

## ACUTE TOXICITY TO FISH (BLUEGILL SUNFISH)

### TEST SUBSTANCE

**Identity:** Mixture containing Perfluorooctanesulfonate, which may also be referred to as PFOS, FC-95, or as a component of FC-206A. (1-Octanesulfonic acid) (CAS # 2795-39-3).

**Remarks:** The 3M production lot number was cc785-22. The test sample is FC-206A. Current information indicates it is a mixture of 0.92% PFOS, 18.75% diethylene glycol butyl ether, 75.44% water, 1.83% hydroxy foamer, 1.75% sodium octyl sulfate, 1.2% poloxyethylene monooctylphenyl ether, 0.06% sodium lauryl sulfate, and 0.05% benzotriazole.

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

### METHOD:

**Method:** U.S. EPA 1975

**Type:** Acute static

**GLP:** No

**Year completed:** 1978

**Species:** *Lepomis macrochirus*

**Supplier:** Dale Fattig Fish Farm, Brady, NE.

**Analytical monitoring:** Temperature, pH, and dissolved oxygen.

**Exposure period:** 96-hours

**Statistical methods:** LC<sub>50</sub> values and 95% confidence intervals were calculated using the U.S. EPA (Duluth) probit computer program.

**Test fish age:** Juveniles

**Length and weight:** Average length = 45 mm  
Average weight = 0.92 g (wet)

**Loading:** 0.8 g/L

**Pretreatment:** None

**Test conditions:**

**Dilution water:** Carbon-filtered well water

**Dilution water chemistry:**

**Hardness:** 235 mg/L as CaCO<sub>3</sub>

**Alkalinity:** 221 mg/L as CaCO<sub>3</sub>

**Conductivity:** 418 µmhos/cm

**Lighting:** A daily photoperiod of 16 hours light and 8 hours dark with a 30-minute transition period was maintained throughout the testing period.

**Stock and test solution preparation:** The test material was added directly to the dilution water on a weight / volume basis.

**Concentrations dosing rate:** Once.

**Stability of the test chemical solutions:** Not noted.

**Exposure vessels:** Glass aquaria (35x30x20 cm) containing 16 L of test solution.

**Number of replicates:** 2

**Number of fish per replicate:** 10

**Number of concentrations:** five plus a blank control

**Water chemistry during the study:**

**pH range (0-96 hours):**

7.9 – 8.0 (control exposure)

7.5 – 7.9 (1350 mg/L exposure)\*

**Temperature range (0-96 hours):** 17 - 18 °C

**Dissolved oxygen range (0-96 hours):**

5.8 – 9.9 mg/L (control exposure)

0.1 – 7.3 mg/L (1350 mg/L exposure)\*

\* 1350 mg/L (intermediate concentration) exposure values used because the highest three concentrations used resulted in total mortality by 48 hours.

**Remarks:** Very low dissolved oxygen concentrations (0.1 – 1.8 mg/L) observed in the exposure concentrations (except for the control) by 48 hours.

## RESULTS

**Nominal concentrations:** Blank control, 1000, 1350, 1800, 2400, and 3200 mg/L (listed in text as g/L)

**Element values:** 96-hour  $LC_{50}$  = 1300 (1100 – 1400) mg/L (Duplicate 1)  
1200 (1000 – 1300) mg/L (Duplicate 2)

Element values based on nominal concentrations.

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

## CONCLUSIONS

The test sample 96-hour  $LC_{50}$  range for bluegill sunfish was determined to be 1200 - 1300 mg/L.

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

## DATA QUALITY

**Reliability:** Klimisch ranking 2. Testing meets the criteria for quality testing. However, sample purity was not properly characterized and the study lacks

analytical confirmation of the amount of fluorochemical proportion in the solution.

#### REFERENCES

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This study was conducted by the 3M Company, Environmental Laboratory, St. Paul, MN, 1978.

#### OTHER

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**Last changed: 6/26/00**



Form 6747-11-A

## TECHNICAL REPORT SUMMARY

Date

11/2/78

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            | Commercial Chemicals                                                | Project Number                    | 9970012600 |
| Report Title       | FC-206A cc 785-22: Results of Aquatic 96-Hr. LC <sub>50</sub> Tests | Report Number                     | 003        |
| To                 | D. L. Bacon                                                         |                                   |            |
| Author(s)          | A. N. Welter                                                        | Employee Number(s)                | 09362      |
| Notebook Reference |                                                                     | No. of Pages Including Coversheet | 6          |

SECURITY ►

☒ Open☐ Closed3M 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 LC<sub>50</sub> value for the bluegill sunfish (*Lepomis macrochirus*) exposed to varying concentrations of FC-206A cc 785-22.

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

The acute static 96-hr. LC<sub>50</sub> for bluegill sunfish (*Lepomis macrochirus*) exposed to varying concentrations of FC-206A cc 785-22 ranged from 1.2 to 1.3 g/l.

Information Liaison  
Initials: SKW

November 2, 1978

## CONCLUSIONS

In a replicated static 96-hour LC<sub>50</sub> aquatic test, the calculated LC<sub>50</sub> values for bluegill sunfish (*Lepomis macrochirus*) were 1.2 and 1.3g/l respectively. Materials possessing an LC<sub>50</sub> of this order of magnitude are considered to present an insignificant hazard based on the NIOSH aquatic toxicity criteria.

## INTRODUCTION

The test substance FC-306A cc 785-22 is an amber liquid. Tests performed on this material evaluated its aquatic toxicity and the data generated form the basis of this report.

## METHODS

### A. Aquatic Testing

The test protocol utilized for this study was 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 15 days prior to testing.

An acute short-term (96-hour) static bioassay was performed on FC-206A. Carbon-filtered well water of known composition was used as the diluent (Attachment 1). All glass aquaria (35 x 30 x 20 cm) containing 16:1 of diluent or diluent plus toxicant comprised a study chamber. Ten blugills, 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 experiments were conducted at 17° C. Mortality, temperature, dissolved oxygen level and pH of all test solutions were recorded. General observations relative to behavioral changes were similarly recorded when appropriate.

These studies were performed in replicate using the following FC-206A (cc 785-22) concentrations: 1.0, 1.35, 1.8, 2.4, and 3.2g/l. LC<sub>50</sub> values with 95% confidence limits were calculated using the US/EPA (Duluth) probit computer program. Organisms used in this study were considered to be generally healthy and free of disease.

\*Dale Fattig Fish Farm, Brady, Nebraska.

#Commercial Fish food of known composition.

November 10, 1978

RESULTS

The bluegill sunfish (*Lepomis macrochirus*) used in this study had an average weight of  $.92 \pm .36$  grams and a length of  $4.5 \pm 0.6$  cm. The loading factor for these studies was 0.6 g/l below the maximum recommended loading factor of 0.8 g/l (1). Temperature and pH ranges throughout this study were 17-18° C. and 7.6-8.0 pH units respectively.

Dissolved oxygen values decreased sharply 48-hour post initiation of this study. Test aquaria solutions originally were clear (normal DO levels), became straw colored (low DO), and at termination of this study had assumed a grayish-white color. This latter color change was accompanied by a relative increase in the dissolved oxygen content. Although the DO levels determined were in the range of 0.1-0.3 mg/l, there was no apparent increase in mortality due to what would be assumed to be a general hypoxic situation. The absence of an increased mortality attributable to low dissolved oxygen content relationship is taken as presumptive evidence that the calculated LC<sub>50</sub> values are reflections of test chemical induced mortality, per se.

In this replicated study, the 96-hour LC<sub>50</sub> values were 1.2, range 1.0-1.3 and 0.3, range 1.1-1.5 g/l respectively. These values translate to LC<sub>50</sub> values of 1200 and 1300 ppm. Chemicals possessing an LC<sub>50</sub> value in this range are considered to be an insignificant hazard to the aquatic environment (2).

REFERENCES

- (1) US/EPA - Methods for Acute Toxicity Test with Fish, Macro-invertebrates and Amphibians, Ecological Research Series 660/3-75-009, 1975.
- (2) Registry of Toxic Effects of Chemical Substances, '76 Edition, Christensen, H. E., and E. J. Fairchild, Eds. p. XV and Ci, U. S. Department of Health, Education and Welfare; National Institute for Occupational Safety and Health, Washington, DC, June, 1976.

*ANW/cen*

ANW/cen

Attachments

# LAB REQUEST NO. 4382 (STANDING)

DATE SHEET

ATTACHMENT #1

319 FORM

SAMPLE DESCRIPTION:  
CARBON FILTERED WELL  
20 - BIOASSAY ROOM

|         | PH  | TOTAL CL2<br>RESIDUAL | C.O.D. | CONDUCTIVITY<br>μmhos/cm | M-ALK. | P-ALK. | TOTAL<br>SOLIDS | TOTAL<br>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                      | 221    | 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      | 287             | 236               | <0.1    |  |  |  |
| 9-01-78 | 7.7 | -                     | <1     | 400                      | 220    | 0      | 276             | 236               | <0.1    |  |  |  |
| 9-07-78 | 7.7 | -                     | <1     | 400                      | 220    | 0      | 287             | 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 | 7.5 | <0.1                  | <1     | 405                      | 220    | 0      | 313             | 232               | <0.1    |  |  |  |

ENVIRONMENTAL ENGINEERING LABORATORY  
AQUATIC TOXICITY DATA SHEET

Type Test Static 96 hr. LC50 Material Tested FC 206 A Lot CC 785-22 ATB

Test Organism Bluegill Vendor Brady, N.Y. Date Rec'd 9/29/78 Batch No. 7-19-78

Avg. Wt. 919 ± 359 grams Avg. Size 4.5 ± .58 cm.

Test Solution Volume 16 Liters/Tank No. Organisms per dose 10

Time Started 11:40 Date Started 10/10/78 Date Completed 10/14/78

Dilution Water Carbon filtered well (Temp. 18 °C., D.O. 7.7 ppm, pH 7.5)  
water

Investigator Alphonse M. Wilton LC 50 (95% Confidence Limits) 1.3 1.1  
(Full Signature) 1.4

| Conc.<br>mg/l.   | Total<br>Amount<br>Added | 24-Hours                                 |      |             | 48-Hours                                 |     |             | 72-Hours                                 |     |             | 96-Hours                                 |     |             |
|------------------|--------------------------|------------------------------------------|------|-------------|------------------------------------------|-----|-------------|------------------------------------------|-----|-------------|------------------------------------------|-----|-------------|
|                  |                          | Total<br>No. Dead                        | pH   | D.O.<br>ppm | Total<br>No. Dead                        | pH  | D.O.<br>ppm | Total<br>No. Dead                        | pH  | D.O.<br>ppm | Total<br>No. Dead                        | pH  | D.O.<br>ppm |
| Control          | 0                        | 0                                        | 7.9  | 6.6         | 0                                        | 7.9 | 5.8         | 1                                        | 7.9 | 5.8         | 1                                        | 7.9 | 5.8         |
| 1.0              | 16                       | 0                                        | 7.85 | 6.3         | 2                                        | 7.6 | 3           | 2                                        | 7.6 | 2           | 2                                        | 7.6 | 1.8         |
| 1.35             | 21.6                     | 0                                        | 7.85 | 6.2         | 3                                        | 7.9 | 1           | 5                                        | 7.7 | 2           | 5                                        | 7.6 | 1.0         |
| 1.8              | 28.8                     | 0                                        | 7.9  | 6.5         | 10                                       |     |             |                                          |     |             |                                          |     |             |
| 2.4              | 38.4                     | 0                                        | 7.9  | 6.0         | 10                                       |     |             |                                          |     |             |                                          |     |             |
| 3.2              | 51.2                     | 5                                        | 7.7  | 3.5         | 10                                       |     |             |                                          |     |             |                                          |     |             |
|                  |                          | Calibrated pH at<br>end of all determin. |      |             | Calibrated pH at<br>end of all determin. |     |             | Calibrated pH at<br>end of all determin. |     |             | Calibrated pH at<br>end of all determin. |     |             |
| Test Temp. °C    |                          | 18°                                      |      |             | 18°                                      |     |             | 17°                                      |     |             | 17°                                      |     |             |
| Signature & Date |                          | A.V. Wilton 10/11/78                     |      |             | A.V. Wilton 10/12/78                     |     |             | A.V. Wilton 10/13/78                     |     |             | A.V. Wilton 10/14/78                     |     |             |

Comments: Saline medium day (2) pronounced day 2 onward, Day 4 solution became  
grey-white in color. DO. with calibrated daily against air and aerated H<sub>2</sub>O

Effects other than Mortality

| Conc.<br>mg/l    | Symptoms or Reactions | Total No. Affected   |                      |                      |                      |
|------------------|-----------------------|----------------------|----------------------|----------------------|----------------------|
|                  |                       | 24-Hr.               | 48-Hr.               | 72-Hr.               | 96-Hr.               |
| Control          |                       | 0                    | 0                    | 0                    | 0                    |
| 1.0              | <u>Surfaceing</u>     | 1                    | 3                    | 8                    | 8                    |
| 1.35             | "                     | 1                    | 7                    | 5                    | 5                    |
| 1.8              | "                     | 6                    | 7                    |                      |                      |
| 2.4              | "                     | 10                   |                      |                      |                      |
| 3.2              | "                     | 5                    | 2                    |                      |                      |
| Signature & Date |                       | A.V. Wilton 10/11/78 | A.V. Wilton 10/12/78 | A.V. Wilton 10/13/78 | A.V. Wilton 10/14/78 |

Comments: \_\_\_\_\_



ENVIRONMENTAL ENGINEERING LABORATORY  
AQUATIC TOXICITY DATA SHEET

Type Test Static 48h LC50 Material Tested FC DCL A Lot cc 785-22  
 Test Organism Bluegill Vendor Brook, No. 6 Date Rec'd 7/29/78 Batch No 7-1978  
 Avg. Wt. .9145, 359 grams Avg. Size 4.5 ± .58 cm.  
 Test Solution Volume 16 Liters/Tank No. Organisms per dose 10  
 Time Started 11:40 AM Date Started 10/10/78 Date Completed 10/17/78  
 Dilution Water Carbon filtered well (Temp. 18 °C, D.O. 7.9 ppm, pH 7.8)  
 Investigator Alphonse M. Walter LC 50 (95% Confidence Limits) 1.2 1.3  
 (Full Signature)

| Conc.<br>mg/l.   | Total<br>Amount<br>Added | 24-Hours             |     |             | 48-Hours               |     |             | 72-Hours             |     |             | 96-Hours             |     |             |
|------------------|--------------------------|----------------------|-----|-------------|------------------------|-----|-------------|----------------------|-----|-------------|----------------------|-----|-------------|
|                  |                          | Total<br>No. Dead    | pH  | D.O.<br>ppm | Total<br>No. Dead      | pH  | D.O.<br>ppm | Total<br>No. Dead    | pH  | D.O.<br>ppm | Total<br>No. Dead    | pH  | D.O.<br>ppm |
| Control          | 7 ppm                    | 0                    | 7.9 | 7.1         | 0                      | 8.0 | 6.1         | 0                    | 8.0 | 6.0         | 0                    | 8.0 | 6.0         |
| 1.0              | 16                       | 0                    | 7.9 | 6.8         | 2                      | 7.6 | .2          | 2                    | 7.7 | .2          | 2                    | 7.7 | 1.7         |
| 1.35             | 21.6                     | 0                    | 7.9 | 7.3         | 5                      | 7.6 | .2          | 8                    | 7.6 | .2          | 8                    | 7.7 | .2          |
| 1.8              | 28.8                     | 0                    | 7.9 | 6.9         | 8                      | 7.6 | .1          | 10                   |     |             |                      |     |             |
| 2.4              | 38.4                     | 0                    | 7.9 | 6.3         | 8                      | 7.6 | .1          | 10                   |     |             |                      |     |             |
| 3.2              | 51.2                     | 2                    | 7.9 | 6.3         | 10                     |     |             |                      |     |             |                      |     |             |
|                  |                          | Calibrated pH at end |     |             | + Calibrated pH at end |     |             | calibrated pH at end |     |             | Calibrated pH at end |     |             |
| Test Temp. °C    |                          | 18°C                 |     |             | 18°                    |     |             | 17°                  |     |             | 17°                  |     |             |
| Signature & Date |                          | ANWalter 10/11/78    |     |             | ANWalter 10/12/78      |     |             | ANWalter 10/13/78    |     |             | ANWalter 10/14/78    |     |             |

Comments: Solution became quite cloudy at day 4. Day 4 solution became grey-white in color. D.O. meter calibrated daily against air and checked 11.0

Effects other than Mortality

EC50

| Conc.<br>mg/l    | Symptoms or Reactions | Total No. Affected |                   |                   |                   |
|------------------|-----------------------|--------------------|-------------------|-------------------|-------------------|
|                  |                       | 24-Hr.             | 48-Hr.            | 72-Hr.            | 96-Hr.            |
| Control          |                       | 0                  | 0                 | 0                 | 0                 |
| 1.0              |                       | 0                  | 8                 | 8                 | 8                 |
| 1.35             | <u>surfacing</u>      | 2                  | 5                 | 2                 | 2                 |
| 1.8              |                       | 5                  | 2                 |                   |                   |
| 2.4              |                       | 10                 | 2                 |                   |                   |
| 3.2              |                       | 6                  | 1                 |                   |                   |
| Signature & Date |                       | ANWalter 10/11/78  | ANWalter 10/12/78 | ANWalter 10/13/78 | ANWalter 10/14/78 |

Comments: