May 5/19/88

DISCUSSION OF INDIVIDUAL STUDIES

NALED

Table of Contents

INTRODUCTION

Naled is a nonsystemic insecticide-acaricide registered for use on terrestrial food crop (field, vegetable, and orchard crops), terrestrial nonfood (livestock and poultry and their surroundings), greenhouse food crop, greenhouse nonfood, domestic outdoor (urban and rural outdoor areas for mosquito control), aquatic food crop, aquatic nonfood, forestry, and indoor (agricultural, domestic, medical, and commercial establishments), use sites. Of the naled applied in the United States, the major use sites are: fruit, nut, vegetable, and field crops (50%); mosquito control (35%); dog flea collars (10%); and livestock (5%). Single active ingredient formulations consist of 3-6% D, 7-15% Impr, 2-7.2 lb/gal and 2.5-58% EC, 12.6 lb/gal and 1.2-20% SC/L, and 1.26-3.34 lb/gal and 0.66-15% RTU. Application rates range from 0.01-4 lb/A, 4.5-6.75 oz/50,000 ft^3 (7.2 lb/gal EC) and 16.67-50 fl. oz/50,000 ft^3 (10% RTU) (greenhouse fumigation), 20 fl. oz/5000 ft^2 (1% RTU), and 800 ppm (sewage). Naled is applied using aircraft and ground equipment including mist sprayers and foggers. Applicators need not be certified to apply naled.

DATA EVALUATION RECORD

NALED STUDY 1

CHEM 034401 Naled

BRANCH EAB

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40034902
Chen, Y.S. 1986a. Hydrolysis products of (ethyl-1-^{14}C)naled in buffer solutions. Laboratory Project Identification 8602408. Unpublished study prepared and submitted by Chevron Chemical Company, Richmond, CA.

SUBST. CLASS = S

DIRECT RVW TIME = 3

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

EDITED BY: TITLE: Task Leader

APPROVED BY: TITLE: Project Manager

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APPROVED BY: S. C. Termes
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May 19th, 1988

SIGNATURE:

CONCLUSIONS:

Degradation - Hydrolysis

This study is unacceptable as presented. Only thin layer chromatography (TLC) was used to identify hydrolysis products, but no actual chromatographic data were included in the report. Identity of degradates should have been confirmed by another methodology besides TLC. Two apparently different unknowns were detected but no attempts were made to characterize these unknowns. The Agency is concerned about the contribution of naled to the concentration of DDVP (that is, dimethyl 0-(2,2-dichlorovinyl) phosphate) in the environment and, therefore, attempts to identify these degradation products (even though they were present at less than 10%) should have been made as DDVP is a degradate of concern.

SUMMARY OF DATA BY REVIEWER:

Although the study is unacceptable for the reasons pointed out above, the reported results indicate that the hydrolysis of naled is a pH-dependent process, with the rate of hydrolysis increasing with increasing pH. The predominant process at pH 9 is demethylation of naled to yield 1,2-dibromo-2,2-dichloroethyl methyl phosphate (desmethyl naled); bromodichloro acetaldehyde (BDCA) (which does not contain the phosphate group) and "unknown 2" are also present in small amounts. At pH 5, the main hydrolysis product in BDCA (which implies that cleavage of the P-O bond and elimination of the C_2-Br is the predominant mechanism) with desmethyl naled and "unknown 1" detected as minor components. At pH 7, however, both BDCA and desmethyl naled (the former in higher amounts than the latter) are present together with small amounts of "unknown 1" and "unknown 2". Thus, at pH 7 both mechanisms appear to predominate. Since the test material was radiolabelled in the C-1 position, the structures of "unknown 1" and "unknown 2" should also contain the C-1.

The reported half-lives were 1.6 hours at pH 9, 15.4 hours at pH 7, and 96 hours at pH 5.

DISCUSSION:

1. TLC data were reported in terms of percent of the recovered from the TLC plates. The registrant stated that total recoveries for all-samples were 97.1-99.8%.

2. Recovery values from fortified samples and method detection limits were not reported.

3. It should be noted that TLC analyses were conducted using silica gel TLC plates. According to supplemental information provided in the photodegradation in air study, naled is unstable on silica gel and TLC analyses using silica gel-coated plates must be completed quickly.

4. Samples were not analyzed for the degradate DDVP. EPA is concerned about human DDVP exposure and the contribution of naled to DDVP concentrations in the environment. Therefore, attempts to identify "unknown 1" and "unknown 2" should have been made to confirm/rule out the contribution of hydrolysis to DDVP formation.

5. At sampling time, 10 ml of test solution were pipetted and acidified immediately with 0.5 ml of concentrated HCl; then, 10 ml of the acidified solution was analysed for naled by TLC (hydrolysis products were also identified, by TLC, after acidification). What evidence does the registrant have that such a drastic change in pH did not affect the nature of the degradates present prior to acidification? What was the purpose of such a drastic change in pH? What was the final pH?

MATERIALS AND METHODS

MATERIALS AND METHODS:

1-[^{14}C]Naled (radiochemical purity 99.4%, specific activity 118,340 dpm/ug, New England Nuclear) was added at 9.4 ppm to sterile aqueous buffered solutions adjusted to pH 5, 7, and 9. The solutions were incubated in brown glass bottles at 25 ± 1°C. The solutions were sampled at intervals up to 336 hours posttreatment.

Solutions were analyzed for total radioactivity by LSC. Solutions were acidified with concentrated hydrochloric acid and analyzed by TLC with nonradiolabeled reference standards on silica gel plates developed with 1-butanol:glacial acetic acid:water (6:1:1). Radioactive areas were located using autoradiography, identified by comparison to nonradiolabeled standards, and quantified by LSC. Nonradiolabeled reference standards were identified by UV light or spraying with silver nitrate. The identification of degradates was confirmed by TLC using the solvent systems chloroform:glacial acetic acid (4:1) and ether.

Naled

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_____ Identity of product inert impurities.

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_____ Description of product 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

__X__ FIFRA registration data.

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

RESULTS AND DISCUSSION

Naled was readily hydrolyzed in basic solution compared with acidic solution. The half-lines for hydrolysis are approximately 1.6 hours, 15.4 hours and 96 hours in pH=9, 7 and 5, respectively (Figures 1-3). These values are consistent with previous studies (1, 2). The hydrolysis products are also straightforward. At pH=9, the major product is desmethyl naled. At pH=5, the major product is BDCA. And at pH=7, both desmethyl naled and BDCA are present in significant amount (Tables I-III). Therefore, demethylation is a dominant reaction in basic solution and de-1,2-dibromo-2,2-dichloro-ethylation is dominant reaction in acidic solution. The pH values at end of each hydrolysis did not change more than 0.1 unit for all solutions. The total recovery for all samples was essentially quantititive (97.1% - 99.8%).

Two minor products were also observed. Unknowing I probably derived from BDCA (Table I) and unknowing 2 probably drived from desmethyl naled (Table III). However, they presented no more than 5% when approximately 90% of naled was hydrolyzed. No attempts were made to identify these two minor products.

REFERENCES

1. Y.S. Chen, Photodegradation of (Ethyl-1-^{14}C) Naled in Water by Long Wavelength UV Light, Chevron Chemical Company Report, October 13, 1986.

2. G. H. Fujie, Naled Hydrolysis in Aqueous Solutions, Chevron Chemical Company Report, October 13, 1986.

3. R. Jentzsch and G. W. Fischer (1978), Journal f. prakt. chemic. Band 320, Heft 4, S. 634-646.

Notebook Reference: 8753

PERTINENT DATA TABLES AND FIGURES

TABLE I

HYDROLYSIS PRODUCTS OF NALED IN pH=5 SOLUTION

Time Interval (Hour)	Percent Distribution			
	Naled	BDCA	Desmethyl Naled	Unknown 1
0	100	-	-	-
6	98.6	1.4	0	0
24	86.7	10.7	0	2.6
48	67.4	28.4	0.3	3.8
72	61.1	34.8	0.3	4.8
96	46.4	48.2	0.7	4.7
168	27.2	68.4	0.8	3.6
336	10.8	86.5	1.3	1.4

TABLE II

HYDROLYSIS PRODUCTS OF NALED IN pH=7 SOLUTION

Time Interval (Hour)	Percent Distribution				
	Naled	BDCA	Desmethyl Naled	Unknown 1	Unknown 2
0	100	-	-	-	-
6	70.9	25.3	3.7	0	0.1
24	33.8	52.2	9.8	2.2	2.0
48	11.0	68.5	13.0	2.4	5.1

TABLE III

HYDROLYSIS PRODUCTS OF NALED IN pH=9 SOLUTION

Time Interval (Hour)	Percent Distribution			
	Naled	BDCA	Desmethyl Naled	Unknown 2
0	100	-	-	-
2	38.7	5.3	54.9	1.0
4	9.2	4.4	84.0	2.4
6	2.2	3.0	91.7	3.1

DATA EVALUATION RECORD

NALED STUDY 2

CHEM 034401 Naled

BRANCH EAB

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40034903
Chen, Y.S. 1986b. Photodegradation of [ethyl-1-^{14}C]naled in water by long wavelength UV light. Laboratory Project Identification 8613174. Prepared and submitted by Chevron Chemical Co., Richmond, CA.

SUBST. CLASS = S

DIRECT RVW TIME = 2

REVIEWED BY: L. Lewis, T. Colvin-Snyder TITLE: Staff Scientists

EDITED BY: TITLE: Task Leader

APPROVED BY: TITLE: Program Manager

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APPROVED BY: S. C. Termes
TITLE: Chemist
ORG: EAB/HED/OPP
TEL: 557-1981

May 19th, 1988

SIGNATURE:

CONCLUSIONS:

Degradation - Photodegradation in Water

This study is unacceptable. The submitted study only provides information on the photodegration of naled in the 320-380 nm region (long wavelength UV light of an artificial light source and not in the complete wavelength range of sunlight (or artificial light source simulating sunlight), as required in the Subdivision N Guidelines. No electronic absorption spectrum of naled under the experimental conditions (solvent, buffer system, etc.) was presented. The radiolabelled naled was introduced in the buffer solutions as an acetone solution (1 ml acetone to 400 ml buffer solution). Acetone is a well-known photosensitizer; however, no comments were made in the study of any possible effects of acetone on the photodegradation of naled. The only methodology to identify degradation products was thin layer chromatography.

SUMMARY OF DATA BY REVIEWER:

Ethyl-labeled [^{14}C]naled (radiochemical purity >99%, specific activity 15,560 dpm/ug), at 25 ppm, degraded with a half-life of 24-48 hours (calculated half-life approximately 30 hours) in a pH 5 buffer solution that was irradiated with a Pyrex-filtered black light fluorescent lamp (320-380 nm, peaking at 365 nm; intensity 2 x 10^3 uW/cm^2) and incubated at 25 C. The major degradates were bromodichloroacetaldehyde (BDCA, maximum 38.5% of the recovered at 72 hours), 2,2-dichlorovinyl dimethyl phosphate (DDVP, 11.9% at 48 hours), and acetic acid (15.7% at 96 hours). Chloroacetic acid (CAA) plus dichloroacetic acid (DCAA) totaled a maximum of 13.4% of the recovered at 96 hours, and unidentified [^{14}C]residues were $\leq$7.4% of the recovered at every sampling interval. $^{14}CO_2$ totaled 8.2% of the recovered radioactivity after 96 hours of irradiation; no other volatile products were trapped. In the dark control, the half-life of naled was approximately 96 hours. During the study, the material balance of the samples ranged from 98.4-100.4% of the applied radioactivity.

DISCUSSION:

1. Artificial light source - The wavelength region provided by the artificial light source was 320-380 nm (long wavelength UV region). Therefore, the use of this light source was inadequate because it did not expand the spectrum of sunlight.

2. The electronic absorption spectrum of naled (and its degradates) in the UV-VIS region and under the same experimental conditions (solvent, buffer system, etc.) was not included in the report.

3. In the characterization of photoproducts at selected intervals, samples were extracted with ethyl acetate after adjustment to pH 1 (with 6N HCl) and then followed by identification by TLC. This change in pH is a drastic one. What evidences does the author have that such a drastic pH change does not result in further reactions of the degradates present at sampling time?

4. The only methodology used to identify photodegradation products was TLC. However, no TLC data (for example, R_f values) were provided.

5. The degradation of naled in the dark control was not detailed; the registrant provided only the calculated half-life of the parent and a list of the degradation products. However, this study was conducted under identical conditions as the hydrolysis study (Chen, 40034902), so data from that study could have been used in interpreting the results of this photodegradation study in water, provided the hydrolysis study was acceptable.

MATERIALS AND METHODS

MATERIALS AND METHODS:

A phosphate buffer solution (pH 5, HPLC grade water) was treated with ethyl-labeled [^{14}C]naled (radiochemical purity >99%, specific activity 15,560 dpm/ug, New England Nuclear) at 25 ppm, then filter-sterilized. The solution was transferred to a Pyrex photoreaction chamber and irradiated with a black light fluorescent lamp (F15T8/B1, GE, wavelengths between 320-380 nm, intensity of 2 x 10^3 uW/cm^2 peaking at 365 nm) for 96 hours. The temperature of the test solution was maintained at 25°C. Air drawn through the solution was passed through xylene and 0.5 N NaOH traps to collect volatiles and CO_2. An additional treated sample was maintained under identical conditions in the dark.

Samples of the test solution were extracted twice with ethyl acetate following adjustment to pH 1 with 6 N HCl. The extracts were combined, concentrated, and aliquots were spotted onto silica gel TLC plates along with known standards. The plates were developed in 1-butanol:glacial acetic acid:water (4:1:1, v:v:v), chloroform:glacial acetic acid (1:1, v:v), and ether, and autoradiographed. Radioactive areas were scraped from the plates and quantified using LSC. Aliquots of the remaining aqueous phase were also characterized using TLC.

Naled

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_____ Description of product 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

__X__ FIFRA registration data.

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

Naled

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PERTINENT DATA TABLES AND FIGURES

TABLE I

NALED PHOTODEGRADATION PRODUCTS AT DIFFERENT INTERVALS IN PRESENCE OF WATER AND AIR

Hour	Percent of Total ^{14}C						
	Naled	DDVP	BDCA	CAA and DCAA	Acetic Acid	CO_2	Aqueous[a]
0	92.6	1.9	4.5	0.8	-	0	0.2
1	88.5	2.9	5.0	1.3	-	0	1.7
2	90.6	2.9	4.0	0.4	-	0	2.1[b]
4	87.8	3.8	4.4	1.0	-	0	3.0[b]
6	83.8	5.3	4.8	1.5	2.3	0	2.3
24	57.9	11.8	11.6	4.3	7.1	1.2	6.1
48	27.8	11.9	29.6	6.9	12.8	3.6	7.4
72	15.7	9.3	38.5	9.2	15.3	5.5	6.5
96	11.8	9.2	36.3	13.4	15.7	8.2	5.4

a - Contained at least 4 different products with R_f = 0.78, 0.68, 0.51, and 0.31 in Solvent System A.

b - May contain acetic acid.

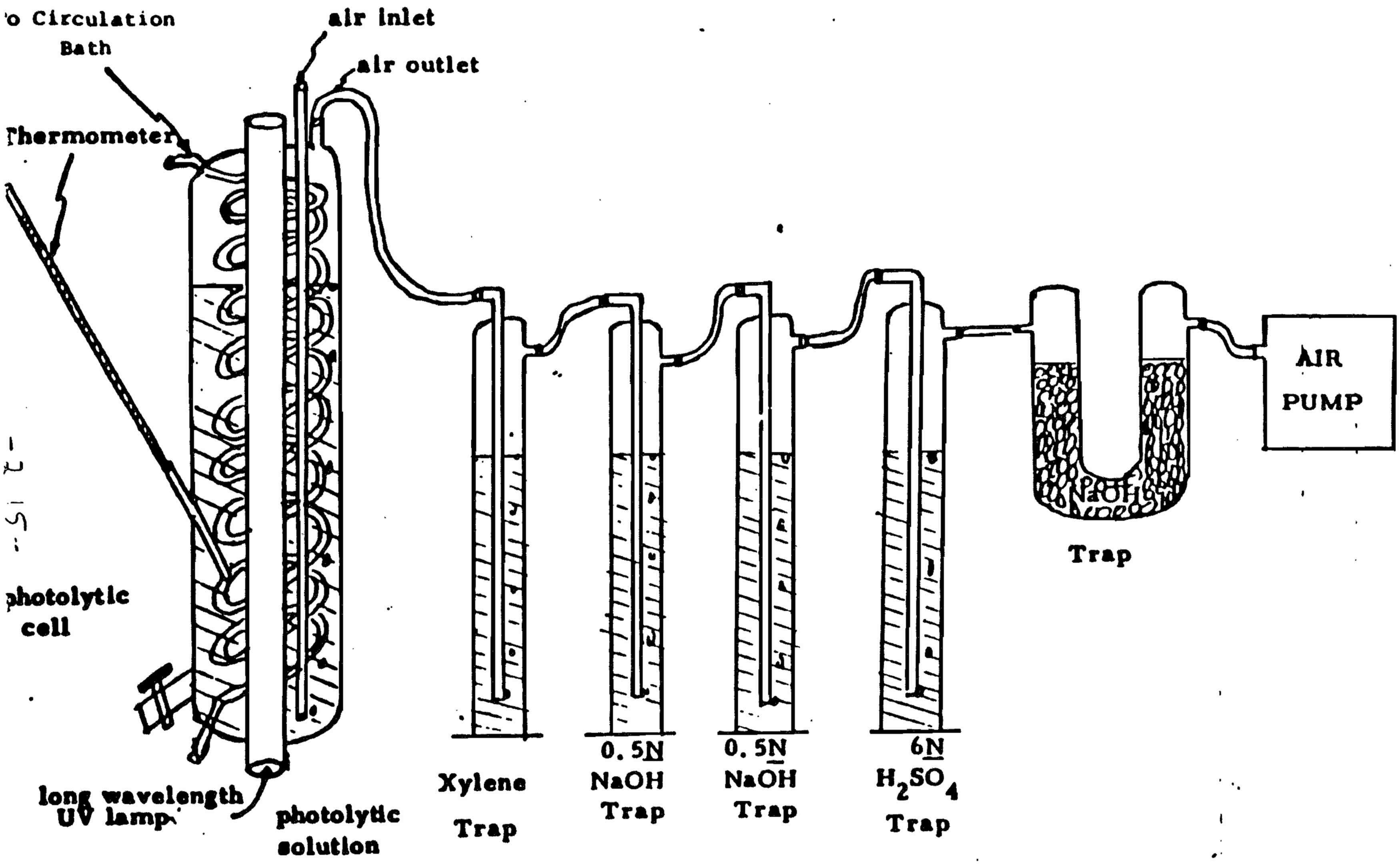

Figure 1. Diagram of Apparatus Used in Photodegradation Study

DATA EVALUATION RECORD

NALED STUDY 3

CHEM 034401 Naled

BRANCH EAB

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40357301
Chen, Y.S. 1987. Naled photolysis on dead cotton leaves. Laboratory Project Identification MEF-0049/8720594. Prepared and submitted by Chevron Chemical Company, Richmond, CA.

SUBST. CLASS = S

DIRECT RVW TIME = 5

REVIEWED BY:	T. Colvin-Snyder	TITLE:	Staff Scientist
EDITED BY:		TITLE:	Task Leader
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APPROVED BY: S. C. Termes
TITLE: Chemist
ORG: EAB/HED/OPP
TEL: 557-1981

SIGNATURE: May 19th, 1988

CONCLUSIONS:

Degradation - Photodegradation on Leaves (in place of Photodegradation on Soil)

This study is scientifically sound and provides supplemental information towards the registration of naled. This study does not fulfill EPA Data Requirements for Registering Pesticides because of the experimental design. The experiment was conducted in two days (0.5- and 2-hours samples taken on one day; 1- and 4-hours samples on another) which had different atmospheric conditions and, therefore, the incubation conditions for all samples were not identical.

SUMMARY OF DATA BY REVIEWER:

Ethyl-2-[^{14}C]naled (radiochemical purity 97.3%, specific activity 20.3 mCi/mMol), applied at 0.281 mg (equivalent to approximately 2 lb ai/A) to the surface of discs of excised, dried cotton leaves, photodegraded with a half-life of <2 hours in sunlight at 24-25 C. (Lattitude: 37°59'02"N; Longitude: 122°20'15"W. The experiments were conducted on two separate days; one day was sunny with patches of clouds and the other was overcast with spotty sunshine). DDVP (2,2-dichlorovinyl dimethyl phosphate) reached a maximum concentration of 37.4% of the applied at 2 hours posttreatment and one unidentified degradate was isolated at 2.5-4.6% throughout the study. Volatilized naled and DDVP totaled 1.6% and 4.9% of the applied, respectively, after 4 hours of irradiation. In the dark control, [^{14}C]naled did not degrade during 2 or 4 hours of incubation.

DISCUSSION:

1. The experimental design was inappropriate to accurately establish the dissipation of naled and the formation of degradates on leaf surfaces. Because the experiment was conducted on separate, meteorologically dissimilar days, the 0.5- and 2-hour samples should not be compared to the 1- and 4-hour samples; there was no normal progression of environmental conditions (air temperature and sunlight intensity and spectrum were lower on the 1- and 4-hour experiment day) with time. As a result, naled degraded less in the 1-hour sample than in the 0.5-hour sample, and less in the 4.0-hour sample than in the 2.0-hour sample.

2. Exposure to the degradate DDVP is of concern to EPA. It cannot be determined from these data if continued exposure to sunlight would result in higher concentrations of DDVP on leaf surfaces.

3. The exact position of the labeled carbon in [^{14}C]naled is unclear; the registrant stated that [^{14}C]naled was labeled in the 2 position, but the registrant indicated that [^{14}C]naled was labeled in the 1 position using a drawing of the structure of naled.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Ethyl-2-[^{14}C]naled (radiochemical purity 97.3%, specific activity 20.3 mCi/mMol, New England Nuclear) was applied at 0.281 mg (equivalent to 2 lb ai/A) to the surface (surface area 12.5 cm^2) of cotton leaves that had been excised from 8-week-old plants, cut into discs (2-inch diameter), and oven-dried between two glass plates at 120°C for 4 hours. The treated discs were incubated on glass plates in borosilicate glass bell jars. Air was drawn by vacuum-suction (approximately 200 mL/min) through each bell jar and then through a dry ice-in-acetone-cooled hexane scrubber to trap volatiles (Figure 1). The bell jars were placed outdoors in Richmond, California (latitude 37°59'02"N, longitude 122°20'15"W), on August 17th, which was a sunny day with patches of cloud and ambient temperatures of 24 to 25°C (Table 1). Additional treated leaf samples in bell jars covered with aluminum foil were incubated outdoors with the irradiated samples to serve as dark controls. Leaves and hexane scrubbers were sampled at 0.5 and 2 hours posttreatment.

The experiment was repeated on August 19th, which was an overcast day with "spotty" sunshine. Air temperatures ranged from 20 to 25°C. Leaves and hexane scrubbers were sampled at 1 and 4 hours posttreatment.

Each bell jar was cooled in a freezer to minimize loss of material through volatilization for ten minutes. The leaves were then removed from the bell jars and extracted twice with acetone. The bell jars and bottom glass plates were also rinsed with acetone and hexane. Aliquots of the combined extracts and rinses (leaf, bell jar, and glass plate) were analyzed for total radioactivity by LSC. Additional aliquots were concentrated and analyzed for degradates by HPLC; the HPLC analyses were confirmed using two-dimensional TLC. For TLC analyses, extracts were spotted on silica gel plates and developed in diethyl ether in the first direction and butan-1-ol:acetic acid:water (6:1:1) in the second direction. Radioactive areas were located using autoradiography, quantified by LSC, and identified by comparison to nonradiolabeled reference standards located using ultraviolet light. The extracted leaves were analyzed for radioactivity by LSC following combustion. The hexane scrubber solutions were analyzed by LSC, and solutions containing sufficient radioactivity ($\geq$0.3% of the applied radioactivity) were concentrated and analyzed by HPLC and TLC as described above.

3

Naled

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_____ A draft product label.

_____ The product confidential statement of formula.

_____ Information about a pending registration action

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

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PERTINENT DATA TABLES AND FIGURES

TABLE I

Sunlight Intensity and Temperature Measurement

DATE	TIME*	µWatts/cm²			ATMOSPHERIC COVER	TEMPERATURE °C
		BLUE REGION	RED REGION	FAR-RED REGION		
August 17	10:00	21	16	55	sunny with patches of clouds	24
	10:30	65	15	55		24
	11:00	86	29	85		25
	11:30	115	48	120		25
	12:00	120	63	133		25
August 19	09:15	15	10	12	overcast with spotty sunshine	20
	09:45	22	26	24		21
	10:15	17	15	16		21
	10:45	16	14	15		22
	11:15	38	42	50		22
	11:45	22	30	30		22
	12:30	70	55	50		24
	13:00	34	25	30		22
	13:15	61	38	42		25

* - 0.5 and 2 hours samples were taken on the 17th, 1 and 4 hours samples were taken on the 19th.

TABLE II

14C DISTRIBUTION AND RECOVERY

INTERVAL (HOURS)	% 14C of Total Applied				Calculated Radiation Exposure[c]	Sample
	Hexane Scrubber	Bell Jar[a]	Leaf Residue[b]	TOTAL Recovery		
0	--	99.5	0.8	100.3	0	dark
0.5	0	107.5	0.5	108.0	0	dark
1.0	0.3	98.8	0.6	99.7	0	dark
2.0	0.2	106.0	1.6	107.8	0	dark
4.0	0.1	99.9	0.7	100.7	0	dark
0.5	0	103.0	1.1	104.1	21.5	light
1.0	1.5	104.3	1.0	106.8	19.0	light
2.0	1.7	86.4	2.2	90.3	168.3	light
4.0	6.7	82.1	3.1	91.9	113.1	light

a - Includes cotton leaf acetone extracts.

b - Nonextractable 14C of the leaf, was not further characterized due to its quantity.

c - The unit is linearly proportional to the UV exposure, uWatts/cm^2/interval(hour), by integrating Table I .

TABLE III

Quantitation of the Naled and Its Photoproducts

INTERVAL (HOUR)	% OF TOTAL 14C APPLIED					SAMPLE
	BELL JAR[a]			HEXANE SCRUBBER		
	NALED	DDVP	UNKNOWN	NALED	DDVP	
0	93.8	2.6	3.1	0	0	dark
0.5	98.0	4.4	5.1	0	0	dark
1.0	90.6	5.4	2.8	NA	NA	dark
2.0	99.5	3.3	3.2	NA	NA	dark
4.0	93.7	3.9	2.6	NA	NA	dark
0.5	75.9	24.2	2.9	0	0	light
1.0	83.2	18.6	2.5	0.5	0.5	light
2.0	44.4	37.4	4.6	0.3	1.4	light
4.0	45.1	33.8	3.2	1.6	4.9	light

NA - Not analyzed due to low 14C content.
a - See Table II.

FIGURE 1
SCHEMATIC DRAWING OF THE APPARATUS

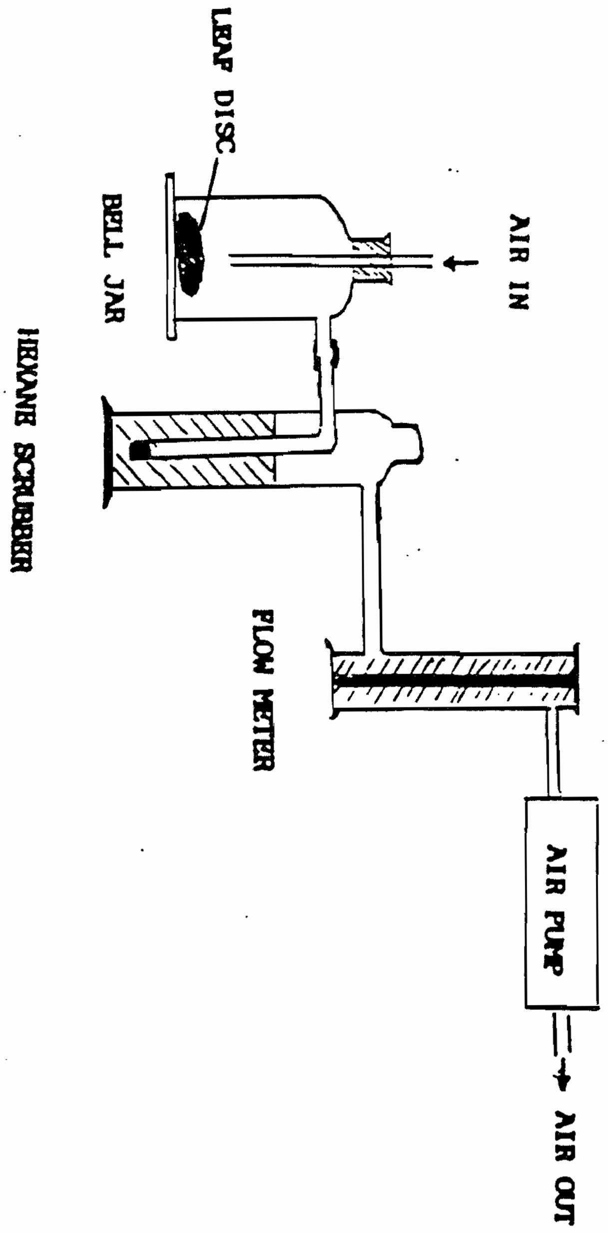
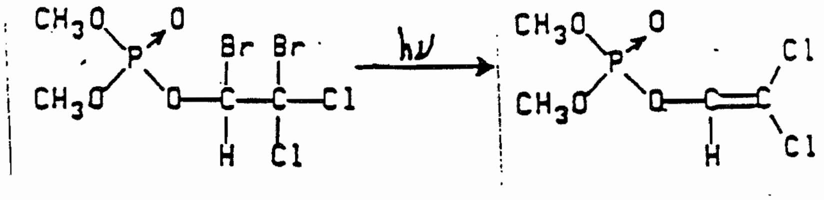


FIGURE 4

PHOTOLYSIS PATHWAY OF NALED ON DEAD COTTON LEAVES



NALED

DDVP

DATA EVALUATION RECORD

NALED STUDY 4

CHEM 034401 Naled

BRANCH EAB

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40050901 CONTENT CAT 01
Teeter, D. 1986. Determination of vapor phase photolysis rate of naled. Laboratory Project ID: ABC 34595. Prepared by Analytical Bio-Chemistry Laboratories, Inc., Columbia, Missouri, and submitted by Chevron Chemical Company, Richmond, CA.

SUBST. CLASS = S

DIRECT RVW TIME = 6

REVIEWED BY: K. Patten TITLE: Staff Scientist

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

APPROVED BY: W. Spangler TITLE: Project Manager

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

APPROVED BY: S. Termes
TITLE: Chemist
ORG: EAB/HED/OPP
TEL: 557-1981

SIGNATURE: May 9th, 1986

CONCLUSIONS:

Degradation - Photodegradation in Air

This study is unacceptable because air samples were never analyzed separately from nonvaporized naled; naled degradation in the vapor phase could not be distinguished from degradation that occurred in material adsorbed to the sides of the glass container. In addition, this study would not fulfill EPA Data Requirements for Registering Pesticides because up to 81.1% of the applied radioactivity was not identified and the spectrum of the artificial light source was not similar to that of natural sunlight.

SUMMARY OF DATA BY REVIEWER:

Ethyl-1-[^{14}C]naled (radiochemical purity >95%), at 29 or 290 ug/72 L bottle (0.403 or 4.03 ug/L), did not photodegrade in borosilicate glass bottles (72-L) irradiated continuously with a G.E. Model RSM sunlamp at 30 C and ambient humidity for 11 days; the degradation rate in both the irradiated samples and dark controls was calculated to be 10.3-10.4 days. The sunlamp produced a discontinuous spectrum of light with major wavelengths at approximately 360, 405, 440, 550, and 575 nm. DDVP (2,2-dichlorovinyl dimethyl phosphate) was detected in "minor" amounts (<10% of the applied) in all samples, including the primary stock solution. Dichloroacetaldehyde (DCA), bromodichloroacetaldehyde (BDCA), and dichloroethanol (DCE) were not detected in any sample; up to 81.1% of the applied radioactivity was not identified. The material balance ranged from 40.9-109% (average 96 ± 15%) of the applied radioactivity during the study.

DISCUSSION:

1. Because the naled in the air was condensed onto the walls of the bottles and the bottles were then extracted, it is impossible to distinguish between degradation that occurred in the air and degradation that occured on the glass. Since the melting point of naled is reported to be approximately 27 C and the study was conducted at 30 C, it is probable that the majority of the naled was either in solution or adsorbed to the walls of the bottle. At no time was the air sampled.

2. The analytical method was inadequate. The samples were analyzed using TLC with only one solvent system, and the retention time of the parent compound changed with each new batch of solvent. Despite the variable retention times, the samples were cochromatographed only against the parent compound. Reference standards of possible degradates were chromatographed only once and on a separate silica gel plate.

   Also, none of the [^{14}C]residues in the extract were mobile, except for those cochromatographing with naled and DDVP. The residues remaining at the origin comprised up to 81.1% of the applied radioactivity. The study author made no further attempt to identify the [^{14}C]residues. The dichloroacetic acid reference standard did not migrate from the origin, therefore the study author concludes that the unidentified origin material was probably dichloroacetic acid, as well as acetic acid and chloroacetic acid which were not tested.

3. The artificial light source produced a discontinuous spectrum that was not similar to sunlight.

4. The study author provided an absorbance spectrum for naled indicating that absorbance above 300 nm is negligible. However, in both the photodegradation in water and on leaf surfaces studies, the reported results indicate that naled degraded more rapidly in the irradiated samples than in the dark controls.

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5. At several sampling intervals, the material balance was poor. This may be due to the study design, since each sample was treated independently (Discussion point 7). Also, it is possible that recovery problems were due to the use of very large containers (72 L), a small amount of naled (0.403 or 4.03 ug/L), and a small amount of extracting solution (100 mL).

6. The vapor pressure of naled was reported to be 2 x 10^{-4} mm of Hg at 20 C; the study was conducted at 30° C. What is the vapor pressure at that temperature?

7. The laboratory notes state that the environmental chamber contained two bottles, one irradiated and one dark control. Therefore, the photodegradation study is actually ten separate sequential experiments that were conducted at different times in a single environmental chamber. Temperature data from the environmental chamber indicate that the experments were conducted between August 13 and October 14, 1986 (in the lab notes, the study author considers each 12-hour irradiation period equivalent to one day). The dates that each experiment were conducted are: 8/13 to 8/15, high concentration for 3 days; 8/26 to 8/29, high for 6 days; 8/29 to 9/3, high for 10 days; 9/3 to 9/5, low for 3 days; 9/5 to 9/8, low for 6 days; 9/9, high for 1 day; 9/9 to 9/10, low for 1 day; 9/12 to 9/17, low for 10 days; 9/24 to 10/1, low for 22 days; 10/3 to 10/14, high for 22 days. Although this method is not incorrect, it requires that the researchers take additional care to insure that treatment rates and incubation conditions are identical between experiments.

8. It is unclear whether the bottles were extracted with methanol or hexane. In the typed summary in the front of the original document, the study author states that the bottles were extracted with methanol. The hand-written laboratory notes in the study appendix state that the bottles were extracted with hexane. Methanol is listed as the solute solvent on the TLC forms.

9. Naled is known to be unstable on silica gel; however, TLC on silica gel plates was used to "identify" photoproducts. No other methodology was used to attempt characterizing photoproducts.

10. For the dark samples, a thick, black plastic bag was used to cover the the vessel. How effective is this method?

11. The author attributed the low ^{14}C recovery on Day 10 samples due to their being allowed to sit too long in the refrigerator before extraction. What stability data does the author have?

5

MATERIALS AND METHODS

MATERIALS AND METHODS:

Ethyl 1-[^{14}C]naled (radiochemical purity 95%, specific activity 1.10 x 10^{-7} dpm/mg, Chevron Chemical Company) in hexane was added at 0.403 or 4.03 ug/L to two 72-L borosilicate glass bottles. The bottles were sealed with borosilicate glass lids and high vacuum grease. One bottle was covered with a thick black plastic bag to serve as a dark control. Both bottles were placed in a mini-environmental chamber maintained at 30 C. The flask to be irradiated was positioned so that the surface of the glass was 40 cm from a G.E. Model RSM sunlamp. Bottles containing 0.403 ug/L of naled were irradiated for 3 days, then removed from the chamber and replaced with two bottles (one exposed and one dark control) that would be irradiated for 6 days. At the completion of the study, bottles containing high concentrations of naled had been irradiated for 0, 0.5, 3, 5, and 11 days and bottles containing low concentrations of naled had been irradiated for 0. 0.5, 1.5, 3, 5, and 7 days.

After exposure, the bottles were placed in a walk-in freezer at approximately -20 C for 2 hours to condense the gaseous products onto the surface of the glass. Then 100 mL of methanol (hexane?) were added to the bottles, the bottles were resealed, and the methanol was swirled vigorously to coat the glass surfaces. The swirling was repeated twice to insure that the solution was homogenous. Ten mL of the methanol were analyzed for total radioactivity by LSC, the remainder was stored frozen at -20 C until later analysis by TLC on silica gel plates developed in chloroform:acetic acid (95:5). The extracts were cochromatographed with naled stock solution; the registrant stated that retention factors "showed large variations with each new batch of mobile solvent mixture". Radioactive areas on the plates were located using a TLC linear scanner. Unlabeled standards of 2,2-dichloroethenyl dimethyl phosphate (DDVP), dichloroacetaldehyde (DCA), bromodichloroacetaldehyde (BDCA), 2,2-dichloroethanol (DCE), and dichloroacetic acid (DCAA) were spotted onto silica gel plates along with a radiolabeled naled standard and the plates were developed in chloroform:acetic acid (95:5). The unlabeled reference standards were visualized using silver nitrate reagent.

Naled

Page ______ is not included in this copy.

Pages __54__ through __57__ are not included in this copy.

The material not included contains the following type of information:

_____ Identity of product inert ingredients.

_____ Identity of product inert impurities.

_____ Description of the product manufacturing process.

_____ Description of product 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

__X__ 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.

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

SUMMARY

The rate of photolysis of Naled when exposed to a G.E. Model RS-2 Sunlamp at 30°C was determined. Light passed through a borosilicate glass filter was used to photolyze 72 liters of Naled-saturated air for periods of time ranging from zero to eleven days. Dark flasks were prepared with each exposed flask in order to observe atmospheric degradation as well as photolysis.

No degradation due to exposure to the sunlamp was observed. A dark reaction of atmospheric degradation was observed, with first-order kinetics and a half-life of 10.8 days. The rate constant is 0.0643/day.

This test was also performed at one-tenth of the saturated vapor concentration. This data set yielded no information. At low concentrations, Naled is unstable in the presence of silica gel, and the compound degradation during analysis by TLC is much greater than the atmospheric degradation.

Evidence indicates that the most prevalent degradation products during the course of the study were dichloroacetic acid, possibly chloroacetic acid, and/or acetic acid.

RESULTS AND DISCUSSION

Refer to Table 1 for Sample Code and Table 2 for Study Results.

All calculations of rate constants have been made from the high concentration set of data. Naled is known to be unstable on silica gel; at the level the samples were analyzed for Naled in the low concentration set, this instability was greater than any atmospheric degradation. Levels of Naled in the low samples after TLC analysis ranged between 20% and 44% in a relatively random pattern except the time 0 sample, which recovered 59%. (The low ^{14}C recovery on the Day 10 samples was due to their being allowed to sit too long in the freezer before extraction).

Light and dark samples of the high concentration portion of this study appear to agree with each other well within typical analytical variability. The only exception is the Day 22 Dark sample, which is approximately 50% low in parent Naled. This point is not included in calculations, because it is not reasonable to expect the dark reaction to suddenly proceed much faster than the reaction in the flask exposed to light. Also, its inclusion in the rest of the data gives rate constants and half-lives for the "dark" and "combined" data sets that are not consistent with the data obtained (i.e., the dark half-life was calculated at 5.26 days, while data from Day 5 shows only about 35% degradation).

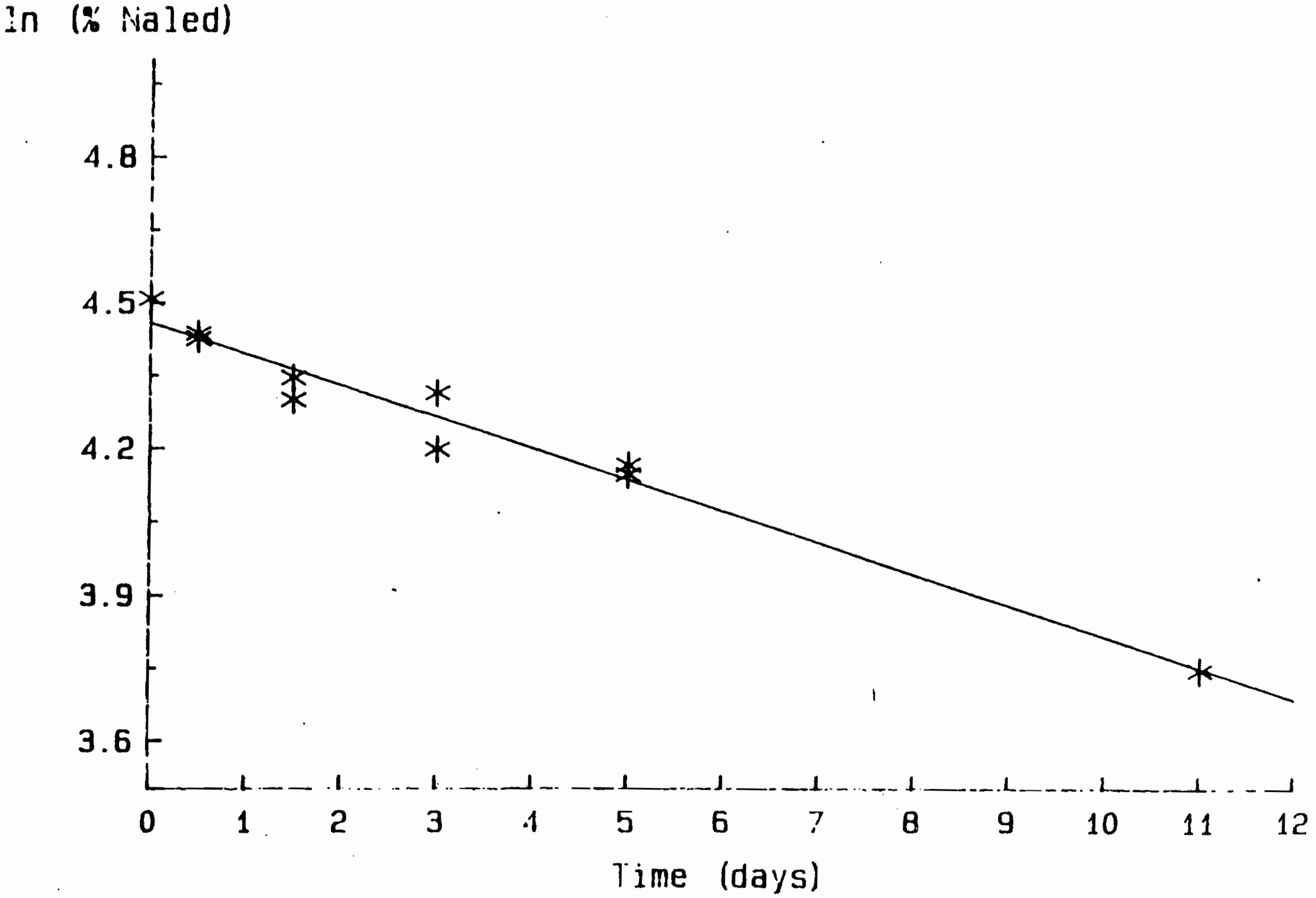
The low ^{14}C recovery of the Day 22 high light sample may be due to diffusion through a poorly sealed lid, or to incomplete extraction. It is also possible that ^{14}C compounds may have been slowly diffusing into the silicon grease sealant.

The protocol requires that an attempt be made to identify all degradation products that occur in amounts greater than 10% of initial concentration. The only peaks other than parent Naled observed greater than 10% in the high concentration samples were those that remained at the origin of the TLC chromatogram. Of the compounds tested as possible degradation products, only dichloroacetic acid remained at the origin. The rest moved well above the origin, and resolved well away from the parent Naled. It is possible that the chloroacetic acid and acetic acid may form from the dichloroacetic acid, and these compounds behave similarly on silica gel during thin layer chromatography.

PERTINENT DATA TABLES AND FIGURES

FIGURE 1

Plot of Naled Degradation



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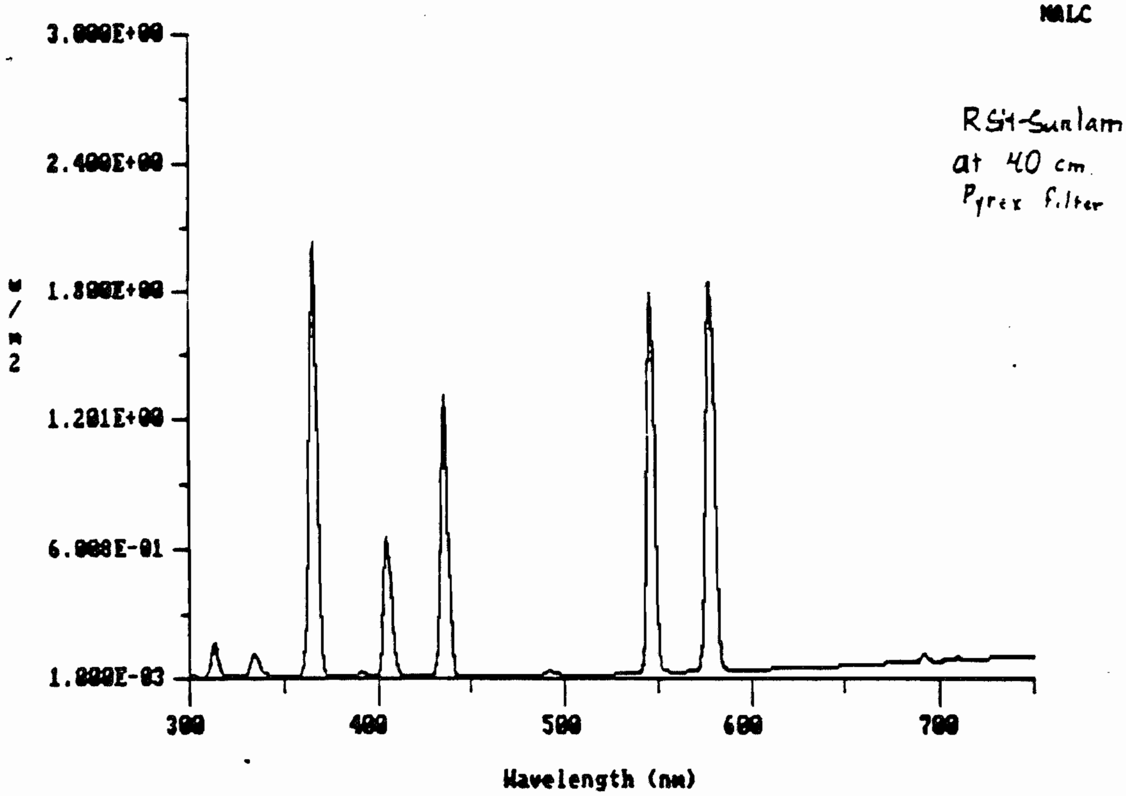
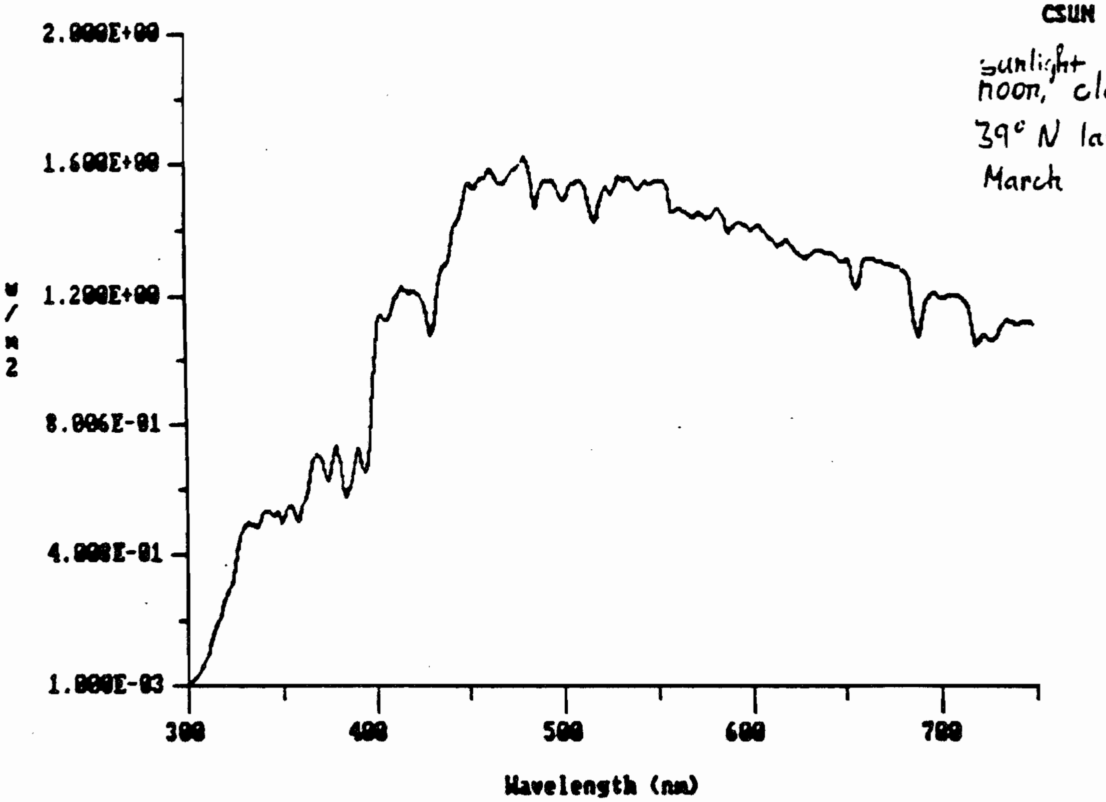


RS4-Sunlamp
at 40 cm
Pyrex filter

**CALSUN 05/05 07:09
LO: 300 HI: 750 INT: 1 MIN: 300NM 4.6892E-03 MAX: 481NM 1.6235E+00



sunlight
noon, clear sky
39° N latitude
March

BAUSCH & LOMB

SPECTRONIC [illegible]

CAT NO [illegible]

[illegible]

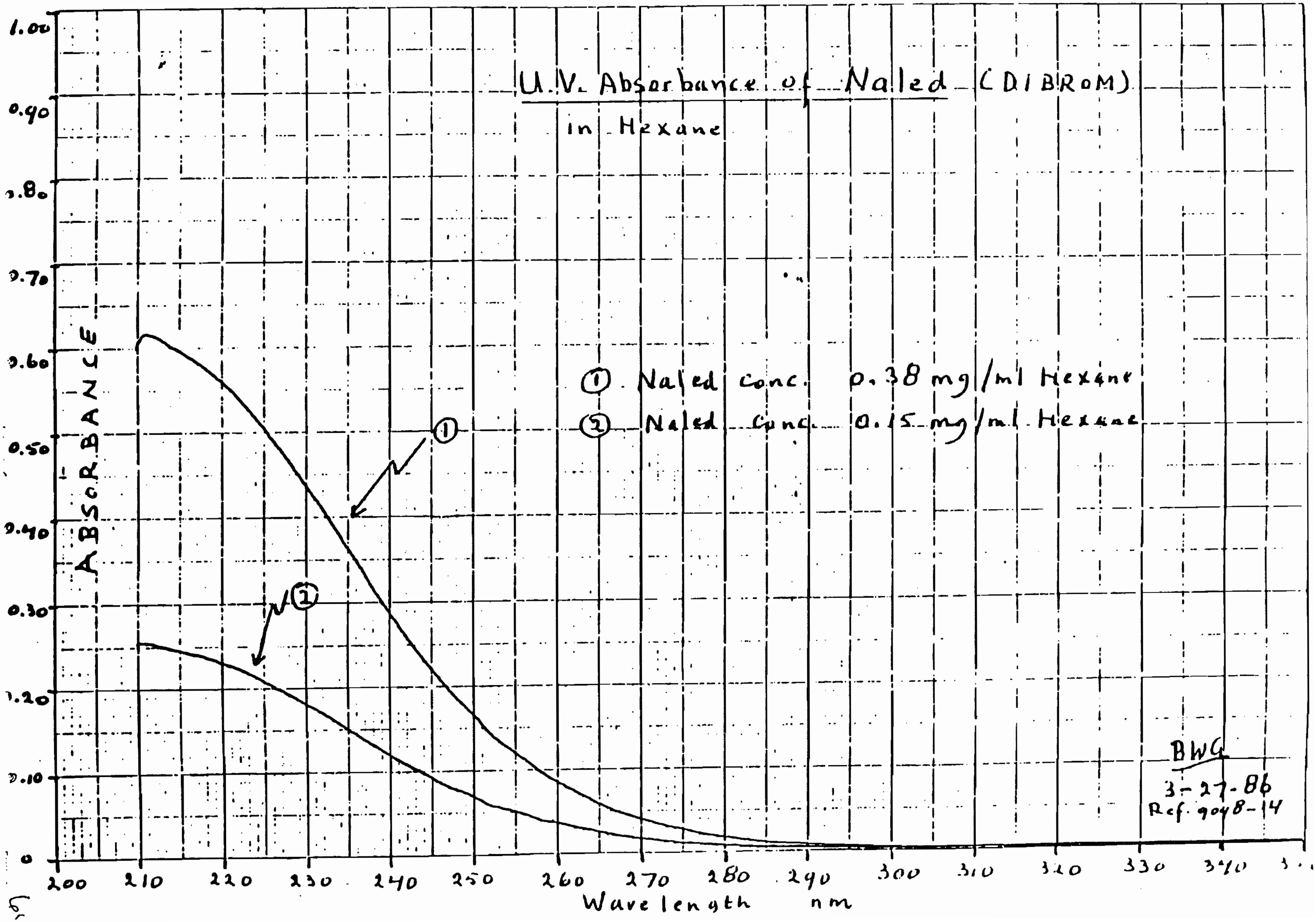


TABLE 2

Study Results

	Real Time	Light			Dark		
Sample Code	(Days)	% Recovery	% Naled	ln % Naled	% Recovery	% Naled	ln % Naled
0-High	0	107	90.8	4.509	---	---	---
1-High	0.5	104	84.4	4.440	102	83.5	4.425
3-High	1.5	103	77.1	4.345	103	73.7	4.300
6-High	3	97.9	74.8	4.315	102	66.6	4.199
10-High	5	94.6	63.3	4.148	93.3	64.5	4.167
22-High	11	70.3	42.4	3.747	101	19.9*	2.991*
0-Low	0	109	59.0	4.078	---	---	---
1-Low	0.5	99.4	39.6	3.679	104	40.8	3.709
3-Low	1.5	101	44.0	3.784	105	27.0	3.296
6-Low	3	102	34.3	3.535	104	41.2	3.718
10-Low	5	87.6	20.1	3.001	40.9	29.4	3.381
14-Low	7	78.1	29.9	3.398	104	26.5	3.277

*This point is considered a statistical outlier and is not included in calculations.

From the high concentration data: ln (% Naled) = -kt + c

Light only: Correlation = -0.9956
$k = 0.0668\ \text{day}^{-1}$
$t_{\frac{1}{2}} = 10.4$ days

Dark only: (Day 0 included) $r = -0.9327$
$k = 0.0670\ \text{day}^{-1}$
$t_{\frac{1}{2}} = 10.3$ days

Both: $r = -0.9824$
$k = 0.0643\ \text{day}^{-1}$
$t_{\frac{1}{2}} = 10.8$ days

DATA EVALUATION RECORD

NALED STUDY 5

CHEM 034401 Naled

BRANCH EAB

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID No MRID
Pack, D.E. 1986a. The aerobic aquatic metabolism of (ethyl-1-^{14}C)naled. Laboratory Project Identification 8607764. Prepared and submitted by Chevron Chemical Company, Richmond, CA. Reg. No. 263621.

SUBST. CLASS = S

DIRECT RVW TIME = 8

REVIEWED BY: T. Colvin-Snyder TITLE: Staff Scientist
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ORG: Dynamac Corporation
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TEL: 468-2500

APPROVED BY: S. Termes
TITLE: Chemist
ORG: EAB/HED/OPP
TEL: 557-1981

SIGNATURE: May 19th, 1988

CONCLUSIONS:

Metabolism - Aerobic Aquatic

This study is unacceptable because material balances were incomplete (between 25.04 and 35.99% of the applied radioactivity was unaccounted at every sampling interval between 1 and 30 days posttreatment). In addition, this study would not fulfill EPA Data Requirements for Registering Pesticides because [^{14}C]residues were incompletely characterized (extracts containing up to 0.23 ppm naled residues were not analyzed for degradates, and degradates present at up to 0.318 ppm were not identified) and the test water was not characterized.

SUMMARY OF DATA BY REVIEWER:

Ethyl 1-[^{14}C]naled (radiochemical purity 99.3%, specific activity 118.340 dpm/ug), at 10 ppm, degraded with a half-life of <1 day in flooded sand soil that was incubated with continuous aeration at 25 C in the dark. Naled was not detected in the soil or water after 14 days posttreatment. The non-volatile degradates DDVP (2,2-dichloroethenyl dimethyl phosphate; ≤0.209 ppm), DCAA (dichloroacetic acid; ≤0.361 ppm), and DCE (dichloroethanol; ≤0.728 ppm) reached maximum concentrations at 1 day posttreatment. DDVP and DCAA were not detected at 30 days posttreatment; DCE was 0.001 ppm at 30 days posttreatment. Several unidentified degradates were isolated at up to 0.318 ppm. Carbon dioxide totaled 71.06% of the applied and organic volatiles totaled 0.15% at 30 days posttreatment.

DISCUSSION:

1. Material balances, based on an observed application rate of 8.861 ppm, were incomplete; 25.04-32.99% of the applied radioactivity was unaccounted for at 1-30 days posttreatment. In addition, although the registrant stated that naled was applied at 10.00 ppm, day 0 samples contained only 8.861 ppm of naled.

2. Degradates were inadequately characterized. The aqueous soil extracts contained up to 0.20 ppm (2.3% of the applied) naled residues but were not analyzed for degradates. In addition, five compounds were isolated on the TLC plates at maximum concentrations ranging from 0.034-0.318 ppm but were not identified. All degradates ≥0.01 ppm must be identified.

3. Only day-3 distillates of vapors trapped during concentration of the methanol and ethyl ether extracts were analyzed for the degradate DCE by HPLC. The registrant assumed that distillates from the other samples contained only DCE and did not contain other degradates.

4. The test water was not characterized; water pH and dissolved oxygen content were not reported.

5. The registrant stated that naled is unstable in the presence of silica gel, so TLC analyses were conducted "as rapidly as possible".

6. The registrant stated that the hydrocarbon traps were discontinued after the first few samplings since no radioactivity was detected; however, data for organic volatiles at all sampling intervals were provided.

7. The rate at which air was bubbled throughout the duration of the study was not specified.

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MATERIALS AND METHODS

MATERIALS AND METHODS:

Ethyl-1-[^{14}C]Naled (radiochemical purity 99.3%, specific activity 20.3 mCi/mMol, New England Nuclear) was applied at 10 ppm to water-flooded (50 g soil:100 mL water) sand soil (Table 1) in 250 mL flasks that were wrapped in aluminum foil and incubated at 25 C. Compressed air was "slowly" and constantly bubbled through the water layer in each flask to maintain aerobic conditions. The soil:water systems were sampled at 1, 3, 7, 14, and 30 days posttreatment.

The water layer was decanted from each soil:water sample and centrifuged, and the resulting soil pellet was returned to the soil fraction. The water was acidified to approximately pH 2 with 6 N sulfuric acid, saturated with ammonium sulfate, and extracted three times with ethyl ether:ethanol (2:1). The extracts and the extracted water were analyzed for total radioactivity by LSC. The soil was extracted three times with methanol, acidified with 1 M sodium bisulfate, and further extracted three times with water. The methanol and water extracts were analyzed separately for total radioactivity by LSC. The ethyl ether:ethanol extracts from the water layer and the methanol extracts from the soil layer were combined and concentrated by rotary evaporation; the solvent vapors were condensed onto a dry-ice-cooled cold trap. The nonvolatilized residues were dissolved in acetone and analyzed for naled and its degradates using two-dimensional TLC on silanized silica gel plates developed with chloroform:acetic acid (19:1) and then in chloroform: acetic acid (1:1). Areas of radioactivity were located using autoradiography, quantified by LSC, and identified by comparison to nonradiolabeled reference standards that had been located using "the appropriate spray reagent". The trapped solvent vapors from the 3-day sample were mixed with nonradiolabeled DCE and then distilled at 100 C; this distillate was analyzed by LSC, found to contain no radioactivity, and then discarded. The residues were further distilled at 80 C and then at 100 C; the resulting fractions were analyzed by LSC and then analyzed for the degradate DCE by reverse-phase HPLC.

In order to quantify volatilization, one flask containing naled-treated soil:water was connected to a series of volatile traps prior to incubation. The compressed air was passed sequentially through a water saturator, the water layer covering the soil, a hydrocarbon trap, and two sodium hydroxide solution traps. The soil:water system was kept under a slight negative pressure using a vacuum. The treated soil:water samples were incubated in the dark at 25 C. The sodium hydroxide trapping solutions were analyzed by LSC at intervals up to 30 days posttreatment. At 30 days posttreatment, the soil and water in the flask were analyzed for total radioactivity by LSC following combustion and LSC, respectively.

Naled

Page ______ is not included in this copy.

Pages _70__ through _74__ are not included in this copy.

The material not included contains the following type of information:

_____ Identity of product inert ingredients.

_____ Identity of product inert impurities.

_____ Description of the product manufacturing process.

_____ Description of product 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

__X__ FIFRA registration data.

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

Naled

Page ______ is not included in this copy.

Pages _76__ through _78__ are not included in this copy.

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_____ Identity of product inert ingredients.

_____ Identity of product inert impurities.

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PERTINENT DATA TABLES AND FIGURES

TABLE 1

SOIL CHARACTERISTICS

Origin	Cranberry Bog in Massachusetts
Classification	Sand
% Sand	92
% Slit	6
% Clay	2
% Organic	4.4
pH	4.3
Cation Exchange Capacity	3.5 meq/100 g
1/3 Bar Value	7.81%
Bulk density	1.13 g/ml

TABLE 2

^{14}C-NALED AEROBIC AQUATIC METABOLISM

VOLATILE METABOLITES

Initial fortification - 54980000 dpm

INTERVAL DAYS	CO_2		ORGANIC		
	DPM	% TOTAL OFF	DPM	% TOTAL OFF	% LEFT
0	0	0.00	0	0.00	100.00
1	8788903	15.99	5311	0.01	84.00
2	12321243	38.40	1275	0.01	61.59
3	6714285	50.61	3100	0.02	49.37
7	6199724	61.88	16929	0.05	38.07
10	1090782	63.87	34635	0.11	36.02
14	752793	65.24	13034	0.14	34.63
21	1405335	67.79	1970	0.14	32.07
28	1364945	70.28	1757	0.14	29.58
30	429568	71.06	2198	0.15	28.80

TABLE 3

^{14}C-NALED AEROBIC AQUATIC METABOLISM

EXTRACTION DATA

INTERVAL DAYS	REPLICATE	% OF DOSE				
		WATER SUPERNATANT	METHANOL EXTRACT	ACIDIC WATER EXTRACT	SOIL RESIDUE	TOTAL RECOVERED
1	A	35.26 35.94	21.01 21.21	2.19 2.16	5.37 5.87	63.83 65.19
	B	32.41 32.90	21.27 21.51	2.30 2.29	5.79 5.14	61.77 61.85
3	A	9.96 9.44	6.51 6.63	1.17 1.22	15.10 15.75	32.74 33.03
	B	10.64 10.81	7.50 7.84	1.15 1.15	10.58 11.33	29.88 31.14
7	A	8.77 8.83	4.58 4.66	0.98 0.67	14.58 12.81	28.92 26.97
	B	8.52 8.67	5.41 5.66	0.67 0.66	15.42 14.57	30.02 29.56
14	A	1.28 1.31	1.75 1.72	0.72 0.70	14.67 15.06	18.42 18.79
	B	4.00 4.02	2.95 2.88	0.71 0.71	15.98 16.09	23.64 23.70
30	A	0.07 0.07	0.91 0.89	0.43 0.43	16.77 14.43	18.18 15.83
	B	0.08 0.08	0.94 0.96	0.43 0.42	15.99 13.69	17.44 15.15

TABLE 4

QUANTITATION DATA

% OF DOSE

SPOT NUMBER	IDENTIFICATION	% OF DOSE									
		DAYS									
		1		3		7		14		30	
1		0.667	0.487	0.767	1.116	2.075	1.375	1.171	3.591	0.839	0.911
2		0.352	0.236	0.379	0.269		0.105		0.330	0.011	
3	DCAA	14.111	9.932	1.019	0.770		0.079	0.131	0.198		
4		0.334	0.406	0.256	0.000		0.650	0.054	0.066	0.050	0.016
5		0.268	0.278	0.100	0.072	0.190	0.224				
6			0.454								
7	DDVP	4.883	3.242	0.189	0.240		0.131	0.018	0.052		
8	Naled	8.296	5.762	0.120	0.356	0.172	0.057				
	TLC TOTAL	28.91	20.80	2.83	2.82	2.44	2.62	1.37	4.24	0.90	0.93
	DCE	24.106	30.331	12.541	14.574	10.273	10.735	1.533	2.322	0.017	0.031
	TOTAL	53.018	51.129	15.372	17.396	12.710	13.357	2.907	6.559	0.917	0.958

TABLE 5

QUANTITATION DATA

PPM FOUND CALCULATED AS NALED

SPOT NUMBER	IDENTIFICATION	PPM FOUND CALCULATED AS NALED									
		DAYS									
		1		3		7		14		30	
1	DCAA	0.059	0.043	0.068	0.099	0.184	0.122	0.104	0.318	0.074	0.081
2		0.031	0.021	0.034	0.024		0.009		0.029	0.001	
3		1.250	0.880	0.090	0.068		0.007	0.012	0.018		
4		0.030	0.036	0.023			0.058	0.005	0.006	0.004	0.001
5		0.024	0.025	0.009	0.006	0.017	0.020				
6			0.040								
7	DDVP	0.433	0.287	0.017	0.021		0.012	0.002	0.005		
8	Naled	0.735	0.511	0.011	0.032	0.015	0.005				
	TLC TOTAL	2.562	1.843	0.251	0.250	0.216	0.232	0.122	0.376	0.080	0.082
	DCE	2.136	2.688	1.111	1.291	0.910	0.951	0.136	0.206	0.001	0.003
	TOTAL	4.698	4.531	1.362	1.542	1.126	1.184	0.258	0.581	0.081	0.085

TABLE 6

QUANTITATION DATA

AVERAGE PPM FOUND BASED ON THE ACTUAL MOLECULAR WEIGHT OF EACH COMPOUND

	AVERAGE PPM FOUND (ACTUAL MW BASIS)					
DAYS	0	1	3	7	14	30
Naled	8.861	0.623	0.021	0.010	0.000	0.000
DDVP	0.000	0.209	0.011	0.007	0.002	0.000
DCAA	0.000	0.361	0.027	0.001	0.005	0.000
DCE	0.000	0.728	0.363	0.281	0.052	0.001

DATA EVALUATION RECORD

CASE GS — NALED STUDY 6 PM

CHEM 034401 Naled

BRANCH EAB

FORMULATION 00- ACTIVE INGREDIENT

Pack, D.E. and Fry, C.E. 1988. "Anaerobic aquatic metabolism of [Ethyl-1-^{14}C]naled" Laboratory Project ID: MEF-0012/8716031. Prepared and submitted by Chevron Chemical Company, Richmond, CA.
EPA Accession No. 40580001

SUBST. CLASS:

DIRECT RVW TIME: (MH) START DATE END DATE

REVIEWED BY: S.C. Termes
TITLE: Chemist
ORG: EAB/HED/OPP
TEL: (703)557-1981

APPROVED BY: S.C. Termes
TITLE: Chemist
ORG: EAB/HED/OPP
TEL: (703)557-1981

SIGNATURE: May 19th, 1988

CONCLUSIONS:

Metabolism- Anaerobic aquatic

This study is unacceptable as presented. Tables I and II and other important information are missing from the submitted report. Therefore, the review of this study cannot be completed at the present time.

SUMMARY OF DATA BY REVIEWER:

No summary of data will be given at the present time until the requested information is submitted and the study is reevaluated.

DISCUSSION:

1. Table I (Soil characteristics) and Table II (Phase I- Quantitation of volatile metabolites) were missing from the report.
2. No analytical data were provided for the bog water.
3. The flow rate of nitrogen was not specified. No apparent attempts were made to monitor the dissolved oxygen (DO) level to insure that anaerobic conditions were maintained at all times throughout the duration of the experiments.

Do the authors have any information on the pH of the leachates as to be able to speculate on the expected $CO_2/HCO_3^-/CO_3^{2-}$ distribution in the leachates?

SUMMARY OF DATA BY REVIEWER:

Aged (0.4-3.0 hours) ethyl 1-[^{14}C]naled residues were mobile in columns of sandy loam, clay loam, and two loam soils (one of them had been classified as sandy loam by the registrant); 5.4-10.98% of the applied radioactivity remained in the soil columns and 36.20-58.50% was in the leachates. [^{14}C]Naled residues were very mobile in sand soil columns; 2.71% of the applied radioactivity remained in the soil and 66.68% was in the leachates. In general, radioactivity remaining in the soil was evenly distributed throughout the columns. In the leachates, naled was not detected ($\leq$0.02 ppm), DDVP (2,2-dichlorovinyl dimethyl phosphate) was $\leq$0.093 ppm, DCE (dichloroethanol) was $\leq$0.085 ppm, DCAA (dichloroacetic acid) was $\leq$1.863 ppm, and carbonate was $\leq$0.282 ppm.

DISCUSSION:

1. The total radioactivity applied to the soil columns prior to leaching was not reported. Soils were treated at 10.00 ppm prior to aging. After leaching, only 46.50-69.39% of the applied radioactivity was accounted for. The registrant attributed the loss of radioactivity to the rapid conversion of naled to carbon dioxide; in a previously reviewed aerobic metabolism study (Pack et al, no MRID, 1980) 51.5% of the applied radioactivity was evolved as carbon dioxide after 3 days of incubation in loamy sand soil treated with naled at 12 ppm. Although $^{14}CO_2$ was an expected degradation product, no provisions were made to detect/monitor $^{14}CO_2$ evolution.

2. The soil columns were leached for several days to two weeks. Since the half-life of naled is extremely short (0.4-3.0 hours), extensive degradation occurred during leaching.

3. After leaching, the columns were drained overnight. It is unclear if solution drained from the columns was collected.

4. Several discrepancies were found in the data presented by the registrant. The data for Tables 12 and 16 (Concentration of metabolites in leachates, soils 6073-39 and 8149-11) were identical; it appears that data for one soil were copied twice and data for the other soil were omitted. Also, the totals for Table 6 were copied from Table 5; however, individual values for each fraction were reported so that correct totals could be calculated.

5. One of the soils (soil code 6073-39) classified by the registrants as a sandy loam soil is a loam soil according to the USDA Soil Textural Classification System and is referred to as such in this review.

6. Soil TLC data were not reviewed for this submission because they were previously reviewed (MRID 00064796, August 1982).

DATA EVALUATION RECORD

NALED Study 7

CHEM 034401 Naled

BRANCH EAB

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID NO MRID
Pack, D.E. 1986b. Soil column leaching of (ethyl-1-^{14}C). Laboratory Project Identification 86077623. Prepared and submitted by Chevron Chemical Company, Richmond, CA.

SUBST. CLASS = S

DIRECT RVW TIME = 7

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

EDITED BY: TITLE: Task Leader

APPROVED BY: TITLE: Project Manager

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

APPROVED BY: S. C. Termes
TITLE: Chemist
ORG: EAB/HED/OPP
TEL: 557-0335

May 19th, 1988

SIGNATURE:

CONCLUSIONS:

Mobility - Leaching and Adsorption/Desorption

This study is unacceptable as presented. Several deficiencies were noted which must be clarified by the registrant prior to reevaluation of the study: (a) why, if conversion of naled to $^{14}CO_2$ was an expected degradation pathway, no provisions were made to detected/monitor $^{14}CO_2$ evolution; (b) after leaching, the columns were drained overnight, but it is unclear if solution drained from the column was collected; (c) discrepancies in data presented (Tables 12 and 16 are identical and appears that data were copied twice and data for another soil may have been omitted; also Tables 5 and 6 show discrepancies in the totals); (d) a metabolite not previously observed was detected by HPLC in the leachates, which was attributed to ^{14}C-carbonate.

7. A species not previously found in degradation studies of naled was detected in HPLC-data from leachates. This species was attributed to an inorganic species, ^{14}C-carbonate. Knowledge of the pH of the leachates should have aided in establishing the distribution of $CO_2/HCO_3^-/CO_3^{2-}$ species in the leachates.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Ethyl 1-[^{14}C]naled (radiochemical purity 99.7%, specific activity 20.3 mCi/mMol, New England Nuclear) was applied at 10 ppm to 50-g samples of sand, sandy loam, clay loam, and two loam (one of these soils had been classified as sandy loam by the registrant) soils. The soil moisture was adjusted to about 75% of field capacity, and then the soil samples were incubated for one half-life (0.4-3.0 hours) of naled at 25 C. Columns (4.9-cm diameter, 28-cm length) of untreated soil saturated with 0.01 M calcium sulfate solution were topped with 2 cm (50 g) of the appropriate aged, treated soil. The soil columns were leached with a total of at least 53 cm (21 inches, 1000 mL) of 0.01 M calcium sulfate solution for several days to two weeks. Leachates were collected in 50-mL (2.6-cm) fractions. After leaching the columns were allowed to drain overnight and were then divided into 5-cm segments.

Leachates were analyzed for total radioactivity by LSC, and for naled and degradates by reverse-phase HPLC. A portion of each soil segment was analyzed for total radioactivity by LSC following combustion. Each soil segment was extracted four times with methanol, and the combined extracts were analyzed by LSC. The methanol extracts were concentrated by rotary evaporation at ambient temperature; the solvent vapors were condensed using a dry-ice-cooled cold trap. The residues were redissolved in methanol and then evaporated under nitrogen. The methanol extracts and condensed vapors were analyzed by LSC. The condensed vapors contained little radioactivity and were not further analyzed. Some of the methanol extracts were analyzed by HPLC, but there was too little radioactivity to detect individual compounds, so HPLC analyses were discontinued. Extracted soil samples were analyzed by LSC following combustion.

Page ______ is not included in this copy.

Pages __93__ through __97__ are not included in this copy.

The material not included contains the following type of information:

_____ Identity of product inert ingredients.

_____ Identity of product inert impurities.

_____ Description of the product manufacturing process.

_____ Description of product 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

__X__ 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.

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

7.12

Naled

Page ______ is not included in this copy.

Pages __99__ through __102__ are not included in this copy.

The material not included contains the following type of information:

_____ Identity of product inert ingredients.

_____ Identity of product inert impurities.

_____ Description of the product manufacturing process.

_____ Description of product 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

__X__ 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.

PERTINENT DATA TABLES AND FIGURES

TABLE I

SOIL CHARACTERISTICS

Soil Code	6073-39	6073-43	6073-48	8149-05	8149-11
Source	Greenville, Mississippi	Dallas Center, Iowa	Greenville, Mississippi	Ocoee, Florida	Fresno, California
Classification	Sandy Loam	Clay Loam	Sandy Loam	Sand	Loam
% Sand	48	30	70	92	48
% Silt	43	38	17	4	40
% Clay	9	32	13	4	12
% Organic	1.5	2.8	1.0	1.9	0.8
pH	5.5	8.1	7.1	6.7	7.2
CEC (meq/100 g)	7.6	27.8	7.5	2.9	6.8
1/3 Bar Value	20.4	32.3	13.4	4.39	14.0
Bulk Density (g/ml)	1.15	1.01	1.23	1.47	1.38

TABLE 2

TOTAL C-14 CONCENTRATION IN LEACHATES

(PPM CALCULATED AS NALED)

SOIL 6073-39

	COLUMN A			COLUMN B		
FRACTION NUMBER	VOLUME mL	% OF DOSE	PPM	VOLUME mL	% OF DOSE	PPM
1	50	.00	.000	50	.00	.000
2	50	0.00	0.000	50	.00	.000
3	50	0.00	0.000	50	.00	.000
4	70	2.58	0.183	52	0.24	0.023
5	50	13.26	1.319	57	11.31	0.987
6	50	12.46	1.240	53	11.05	1.037
7	50	6.54	0.651	50	8.75	0.871
8	50	2.27	0.225	50	4.18	0.416
9	50	0.35	0.035	50	1.50	0.149
10	50	0.09	0.009	50	0.60	0.060
11	50	0.08	0.008	50	0.08	0.008
12	50	0.07	0.007	50	0.07	0.007
13	50	0.07	0.007	50	0.07	0.007
14	51	0.06	0.006	54	0.06	0.005
15	50	0.02	0.002	57	0.07	0.006
16	70	0.03	0.002	50	0.04	0.004
17	52	0.01	0.001	50	0.02	0.002
18	50	0.02	0.002	51	0.01	0.001
19	50	0.02	0.002	59	0.02	0.001
20	56	0.01	0.001	51	0.02	0.002
21	40	0.01	0.001			
TOTAL	1089	37.94		1034	38.08	

TABLE 3

TOTAL C-14 CONCENTRATION IN LEACHATES

(PPM CALCULATED AS NALED)

SOIL 6073-43

	COLUMN A			COLUMN B		
FRACTION NUMBER	VOLUME mL	% OF DOSE	PPM	VOLUME mL	% OF DOSE	PPM
1	50	0.00	0.000	50	0.00	0.000
2	50	0.00	0.000	50	0.00	0.000
3	50	3.73	0.403	50	3.45	0.372
4	50	5.70	0.615	50	6.10	0.659
5	50	6.21	0.670	50	7.36	0.794
6	50	7.13	0.769	50	8.65	0.934
7	50	7.74	0.835	50	9.49	1.024
8	50	6.41	0.692	50	7.19	0.776
9	50	5.04	0.544	50	4.90	0.529
10	50	3.78	0.408	50	2.76	0.298
11	50	2.59	0.279	50	1.92	0.207
12	50	1.88	0.203	50	1.43	0.155
13	50	1.39	0.150	50	1.13	0.122
14	50	1.03	0.111	50	0.94	0.102
15	50	0.82	0.089	50	0.82	0.089
16	50	0.61	0.066	50	0.70	0.075
17	50	0.57	0.062	50	0.56	0.060
18	50	0.57	0.061	50	0.48	0.052
19	50	0.57	0.061	50	0.43	0.046
20	50	0.48	0.052	50	0.40	0.044
21	50	0.36	0.039	50	0.41	0.044
22	50	0.32	0.034	50	0.37	0.040
23	81	0.44	0.029	24	0.13	0.030
TOTAL	1081	57.37		1024	59.63	

TABLE 4

TOTAL C-14 CONCENTRATION IN LEACHATES

(PPM CALCULATED AS NALED)

SOIL 6073-48

FRACTION NUMBER	COLUMN A			COLUMN B		
	VOLUME mL	% OF DOSE	PPM	VOLUME mL	% OF DOSE	PPM
1	50	.00	.000	50	.00	.000
2	50	0.11	0.011	50	.00	.000
3	52	2.38	0.228	50	0.60	0.059
4	51	4.16	0.406	50	6.00	0.597
5	50	4.68	0.465	50	16.47	1.638
6	51	3.02	0.294	50	12.61	1.254
7	50	1.44	0.144	53	6.12	0.575
8	50	1.04	0.103	51	3.14	0.306
9	52	0.68	0.065	53	2.11	0.198
10	50	0.60	0.060	50	1.36	0.135
11	52	0.37	0.036	50	1.03	0.102
12	52	0.40	0.039	55	0.78	0.070
13	50	0.27	0.027	51	0.54	0.053
14	56	0.21	0.019	49	0.48	0.049
15	53	0.17	0.016	50	0.21	0.021
16	52	0.15	0.014	52	0.24	0.023
17	57	0.11	0.010	50	0.17	0.017
18	51	0.10	0.009	52	0.17	0.016
19	59	0.07	0.006	51	0.18	0.017
20	43	0.03	0.004	60	0.16	0.013
21				25	0.06	0.011
TOTAL	1031	19.99		1052	52.41	

7.21

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TABLE 6

TOTAL C-14 CONCENTRATION IN LEACHATES

(PPM CALCULATED AS NALED)

SOIL 8149-11

	COLUMN A			COLUMN B		
FRACTION NUMBER	VOLUME mL	% OF DOSE	PPM	VOLUME mL	% OF DOSE	PPM
1	50	0.01	0.001	50	3.79	0.372
2	51	5.32	0.512	50	6.71	0.659
3	37	20.49	2.719	51	8.09	0.779
4	57	19.75	1.701	21	9.51	2.223
5	53	2.90	0.269	72	10.43	0.711
6	52	0.68	0.065	49	7.90	0.791
7	51	0.68	0.065	51	5.39	0.519
8	60	0.32	0.026	60	3.04	0.248
9	65	0.19	0.014	50	2.11	0.207
10	50	0.16	0.016	24	1.58	0.323
11	75	0.10	0.007	60	1.25	0.102
12	53	0.06	0.005	46	1.03	0.110
13	35	0.04	0.006	53	0.90	0.084
14	52	0.06	0.005	52	0.77	0.073
15	50	0.06	0.006	67	0.61	0.045
16	21	0.02	0.005	48	0.53	0.054
17	75	0.08	0.005	50	0.47	0.046
18	55	0.05	0.004	41	0.44	0.053
19	22	0.02	0.003	58	0.45	0.038
20	63	0.04	0.003	46	0.40	0.043
21	17	0.01	0.003	9	0.15	0.080
TOTAL	1013	60.35		1081	73.01	

51.03 65.55

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TABLE 5

TOTAL C-14 CONCENTRATION IN LEACHATES

(PPM CALCULATED AS NALED)

SOIL 8149-05

	COLUMN A			COLUMN B		
FRACTION NUMBER	VOLUME mL	% OF DOSE	PPM (NALED)	VOLUME mL	% OF DOSE	PPM (NALED)
1	50	0.00	0.000	50	0.00	0.000
2	50	0.00	0.000	50	.00	.000
3	50	3.39	0.366	57	0.62	0.059
4	50	11.09	1.196	54	15.23	1.522
5	50	18.64	2.012	53	19.70	2.006
6	50	11.35	1.225	58	21.25	1.977
7	50	5.88	0.635	62	6.82	0.593
8	50	3.43	0.371	55	3.54	0.348
9	53	1.63	0.166	56	1.48	0.143
10	55	1.23	0.121	63	1.30	0.111
11	50	1.00	0.107	50	1.06	0.115
12	51	1.04	0.110	60	1.04	0.094
13	54	0.78	0.078	52	0.40	0.041
14	50	0.33	0.036	50	0.16	0.018
15	50	0.18	0.020	54	0.08	0.008
16	50	0.13	0.014	52	0.12	0.012
17	50	0.08	0.009	54	0.06	0.006
18	50	0.06	0.007	51	0.05	0.005
19	50	0.05	0.005	50	0.04	0.005
20	50	0.05	0.005	50	0.04	0.004
TOTAL	1013	60.35		1081	73.01	

TABLE 8

SOIL SEGMENTS AND EXTRACTS

SOIL WEIGHTS AND TOTAL C-14 AS % OF DOSE

SOIL 6073-43

		% OF DOSE		
SEGMENT NUMBER	DRY WEIGHT	SOIL BEFORE EXTRACTION	METHANOL EXTRACT	SOIL AFTER EXTRACTION
		COLUMN A		
1	75.74	2.04	0.99	0.99
2	78.07	0.89	0.08	1.05
3	81.42	0.87	0.06	0.90
4	89.34	0.93	0.07	0.98
5	103.40	1.05	0.03	0.48
TOTAL	427.98	5.79	1.23	4.40
		COLUMN B		
1	91.34	1.44	0.73	0.82
2	48.21	0.65	0.06	0.58
3	79.43	0.91	0.04	0.76
4	83.16	0.88	0.04	0.83
5	94.04	1.05	0.05	1.03
6	36.68	0.79	0.04	0.20
TOTAL	432.85	5.72	0.97	4.21

[redacted]

TABLE 7

SOIL SEGMENTS AND EXTRACTS

SOIL WEIGHTS AND TOTAL C-14 AS % OF DOSE

SOIL 6073-39

SEGMENT NUMBER	DRY WEIGHT	% OF DOSE SOIL BEFORE EXTRACTION	METHANOL EXTRACT	SOIL AFTER EXTRACTION
		COLUMN A		
1	122.55	2.05	0.38	1.38
2	133.04	2.64	0.14	2.11
3	138.72	2.78	0.09	1.93
4	135.45	2.27	0.09	2.00
5	141.21	1.75	0.06	1.59
6	119.37	0.57	0.01	0.61
TOTAL	811.15	12.05	0.76	9.62
		COLUMN B		
1	132.91	1.76	0.46	1.50
2	127.91	2.02	0.15	1.70
3	133.66	1.98	0.14	1.74
4	130.63	1.53	0.12	1.92
5	174.46	1.71	0.07	2.60
6	111.58	0.73	0.05	0.71
TOTAL	811.15	9.74	0.99	10.17

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[redacted]

TABLE 10

SOIL SEGMENTS AND EXTRACTS

SOIL WEIGHTS AND TOTAL C-14 AS % OF DOSE

SOIL 9143-05

SEGMENT NUMBER	DRY WEIGHT	% OF DOSE SOIL BEFORE EXTRACTION	METHANOL EXTRACT	SOIL AFTER EXTRACTION
		COLUMN A		
1	163.98	0.97	0.41	0.38
2	142.31	0.39	0.05	0.51
3	129.86	0.37	0.08	0.38
4	121.47	0.37	0.04	0.24
5	147.72	0.47	0.07	0.28
6	143.33	0.41	0.03	0.61
TOTAL	859.68	2.98	0.68	2.40
		COLUMN B		
1	150.27	0.48	0.32	0.49
2	152.23	0.53	0.04	0.31
3	134.51	0.44	0.02	0.21
4	133.22	0.44	0.02	0.32
5	137.57	0.39	0.03	0.30
6	105.97	0.52	0.10	0.18
TOTAL	813.77	2.80	0.53	1.81

TABLE 9

SOIL SEGMENTS AND EXTRACTS

SOIL WEIGHTS AND TOTAL C-14 AS % OF DOSE

SOIL 6073-48

SEGMENT NUMBER	DRY WEIGHT	% OF DOSE		
		SOIL BEFORE EXTRACTION	METHANOL EXTRACT	SOIL AFTER EXTRACTION
		COLUMN A		
1	143.98	4.31	0.34	3.96
2	131.31	2.63	0.18	2.46
3	126.06	2.47	0.10	1.48
4	121.09	2.34	0.09	1.66
5	109.15	2.62	0.08	1.33
TOTAL	631.59	14.37	0.78	10.89
		COLUMN B		
1	96.74	1.63	0.27	1.66
2	127.21	1.55	0.10	1.90
3	124.35	1.03	0.09	1.11
4	127.50	1.01	0.05	1.01
5	130.57	1.45	0.06	1.18
6	99.98	1.43	0.09	1.40
TOTAL	706.35	8.10	0.66	8.27

TABLE 12

CONCENTRATION OF METABOLITES IN LEACHATES

PPM CALCULATED AS NALED

SOIL 6073-39

FRACTION NUMBER	PPM			
	CARBONATE	DCE	DCAA	DDVP
	COLUMN A			
1				
2				
3				
4	0.007	0.001	0.175	
5	0.103	0.078	1.080	0.058
6	0.282	0.085	0.780	0.093
7	NA	NA	NA	NA
8	0.052	0.012	0.142	0.019
9	0.001	0.001	0.027	0.005
10	0.003		.000	0.005
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
	COLUMN B			
1				
2				
3				
4			0.023	
5	0.029	0.042	0.889	0.027
6	0.168	0.069	0.716	0.084
7	0.079	0.049	0.589	0.054
8	0.064	0.022	0.308	0.021
9	0.067	0.004	0.070	0.008
10	0.029	0.001	0.027	0.002
11			0.008	
12				
13				
14				
15				
16				
17				
18				
19				
20				

[redacted]

TABLE 11

SOIL SEGMENTS AND EXTRACTS

SOIL WEIGHTS AND TOTAL C-14 AS % OF DOSE

SOIL 8149-11

		% OF DOSE		
SEGMENT NUMBER	DRY WEIGHT	SOIL BEFORE EXTRACTION	METHANOL EXTRACT	SOIL AFTER EXTRACTION
		COLUMN A		
1	150.73	2.18	0.17	2.41
2	150.84	1.69	0.07	1.51
3	153.87	2.70	0.09	2.41
4	148.60	2.05	0.08	1.84
5	154.82	1.44	0.04	1.42
6	104.79	0.99	0.03	0.89
TOTAL	863.65	11.05	0.48	10.48
		COLUMN B		
1	141.79	1.81	0.11	2.41
2	153.14	1.34	0.05	1.51
3	147.98	1.40	0.04	2.41
4	159.77	2.55	0.08	1.84
5	152.06	2.23	0.08	1.42
6	100.15	0.98	0.03	0.89
TOTAL	854.89	10.32	0.40	10.48

TABLE 14

CONCENTRATION OF METABOLITES IN LEACHATES

PPM CALCULATED AS NALED

SOIL 6073-48

COLUMN A

FRACTION NUMBER	PPM			
	CARBONATE	DCE	DCAA	DDVP
1				
2				
3	0.018	0.001	0.208	0.001
4	0.043	0.006	0.348	0.009
5	0.066	0.006	0.384	0.010
6	0.073	0.008	0.206	0.003
7	0.080	0.007	0.111	
8	0.086	0.005	0.013	
9	0.058	0.002	0.005	
10	0.059	0.000	0.002	
11	0.036	0.000		
12	0.038	.000		
13	0.027	0.000		
14	0.019	.000		
15	0.016			
16	0.014			
17	0.010			
18	0.009			
19				
20				

COLUMN B

FRACTION NUMBER	CARBONATE	DCE	DCAA	DDVP
1				
2				
3	0.001		0.058	
4	0.117	0.005	0.469	0.006
5	0.100	0.028	1.476	0.032
6	0.210	0.031	0.973	0.040
7	0.098	0.014	0.430	0.032
8	0.063	0.004	0.220	0.018
9	0.056	0.001	0.132	0.009
10	0.053	0.001	0.077	0.004
11	0.051	.000	0.048	0.002
12	0.041	.000	0.023	0.001
13	0.030	0.000	0.021	0.001
14	0.036	0.001	0.011	0.001
15	0.020		.000	
16	0.023			
17	0.017			
18	0.016			
19	0.017			
20				

7.31
-~~6.29~~

TABLE 13

CONCENTRATION OF METABOLITES IN LEACHATES

PPM CALCULATED AS NALED

SOIL 6073-43

FRACTION NUMBER	PPM			
	CARBONATE	DCE	DCAA	DDVP
		COLUMN A		
1				
2				
3	0.004		0.398	0.001
4	0.003	0.001	0.606	0.001
5		0.001	0.664	0.004
6	0.006	0.001	0.724	
7	0.008	0.001	0.818	0.007
8	0.018	0.001	0.658	0.003
9	0.028	.000	0.503	
10	0.031	0.001	0.373	0.003
11	0.044		0.232	0.003
12	0.025		0.174	0.003
13				
14				
15				
16				
17				
18				
19				
20				
21				
		COLUMN B		
1				
2				
3	0.003	0.000	0.369	
4	0.003	0.001	0.650	0.001
5	0.002	0.001	0.790	0.001
6	0.012	0.001	0.910	0.002
7	0.014	0.002	1.005	0.004
8	0.031	0.002	0.739	0.003
9	0.036	0.001	0.485	0.007
10	0.049	0.001	0.243	0.006
11	0.031	.000	0.169	0.007
12	0.033		0.118	0.004
13				
14				
15				
16				
17				
18				
19				
20				
21				

[redacted]

TABLE 16

CONCENTRATION OF METABOLITES IN LEACHATES

PPM CALCULATED AS NALED

SOIL 8149-11

FRACTION NUMBER	PPM CARBONATE	PPM DCE	PPM DCAA	PPM DDVP
		COLUMN A		
1				
2				
3				
4	0.007	0.001	0.175	
5	0.103	0.078	1.080	0.058
6	0.282	0.085	0.780	0.093
7	NA	NA	NA	NA
8	0.052	0.012	0.142	0.019
9	0.001	0.001	0.027	0.005
10	0.003		.000	0.005
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
		COLUMN B		
1				
2				
3				
4			0.023	
5	0.029	0.042	0.889	0.027
6	0.168	0.069	0.716	0.084
7	0.079	0.049	0.689	0.054
8	0.064	0.022	0.308	0.021
9	0.067	0.004	0.070	0.008
10	0.029	0.001	0.027	0.002
11			0.008	
12				
13				
14				
15				
16				
17				
18				
19				
20				

7.33

- ~~6.31~~ -

[redacted]

TABLE 15

CONCENTRATION OF METABOLITES IN LEACHATES

PPM CALCULATED AS NALED

SOIL 8149-05

FRACTION NUMBER	PPM			
	CARBONATE	DCE	DCAA	DDVP
COLUMN A				
1				
2				
3	0.001	.000	0.364	
4	0.027	0.011	1.141	
5	0.066	0.016	1.863	0.040
6	0.100	0.014	1.018	0.055
7	0.093	0.004	0.492	0.032
8	0.092	0.001	0.252	0.017
9	0.073	0.000	0.090	0.000
10	0.039	0.000	0.082	0.000
11	0.072	0.000	0.032	.000
12	0.065	0.000	0.045	
13	0.041	0.000	0.037	
14	0.028	0.000	0.008	
15				
16				
17				
18				
19				
20				
21				
COLUMN B				
1				
2				
3	0.000	.000	0.058	
4	0.035	0.013	1.465	0.010
5	0.056	0.015	1.845	0.000
6	0.156	0.015	1.668	0.092
7	0.046	0.003	0.482	0.046
8	0.081		0.226	0.032
9	0.035		0.107	
10	0.000		0.000	
11	0.065		0.047	
12	0.046		0.047	
13	0.031		0.010	
14	0.012		0.006	
15				
16				
17				
18				
19				

7,32

3.6 and would cause naled to be classified as very mobile in soil. No calculation of K_d values for the degradate DDVP was presented. Therefore, this study is unacceptable.

In the previously reviewed study (Pack, 1980, MRID 00064796), [14C]naled (specific activity unspecified, Chevron Chemical Co.) was spotted on air-dried soil thin layer chromatography plates prepared from sieved (100-mesh) clay loam, sandy loam, loamy sand, and clay soils (Table 1) along with the reference compounds paraquat (class 1, immobile) and acephate (class 4-5, mobile). The plates were developed with deionized water and air dried. Autoradiograms were prepared for plate visualization and R_fs were measured.

In this new submission, in order to calculate K_d values, R_f values were remeasured at the center of the TLC spots, and then the following equation was used:

$$K_d = (1/R_f - 1)/(d_s - 1)$$

The above equation was derived from the Hamaker relationship between K_d and Rf:

$$R_f = 1/(1 + (K_{oc})(\%oc/100)(d_s)(1/ {}^{2/3} -1))$$

Where: = soil pore fraction = 0.5
d_s = density of the soil solid = 2.5
oc = organic carbon content of the soil
K_{oc} = adsorption coefficient on the basis of organic carbon = $(K_d/\%oc)(100)$
K_d = $(K_{oc})(\%oc/100)$

However, manipulation of the Hamaker equation yields the following equation:

$$K_d = (1/R_f - 1)/(d_s(1/ {}^{2/3} - 1)$$

Substitution of numerical values in both equations yields similar results for calculated K_d values; i.e. $(d_s(1/ {}^{2/3} - 1)$ is approximately equal to $(d_s - 1)$ when $d_s = 2.5$ and $= 0.5$.

DATA EVALUATION RECORD

NALED Study 8

CHEM 034401 Naled

BRANCH EAB

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40279200
Pack, D.E. 1987. Estimation of soil adsorption coefficient of naled from TLC data. Laboratory Project Identification MEF-0051/8711318. Unpublished study prepared and submitted by Chevron Chemical Co., Richmond, CA.

SUBST. CLASS = S

DIRECT RVW TIME = 5

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

EDITED BY: TITLE: Task Leader

APPROVED BY: TITLE: Project Manager

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

APPROVED BY: S. C. Termes
TITLE: Chemist
ORG: EAB/HED/OPP
TEL: 557-1981

SIGNATURE:

May 9th, 1988

CONCLUSIONS:

Mobility - Leaching and Adsorption/Desorption

This soil mobility (soil TLC) study (Pack, 1980, MRID 00064796) was originally reviewed (Dynamac Corporation) in a report dated August, 1982. The registrant has subsequently calculated K_d values from the R_f values observed in the 1980 study. The mobility classification from the 1980 study (based on R_f values) and the mobility classification based on K_d values calculated from those R_f values (remeasured at the center of the TLC spot) differ. Based on R_f values (1980 study), naled was classified as slightly mobile (Helling and Turner's mobility classes 2-3) and the degradate dichlorvos (DDVP) as moderately mobile (classes 3-4). In the recalculated K_d values for naled (MRID 40279200), the calculated K_d values ranged from 1.3 to

Naled

Page ______ is not included in this copy.

Pages _123_ through _126_ are not included in this copy.

The material not included contains the following type of information:

_____ Identity of product inert ingredients.

_____ Identity of product inert impurities.

_____ Description of the product manufacturing process.

_____ Description of product 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

__X__ FIFRA registration data.

_____ The document is a duplicate of page(s) _________

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

MATERIALS AND METHODS

RESULTS

Photographs of the autoradiograms are shown in Figures 1-4. The measured R_f's are given in Table 2. The data show that naled is slightly mobile (Helling and Turner's mobility classes 2-3) and that dichlorvos is moderately mobile (classes 3-4).

RESULTS

The remeasured R_f values and the calculated K_D values are given in Table 2. The K_D ranges from 1.3 to 3.6.

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

TABLE 2

Frontal R_f Values Found

Compound	Frontal R_f			
	Blendon sandy loam	Nicolett clay loam	Oakley loamy sand	Stockton adobe clay
Naled	0.48	0.28	0.41	0.48
Dichlorvos	0.80	0.56	0.80	0.80
Paraquat	0.02	0.02	0.02	0.02
Acephate	0.93	0.88	0.97	0.93

8.11
- 7.11

PERTINENT DATA TABLES AND FIGURES

TABLE 2

CALCULATED K_D VALUES

SOIL	R_f	K_D
Blendon sandy loam	0.35	1.3
Nicolett clay loam	0.16	3.6
Oakley loamy sand	0.28	1.8
Stockton adobe clay	0.16	3.6

TABLE 1

Classification, Properties and Source of Soils Used for Thin-Layer Chromatograms

Soil Classification (Series, Texture)	Blendon sandy loam	N t c n	Oakley loamy sand	Stockton adobe clay
pH	5.6	7.	7.3	4.5
% organic matter	1.4	.7	1.4	2.4
% sand	68	23	85	28
% silt	16	40	6	26
% clay		37	9	46
Water Holding Capacity (%)	1.	38	2.4	34
Cation Exch. Cap. (meq/100 g)	7.	21	7.5	25
Field Source	Merrick C ty NE	Dallas Center IA	Oakley CA	Biggs CA

8,12

clay loam, sandy loam, loamy sand, and two sand soils; Freundlich K (adsorption) values were 0.015-0.46. The study was conducted in 1:5 soil:solution slurries (1 mM calcium sulfate) slurries with dichloroacetic acid concentrations of 0.1, 0.2, 0.5, and 1.0 ppm.

DISCUSSION:

1. The soil:solution slurries were equilibrated for one hour; however, preliminary studies were not conducted to determine if equilibration was in fact established in one horu. The registrants referenced two batch equilibrium studies using lindate and RE-20615 as evidence that one hour is sufficient for equilibration. Since equilibration time is dependent on several factors including the pesticide being studied, the soil:solution ration, and the termperature during the study, these references cannot be accept as evidence that one hour is sufficient to establish equilibrium of dichloroacetic acid solution:soil slurries under these experimetnal conditions.

2. The study was conducted using 2mM (0.001 M) calcium sulfate solutions instead of 0.01 M calcium ion solutions.

3. The desorption phase for this degradate was not studied, and, therefore, no K (desorption) values were calculated.

DATA EVALUATION RECORD

NALED STUDY 9

CHEM 034401 Naled

BRANCH EAB

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40034905 CONTENT CAT 01
Pack, D. E. 1986.
Freundlich adsorption isotherms of dichloroacetic acid.
Laboratory Project ID: 8614820. Unpublished study prepared and submitted by Chevron Chemical Company, Richmond, CA.

SUBST. CLASS = S

DIRECT RVW TIME = 2

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

EDITED BY: TITLE: Staff Scientist

APPROVED BY: TITLE: Program Manager

ORG: Dynamac Corp., Rockville, MD
TEL: 468-2500

APPROVED BY: S. C. Termes
TITLE: Chemist
ORG: EAB/HED/OPP
TEL: 557-1981

SIGNATURE: May 19th, 1988

CONCLUSIONS:

Mobility - Leaching and Adsorption

This study is unacceptable because it was not established that the equilibration time of one hour was sufficient for the soil:solution slurries to reach equilibrium. In addition, this study would not fulfill EPA Data Requirements for Registering Pesticides because desorption of this degradate was not addressed. Dichloroacetic acid (DCAA) is a major degradate found in leachates (soil column study).

SUMMARY OF DATA BY REVIEWER:

Based on batch equilibrium studies, 1.2-[^{14}C]Dichloroacetic acid (radiochemical purity 96%, specific activity 364,3000 dpm/ug), was very mobile in

Naled

Page ______ is not included in this copy.

Pages _136_ through _138_ are not included in this copy.

The material not included contains the following type of information:

_____ Identity of product inert ingredients.

_____ Identity of product inert impurities.

_____ Description of the product manufacturing process.

_____ Description of product 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

__X__ FIFRA registration data.

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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.

MATERIALS AND METHODS

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

RESULTS

The adsorption data are given in Table 2. Log-log plots are shown in Figures 1-5.

The adsorption of solutes on solids can be described by the Freundlich adsorption equation (8):

$$\frac{x}{m} = KC^{1/n} \qquad (1)$$

Where x = weight of adsorbed chemical
m = weight of solid adsorbent
C = concentration in equilibrium solution
K and n are constants

Values for K and 1/n may be found by taking logarithms of both sides of equation (1):

$$\log \frac{x}{m} = \log K + \frac{1}{n} \log C \qquad (2)$$

A plot of log x/m vs. log C should give a straight line, with slope 1/n and intercept log K. If x/m is in units of µg/g and C in µg/ml, then K is the µg of chemical adsorbed per gram solid when the equilibrium solution concentration is 1 µg/ml.

The Freundlich adsorption constants were calculated by linear regression analysis on the log transformed data according to equation (2). The calculated K and 1/n values are included in Tables 2-4. The correlation coefficients R were also calculated by the linear regression analysis and are also given in the tables. The closer R is to unity, the better the correlation between x/m and C is. Note that the correlation is excellent in all cases.

K is a measure of the amount adsorbed which is in equilibrium with a solution of unit concentration, the higher the value of K, the stronger the adsorption. Thus, K-values compare both the capacity of different soils to adsorb a particular chemical and the relative adsorbability of different chemicals on the same soil.

The values show that DCAA is weakly adsorbed. These results corrobrate completely the results found in the soil column leaching study (1).

ACKNOWLEDGEMENT

I thank A. D. Hunziker and C. E. Fry for technical assistance.

NOTEBOOK REFERENCE: 9248

D E Pack

D. E. PACK

9.8

PERTINENT DATA TABLES AND FIGURES

REFERENCES

1. Pack, D. E., **Soil Column Leaching of [Ethyl-1-^{14}C]naled (DIBROM),** Chevron Chemical Company report, June 19, 1986.

2. Pack, D. E., **The Soil Metabolism of ^{14}C-Naled (DIBROM),** Chevron Chemical Company report, November 25, 1980.

3. Pack, D. E., **The Aerobic Aquatic Metabolism of [Ethyl-1-^{14}C]Naled (DIBROM),** Chevron Chemical Company report, May 12, 1986.

4. Pack, D. E., **Mobility of Naled and Dichlorvos in Soil as Determined By Soil Thin-Layer Chromatography,** Chevron Chemical Company report, August 29, 1980.

5. Pack, D. E., **[Ethyl-1,2-^{14}C]captafol (DIFOLATAN) Hydrolysis Products,** Chevron Chemical Company report, February 20, 1986.

6. Kay, B. D., **Adsorption and Movement of Lindane in Soils, Soil Science,** 104, 314 (1967).

7. Warnock, R. W., **Adsorption of RE-20615 on Soil,** Chevron Chemical Company report, December 22, 1977.

8. Freundlich, H., Colloid and Capillary Chemistry, Methuen, London, 1926.

9.9

TABLE 2

FREUNDLICH ISOTHERM CONSTANTS

Soil	Initial Aqueous ppm	Counts*	Equilibrium Concentration		K	l/n	r**
			Solution µg/mL	Soil µg/g			
03 Sand (Cranberry bog)	0.1	13694	0.0902	0.0488	0.46	0.95	0.9988
	0.2	13871	0.1828	0.0860			
	0.5	13851	0.4563	0.2183			
	1.0	13884	0.9149	0.4257			
05 Sand	0.1	14859	0.0966	0.0170	0.17	1.04	0.9391
	0.2	14838	0.1929	0.0353			
	0.5	14902	0.4844	0.0778			
	1.0	14850	0.9655	0.1725			
07 Loamy Sand	0.1	15047	0.0978	0.0111	0.15	1.17	0.9947
	0.2	15085	0.1960	0.0199			
	0.5	15016	0.4878	0.0608			
	1.0	14900	0.9681	0.1594			
43 Clay Loam	0.1	15369	0.0943	0.0287	0.3191	1.072	0.9870
	0.2	15591	0.1912	0.0438			
	0.5	15293	0.4690	0.1552			
	1.0	15325	0.9399	0.3005			
48 Sandy Loam	0.1	15217	0.0962	0.0188	0.24	1.14	0.9719
	0.2	15280	0.1933	0.0336			
	0.5	15256	0.4825	0.0876			
	1.0	14935	0.9447	0.2767			

* - Average of duplicate samples
** - r = correlation coefficient

TABLE I

SOIL CHARACTERISTICS

Soil Code	03	05	05	43	48
Reference No.	8149-03	8149-05	8149-07	6073-43	6073-48
Source	MS (Cranberry Bog)	Ocoee, FL	NC	Dallas Center, IA	Greenville MS
Classification	Sand	Sand	Loamy Sand	Clay Loam	Sandy Loam
% Sand	92	92	88	30	70
% Silt	6	4	6	38	17
% Clay	2	4	6	32	13
% Organic	4.4	1.9	1.6	2.8	1.0
CEC (Meq/100g)	3.5	2.9	2.5	27.8	7.5
pH	4.3	6.7	6.2	8.1	7.1
1/3 Bar %	7.81	4.39	5.56	32.3	13.4
Bulk Density g/mL	1.13	1.47	1.44	1.01	1.23

FIGURE 2

SOIL 8149-05

FREUNDLICH SOIL ADSORPTION ISOTHERM

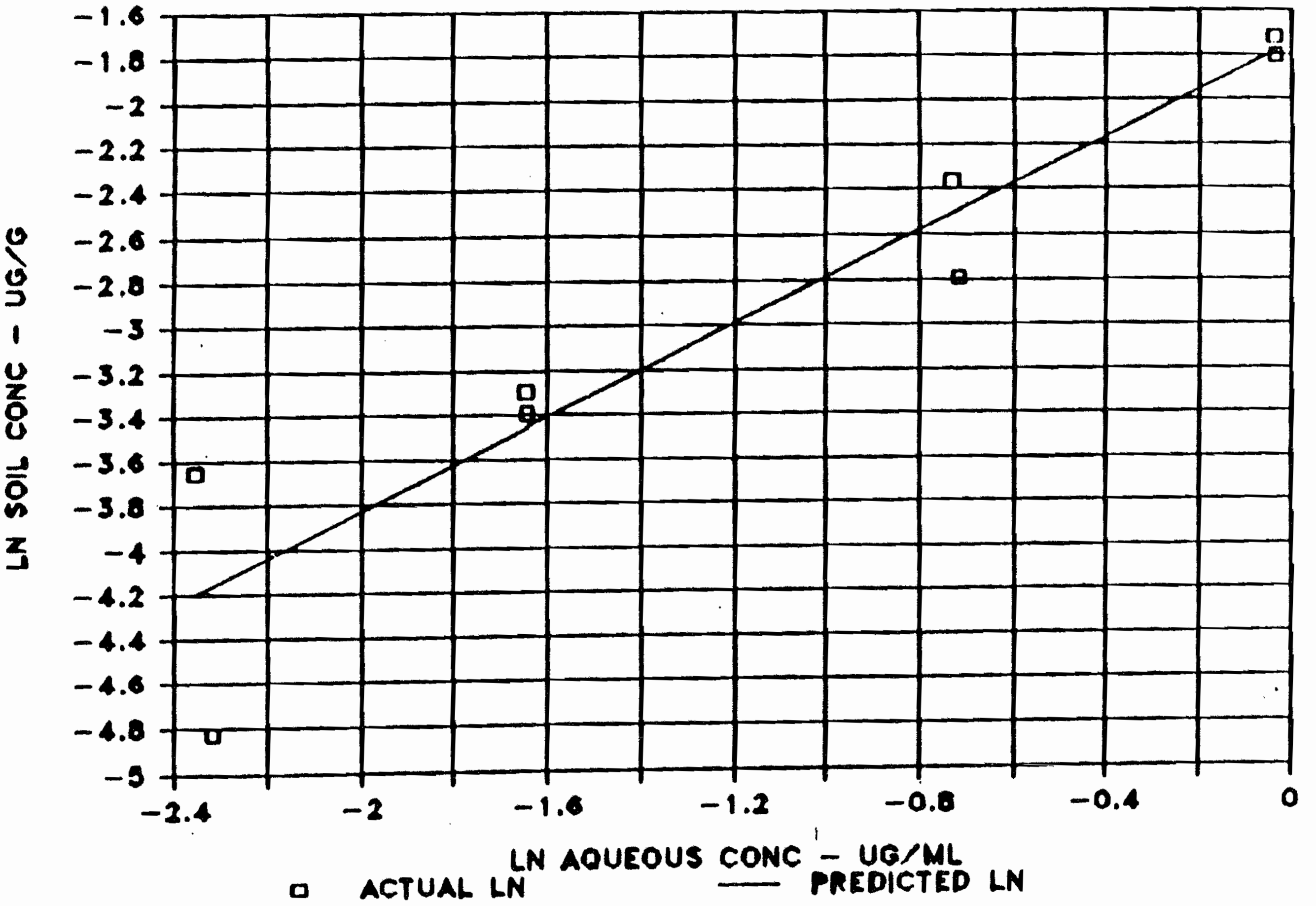

FIGURE 1

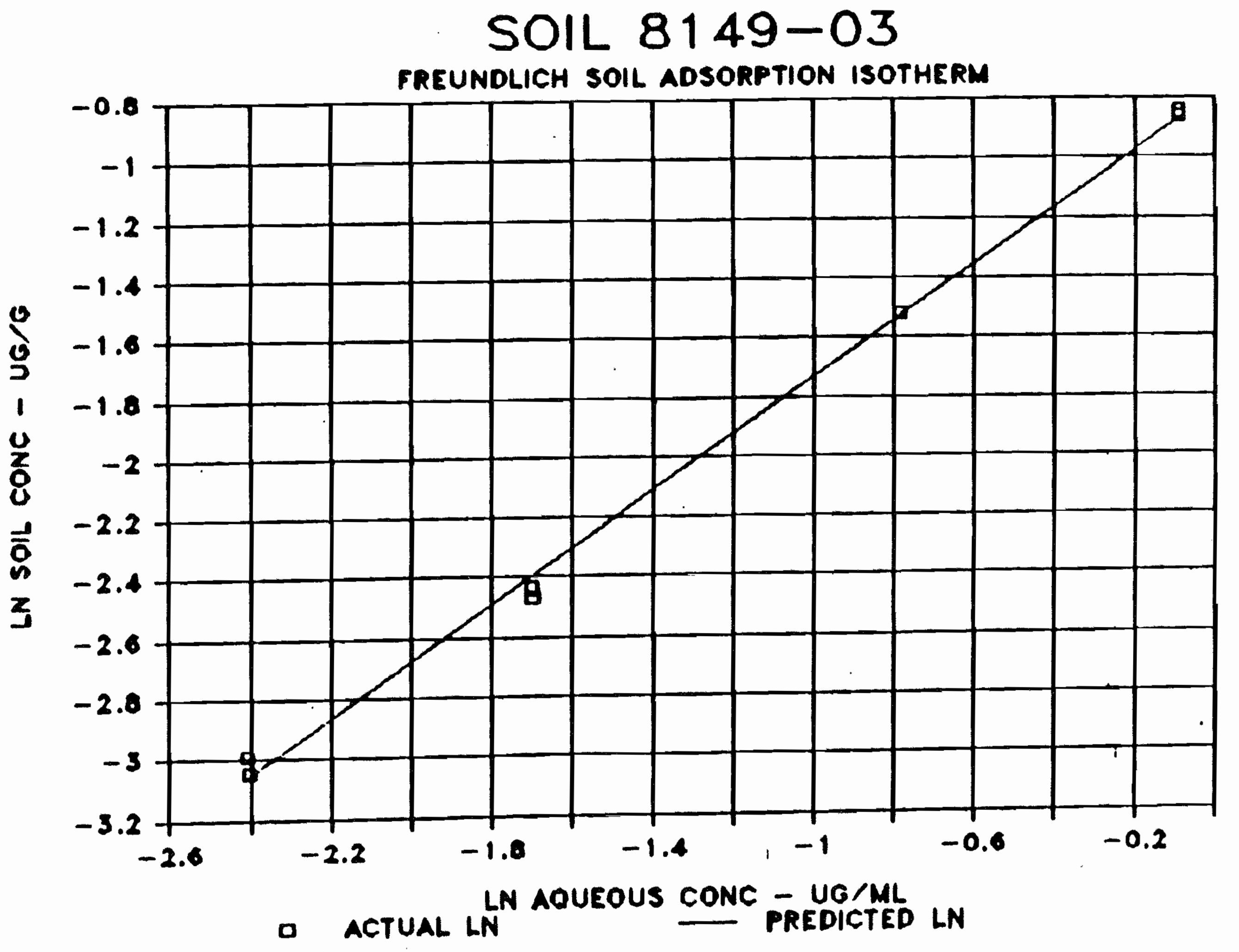

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FIGURE 3

SOIL 8149−07

FREUNDLICH SOIL ADSORPTION ISOTHERM

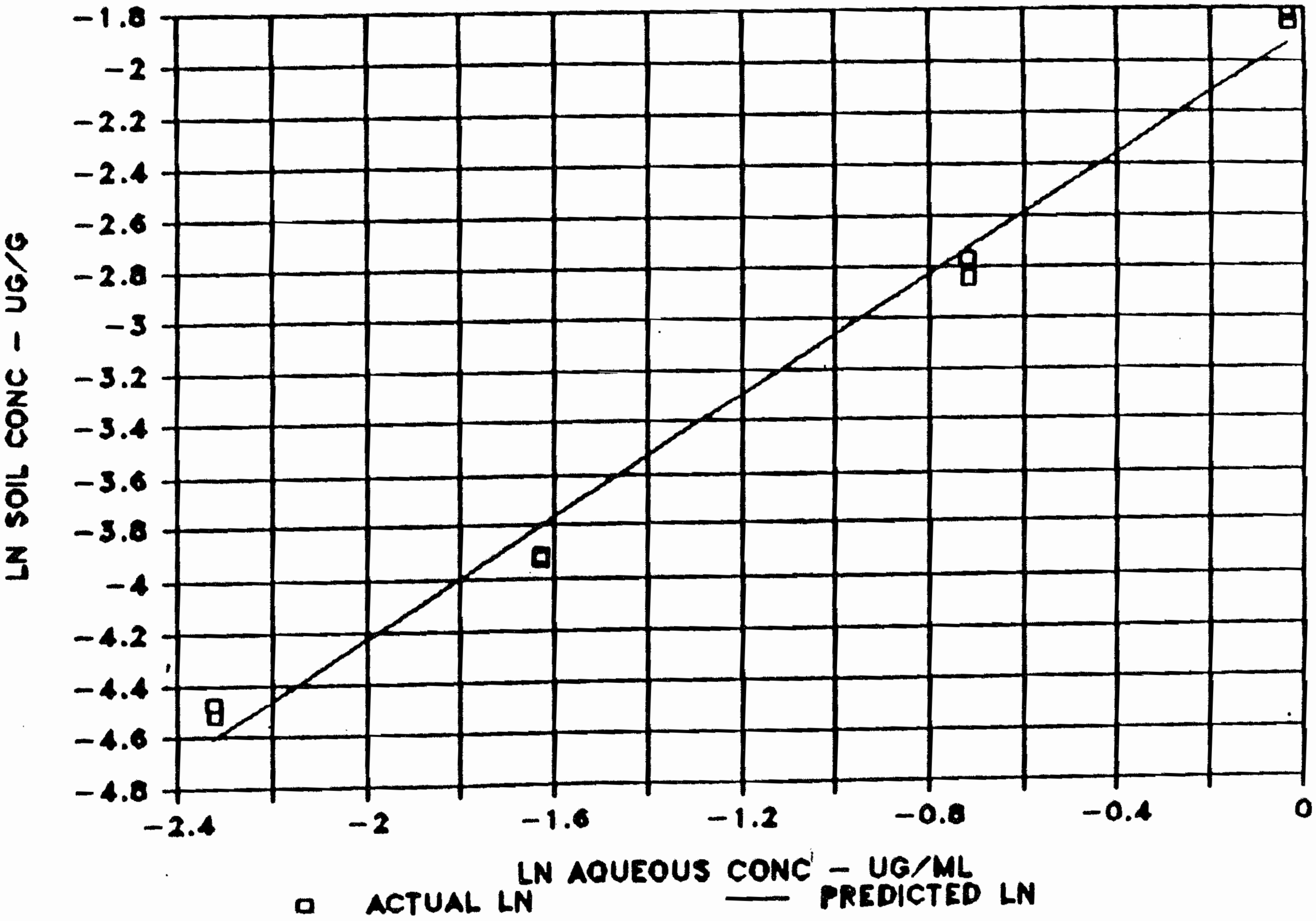

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FIGURE 4

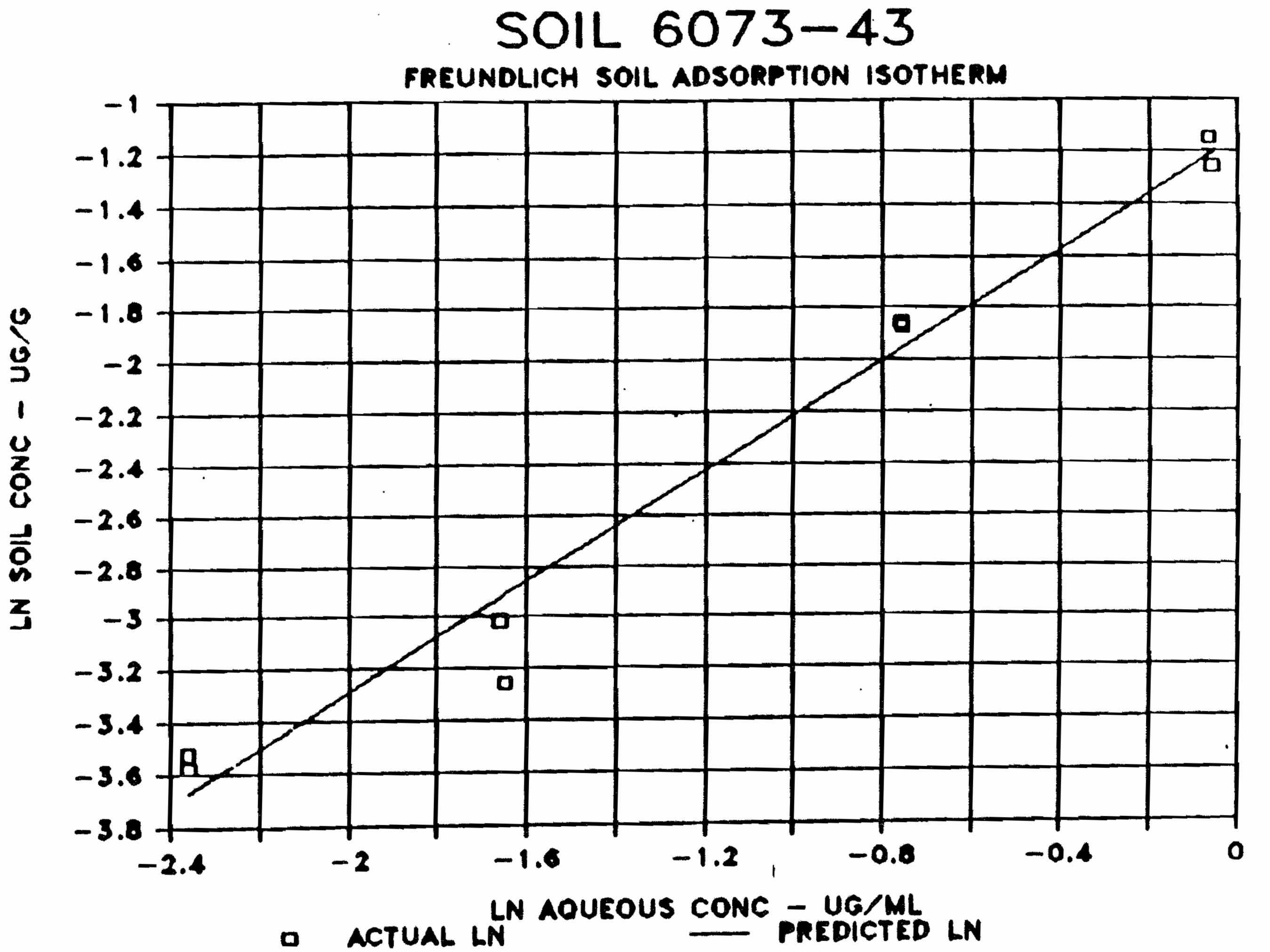
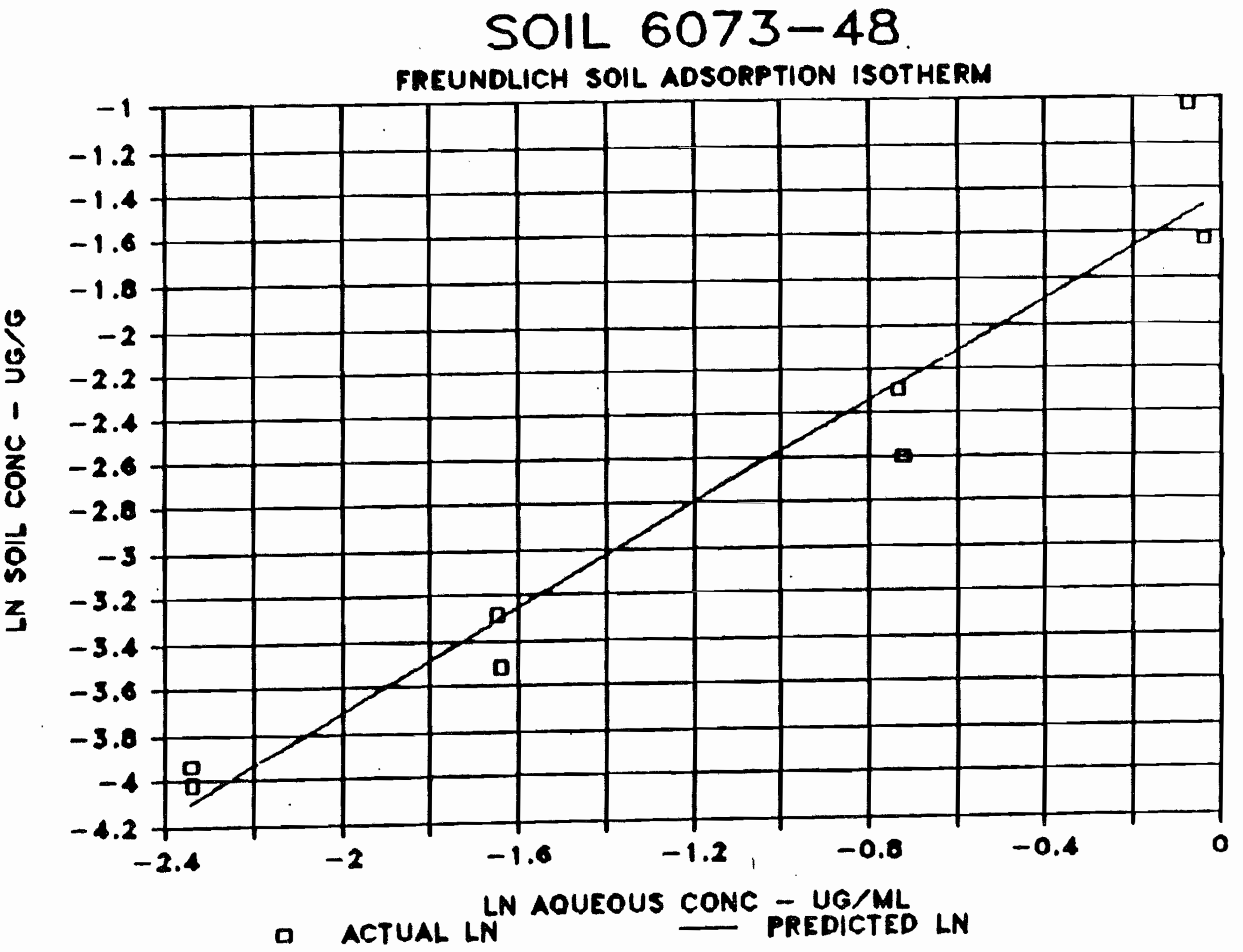

SOIL 6073−43
FREUNDLICH SOIL ADSORPTION ISOTHERM
LN SOIL CONC − UG/G
−1
−1.2
−1.4
−1.6
−1.8
−2
−2.2
−2.4
−2.6
−2.8
−3
−3.2
−3.4
−3.6
−3.8
−2.4
−2
−1.6
−1.2
−0.8
−0.4
0
LN AQUEOUS CONC − UG/ML
□ ACTUAL LN
—— PREDICTED LN

FIGURE 5


SOIL 6073-48
FREUNDLICH SOIL ADSORPTION ISOTHERM
LN SOIL CONC - UG/G
-1
-1.2
-1.4
-1.6
-1.8
-2
-2.2
-2.4
-2.6
-2.8
-3
-3.2
-3.4
-3.6
-3.8
-4
-4.2
-2.4
-2
-1.6
-1.2
-0.8
-0.4
0
LN AQUEOUS CONC - UG/ML
□ ACTUAL LN
PREDICTED LN

DATA EVALUATION RECORD

CASE GS -- NALED STUDY 10 PM

CHEM 034401 Naled

BRANCH EAB

FORMULATION 00- DIBROM 14 Concentrate

Lee, S-L. 1988. "Aquatic field dissipation of DIBROM". Laboratory Project ID: R196T71178. Prepared and submitted by Chevron Chemical Company, Richmond, CA. EPA Accession No. 40494101

SUBST. CLASS:

DIRECT RVW TIME: (MH) START DATE END DATE

REVIEWED BY: S.C. Termes
TITLE: Chemist
ORG: EAB/HED/OPP
TEL: (703)557-1981

APPROVED BY: S.C. Termes
TITLE: Chemist
ORG: EAB/HED/OPP
TEL: (703)557-1981

SIGNATURE: May 19th, 1988

CONCLUSIONS:

Field Dissipation- Aquatic

This study is unacceptable as presented. The study may be acceptable if the registrant submits additional information and the submitted information is acceptable to the Agency.

SUMMARY OF DATA BY REVIEWER:

Two test sites (farm ponds in Titusville, FL and in Lexington, MS), were chosen for the study. Each test site had one treated and one untreated pond about one half acre each. DIBROM 14 CONCENTRATE (85% a.i.), applied in five aerial applications at a maximum label rate of 0.4 lb a.i./A in a period of three weeks, was used as the test material. Levels of naled and its degradate DDVP were measured in pond water and soil/sediment cores (down to 15 cm). Collected water samples were analyzed within 24-hr of being received (some samples were analyzed within 8-days of receiving; stability data for naled and DDVP in frozen water samples indicate that these materials are stable for up to 8-days). Levels of naled and DDVP in pond water (for both sites) were below 0.1 ppm. No naled or DDVP were detected in sediments (detection limit 0.01 ppm). It was determined that naled and DDVP were unstable in frozen sediment samples (in 6-days of storage, 50% or less remained in samples). No dissipation rates were established because the residues in pond water were two low and because no residues were found in sediment samples.

DISCUSSION:

1. No data on soil/sediment characteristics were included in the report.

2. No analytical data was provided for pond water.

3. Although in the description of the sampling procedures it is said that "upon retrieval of the core, the samples were separated into 0-7.5 cm and 7.5-15 cm sections" (then the respective sections at each sampling site were combined), no data for each of these separate sections appear in the report.

4. Dichloroacetic acid (DCAA) and dichloroethanol (DCE) were identified as degradation products in both aerobic/anaerobic aquatic metabolism studies, but no apparent attempts were made to identify these metabolites in the aquatic field dissipation study.

5. The test sites were not satisfactorily described.

AQUATIC FIELD DISSIPATION OF DIBROM

Summary of Naled and DDVP Residues in Pond Water Following Five Aerial Applications of DIBROM 14 C at 0.4 lb ai/A to the test site in Floria (T-7117)

Treatment to Sampling Interval	Naled	DDVP	Total as naled equivalents
		*(ppb)	
Pretreatment	nd	nd	nd
First Application (November 10, 1987)			
1 hr	6	4	13
1 day	2	6	10
Second Application (November 12, 1987)			
1 hr	9	4	16
1 day	nd	2	3
Third Application (November 16, 1987)			
1 hr	1	2	4
1 day	nd	5	9
Fourth Application (November 19, 1987)			
1 hr	3	2	6
1 day	nd	nd	nd
Fifth Application (November 23, 1987)			
1 hr	4	1	6
2 hr	7	2	10
4 hr	6	4	13
6 hr	5	2	8
8 hr	4	4	11
1 day	1	5	10
2 days	nd	1	2
3 days	nd	nd	nd
4 days	nd	nd	nd
7 days	nd	nd	nd

nd - not detectable (<1 ppb)
<u>Test site</u> - Titusville, Florida
* Average value of samples collected at two quadrants and upper, mid-depth, lower levels (see Appendix B protocol for details of sampling procedure).

AQUATIC FIELD DISSIPATION OF DIBROM

Summary of Naled and DDVP Residues in Pond Water Following Five Aerial Applications of DIBROM 14 C at 0.4 lb ai/A to the test site in Mississippi (7118)

Treatment to sampling interval	Naled	DDVP	Total as naled equivalents
		*(ppb)	
Pretreatment	nd	nd	nd
First Application (November 2, 1987)			
1 hr	nd	1	2
1 day	nd	nd	nd
Second Application (November 5, 1987)			
1 hr	nd	nd	nd
1 day	nd	nd	nd
Third Application (November 9, 1987)			
1 hr	nd	1	2
1 day	nd	1	2
Fourth Application (November 12, 1987)			
1 hr	1	4	8
1 day	nd	3	5
Fifth Application (November 17, 1987)			
1 hr	3	3	8
2 hr	2	4	9
4 hr	1	5	10
6 hr	nd	4	7
8 hr	nd	5	9
1 day	nd	4	7
2 days	nd	3	5
3 days	nd	2	3
4 days	nd	nd	nd
7 days	nd	nd	nd
10 days	nd	nd	nd
14 days	nd	nd	nd

nd - not detectable (<1 ppb)
<u>Test site</u> - Lexington, Mississippi.
* Average value of samples collected at two quadrants and upper, mid-depth, lower levels (see Appendix B protocol for details of sampling procedure).

No residue of naled or DDVP was detected in sediment samples collected from both test sites. The following table provides information about frequency of applications and samplings.

Summary of Naled and DDVP Residues in Sediment Following Five Aerial Applications of DIBROM 14 C at 0.4 lb ai/A to test sites in Florida and Mississippi

Treatment to sampling interval	Residues detected Naled	 DDVP
<u>T- 7117</u> <u>Titusville,</u> <u>Florida</u>		
Pretreatment	nd	nd
First Application (November 10, 1987)		
1 hr	nd	nd
1 day	nd	nd
Second Application (November 12, 1987)		
1 hr	nd	nd
1 day	nd	nd
Third Application (November 16, 1987)		
1 hr	nd	nd
1 day	nd	nd
Fourth Application (Novemver 19, 1987)		
1 hr	nd	nd
1 day	nd	nd
Fifth Application (November 23, 1987)		
1 hr	nd	nd
7 days	nd	nd
<u>T- 7118</u> <u>Lexington,</u> <u>Mississippi</u>		
pretreatment	nd	nd
First Application (November 2, 1987)		
1 hr	nd	nd
1 day	nd	nd
Second Application (November 5, 1987)		
1 hr	nd	nd
1 day	nd	nd
Third Application (November 9, 1987)		
1 hr	nd	nd
1 day	nd	nd
Fourth Application (Novemver 12, 1987)		
1 hr	nd	nd
1 day	nd	nd
Fifth Application (November 17, 1987)		
1 hr	nd	nd
7 days	nd	nd
10 days	nd	nd
14 days	nd	nd

nd - not detectable (<0.01 ppm)

Naled

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Pages _155_ through _168_ are not included in this copy.

The material not included contains the following type of information:

_____ Identity of product inert ingredients.

_____ Identity of product inert impurities.

_____ Description of the product manufacturing process.

_____ Description of product 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

__X__ FIFRA registration data.

_____ The document is a duplicate of page(s) _________

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

DATA EVALUATION RECORD

NALED STUDY 11

CHEM 034401 Naled

BRANCH EAB

FORMULATION--12--EMULSIFIABLE CONCENTRATE (EC)

FICHE/MASTER ID 40304301 CONTENT CAT 01

Selman, F. and Williams, M. 1987. Dissipation study on Dibrom 14 concentrate for forestry uses. ABC Report No. 33770. Prepared by Analytical Bio-Chemistry Laboratories, Inc., Columbia, MO; and submitted by Chevron Chemical Co., Richmond, CA.

SUBST. CLASS = S

DIRECT RVW TIME = 9

REVIEWED BY: K. Patten TITLE: Staff Scientist

EDITED BY: TITLE: Staff Scientist

APPROVED BY: TITLE: Program Manager

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TEL: 468-2500

APPROVED BY: S. C. Termes
TITLE: Chemist
ORG: EAB/HED/OPP
TEL: 557-1981

SIGNATURE: May 19th, 1988

CONCLUSIONS:

Field Dissipation - Forestry

This study is scientifically sound and provides supplemental information towards the registration of naled. This study does not fulfill EPA Data Requirements for Registering Pesticides because insufficient data were provided to accurately establish a pattern of dissipation of naled and its primary degradate DDVP in a forest environment, and no frozen storage stability data were provided to confirm that samples did not degrade prior to analysis.

SUMMARY OF DATA BY REVIEWER:

A 24-A loblolly pine forest in Madison County, Georgia, was treated aerially with naled (Dibrom 14 Concentrate, 14% EC) at 0.4 lb ai/A on November 11, 1985. Prior to spraying, cellulose filter paper sheets had been placed at the top of the canopy, at shrub height (approximately 5 feet), and at ground level. At 1-2 hours posttreatment, naled on the filter paper ranged from <0.01 to 0.15 ug/cm^2 and DDVP (as naled equivalents) ranged from <0.01 to 0.24 ug/cm^2. In general, the higher concentrations were in samples from the top of the canopy. Naled plus DDVP were <5 ppb in stream (0, 1, and 3 days posttreatment) and pond water (0, 1 and 7 days posttreatment) samples, and <50 ppb in exposed and litter-covered soil (0- to 3- and 3- to 6-inch depths; 0, 1, 3, and 5 days posttreatment) and stream sediment (0 days posttreatment).

DISCUSSION:

1. Insufficient data were provided to accurately determine the pattern of dissipation of naled and DDVP in the forest environment. Many of the 0-7 day samples could not be analyzed because of problems with the analytical method (terpenes in the highly organic samples interfered with and eventually destroyed the GC column).

2. Although the protocol specified that foliage, water, leaf litter, sediment, and soil were to be sampled at 0, 1, 3, 5, 7, 30, and 90 days after treatment, the registrant stated that the analyses were terminated with the 3-7 day samples when no residues were found (except on the filter paper).

3. The first significant rainfall did not occur until days 9 and 10 posttreatment (0.91 and 1.06 inches, respectively), at which time little or no naled and DDVP probably remained on the foliage (based on data from the photodegradation on plant leaves and aerobic soil metabolism studies the half-lives of naled and DDVP were <1 day). The only rainfall received prior to day 9 was on day 1, when 0.15 inches of rain were received in three distinct events; it is likely that the day 1 rainfall was insufficient to wash the leaves and transport the washes to the forest floor.

3. Samples and/or sample extracts were stored frozen for various lengths of time before analysis. However no storage stability data were provided to show that naled and DDVP were stable in the samples and samples extracts for the length of storage. The study author did state that after 7 months of freezer storage, the naled in samples of the solution applied to the forest site had degraded to low levels.

4. Although the registrant stated that soil was sampled from the 0- to 3-, 3- to 6-, 6- to 9-, and 9- to 12-inch depths, only data from the 0- to 3-and 3- to 6-inch depths were reported.

5. Data on the effect of naled on insect population was not reviewed because it is not pertinent to the EAB.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Twenty-four acres of a loblolly pine forest located in Madison County, Georgia, were selected for the study. The area to be treated contained both a stream and a pond, was 600-680 feet in elevation with a 6-15% slope, and was underlain with deep (>60 inches) sandy loam and sandy clay loam soils. For sampling purposes, the area was divided into quadrants. Prior to treatment, 100 m^2 areas in each quadrant were cleared of vegetation to provide exposed soil. Cellulose filter paper sheets (18 x 11 inches) were placed at the top of the canopy, at shrub height (approximately 5 feet), and at ground level; at least one sheet was placed in the cleared area in each quadrant. Naled (Dibrom 14 Concentrate, 14% EC, Chevron Chemical Company), at 0.4 lb ai/A, was sprayed onto the site by aircraft on November 11, 1985. For safety, the area was not entered for sampling until 1-2 hours posttreatment.

The filter paper sheets were collected, placed in individual sample bottles, and stored on dry ice. Foliage samples from the top, middle, and lower third of the loblolly pine canopy (six per quadrant); leaf litter (three per quadrant); exposed and litter-covered soil (0- to 3- and 3- to 6-inch depths); stream and pond water; and stream and pond sediment were collected from the treated site and from a similar control site up to 84 days posttreatment. Samples were placed on dry ice, then frozen until analysis. Soil, leaf litter, and foliage samples were sampled while frozen; sediment samples were allowed to thaw prior to analysis.

Water samples were extracted three times with hexane:methylene chloride (10:90); the extract was concentrated and analyzed using GLC. Soil, sediment, leaf litter, and foliage samples were extracted twice by shaking with hexane:methylene chloride (10:90); the extracts were analyzed using GLC. Some samples required additional cleanup and were analyzed using gel permeation chromatography. The detection limits for naled and DDVP were 0.5 ng/cm^2 for filter paper, 1 ppb for water, and 25 ppb for soil. Recovery from fortified soil ranged from 33 to 92% for naled and from 94 to 156% for DDVP. Recovery from fortified water samples ranged from 80 to 146% for naled and from 70 to 103% for DDVP.

Naled

Page ______ is not included in this copy.

Pages _173_ through _179_ are not included in this copy.

The material not included contains the following type of information:

_____ Identity of product inert ingredients.

_____ Identity of product inert impurities.

_____ Description of the product manufacturing process.

_____ Description of product 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

__X__ FIFRA registration data.

_____ The document is a duplicate of page(s) _________

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

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

Naled

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Pages _181_ through _182_ are not included in this copy.

The material not included contains the following type of information:

_____ Identity of product inert ingredients.

_____ Identity of product inert impurities.

_____ Description of the product manufacturing process.

_____ Description of product quality control procedures.

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_____ Sales or other commercial/financial information.

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_____ The product confidential statement of formula.

_____ Information about a pending registration action

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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.

PERTINENT DATA TABLES AND FIGURES

TABLE 1

Soil Characterization Data.

	Soil Depth (inches)			
	0-12	12-24	24-36	36-48
Madison Sandy Loam				
% Organic Matter	0.80	0.27	0.07	0.07
pH	5.4	5.5	5.5	5.5
CEC (meq/100 g)	5.9	5.2	3.6	3.5
% Sand	36.0	34.0	52.0	58.0
% Silt	18.0	24.0	28.0	30.0
% Clay	46.0	42.0	20.0	12.0
Texture	Clay	Clay	Sandy Loam	Sandy Loam
Cecil Sandy Loam				
% Organic Matter	1.50	0.20	0.20	0.27
pH	5.1	5.5	5.3	5.2
CEC (meq/100 g)	4.3	4.4	5.0	5.1
% Sand	64.0	56.0	44.0	36.0
% Silt	20.0	14.0	22.0	22.0
% Clay	16.0	30.0	34.0	42.0
Texture	Sandy Loam	Sandy Clay Loam	Sandy Clay Loam	Clay

11.16
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Table 1. Naled and DDVP (µg/cm^2 as naled equivalents) on filter paper surfaces 1-2 hours after a loblolly pine forest was sprayed with naled (Dibrom 14 Concentrate) at 0.4 oz ai/A.[a]

Site	Replicate	Total	Naled	DDVP as naled
		Top of canopy		
1	A	0.31	0.07	0.24
	B	0.14	0.02	0.12
	C	0.07	0.02	0.05
2	A	0.02	0.02	<0.01
	B	0.07	0.04	0.03
	C	0.13	0.06	0.07
3	A	0.30	0.15	0.15
	B	0.18	0.06	0.12
	C	0.22	0.13	0.09
4	A	<0.01	<0.01	<0.01
	B	<0.01	<0.01	<0.01
	C	<0.01	<0.01	<0.01
		Shrub height (~5 feet)		
1	A	0.08	0.06	0.02
	B	0.10	0.07	0.03
	C	0.06	0.04	0.02
2	A	0.06	0.04	0.02
	B	0.08	0.06	0.02
	C	0.04	0.04	<0.01
3	A	0.14	0.11	0.03
	B	0.10	0.08	0.02
	C	0.17	0.14	0.03
4	A	<0.01	<0.01	<0.01
	B	<0.01	<0.01	<0.01
	C	<0.01	<0.01	<0.01
		Ground level under canopy		
1	A	0.05	0.03	0.02
	B	0.11	0.06	0.05
	C	0.10	0.07	0.03
2	A	0.10	0.07	0.03
	B	0.10	0.07	0.03
	C	0.07	0.05	0.02
3	A	0.15	0.10	0.05
	B	0.01	0.01	<0.01
	C	0.01	0.01	<0.01
4	A	<0.01	<0.01	<0.01
	B	<0.01	<0.01	<0.01
	C	<0.01	<0.01	<0.01
		Ground level (exposed surface)		
1	--	0.11	0.01	0.10
2	--	0.10	0.01	0.09
3	--	0.08	0.01	0.07
4	--	0.16	0.02	0.14

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[a] Data are reformated from pages 9-10 in the original document.

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TABLE 9

Analysis of Stream and Pond Water and Stream Sediment for Naled and DDVP.

Sample Code	Test Day	Location	Naled (ppb)	DDVP as Naled[1] (ppb)	Total (ppb)
0-T-1-WS-A	0	Stream	<5	<5	<5
0-T-1-WS-B	0	Stream	<5	ND	<5
1-T-1-WS-A	1	Stream	ND	<5	<5
1-T-1-WS-B	1	Stream	ND	ND	ND
0-T-2-WS-A	0	Stream	<5	<5	<5
0-T-2-WS-B	0	Stream	<5	<5	<5
1-T-2-WS-A	1	Stream	ND	ND	ND
1-T-2-WS-B	1	Stream	ND	ND	ND
0-T-3-WS-A	0	Stream	<5	ND	<5
0-T-3-WS-B	0	Stream	ND	<5	<5
1-T-3-WS-A	1	Stream	ND	ND	ND
1-T-3-WS-B	1	Stream	<5	ND	<5
0-T-4-WS-A	0	Stream	ND	ND	ND
0-T-4-WS-B	0	Stream	ND	ND	ND
1-T-4-WS-A	1	Stream	<5	ND	<5
1-T-4-WS-B	1	Stream	ND	<5	<5
0-T-5-WS-A	0	Stream	ND	ND	ND
0-T-5-WS-B	0	Stream	ND	ND	ND
1-T-5-WS-A	1	Stream	ND	<5	<5
1-T-5-WS-B	1	Stream	ND	<5	<5
7-T-1-WS-A	7	Stream	ND	ND	ND
7-T-1-WS-B	7	Stream	ND	ND	ND
7-T-2-WS-A	7	Stream	ND	ND	ND
7-T-2-WS-B	7	Stream	ND	ND	ND

(continued on next page)

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[1] DDVP residues expressed as Naled by multiplying DDVP residues by molecular weight conversion of 1.72.

11.18

TABLE 9 (continued)

Analysis of Stream and Pond Water and Stream Sediment for Naled and DDVP.

Sample Code	Test Day	Location	Naled (ppb)	DDVP as[1] Naled (ppb)	Total (ppb)
7-T-3-WS-A	7	Stream	ND	ND	ND
7-T-3-WS-B	7	Stream	ND	ND	ND
7-T-4-WS-A	7	Stream	ND	ND	ND
7-T-4-WS-B	7	Stream	ND	ND	ND
7-T-5-WS-A	7	Stream	ND	ND	ND
7-T-5-WS-B	7	Stream	ND	ND	ND
0-T-PW-A	0	Pond	ND	ND	ND
0-T-PW-B	0	Pond	ND	ND	ND
1-T-PW-A	1	Pond	ND	ND	ND
1-T-PW-B	1	Pond	ND	ND	ND
3-T-PW-A	3	Pond	ND	ND	ND
3-T-PW-B	3	Pond	ND	ND	ND
0-T-1-SS-A	0	Stream	ND	ND	ND
0-T-1-SS-B	0	Stream	<50	ND	<50

[1] DDVP residues expressed as Naled by multiplying DDVP residues by molecular weight conversion of 1.72.

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TABLE 10

Analysis of Soil Samples for Naled and DDVP.

Sample Code	Test Day	Location	Naled (ppb)	DDVP as[1] Naled (ppb)	Total (ppb)
		0-3 Inch Depth			
0-T-1-SL-A(0-3)	0	Under Litter	<50	ND	<50
0-T-2-SL-A(0-3)	0	Under Litter	ND	<50	<50
0-T-3-SL-A(0-3)	0	Under Litter	ND	ND	ND
0-T-4-SL-A(0-3)	0	Under Litter	<50	<50	<50
1-T-1-SL-A(0-3)	1	Under Litter	ND	ND	ND
1-T-2-SL-A(0-3)	1	Under Litter	ND	ND	ND
1-T-3-SL-A(0-3)	1	Under Litter	ND	ND	ND
1-T-4-SL-A(0-3)	1	Under Litter	ND	ND	ND
3-T-1-SE-A(0-3)	3	Exposed Soil	ND	ND	ND
3-T-2-SE-A(0-3)	3	Exposed Soil	ND	ND	ND
3-T-3-SE-A(0-3)	3	Exposed Soil	ND	ND	ND
3-T-4-SE-A(0-3)	3	Exposed Soil	ND	ND	ND
5-T-1-SL-A(0-3)	5	Under Litter	ND	ND	ND
5-T-2-SL-A(0-3)	5	Under Litter	ND	ND	ND
5-T-3-SL-A(0-3)	5	Under Litter	ND	ND	ND
5-T-4-SL-A(0-3)	5	Under Litter	<50	ND	<50
		3-6 Inch Depth			
0-T-1-SL-A(3-6)	0	Under Litter	ND	ND	ND
0-T-2-SL-A(3-6)	0	Under Litter	ND	ND	ND
0-T-3-SL-A(3-6)	0	Under Litter	<50	ND	<50
0-T-4-SL-A(3-6)	0	Under Litter	<50	ND	<50
0-T-1-SE-A(3-6)	0	Exposed Soil	ND	ND	ND
0-T-2-SE-A(3-6)	0	Exposed Soil	<50	ND	<50
0-T-3-SE-A(3-6)	0	Exposed Soil	ND	<50	<50
0-T-4-SE-A(3-6)	0	Exposed Soil	<50	<50	<50
1-T-1-SL-A(3-6)	1	Under Litter	ND	ND	ND
1-T-2-SL-A(3-6)	1	Under Litter	ND	ND	ND
1-T-3-SL-A(3-6)	1	Under Litter	ND	ND	ND
1-T-4-SL-A(3-6)	1	Under Litter	<50	ND	<50

(continued on next page)

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[1] DDVP residues expressed as Naled by multiplying DDVP residues by molecular weight conversion of 1.72.

11.20

TABLE 10 (continued)

Analysis of Soil Samples for Naled and DDVP.

Sample Code	Test Day	Location	Naled (ppb)	DDVP as Naled[1] (ppb)	Total (ppb)
		3-6 Inch Depth (continued)			
1-T-1-SE-A(3-6)	1	Exposed Soil	ND	<50	<50
1-T-2-SE-A(3-6)	1	Exposed Soil	ND	ND	ND
1-T-3-SE-A(3-6)	1	Exposed Soil	ND	ND	ND
1-T-4-SE-A(3-6)	1	Exposed Soil	ND	ND	ND
3-T-1-SE-A(3-6)	3	Exposed Soil	ND	ND	ND
3-T-2-SE-A(3-6)	3	Exposed Soil	ND	ND	ND
3-T-3-SE-A(3-6)	3	Exposed Soil	ND	ND	ND
3-T-4-SE-A(3-6)	3	Exposed Soil	ND	ND	ND
5-T-1-SL-A(3-6)	5	Under Litter	ND	105	105
5-T-2-SL-A(3-6)	5	Under Litter	ND	ND	ND
5-T-3-SL-A(3-6)	5	Under Litter	ND	ND	ND
5-T-4-SL-A(3-6)	5	Under Litter	ND	ND	ND

[1] DDVP residues expressed as Naled by multiplying DDVP residues by molecular weight conversion of 1.72.

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139

TABLE 1

Soil Characterization Data.

	Soil Depth (inches)			
	0-12	12-24	24-36	36-48
Madison Sandy Loam				
% Organic Matter	0.80	0.27	0.07	0.07
pH	5.4	5.5	5.5	5.5
CEC (meq/100 g)	5.9	5.2	3.6	3.5
% Sand	36.0	34.0	52.0	58.0
% Silt	18.0	24.0	28.0	30.0
% Clay	46.0	42.0	20.0	12.0
Texture	Clay	Clay	Sandy Loam	Sandy Loam
Cecil Sandy Loam				
% Organic Matter	1.50	0.20	0.20	0.27
pH	5.1	5.5	5.3	5.2
CEC (meq/100 g)	4.3	4.4	5.0	5.1
% Sand	64.0	56.0	44.0	36.0
% Silt	20.0	14.0	22.0	22.0
% Clay	16.0	30.0	34.0	42.0
Texture	Sandy Loam	Sandy Clay Loam	Sandy Clay Loam	Clay

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DATA EVALUATION RECORD

NALED STUDY 12

CHEM 034401 Naled

BRANCH EAB

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40034905 CONTENT CAT 01
Cheng, H.M. 1986. Naled accumulation study: Rotational crops (confined). Laboratory Project ID: 8607629A. Prepared and submitted by Chevron Chemical Company, Richmond, CA.

SUBST. CLASS = S

DIRECT RVW TIME = 6

REVIEWED BY: K. Patten TITLE: Staff Scientist

EDITED BY: TITLE: Staff Scientist

APPROVED BY: TITLE: Program Manager

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SIGNATURE: May 19th, 1988

CONCLUSIONS:

Confined Accumulation - Rotational Crops

This study is not acceptable as presented because a) residues in the soil were not characterized, except at time 0; b) at time 0, the measured concentration of naled residues in the 0- to 6-inch soil depth was 0.52 ppm instead of the approximately 1 ppm expected for the 2 lb ai/A application; c) soil textural analysis could not be confirmed; d) immature plant samples were not analyzed. The purpose of the reviewed study was to provide residue information on the amount and nature of residues taken up by rotational crops requested in a previous review (8/5/86).

For accepting the study, points 2 through 7 in the DISCUSSION should be addressed by the registrant.

SUMMARY OF DATA BY REVIEWER:

[^{14}C]Naled residues were ≤0.03 ppm (naled equivalents) in mature lettuce (tops and roots), wheat (grain, bran, and straw), and carrots (tops and roots) planted in plastic pots (14-inch diameter, 18-inch depth) of loam soil 30 days after the soil was surface-treated with ethyl [1-^{14}C]naled (radiochemical purity >99%) at approximately 2 lb ai/A. [^{14}C]Naled residues were 0.07 ppm in wheat roots. The study was conducted in a greenhouse maintained on a 14-hour, 80°F light:10-hour, 70°F dark cycle. In the upper 6 inches of the soil, [^{14}C]naled residues were 0.52 ppm at time 0, 0.03 ppm at planting (30 days posttreatment), and 0.01 ppm at harvest of the crops (3-4 months posttreatment).

DISCUSSION:

1. The application rate in this study is 2 lb ai/A. According to the 1987 Index, the only sites treated at >2 lb ai/A are orchard crops (peaches, walnuts, and tangerines), which would be unlikely to be in a rotation. As long as no field treated at >2 lb ai/A is rotated, this study may be made acceptable in support of data requirements for accumulation in confined rotational crops if the requested data are found acceptable.

2. [^{14}C]Residues in the soil were not characterized except at time 0. The study author stated that at time 0, 85% of the residues could be extracted with methanol and acetone and 95% of the extract was parent naled. Is there any evidence of presence of DDVP?

3. At time 0, the measured concentration of naled residues in the 0- to 6-inch soil depth was 0.52 ppm. For an application of 2 lb ai/A, the measured concentration should have been approximately 1 ppm.

4. The moisture content of the soil during aging was not reported.

5. Immature plant samples were not analyzed.

6. The study author stated that attempts were made to characterize the [^{14}C]residues in the wheat roots and straw; however, there was insufficient wheat root mass to extract and <10% of the radioactivity in the wheat straw (<0.003 ppm) was extractable with acetone and methanol:water.

7. The soil textural analysis (sand, silt, and clay) summed to 92% rather than 100%. The textural classification could not be confirmed.

8. No raw data were provided to support the conclusions. This was a revised document, resubmitted in response to EAB comments. Raw data was provided with the original submission (8/5/86 review).

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MATERIALS AND METHODS

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MATERIALS AND METHODS:

Ethyl [1-^{14}C]naled (radiochemical purity >99%, specific activity 12.96 mCi/mMol, Chevron Chemical Company) was applied at approximately 2 lb ai/A to the surface of loam soil (40% sand, 40% silt, 12% clay, 0.8% organic matter, pH 7.2, CEC 6.8 meq/100 g) that was contained in plastic pots (14-inch width, 18-inch depth) in a greenhouse. The greenhouse was on a 14 hour:10 hour light:dark cycle; temperatures varied from 80°F during the daylight to 70°F at night. At 30 days posttreatment, the pots were planted to Long Imperator 58 carrots, Vanguard 75 lettuce or Yecorn Rojo wheat. The plants were fertilized regularly and harvested at maturity (approximately 2 months postplanting for lettuce, 2.5 months for wheat, and 3 months for carrots). Soil (0- to 6-inch depth) was sampled at 0 and 30 days posttreatment and at the time of plant harvest.

Plant samples were ground with dry ice, and plant and soil subsamples were analyzed for total radioactivity by LSC following combustion. Soil samples from time 0 and the wheat straw were extracted sequentially twice with acetone and twice with methanol:water (2:1, v:v). Aliquots of the extracts were analyzed by LSC. The soil extracts were further analyzed by TLC on silica gel plates developed in chloroform:acetic acid (4:1 and 10:1, v:v) and methylene chloride:acetone (3:1, v:v). The plates were visualized using autoradiography, and radioactive compounds were identified by comparison to reference standards. The extracted soil was analyzed for unextractable [^{14}C]residues by LSC following combustion.

MATERIALS AND METHODS

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

PERTINENT DATA TABLES AND FIGURES

Naled

Page ______ is not included in this copy.

Pages _196_ through _200_ are not included in this copy.

The material not included contains the following type of information:

_____ Identity of product inert ingredients.

_____ Identity of product inert impurities.

_____ Description of the product manufacturing process.

_____ Description of product 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

__X__ FIFRA registration data.

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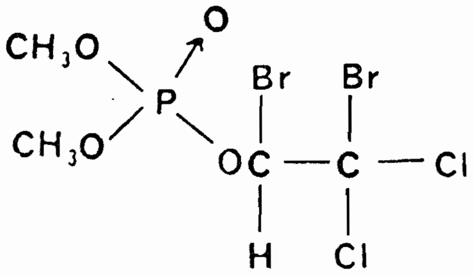
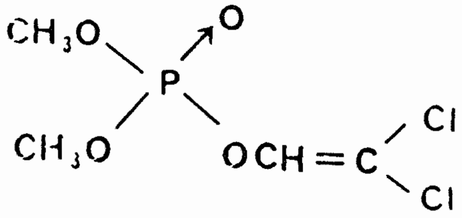
The information not included is generally considered confidential by product registrants. If you have any questions, please contact the individual who prepared the response to your request.

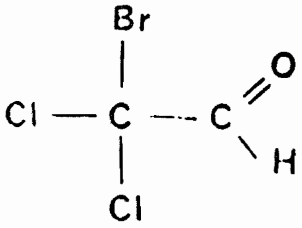
STRUCTURES OF NALED AND POTENTIAL DEGRADATES



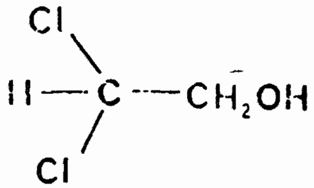
Naled
(1,2-Dibromo-2,2-dichloroethyl
dimethyl phosphate)



DDVP
(2,2-Dichlorovinyl dimethyl
phosphate)



BDCA
(Bromodichloroacetaldehyde)



DCE
(Dichloroethanol)

$Cl_2CH\ CO_2H$

DCAA
(Dichloroacetic acid)

$ClCH_2CO_2H$

DCA
(Chloroacetic acid)