

ACUTE TOXICITY TO AQUATIC INVERTEBRATES (E.G., DAPHNIA)

TEST SUBSTANCE

Identity: Perfluorooctylsulfonate, didecyldimethylammonium salt; may also be referred to as Fluoroalkyl ammonium derivative. [1-Decaminium, N-decyl-N,N-dimethyl-, salt with 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-heptadecafluoro-1-octanesulfonic acid (1:1), CAS # 251099-16-8]

Remarks: The 3M production lot number was Lot 1. The test sample is L-14394 referred to by the test laboratory as P3025. The sample was labeled F-11615, Lot 1. The test sample is a mixture of the test substance in water (approximately 30-40% test substance, 60-70% water, and 0-5% of residual perfluorochemicals). All values reported relate to this mixture. The test sample appears to be a 2-phase dispersion (clear liquid with opaque solid) which rapidly separates after agitation. No calculations were made to adjust for the actual concentration of the test substance in the test sample.

METHOD

Method: OECD 202

Test type: Static acute

GLP: No

Year Completed: 1996

Species: *Daphnia magna*

Analytical monitoring: DO, pH, temperature and conductivity were monitored daily.

Statistical methods: EL₅₀ values calculated using Trimmed Spearman-Kärber method. NOEL value calculated using Steel's Many-One Rank test.

Test daphnid source: Obtained from cultures maintained by ASci Corporation, Duluth, MN.

Test daphnid age at study initiation: < 24-hours

Test conditions

Dilution water: Dechlorinated City of Duluth, MN tap water. Water was aerated for 24-hours prior to use in the test.

Dilution water chemistry:

hardness: 44 mg/L as CaCO₃

pH: 8.04

Lighting: Cool-white fluorescent bulbs. Photoperiod of 16-hours light, 8-hours dark. No transition period noted.

Stock and test solutions preparation: Water-accommodated fractions. Test solutions were prepared individually for each concentration by mass addition of vigorously shaken test substance in 1 L of dilution water. The solutions were vigorously stirred for 23-hours (vortex 1/2 to 1/3 solution depth). The aqueous phase was siphoned from the vessel at mid-depth after settling for 1-hour.

Exposure vessels: 250 mL borosilicate glass beakers containing 200 mL of test solution. The solutions were kept covered during the test.

Number of replicates: Four

Number of daphnids per replicate: Five

Number of concentrations: five plus a negative control

Water chemistry during the study:

Dissolved oxygen range (0 – 48 hours):

8.6 – 9.1 mg/L (control exposure)

8.0 – 8.8 mg/L (50 mg/L exposure)

pH range (0 – 48 hours)

8.04 – 8.11 (control exposure)

7.92 – 8.00 (50 mg/L exposure)

Test temperature range (0 – 48 hours)

20.9 – 21.0°C

Conductivity range (0 – 48 hours):

142 – 155 µmhos/cm (control exposure)

120 – 124 µmhos/cm (50 mg/L exposure)

Element basis: mortality and immobilization

RESULTS

Nominal loading concentrations: Bk control, 3.13, 6.25, 12.5, 25, 50 mg/L

Element value: 24-hour EL_{50} = 27.0 (18.7-39.0) mg/L

48-hour EL_{50} = 11.3 (9.6-13.2) mg/L

48-hour NOEL = 6.25 mg/L

All element values based on nominal concentrations.

Statistical Evaluation: The EL_{50} values and 95% confidence intervals were calculated by the Trimmed Spearman-Kärber method. The NOEL was calculated using Steel's Many-One Rank test using the TOXSTAT statistical software Version 3.2, University of Wyoming.

Biological observations: Daphnids in the negative control, and the 3.13 and 6.25 mg/L treatments appeared healthy and normal throughout the test with no mortality, immobility or overt clinical signs of toxicity. The effects noted in this study were mortality; no immobilization was noted at any test concentration. The number of surviving organisms were determined visually and recorded initially and after 24 and 48 hours.

Cumulative percent mortality:

Nominal Loading Test Conc., mg/L	24-hours	48-hours
Neg. Control	0	0
3.13	0	0
6.25	0	0
12.5	5	70
25	50	95
50	75	100

Control response: satisfactory

Remarks: Values reported are for the test sample. No calculations were made to adjust for the concentration of the test substance in the test sample.

CONCLUSIONS

The test substance 48-hour EL50 for *Daphnia magna* was determined to be 11.3 mg/L with a 95% confidence interval of 9.6-13.2 mg/L. The 48-hour no observed effect level (NOEL) was 6.25 mg/L.

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

DATA QUALITY

Reliability: Klimisch ranking 2. The study lacks analytical measurement of test substance concentrations in the test solutions and sample purity is not sufficiently characterized. Additionally, data is for a mixture and toxicity cannot be positively attributed to didecyldimethylammonium Perfluorooctylsulfonate salt alone.

REFERENCES

This study was conducted at AScl Corporation, Environmental Testing Division, Duluth, MN, at the request of the 3M Company.

OTHER

Last changed: 5/24/00

STUDY TITLE

ACUTE TOXICITY OF P3025 DEVELOPMENTAL MATERIAL
TO DAPHNIA MAGNA

DATA STANDARD

OECD GUIDELINE 202

STUDY COMPLETED

January 10, 1997

TESTING FACILITY

ASCI Corporation/ASCI-Duluth
Environmental Testing Division
4444 Airpark Boulevard
Duluth, MN 55811

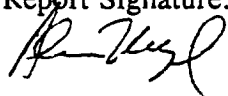
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STUDY IDENTIFICATION NUMBERS

ASCI Study ID# 5010-068

3M Company Lab Request# P3025

1.0 GENERAL INFORMATION

Study Title	Acute Toxicity of P3025 Developmental Material to <i>Daphnia magna</i> .	
Data Standard	OECD Guideline 202.	
Sponsor	3M Environmental Technology and Safety Services, Building 2-3E-09, 935 Bush Avenue, St. Paul, MN 55133.	
Sponsor's Representative	Susan A. Beach, 3M Environmental Technology and Safety Services, Building 2-3E-09, 935 Bush Avenue, St. Paul, MN 55133; Tel No. (612) 778-7452.	
Sponsor's Lab Request#	P3025.	
Testing Facility	ASCI Corporation/ASCI-Duluth Environmental Testing Division, 4444 Airpark Boulevard, Duluth, MN 55811; Tel. No. (218) 722-4040.	
Principal Investigator	Joe Dierkes.	
Project Director	Joe Amato.	
Testing Facility Director	Donald Mount.	
Definitive Test Dates	December 11-13, 1996.	
Data Validation and Report Review	Alan Mozol.	Report Signature: 
Retention of Raw Data and Final Report	Two years from the date study is completed.	
Location of Raw Data and Final Report	ASCI Corporation/ASCI-Duluth Environmental Testing Division, 4444 Airpark Boulevard, Duluth, MN 55811; Tel. No. (218) 722-4040.	

2.0 OBJECTIVE(S)

To determine the 48-h EL_{50} (effective loading) and 48-h NOEL (no observed effect loading) of the test substance, L-14394 Developmental Material, for *Daphnia magna*.

This study was conducted according to the ASCI study plan retained by the Sponsor and OECD Guideline 203 (OECD 1993). The test solutions were water-accommodated fractions (WAF) prepared following Girling, Whale, & Adema 1994.

3.0 TEST METHODS

Test Substance	Properties: P_{3025} two-phase liquid before agitated, light colored, nearly odorless slurry after agitation, specific gravity ca. 1.2 g/cc, and insoluble in water. Test loadings: 0 (dilution water control), 3.13 mg/L, 6.25 mg/L, 12.5 mg/L, 25 mg/L, and 50 mg/L. Test loading solutions were prepared individually for each concentration. The solutions were prepared by mass addition of vigorously shaken test substance in 1 L of dilution water. The solutions were vigorously stirred (vortex 1/3 to 1/2 solution depth) for 23 h. The aqueous phase was siphoned from the vessel at mid depth.
Test Organisms	Source: Healthy, less than 24-h old <i>Daphnia magna</i> neonates obtained from ASCI stock cultures maintained in water of test dilution water quality. Use in test: Gravid adults isolated in dilution water 24 h before the test initiation. Used 5 organisms/replicate and 4 replicates/test concentration. Handled with a smooth bore pipet with an inside diameter of 6 mm. Did not feed during the test.
Dilution Water	Source: Dechlorinated City of Duluth, MN, tap water. The most recent chemical analysis is in Raw Data Package. Characteristics: At test time, the water had a hardness of 44 mg/L (as $CaCO_3$) and a pH of 8.04. Aerated for 24 h prior to using in the test.

Exposure Chambers	<i>Type:</i> 250-ml borosilicate glass beakers. <i>Use in test:</i> 200 ml of dilution water or test solution/chamber. During the test, kept covered, except when experimental observations were made.
Incubation	<i>Duration:</i> 48 h. <i>Daily photoperiod:</i> 16 h light and 8 h dark, maintained using cool-white fluorescent bulbs. <i>Temperature:</i> 21°C.
Observations	<i>Biological:</i> Daily- gently agitated the exposure chambers and then counted immobilized and dead test organisms (immobilized organisms were those that did not swim within 15 seconds after exposure chambers were gently agitated). Did not remove any daphnids from the exposure chambers at 24 h. <i>Water chemistry:</i> (1) At test initiation- total hardness and alkalinity, (2) daily- temperature, pH, DO, and conductivity. All determinations were made according to APHA methods (1995).
Endpoint Calculations	<i>EL₅₀:</i> Trimmed Spearman-Kärber method. <i>NOEL:</i> Steel's Many-One Rank Test (TOXSTAT statistical software, Version 3.2, University of Wyoming).

3.0 RESULTS

Test Substance Toxicity (Pertinent data are in Table 1)	24-h EL_{50}: 27.0 mg/L (18.7-39.0 mg/L). 48-h EL_{50}: 11.3 mg/L (9.6-13.2 mg/L). 48-h NOEL: 6.25 mg/L.
Ranges of Water Chemistry Parameters	Total hardness (as $CaCO_3$ mg/L): 41-44. Alkalinity (as $CaCO_3$ mg/L): 42-45. Temperature ($^{\circ}C$): 20.9-21.0$^{\circ}C$. DO (mg/L): 7.9-9.1. pH: 7.92-8.11. Specific conductivity (μmhos/cm): 120-155.

4.0 TEST QA/QC

QA/QC Criteria	Data
Test organism dead, immobilized or trapped in control exposure must not be more than 10%.	0% dead, immobilized or trapped.
The lowest DO concentration at test termination must be at least 60% of the air saturation value at test temperature.	The lowest DO concentration measured was 89% of the air saturation value at 21.0 $^{\circ}C$.
Test duration must be 48 h.	The test duration was 48 h.
Reference substance toxicity test (this test is optional).	Current control chart for <i>D. magna</i> acute testing is included in the raw data package.

5.0 REFERENCES

American Public Health Association (APHA). 1995. Standard Methods for the Examination of Water and Wastewater. APHA, Washington, D.C.

Girling, A.E., G.F. Whale, and D.M.M. Adema. 1994. A Guideline Supplement for Determining the Aquatic Toxicity of Poorly Water-Soluble Complex Mixtures Using Water-Accommodated Fractions. Chemosphere 29(12):2645-2649.

Hamilton, M.A., R.C. Russo, and R.V. Thurston. 1977. Trimmed Spearman-Kärber Method for Estimating Median Lethal Concentrations in Toxicity Bioassays. Environ. Sci. Technol. 11(7):714-719; Correction 12(4):417 (1978).

Organization for Economic Cooperation and Development (OECD). 1993. OECD Guidelines for Testing of Chemicals. OECD Publication Information Center, Washington, D.C.

University of Wyoming. Toxstat Version 3.2. Cheyenne, Wyoming 82001.

TABLE 1. ACUTE TOXICITY OF *p3c35* DEVELOPMENTAL MATERIAL TO *DAPHNIA MAGNA*

Test Substance Loading (mg/L)	Cumulative Number and Percent of Affected Organisms ^a	
	24 h	48 h
0	0 (0)	0 (0)
3.13	0 (0)	0 (0)
6.25	0 (0)	0 (0)
12.5	1 (5)	14 (70)
25	10 (50)	19 (95)
50	15 (75)	20 (100)

^a Percentages are in parenthesis.

**EFFECT AND NO-EFFECT LOADINGS WITH
CORRESPONDING 95% CONFIDENCE INTERVALS**

EL ₅₀ and NOEL	Data (mg/L)	95% Confidence Interval
24-h EL ₅₀	27.0	18.7-39.0
48-h EL ₅₀	11.3	9.6-13.2
48-h NOEL	6.25	Not Applicable