

ACUTE TOXICITY TO AQUATIC INVERTEBRATES (E.G. DAPHNIDS)

TEST SUBSTANCE

Identity: N-methylperfluorooctane sulfonamidoethanol; may also be referred to as N-MeFOSE Alcohol, FC-790, or FM-3925. (1-Octanesulfonamide, N-methyl-1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-heptadecafluoro-N-(2-hydroxyethyl)-, CAS # 24448-09-7)

Remarks: Material is an off-white, waxy solid. Sample purity and lot number were not recorded.

METHOD**Summary of 2 Studies**

Method: U.S. EPA 660/3-75-009, April, 1975.

Type: Static acute

GLP: No

Year completed: 1978

Species: *Daphnia magna*

Analytical monitoring: Nominal concentrations only. Temperature, pH and dissolved oxygen recorded

Statistical methods: Probit analysis. Concentrations tested exceed water solubility of the test substance.

Test daphnid source: Obtained from the Mogul Corporation.

Test daphnid age at study initiation: Neonates

Test conditions:

Dilution water: Carbon-filtered well water

Dilution water chemistry:

Hardness*:	236 mg/L as CaCO ₃
DO:	8.9 mg/L
COD*:	<1 mg/L
Alkalinity*:	221 mg/L as CaCO ₃
pH:	8.7
Conductivity*:	420 µmhos/cm
Total Solids*:	286 mg/L

* Values taken as averages from general laboratory water quality testing in 1978.

Stock and test solution preparation: Not noted in report.

Exposure vessels: Test vessels were described as jars. Each test vessel contained 100 mL of test solution.

Number of replicates: one

Number of daphnids per replicate: 20

Water chemistry during the study:

Dissolved oxygen (recorded at 48 hours or at 24 hours when all test organisms were dead):

7.8 mg/L (control exposure Test 1)

7.6 mg/L (control exposure Test 2)

7.5 mg/L (1000 mg/L exposure Test 1)

8.5 mg/L (8700 mg/L exposure Test 2)

pH (recorded at 48 hours or at 24 hours when all test organisms were dead):

8.6 (control exposure Test 1)

8.7 (control exposure Test 2)

8.2 (1000 mg/L exposure Test 1)

8.6 (8700 mg/L exposure Test 2)

Test temperature range (0 – 48hours:

19 – 20°C

Element basis: mortality and immobilization

Remarks: A first study was conducted at concentrations too low to induce sufficient mortality to allow the calculation of an LC50. The study was repeated at higher test substance concentrations.

The test substance was noted to precipitate out gradually over the course of the test. It was generally noted that more precipitate formed in the higher concentration test solutions.

RESULTS

Nominal concentrations:

Test 1: One blank and one solvent control, 100, 180, 320, 560, and 1000 mg/L

Test 2: One blank and one solvent control, 650, 1000, 1550, 2400, 3700, 5600, 8700 mg/L.

Element value:

Test 1 48-hour EC₅₀ (95% CI): Not calculated.

Test 2 48-hour EC₅₀ (95% CI): 1352 (1194-1533) mg/L

Statistical Evaluation: The EC₅₀ value and 95% confidence intervals were calculated using probit analysis.

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Test 1 Percent Mortality/Immobilization

Nominal Test Concentration, mg/L	24-Hours	48-Hours
Negative Control	0	10
Solvent Control	0	0
100	0	5
180	0	0
320	0	10
560	0	0
1000	0	10

Test 2 Percent Mortality/Immobilization

Nominal Test Concentration, mg/L	24-Hours	48-Hours
Negative Control	0	5
Solvent Control	0	0
650	0	0
1000	10	20
1550	60	60
2400	100	100
3700	100	100
5600	100	100
8700	100	100

CONCLUSIONS

The test sample 48-hour EC₅₀ for *Daphnia magna* was determined to be greater than the test substance solubility limit.

Submitter: 3M Company, Environmental Laboratory, P.O. Box 33331, St. Paul, Minnesota, 55133

DATA QUALITY

Reliability: Klimisch ranking 3. These studies lack analytical data for determination of the test substance concentration in the test solutions and determination of the sample purity. The tests should have been replicated. There was no description of stock solution preparation. Testing was also done well above the solubility limit of the test substance with the aid of acetone to enhance solubility.

REFERENCES

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These studies were conducted by the 3M Company, Environmental Laboratory, 3M Lab Request Number 4197, 1978.

OTHER

Submitter: 3M Company, Environmental Laboratory, P.O. Box 33331, St. Paul, Minnesota, 55133

Last changed: 5/12/00

006601



Form 6747-11-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
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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

006602

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 LC₅₀ 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^o 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).

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⁺ Dale Fattig Fish Hatchery, Brady, Nebraska

^o 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, LC₅₀ 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 LC₅₀ 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 Amphibians, Ecological Research Series 660/3-75-009, 1975.

PH
TOTAL CL ₂ RESIDUAL
C.O.D.
CONDUCTIVITY μmhos/cm
M-ALK.
P-ALK.
TOTAL SOLIDS
TOTAL HARDNESS
AMMONIA

7.8	-	<1	430	221	0	287	236	<0.1
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7.7	-	<1	440	219	0	288	23%	<0.1
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7.7	-	21	420	221	0	287	236	20.1
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7.7	-	41	420	222	0	293	225	50.1
7.8	-	41	415	231	0	284	231	50.1

7.6	=	5	443	2.21	0	1.76	3.46	50.1
8.5	=	11	402	1.54	0	1.76	3.46	50.1

7.7	=	51	400	210	0	210	210	210
7.7	=	51	400	210	0	210	210	210

7.7	50.1	41		222	0		232	40.
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7.7	50.1		405	222	0		232	50.1
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ENVIRONMENTAL ENGINEERING LABORATORY
AQUATIC TOXICITY DATA SHEET

ATTACHMENT #2

LIR 4197

#2

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.T. Elmerhaw LC 50 (95% Confidence Limits) _____

mg/L Conc.	No. Organisms Exposed	24 Hrs.			48 Hrs.			72 Hrs.			96 Hrs.		
		No. Dead	pH	D.O. ppm	No. Dead	pH	D.O. ppm	No. Dead	pH	D.O. ppm	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. <u>18°C</u>		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).

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ENVIRONMENTAL ENGINEERING LABORATORY
AQUATIC TOXICITY DATA SHEET

ATTACHMENT #2

4197

Type Test 96-Hr. Static Material Tested EC-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. 2.9 ppm, pH 7.8)
Investigator E. Pritchard LC 50 (95% Confidence Limits) _____

C.	No. Organisms Exposed	24 Hrs.			48 Hrs.			72 Hrs.			96 Hrs.		
		No. Dead	pH	D.O. ppm	No. Dead	pH	D.O. ppm	No. Dead	pH	D.O. ppm	No. Dead	pH	D.O. ppm
Control	20	0	7.8	5.8	0	7.8	4.1	1	7.8	3.4	1	7.9	3.5
100 mg/l	20	1	7.8	5.3	2	7.8	4.1	3	7.8	3.7	3	7.9	3.5
50 mg/l	20	1	7.8	5.4	1	7.8	3.6	1	7.8	3.0	1	7.8	3.1
25 mg/l	20	0	8.0	5.2	0	8.0	3.7	0	7.9	2.9	0	7.9	3.0
12.5 mg/l	20	0	7.9	5.8	0	7.9	4.4	0	7.9	3.9	3	7.9	3.8
6.25 mg/l	20	0	8.0	6.2	1	8.0	5.0	1	8.0	4.4	1	7.9	4.0
3.125 mg/l	20	1	7.9	6.1	2	7.9	4.3	3	7.9	3.9	3	7.8	3.7
1.5625 mg/l	20	1	7.8	5.2	1	7.8	3.8	1	7.8	3.5	1	7.9	3.4
													3.9
Temp.	18°C			18°C			18°C			20°C			

Notes: Used 8 ml of acetone for each conc. (0.5 ml acetone per liter)
Used 8 ml of acetone in (1) control
The two (2) concs. 180 mg/l and 220 mg/l The chemical precipitated out within 24 Hrs. The remaining conc. precipitated out within 48 Hrs.

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L.R. 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~~ ^l 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. El-Nabawawy

mg/L Conc.	No. Organisms Exposed	24 Hrs.			48 Hrs.			72 Hrs.			96 Hrs.		
		No. Dead	pH	D.O. ppm	No. Dead	pH	D.O. ppm	No. Dead	pH	D.O. ppm	No. Dead	pH	D.O. ppm
Control	20	0			2	8.6	7.9						
Control + Acute	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 used in this test were
from Mogul Corp. on 4-17-78. Used
in this study.

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ENVIRONMENTAL ENGINEERING LABORATORY
AQUATIC TOXICITY DATA SHEET

ATTACHMENT #3

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

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

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

Time Started 3:00 PM Date Started 4-27-78 Date Completed 4-29-78

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

Investigator H. Pritchard LC 50 (95% Confidence Limits) 1353 mg/l (1194-1633)
M. El-Nabawany

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						
50	20	0			0	8.7	7.4						
150	20	0			0	8.5	7.2						
300	20	2			4	8.5	7.0						
550	20	12			12	8.6	7.2						
1100	20	20	8.6	8.5	20	—	—						
1700	20	20	8.6	8.5	20	—	—						
2600	20	20	8.6	8.5	20	—	—						
3700	20	20	8.6	8.5	20	—	—						
Avg. Temp.		19°C			19°C								

Comments:

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

006609

LR 4197

D

RUN

PROBIT.B20

WEDSDAY, MAY 11, 1978 15:34:51

ENVIRONMENTAL RESEARCH LAB. - DULUTH

PROBIT ANALYSIS FOR BIOASSAY 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

CONC	LOG CONC.	FRACTION RESPONDING
650	2.812913	0
1000	3	0.2
1550	3.190332	0.6
2400	3.380211	1

CHI-SQ PROBABILITY IS > 0.05

FITTED FRACTION

0.005900574

0.1499294

0.6809147

0.9757905

Daphnia Magna

NO OF ITERATIONS

INTERCEPT (A)

SLOPE (B)

CHI SQ FOR LINEARITY

CHI SQ FOR PROBABILITY

6

-24.7904

7.917881

1.61074

0.4469226

(HETEROGENEITY FACTOR (H) = 1

LC-50 = 1351.882

G = 0.1348014

95 PCT. LOWER CONF. LIMIT =

1193.531

95 PCT. UPPER CONF. LIMIT =

1533.081

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