3M Corporate Toxicology and Regulatory Services

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AR226 - 3624

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February 13, 2006

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EPA East – Room 6428 Attn: Section 8(e)
Office of Pollution Prevention and Toxics
US EPA
1200 Pennsylvania Avenue NW
Washington DC 20460-0001

No CBI

RE: TSCA 8(E) Supplemental Notice: N-methylperfluorooctane sulfonamidoethanol, Docket No. 8EHQ-1004-15865A

Dear Sir/Madam:

3M is submitting this notice to supplement its previous submissions on N-methylperfluorooctane sulfonamidoethanol with reference to a cover letter dated October 26, 2004, and an e-mail from Terry Obryan dated January 11, 2006.

In that submission, 3M submitted water solubility and octanol/water partition coefficient data on this chemical. EPA has questioned whether the values are correct as the reported water solubility is more than three orders of magnitude higher than the EPIWIN estimated value, as well as estimated values for analogs. In addition EPA questions whether the identity of the chemical is accurate since there is handwritten information on the page.

In this letter, 3M confirms that the data submitted is data on N-methylperfluorooctane sulfonamidoethanol and that the data are correct. The EPIWIN model training set does not include partially fluorinated molecules such as this one. Therefore, the estimated Kow of this chemical using EPIWIN is likely higher than that measured due to this model's tendency to overestimate the octanol solubility and underestimate the water solubility because of the large perfluorinated chain contained in the molecule. In addition, the ecotoxicity studies were conducted in excess of water solubility and this material is apparently not acutely toxic at the solubility limit.

Final study reports are enclosed.

If you have any questions, please do not hesitate to contact me at 651-737-1374.

Sincerely,

Deanna Luebker, MS, PhD
3M TSCA 8(e) Coordinator

Enclosures

8EHQ-04-15865

MRID 292982

AR 226 _ 3623

3M Form 6747-11-A

TECHNICAL REPORT SUMMARY

Date: 1/10/79

TO: TECHNICAL COMMUNICATIONS CENTER – 201-2CN

(Important – If report is printed on both sides of paper, send two copies to TCC.)

Division: Environmental Laboratory (EE & PC)	Dept. Number: 0535
Project: Premanufacturing Notice FC-790	Project Number: 9970012600
Report Title: Distribution Coefficient of FM 3925 in n-Octanol/Water	Report Number: 015
To: V. Pothapragada	
Author(s): E. A. Reiner	Employee Number(s): 47816
Notebook Reference: 48838, pp. 6, 8, and 9.	No. of Pages Including Coversheet:

SECURITY ▶ ☐ Open (Company Confidential) ☐ Closed (Special Authorization)

3M CHEMICAL REGISTRY ▶ New Chemicals Reported ☐ Yes ☒ No

KEYWORDS: (Select terms from 3M Thesaurus. Suggest other applicable terms.)

Distribution Coefficient

Partition Coefficient

Bioconcentration

Fluorochemical

CURRENT OBJECTIVE:

To determine the distribution coefficient of FM 3925 between n-octanol and water.

REPORT ABSTRACT: (200-250 words) This abstract information is distributed by the Technical Communications Center to alert 3M'ers to Company R&D. It is Company confidential material.

The distribution coefficient of FM 3925, lot 505, between n-octanol and water was measured according to a modification of the Environmental Laboratory procedure. The distribution coefficient was determined to be 56,800. This result suggests that this material is likely to accumulate in aquatic organisms to levels above the ambient aquatic concentration.

Information Liaison Initials: SKW

Introduction

Living organisms possess the ability to concentrate and accumulate lipophilic organic compounds either directly from their environment or from their food source, a phenomenon which is well document (1,2).

In 1975 Neely, et al (3), demonstrated that the partition coefficient can be used to estimate this bioconcentration potential. As a result, it has become a common procedure to estimate bioconcentration potential from a compound's partition or distribution coefficient.

Methods

The distribution coefficient of FM 3925, lot 505, was determined by a modification of the 3M Environmental Laboratory protocol. The modifications and unique features of this study are described below.

FM 3925, lot 505, was used in the distribution coefficient determination as it was received without any further purification. Gas chromatographic analysis of this compound shows that this compound elutes as three overlapping peaks (Figure 1). The combined area of these peaks was used in the determination of sample concentrations.

Sample analysis was performed on a Hewlet-Packard Model 5713 gas chromatograph, a ^{63}Ni electron capture detector. Separations were obtained using a 6 ft. by 1/8 in. O.D. stainless steel column containing 10% Carbowax 20M on 60/80 mesh Chromosorb W-AW. Injection port, oven and detector temperatures were set at 200°, 180°, and 300° C., respectively. The gas flow rate was 40 ml/min. of a 95% Argon 5% methane mixture.

Two 25-ml n-octanol solutions of FM 3925 were added to amber glass jars, each containing 900 ml of deionized water. These n-octanol solutions contained 1000 ppm (0.018M) and 2000 ppm (0.036M) of FM 3925. High FM 3925 concentrations were used because earlier attempts at determining the distribution coefficient with concentrations below 0.01M were unsuccessful since our analytical procedures were not sufficiently sensitive to detect the FM 3925 re-extracted from the water phase into ethyl acetate.

The jars used in the distribution coefficient determination were shaken ∿16 hours at room temperature (∿22° C.). The octanol phase was drawn off and the water phase was placed in 250-ml polycarbonate centrifuge tubes and centrifuged at 10,000 rpm (13,700 x G) for 15 minutes. Following centrifugation, 600 ml of the water phase was transferred by pipet to a separatory funnel where it was, in turn, drawn off into a second separatory funnel to ensure the removal of octanol contamination transferred by the pipetting procedure.

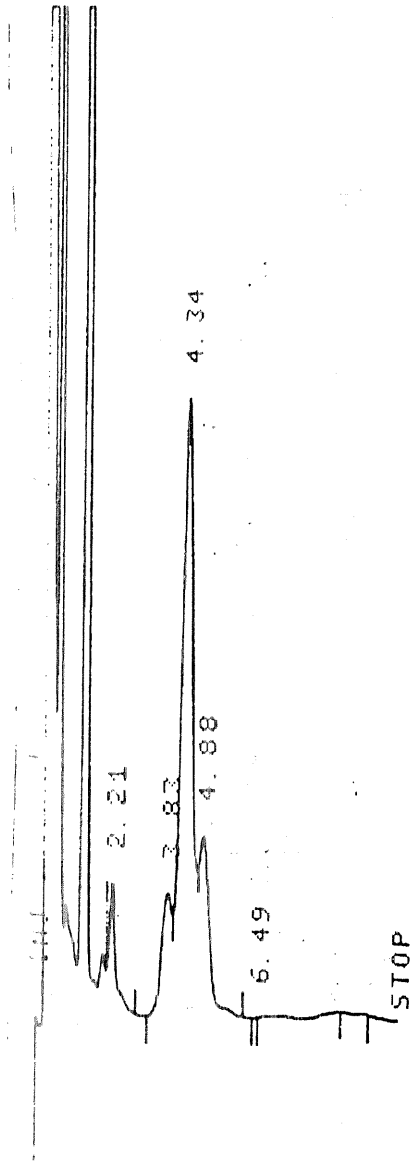

Figure 1. Gas Chromatogram of a 5 µl injection of a 1 ppm solution of FM 3925 (#081, 7/13/78). Peaks at 3.83, 4.34, and 4.88 minutes represent FM 3925. Gas chromatographic conditions are given in text.

The water phase was extracted three times with ethyl acetate, and the ethyl acetate extract was concentrated to 15 ml at room temperature under nitrogen. The FM 3925 concentration in the ethyl acetate was determined by electron capture gas chromatography. A typical gas chromatogram of the water phase is shown in Figure 2. The octanol phase from the distribution coefficient determination was not analyzed for FM 3925 content. Its FM 3925 concentration was assumed to be unchanged. This assumption was justified since measurement of the FM 3925 content from the octanol phase from previous attempts to determine the distribution coefficient had shown that the FM 3925 concentration was not detectably lowered by water extraction. A gas chromatogram showing the octanol phase after extraction is shown in Figure 3.

Results and Discussions

FM 3925 concentrations in the water phases shaken with the 1000 ppm and 2000 ppm FM 3925 n-octanol solutions were 0.0189 ppm and 0.033 ppm respectively. This data corresponds to distribution coefficients of 52,900 and 60,600. The average distribution coefficient for FM 3925 in n-octanol/water is 56,800.

Based on the method of Neely, Branson & Blair (3), this corresponds to a predicted bioconcentration factor between fish muscles and water of 500. Our laboratory findings on the structurally similar compound, FM 3422, gave values in this same range (4). Total body accumulation of FM 3422 into juvenile channel catfish indicated a bioconcentration factor of ~500. Upon transfer to clear water, there was 50% clearance of the bioconcentrated FM 3422 within four days.

The lower distribution coefficient for FM 3925 compared to FM 3422 (56,800 vs. >100,000), and the greater water solubility of FM 3925 (0.82 mg/l vs. 0.05 mg/l) (5), suggest that FM 3925 will bioconcentrate to a lesser extent than FM 3422.

References:

(1) Burnett, R. Science 174:606, 1971.

(2) Gustafson, C. G. Env. Sci. & Tech. 4:814, 1970.

(3) Neely, W. B., D. R. Branson, and G. E. Blair, Env. Sci. & Tech. 8:1113, 1974.

(4) Welter, A. N. Evaluation of the Bioconcentration Potential of FM 3422, 3M Technical Report, August 16, 1978.

(5) Welter, A. N., E. A. Reiner, Solubility of FM 3925, 3M Technical Report, January 8, 1979.

EAR/cen

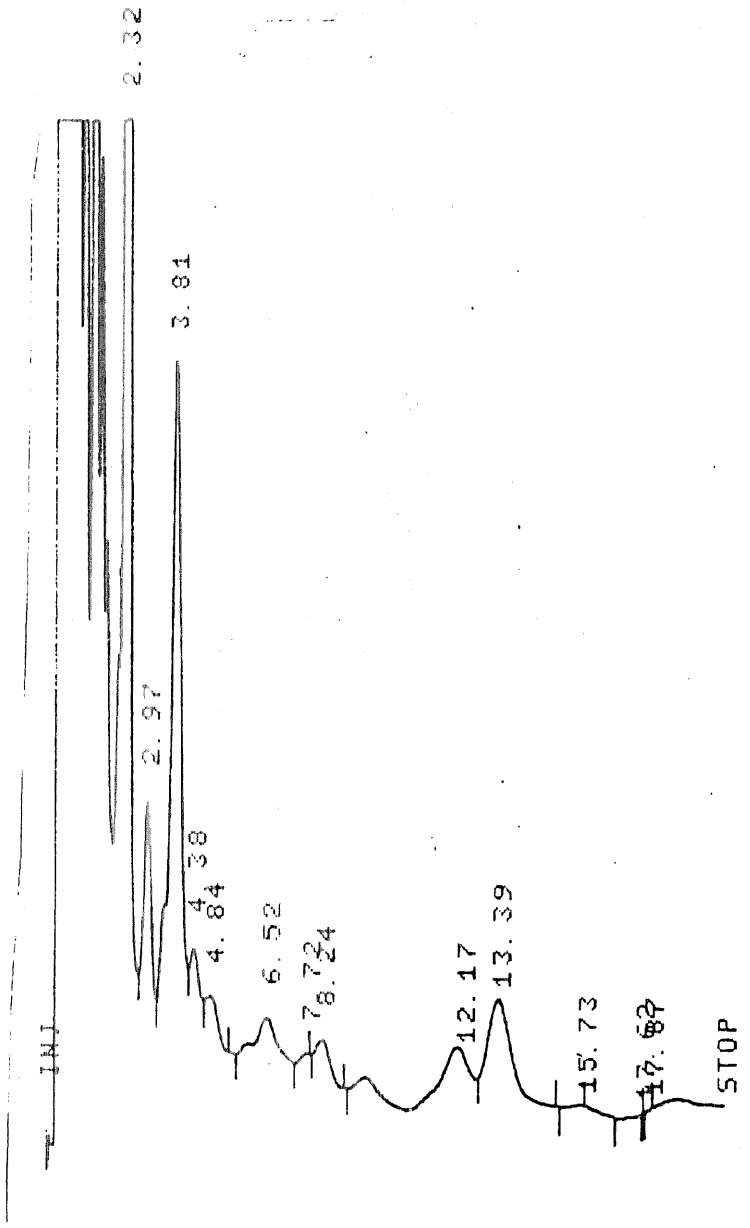

Figure 2. Gas Chromatogram of the water phase from an n-octanol/water distribution coefficient for FM 3925 (#082, 7/13/78). Peaks at 3.81, 4.38, and 4.84 minutes are taken to represent FM 3925. Gas chromatographic conditions are given in text.

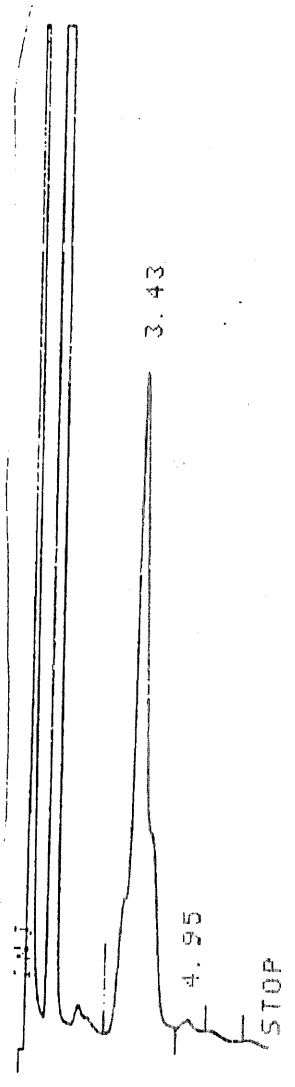

Figure 3 . Gas Chromatogram of the octanol phase from an n-octanol/water distribution coefficient determination for FM 3925 (#163, 6/20/78). The peak at 3.43 minutes and shoulders before and after this peak represent FM 3925. Gas Chromatographic conditions are given in text.

AK226-2626

3M Form 5747-11-A

TECHNICAL REPORT SUMMARY

Date: Jan. 8, 1979

TO: TECHNICAL COMMUNICATIONS CENTER -- 201-2CN

(Important -- If report is printed on both sides of paper, send two copies to TCC.)

Division: Environmental Laboratory	Dept. Number: 0535
Project: Fate of Fluorochemical Project	Project Number: 9970012600
Report Title: Aquatic Toxicity Studies: FM 3925	Report Number: 018
To: D. L. Bacon	
Author(s): A. N. Welter	Employee Number(s): 09362
Notebook Reference:	No. of Pages Including Coversheet:

SECURITY ▶ ☐ Open (Company Confidential) ☐ Closed (Special Authorization)

3M CHEMICAL REGISTRY ▶ New Chemicals Reported ☐ Yes ☒ No

KEYWORDS: (Select terms from 3M Thesaurus. Suggest other applicable terms.)

EE & PC Div.
(Environmental Lab)
Toxicity
(Aquatic)

CURRENT OBJECTIVE:

To determine the acute 96 hour LC_{50} value for bluegill sunfish (*Lepomis macrochirus*) and the 48 hour LC_{50} value for *Daphnia magna* exposed to varying concentrations of FM 3925.

REPORT ABSTRACT: (200-250 words) This abstract information is distributed by the Technical Communications Center to alert 3M'ers to Company R&D. It is Company confidential material.

The limited water solubility of FM 3925 prohibited a definitive determination of the aquatic toxicity of this test material. Both vertebrate and invertebrate LC_{50} values exceed the water solubility of FM 3925.

cc: R. A. Prokop
D. Ricker
R. L. Bohon
M. T. Elnabarawy
A. Mendel
E. A. Reiner
S. K. Welsh

Information Liaison Initials: SKW

CONCLUSION

The limited water solubility of FM 3925 prohibited a definitive determination of the aquatic toxicity of this test material. Both vertebrate and invertebrate LC_{50} values exceed the water solubility of FM 3925.

INTRODUCTION

The test substance FM 3925 is an off-white solid. Tests performed on this material evaluated its aquatic toxicity and the data obtained form the basis of this report.

METHODS

A. Aquatic Testing

The test protocols utilized for these studies were modeled after that described by US/EPA (1975).

B. Acclimation Procedure

The bluegill sunfish *(Lepomis macrochirus)* were obtained from a private hatchery+. Stock fish were held in a fiberglass holding tank filled with carbon-filtered well water at 14-15°C. A daily photoperiod of 16 hours light and 8 hours dark with a 30-minute transition period was maintained throughout the acclimation and testing period. The fish were fed Tetra-Min° daily, food being withheld 48 hours prior to and throughout the test period. Fish were acclimated for 14 days prior to testing.

An acute short-term (96-hour) static bioassay was performed on FM 3925. Carbon-filtered well water of known composition was used as the diluent (Attachment 1). All glass aquaria (35x20x20 cm) containing 16 liters of diluent or diluent plus toxicant comprised a study chamber. Twenty bluegill sunfish, uniform in size, were tested at each chemical concentration. Test fish were randomly allocated to each test chamber within 30 minutes following toxicant addition. Bluegill sunfish experiments were conducted at 20-21° C. Mortality, temperature, dissolved oxygen level and pH of all test solutions were measured at 24-hour intervals or until total mortality had occurred. General observations relative to behavioral changes were similarly recorded when appropriate.

These studies were performed in replicate. Organisms used in this study were considered to be generally healthy and free of disease.

The *Daphnia magna* used in this study were obtained on April 19, 1978 from the Mogul Corp. Test protocol used was that described by USEPA (1975).

+ Dale Fattig Fish Hatchery, Brady, Nebraska

° Commercial Fish food of known composition

RESULTS

The bluegill sunfish (*Lepomis macrochirus*) used in this study had an average weight of 0.5 g and length of 4.0 cms. The loading factor for these studies was approximately 0.63 g/l, below the maximum recommended loading factor of 0.8 g/l[1]. Test temperatures ranged from 18 - 20°C, pH varied from 7.8 - 8.2 pH units and oxygen saturation at 96 hours in the individual experiments were 34 and 56%.

In this replicated study, LC_{50} values were not calculated since these values would exceed the water solubility limits of FM 3925. (Attachment 2).

The *Daphnia magna* study was conducted at a temperature of 19°C., pH range of 8.2 - 8.7 and a minimal 48 hour oxygen saturation of 79%. Projected LC_{50} values exceeded the water solubility of FM 3925. (Attachment 3).

The data reported herein are based on studies performed by M. T. Elnabarawy and L. H. Pritchard.

REFERENCES

1. US/EPA - Methods for Acute Toxicity Test with Fish Macro-invertebrates and Amphibrians, Ecological Research Series 660/3-75-009, 1975.

LAB REQUEST NO. 4382 (STANDING) | DATE | SHEET

ATTACHMENT #1

IS FORM

LE DESCRIPTION:
ON FILTERED WELL
— BIOASSAY ROOM

	PH	TOTAL Cl_2 RESIDUAL	C.O.D.	CONDUCTIVITY micromhos/cm	M-ALK.	P-ALK.	TOTAL SOLIDS	TOTAL HARDNESS	AMMONIA			
7-18-78	7.8	-	<1	430	221	0	287	236	<0.1			
7-25-78	7.7	-	<1	440	219	0	288	236	<0.1			
7-31-78	7.7	-	<1	420	231	0	287	236	<0.1			
8-14-78	7.7	-	<1	420	222	0	293	236	<0.1			
8-21-78	7.6	-	<1	445	221	0	[illegible]	236	<0.1			
9-01-78	7.7	-	<1	400	[illegible]	0	276	[illegible]	<0.1			
9-07-78	7.7	-	<1	400	220	0	[illegible]	236	<0.1			
9-15-78	7.7	<0.1	<1		222	0		232	<0.1			
9-22-78	7.7	<0.1		405	222	0		232	<0.1			
9-29-78												

Lik 4/97

#2

ATTACHMENT #2

ENVIRONMENTAL ENGINEERING LABORATORY
AQUATIC TOXICITY DATA SHEET

Type Test 96-Hour Static Material Tested FM 3925

Test Organism Bluegill Sunfish Avg. Wt. .5 grams Avg. Size 4 cm

Test Solution Volume 16 Liters/Tank No. Organisms per dose 20

Time Started 2:00 PM Date Started 4-11-78 Date Completed 4-15-78

Dilution Water Carbon Filter - Well Water (Temp. 18 °C., D.O. 8.9 ppm, pH 7.9)

Investigator M.J. Elmshorany LC 50 (95% Confidence Limits)

mg/l Conc.	No. Organisms Exposed	24 Hrs. No. Dead	pH	D.O. ppm	48 Hrs. No. Dead	pH	D.O. ppm	72 Hrs. No. Dead	pH	D.O. ppm	96 Hrs. No. Dead	pH	D.O. ppm
Control	20	0	8.2	6.1	0	8.2	6.0	0	8.2	6.0	0	8.2	5.7
Control	20	0	7.8	6.5	0	7.8	6.2	0	7.8	6.0	0	7.9	5.7
13	20	0	7.9	6.3	0	7.9	6.0	1	7.9	6.0	1	7.9	5.8
22	20	0	7.9	6.0	0	7.9	5.9	2	7.9	5.8	2	7.9	5.8
36	20	0	7.8	6.0	0	7.8	5.9	2	7.8	5.3	2	7.8	5.0
60	20	0	7.8	6.0	0	7.8	5.9	3	7.9	5.3	3	7.9	5.1
100	20	0	7.8	6.0	0	7.9	5.6	7	7.9	5.3	7	7.9	5.0
Avg. Temp. ~~18°C~~		18°C			18°C			18°C			19°C		

Comments: * Control tank contained 8 ml acetone (.5 ml/liter)

- Known amounts of FM-3925 (a solid material) were dissolved in 8 ml acetone (.5 ml acetone per liter of water) before mixing with dilution water.
- Test material gradually precipitated out (settled out in the bottom); precipitated material increased with concentrations (on initial basis).

4197

ENVIRONMENTAL ENGINEERING LABORATORY
AQUATIC TOXICITY DATA SHEET

ATTACHMENT #2

Type Test 96-Hr. Static Material Tested FC-3925

Test Organism Bluegill Sunfish Avg. Wt. 0.5 grams Avg. Size 4 cm

Test Solution Volume 16 Liters/Tank No. Organisms per dose 20

Time Started 3:30 PM Date Started 4-18-78 Date Completed 4-22-78

Dilution Water Carbon Filter - Well Water (Temp. 17°C, D.O. 8.9 ppm, pH 7.8)

Investigator L. Pritchard LC 50 (95% Confidence Limits)

...c.	No. Organisms Exposed	24 Hrs. No. Dead	pH	D.O. ppm	48 Hrs. No. Dead	pH	D.O. ppm	72 Hrs. No. Dead	pH	D.O. ppm	96 Hrs. No. Dead	pH	D.O. ppm
...tro	20	0	7.8	5.8	0	7.8	4.1	1	7.8	3.4	1	7.9	3.5
[illegible]	20	1	7.8	5.3	2	7.8	4.1	3	7.8	3.7	3	7.9	3.5
[illegible]	20	1	7.8	5.4	1	7.8	3.6	1	7.8	3.0	1	7.8	3.1
[illegible]	20	0	8.0	5.2	0	8.0	3.7	0	7.9	3.9	0	7.9	3.0
[illegible]	20	0	7.9	5.8	0	7.9	4.4	0	7.9	3.9	3	7.9	3.8
[illegible]	20	0	8.0	6.2	1	8.0	5.0	1	8.0	4.4	1	7.9	4.0
[illegible]	20	1	7.9	6.1	2	7.9	4.3	3	7.9	3.9	3	7.8	3.7
[illegible]	20	1	7.8	5.2	1	7.8	3.8	1	7.8	3.5	1	7.9	3.4
													[illegible]
... Temp.		18°C			18°C			18°C			20°C		

...ments: Used 8 ml of acetone for each conc. (.5 ml acetone per liter)
Used 8 ml of acetone in (1) control
The two (2) conc. 180 mg/l and 320 mg/l the chemical
...cipitated out within 24 Hrs. The remaining
conc. percipitated out within 48 Hrs.

LR. 4197

ENVIRONMENTAL ENGINEERING LABORATORY
AQUATIC TOXICITY DATA SHEET

ATTACHMENT #3

Type Test 48-Hr. Daphnia Magna Material Tested FM-3925 (2)

Test Organism Daphnia Magna Avg. Wt. * grams Avg. Size * cm

Test Solution Volume 100 ml ~~Liters/Tank~~ Jar No. Organisms per dose 20

Time Started 2:00 pm Date Started 4-24-78 Date Completed 4-26-78

Dilution Water Carbon Filter - Well Water (Temp. 19 °C., D.O. 8.7 ppm, pH 8.7)

Investigator L. Pritchard LC 50 (95% Confidence Limits) _____
M. Elwabanawy

mg/l Conc.	No. Organisms Exposed	24 Hrs. No. Dead	pH	D.O. ppm	48 Hrs. No. Dead	pH	D.O. ppm	72 Hrs. No. Dead	pH	D.O. ppm	96 Hrs. No. Dead	pH	D.O. ppm
Control	20	0			2	8.6	7.8						
Control + Water	20	0			0	8.6	7.2						
100	20	0			1	8.5	7.5						
180	20	0			0	8.5	7.5						
320	20	0			2	8.5	7.1						
560	20	0			0	8.3	7.6						
1000	20	0			2	8.2	7.5						
Avg. Temp.		19°C			19°C								

Comments:

* Daphnia Magna use in this test were [illegible] from Mogul Corp. on 4-1978. Used [illegible] in this study.

ENVIRONMENTAL ENGINEERING LABORATORY
AQUATIC TOXICITY DATA SHEET

ATTACHMENT #3

Type Test 48 Hr. Daphnia Magna Material Tested FM-3925 #2

Test Organism Daphnia Magna Avg. Wt. * grams Avg. Size * cm

Test Solution Volume 100 ml ~~[illegible]~~ Jar No. Organisms per dose 20

Time Started 3:00 pm Date Started 4-37-78 Date Completed 4-39-78

Dilution Water Carbon Filter - Well Water (Temp. 20 °C., D.O. 8.9 ppm, pH 8.7)

Investigator L. Pritchard
M. Elwabanawy

LC 50 (95% Confidence Limits) 1353 mg/l (1194-1633)

mg/l Conc.	No. Organisms Exposed	24 Hrs. No. Dead	pH	D.O. ppm	48 Hrs. No. Dead	pH	D.O. ppm	72 Hrs. No. Dead	pH	D.O. ppm	96 Hrs. No. Dead	pH	D.O. ppm
Control	20	0			1	8.7	7.6						
[illegible]	20	0			0	8.7	7.4						
250	20	0			0	8.5	7.2						
1000	20	2			4	8.5	7.0						
550	20	12			12	8.6	7.2						
4100	20	20	8.6	8.5	20	—							
5700	20	20	8.6	8.5	20	—							
6600	20	20	8.6	8.5	20	—							
7000	20	20	8.6	8.5	20	—							
Avg. Temp.		19°C			19°C								

Comments:

* Daphnia Magna used in this test were obtained from Mogul Corp. on 4-19-78. Used neonates in this study.

LR 4197

D

RUN

PROBIT.B20
THURSDAY, MAY 11, 1978 15:34:51

FM-3925

ENVIRONMENTAL RESEARCH LAB. - DULUTH

PROBIT ANALYSIS FOR DIORSSAY DATA

NO OF CURVES ? 1
NO. OF CONC. FOR CURVE 1 ? 4
INPUT DATA-CONC.,NO. EXPOSED,NO.DEAD
? 650,20,0
? 1000,20,4
? 1550,20,12
? 2400,20,20

Daphnia Magna

CONC. mg/l	LOG CONC.	FRACTION RESPONDING	FITTED FRACTION
650	2.812913	0	0.005900574
1000	3	0.2	0.1499294
1550	3.190332	0.6	0.6809147
2400	3.380211	1	0.9757905

CHI-SQ PROBABILITY IS > 0.05

NO OF ITERATIONS	INTERCEPT (A)	SLOPE (B)	CHI SQ FOR LINEARITY	CHI SQ FOR PROBABILITY
6	-24.7904	7.917881	1.61074	0.4469226

HETEROGENIETY FACTOR (H) = 1 G = 0.1348014

LC-50 = 1351.882

95 PCT. LOWER CONF. LIMIT = 1193.531
95 PCT. UPPER CONF. LIMIT = 1533.081