additional study is needed to establish the mobility of 2-methylphosphinico-acetic acid in one soil.

7/ Registrant may request a waiver of the laboratory volatility data requirement by showing that Ignite has a very low vapor pressure (eg, $<1 \times 10^{-6}$) or is non-toxic.

8/ Depending on the results of the laboratory volatility studies (163-2).

9/ Currently not being imposed for this product pursuant to PR Notice 82-1, 1/12/82.

10/ All data are required if the results from the aerobic soil metabolism (162-1) and/or soil field dissipation studies (164-1) show that >50% Ignite remains in the soil prior to the recommended subsequent application.

11/ Depending on the results of the accumulation studies in confined rotational crops (165-1).

DYNAMAC
CORPORATION

GLUFOSINATE AMMONIUM

Task 1: Review and Evaluation of Individual Studies

Task 2: Environmental Fate Assessment

September 8, 1988

Final Report

Contract No. 68-02-4250

Submitted to:
Environmental Protection Agency
Arlington, VA 22202

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

5

GLUFOSINATE AMMONIUM

Table of Contents

INTRODUCTION

Glufosinate ammonium is a nonselective foliage-applied herbicide developed to control a broad spectrum of emerged annual and perennial grass and broadleaf weeds on field and vegetable crops, orchards, vineyards, terrestrial nonfood sites (including dry ditches and canals, and ditch banks), domestic outdoor sites, and greenhouses. It will also control or suppress certain woody and herbaceous plant species. Glufosinate ammonium is primarily a contact herbicide with limited systemic activity; plants that have not emerged will not be controlled and there is reported to be no residual activity. Glufosinate ammonium is formulated as a 1.67 lb/gallon (16.22%) aqueous soluble, and it may be tank-mixed with numerous other pesticides.

Application rates vary with the type and maturity of the weeds being controlled. On terrestrial food crop sites other than minimum tillage systems, the maximum application is 1.46lb a.i./A (7.0 pints/A), with no more than 4.51lb a.i./A (21.6 pints/A) applied to any site in a given year. On minimum tillage systems, no more than 1.50lb a.i./A (7.2 pints/A/year) should be applied. On terrestrial nonfood sites, the maximum application is 1.46lbai/A (7.0 pints/A), with no more than 5.85 lb a.i./A (28 pints/A) applied to any site in a given year. The label states that treated soybean or corn forage, or orchard cover crops should not be grazed; that glufosinate ammonium should not be applied through irrigation systems or by means of aerial equipment; that it should not be applied within 14 days of nut, apple, or grape harvest; and that treated areas should not be replanted with cereal grains for at least 120 days following the application of glufosinate ammonium.

DATA EVALUATION RECORD

STUDY 1

CHEM 128850 Glufosinate ammonium §161-1

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40345656
Goerlitz, G., C. Kloeckner, and U. Eyrich. 1986. Abiotic hydrolysis as a function of pH and amendment, and separation of potential hydrolysis products of HOE 039866 from the active ingredient by HPLC. Project Nos. (B)277/85 and (B)110/86. Prepared by Hoechst AG, Frankfurt, Federal Republic of Germany, and submitted by Hoechst Celanese Corporation, Somerville, NJ.

DIRECT REVIEW TIME = 10

REVIEWED BY: W. Higgins TITLE: Staff Scientist

EDITED BY: K. Patten K. Patten TITLE: Task Leader

APPROVED BY: W. Spangler W. Spangler TITLE: Project Manager

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

APPROVED BY: P. Datta
TITLE: Chemist
ORG: EAB/HED/OPP
TEL: 557-9733

SIGNATURE: PRDatta 9/22/88

CONCLUSIONS:

Degradation - Hydrolysis

This study is acceptable and fulfills EPA Data Requirements for Registering Pesticides because glufosinate ammonium does not hydrolyze in sterile aqueous solutions buffered to pH 5, 7, and 9.

SUMMARY OF DATA BY REVIEWER:

Glufosinate did not hydrolyze in sterile, buffered, aqueous solutions (pH 5, 7, and 9) that were treated with glufosinate ammonium (purity 99.5%) at 232-236 ppm and incubated in the dark at 25 ± 0.1°C for 30 days. The hydrolytic half-life of glufosinate in solutions of pH 5, 7, and 9 was estimated by the registrant to be >300 days. During the study, the material balances ranged from 97.1 to 103.0% of the applied.

DISCUSSION:

The registrant calculated the half-lives of glufosinate in the pH 5, 7, and 9 solutions using linear regression model, but had no confidence in the resulting values. The calculated half-lives were 6.08 years (r^2 = 0.1206) for the pH 5 solution, 44.83 years (r^2 = 0.0066) for the pH 7 solution, and 2.14 years (r^2 = 0.8750) for the pH 9 solution.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Glufosinate ammonium (Hoe 039866, purity 99.5%, Hoechst AG) was added at 232-236 ppm to sterile (boiled), buffered, aqueous solutions adjusted to pH 5 (citric acid plus sodium hydroxide), 7 (potassium dihydrogen-phosphate plus sodium hydroxide, and 9 (potassium chloride, boric acid, and sodium hydroxide). The solutions were incubated in the dark at 25 ± 0.1°C. Duplicate aliquots of the solutions were sampled at intervals between 0 and 30 days posttreatment.

The samples were analyzed by HPLC with UV absorption detection. The HPLC detection limit was ≈10 ppm (≈4% of the initial concentration).

RIN 5218-93

EFGWB Reviews of Glufosinate (128850)

Page_____ is not included in this copy.

Pages 12 through 13 are not included.

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.

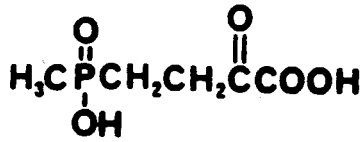
____ Information about a pending registration action.

✓ FIFRA registration data.

____ The document is a duplicate of page(s) _____.

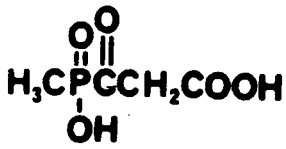
____ The document is not responsive to the request.

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.



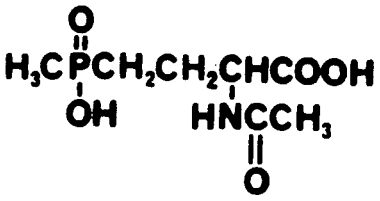
4-Methylphosphinico-2-oxo-butanoic acid

Hoe 065594



3-Methylphosphinico-3-oxo-propionic acid

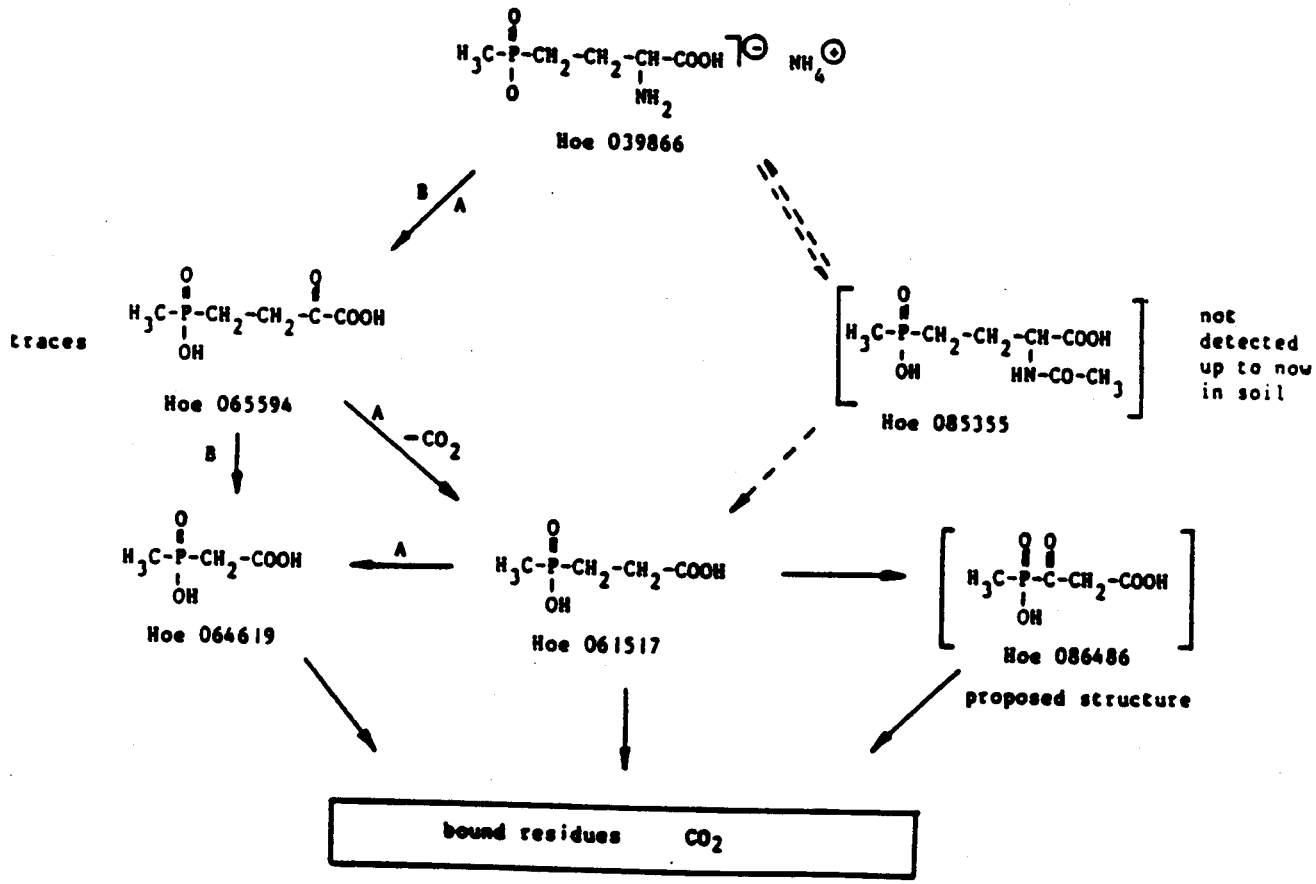
Hoe 086486



2-Acetamido-4-methylphosphinico-butanoic acid

Hoe 085355

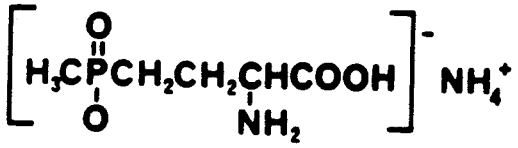
PROPOSED DEGRADATION SCHEME FOR HOE-039866 IN SOIL



APPENDIX

GLUFOSINATE AMMONIUM AND ITS DEGRADATES

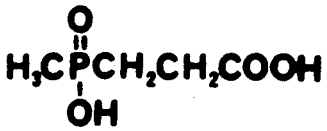
REGISTRANT-PROPOSED DEGRADATION PATHWAY



Glufosinate ammonium

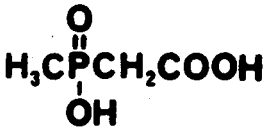
Hoe 039866

Ammonium-DL-homoalanin-4-yl(methyl)phosphinate



3-Methylphosphinico-propionic acid

Hoe 061517



2-Methylphosphinico-acetic acid

Hoe 064619

3. If the partially acceptable studies identified in Table A (§162-1 and §163-1) cannot be made acceptable by the submission of additional information/data, then new studies must be submitted.

4. In Table A, "Yes" with Footnote 4 means the submitted study does not satisfy the data requirement. If the registrant can provide a satisfactory response to the comments in the individual DER of the study then, at the discretion of EFGWB the data may be acceptable after reevaluation. Otherwise, a new study must be conducted in accordance with Subdivision N Guidelines.

5. Since the Ignite labels do not permit aerial and/or mist blower applications and since Ignite has been classified in toxicological category #3, spray drift monitoring data under §158.440 are not required.

6. Since Ignite is a new chemical, it is not on the Ground Water Team's data call-in list of 141 pesticides. Since insufficient data on aerobic/anaerobic soil metabolism and field (soil) dissipation of Ignite and its major degradates have been submitted to date, the Ground Water Team of EFGWB defers the requirements for ground water monitoring studies at this time.

cc: Branch Chiefs

RIN 5218-93

EFGWB Reviews of Glufosinate (128850)

Page_____ is not included in this copy.

Pages 19 through 29 are not included.

The material not included contains the following type of information:

____ Identity of product inert ingredients.

____ Identity of product impurities.

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____ The product confidential statement of formula.

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DATA EVALUATION RECORD

STUDY 2

CHEM 128850 Glufosinate ammonium §161-2

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40345657
Stumpf, K. and C. Schink. 1986. HOE 039866-^{14}C: Photodegradation study in water. Project No. CB052/86. Report No. A34306. Prepared by Hoechst AG, Frankfurt, Federal Republic of Germany, and submitted by Hoechst Celanese Corporation, Somerville, NJ.

DIRECT REVIEW TIME = 15

REVIEWED BY: W. Higgins TITLE: Staff Scientist

EDITED BY: K. Patten K. Patten TITLE: Task Leader

APPROVED BY: W. Spangler W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation
Rockville, MD

TEL: 468-2500

APPROVED BY: P. Datta
TITLE: Chemist
ORG: EAB/HED/OPP
TEL: 557-9733

SIGNATURE: PRDatta 9/22/88

CONCLUSIONS:

Degradation - Photodegradation in Water

This study is scientifically sound and provides supplemental information towards the registration of glufosinate ammonium. This study does not fulfill EPA Data Requirements for Registering Pesticides because the study was terminated after 120 hours (rather than being conducted for 30 days) and the artificial light source was not similar to sunlight.

SUMMARY OF DATA BY REVIEWER:

[^{14}C]Glufosinate did not degrade in sterile, aqueous buffered solutions (pH 5, 7, and 9) treated with [3,4-^{14}C]glufosinate ammonium (radiochemical purity 98.4%) at ≈9 ppm and irradiated with a mercury vapor lamp (energy output of 1470 W/m^2) for 120 hours. After 120 hours, 95.8-100% of the applied was glufosinate and <0.3% had been evolved as $^{14}CO_2$. "Some" degradation (not quantified) was reported in the pH 9 solution

after 168 and 240 hours of irradiation. The material balances ranged from 89.6 to 100% of the applied.

DISCUSSION:

1. The study was conducted for an insufficient length of time (120 hours instead of 30 days). The registrant stated that 120 hours of irradiation with the mercury vapor lamp was equivalent to 30 days of natural sunlight irradiation. The intensity of natural sunlight was reported as 440 W/m^2 and that of the lamp was calculated as 1470 W/m^2. However, the irradiation spectrum of the lamp had discrete peaks of high intensity at several wavelengths and did not approximate the irradiation spectrum of natural sunlight. Therefore, the reviewer does not accept the claim that 1 hour of irradiation with the mercury vapor lamp was equivalent to 3.4 hours of natural sunlight irradiation.

2. In the first replication of the pH 9 solution photolysis study, the degradate HOE 061517 (3-methyl-phosphinicopropionic acid) was detected at 13.1 and 19.9% of the applied after 90 and 120 hours of irradiation, respectively. The registrant attributed the formation of this degradate to microbial degradation of the test substance, but did not provide evidence that the sterility of the test solution had been violated. Upon repeating the photolysis study at pH 9, 100% of the applied was parent material after 96 and 120 hours of irradiation, but "some" degradation (not quantified) was noted at 168 and 240 hours. The registrant chose to ignore the first pH 9 study and the 168- and 240-hour samples from the second pH 9 study, and stated that the half-life of glufosinate in all solutions was >300 days.

3. The registrant-calculated half-life of >300 days was based on the assumption that 1 hour of irradiation with the mercury vapor lamp was equivalent to 3.4 hours of sunlight and on discarding the 168- and 240-hour pH 9 samples.

4. The registrant stated that the test substance and the buffers do not absorb light at wavelengths >290 nm. However, absorption spectra were provided for only the test substance (Figure 7) and the acetate buffer (Figure 8). The registrant should provide absorption spectra for the test substance dissolved in each of the buffer solution.

5. No information was provided on how the dark controls were maintained.

6. The registrant stated that lamp specifications were taken from a technical data sheet; the intensity and wavelength spectrum emitted by the lamp under the study conditions were not measured.

7. The method detection limit was not reported.

MATERIALS AND METHODS

MATERIALS AND METHODS:

[3,4-^{14}C]Glufosinate ammonium (Hoe 039866, radiochemical purity 98.4%, specific activity 5.72 mCi/g, Hoechst AG) was added at ≈9 ppm to autoclave-sterilized aqueous buffered solutions adjusted to pH 5 (sodium acetate plus acetic acid), 7 (potassium dihydrogen phosphate plus sodium hydroxide), and 9 (potassium chloride, boric acid, and sodium hydroxide). Aliquots of the solutions were transferred to a photoreactor (Figures 1, 2, and 3) and irradiated continuously with a mercury vapor lamp (TQ 150 Z 3, Original Hanau Quarzlampen GmbH, 33.8 W, intensity 1470 W/m^2) for 120 hours. The lamp was inside a dip pipe made of Solidex glass that filtered out wavelengths <290 nm, and the solution surrounded the lamp. The photoreactor was connected to tubes containing methanol/ethanolamine, sulfuric acid, and diethylene glycol volatile trapping solutions. The reaction compartment was maintained at 25 ± 2°C. Additional aliquots of the buffer solutions were transferred to a photoreactor and incubated in the dark to serve as controls (no additional information on the dark control incubation conditions was provided). The irradiated and dark control test solutions were sampled at 0, 8, 24, 48, 72, 96, and 120 hours posttreatment. Volatile trapping solutions were sampled at the end of the experiment.

Total radioactivity in the test solutions and trapping solutions was quantified by LSC. Additional samples of the test solutions were also analyzed by HPLC with radioactivity detection.

The study was repeated with the pH 9 solution; the solution was sampled after 0, 72, 96, 120, 168, and 240 hours of irradiation and analyzed as described.

RIN 5218-93

EFGWB Reviews of Glufosinate (128850)

Page_____ is not included in this copy.

Pages 34 through 50 are not included.

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.

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____ 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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DATA EVALUATION RECORD

STUDY 3

CHEM 128850 Glufosinate ammonium §161-3

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40345658
Stumpf, K. and C. Schink. 1987. HOE 039866-^{14}C-Photodegradation on soil. Prepared by Hoechst AG, Frankfurt, FRG, and submitted by Hoechst Celanese Corporation, Somerville, NJ.

DIRECT REVIEW TIME = 12

REVIEWED BY: J. Harlin TITLE: Staff Scientist

EDITED BY: K. Patten TITLE: Task Leader

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation
Rockville, MD

TEL: 468-2500

APPROVED BY: P. Datta
TITLE: Chemist
ORG: EAB/HED/OPP
TEL: 557-9733

SIGNATURE:

CONCLUSIONS:

Degradation - Photodegradation on Soil

This study is scientifically sound and provides supplemental information towards the registration of glufosinate ammonium. This study does not fulfill EPA Data Requirements for Registering Pesticides because the study was terminated after 45 hours (rather than being conducted for 30 days).

SUMMARY OF DATA BY REVIEWER:

On loamy sand soil treated with [3,4-^{14}C]glufosinate ammonium (radiochemical purity 98%) at 1 mg/plate (≈1 kg/ha), [^{14}C]glufosinate declined to 87.5% of the applied radioactivity during 45 hours of continuous irradiation with a xenon arc lamp (820 W/m^2, 300-800 nm) at 20-30°C. The major degradate 3-methyl phosphonicopropionic acid (HOE 061517) accounted for up to 9.6% of the applied (maximum at 30 hours). Cumulative $^{14}CO_2$ and other volatile degradates totaled 4.54 and 0.18% of the applied,

respectively, and unextractable [^{14}C]residues were 3.6% of the applied at 45 hours posttreatment. One unidentified degradate was isolated at 2.8% of the applied after 4 hours of irradiation. In the dark control, [^{14}C]glufosinate accounted for 94.7% of the applied at 45 hours post-treatment; 3-methyl phosphonicopropionic acid was detected once, at 4 hours posttreatment (3.4% of the applied). Material balances for all samples ranged from 92.4 to 100% during the study.

DISCUSSION:

1. The study was terminated after 45 hours of exposure, rather than being conducted 30 days. The registrant stated that 45 hours of continuous artificial irradiation were equivalent to 30 days exposure (12 hours of sunlight per day), assuming that 1 hour of irradiation with the xenon arc lamp was equivalent to 8 hours of irradiation with natural sunlight. However, no data were provided in the original document to substantiate this claim. In the photodegradation in water study, the registrant stated that the intensity of natural sunlight was 440 W/m^2.

2. The treated soil samples were irradiated continuously for 45 hours. Continuous irradiation of the samples may distort the photolytic results when compared to natural sunlight conditions; the samples should have been irradiated on a 12-hour light:dark cycle.

3. The registrant-calculated half-life of >300 days was based on the claim that 1 hour of irradiation with the artificial light used in this study was equivalent to 8 hours of sunlight, and on the assumption that one day is equal to 12 hours of sunlight. Therefore, each "day" is equal to 1.5 hours of irradiation with the artificial light.

4. The temperature in the quartz glass dish containing the soil plates varied from 20-30°C, while the temperature on the surface of the glass dishes were ≈40°C. The temperature of the soil surface was apparently not measured; it may have been closer to 40°C than 20-30°C, since exposed surfaces tend to become hotter than the surrounding air. Also, it was stated that due to technical reasons (unspecified), the temperature was not measured during irradiation of the soil plates, but in a separate run.

5. The study author stated that the UV-absorption spectrum of glufosinate shows absorption only at 190 nm (Figure 9); therefore, photodegradation of glufosinate was not expected.

6. Recovery from fortified samples and detection limits were not provided.

MATERIALS AND METHODS

MATERIALS AND METHODS:

A slurry of air-dried, sieved (250 µm) sandy loam soil (58.9% sand, 30.9% silt, 10.2% clay, organic matter 1.6%, pH 5.7, CEC 5.8 meq/100 g) and water was spread on glass plates and air-dried. [^{3},4-^{14}C]Glufosinate ammonium (Hoe 039866; radiochemical purity 98%, specific activity 44.21 mCi/g, Hoechst AG) mixed with unlabeled glufosinate ammonium (purity 99.5%) in water was applied to the soil at 1 mg/plate (equivalent to ≈1 kg/ha), and the plates were dried under red light. The treated soil samples were placed in quartz glass boxes on top of cooling tables within a photoreactor chamber (Hanau Suntest, Figures 1-3). Additional plates were placed in the quartz glass boxes and the boxes were wrapped in aluminum foil to exclude light. The samples were irradiated continuously with a xenon arc lamp (D-6450 Hanau, Hanau Quarzlampen GmbH Company) emitting light of 820 W/m^{2} intensity between 300-830 nm. Measured intensities and spectral distribution of the light source are presented in Figure 4; the quartz glass enclosing the samples was used to filter out wavelengths <290 nm. The registrant stated that 24 hours of irradiation with the xenon lamp was equivalent to 16 days of natural sunlight with 12 hours of sunlight per day. Air was passed into the quartz glass box containing the samples, then through a series of gas traps containing 2-aminoethanol:2-methoxyethanol (3:7) to trap $^{14}CO_2$ and 2-methyoxyethanol to trap other volatiles. The temperature within the glass box varied between 20-30°C, and the temperature on the surface of the box was ≈40°C. Irradiated and dark control soil plates and the gas trapping solutions were sampled after 0, 4, 7, 16, 30, and 45 hours of irradiation.

Soil was scraped from the glass plate and transferred to a flask. The plates were washed with water and the washings were transferred to the flask containing the soil. The soils were then extracted three times with water at 70°C. Total [^{14}C]residues in each extract were determined by LSC; the extracts were combined and again analyzed by LSC. The combined extracts were diluted with water (samples with very low radioactivity were concentrated), and aliquots were analyzed for glufosinate and its degradates by anion exchange HPLC with radioactivity detection. [^{14}C]Compounds were identified by comparison to reference standards. The extracted soil was analyzed for unextractable radioactivity by LSC following combustion. The trapping solutions were diluted with methanol and analyzed for total radioactivity by LSC.

RIN 5218-93

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DATA EVALUATION RECORD

STUDY 4

CHEM 128850 Glufosinate ammonium §162-1

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40345659-A
Stumpf, K. 1987a. HOE 039866-^{14}C: Aerobic soil metabolism. Study No. CB060/86. Prepared by Hoechst AG, Frankfurt, Germany, and submitted by Hoechst Celanese Corp., Somerville, NJ.

DIRECT REVIEW TIME = 16

REVIEWED BY: L. Binari TITLE: Staff Scientist

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CONCLUSIONS:

Metabolism - Aerobic Soil

This study is acceptable and partially fulfills EPA Data Requirements for Registering Pesticides by providing information on the aerobic soil metabolism of glufosinate ammonium. However, the study was terminated after 120 days, before the patterns of formation and decline of the degradates 3-methylphosphinico-propionic acid and 2-methylphosphinico-acetic acid were established.

SUMMARY OF DATA BY REVIEWER:

[^{14}C]Glufosinate degraded with a half-life of ≈8-16 days in a silt loam and two sandy loam soils that were treated with [3,4-^{14}C]glufosinate ammonium (radiochemical purity 98.7%) at ≈7.5 ppm and incubated at 22 ± 2°C in the dark. The registrant-calculated half-lives were 20.7-23.3 days for the three soils. At 95-98 days posttreatment in the three soils, . . .

glufosinate comprised 2.9-7.4% of the applied radioactivity,

3-methylphosphinico-propionic acid (Hoe 061517) comprised 39.4-51.7%,

2-methylphosphinico-acetic acid (Hoe 064619) comprised 6.3-18.5%, and

3-methylphosphinico-3-oxo-propionic acid (Hoe 086486; tentative identification) comprised <1-5.4%;

$^{14}CO_2$ totaled 4.3-11.9% of the applied in the silt loam and Mississippi sandy loam soils, and 25.3-28.2% in the Frankfurt sandy loam soil. Between 98 and 120 days posttreatment (Frankfurt sandy loam soil only), glufosinate had decreased to 1.1% of the applied; 3-methylphosphinico-propionic acid and 2-methylphosphinico-acetic acid had decreased slightly, then increased to 37.0-40.8 and 4.8-5.0% of the applied, respectively; 3-methylphosphinico-3-oxo-propionic acid had decreased to <1% of the applied; and $^{14}CO_2$ totaled 31.4-35.4% of the applied.

DISCUSSION:

1. The study was terminated before the patterns of formation and decline of the degradates 3-methylphosphinico-propionic acid and 2-methylphosphinico-acetic acid were established. The duration for an aerobic soil metabolism study should be 1 year or until the pattern of decline of the test substance and the patterns formation and decline of degradates are established. An additional study that establishes the pattern of decline of 3-methylphosphinico-propionic acid, the major degradate of glufosinate ammonium, has been submitted by the registrant (Study 5). No additional information has been provided on 2-methylphosphinico-acetic acid.

2. Material balances for the silt loam soil were incomplete; up to 26.1% of the applied radioactivity was unaccounted for by 95 days posttreatment. Material balances for the Frankfurt sandy loam soil were variable but appear to be adequate to establish the half-life of the test substance.

3. The registrant reported that 40% of soil moisture capacity is approximately the lower limit of 75% of soil moisture capacity at 0.33 bar.

4. An additional experiment was performed in which a sample of Frankfurt sandy loam soil was amended with 5 g/kg of alfalfa meal (Lucerne) prior to the preincubation period, in order to provide the microorganism with additional carbon. The half-life of glufosinate in the amended soil was 16-32 days, and the registrant-calculated half-life was 14.6 days.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Experiment 1 (Soil analysis)

Samples of a silt loam and two sandy loam soils (Table 1) were sieved (2 mm), moistened to 40% of their moisture-holding capacity, and maintained in the dark at 22 ± 2°C for 1-2 weeks. Following this period, the soils were treated with [3,4-^{14}C]glufosinate ammonium (Hoe 039866; radiochemical purity 98.7%, specific activity 7.4 mCi/g, Hoechst AG) at ≈7.5 ppm (1.95 kg/ha), then moistened to 60% of their moisture-holding capacity. The treated soils were incubated in the dark at 22 ± 2°C in flasks sealed with cotton-wool plugs. Soil samples were taken at 0, 4, 8, 16, 32, 64, 95/98, and 120 (sandy loam V only) days posttreatment.

The soil was extracted twice with water by heating at 70°C for 3 hours. Extracts were separated from the soil by centrifugation, analyzed for radioactivity by LSC, then combined and analyzed for glufosinate and its degradates by HPLC with radioactivity detection. To confirm degradate characterizations, extracts were methylated and/or acetylated and analyzed by GC/MS. Unextractable [^{14}C]residues remaining in the soil were quantified by combustion and LSC.

Experiment 2 (Volatile determination)

The silt loam and two sandy loam soils were prepared and treated with [3,4-^{14}C]glufosinate ammonium as described in Experiment 1. The treated soils were incubated in the dark at 22 ± 2°C in flasks attached to a gas collection system having two successive traps; the first contained ethylene glycol and the second contained methanol:ethanolamine (7:3) (Figure 1). The flasks were purged with air (flow rate unspecified) daily for 8 hours. Gas trap solutions were sampled at 4, 8, 16, 32, 49, 64, 81, 95/98, and 120 days posttreatment. The sandy loam V soil was sampled at 120 days posttreatment (the other soils were not sampled).

Radioactivity in the gas trap solutions was quantified using LSC. The soil was extracted and analyzed as described in Experiment 1.

Table 1. Soil characteristics.

Soil type	Source	Sand	Silt	Clay	Organic matter	pH	CEC (meq/100 g)
		%					
Sandy loam V	Frankfurt, Germany	58.9	30.9	10.2	1.6	5.7	5.8
Sandy loam S	Leland, Mississippi	63.8	31.1	5.1	1.5	6.3	3.9
Silt loam	Frankfurt, Germany	26.8	57.0	16.2	0.9	7.4	13.0

[a] Data compiled from page 13 of the original document.

STUDY AUTHOR(S)'S RESULTS AND/OR CONCLUSIONS

RIN 5218-93

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DATA EVALUATION RECORD

STUDY 5

CHEM 128850 Glufosinate ammonium §162-1

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40345659-B

Stumpf, K. 1987b. Hoe 061517-^{14}C: Degradation in soil. Study No. CB065/86. Prepared by Hoechst AG, Frankfurt, Germany, and submitted by Hoechst Celanese Corp., Somerville, N.J.

DIRECT REVIEW TIME = 16

REVIEWED BY: L. Binari TITLE: Staff Scientist

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This study was designed to investigate the degradation of 3-methylphosphinico-propionic acid, the primary soil degradate of glufosinate, under aerobic soil conditions. It is being reviewed as part of the glufosinate ammonium data package.

CONCLUSIONS:

Metabolism - Aerobic Soil

This study is acceptable and partially fulfills EPA Data Requirements for Registering Pesticides by providing information on the aerobic soil metabolism of 3-methylphosphinico-propionic acid, the major degradate of glufosinate ammonium.

SUMMARY OF DATA BY REVIEWER:

[3-^{14}C]3-Methylphosphinico-propionic acid (Hoe 061517; radiochemical purity 96%), at ≈1.6 kg/ha, degraded with a half-life of >120 days in

88

sandy loam soil incubated at 22 ± 2°C in the dark. At 120 days post-treatment, 3-methylphosphinico-propionic acid comprised ≈56% of the applied radioactivity, $^{14}CO_2$ totaled ≈29% of the applied, and the only nonvolatile degradate isolated, 2-methylphosphinico-acetic acid (Hoe 064619), comprised <1% of the applied.

DISCUSSION:

1. The degradation of 3-methylphosphinico-propionic acid (the primary soil metabolite of glufosinate) in sandy loam soil under aerobic conditions was considerably slower in this study than in Study 4 (MRID 40345659-A), which investigated the aerobic soil metabolism of glufosinate ammonium. The registrant suggested that the differences were due to changed reaction kinetics and/or rate-limiting degradation steps.

2. The treatment rate for laboratory studies should be expressed in terms of µg of test compound per g of soil (i.e., ppm), rather than kg/ha.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Experiment 1 (Soil analysis)

Sandy loam soil (58.9% sand, 30.9% silt, 10.2% clay, 1.6% organic matter, pH 5.7, CEC 5.8 meq/100 g) was sieved (2 mm), moistened to 40% of its moisture capacity, and maintained in the dark at 22 ± 2°C for one week. Following this period, the soil was treated with [3-^{14}C]3-methylphosphinico-propionic acid (Hoe 061517; radiochemical purity 96%, specific activity 10.3 mCi/g, Hoechst AG) at ≈1.6 kg/ha, then moistened to 60% of its moisture capacity. The treated soil was incubated in the dark at 22 ± 2°C in flasks sealed with cotton-wool plugs. Soil samples were taken at 0, 4, 8, 16, 32, 64, 98, and 120 days posttreatment.

Radioactivity in the gas trap solutions was quantified by LSC. Soil was extracted twice with water at 70°C. Extracts were separated from the soil by centrifugation, then analyzed for radioactivity by LSC and for 3-methylphosphinico-propionic acid and its degradates by HPLC with radioactivity detection. To confirm degradate characterizations, extracts were methylated and/or acetylated and analyzed by GC/MS. Unextractable [^{14}C]-residues remaining in the soil were quantified by combustion and LSC.

Experiment 2 (Volatile determination)

The sandy loam soil was prepared and treated with [3-^{14}C]3-methylphosphinico-propionic acid as described in Experiment 1. The treated soil was incubated in the dark at 22 ± 2°C in flasks attached to a gas collection system having two successive traps; the first contained ethylene glycol and the second contained methanol:ethanolamine (7:3) (Figure 1). The flasks were purged with air (flow rate unspecified) daily for 8 hours. Gas trap solutions were sampled at 4, 8, 16, 32, 49, 64, 81, 98, and 120 days posttreatment. Soil was sampled at 120 days posttreatment and analyzed as described above. Radioactivity in the gas trap solutions was quantified by LSC.

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DATA EVALUATION RECORD

STUDY 6

CHEM 128850 Glufosinate ammonium § 162-1

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40345659-C
Gildemeister, H. and H.J. Jordan. 1986b. HOE 039866-^{14}C: Aerobic soil metabolism study. Report No. CB066/85. Performed by Hoechst AG, Frankfurt, Federal Republic of Germany, and submitted by Hoechst Celanese Corporation, Somerville, NJ.

FICHE/MASTER ID 40345659-D
Gildemeister, H. and H.J. Jordan. 1986a. Amendment to HOE 039866-^{14}C: Aerobic soil metabolism study. Report No. A33847. Performed by Hoechst AG, Frankfurt, Federal Republic of Germany, and submitted by Hoechst Celanese Corporation, Somerville, NJ.

DIRECT REVIEW TIME = 5

REVIEWED BY: W. Higgins TITLE: Staff Scientist

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CONCLUSIONS:

Metabolism - Aerobic Soil

When previously reviewed (07/30/86), this study did not fulfill data requirements because chromatograms and retention time ranges for degradate 3-methylphosphinico-propionic acid (Hoe 061517) had not been provided, HPLC results for the parent material and 3-methylphosphinico-propionic acid were not validated through an acceptable method such as GC/MS, and further work was needed to determine the half-life of 3-methylphosphinico-propionic acid in soil. The registrant has responded by providing chromatograms and retention time ranges for 3-methylphos-

phinico-propionic acid and information on the confirmation of the identity of 3-methylphosphinico-propionic acid by GC/MS. Additional discussion was provided on the pattern of formation and decline of 3-methylphosphinico-propionic acid.

Based on the additional information provided, this study is scientifically sound and provides supplemental information towards the registration of glufosinate ammonium. This study does not fulfill EPA Data Requirements for Registering Pesticides because the study duration (96 days) was insufficient to define the pattern of formation and decline of degradates M1 and M3. Based on the data provided, degradate M1 should have been identified.

DATA EVALUATION RECORD

STUDY 7

CHEM 128850 Glufosinate ammonium §162-1

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40501018
Smith, A.E. 19??. Persistence and transformation of the herbicide ^{14}C-glufosinate-ammonium in prairie soils under laboratory conditions. Prepared by Agriculture Canada Research Station, Regina, Saskatchewan, Canada, and submitted by Hoechst Celanese Corporation, Somerville, NJ.

DIRECT REVIEW TIME = 12

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CONCLUSIONS:

Metabolism - Aerobic Soil

This study is unacceptable because material balances were incomplete (up to 71% of the applied radioactivity was unaccounted for). In addition, this study would not fulfill EPA Data Requirements for Registering Pesticides because degradates were not characterized.

The supplemental $^{14}CO_2$ evolution experiment did not adequately account for the missing material because the soil was sampled only once, at 111 days posttreatment, and the data were not comparable to the primary soil degradation experiment because the experimental design was different than that used in the primary experiment.

SUMMARY OF DATA BY REVIEWER:

Experiment 1 (Soil analysis)

[^{14}C]Glufosinate degraded with half-lives of ≈3 days in a sandy loam soil and 3-7 days in clay and clay loam soils that were treated with [3,4-^{14}C]glufosinate ammonium (radiochemical purity 95%) at 2 ppm, then incubated at 20 ± 1°C and 85% of field moisture capacity in the dark. At 84 days posttreatment, glufosinate comprised 2-4% and unidentified degradates comprised 25-49% of the applied radioactivity.

Experiment 2 ($^{14}CO_2$ evolution)

At 90 days posttreatment, 28, 44, and 55% of the applied radioactivity had evolved as $^{14}CO_2$ from clay loam, clay, and sandy loam soils, respectively, that were treated with [3,4-^{14}C]glufosinate ammonium (radiochemical purity 95%) at 2 ppm and incubated at 20 ± 1°C and 85% of field moisture capacity in the dark.

DISCUSSION:

General

1. The test substance was a mixture of [^{14}C]glufosinate ammonium and unlabeled formulated glufosinate ammonium; the formulation used to dilute the radioactive material was not specified.

2. CEC values for the test soils were not provided.

3. The soil moisture content was maintained at 85% of field capacity rather that 75%.

Experiment 1 (Soil analysis)

1. Material balances were incomplete because only soluble [^{14}C]residues were measured; the soils were not combusted before extraction to determine total [^{14}C]residues remaining in the soil or after extraction to quantify unextractable [^{14}C]residues. Up to 71% of the applied radioactivity was unaccounted for.

2. Degradates were not characterized.

3. The concentration of glufosinate and its degradates was normalized to account for 100% of the [^{14}C]residues applied to the TLC plates; however, since TLC plate recoveries were not reported, it could not be determined if material was lost during TLC analysis.

4. It was not specified that reference compounds were cochromatographed with the samples.

Experiment 2 ($^{14}CO_2$ evolution)

1. Although data from this study were intended to supplement Experiment 1, the experimental designs were sufficiently different that it is uncertain whether the results are comparable. In this experiment, the soil was analyzed only once, at 111 days posttreatment (21 days after chloroform treatment). At that time, the data reported were "radioactivity released as CO_2" (presumably radioactivity released between 0 and 90 days), "aqueous extractable radioactivity", "radioactivity in the biomass" (presumably radioactivity released between 90 and 111 days), "radioactivity in the fulvic, humic, and humin soil fractions", and "total radioactivity"; none of these data are comparable to data from Experiment 1. The aqueous extractable radioactivity data, the only data obtained in both Experiments 1 and 2, were obtained at 111 days posttreatment, 21 days after chloroform fumigation of the soil.

2. Data resulting from the chloroform fumigation-incubation technique were not reviewed in detail because they are not pertinent to EPA data requirements for an aerobic soil metabolism study.

Additional

1. From an additional experiment conducted using air-dried soils (moisture content 10% of field capacity) incubated at 20 ± 1°C, it was reported that glufosinate underwent no detectable breakdown; however, no data were provided.

2. An additional experiment conducted at 10 ± 1°C was not reviewed because the experimental design is not pertinent to current data requirements.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Experiment 1 (Soil analysis)

Clay, clay loam, and sandy loam soils (Table 1) were sieved (2 mm), placed in polystyrene foam cartons, moistened to 85% of field capacity, and maintained in the dark at 20 ± 1°C for 7 days. Following this period, the soils were treated with a mixture of [3,4-^{14}C]glufosinate ammonium (radiochemical purity 95%, specific activity μCi/mg, Hoechst Aktiengesellschaft) and unlabeled formulated glufosinate ammonium (200 g/L, formulation not further described) at 2 ppm (1 kg/ha). The treated soil was mixed and incubated in the dark at 20 ± 1°C in lightly capped cartons; the soil moisture was maintained at 85% of field capacity by the addition of water every other day. Soil samples were taken at 0.1, 3, 7, 14, 28, 56, and 84 days posttreatment.

Soil samples were extracted with water and calcium hydroxide for ≈2 hours. Extracts were analyzed for radioactivity by LSC. Aliquots of the extracts were acidified with 12 N hydrochloric acid and evaporated to dryness. The residue was dissolved in water and analyzed by TLC on cellulose plates developed in 2-propanol:acetone:? N hydrochloric acid (30:7.5:12.5) or n-butanol:acetone:? N hydrochloric acid (25:10:15). Radioactive areas were located and quantified using a TLC linear analyzer. Unlabeled reference compounds were visualized under UV light.

Experiment 2 ($^{14}CO_2$ evolution)

The clay, clay loam, and sandy loam soils were prepared and treated with the [3,4-^{14}C]glufosinate ammonium:formulated glufosinate ammonium mixture as described in Experiment 1. Each foam container containing a treated soil sample was placed in a Mason jar which held a vial of 0.2 N sodium hydroxide solution to absorb evolved $^{14}CO_2$. The treated soils were incubated in the dark at 20 ± 1°C and 85% of field moisture capacity. Samples of the sodium hydroxide solution were taken periodically (intervals unspecified) up to 90 days posttreatment. The sodium hydroxide solution was analyzed for radioactivity by LSC.

At 90 days posttreatment, the soils were sterilized with chloroform and incubated for an additional 21 days. The soils were then extracted as described previously. Total radioactivity in the extracts was determined by LSC; total radioactivity in the extracted soil was determined by LSC following combustion.

Table I. Composition and Physical Characteristics of Soils

soil	composition, % clay	silt	sand	organic content	field capacity, %	pH
clay	70	25	5	4.2	40	7.7
clay loam	30	40	30	11.7	35	6.0
sandy loam	10	25	65	4.0	20	7.6

STUDY AUTHOR(S)'S RESULTS AND/OR CONCLUSIONS

RIN 5218-93

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DATA EVALUATION RECORD

STUDY 8

CHEM 128850 Glufosinate ammonium §162-2

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40501014
Gildemeister, H. 1987. Hoe 039866-^{14}C: Anaerobic soil metabolism study. Project No. CB008/86, Report No. A36191. Unpublished study prepared by Hoechst AG, Frankfurt, Germany, and submitted by Hoechst Celanese Corp., Somerville, NJ.

DIRECT REVIEW TIME = 12

REVIEWED BY: L. Binari TITLE: Staff Scientist

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SIGNATURE: PRDatta 9/22/88

CONCLUSIONS:

Metabolism - Anaerobic Soil

This study is scientifically sound and provides supplemental information towards the registration of glufosinate ammonium. This study does not fulfill EPA Data Requirements for Registering Pesticides because two degradates detected at ≈0.04 ppm were not identified.

SUMMARY OF DATA BY REVIEWER:

Metabolism - Anaerobic Soil

[^{14}C]Glufosinate degraded with a half-life of 45-60 days in a silt loam soil treated with [3,4-^{14}C]glufosinate ammonium (radiochemical purity 99.5%) at approximately 8.8 ppm and incubated anaerobically (flooding plus N_2 atmosphere) at 22 ± 2°C in the dark for 60 days following 30 days of aerobic incubation. The registrant-calculated half-life was 56 days.

After 60 days of anaerobic incubation, glufosinate comprised 12.8% of the applied radioactivity, 3-methyl-phosphinico-propionic acid comprised 41.2% of the applied, and two unknowns each comprised approximately 5% (≈0.04 ppm) of the applied.

DISCUSSION:

1. Two degradates, each comprising ≈5% of the applied (0.04 ppm), were not identified. The study author stated that further investigations were underway to identify these compounds.

2. The decline in material balance at day 45 of anaerobic incubation (due to a cracked incubation flask) did not appear to significantly affect the reported results.

3. Although it was not clearly stated in the Materials and Methods section of the original report, the data indicate that corresponding water samples and soil extracts were combined prior to HPLC analysis.

4. The detection limit for the HPLC method was not specified.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Experiment 1 (Soil analysis)

Air-dried silt loam soil (8.7% sand, 73.1% silt, 18.2% clay, 1.0% organic matter, pH 5.9, CEC 16.1 meq/100 g) was sieved (1 mm), moistened to 80% of field capacity, and maintained in the dark at 22 ± 2°C for 2 weeks. Following this period, the soil was treated with [3,4-^{14}C]glufosinate ammonium (Hoe 039866; radiochemical purity 99.5%, specific activity 16.8 mCi/g, Hoechst AG) at ≈8.8 ppm. The treated soil was incubated in the dark at 22 ± 2°C in flasks sealed with cotton-wool plugs for 30 days. Following the 30-day aerobic incubation, anaerobic conditions were established by flooding the soil with distilled water containing peptone (1:15 w/v). The flasks were purged with nitrogen and sealed with ground-in stoppers. Soil samples were taken after 0 and 30 days of aerobic incubation and soil:water samples were taken after 0, 15, 30, 45, and 60 days of anaerobic incubation (30, 45, 60, 75, and 90 days posttreatment).

Soil samples taken during the aerobic portion of the study were extracted with distilled water for 6 hours at temperatures not exceeding 75°C. The extracts were analyzed for total [^{14}C]residues by LSC and for specific compounds by HPLC with radioactivity detection. Unextractable [^{14}C]residues remaining in the soil were quantified by LSC following combustion.

Samples taken during the anaerobic portion of the study were separated into water and soil fractions. The water fraction was analyzed for total radioactivity by LSC. The soil fraction was extracted with water, and the extract and extracted soil were analyzed as described above. Corresponding water samples and soil extracts were combined and analyzed for glufosinate and its degradates by HPLC with radioactivity detection.

Experiment 2 (Volatile determination)

Silt loam soil was prepared and treated with [3,4-^{14}C]glufosinate ammonium as described in Experiment 1. The treated soil was incubated aerobically for 30 days in the dark at 22 ± 2°C in flasks attached to a gas collection system having three successive traps containing sulfuric acid, ethylene glycol, and methanol:ethanolamine (7:3) (Figure 1). The flasks were purged with air (flow rate unspecified) daily for 8 hours. Following the 30-day aerobic incubation, anaerobic conditions were established as described above and the flasks were reattached to the gas collection system; the flasks were purged with nitrogen daily for 1 hour. Gas trap solutions were sampled after 8, 15, 21, and 30 days of aerobic incubation, and at various intervals up to day 60 of anaerobic incubation. The solutions were analyzed for total radioactivity by LSC.

STUDY AUTHOR(S)'S RESULTS AND/OR CONCLUSIONS

RIN 5218-93

EFGWB Reviews of Glufosinate (128850)

Page_____ is not included in this copy.

Pages 123 through 130 are not included.

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DATA EVALUATION RECORD

STUDY 9

CHEM 128850 Glufosinate ammonium §162-4

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40345660
Gildemeister, H., H. Jordan, and C. Schink. 1987. Hoe 039866-^{14}C: Aerobic aquatic metabolism study. Project No. CB064/86 and Report No. A35713. Unpublished study prepared by Hoechst AG, Frankfurt, Germany, and submitted by Hoechst Celanese Corp., Somerville, NJ.

DIRECT REVIEW TIME = 12

REVIEWED BY: L. Binari TITLE: Staff Scientist

EDITED BY: K. Patten K. Patten TITLE: Task Leader

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SIGNATURE: P Datta 9/22/88

CONCLUSIONS:

Metabolism - Aerobic Aquatic

This study is acceptable and fulfills EPA Data Requirements for Registering Pesticides by providing information on the degradation of glufosinate ammonium in a gravel-pit water:sand sediment system maintained under aerobic conditions.

SUMMARY OF DATA BY REVIEWER:

Under aerobic aquatic conditions, [^{14}C]glufosinate degraded with a half-life of 64 days in a gravel-pit water:sand sediment system that had been treated with [3,4-^{14}C]glufosinate ammonium (radiochemical purity 98.4%) at approximately 2 kg/ha. The registrant-calculated half-life was 29.1-35.2 days. At 94 days posttreatment, . . .

glufosinate comprised approximately 23% of the applied radioactivity,

3-methylphosphinico-propionic acid comprised 36% of the applied,

2-methylphosphinico-acetic acid comprised 18% of the applied, and

3-methylphosphinico-3-oxo-propionic acid (tentative identification) comprised 5% of the applied.

DISCUSSION:

1. The portion of this study investigating the degradation of glufosinate in river water and silt loam sediment was not addressed in the Summary of Data by Reviewer section of this report because the data were too variable to accurately assess the degradation of glufosinate and material balances were incomplete (up to 42% of the applied radioactivity was unaccounted for). Therefore, this portion of the study would not fulfill data requirements.

2. The dissolved oxygen content and hardness of the test waters were not provided.

3. The detection limit of the HPLC method was not reported.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Experiment 1

Silt loam (20.9% sand, 57.8% silt, 21.3% clay, 1.9% organic matter, pH 6.2, CEC 5.6 meq/100 g) and sand (93.4% sand, 6.6% silt, 0% clay, 1.5% organic matter, pH 7.5, CEC 2.1 meq/100 g) sediments from a river and gravel-pit, respectively, in Frankfurt, Germany, were separately mixed with water (ratio unspecified) and sieved (2 mm) prior to use. Samples of the sieved silt loam and sand sediments were flooded with river water (pH 6.2) and gravel-pit water (pH 7.5), respectively, to a depth of approximately 2 cm, then treated with [3,4-^{14}C]glufosinate ammonium (Hoe 039866; radiochemical purity 98.4%, specific activity 5.72 mCi/g, Hoechst AG) at ≈2 kg/ha. The treated water:sediment systems were incubated in the dark at 22 ± 2°C in flasks attached to a gas collection system having two successive traps; the first contained ethylene glycol and the second contained methanol:ethanolamine (7:3) (Figure 1). The flasks were purged with air (flow rate unspecified) daily for 8 hours. The water:sediment systems were continuously stirred during incubation. Gas trap solutions were sampled at 4, 8, 16, 32, 64, 84, and 94 days posttreatment. Water and sediment were sampled at 94 days posttreatment.

Radioactivity in the gas trap solutions was quantified using LSC. Water was separated from the sediment by centrifugation, and the water was analyzed for total radioactivity by LSC and for gluphosinate and its degradates by HPLC with radioactivity detection. Reference compounds were cochromatographed with the samples.

The sediment was extracted twice with distilled water by heating at 70°C for 3 hours. Extracts were separated from the sediment by centrifugation, combined, and analyzed by LSC and HPLC. Unextractable [^{14}C]residues remaining in the sediment were quantified by combustion and LSC.

Experiment 2

Silt loam and sand sediments were prepared, flooded, and treated with [3,4-^{14}C]glufosinate ammonium as described in Experiment 1. The treated water:sediment systems were incubated in the dark at 22 ± 2°C in flasks loosely sealed with cotton-wool plugs on a vibrating table. Water and sediment were sampled at 0, 4, 8, 16, 32, 64, and 94 days posttreatment and analyzed as described above.

STUDY AUTHOR(S)'S RESULTS AND/OR CONCLUSIONS

RIN 5218-93

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DATA EVALUATION RECORD

STUDY 10

CHEM 128850 Glufosinate ammonium §163-1

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40345662
Goerlitz, G. 1985. Adsorption/desorption in the system-soil/water, for HOE 039866 and HOE 061517. Prepared by Hoechst AG, Frankfurt, FRG, and submitted by Hoechst Celanese Corporation, Somerville, NJ.

DIRECT REVIEW TIME = 16

REVIEWED BY: J. Harlin TITLE: Staff Scientist

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SIGNATURE: P.R. Datta 9/22/88

CONCLUSIONS:

Mobility - Leaching and Adsorption/Desorption

This study is acceptable and partially fulfills EPA Data Requirements for Registering Pesticides by providing information on the mobility (batch equilibrium) of unaged glufosinate and unaged 3-methylphosphinico-propionic acid (Hoe 061517) in a sand, two silt loam, and a "volcanic ash" clay soil.

SUMMARY OF DATA BY REVIEWER:

Based on batch equilibrium experiments, [^{14}C]glufosinate was very mobile in sand and two silt loam soils and mobile in a "volcanic ash" clay soil when equilibrated at 22°C in 1:5 soil:calcium chloride solution slurries that contained 0.4-4.0 mg/L of [3,4-^{14}C]glufosinate ammonium (radiochemical purity 98%). Freundlich K_{ads} values were 0.08 for the sand, 0.98 for one silt loam, 3.48 for the second silt loam, and 52.85 for the "volcanic ash" clay soil; respective K_{oc} values were 9.6, 156, 352, and 1229. K_{des}

values for the two silt loam and the "volcanic ash" clay soils ranged from 4.4 to 152.9 following the first desorption step and 9.4 to 308 following the second desorption step (desorption was not studied in the sand soil).

Based on batch equilibrium experiments, [^{14}C]3-methylphosphinico-propionic acid was very mobile in sand and two silt loam soils and slightly mobile in a "volcanic ash" clay soil when equilibrated at 22°C in 1:5 soil:calcium chloride solution slurries that contained 0.4-4.0 mg/L of [3-^{14}C]3-methylphosphinico-propionic acid (radiochemical purity 99%). Freundlich K_{ads} values were <0.1 for the sand, 0.53 for one silt loam, 1.56 for the second silt loam, and 133 for the "volcanic ash" clay soil; respective K_{oc} values were <10, 84.1, 158, and 3093. K_{des} values for the one silt loam and the "volcanic ash" clay soils were 3.2 and 478 following the first desorption step, and 8 and 909 following the second desorption step (desorption was not studied in the sand and one silt loam soil).

DISCUSSION:

1. Based on a preliminary study using the "volcanic ash" clay soil, it was determined that equilibrium was established in 48 hours. Although a slight increase in the concentration of the active ingredient occurred after 72 hours, the study author attributed the increase to analytical inaccuracy.

2. The study author stated that glufosinate adsorption was strongly correlated with the organic matter content of the soils; correlation coefficients were 0.9774-0.9998 for the two silt loam and the "volcanic ash" clay soils and 0.5933 for the sand soil. However, the correlation must be considered tentative because three of the four soils (the sand and two silt loams) had organic matter contents clustered between 0.63 and 0.99. If soils with a range of organic matter contents had been studied, the correlation would have had more value.

3. The study author stated that glufosinate desorption experiments using the sand soil and 3-methylphosphinico-propionic acid desorption experiments using the sand and one silt loam soils were not conducted because the small amount (<5% of that in solution) of glufosinate adsorbed to the soil was near the detection limit, so too few [^{14}C]residues remained in the system during desorption to accurately measure.

4. Detailed results for the 3-methylphosphinico-propionic acid experiment were not provided; only calculated K, K_{oc}, and K_{clay} values were provided. The K values reported in the Discussion section of the original document do not match the K values reported in the table. It is possible that the values in the Discussion section are for the 4.0 mg/L treatment used in the desorption portion of the study and the values in the table are for all treatments, but the study author is not clear on this point. Although the values do not agree, the mobility classification for both sets of values is identical.

5. The clay soil was described as a volcanic ash, which is not typical of most soils in the continental U.S.. Glufosinate was less mobile in this soil than in the three soils more typical of the U.S.; however, this could be a result of the higher organic matter and clay content of the soil. The soil will be accepted in this case, but future studies should be conducted with soils typical of the area in which the pesticide is to be used in the U.S..

6. The pH of the test solutions was not reported.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Two silt loam soils, a sand soil, and a "volcanic ash" clay soil were air-dried, sieved (2 mm), and mixed with 0.01 M calcium chloride solutions (10 g soil:50 mL solution) containing either [3,4-^{14}C]glufosinate ammonium (Hoe 039866; radiochemical purity 98%, specific activity 49.43 mCi/g, Hoechst) or [3-^{14}C]3-methylphosphinico-propionic acid (Hoe 061517; radiochemical purity 99%, specific activity 24.7 mCi/g, Hoechst) at approximately 0.4, 0.8, 2.0, and 4.0 mg/L. The soil:solution slurries were gently shaken in stoppered flasks for 48 hours in a water bath maintained at 22 ± 0.1°C in the dark. Following equilibration, the solutions were filtered and aliquots of the filtrates were analyzed by LSC. Additional aliquots were analyzed for degradates using HPLC.

To determine desorption, pesticide-free 0.01 M calcium chloride solution was added to the soil that had been equilibrated at ≈4.0 mg/L to replace the filtrate removed after adsorption. Samples were equilibrated for 48 hours and aliquots of the aqueous fraction were analyzed by LSC. The desorption phase was repeated once. The remaining soil was analyzed for total radioactivity by LSC following combustion.

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Pages 157 through 180 are not included.

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____ The product confidential statement of formula.

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DATA EVALUATION RECORD

STUDY 11

CHEM 128850 Glufosinate ammonium §163-1

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40345661
Gildemeister, H. and U. Scheinkoenig. 1986a. HOE 039866-^{14}C: Leaching study and amendment. Project No. (B) 173185. Report Nos. A31970 and A33846. Prepared by Hoechst AG, Frankfurt, Germany, and submitted by Hoechst Celanese Corporation, Somerville, NJ.

DIRECT REVIEW TIME = 16

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EDITED BY: K. Patten K. Patten TITLE: Task Leader

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CONCLUSIONS:

Mobility - Leaching and Adsorption/Desorption

This study is unacceptable because the soils were sieved through 250-500 µm mesh screens, which removes a portion of the sand fraction and may cause the pesticide to appear less mobile. In general, this study was conducted according to current EPA Data Requirements for Registering Pesticides for mobility (soil TLC) studies.

SUMMARY OF DATA BY REVIEWER:

Based on soil TLC analysis and Helling's classification system, unaged [^{14}C]glufosinate was mobile (60-80% between R_f 0.65 and 0.89) in a sand and one silt loam soil, low to intermediately mobile (82.5% between R_f 0.10 and 0.64) in a second silt loam soil, and immobile (97% between R_f 0.00 and 0.09) in a "volcanic ash" clay soil that had been sieved through 250-500 µm mesh screens.

Based on soil TLC analysis and Helling's classification system, aged (30 days) [^{14}C]glufosinate residues were intermediately mobile to mobile (74.1% between R_f 0.35 and 0.89) in a silt loam soil, intermediately mobile (60.7% between R_f 0.35 and 0.64) in a sand soil, immobile to mobile (44.0% between R_f 0.00 and 0.09; 14.9-21.0% each between R_f 0.10-0.34, 0.35-0.64, and 0.65-0.89) in a second silt loam soil, and immobile (89.1% between R_f 0.00 and 0.09) in a "volcanic ash" clay soil that had been sieved through 250-500 µm mesh screens. Glufosinate and its degradate, 3-methylphosphinico-propionic acid, accounted for approximately 45.5 and 54.5% of the radioactivity in extracts of the aged soil that were applied to the soil TLC plates (9.8-15.7% and 11.7-18.7% of the radioactivity applied to the soil before incubation).

DISCUSSION:

1. The soil was sieved through a 250- or 500-µm mesh screens rather than the more typical 2000-µm mesh screen, so that a significant portion of the sand fraction (0.05-2.00 mm) may have been removed. In the present study, over-sieving the soils appears to have decreased the observed mobility of glufosinate residues in those soils. The same soils were used in this study as in the batch equilibrium study (Study 10), except that in the batch equilibrium study the soils were sieved through 2-mm mesh screens. In the batch equilibrium study, [^{14}C]glufosinate was very mobile in the sand and both silt loam soils, and mobile in the "volcanic ash" clay soil. In contrast, in this TLC study, [^{14}C]glufosinate was mobile in the sand and one silt loam soil, low to intermediately mobile in the second silt loam soil, and immobile in the "volcanic ash" clay soil.

2. The clay soil was described as a volcanic ash, which is not typical of most soils in the continental U.S.. Glufosinate was less mobile in this soil than in the three soils more typical of the U.S.; however, this could be a result of the higher organic matter and clay content of the soil. The soil will be accepted in this case, but future studies should be conducted with soils typical of the area in which the pesticide is to be used in the U.S..

3. A previous study dated September 4, 1985, "HOE 039866 ^{14}C: Leaching study" (Report No. 173/85, A31970) that was appended to this study had been previously review by EAB and was not accepted to fulfill data requirements. The study was not reconsidered in this report because the data requirement for mobility has been fulfilled by the batch equilibrium study (40345662, Study 10).

MATERIALS AND METHODS

MATERIALS AND METHODS:

Silt loam soil (silt loam I) was treated with [3,4-^{14}C]glufosinate ammonium (radiochemical purity 98.2%, specific activity 21.87 mCi/g, Hoechst) at 7.5 ppm. The treated soil was incubated in the dark in cotton-wool plug-sealed flasks at 40% of moisture holding capacity and 22°C for 30 days; the soil was remoistened at 2-3 day intervals.

Meanwhile, sand, volcanic ash, and two silt soils (Table 1) were air-dried, sieved through either 250 µm (medium- or fine-textured soils) or 500 µm (coarse-textured soils) mesh screens, and mixed with distilled water to produce slurries. The slurries were spread on glass plates (10 x 20 cm; two plates per soil type) to a thickness of 500 µm (medium- and fine-textured soils) or 750 µm (coarse-textured soils), then air-dried.

Following incubation, the treated, aged soil samples were mixed with water and emulsifier Hoe OS 1728 and transferred to closed dialyzing tubes, which were placed in 400 mL water in covered weighed containers for 24 hours. The soil:water slurries were extracted with water and allowed to settle. The supernatant was decanted and combined with the container water (extract I). The remaining soil:water slurry was mixed with distilled water, stirred, and after 24 hours, the water was decanted (extract II). Aliquots of the two extracts were analyzed separately for total radioactivity by LSC. Additional aliquots of both extracts were analyzed for glufosinate and its degradates by HPLC and identified by comparison to reference standards. The extracted soil samples were air-dried and analyzed for unextractable radioactivity by LSC following combustion.

Aliquots of the first extract (66748 dpm, concentration not reported in ppm) were spotted on the soil TLC plates along with undegraded [^{14}C]glufosinate and two reference compounds (chloridazon and buturon). The plates were air-dried, then developed in distilled water to a distance of 10 cm. Following development, the plates were air-dried and analyzed for glufosinate, its degradates, and the reference compounds using a TLC linear analyzer.

RIN 5218-93

EFGWB Reviews of Glufosinate (128850)

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DATA EVALUATION RECORD

STUDY 12

CHEM 00128850 Glufosinate ammonium §164-1

FORMULATION--??--AQUEOUS SOLUBLE

FICHE/MASTER ID 40345663
Horton, W.E. and R.L. Graney. 1987b. Determination of Ignite non-selective herbicide (Hoe 039866) and its metabolite (Hoe 061517) residues in soil from Quantico, Maryland. Performed by Dr. R. Ritter, Agronomy Dept., Univ. of Maryland, College Park, MD, and submitted by Hoechst Celanese Corporation, Somerville, NJ.

DIRECT REVIEW TIME = 16

REVIEWED BY: W. Higgins TITLE: Staff Scientist

EDITED BY: K. Patten K. Patten TITLE: Task Leader

APPROVED BY: W. Spangler W. Spangler TITLE: Project Manager

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CONCLUSIONS:

Field Dissipation - Terrestrial

This study is scientifically sound and provides supplemental information towards the registration of glufosinate ammonium. This study does not fulfill EPA Data Requirements for Registering Pesticides because the pattern of formation and decline of 2-methylphosphinico-acetic acid, a major degradate of glufosinate ammonium, was not addressed and acceptable freezer storage stability data were not provided.

SUMMARY OF DATA BY REVIEWER:

Glufosinate dissipated with an initial half-life of <3 days and a second half-life of 14-30 days in three unvegetated field plots of loamy sand soil in Quantico, Maryland, that were treated with glufosinate ammonium (Ignite, 1.67 lb/gal aqueous soluble formulation) at 3.0 lb ai/A on May

13, 1986; the registrant-calculated half-life was 20.2 days. In the 0- to 10-cm layer of soil, glufosinate declined from an average of 1.06 ppm (maximum 1.42 ppm) immediately posttreatment to 0.54 ppm by 3 days and <0.05 ppm (detection limit) by 93 days. In the 0- to 10-cm soil depth, the degradate 3-methylphosphinico-propionic acid (Hoe 061517) reached a maximum of 0.23 ppm on day 30 and declined to <0.05 ppm by day 93. Neither glufosinate nor 3-methylphosphinico-propionic acid were detected in soil samples taken deeper than 10 cm (soil was sampled to 75 cm) at any time during the study (duration of 147 days).

DISCUSSION:

1. The soil samples were only analyzed for glufosinate and 3-methylphosphinico-propionic acid (Hoe 061517). However, in an aerobic soil metabolism study (Study 4, MRID 40345659-A), 2-methylphosphinico-acetic acid (Hoe 064619) was also a major degradate, accounting for up to 18.5% of the applied after 95 days of aerobic incubation. Therefore, the soil should have also been analyzed for this degradate in the field dissipation study.

2. Soil samples were stored frozen for unspecified periods of time prior to analysis. Although a freezer storage stability study has been submitted, it is not acceptable due to variability of data and poor recovery. An acceptable freezer storage stability study must be received in order for data from freezer-stored samples to be considered acceptable.

3. The registrant-calculated half-life was 20.2 days; however, the observed half-life was <3 days. The discrepancy apparently was caused by the pattern of degradation of glufosinate. Glufosinate degraded very rapidly through day 3, then appeared to stabilize until day 30.

4. Recovery values from fortified soil samples were extremely variable, ranging from 56 to 120% of the applied for glufosinate and from 61 to 102% for Hoe 061517.

5. There are discrepancies in the data for degradate Hoe 061517 on day 14. Table 1 lists the concentration of Hoe 061517 in soil samples taken on day 14 as 0.07 ppm. In Table 3, the average value of samples taken on day 14 was listed as 0.21 ppm. However, the values of the individual replicates were 0.22, 0.13, and 0.17 ppm, the average of which is 17.3 ppm. Therefore, the reviewer is assuming that the actual value should be 17.3 ppm.

6. The depth to the water table was 10 feet.

7. During the study, rainfall totaled 17.76 inches, and air temperatures ranged from 35 to 94°C.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Glufosinate ammonium (Ignite, 1.67 lb/gal aqueous soluble formulation) was applied at 3.0 lb ai/A to three unvegetated field plots (20 x 20 feet, 1% slope) of loamy sand soil (85.2% sand, 10.4% silt, 4.4% clay, 0.9% organic matter, CEC 2.83 meq/100 g) located in Quantico, MD, on May 13, 1986. Soil samples were taken immediately prior to treatment and at intervals between 0 and 147 days posttreatment. Soil samples were taken using soil probes (0.75-1.0 inch in diameter) to a maximum depth of 75 cm and were stored frozen until analysis.

Soil samples were analyzed using Hoechst Analytical Method Number AL 38/85. Each sample was homogenized in the presence of an emulsifier (Hoe S 1728) and water. The homogenate was placed in a dialysis tube, then placed in water and allowed to stand for 24 hours. The dialyzate was evaporated to dryness; the residue was dissolved in acetic acid, then methylated by adding trimethyl orthoacetate and refluxing for 4 hours. To remove excess trimethyl orthoacetate while not losing volatile derivatives, toluene was added and the sample was concentrated by rotary evaporation; this procedure was repeated twice more. The concentrated sample was diluted with acetic acid methyl ester, then analyzed for glufosinate and Hoe 061517 by GC with phosphorus-sensitive flame photometer detection. The detection limit was 0.05 ppm. Recovery from fortified soil samples ranged from 56 to 120% of the applied for glufosinate and from 61 to 102% for Hoe 061517.

STUDY AUTHOR(S)'S RESULTS AND/OR CONCLUSIONS

RIN 5218-93

EFGWB Reviews of Glufosinate (128850)

Page_____ is not included in this copy.

Pages 197 through 204 are not included.

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____ Description of quality control procedures.

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____ Sales or other commercial/financial information.

____ A draft product label.

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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.

DATA EVALUATION RECORD

STUDY 13

CHEM 00128850 Glufosinate ammonium §164-1

FORMULATION--??--AQUEOUS SOLUBLE

FICHE/MASTER ID 40345664
Horton, W.E. and R.L. Graney. 1987c. Determination of Ignite non-selective herbicide (Hoe 039866) and its metabolite (Hoe 061517) residues in soil from Sunnyside, Washington. Performed by Northwest Agricultural Research, Sunnyside, WA, and submitted by Hoechst Celanese Corporation, Somerville, NJ.

DIRECT REVIEW TIME = 8

REVIEWED BY: W. Higgins TITLE: Staff Scientist

EDITED BY: K. Patten K. Patten TITLE: Task Leader

APPROVED BY: W. Spangler W. Spangler TITLE: Project Manager

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CONCLUSIONS:

Field Dissipation - Terrestrial

This study is scientifically sound and provides supplemental information towards the registration of glufosinate ammonium. This study does not fulfill EPA Data Requirements for Registering Pesticides because the pattern of formation and decline of 2-methylphosphinico-acetic acid, a major degradate of glufosinate ammonium, was not addressed and acceptable freezer storage stability data were not provided.

SUMMARY OF DATA BY REVIEWER:

Glufosinate dissipated with a half-life of 7-14 days in unvegetated field plots of loamy sand soil in Sunnyside, Washington, that were treated with glufosinate ammonium (Ignite, 1.67 lb/gal aqueous soluble formulation) at 2.0 lb ai/A on June 23, 1985; the registrant-calculated half-life was 15.0 days. In the 0- to 3-inch layer of soil, glufosinate declined from

205

a maximum of 1.72 ppm at 3 days posttreatment to 0.56 ppm at 14 days and <0.05 ppm at 90 days. In the 0- to 3-inch soil depth, the degradate 3-methylphosphinico-propionic acid (Hoe 061517) was detected at a maximum of 0.29 ppm on day 30 and declined to 0.23 ppm by day 90. Neither glufosinate nor 3-methylphosphinico-propionic acid were detected in any soil samples taken deeper than 3 inches (soil was sampled to 24 inches) at any time during the study (duration of 90 days).

DISCUSSION:

1. The soil samples were only analyzed for glufosinate and 3-methylphosphinico-propionic acid (Hoe 061517). However, in an aerobic soil metabolism study (Study 4, MRID 40345659-A), 2-methylphosphinico-acetic acid (Hoe 064619) was also a major degradate, accounting for up to 18.5% of the applied after 95 days of aerobic incubation. Therefore, the soil should have also been analyzed for this degradate in the field dissipation study.

2. Soil samples were stored frozen for approximately 12 to 16 months prior to analysis. Although a freezer storage stability study has been submitted, it is not acceptable due to variability of data and poor recovery. The study author also attempted to illustrate freezer storage stability by comparing the glufosinate concentration in soil samples taken immediately posttreatment (frozen 16 months prior to analysis) to the theoretical concentration of glufosinate in soil immediately posttreatment. However, theoretical values are not adequate to substantiate application rates; analysis of immediate posttreatment samples are necessary to verify application rate and the composition of the test substance. Therefore, the study author's explanation is not adequate. An acceptable freezer storage stability study must be received in order for data from freezer-stored samples to be considered acceptable.

3. The field test data indicate that there were three replicate plots treated with glufosinate ammonium; however, only one set of data were provided, apparently average values from the three plots. It would have been preferable if separate data for each plot had been provided in addition to the averaged values (as was done for Study 12, MRID 4034-5663), so that between-plot variability could be determined.

5. The depth to the water table was 5-15 feet.

6. During the study, rainfall totaled 1.6 inches, and air temperature ranged from 37 to 102°C. Soil temperatures were not reported. Meteorological data was collected approximately 9 miles from the actual test site. It is preferred that such information be measured at the test site, since rainfall and temperatures can vary between sites in close proximity.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Glufosinate ammonium (Ignite, 1.67 lb/gal aqueous soluble formulation) was applied at 2.0 lb ai/A to three unvegetated field plots (8 x 15 feet, slope <1%) of loamy sand soil (84.4% sand, 9.6% silt, 6.0% clay, 0.8% organic matter, pH 6.8, CEC 6.4 meq/100 g) located in Sunnyside, WA, on June 23, 1985. Soil samples were taken immediately prior to treatment and at intervals between 0 and 90 days posttreatment. Samples were collected by digging a hole with a shovel to expose the soil horizons and removing the appropriate soil layers with a knife. Triplicate soil samples were taken from each horizon and composited in the field. Samples were stored frozen until analysis.

Soil samples were analyzed by Hoechst Analytical Method Number AL 38/85. Each sample was homogenized in the presence of an emulsifier (Hoe S 1728) and water. The homogenate was placed in a dialysis tube, then placed in water and allowed to stand for 24 hours. The dialyzate was evaporated to dryness; the residue was dissolved in acetic acid, then methylated by adding trimethyl orthoacetate and refluxing for 4 hours. To remove excess trimethyl orthoacetate while not losing volatile derivatives, toluene was added and the sample was concentrated by rotary evaporation; this procedure was repeated twice more. The concentrated sample was diluted with acetic acid methyl ester, applied to a silica gel column, and eluted with methanol. The eluate was concentrated, diluted with methanol, and analyzed for methylated derivatives of glufosinate and Hoe 061517 by GC using phosphorus-sensitive flame photometric detection. The detection limit was 0.05 ppm. Recovery from fortified soil samples ranged from 66 to 100% of the applied for glufosinate and from 60 to 99% for Hoe 061517.

STUDY AUTHOR(S)'S RESULTS AND/OR CONCLUSIONS

RIN 5218-93

EFGWB Reviews of Glufosinate (128850)

Page_____ is not included in this copy.

Pages 210 through 217 are not included.

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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DATA EVALUATION RECORD

STUDY 14

CHEM 00128850 Glufosinate ammonium §164-1

FORMULATION--??--AQUEOUS SOLUBLE

FICHE/MASTER ID 40345665
Horton, W.E. and R.L. Graney. 1987a. Determination of Ignite non-selective herbicide (Hoe 039866) and its metabolite (Hoe 061517) residues in soil from Geneseo, Illinois. Performed by Van Der Schaaf Agricultural Research Inc., Geneseo, IL, and submitted by Hoechst Celanese Corporation, Somerville, NJ.

DIRECT REVIEW TIME = 8

REVIEWED BY: W. Higgins TITLE: Staff Scientist

EDITED BY: K. Patten K. Patten TITLE: Task Leader

APPROVED BY: W. Spangler W. Spangler TITLE: Project Manager

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APPROVED BY: P. Datta
TITLE: Chemist
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TEL: 557-9733

SIGNATURE: P.R. Datta 9/22/88

CONCLUSIONS:

Field Dissipation - Terrestrial

This study is scientifically sound and provides supplemental information towards the registration of glufosinate ammonium. This study does not fulfill EPA Data Requirements for Registering Pesticides because the pattern of formation and decline of 2-methylphosphinico-acetic acid, a major degradate of glufosinate ammonium, was not addressed and acceptable freezer storage stability data were not provided.

SUMMARY OF DATA BY REVIEWER:

Glufosinate dissipated with a registrant-calculated half-life of 6.3 days in three unvegetated field plots of silt loam soil in Geneseo, Illinois, that were treated with glufosinate ammonium (Ignite, 1.67 lb/gal aqueous soluble formulation) at 3.0 lb ai/A on May 14, 1986. In the 0- to 10-cm layer of soil, glufosinate declined from an average of 0.77 ppm at 1-day

posttreatment to 0.11 ppm at 15 days and ≤0.06 ppm at 30 days. In the 0- to 10-cm soil depth, 3-methylphosphinico-propionic acid (Hoe 061517) was detected at a maximum of 0.39 ppm on day 30 and declined to ≤0.08 ppm by day 90. Neither glufosinate nor 3-methylphosphinico-propionic acid were detected in any soil samples taken deeper than 10 cm (soil was sampled to 75 cm) at any time during the study (duration of 149 days).

DISCUSSION:

1. The soil samples were only analyzed for glufosinate and 3-methylphosphinico-propionic acid (Hoe 061517). However, in an aerobic soil metabolism study (Study 4, MRID 40345659-A), 2-methylphosphinico-acetic acid (Hoe 064619) was also a major degradate, accounting for up to 18.5% of the applied after 95 days of aerobic incubation. Therefore, the soil should have also been analyzed for this degradate in the field dissipation study.

2. Soil samples were stored frozen for unspecified periods of time prior to analysis. Although a freezer storage stability study has been submitted, it is not acceptable due to variablilty of data and poor recovery. An acceptable freezer storage stability study must be received in order for data from freezer-stored samples to be considered acceptable.

3. The concentration of glufosinate in all three plots was greater on day 1 than immediately posttreatment, and greater on day 7 than on day 3; it is therefore difficult to establish the half-life for glufosinate more accurately than occurring between 3 and 15 days posttreatment. The data are not so variable as to invalidate the study, because ≥≈80% of the applied dissipated by day 15 and ≥≈90% dissipated by day 30.

4. The depth to the water table was 45-85 feet.

5. During the study, rainfall totaled 30.54 inches, and air and soil temperatures ranged from 35 to 95°C and 47 to 78°C, respectively.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Glufosinate ammonium (Ignite, 1.67 lb/gal aqueous soluble formulation) was applied at 3.0 lb ai/A to three unvegetated field plots (30 x 10 feet, slope <1%) of silt loam soil (23.2% sand, 58.0% silt, 18.8% clay, 3.2% organic matter, pH 6.2, CEC 16.6 meq/100 g) located in Geneseo, Illinois, on May 14, 1986. Soil samples were taken immediately prior to treatment and at intervals between 0 and 149 days posttreatment. Samples were taken using a hydraulic probe (1-inch diameter) to a maximum depth of 75 cm and were stored frozen until analysis.

Soil samples were analyzed by Hoechst Analytical Method Number AL 38/85. Each sample was homogenized in the presence of an emulsifier and water. The homogenate was placed in a dialysis tube, then placed in water and allowed to stand for 24 hours. The dialyzate was evaporated to dryness. To remove possible traces of water, ethyl acetate was added to the residue and then evaporated off. The residue was dissolved in acetic acid and then methylated by adding trimethyl orthoacetate and refluxing for 2 hours. The solution was then diluted with toluene and concentrated by rotary evaporation; this procedure was performed three times. The concentrated solution was diluted with toluene and methyl acetate (50:50), applied to a silica gel column, and eluted with methyl acetate:-methanol (80:20). The eluate was concentrated under a stream of nitrogen to 1.0 mL. This sample was analyzed for glufosinate and Hoe 061517 by GC with PN-detection. The detection limit was 0.05 ppm. Recovery from fortified soil samples ranged from 67 to 81% of the applied for glufosinate and from 61 to 80% for Hoe 061517.

STUDY AUTHOR(S)'S RESULTS AND/OR CONCLUSIONS

RIN 5218-93

EFGWB Reviews of Glufosinate (128850)

Page_____ is not included in this copy.

Pages 223 through 229 are not included.

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____ Identity of product inert ingredients.

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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.

____ Information about a pending registration action.

✓ FIFRA registration data.

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DATA EVALUATION RECORD

STUDY 15

CHEM 128850 Glufosinate ammonium §165-1

FORMULATION--90--FORMULATION NOT IDENTIFIED

FICHE/MASTER ID 40501015
Schwalbe-Fehl, M. 1987a. Residue determination in rotational crops sown 30 days after treatment of soil. Study No. CM056/86 and Report No. A36703. Prepared by Hoechst AG, Frankfurt, FRG, and submitted by Hoechst Celanese Corporation, Somerville, NJ.

DIRECT RVW TIME = 8

REVIEWED BY: W. Higgins TITLE: Staff Scientist

EDITED BY: K. Patten K. Patten TITLE: Task Leader

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SIGNATURE: P.Datta 9/22/88

CONCLUSIONS:

Confined Accumulation - Rotational Crops

This study is unacceptable because the concentration of glufosinate in the soil was variable, so that the application rate could not be confirmed and the extent of pesticide uptake by the crops in relation to the concentration of pesticide in the soil was uncertain. In addition, this study would not fulfill EPA Data Requirements for Registering Pesticides because [^{14}C]residues were not characterized in all plants containing >0.01 ppm of residues.

Summary of Data By Reviewer:

[1^{4}C]Glufosinate ammonium residues accumulated in mature spinach (0.023 ppm in leaves), radishes (0.023 ppm in tubers, 0.025 ppm in roots, <0.021 ppm in leaves), wheat (0.247 ppm in straw, 0.024 ppm in grain, 0.312 ppm in chaff), and carrots (0.016 ppm in leaves, 0.012 ppm in tubers) planted

in sandy loam soil 30 days after the soil had been treated with formulated [3,4-^{14}C]glufosinate ammonium (radiochemical purity 98%) at 1 kg ai/ha. In wheat straw and chaff, 57.5-66.1% of the recovered radioactivity (≈100% of the extractable radioactivity) was 3-methylphosphinico-propionic acid (Hoe 061517) and 29.7-37.8% of the recovered radioactivity was unextractable in cold water. The concentrations in the plants were reported on a wet weight basis (except for the wheat straw and chaff that were dry at harvest) and were corrected for radioactivity in the control plants.

In the 0- to 5-cm soil depth, total [^{14}C]residues were 0.680 ppm immediately after treatment, 0.803 ppm at 30 days posttreatment (planting; 0.574 ppm glufosinate and 0.094 ppm Hoe 061517), 1.59 ppm at 74 days (radish harvest), 0.723 ppm at 89 days (spinach harvest), and 1.07 ppm at 172 days (wheat and carrot harvest). Between 30 and 172 days posttreatment, glufosinate decreased from 86 to 43% of the hot water-extractable radioactivity, 3-methylphosphinico-propionic acid increased from 14 to 50%, and 2-methylphosphinico-acetic acid (Hoe 064619) comprised up to 7.7% (maximum at 89 days posttreatment). Between 30 and 172 days posttreatment, unextractable [^{14}C]residues ranged from 16.9-24.7% of the applied radioactivity (0.17-0.27 ppm). [^{14}C]Residues were not found below the 5-cm soil depth, except on days 74 and 172 at 0.049 and 0.119 ppm, respectively.

DISCUSSION:

1. The concentration of [^{14}C]residues in the soil was quite variable, probably because of uneven application to the soil. The application of glufosinate ammonium to the soil was monitored by placing small (4 cm^2) filter paper discs on the soil surface prior to treatment. The discs were analyzed for total radioactivity by LSC shortly after treatment; recovery was 102 ± 57%.

   The variability of [^{14}C]residues in the soil made it difficult to accurately confirm the application rate and to determine the extent of pesticide uptake in the crops in relation to the concentration of residues in the soil.

2. Freezer storage stability data were not provided for unextracted plant tissue. Data were referenced, but the referenced material was not received by the reviewer. Freezer storage stability data for unextracted soil were provided by the registrant (Study 19), but the data were considered too variable and the recovery was poor.

3. The control plants contained up to 0.201 ppm of [^{14}C]residues. The registrant suggested that the control plants, which were kept in the same growth chamber as the treated plants, absorbed CO_2 from the atmosphere. This is logical, because CO_2 was determined to be a major degradate of glufosinate ammonium in the aerobic soil metabolism study; however, the data provided in this study do not support this theory. The concentration of [^{14}C]residues in the soil is greater at 172 days posttreatment than immediately posttreatment, which does not suggest that CO_2 was

evolved. The registrant did not take air samples to confirm that significant quantities of CO_2 were evolved.

4. The [^{14}C]glufosinate ammonium was mixed with a "blank formulation", and therefore must be considered a formulated product. The "blank formulation" was not described.

5. The plants were not sampled when immature.

6. Recoveries from fortified soil samples were not reported.

7. During the study, each container of soil was top-watered with 125.7 L and bottom-watered with 149 L of water. In the growth chamber, average air temperatures ranged from 3 to 28°C and relative humidity ranged from 50 to 90%.

MATERIALS AND METHODS

Materials and Methods:

[3,4-^{14}C]Glufosinate ammonium (Hoe 039866; radiochemical purity 98%, specific activity 44.19 mCi/g, Hoechst AG) was mixed with nonlabeled glufosinate ammonium (purity 99.6%) and "blank formulation" to produce a formulated test substance containing 18% active ingredient (1.9% radiolabeled ai). The formulated test substance was hand-sprayed at ≈1 kg ai/ha (70 mg/container) to the surface of sandy loam soil (59.1% sand, 32.1% silt, 8.8% clay, 2.27% organic matter, pH 5.65, CEC 6.22 meq/100 g) contained within stainless steel cylinders (1.0 m diameter, 0.5 m deep) located in a greenhouse. At the time of treatment, the soil surface was approximately one-third covered with weeds. The treated soil was allowed to age for 30 days, during which time average air temperatures ranged from 12-26°C and the soil was kept moist with ≈23.5 L/container of surface-applied water.

At 30 days posttreatment, the upper soil layer of each container was carefully loosened without removing the dried weeds, then planted with spinach, radishes, carrots, or wheat. Additional soils that had not been treated with [^{14}C]glufosinate ammonium were planted to serve as a control. All containers were transferred to a single growth chamber for the duration of the study. The plants were harvested at maturity: 74 days posttreatment (44 days postplanting) for the radishes (tubers, leaves, and roots); 89 days posttreatment for the spinach (leaves); and 172 days for wheat (straw, grain, and chaff) and carrots (tubers and leaves). Duplicate or triplicate soil samples (30-cm cores) were taken immediately after treatment, at the time of planting, and at the time of harvest of each crop. Soil samples were stored frozen for up to 3 months posttreatment prior to analysis. Plants were separated into their various parts, weighed, and cut into small pieces. Plants were subsampled for total radioactivity analysis by LSC following combustion; the remaining material was frozen for up to 6 months until further analysis.

The soil cores were thawed, divided into 5-cm sections, air-dried, and finely ground; subsamples were analyzed for total radioactivity by LSC following combustion. Additional portions of soil were extracted four times with hot water (70°C, pH 6). The water extracts were combined, concentrated, and analyzed by HPLC with UV and radioactivity detection. The extracted soil was analyzed for unextractable radioactivity by LSC following combustion.

The wheat straw and chaff (the only plant parts containing ≥0.025 ppm [^{14}C]residues) were thawed, homogenized, and extracted four times with cold water. The water extracts were combined, concentrated, and centrifuged to remove fine solid material. The radioactivity in the extracted plant tissue and the residue after centrifugation was determined by LSC following combustion. The water extracts were purified by anion exchange ion chromatography; the eluates were concentrated and analyzed by HPLC. [^{14}C]Residues were identified by GC/MS. Recoveries of glufosinate and its degradate 3-methyl phosphinico-propionic acid from fortified plant samples were 96.5 and 70.1%, respectively, and the detection limit ranged from 0.01-0.03 ppm.

RIN 5218-93

EFGWB Reviews of Glufosinate (128850)

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Pages 235 through 254 are not included.

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____ Identity of product inert ingredients.

____ Identity of product impurities.

____ Description of the product manufacturing process.

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____ Sales or other commercial/financial information.

____ A draft product label.

____ The product confidential statement of formula.

____ Information about a pending registration action.

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DATA EVALUATION RECORD

STUDY 16

CHEM 128850 Glufosinate ammonium §165-1

FORMULATION--90--FORMULATION NOT IDENTIFIED

FICHE/MASTER ID 40501016
Schwalbe-Fehl, M. 1987b. Residue determination in rotational crops sown 121 days after treatment of soil. Study No. CM088/85 and Report No. A35297. Prepared by Hoechst AG, Frankfurt, FRG, and submitted by Hoechst Celanese Corporation, Somerville, NJ.

DIRECT RVW TIME = 8

REVIEWED BY: W. Higgins TITLE: Staff Scientist

EDITED BY: K. Patten TITLE: Task Leader

APPROVED BY: B. Spangler TITLE: Project Manager

ORG: Dynamac Corporation
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TEL: 468-2500

APPROVED BY: P. Datta
TITLE: Chemist
ORG: EAB/HED/OPP
TEL: 557-9733

SIGNATURE:

CONCLUSIONS:

Confined Accumulation - Rotational Crops

This study is unacceptable because the concentration of glufosinate in the soil was variable, so that the application rate could not be confirmed and the extent of pesticide uptake by the crops in relation to the concentration of pesticide in the soil was uncertain. In addition, this study would not fulfill EPA Data Requirements for Registering Pesticides because [^{14}C]residues were not characterized in all plant parts containing >0.01 ppm of residues and [^{14}C]residues were not characterized in all soil samples.

Summary of Data By Reviewer:

[^{14}C]Glufosinate ammonium residues accumulated in mature spinach (0.019 ppm in leaves, 0.010 ppm in stems), radishes (0.045 ppm in leaves, 0.048 ppm in tubers), wheat (0.438 ppm in straw, 0.061 ppm in grain/husks,

233

0.285 ppm in "rest of ears"), and carrots (0.102 ppm in leaves, 0.012 ppm in tubers) planted in silt loam soil 121 days after the soil had been treated with formulated [3,4-^{14}C]glufosinate ammonium (radiochemical purity 99%) at 1.5 kg/ha. In the wheat straw, ≈100% of the water-extractable methanol-soluble radioactivity (81.1% of the recovered) was 3-methylphosphinico-propionic acid (Hoe 061517) and 7.3% was unextractable in water. The concentrations in the plants were reported on a wet weight basis (except for the wheat straw and chaff that were dry at harvest) and were corrected for radioactivity in the control plants.

In the 0- to 5-cm soil depth, total [^{14}C]residues averaged 0.73 ppm immediately posttreatment, 0.90 ppm at 121 days posttreatment (planting), 2.61 ppm at 158 days posttreatment, and 2.70 ppm at 245 days posttreatment. At 158 and 245 days posttreatment, 87.3-88.3% of the applied radioactivity in the upper 5 cm of soil was water-extractable; [^{14}C]-glufosinate comprised 18.3-20.6% of the recovered and 3-methylphosphinico-propionic acid comprised 57.8-79.4% of the recovered. An unidentified compound, detected only at 158 days posttreatment, comprised 23.8% of the recovered (0.54 ppm). At all sampling intervals, [^{14}C]residues were ≤0.023 ppm in the 5- to 10-cm depth and were not detected (<0.014 ppm) in the 10- to 15-cm depth.

Discussion:

1. The concentration of [^{14}C]residues in the soil was quite variable, probably because of uneven application to the soil. The distribution of glufosinate ammonium to the soil was determined in a separate experiment. Soil (0.7 m^2) was treated with glufosinate ammonium at 1 kg/ha in the same manner as the experimental samples. Twenty soil cores were taken from the upper 5 cm and analyzed for total radioactivity by LSC following combustion. Recovery ranged from 1.52-5.68 ppm (average 2.91 ± 1.00).

   The variability of [^{14}C]residues in the soil made it difficult to accurately confirm the application rate and to determine the extent of pesticide uptake in the crops in relation to the concentration of residues in the soil.

2. [^{14}C]Residues in the soil were characterized only at the time of harvest. One degradate in the day 158 soil sample was not characterized; the study author stated that attempts at identification were continuing.

3. It was not specified how the plant and soil samples were stored prior to analysis.

4. The control plants contained up to 321 dpm/g of [^{14}C]residues.

5. The [^{14}C]glufosinate ammonium was mixed with a "blank formulation", and therefore must be considered a formulated product. The "blank formulation" was not described.

6. The plants were not sampled when immature.

7. Recoveries from fortified soil samples were not reported.

8. During the study, each container of soil was top-watered with 123 L and bottom-watered with 145.2 L of water. Air temperatures ranged from 9 to 30°C.

MATERIALS AND METHODS

Materials and Methods:

[3,4-^{14}C]Glufosinate ammonium (Hoe 039866; radiochemical purity 99%, specific activity 47.6 mCi/g, Hoechst AG) was mixed with nonlabeled glufosinate ammonium (purity 99.5%) and "blank formulation" to produce a formulated test substance containing 18% active ingredient (3.0% radiolabeled ai). The formulated test substance was hand-sprayed at ≈1.52 kg ai/ha (106.1 mg/container) to the surface of silt loam soil (24.8% sand, 59.8% silt, 15.4% clay, 1.5% organic matter, pH 7.5, CEC 13.2 meq/100 g) contained within stainless steel cylinders (1.0 m diameter, 0.5 m deep) located in a greenhouse. The treated soil was allowed to age for 121 days, during which time average air temperatures ranged from 13-25°C and the soil was kept moist with ≈55 L/container of surface-applied water.

At 121 days posttreatment, the treated soils were planted with spinach, radishes, carrots, or wheat. Additional soils that had not been treated with [^{14}C]glufosinate ammonium were planted to serve as a control. The soils were kept in the greenhouse until the plants emerged, then were moved outdoors under cover to exclude rain. The plants were harvested at maturity: 158 days posttreatment (37 days postplanting) for the radishes (tubers and leaves); 156 days posttreatment for the spinach (leaves and stems); and 241 days for wheat (straw, grain/husks, and "rest of the ears") and carrots (tubers and leaves).

Soil samples (30-cm cores) were taken immediately after treatment, at the time of planting, and at the time of harvest of each crop, and sectioned into 0- to 5-, 5- to 10-, and 10- to 15-cm segments. The soils were air-dried and finely ground; subsamples were analyzed for total radioactivity by LSC following combustion. Additional portions of soil from 158 and 245 days posttreatment were extracted three times with hot water. The water extracts were combined, concentrated, and analyzed by HPLC with UV and radioactivity detection. The extracted soil was analyzed for unextractable radioactivity by LSC following combustion.

Plants were separated into their various parts, weighed, and cut into small pieces. Plants were subsampled for total radioactivity analysis by LSC following combustion; the remaining material was frozen until further analysis. Then, the wheat straw was thawed, homogenized, and extracted twice with cold water. The water extracts were combined, concentrated, and centrifuged to remove fine solid material. The radioactivity in the extracted plant tissue and the residue after centrifugation was determined by LSC following combustion. "Natural constituents" were precipitated from the water extracts using methanol. The resulting supernatants were purified by anion exchange ion chromatography; the eluates were concentrated and analyzed by HPLC. [^{14}C]Residues were identified by GC/MS.

RIN 5218-93

EFGWB Reviews of Glufosinate (128850)

Page_____ is not included in this copy.

Pages 260 through 274 are not included.

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____ 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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DATA EVALUATION RECORD

STUDY 17

CHEM 128850 Glufosinate ammonium §165-4

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40501017
Fischer, R. and M. Schwalbe-Fehl. 1985. HOE-039866-^{14}C-Flow-through bio-accumulation study with bluegill sunfish (Lepomis macrochirus). Study No. CM056/85, Report No. A32789. Prepared by Hoechst AG, Frankfurt, FRG, and submitted by Hoechst Celanese Corporation, Somerville, NJ.

DIRECT REVIEW TIME = 10

REVIEWED BY: L. Binari TITLE: Staff Scientist

EDITED BY: K. Patten TITLE: Task Leader

APPROVED BY: W. Spangler TITLE: Project Manager

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APPROVED BY: P. Datta
TITLE: Chemist
ORG: EAB/HED/OPP
TEL: 557-9733

SIGNATURE:

CONCLUSIONS:

Laboratory Accumulation - Fish

This study is scientifically sound and provides supplemental information towards the registration of glufosinate ammonium. This study does not fulfill EPA Data Requirements for Registering Pesticides because residues in the fish were not characterized,and the water samples from 3-21 day exposure period were not analyzed.

SUMMARY OF DATA BY REVIEWER:

Total [^{14}C]glufosinate residues did not accumulate in bluegill sunfish during 28 days of exposure to [3,4-^{14}C]glufosinate ammonium residues at 0.1 ppm in a flow-through system. The maximum concentration of [^{14}C]-residues (day 28) was 0.012 ppm in edible tissues (body, bones), 0.034 ppm in nonedible tissues (head, fins, viscera), and 0.018 ppm in whole

fish. After 14 days of depuration, [^{14}C]residues in edible tissues, nonedible tissues, and whole fish were <0.004, 0.006, and 0.005 ppm, respectively. No mortality of the fish in the treated or untreated aquaria was observed during the study.

Total [^{14}C]residues in the treated water ranged from 0.06 to 0.108 ppm during the exposure period. Analysis of the 3- and 21-day water samples from the exposure period found only glufosinate (data were not provided).

DISCUSSION:

1. [^{14}C]Residues in the fish were not characterized. The study authors stated that low levels of radioactivity in the fish tissues precluded degradate characterization; however, the low concentration of [^{14}C]-residues in the fish is, in part, a result of the low treatment level. The LC_{50} value and no-observed-effect level of glufosinate for bluegill sunfish were not reported so it could not be determined if the fish were exposed at the recommended concentration (10% of the 96-hour LC_{50}).

2. The bioaccumulation of residues had not plateaued when the exposure period was terminated.

3. It was reported that only glufosinate was detected in the 3- and 21-day water samples from the exposure period; however, no data were provided.

4. It was reported that "results were corrected by subtracting the radioactivity concentrations of the control samples from those of the treated fish." Results from the analysis of the control fish were not provided, but should be to establish the concentrations of radioactivity present. It was reported that control water samples did not contain any significant radioactivity.

5. Bioconcentration factor values should be provided for each fish tissue at every sampling interval during the exposure period. The study authors only reported that nominal BCF values ranged from 0.05 to 0.3.

6. Recoveries of glufosinate from fortified fish tissues were not reported.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Bluegill sunfish (Lepomis macrochirus, weight 2.0-3.5 g) were held in tanks on a 16-hour daylight photoperiod for at least 14 days prior to initiation of the study. Flow-through aquatic exposure systems were prepared using two 36-L stainless steel tanks. Aerated, filtered (sand and charcoal filters) tap water (pH 7.9-8.5, dissolved oxygen 2.3-10.9 mg/L, alkalinity 239-272 mg/L as $CaCO_3$, hardness 304-393 mg/L as $CaCO_3$, temperature 21.1-23.3°C; Table 4) was provided to each aquarium at a rate of 4.8 turnovers per day.

Bluegill sunfish (100) were placed in each aquarium, and one aquarium was continuously treated with [3,4-^{14}C]glufosinate ammonium (radiochemical purity 98%, specific activity 3.98 mCi/g, Hoechst AG) at 0.1 ppm. The second aquarium served as an untreated control. Following a 28-day exposure period, fish remaining in the [^{14}C]glufosinate ammonium-treated aquarium were transferred to an aquarium containing untreated water for a 14-day depuration period. From the control and treated aquaria, water was sampled daily and fish (5 or 20) were sampled on days 0, 1, 3, 7, 14, 21, and 28 of the exposure period. During the depuration, water samples and fish were taken on days 1, 3, 7, 10, and 14. Fish were frozen until analysis.

Radioactivity in the water samples was quantified by LSC. Water samples from days 3 and 21 of the exposure period were analyzed for glufosinate and its degradates by HPLC with radioactivity detection.

Whole fish (2) were analyzed for total radioactivity by LSC following combustion. Additional fish (3) were dissected into edible tissues (body, bones) and nonedible tissues (head, fins, viscera), and the two fractions were analyzed for radioactivity by LSC following combustion. The detection limit was 0.004 ppm.

STUDY AUTHOR(S)'S RESULTS AND/OR CONCLUSIONS

RIN 5218-93

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Pages 280 through 291 are not included.

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____ Identity of product impurities.

____ Description of the product manufacturing process.

____ Description of quality control procedures.

____ Identity of the source of product ingredients.

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____ A draft product label.

____ The product confidential statement of formula.

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DATA EVALUATION RECORD

STUDY 18

CHEM 128850 Glufosinate ammonium

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40345666
Specht, W., K. Kunzler, and H. Idstein. 1986. Method verification of the analytical method for the analysis of HOE 039866 and HOE 061517 in crops and soil and validation of the analytical method AL 38/85 for the residue analysis of HOE 039866 in plant materials, water, and soil. Project Nos. 31-A and (B)104/86. Prepared by Institut Fur Ruckstandsanalytik, Hamburg, and Hoechst AG, Frankfurt, Federal Republic of Germany, and submitted by Hoechst Celanese Corporation, Somerville, NJ.

DIRECT REVIEW TIME = 8

REVIEWED BY: L. Binari TITLE: Staff Scientist

EDITED BY: K. Patten K. Patten TITLE: Task Leader

APPROVED BY: W. Spangler W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation
Rockville, MD
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APPROVED BY: P. Datta
TITLE: Chemist
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TEL: 557-9733

SIGNATURE: P R Datta

CONCLUSIONS:

Ancillary Study - Methodology

This method may be acceptable for determining the concentration of glufosinate and 3-methylphosphinico-propionic acid (Hoe 061517) in soil and plant material.

SUMMARY OF DATA BY REVIEWER:

Experiment 1

Recoveries of glufosinate and 3-methylphosphinico-propionic acid (Hoe 061517) from soil (uncharacterized) fortified with glufosinate ammonium

292

(Hoe 039866) and 3-methylphosphinico-propionic acid (Hoe 061517) (purities >98%) at 0.08-0.6 ppm ranged from 68 to 86%. Recoveries of glufosinate and 3-methylphosphinico-propionic acid from plant samples, including potatoes; French bean plants, beans, straw, husks, and seeds; pea husks and seeds; corn plants, cob, and grain; broad bean beans, husks, and seeds; and sunflower plants and seeds, ranged from 68 to 93%.

Experiment 2

Final recoveries of radioactivity from soja seed and corn meal fortified (concentration unspecified) with [3,4-^{14}C]glufosinate ammonium (radiochemical purity 98%) ranged from 78 to 86 and 94 to 96%, respectively; GC analysis detected similar concentrations of glufosinate (75.9-82.9% of applied).

Comparison of several types of dialysis tubing (Spectropor membrane tubing, mw cutoff 6-8000; Thomapor "Special", mw cutoff 1000; and Nadir-Kalle) yielded comparable concentrations of radioactivity (≈69-137% of applied) in dialyzates from fortified water and plant samples, including hazelnut, corn cob and meal, olive soapstock, and soja seed.

It was reported that capillary GC columns did not provide a stable response as compared to packed GC columns. The capillary columns were only recommended if the separation capacity of the packed system was not sufficient.

DISCUSSION:

Experiment 1

Soil characteristics, including textural analysis and classification, organic matter content, pH, and CEC, were not provided.

Experiment 2

1. Fortification levels were not reported.

2. The results were inadequately discussed; for example, the relationship between Tables 5.1.3.2 and 5.1.3.2.1 is unclear.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Experiment 1

Plant and soil samples were homogenized and a subsample was fortified with glufosinate ammonium or 3-methylphosphinico-propionic acid (purities 98.3 and 100%, respectively; Hoechst AG) at ≈0.08-3.0 ppm. The fortified sample was then homogenized in the presence of an emulsifier (Hoe S 1728) and water. The homogenate was placed in a dialysis tube, then placed in water and allowed to stand for 24 hours. The dialyzate was evaporated to dryness; the residue was dissolved in acetic acid, then methylated by adding trimethyl orthoacetate and refluxing for 4 hours. To remove excess trimethyl orthoacetate while not losing volatile derivatives, toluene was added and the sample was concentrated by rotary evaporation; this procedure was repeated twice more. The concentrated sample was diluted with acetic acid, applied to a silica gel column, and eluted with methanol. The eluate was concentrated, diluted with acetone, and analyzed for methylated derivatives by GC using flame photometric detection. Glufosinate and its degradates were identified by comparison to authentic standards and quantified from peak areas or peak heights. The detection limit was 0.05 ppm.

Experiment 2

Water and plant samples were fortified (level unspecified) with [3,4-^{14}C]glufosinate ammonium (radiochemical purity 98%, specific activity 51.3 mCi/g, Hoechst AG). The samples were dialyzed and analyzed as described in Experiment 1 with recoveries of radioactivity being monitored at several stages of the procedure (see Section 5.1.1 of the original document for a flow-diagram). In addition, several types of dialysis tubing were compared for recovery of [^{14}C]residues, and the sensitivities of packed and capillary GC columns were compared. [^{14}C]Residues were quantified by LSC.

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____ A draft product label.

____ The product confidential statement of formula.

____ Information about a pending registration action.

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DATA EVALUATION RECORD

STUDY 19

CHEM 128850 Glufosinate

FORMULATION--90--FORMULATION NOT IDENTIFIED

FICHE/MASTER ID 40345667
Horton, W.E. 1987. Summary of the storage stability of Hoe 039866 and Hoe 061517 in soil. Submitted by Hoechst Celanese Corporation, Somerville, NJ.

DIRECT REVIEW TIME = 4

REVIEWED BY:	T. Colvin-Snyder	TITLE:	Staff Scientist
EDITED BY:	K. Patten K. Patten	TITLE:	Task Leader
APPROVED BY:	W. Spangler W. Spangler	TITLE:	Project Manager
ORG:	Dynamac Corporation Rockville, MD		
TEL:	468-2500		

APPROVED BY: P. Datta
TITLE: Chemist
ORG: EAB/HED/OPP
TEL: 557-9733

SIGNATURE: PKDatta 9/22/88

CONCLUSIONS:

Ancillary Study - Freezer Storage Stability

This study is unacceptable because recoveries from time 0 samples for both glufosinate and 3-methylphosphinico-propionic acid were poor (recoveries were as low as 65% for glufosinate and 60% for 3-methylphosphinico-propionic acid), recoveries from soil within each sampling interval were too variable to accurately assess the stability of glufosinate and 3-methylphosphinico-propionic acid in the soils (recoveries for the same test substance at the same sampling interval varied by as much as 29% of the applied), and the number of sampling intervals was inadequate (soils were only sampled at 0 and 6 months posttreatment). In addition, the maximum fortification rates (0.20 and 0.10 ppm for glufosinate and 3-methylphosphinico-propionic acid, respectively) were much lower than concentrations that were detected in soil treated at typical application rates, the test substances were not characterized, and the soil was not characterized.

SUMMARY OF DATA BY REVIEWER:

Recoveries of glufosinate in uncharacterized soil fortified with glufosinate ammonium (Hoe 039866, test substance uncharacterized) at 0.05-0.20 ppm were 71 ± 8% of the applied at time 0 and 74 ± 15% after 6 months of storage at -20°C. Recoveries of 3-methylphosphinico-propionic acid (Hoe 061517, test substance uncharacterized) in uncharacterized soil fortified at 0.05 and 0.10 ppm were 74 ± 17% at time 0 and 58 ± 7% after 6 months of storage at -20°C.

DISCUSSION:

1. Recoveries from time 0 samples for both glufosinate and 3-methylphosphinico-propionic acid were poor; recoveries were as low as 65% for glufosinate and 60% for 3-methylphosphinico-propionic acid.

2. Recoveries from soil within each sampling interval were too variable to accurately assess the stability of glufosinate and 3-methylphosphinico-propionic acid in the soils; recoveries for the same test substance at the same sampling interval varied by as much as 29% of the applied.

3. The number of sampling intervals was inadequate; soils were only sampled at 0 and 6 months posttreatment.

4. The maximum fortification rate (0.20 and 0.10 ppm for glufosinate and 3-methylphosphinico-propionic acid, respectively) were much lower than concentrations that would be expected in soils treated at typical application rates.

5. The test substances were not characterized; the purities and formulations of the test substances were not reported, and it was not specified if the test substances were radiolabeled.

6. The soil was not characterized; the USDA soil texture, soil textural analysis, percentage of organic matter, pH, and CEC were not reported.

7. The freezer storage stability of 2-methylphosphinico-acetic acid, an important glufosinate degradate, was not studied.

8. The analytical methods used to extract and analyze the soil were not provided; however, it is assumed that the author used the same analytical methodology used in the terrestrial field dissipation studies (Studies 12, 13, and 14).

9. The study author stated that data from a terrestrial field dissipation study in which soil samples were stored for 16 months demonstrate that glufosinate is stable in soil because 82 and 92% of the "expected residues" in time 0 soil samples were present as glufosinate equivalents in soil treated at 2.0 lb/A; and low levels of Hoe 061517, the expected degradate of instability, were found. Since this is a theoretical recovery of "expected residues" (the actual amount of glufosinate applied to the soil is not known), and total residues rather than parent glufosi-

nate were measured, these data do not provide evidence of the stability of glufosinate in soil.

10. The author stated, based on a journal article submitted with this report (Egli, H. 1982. J. Agric. Food Chem. 30:861-868), that residue stability at -20°C can be derived from the hydrolytic half-life of the chemical in neutral solution. The author of this report further stated that "This literature study indicated that residues would be stable for at least one year if the hydrolysis half-life at 50°C or 70°C was greater than 1 day or 10 days, respectively." The author then offered the glufosinate hydrolytic half-lives of >300 days at 25°C and >5 days at 50°C as evidence of freezer storage stability. However, in the conclusions section of the published journal article, it is stated that "If the half-life time of a compound is above 10 days at 70°C, its residues (if any) are stable for at least 1 year at -20°C. Residues of compounds with half-life times below 10 days (70°C) but above 1 day (50°C) are of uncertain stability..." Therefore, based on the journal article, the hydrolysis data presented by the study author only demonstrate that glufosinate residues are of uncertain stability at -20°C, since no data for hydrolysis at 70°C were presented.

    Also, the journal article should be used only to suggest whether a given compound might be stable when stored frozen. Except for the compounds which were actually under study (glufosinate was not studied), the correlation between hydrolytic half-lives and storage stability should be considered theoretical only, and therefore unacceptable for the purpose of registration.

MATERIALS AND METHODS

STUDY AUTHOR(S)'S RESULTS AND/OR CONCLUSIONS

PERTINENT DATA TABLES AND FIGURES

MATERIALS AND METHODS:

Soil samples (uncharacterized) were fortified with glufosinate (Hoe-039866, uncharacterized) at 0.05, 0.10, and 0.20 ppm. Additional soil samples (uncharacterized) were fortified with the degradate 3-methylphosphinico-propionic acid (Hoe-061517, uncharacterized) at 0.05, and 0.10 ppm. The soils were stored frozen at -20°C for six months. The soil samples were analyzed for glufosinate or 3-methylphosphinico-propionic acid immediately posttreatment and at 6 months posttreatment; the analytical methods were not provided.

RIN 5218-93

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EXECUTIVE SUMMARY

The following findings are derived from those reviewed studies which have met the requirements of 40 CFR Part 158.130 and the guidance of Subdivision N, and were also deemed acceptable.

Hydrolysis

Glufosinate did not hydrolyze in sterile, buffered, aqueous solutions (pH 5, 7, and 9) that were treated with glufosinate ammonium (purity 99.5%) at 232-236 ppm and incubated in the dark at 25 ± 0.1°C for 30 days. The hydrolytic half-life of glufosinate in solutions of pH 5, 7, and 9 was estimated by the registrant to be >300 days. During the study, the material balances ranged from 97.1 to 103.0% of the applied.

Aerobic soil metabolism

[^{14}C]Glufosinate degraded with a half-life of ≈8-16 days in a silt loam and two sandy loam soils that were treated with [3,4-^{14}C]glufosinate ammonium (radiochemical purity 98.7%) at ≈7.5 ppm and incubated at 22 ± 2°C in the dark. The registrant-calculated half-lives were 20.7-23.3 days for the three soils. At 95-98 days posttreatment in the three soils, glufosinate comprised 2.9-7.4% of the applied radioactivity, 3-methylphosphinico-propionic acid (Hoe 061517) comprised 39.4-51.7%, 2-methylphosphinico-acetic acid (Hoe 064619) comprised 6.3-18.5%, and 3-methylphosphinico-3-oxo-propionic acid (Hoe 086486; tentative identification) comprised <1-5.4%; $^{14}CO_2$ totaled 4.3-11.9% of the applied in the silt loam and Mississippi sandy loam soils, and 25.3-28.2% in the Frankfurt sandy loam soil. Between 98 and 120 days posttreatment (Frankfurt sandy loam soil only), glufosinate had decreased to 1.1% of the applied; 3-methylphosphinico-propionic acid and 2-methylphosphinico-acetic acid had decreased slightly, then increased to 37.0-40.8 and 4.8-5.0% of the applied, respectively; 3-methylphosphinico-3-oxo-propionic acid had decreased to <1% of the applied; and $^{14}CO_2$ totaled 31.4-35.4% of the applied.

[3-^{14}C]3-Methylphosphinico-propionic acid (Hoe 061517; radiochemical purity 96%), at ≈1.6 kg/ha, degraded with a half-life of >120 days in sandy loam soil incubated at 22 ± 2°C in the dark. At 120 days posttreatment, 3-methylphosphinico-propionic acid comprised ≈56% of the applied radioactivity, $^{14}CO2$ totaled ≈29% of the applied, and the only nonvolatile degradate isolated, 2-methylphosphinico-acetic acid (Hoe 064619), comprised <1% of the applied.

Aerobic aquatic metabolism

Under aerobic aquatic conditions, [^{14}C]glufosinate degraded with a half-life of 64 days in a gravel-pit water:sand sediment system that had been treated with [3,4-^{14}C]glufosinate ammonium (radiochemical purity 98.4%) at approximately 2 kg/ha. The registrant-calculated half-life was 29.1-35.2 days. At 94 days posttreatment, glufosinate comprised approximately 23% of the applied radioactivity, 3-methylphosphinico-propionic acid comprised 36% of the applied, 2-methylphosphinico-acetic acid comprised 18% of the applied, and 3-methylphosphinico-3-oxo-propionic acid (tentative identification) comprised 5% of the applied.

Mobility - Leaching and adsorption/desorption

Based on batch equilibrium experiments, [^{14}C]glufosinate was very mobile in sand and two silt loam soils and mobile in a "volcanic ash" clay soil when equilibrated at 22°C in 1:5 soil:calcium chloride solution slurries that contained 0.4-4.0 mg/L of [3,4-^{14}C]glufosinate ammonium (radiochemical purity 98%). Freundlich K_{ads} values were 0.08 for the sand, 0.98 for one silt loam, 3.48 for the second silt loam, and 52.85 for the "volcanic ash" clay soil; respective K_{oc} values were 9.6, 156, 352, and 1229. K_{des} values for the two silt loam and the "volcanic ash" clay soils ranged from 4.4 to 152.9 following the first desorption step and 9.4 to 308 following the second desorption step (desorption was not studied in the sand soil).

Based on batch equilibrium experiments, [^{14}C]3-methylphosphinico-propionic acid was very mobile in sand and two silt loam soils and slightly mobile in a "volcanic ash" clay soil when equilibrated at 22°C in 1:5 soil:calcium chloride solution slurries that contained 0.4-4.0 mg/L of [3-^{14}C]3-methylphosphinico-propionic acid (radiochemical purity 99%). Freundlich K_{ads} values were <0.1 for the sand, 0.53 for one silt loam, 1.56 for the second silt loam, and 133 for the "volcanic ash" clay soil; respective K_{oc} values were <10, 84.1, 158, and 3093. K_{des} values for the one silt loam and the "volcanic ash" clay soils were 3.2 and 478 following the first desorption step, and 8 and 909 following the second desorption step (desorption was not studied in the sand and one silt loam soil).

The following findings are derived from those reviewed studies which have not met the requirements of 40 CFR 158.30 and/or the Subdivision N Guidelines, but which have been deemed good studies following generally sound scientific practice. They thereby provide supplemental information on the fate of the pesticide.

Photodegradation in water

[^{14}C]Glufosinate did not degrade in sterile, aqueous buffered solutions (pH 5, 7, and 9) treated with [3,4-^{14}C]glufosinate ammonium (radiochemical purity 98.4%) at ≈9 ppm and irradiated with a mercury vapor lamp (energy output of 1470 W/m^2) for 120 hours. After 120 hours, 95.8-100% of the applied was glufosinate and <0.3% had been evolved as $^{14}CO_2$. "Some" degradation (not quantified) was reported in the pH 9 solution after 168 and 240 hours of irradiation. The material balances ranged from 89.6 to 100% of the applied.

Photodegradation on soil

On loamy sand soil treated with [3,4-^{14}C]glufosinate ammonium (radiochemical purity 98%) at 1 mg/plate (≈1 kg/ha), [^{14}C]glufosinate declined to 87.5% of the applied radioactivity during 45 hours of continuous irradiation with a xenon arc lamp (820 W/m^2, 300-800 nm) at 20-30°C. The major degradate 3-methyl phosphonicopropionic acid (HOE 061517) accounted for up to 9.6% of the applied (maximum at 30 hours). Cumulative $^{14}CO_2$ and other volatile degradates totaled 4.54 and 0.18% of the applied, respectively, and unextractable [^{14}C]-

residues were 3.6% of the applied at 45 hours posttreatment. One unidentified degradate was isolated at 2.8% of the applied after 4 hours of irradiation. In the dark control, [^{14}C]glufosinate accounted for 94.7% of the applied at 45 hours posttreatment; 3-methyl phosphonicopropionic acid was detected once, at 4 hours posttreatment (3.4% of the applied). Material balances for all samples ranged from 92.4 to 100% during the study.

Anaerobic soil metabolism

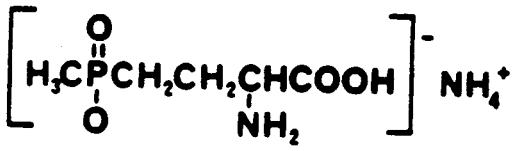
[^{14}C]Glufosinate degraded with a half-life of 45-60 days in a silt loam soil treated with [3,4-^{14}C]glufosinate ammonium (radiochemical purity 99.5%) at approximately 8.8 ppm and incubated anaerobically (flooding plus N_2 atmosphere) at 22 ± 2°C in the dark for 60 days following 30 days of aerobic incubation. The registrant-calculated half-life was 56 days. After 60 days of anaerobic incubation, glufosinate comprised 12.8% of the applied radioactivity, 3-methyl-phosphinico-propionic acid comprised 41.2% of the applied, and two unknowns each comprised approximately 5% (≈0.04 ppm) of the applied.

Terrestrial field dissipation studies

Glufosinate dissipated with an initial half-life of <3 days and a second half-life of 14-30 days in three unvegetated field plots of loamy sand soil in Quantico, Maryland, that were treated with glufosinate ammonium (Ignite, 1.67 lb/gal aqueous soluble formulation) at 3.0 lb ai/A on May 13, 1986; the registrant-calculated half-life was 20.2 days. In the 0- to 10-cm layer of soil, glufosinate declined from a average of 1.06 ppm (maximum 1.42 ppm) immediately posttreatment to 0.54 ppm by 3 days and <0.05 ppm (detection limit) by 93 days. In the 0- to 10-cm soil depth, the degradate 3-methylphosphinico-propionic acid (Hoe 061517) reached a maximum of 0.23 ppm on day 30 and declined to <0.05 ppm by day 93. Neither glufosinate nor 3-methylphosphinico-propionic acid were detected in soil samples taken deeper than 10 cm (soil was sampled to 75 cm) at any time during the study (duration of 147 days).

Glufosinate dissipated with a half-life of 7-14 days in unvegetated field plots of loamy sand soil in Sunnyside, Washington, that were treated with glufosinate ammonium (Ignite, 1.67 lb/gal aqueous soluble formulation) at 2.0 lb ai/A on June 23, 1985; the registrant-calculated half-life was 15.0 days. In the 0- to 3-inch layer of soil, glufosinate declined from a maximum of 1.72 ppm at 3 days posttreatment to 0.56 ppm at 14 days and <0.05 ppm at 90 days. In the 0- to 3-inch soil depth, the degradate 3-methylphosphinico-propionic acid (Hoe 061517) was detected at a maximum of 0.29 ppm on day 30 and declined to 0.23 ppm by day 90. Neither glufosinate nor 3-methylphosphinico-propionic acid were detected in any soil samples taken deeper than 3 inches (soil was sampled to 24 inches) at any time during the study (duration of 90 days).

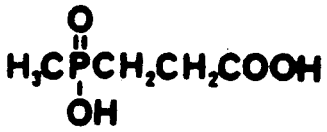
Glufosinate dissipated with a registrant-calculated half-life of 6.3 days in three unvegetated field plots of silt loam soil in Geneseo, Illinois, that were treated with glufosinate ammonium (Ignite, 1.67 lb/gal aqueous soluble formulation) at 3.0 lb ai/A on May 14, 1986. In the 0- to 10-cm layer of soil, glufosinate declined from an average of 0.77 ppm at 1-day posttreatment to 0.11 ppm at 15 days and ≤0.06 ppm at 30 days. In the 0- to 10-cm soil



Glufosinate ammonium

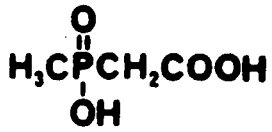
Hoe 039866

Ammonium-DL-homoalanin-4-yl(methyl)phosphinate



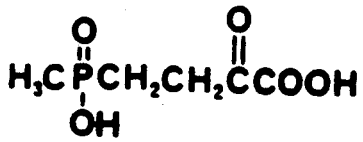
3-Methylphosphinico-propionic acid

Hoe 061517



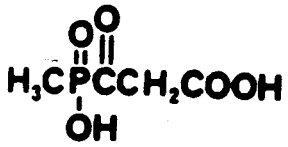
2-Methylphosphinico-acetic acid

Hoe 064619



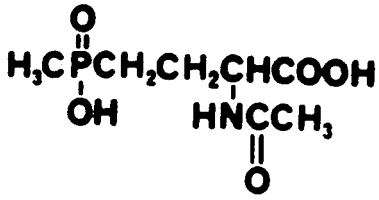
4-Methylphosphinico-2-oxo-butanoic acid

Hoe 065594



3-Methylphosphinico-3-oxo-propionic acid

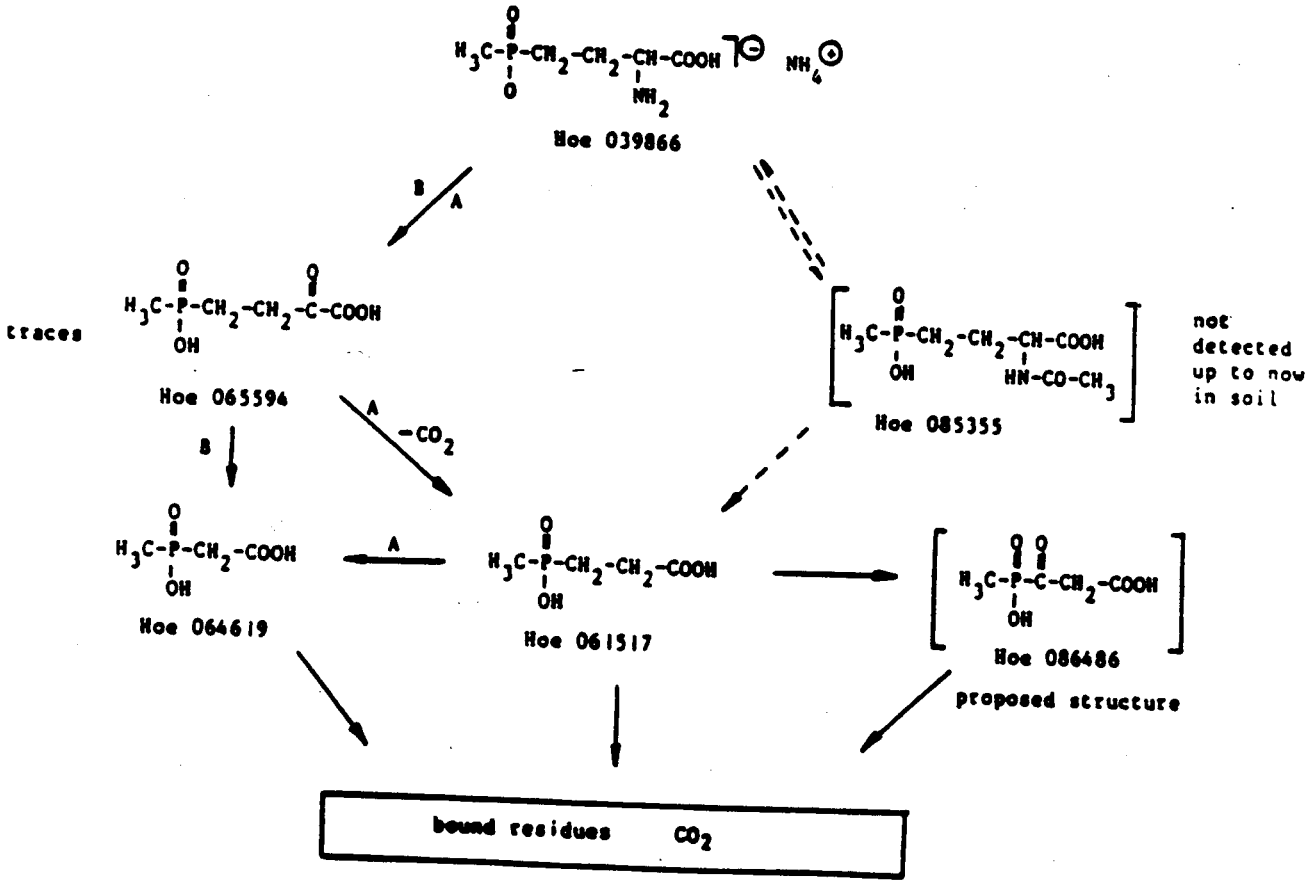
Hoe 086486



2-Acetamido-4-methylphosphinico-butanoic acid

Hoe 085355

PROPOSED DEGRADATION SCHEME FOR HOE-039866 IN SOIL



depth, 3-methylphosphinico-propionic acid (Hoe 061517) was detected at a maximum of 0.39 ppm on day 30 and declined to ≤0.08 ppm by day 90. Neither glufosinate nor 3-methylphosphinico-propionic acid were detected in any soil samples taken deeper than 10 cm (soil was sampled to 75 cm) at any time during the study (duration of 149 days).

Accumulation in laboratory fish

Total [^{14}C]glufosinate residues did not accumulate in bluegill sunfish during 28 days of exposure to [3,4-^{14}C]glufosinate ammonium residues at 0.1 ppm in a flow-through system. The maximum concentration of [^{14}C]residues (day 28) was 0.012 ppm in edible tissues (body, bones), 0.034 ppm in nonedible tissues (head, fins, viscera), and 0.018 ppm in whole fish. After 14 days of depuration, [^{14}C]residues in edible tissues, nonedible tissues, and whole fish were <0.004, 0.006, and 0.005 ppm, respectively. No mortality of the fish in the treated or untreated aquaria was observed during the study. Total [^{14}C]-residues in the treated water ranged from 0.06 to 0.108 ppm during the exposure period. Analysis of the 3- and 21-day water samples from the exposure period found only glufosinate (data were not provided).

RECOMMENDATIONS

Available data are insufficient to fully assess the environmental fate of the glufosinate ammonium. The submission of data required for full registration of the glufosinate ammonium on terrestrial food crop (field and vegetable crops, orchard crops, vineyards), terrestrial nonfood (around buildings at industrial, recreational, and farm sites; dry ditches and canals;, right-of-ways; fallow areas; ornamentals), greenhouse, and domestic outdoor use sites is summarized below:

The following data are required:

Photodegradation studies in water: One study (Stumpf and Schink, 40345657) was reviewed and provides supplemental information towards the registration of glufosinate ammonium. This study does not fulfill data requirements because the study was terminated after 120 hours (rather than being conducted for 30 days) and the artificial light source was not similar to sunlight.

Photodegradation studies on soil: One study (Stumpf and Schink, 40345658) was reviewed and provides supplemental information towards the registration of glufosinate ammonium. This study does not fulfill data requirements because the study was terminated after 45 hours (rather than being conducted for 30 days).

Anaerobic soil metabolism studies: One study (Guldemeister, 40501014) was reviewed and provides supplemental information towards the registration of glufosinate ammonium. This study does not fulfill data requirements because two degradates detected at ≈0.04 ppm were not identified.

Laboratory volatility studies: No data were reviewed, but all data are required because glufosinate ammonium is intended for field and vegetable

crop, orchard, and greenhouse uses. If the registrant provides information demonstrating that glufosinate does not pose a potential danger to workers (either by demonstrating that the pesticide has a very low vapor pressure or is nontoxic), this data requirement may be waived.

Terrestrial field dissipation studies: Three studies (Horton and Graney, 40345663, 40345664, 40345665) were reviewed and provide supplemental information towards the registration of glufosinate ammonium. These studies do not fulfill data requirements because the pattern of formation and decline of 2-methylphosphinico-acetic acid, a major degradate of glufosinate ammonium, was not addressed and acceptable freezer storage stability data were not provided.

Confined accumulation studies on rotational crops: Two studies (Schwalbe-Fehl, 40501015, 40501016) were reviewed and are unacceptable because the concentration of glufosinate in the soil was variable, so that the application rates could not be confirmed and the extent of pesticide uptake by the crops in relation to the concentration of pesticide in the soil was uncertain. In addition, these studies would not fulfill data requirements because [^{14}C]-residues were not characterized in all plants containing >0.01 ppm of residues.

Laboratory studies of pesticide accumulation in fish: One study (Fischer and Schwalbe-Fehl, 40501017) was reviewed and provides supplemental information towards the registration of glufosinate ammonium. This study does not fulfill data requirements because residues in the fish were not characterized, and the water samples from the 3- and 21- day exposure period were not analyzed.

The following data requirements are partially fulfilled:

Aerobic soil metabolism studies: Four studies were reviewed. One study (Smith, 40501018) was unacceptable and one study (Guldemeister and Jordan, 40345659-C and D) provided supplemental information only. A third study (Stumpf, 40345659-A) was acceptable and partially fulfills data requirements by providing information on the aerobic soil metabolism of glufosinate ammonium. However, the study was terminated after 120 days, before the patterns of formation and decline of the degradates 3-methylphosphinico-propionic acid and 2-methylphosphinico-acetic acid were established. A fourth study (Stumpf, 40345659-B) was acceptable and partially fulfills data requirements by providing information on the aerobic soil metabolism of 3-methylphosphinico-propionic acid, the major degradate of glufosinate ammonium. An additional study is needed to establish the pattern of decline of 2-methylphosphinico-acetic acid.

Leaching and adsorption/desorption studies: Two studies were reviewed. One study (Guldemeister and Scheinkoenig, 40345661) was unacceptable. A second study (Goerlitz, 40345662) was acceptable and partially fulfills data requirements by providing information on the mobility (batch equilibrium) of unaged glufosinate and unaged 3-methylphosphinico-propionic acid (Hoe 061517) in a sand, two silt loam, and a "volcanic ash" clay soil. An additional study is needed to establish the mobility of 2-methylphosphinico-acetic acid in one soil.

The following data requirements are fulfilled:

Hydrolysis studies: One study (Goerlitz et al., 40345656) was reviewed and fulfills data requirements because glufosinate ammonium did not hydrolyze in sterile aqueous solutions buffered to pH 5, 7, and 9.

Aerobic aquatic metabolism studies: One study (Guldemeister et al., 40345660) was reviewed and fulfills data requirements by providing information on the degradation of glufosinate ammonium in a gravel-pit water:sand sediment system maintained under aerobic conditions.

The following data requirements are deferred or are not required for presently registered uses:

Photodegradation in air studies: No data were reviewed. The data requirement is deferred pending the receipt of acceptable laboratory volatility studies.

Anaerobic aquatic metabolism studies: No data were reviewed; however, no data are required because glufosinate ammonium has no aquatic use, forestry use, or any aquatic impact use involving direct discharges of treated water into outdoor aquatic sites.

Field volatility studies: No data were reviewed. The data requirement is deferred pending the receipt of acceptable laboratory volatility studies.

Aquatic field dissipation studies: No data were reviewed; however, no data are required because glufosinate ammonium has no aquatic food crop use, aquatic nonfood use (including antifouling paints, ditchbanks, and shorelines), or aquatic impact use involving direct discharge of treated water into outdoor aquatic sites

Forestry dissipation studies: No data were reviewed. No data are required because the pesticide has no forestry use.

Dissipation studies for combination products and tank mix uses: No data were reviewed; however, no data are required because data requirements for combination products and tank mix uses are currently not being imposed.

Long-term field dissipation studies: No data were reviewed. No data are required because, based on the results from the field dissipation/aerobic soil metabolism studies, >50% of the applied dissipates in soil prior to the recommended subsequent application.

Field accumulation studies on rotational crops: No data were reviewed. The data requirement is deferred pending the receipt of acceptable accumulation studies in confined rotational crops.

Accumulation studies on irrigated crops: No data were reviewed; however, no data are required because the test substance is not intended for aquatic food crop or aquatic nonfood uses, for uses in and around holding ponds used for

irrigation purposes, or for uses involving effluents or discharges to water used for crop irrigation.

Field accumulation studies on aquatic nontarget organisms: No data were reviewed. No data are required because, based on an acceptable accumulation study in laboratory fish, glufosinate does not bioaccumulate in fish.

REFERENCES

Fischer, R. and M. Schwalbe-Fehl. 1985. HOE-039866-^{14}C-Flow-through bioaccumulation study with bluegill sunfish (Lepomis macrochirus). Study No. CM056/85, Report No. A32789. Prepared by Hoechst AG, Frankfurt, FRG, and submitted by Hoechst Celanese Corporation, Somerville, NJ. (40501017)

Gildemeister, H. 1987. Hoe 039866-^{14}C: Anaerobic soil metabolism study. Project No. CB008/86, Report No. A36191. Unpublished study prepared by Hoechst AG, Frankfurt, Germany, and submitted by Hoechst Celanese Corp., Somerville, NJ. (40501014)

Gildemeister, H. and H.J. Jordan. 1986a. Amendment to HOE 039866-^{14}C: Aerobic soil metabolism study. Report No. A33847. Performed by Hoechst AG, Frankfurt, Federal Republic of Germany, and submitted by Hoechst Celanese Corporation, Somerville, NJ. (40345659-D)

Gildemeister, H. and H.J. Jordan. 1986b. HOE 039866-^{14}C: Aerobic soil metabolism study. Report No. CB066/85. Performed by Hoechst AG, Frankfurt, Federal Republic of Germany, and submitted by Hoechst Celanese Corporation, Somerville, NJ. (40345659-C)

Gildemeister, H. and U. Scheinkoenig. 1986. HOE 039866-^{14}C: Leaching study and amendment. Project No. (B) 173185. Report Nos. A31970 and A33846. Prepared by Hoechst AG, Frankfurt, Germany, and submitted by Hoechst Celanese Corporation, Somerville, NJ. (40345661)

Gildemeister, H., H. Jordan, and C. Schink. 1987. Hoe 039866-^{14}C: Aerobic aquatic metabolism study. Project No. CB064/86 and Report No. A35713. Unpublished study prepared by Hoechst AG, Frankfurt, Germany, and submitted by Hoechst Celanese Corp., Somerville, NJ. (40345660)

Goerlitz, G. 1985. Adsorption/desorption in the system-soil/water, for HOE 039866 and HOE 061517. Prepared by Hoechst AG, Frankfurt, FRG, and submitted by Hoechst Celanese Corporation, Somerville, NJ. (40345662)

Goerlitz, G., C. Kloeckner, and U. Eyrich. 1986. Abiotic hydrolysis as a function of pH and amendment, and separation of potential hydrolysis products of HOE 039866 from the active ingredient by HPLC. Project Nos. (B)277/85 and (B)110/86. Prepared by Hoechst AG, Frankfurt, Federal Republic of Germany, and submitted by Hoechst Celanese Corporation, Somerville, NJ. (40345656)

Horton, W.E. 1987. Summary of the storage stability of Hoe 039866 and Hoe

061517 in soil. Submitted by Hoechst Celanese Corporation, Somerville, NJ. (40345667)

Horton, W.E. and R.L. Graney. 1987a. Determination of Ignite non-selective herbicide (Hoe 039866) and its metabolite (Hoe 061517) residues in soil from Geneseo, Illinois. Performed by Van Der Schaaf Agricultural Research Inc., Geneseo, IL, and submitted by Hoechst Celanese Corporation, Somerville, NJ. (40345665)

Horton, W.E. and R.L. Graney. 1987b. Determination of Ignite non-selective herbicide (Hoe 039866) and its metabolite (Hoe 061517) residues in soil from Quantico, Maryland. Performed by Dr. R. Ritter, Agronomy Dept., Univ. of Maryland, College Park, MD, and submitted by Hoechst Celanese Corporation, Somerville, NJ. (40345663)

Horton, W.E. and R.L. Graney. 1987c. Determination of Ignite non-selective herbicide (Hoe 039866) and its metabolite (Hoe 061517) residues in soil from Sunnyside, Washington. Performed by Northwest Agricultural Research, Sunnyside, WA, and submitted by Hoechst Celanese Corporation, Somerville, NJ. (40345664)

Schwalbe-Fehl, M. 1987a. Residue determination in rotational crops sown 30 days after treatment of soil. Study No. CM056/86 and Report No. A36703. Prepared by Hoechst AG, Frankfurt, FRG, and submitted by Hoechst Celanese Corporation, Somerville, NJ. (40501015)

Schwalbe-Fehl, M. 1987b. Residue determination in rotational crops sown 121 days after treatment of soil. Study No. CM088/85 and Report No. A35297. Prepared by Hoechst AG, Frankfurt, FRG, and submitted by Hoechst Celanese Corporation, Somerville, NJ. (40501016)

Smith, A.E. 19??. Persistence and transformation of the herbicide ^{14}C-glufosinate-ammonium in prairie soils under laboratory conditions. Prepared by Agriculture Canada Research Station, Regina, Saskatchewan, Canada, and submitted by Hoechst Celanese Corporation, Somerville, NJ. (40501018)

Specht, W., K. Kunzler, and H. Idstein. 1986. Method verification of the analytical method for the analysis of HOE 039866 and HOE 061517 in crops and soil and validation of the analytical method AL 38/85 for the residue analysis of HOE 039866 in plant materials, water, and soil. Project Nos. 31-A and (B)104/86. Prepared by Institut Fur Ruckstandsanalytik, Hamburg, and Hoechst AG, Frankfurt, Federal Republic of Germany, and submitted by Hoechst Celanese Corporation, Somerville, NJ. (40345666)

Stumpf, K. 1987a. HOE 039866-^{14}C: Aerobic soil metabolism. Study No. CB060/86. Prepared by Hoechst AG, Frankfurt, Germany, and submitted by Hoechst Celanese Corp., Somerville, NJ. (40345659-A)

Stumpf, K. 1987b. Hoe 061517-^{14}C: Degradation in soil. Study No. CB065/86. Prepared by Hoechst AG, Frankfurt, Germany, and submitted by Hoechst Celanese Corp., Somerville, N.J. (40345659-B)

Stumpf, K. and C. Schink. 1986. HOE 039866-^{14}C: Photodegradation study in water. Project No. CB052/86. Report No. A34306. Prepared by Hoechst AG, Frankfurt, Federal Republic of Germany, and submitted by Hoechst Celanese Corporation, Somerville, NJ. (40345657)

Stumpf, K. and C. Schink. 1987. HOE 039866-^{14}C: Photodegradation on soil. Prepared by Hoechst AG, Frankfurt, FRG, and submitted by Hoechst Celanese Corporation, Somerville, NJ. (40345658)

The following reports were not reviewed because they contain summary data only:

Horton, W.E. 1987. Summary of environmental fate data to support the terrestrial non-food crop use of Ignite non-selective herbicide. Submitted by Hoechst Celanese Corp., Somerville, NJ. (40345655)

Graney, R.L. 1987. Summary of environmental fate data to support the terrestrial food crop use of Ignite non-selective herbicide. Submitted by Hoechst Celanese Corp., Somerville, NJ. (40501013)

The following report was not reviewed because the experimental design is not pertinent to current environmental fate data requirements (ecosystem modeling study):

Graney, R.L. 1987. Potential aquatic environmental concentration and hazard evaluation for HOE-039866 active ingredient. Submitted by Hoechst Celanese Corp., Somerville, NJ. (40501019)