ACUTE TOXICITY TO FISH (FATHEAD MINNOW)

TEST SUBSTANCE

Identity: Mixture containing perfluorooctanesulfonate which may also be referred to as PFOS, FC-95, or as a component of FC-201F. (1-Octanesulfonic acid, 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-heptadecafluoro-, potassium salt, CAS # 2795-39-3)

Remarks: The 3M production lot number was 1. The test sample is FC-201F (F-11129). Current information indicates that FC-201F is a mixture of 3.0% PFOS, 36.0% diethylene glycol butyl ether, 36.6% water, 8.25% acrylic foamer, 7.0% sodium octyl sulfate, 6.0% sulfonated alkyl ether sodium salts, 3.0% triethanolamine, and 0.15% tolyl triazole.

The following summary applies to a mixture with incompletely characterized concentration of impurities. Data may not accurately reflect toxicity of the fluorochemical component of the test sample.

METHOD:

Method: OECD Guideline 203.
Test type: Acute static
GLP: No
Year Completed: 1992
Species: *Pimephales promelas*
Supplier: ECT, Superior, WI, maintained at AScI-DETD, Duluth, MN.
Analytical monitoring: Temperature, pH, DO, and conductivity
Exposure period: 96-hours
Statistical methods: LC_{50} values calculated using the Trimmed Spearman-Karber method.
Test fish age: Juveniles (~30 days)
Length and weight: Length = 0.8-1.6 cm with a mean of 1.1 cm
Weight = 0.001-0.044 g with a mean of 0.013 g
Loading: 0.13 g/L
Pretreatment: Not noted.
Test conditions:
Dilution water: Shallow well water from the Two Harbors, MN area.
Dilution water chemistry:
Hardness: 155 mg/L (as $CaCO_3$)
pH: 8.25
Lighting: Cool-white fluorescent lights providing a daily photoperiod of 16 hours light and 8 hours dark.

Stock and test solution preparation: All test solutions were prepared by direct additions.
Concentrations dosing rate: Once
Stability of the test chemical solution: Described as stable.
Exposure vessels: 4.5 L rectangular glass tanks (20 cm L x 15 cm W x 15 cm H) with 1 liter of test solution.
Number of replicates: two
Number of fish per replicate: 10
Number of concentrations: 5 plus a blank control
Water chemistry during the study:

Conductivity range (0-96 hours):
346 – 371 µmhos/cm (control exposure)
396 – 437 µmhos/cm (1000 mg/L exposure)

pH range (0-96 hours):
8.24 – 8.41 (control exposure)
7.86 – 8.36 (1000 mg/L exposure)

Temperature range (0-96 hours):
20.7 – 21.5°C

Dissolved oxygen range (0-96 hours):
7.4 – 8.6 mg/L (control exposure)
4.6 – 7.9 mg/L (1000 mg/L exposure)

RESULTS

Nominal concentrations: Bk control, 120, 204, 346, 588 and 1,000 mg/L
Element value: 96-hour LC_{50} = >1,000 mg/L
96-hour NOEC = 588 mg/L

Element values based on nominal concentrations.

Remarks: Testing was conducted on the mixture as described in the Test Substance Remarks field. The value reported applies to that mixture and not the fluorochemical proportion alone.

CONCLUSIONS

The FC-201F 96-hour LC_{50} for Fathead minnow (*Pimephales promelas*) was determined to be >1,000 mg/L. The 96-hour No Observed Effects Concentration (NOEC) was 588 mg/L.

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

DATA QUALITY

Reliability: Klimisch ranking 3. Dissolved oxygen fell below an acceptable level at 24 and 48 hours for the highest test concentration in one replicate. The range finding test results did not agree with the definitive test. The range finding test resulted in 100% mortality by 24 hours at 1,000 mg/L, whereas the definitive test had only 30% mortality by 96 hours at 1,000 mg/L. Testing was redone to determine if there is a relationship between the surface area of the exposure chamber to the volume of the dilution water / test solution. The sample purity was not properly characterized and the study lacks analytical confirmation of the amount of fluorochemical proportion in solution.

REFERENCES

This study was conducted by AScI Corporation, Duluth Environmental Testing Division, Duluth, MN at the request of the 3M Company, Lab Request number K1792, 1992.

OTHER

Last changed: 6/26/00

STUDY TITLE

ACUTE TOXICITY OF FC-201F TO FATHEAD MINNOW (*PIMEPHALES PROMELAS*)

DATA STANDARD

OECD GUIDELINE 203

STUDY COMPLETED

August 24, 1992

TESTING FACILITY

AScI Corporation
AScI-Duluth Environmental Testing Division
112 East Second Street
Duluth, MN 55805

Tel. No. (218) 722-4040

STUDY IDENTIFICATION NUMBERS

AScI Study ID# 5030-015-03

3M Company Lab Request# K1792

1.0 GENERAL INFORMATION

Study Title	Acute Toxicity of FC-201F to Fathead Minnow (*Pimephales promelas*)	
Data Standard	OECD Guideline 203.	
Sponsor	3M Environmental Laboratory, Building 2-3E-09, 935 Bush Avenue, St. Paul, MN 55106.	
Sponsor's Representative	Susan A. Beach, 3M Environmental Laboratory, Building 2-3E-09, 935 Bush Avenue, St. Paul, MN 55106; Tel No. (612) 778-7452.	
Sponsor's Lab Request#	K1792.	
Testing Facility	AScI Corporation/AScI-Duluth Environmental Testing Division, 112 East Second Street, Duluth, MN 55805; Tel. No. (218) 722-4040.	
Principal Investigator	Jo A. Thompson	
Project Director	Dinesh Vaishnav	
Testing Facility Director	Donald Mount	
Definitive Test Dates	July 9-13, 1992.	
Data Validation and Report Review	Dinesh Vaishnav 8/24/92	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, 112 East Second Street, Duluth, MN 55805; Tel. No. (218) 722-4040.	

2.0 OBJECTIVE(S)

To determine the 96-h LC50 and 96-h NOEC (no observed effect concentration) of the test substance, FC-201F, for fathead minnow (*Pimephales promelas*) under static test conditions.

This study was conducted according to the AScI study plan retained by the Sponsor and OECD Guideline 203 (OECD 1984).

3.0 TEST METHODS

Test Substance	*Properties*: FC-201F, orange/red liquid, relative density 1.1 (water = 1), stable, and miscible with water. *Test concentrations*: 0.0 (dilution water control), 120, 204, 346, 588, and 1000 mg/L, prepared by direct additions. By 24 h, all test solutions, except the control, were foamy.
Test Organisms	*Source*: Healthy, juvenile fathead minnows (*Pimephales promelas*) obtained from a commercial supplier. *Holding/acclimation:* Held and acclimated for 15 days, during which exposed to the dilution water for 14 days. 0% mortality occurred during holding/acclimation period. At test termination, they were 0.8-1.6 cm in length and 0.001-0.044 g in weight with a mean of 0.013 g. *Use in test*: Did not feed 24 h before the test initiation. Used 10 organisms/replicate and 2 replicates/test concentration.

Dilution Water	*Source*: Shallow well water from the Two Harbors, Minnesota area. The most recent chemical analysis is in Raw Data Package. *Characteristics*: At test time, the water had a hardness of 155 mg/L (as $CaCO_3$) and a pH of 8.25. Aerated for 24 h prior to using in the test.
Exposure Chambers	*Type*: 4.5-L rectangular glass tanks, each measuring about 20 cm length X 15 width X 15 cm height. *Use in test*: 1 L of dilution water or test solution/chamber. During the test, kept covered, except when experimental observations were made. Gentle aeration was provided throughout the test.
Incubation	*Duration*: 96 h. *Daily photoperiod*: 16 h light and 8 h dark, maintained using cool white fluorescent lamps. *Temperature*: 20.7-21.5°C.
Observations	*Biological*: 3 h, 6 h, and daily- Test organisms with abnormal behavior and signs of stress, and dead test organisms. Removed dead test organisms when observations were made. *Water chemistry*: (1) At test initiation- total hardness and alkalinity, (2) daily- temperature, DO, and pH, and (3) at test initiation and termination- specific conductivity. All determinations were made according to APHA methods (1989).
Endpoint Calculations	*LC50*: Trimmed Spearman-Karber method (Hamilton et. al. 1977). *NOEC*: Dunnett's procedure (TOXSTAT statistical software, Version 3.1, University of Wyoming 1989).

3.0 RESULTS

Test Substance Toxicity (Pertinent data are in Table 1)	*24-h LC50*: >1000 mg/L. *48-h LC50*: >1000 mg/L. *72-h LC50*: >1000 mg/L. *96-h LC50*: >1000 mg/L. *96-h NOEC*: 588 mg/L.
Ranges of Water Chemistry Parameters	*Total hardness (as $CaCO_3$ mg/L)*: 155-160.
	Alkalinity (as $CaCO_3$ mg/L): 125-140.
	Temperature (°C): 20.7-21.5.
	DO (mg/L): 4.6-8.0.
	pH: 7.86-8.54.
	Specific conductivity (µmhos/cm): 346-437.

4.0 TEST QA/QC

QA/QC Criteria	Data
Mortality in control exposure must not be more than 10%.	0% mortality.
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 53% of the air saturation value at 21.0°C.
Test duration must be 96 h.	The test duration was 96 h.
Reference Substance Toxicity Test (this test is optional).	Final report for the most recent test with sodium chloride is in Raw Data Package.

5.0 DEVIATIONS

1. The DO fell below 60% of the air saturation at 24 h and 48 h in one replicate of the highest test concentration. The dead fish were removed and aeration was adjusted.

6.0 ADDITIONAL STUDY

In the range-finding test, which was conducted using 1 L dilution water in 1-L borosilicate glass beaker, 100% mortality occurred by 24 h at 1,000 mg/L test substance. However, in the definitive test, which was conducted as described before, only 30% mortality occurred by 96 at 1,000 mg/L test substance. As a result, the range-finding test was repeated and the data showed that by 3 h 100% mortality had occurred at 1,000 mg/L test substance. The information for the repeat test is in raw data package.

7.0 REFERENCES

American Public Health Association (APHA). 1989. Standard Methods for the Examination of Water and Wastewater. APHA, Washington, D.C.

Hamilton M.A., R.C. Russo and R.V. Thurston. 1977. Trimmed Spearman-Karber method for estimating median lethal concentration in toxicity bioassays. Environ. Sci. Technol. 11:714-719.

Organization for Economic Cooperation and Development (OECD). 1981/1984. OECD Guidelines for Testing of Chemicals. OECD Publication Information Center, Washington, D.C.

University of Wyoming. 1989. Toxstat Version 3.1. Fish Physiology and Toxicology Laboratory, Department of Zoology and Physiology. University of Wyoming, Laramie, WY.

Table 1. Acute toxicity of FC-201F to fathead minnows (*P. promelas*)

Test substance nominal concn (mg/L)	Cumulative number of dead organisms and % mortality[a]					
	3 h	6 h	24 h	48 h	72 h	96 h
0.0 (control)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)
120	0 (0)	0 (0)	0 (0)	1 (5)	1 (5)	2 (10)
204	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	1 (5)
346	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	1 (5)
588	1 (5)	1 (5)	1 (5)	1 (5)	1 (5)	1 (5)
1,000	1 (5)	1 (5)	5 (25)	5 (25)	6 (30)	6 (30)

[a]Percentages are in parenthesis.

Effect and no-effect concentrations with corresponding 95% confidence intervals

LC50 and NOEC	Data (mg/L)[b]	95% Confidence interval
24-h LC50	>1000	not calculable
48-h LC50	>1000	not calculable
72-h LC50	>1000	not calculable
96-h LC50	>1000	not calculable
96-h NOEC	588	Not applicable

[b]LC50 values were calculated based on the test substance nominal concentration.