MEMORANDUM July 20, 1999

Subject: Review/Evaluation of Naphthalene Data for Reregistration

From: John Jordan, Ph.D. Microbiologist John Jordan
EFED/ERB III

To: Todd Peterson , CRM
SRRD

Thru: Dan Rieder, Chief 8/2/99
ERB III
EFED

Naphthalene - chemical code 055801 - (D179120)

EFED received two aerobic soil metabolism and one aqueous photolysis study for review and evaluation. Neither the two aerobic soil metabolism studies nor the aqueous photolysis study is acceptable.

162-1 Aerobic Soil Metabolism Studies. MRIDs No. 00156557 and 00160093

The material balances in the two aerobic soil metabolism studies were not within the acceptable range (70 - 100%) ,e.g., only from 55% to 69 % of the applied was recovered in one study and 39% to 47% in the other. The studies do not satisfy the requirement, because of low recovery. Because the aerobic soil metabolism study is required, the registrant should design a study that permits control of the volatile compound in order to obtain an acceptable mass balance.

161-2 Photodegradation in Water. MRID- No. 423358-04.

The study is not acceptable for satisfying the data requirement. The validity of the reported material balances could not be confirmed , because an error in calculation was apparent. However, the study is not required at this time under the present use.

A summary of the data gaps from the 1981 Registration Standard indicated that for the outdoor domestic use the following studies are required: hydrolysis, aerobic soil metabolism, terrestrial field dissipation, and adsorption/desorption. However, the hydrolysis study requirement has been waived, because the Merck Index, 11th edition, 1989, indicated that naphthalene is insoluble in water.

NAPHTHALENE USE PATTERN

Based on information from EFED files, major uses of naphthalene are :

- Clothes Moths
- Carpet Beetles

Minor uses are:

- Bulb storage protection and planting
- Fumigation of museum specimens

Repellent: bats, pigeons, squirrels, sparrows and rabbits in gardens.

There are no registered food uses

vp=4.92 X 10-2 mm Hg @ 20 degrees C (volatile at room temperature)

DYNAMAC
CORPORATION
Environmental Services

NAPHTHALENE

TASK 1: REVIEW AND EVALUATION OF INDIVIDUAL STUDIES

March 15, 1995

Initial Draft Report

Contract No. 68D20057

Submitted to:
Environmental Protection Agency
Arlington, VA 22202

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

NAPHTHALENE

Table of Contents

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Introduction

Scientific Studies

INTRODUCTION

Naphthalene is an insecticidal soil fumigant and moth repellent registered for use in greenhouse and domestic indoor/outdoor sites. Single active ingredient formulations include pelleted/tableted.

DATA EVALUATION RECORD

STUDY 1

CHEM 055801 Naphthalene §161-2

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 42335804
Blaushild, D. 1992. Aqueous photolytic degradation of ^{14}C naphthalene. CHMR Study No. 022/013/002/91; Landis Protocol No. 1612-91-058-01-28B-05. Unpublished study performed by Center for Hazardous Materials Research, Pittsburgh, PA; and submitted by Landis International, Inc., Valdosta, GA.

DIRECT REVIEW TIME = 45

REVIEWED BY: W. Martin TITLE: Staff Scientist

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M. Anderson Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

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TLL
6/24

SIGNATURE: J. A. Jordan
6/25/99

CONCLUSIONS:

Degradation - Photodegradation in Water

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

2. These data are of uncertain value and should not be used to predict the behavior of naphthalene in the environment.

3. This study is unacceptable for the following reason:

   the validity of the reported material balances cannot be confirmed; it appeared that a major error was committed when the data were calculated. The reported material balances are incomplete, declining from 114.77% of the applied at 24 hours posttreatment to 86.09% at 360 hours.

In addition, this study does not meet Subdivision N guidelines for the following reason:

> the only [^{14}C]compound identified during the study was naphthalene. [^{14}C]Residues in solution that were not soluble in diethyl ether (up to 96.37% of the residues in solution), [^{14}C]residues in diethyl ether extracts that were analyzed by HPLC but were not naphthalene (up to 12.5% of the residues in solution), and [^{14}C]residues trapped as volatiles (totalling up to 12.3% of the applied) were not characterized.

4. In order for this study to be reconsidered for use in meeting the aqueous photolysis data requirement, the volume of the collected sample and volume of the remaining bulk irradiated sample at each sampling interval must be reported; values included in calculated material balance data must be clearly labeled as to source (i.e., removed from the bulk solution at time x, removed from the bulk solution between time 0 and time x, remaining in solution at time x, volatilized from the solution during the immediate interval prior to time x, volatilized from the solution between time 0 and time x, total DPM); the "major" [^{14}C]degradate isolated by HPLC must be identified if present at >10% of the applied; [^{14}C]residues in the irradiated solution that were not soluble in diethyl ether must be characterized; and volatilized [^{14}C]residues trapped by the activated charcoal must be characterized.

METHODOLOGY:

[1-^{14}C]Naphthalene (radiochemical purity 97.68%, specific activity 10.1 mCi/mMol, Sigma Chemical), dissolved in 5 mL of 1,4-dioxane, was added to 495 mL of a filter-sterilized (0.2 um) 0.01 M phosphate buffer (pH 7) solution; the final concentration of naphthalene in solution was approximately 36 ppb, and the final concentration of the 1,4-dioxane cosolvent was 1% by volume. An aliquot (300 mL) of the treated buffer solution was transferred to a sterile 350-mL borosilicate glass photolysis vessel, and the vessel was then placed in a Heraeus Suntest CPS Photoionization Chamber (Figure 3). "Twenty one milliliter aliquots [of the remaining treated solution] were added to 6 separate sterilized 40 mL vials"; the vials were sealed with PTFE-lined screw caps, wrapped in aluminum foil, and incubated in the dark at 24.9 ± 0.1 C to serve as dark controls. The bulk treated solution was stirred constantly with a magnetic stirrer, and was continuously irradiated for 15 days using a xenon lamp; the light source had an emission spectrum between 280 and 760 nm and a mean light intensity of 30.1-30.6 x 10^{-3} watts/cm^2 that approximated that of natural sunlight in Phoenix, Arizona (season of year unspecified; Table 2 and Figure 4). The temperature of the irradiated solution was maintained at 24 C by circulating cooling water through a glass

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coil (6-mm id) inside the photolysis vessel; to monitor the temperature, a thermocouple was located in a cavity on one side of the vessel [page 12]. Prior to each sampling, the irradiated test vessel was flushed (12.5-16 mL/minute) with humidified air for 15 minutes; the air was exhausted sequentially through single tubes of toluene, 0.1 N NaOH, and 0.1 N H_2SO_4 trapping solutions, and an activated charcoal trap (6-14 mesh). Aliquots (volume not specified) of the bulk test solution were collected for analysis after 0, 5.5, 24, 48, 168, and 360 hours of irradiation; single vials of the dark control solutions were collected at each sampling interval. The trapping solutions were replaced at each sampling interval; the charcoal trap was changed after 24, 48, 168, and 360 hours. The irradiated and dark control solutions, and the trapping solutions were stored at approximately 4 C until analysis (length of storage not reported). At the termination of the study, the photolysis vessel and associated tubing and septa were rinsed with diethyl ether.

Aliquots of each sample were analyzed for total radioactivity using LSC. Additional aliquots (20 mL) were extracted three times with diethyl ether; the extracts were combined and concentrated under a nitrogen stream. The resulting residues were diluted with acetonitrile, and aliquots of the acetonitrile solution were analyzed by reverse-phase HPLC using a Waters Nova-Pak C-18 column eluted with water:acetonitrile (65:35 to 0:100 to 35:65, v:v; both solutions contained 0.1% trifluoroacetic acid). The column was equipped with UV (260 nm) and radioactive flow detection, and eluate fractions were collected and analyzed by LSC. HPLC column recoveries ranged from 77.7 to 110.6%.

Aliquots of the trapping solutions were analyzed for total radioactivity using LSC. The charcoal traps were extracted with carbon disulfide, and aliquots of the extract were analyzed using LSC. A subsample of the extracted charcoal was analyzed by LSC following combustion. Aliquots of the diethyl ether equipment rinses were analyzed using LSC.

DATA SUMMARY:

[1-^{14}C]Naphthalene (radiochemical purity 97.68%), at approximately 36 ppb, photodegraded with a registrant-calculated half-life of 4.5 days (assuming 12 hours of irradiation/day) in a sterile, aqueous pH 7 buffer solution that was continuously irradiated at 24 C for 15 days with a xenon lamp. The artificial light had an emission spectrum (280-760 nm) and a mean light intensity (30.1-30.6 x 10^{-3} watts/cm^2) that approximated that of natural sunlight in Phoenix, Arizona (season of year unspecified; Table 2 and Figure 4). In contrast, [^{14}C]naphthalene did not degrade in the dark control solutions incubated at 24.9 ± 0.1 C.

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In the irradiated solution, [^{14}C]naphthalene was 33.83-33.98 ng/mL through 5.5 hours posttreatment, 21.92 ng/mL at 24 hours, 9.27 ng/mL at 48 hours, and 0.14 ng/mL at 360 hours (Table 5). Uncharacterized extractable [^{14}C]residues totaled 5.12% of the radioactivity recovered in the irradiated solution immediately posttreatment, ≤1.32% at 5.5 and 24 hours, 11.62% at 48 hours, 6.00% at 168 hours, and 12.50% at 360 hours (reviewer-calculated from Table 8). Uncharacterized water-soluble [^{14}C]residues totaled 10.12% of the radioactivity recovered in the irradiated solution at 24 hours, 25.21% at 48 hours, and 96.37% at 360 hours (Table 8). The study author stated that "The major degradate appears to be hydroxylated naphthalene because of its water solubility" [page 11]; data supporting this conclusion were not provided. [^{14}C]Volatiles trapped in NaOH and H_2SO_4 solutions totaled 0.05% of the applied at 360 hours posttreatment, and [^{14}C]residues trapped in an activated charcoal trap totaled 11.0 and 12.3% after 24 and 360 hours, respectively (reviewer-calculated from Table 4). The study author reported that material balances were 100-114.77% of the applied through 24 hours, 91.18% at 48 hours, and 86.09% at 360 hours (Table 4).

In the dark controls, [^{14}C]naphthalene comprised 93.85-100% of the radioactivity recovered from HPLC at all sampling intervals (Table 7). Material balances were reported to be 91.16%-124.03% of the applied through 528 hours posttreatment, with the exception of 64.18% at 360 hours.

COMMENTS:

1. The material balances reported by the study author in Table 4 declined from 100-114.77% of the applied at 0-24 hours posttreatment to 86.09% at 360 hours. However, the validity of these numbers is uncertain because they cannot be reproduced. From the data presented in Table 4, it appears that when calculating the material balance for the system at time x, the study author included <u>all</u> radioactivity that had been previously removed from the solution by sampling (the steadily increasing "DPM test solution sample") and <u>none</u> of the radioactivity that had volatilized from the sample prior to the immediate sampling interval. This mistake may be confirmed by the data in Table 5, from which it can be determined that, of the total radioactivity isolated in the test vessel at 0 hours, 85.38% remained in the vessel at 360 hours; this value is identical to the "% initially applied" reported in Table 4 for the 360-hour sampling interval if one excludes the volatiles collected between 168 and 360 hours. The same correlation can be made for all sampling intervals. Unfortunately, since the volume of the samples collected from the irradiated bulk sample at each sampling interval was not reported, the data in Tables 4 and 5 cannot be converted to similar units for direct comparison.

   Also, the study author stated that the tubing and photolysis vessel were rinsed with diethyl ether after the study, and aliquots of the

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rinses were analyzed by LSC to "determine complete material balance". No further reference is made to the diethyl ether rinse, and it could not be determined if the results were included when the material balance was calculated.

2. The only [^{14}C]compound identified during the study was naphthalene. [^{14}C]Residues in solution that were not soluble in diethyl ether (up to 96.37% of the residues in solution), [^{14}C]residues in the diethyl ether extracts that were analyzed by HPLC but were not naphthalene (up to 12.50% of the residues in solution), and [^{14}C]residues trapped as volatiles (totalling up to 12.3% of the applied) were not characterized (data calculated from Tables 4 and 8). The study author stated that "The major degradate appears to be hydroxylated naphthalene because of its water solubility" [page 11], but hydroxylated naphthalene was not quantified, and data supporting this conclusion were not provided.

3. The data in Table 5 are incorrectly described. Based on the extraction efficiency data in Table 8, data (for which no units are reported) in the columns labeled "HPLC Data, Percent Naphthalene" and "HPLC Data, Percent Unknown" are not in terms of "% recovered from HPLC" but in terms of "% recovered in the irradiated solution" at individual sampling intervals. The "HPLC Data, Percent Unknown" value includes all residues in solution that are not naphthalene, both those that were not characterized as naphthalene by HPLC and those that were not extracted from the buffer solution by diethyl ether and were therefore never characterized.

4. The study author stated that "In the preliminary experiment, it was found that if the test substance was purged continuously (at 20 ml/min. air flow), all the naphthalene was purged from the solution...The loss of ^{14}C [from the irradiated solution in the definitive experiment] is assumed to be due to volatilization of naphthalene when the system is purged. This assumption is supported by the fact that all of the ^{14}C in the control samples was accounted for [pages 18-19]." This statement was interpreted to mean that the study author believed that naphthalene would not have volatilized from the irradiated solution if there had been no attempt to collect headspace gases. If this hypothesis originated before the definitive experiment was initiated, a static rather than flow-through air sampling system should have been employed.

5. At 168 hours, only 17% of the radioactivity adsorbed by the charcoal trap was extracted with carbon disulfide. This is contrary to the outcome of extractions at other sampling intervals, when 100% of the radioactivity was extracted from the charcoal traps. No explanation was provided.

6. The xenon lamp was compared to sunlight at Phoenix, Arizona (Figure 4). The season of the year the sunlight was analyzed and the total irradiance were not reported for the purposes of comparison.

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7. The study author stated that the solubility of naphthalene in water at 25 C was "estimated to be" 32 ppb, and in pH 7 water containing 1% dioxane was shown to be 46 ppb [page 12]. According to information provided in the study protocol, the published solubility of naphthalene in water is 30 mg/L. No explanation was provided for the significant difference between the "estimated" and published values.

8. The adsorption spectrum of naphthalene in a pH 7 buffer solution containing 1% dioxane was not provided.

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Page_____ is not included in this copy.

Pages 12 through 23 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.

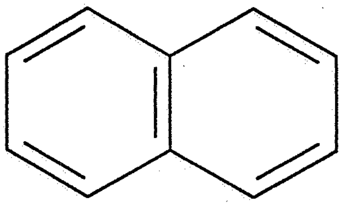
REFERENCES

The following study was reviewed:

Blaushild, D. 1992. Aqueous photolytic degradation of ^{14}C naphthalene. CHMR Study No. 022/013/002/91; Landis Protocol No. 1612-91-058-01-28B-05. Unpublished study performed by Center for Hazardous Materials Research, Pittsburgh, PA; and submitted by Landis International, Inc., Valdosta, GA. (42335804)

APPENDIX

NAPHTHALENE



Naphthalene

(89)

received 9/19/94

DYNAMAC CORPORATION
Environmental Services

NAPHTHALENE

Naphthalene

TASK 1: REVIEW AND EVALUATION OF INDIVIDUAL STUDIES

September 30, 1994

2 aerobic soil metabolism

Initial Draft Report

Contract No. 68D20057

Submitted to:
Environmental Protection Agency
Arlington, VA 22202

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

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NAPHTHALENE

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Introduction

Scientific Studies

INTRODUCTION

Naphthalene is an insecticidal soil fumigant and moth repellent registered for use in greenhouse and domestic indoor/outdoor sites. Single active ingredient formulations include pelleted/tableted.

DATA EVALUATION RECORD

STUDY 1

CHEM 055801 Naphthalene §162-1

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 00156557
Martinson, J.P. 1985. Biodegradation of naphthalene in clay loam soil. Biospherics Project No. 85E-413. Unpublished study performed by Biospherics Incorporated, Rockville, MD, and submitted by W.R. Landis Associates, Inc., Valdosta, GA.

DIRECT REVIEW TIME = 29

REVIEWED BY: K. Ferguson TITLE: Task Leader

EDITED BY: C. Cooke TITLE: Staff Scientist
M. Anderson Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

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TEL: 301-417-9800

APPROVED BY: L. Liu
TITLE: Chemist
ORG: EFGWB/EFED/OPP
TEL: 703-305-5372

SIGNATURE: John H. Jordan 6/7/99
Nhung L Nguyen 6/8/99

CONCLUSIONS:

Metabolism - Aerobic Soil

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

2. These data are of uncertain value and should not be used to predict the behavior of naphthalene and its degradates in the environment.

3. This study is unacceptable for the following reasons:

   The experimental design was inappropriate to establish the pattern of dissipation of naphthalene in aerobic soil. Prior to the termination of the study, only the scintillation cocktail "trapping solutions" were analyzed. The soil was not sampled until the study was terminated, at 15 days posttreatment for Trial 1 and at 32 days for Trial 2.

The material balance was incomplete; only 39.0-47.9% of the radioactivity that was theoretically applied to the soil was recovered at the termination of the experiments.

4. Because the experimental design was inappropriate and the material balance was incomplete, the problems with this study cannot be resolved with the submission of additional data. Therefore, a new study is required.

METHODOLOGY:

Portions (50 g dry weight) of sieved (2 mm) clay loam soil (21.8% sand, 50.4% silt, 27.8% clay, 1.6% organic matter, pH 7.6, CEC 20 meq/100 g) were weighed into two 250-mL glass Erlenmeyer flasks containing [1-^{14}C]naphthalene (radiochemical purity 97.8%, specific activity 6.12 mCi/mMol, Pathfinder Laboratories) dissolved in an unspecified solvent. The theoretical application rate was 10.0 ppm, and the moisture content of the soil was 70% of field capacity. A third flask of soil was prepared as described using [^{14}C]glucose rather than [^{14}C]naphthalene. The soil was "shaken gently", then the flasks were covered with aluminum foil and sealed with a screw cap that was fitted with inlet and outlet ports. The outlet port was attached to a volatile trapping system consisting of two tubes of Maxifluor scintillation cocktail and two tubes of Harvey 14-C scintillation cocktail. The flasks were flushed with compressed air (time period unspecified) each day through 15 days posttreatment; the trapping solutions were collected and replaced after each flushing. The soil samples were analyzed at 15 days posttreatment. Soil temperatures during incubation were not reported.

The experiment was repeated as described, except that the flasks were flushed with compressed air each day through 9 days posttreatment, then were flushed on 11, 12, 13, 25, 28, and 32 days. The soil samples were analyzed at 32 days posttreatment.

Subsamples of the test soil were analyzed for total radioactivity using LSC following combustion. Additional subsamples were extracted with methanol:water (9:1) by refluxing for 4 hours. The extract was decanted, then "refrigerated to sediment solids." Aliquots of the refrigerated extracts were analyzed, without concentration, for total radioactivity using LSC and for specific compounds by HPLC using an Applied Science ODS column eluted with acetonitrile:water (1:1); the column was equipped with UV (221 nm) detection, and aliquots of the eluate were collected and analyzed using LSC. The extracted soil was dried at room temperature (length of drying not specified), then analyzed for unextracted [^{14}C]residues using LSC following combustion.

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REVIEW EVALUATION SHEET

EFGWB REVIEWER NAME:

CHEMICAL:

DP BARCODE:

MRID#:

EFGWB #:

PC CODE:

STUDY TYPE: Guideline #

DYNAMAC REVIEWER(S):

DYNAMAC QA/QC PERSON:

1) Please rate on a scale of 5 (= <u>excellent</u>, requiring no changes or only minor "cosmetic" changes) to 1 (= <u>unacceptable</u>, requiring major rewrite because of misinterpretation of studies) by circling the appropriate number below. Note that these ratings refer to technical changes of meaning or content, not style.

   5 = minor changes (e.g., changes in conclusions to reflect in-house knowledge not available to Dynamac reviewer).

   4 = changes in conclusions (e.g., decision of EFGWB reviewer to use information in the study even though the study has been deemed unacceptable to fulfill guidelines criteria).

   3 = changes in conclusions and discussion (e.g., change in discussion emphasis).

   2 = changes in conclusions, discussion and methods (e.g., substantial changes throughout entire DER).

   1 = complete rereview (original DER unusable -- note that the EFGWB reviewer has the option of returning a marked-up copy for Dynamac redo in this case)

<u>OPTIONAL (for EFGWB internal use only)</u>

2) The errors appeared to be due to:

   a) lack of editorial quality control (e.g. many typos)
   b) lack of scientific quality control (e.g. data misinterpreted)
   c) random factors (e.g. missing page)
   d) other (explain)

3) The major errors were (give details):

The scintillation cocktail trapping solutions were analyzed for total radioactivity using LSC.

DATA SUMMARY:

In a 15-day experiment, [1-^{14}C]naphthalene (radiochemical purity 97.8%), at a theoretical application rate of 10.0 ppm, dissipated with an observed half-life of <15 days in clay loam soil that was incubated at 70% of field moisture capacity (temperature unspecified) in the dark in flasks that were flushed with compressed air (time period unspecified) each day. In the soil at 15 days posttreatment (the only interval at which soil was analyzed), [^{14}C]naphthalene was 2.4-6.4% of the theoretical application, unidentified [^{14}C]compounds extracted from the soil totaled 2.0-2.5%, unextracted soil [^{14}C]residues totaled 13.5-14.0%, $^{14}CO_2$ totaled 12.7-19.0%, and uncharacterized volatile [^{14}C]residues totaled 0.3-0.5% (Tables 1 and 3). Material balances at 15 days posttreatment were 44.6-47.9% of the theoretical application.

In a 32-day experiment, [1-^{14}C]naphthalene (radiochemical purity 97.8%), at a theoretical application rate of 10.0 ppm, dissipated with an observed half-life of <32 days in clay loam soil that was incubated at 70% of field moisture capacity (temperature unspecified) in the dark in flasks that were flushed with compressed air (time period unspecified) each day through 9 days posttreatment, then were flushed on 11, 12, 13, 25, 28, and 32 days. In the soil at 32 days posttreatment (the only interval at which soil was analyzed), [^{14}C]naphthalene was 13.5-13.8% of the theoretical application, unidentified [^{14}C]compounds extracted from the soil totaled 1.3-1.8%, unextracted soil [^{14}C]residues totaled 15.3-16.9%, $^{14}CO_2$ totaled 7.0-14.0%, and uncharacterized volatile [^{14}C]residues totaled $\leq$0.2% (Tables 2 and 3). Material balances at 32 days posttreatment were 39.0-43.9% of the theoretical application.

COMMENTS:

1. The experimental design was inappropriate to establish the pattern of dissipation of naphthalene in aerobic soil; only the evolution of volatiles was measured on a regular basis. The soil was not sampled until the study was terminated (at 15 days posttreatment for Trial 1 and at 32 days for Trial 2), at which time naphthalene was only $\leq$14% of the radioactivity that was theoretically applied to the soil.

2. The material balance was incomplete; only 39.0-47.9% of the radioactivity that was theoretically applied to the soil was recovered at the termination of the experiments. The study author suggested that there may have been losses of $^{14}CO_2$, since the material balances in soil treated with [^{14}C]glucose were only 62.5-66.1% of the theoretical application. The study author also stated that "Although our references recommended toluene based traps for volatile

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chemicals and Maxifluor is trimethylbenzene based, a solvent may not be the proper means to contain a highly volatile material like naphthalene." During the two experiments, [^{14}C]volatiles other than $^{14}CO_2$ totaled $\leq$0.5% of the theoretical application. In contrast, in a related aerobic soil metabolism study in which polyurethane foam plugs were used to trap volatiles (MRID 00160093, Study 2 of this submission), volatilized organic [^{14}C]residues were 41.1-48.1% of the theoretical application at 2 days posttreatment and 43.7-51.0% at 30 days; $\geq$96.6% of the recovered organic volatiles were identified as [^{14}C]naphthalene.

3. Soil temperatures during incubation were not reported.

4. The study author stated that aliquots of the [^{14}C]naphthalene solutions that were analyzed using LSC "served as a dose check." It was not specified whether it was the stock or treatment solutions that were analyzed, and it was not specified when the solutions were analyzed in relation to the time of treatment.

5. The difference between experiments 1 and 2 was that in experiment 1, the [^{14}C]naphthalene was diluted with unlabeled naphthalene prior to use. Although in both experiments the soils were treated with naphthalene at 10.0 ppm, in experiment 1 the application was 13052 dpm/g, and in experiment 2 the application was 986000 dpm/g.

6. The study author refers to "Figure 1" when describing the test system [page 000004]. However, Figure 1 illustrates the structure of naphthalene; no illustration of the test system was included with the study.

7. The study author stated that the "control" soil that was treated with [^{14}C]glucose and incubated with the treated soil was intended to establish the microbial viability of the soil.

8. Units for the cation exchange capacity for the test soil were not reported. The Dynamac reviewer assumed the units to be milliequivalents per 100 grams of soil (meq/100 g).

Page_____ is not included in this copy.

Pages 35 through 43 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.

DATA EVALUATION RECORD

STUDY 2

CHEM 055801 Naphthalene §162-1

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 00160093
Tai, S. 1986. Naphthalene aerobic soil metabolism in silty clay loam soil. Biospherics Project No. 85E-413. Unpublished study performed by Biospherics Incorporated, Rockville, MD, and submitted by W.R. Landis Associates, Inc., Valdosta, GA.

DIRECT REVIEW TIME = 37

REVIEWED BY: K. Ferguson TITLE: Task Leader

EDITED BY: C. Cooke TITLE: Staff Scientist
M. Anderson Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

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TITLE: Chemist
ORG: EFGWB/EFED/OPP
TEL: 703-305-5372

SIGNATURE: John H. Jordan 6/7/99

CONCLUSIONS:

Metabolism - Aerobic Soil

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

2. These data are of uncertain value and should not be used to predict the behavior of naphthalene and its degradates in the environment.

3. This study is unacceptable for the following reasons:

   The material balance was incomplete; only 55.3-69.0% of the radioactivity that was theoretically applied to the soil was recovered at 2 through 30 days posttreatment.

The application rate for samples collected between 2 and 30 days posttreatment was never confirmed; the "immediate posttreatment" samples were not treated until 2 days after the treatment of the other samples.

The soil was not sampled frequently enough to accurately establish the pattern of dissipation of naphthalene. By the first sampling interval (2 days posttreatment), only 6.4-9.9% of the radioactivity that was theoretically applied to the soil was identified as naphthalene.

It was not demonstrated that naphthalene was stable in the sample extracts during storage.

4. Because the material balance was incomplete, the application rate was not confirmed, and the soil was not sampled frequently enough to accurately establish the pattern of dissipation of naphthalene, the problems with this study cannot be resolved with the submission of additional data. Therefore, a new study is required.

METHODOLOGY:

Portions (10 g dry weight) of sieved (2 mm) silty clay loam soil (15.4% sand, 53.0% silt, 31.6% clay, 1.5% organic matter, pH 7.6, CEC 20.96 meq/100 g) were weighed into 14 glass incubation jars containing a mixture of distilled, deionized water and [1-^{14}C]naphthalene (radiochemical purity 99.7%, specific activity 6.12 mCi/mMol, Pathfinder Laboratories), dissolved in 2-propanol; the theoretical application rate was 10.4 ppm, and the final moisture content of the soil was 50.3% (5 g water/10 g dry soil). The soil was swirled to "mix thoroughly", then the samples were placed inside one of two test chambers, each of which consisted of a foil-wrapped 3-L glass jar lined with two thin sheets of polyurethane foam (Figure 2). The jar inlet and outlet glass tubes were blocked with polyurethane foam plugs for trapping volatiles, and the jar was sealed with a hard plastic wide-mouth screw cap and attached to a continuous air-flow system. Humidified, CO_2-free air was drawn (7.5-16.7 mL/minute) through individual test chambers, then through the polyurethane foam plugs and a 1 N NaOH trapping solution (one tube). The samples were incubated in a fume hood at 20.5-25.5 C; soil temperatures were monitored continuously using a probe inserted in a flask of soil in the hood. Duplicate jars of soil, one from each test chamber, were collected for analysis at 2, 5, 7, 14, and 30 days posttreatment. Also, at 2 days posttreatment, additional untreated soil was treated as previously described and analyzed immediately to serve as the 0-day sample. The foam sheets lining the test chambers were removed and replaced at 2, 5, and 7 days posttreatment; the polyurethane foam plugs were replaced at 7 days; and the NaOH solutions at each chamber outlet were replaced at 2, 5, 7, and 14 days. The sheets, plugs, and NaOH solutions were collected at 30 days.

The soil, foam sheets, and foam plugs were extracted immediately upon sampling, and the extracts were stored frozen until analysis. Soil samples collected through 7 days posttreatment were sequentially extracted with acetonitrile by shaking for 30 minutes and with methanol:water (9:1) by refluxing for 4 hours; after each extraction, the extract was decanted through a vacuum filter funnel. Soil samples collected at 14 and 30 days were only extracted with methanol:water (9:1) by refluxing as described. Aliquots of the individual extracts were analyzed for total radioactivity using LSC, then the extracts from each sample were combined. The combined extracts were analyzed, without concentration, for specific compounds by HPLC using a Hypersil ODS C-18 column eluted with acetonitrile:water (6:4); the column was equipped with UV (221 nm) detection, and aliquots of the eluate were collected and analyzed using LSC. [^{14}C]Residues were identified by comparison to the retention time of unlabeled naphthalene. The extracted soil was air-dried for approximately 5 minutes to allow the methanol to evaporate, then was analyzed for unextracted [^{14}C]residues using LSC following combustion.

The polyurethane foam liners and plugs were extracted with acetonitrile by compressing the foam several times in the solvent. Aliquots of the extracts were analyzed for total radioactivity using LSC; additional aliquots were analyzed using HPLC as described.

Aliquots of the NaOH trapping solutions were analyzed for total radioactivity using LSC; [^{14}C]residues in the NaOH trapping solution were identified as CO_2 by precipitation with barium chloride.

DATA SUMMARY:

[1-^{14}C]Naphthalene (radiochemical purity 99.7%), applied to silty clay loam soil at a theoretical application rate of 10.4 ppm, dissipated from the soil with an observed half-life of <2 days when incubated in a continuous air-flow system in the dark at a soil moisture content of 50.3% and a temperature of 20.5-25.5 C. In the soil, [^{14}C]naphthalene was 83.0-86.6% of the theoretical application immediately posttreatment, 6.4-9.9% at 2 days, 1.3-2.6% at 5 and 7 days, and $\leq$1.0% at 14 and 30 days (Table 3). Unidentified [^{14}C]compounds extracted from the soil totaled 1.4-1.9% of the theoretical application immediately posttreatment, and $\leq$1.2% at all later sampling intervals. Unextracted soil [^{14}C]residues ranged from 3.7 to 9.0% of the applied at 2 through 30 days, with no clear pattern of increase or decline. > The majority of the [^{14}C]residues applied to the soil were lost by volatilization. < Volatilized organic [^{14}C]residues, of which $\geq$96.6% were identified as [^{14}C]naphthalene, increased to 41.1-48.1% of the theoretical application at 2 days posttreatment and 43.7-51.0% at 30 days; $^{14}CO_2$ totaled 1.5-2.1% at 2 days and 8.2-13.3% at 30 days (Tables 1 and 5). Material balances were 85.1-88.2% of the theoretical application immediately posttreatment, and ranged from 55.3 to 69.0% at 2 through 30 days.

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COMMENTS:

1. The material balance was incomplete. The study author could account for only 85.1-88.2% of the theoretical application "immediately posttreatment", and for only 55.3-69.0% at 2 through 30 days. It was suggested that the material losses were due to the extreme volatility of naphthalene; i.e., "in the time required to dose the soils and assemble the test apparatus, a significant amount of radioactivity was lost to the atmosphere" [page 12].

   Also, the application rate for samples collected between 2 and 30 days posttreatment was never confirmed; the "immediate posttreatment" samples, in fact, were not treated at the same time as other samples, but were treated and extracted at the 2-day posttreatment sampling interval.

2. The soil was not sampled frequently enough to accurately establish the pattern of dissipation of naphthalene. By the first sampling interval (2 days posttreatment), only 12.1-19.4% of the radioactivity theoretically applied to the soil remained in the soil, and the concentration of naphthalene in the soil was only 6.4-9.9%.

3. The length of frozen storage of the extracts was not reported. The study author did not demonstrate that naphthalene was stable (i.e., would not volatilize) in the sample extracts during storage.

4. The soil was maintained at 50% by weight (5 g water/10 g dry soil) rather than at 75% of field moisture capacity (34.7% by weight). In addition, the study author reported that the moisture loss during the 30-day study was 7% [page 7].

5. Five untreated ("control") soil samples were incubated in a third test chamber as described for the treated soil. The apparent purpose of the control soil was to establish background counts for combustion, extraction, and HPLC analyses. However, "due to large variations on the control soil radioactivity, the control soil sample was not used to provide background counts..." [page 15].

6. The 7-day samples were refluxed overnight, rather than for 4 hours.

7. To determine microbial viability, portions of the control soil were analyzed prior to the initiation of the experiment and at 7 and 30 days posttreatment. In the control soil, numbers of bacteria ranged from 23.6 x 10^6 to 69 x 10^6 colony forming units/g soil and numbers of fungi ranged from 20 x 10^4 to 27 x 10^4 colony forming units/g soil.

8. The study author stated that the test system was made with glass, Teflon, and hard plastic to prevent as much adsorption as possible.

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9. Units for the cation exchange capacity for the test soil were not reported. The Dynamac reviewer assumed the units to be milliequivalents per 100 grams of soil (meq/100 g).

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REFERENCES

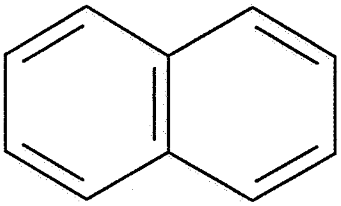
The following studies were reviewed:

Martinson, J.P. 1985. Biodegradation of naphthalene in clay loam soil. Biospherics Project No. 85E-413. Unpublished study performed by Biospherics Incorporated, Rockville, MD, and submitted by W.R. Landis Associates, Inc., Valdosta, GA. (00156557)

Tai, S. 1986. Naphthalene aerobic soil metabolism in silty clay loam soil. Biospherics Project No. 85E-413. Unpublished study performed by Biospherics Incorporated, Rockville, MD, and submitted by W.R. Landis Associates, Inc., Valdosta, GA. (00160093)

APPENDIX

NAPHTHALENE



Naphthalene

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