

AR226-1766

Wildlife International, Ltd.

Project Number 454A-109

- 1 -

PFOS:  
A SEMI-STATIC LIFE-CYCLE TOXICITY TEST  
WITH THE CLADOCERAN (*Daphnia magna*)

ADDENDUM

WILDLIFE INTERNATIONAL, LTD. PROJECT NUMBER: 454A-109  
3M LAB REQUEST NO. U2723

U.S. Environmental Protection Agency  
Series 850 – Ecological Effects Test Guidelines  
OPPTS Number 850.1300  
and  
OECD Guideline 211

This study was reported using a test substance purity of 90.49%. The test substance characterization was revised on 9/7/00 and the purity was determined to be 86.9%. The Sponsor requested that the results be revised based on the most recent purity analyses. The results previously presented were simply corrected by the ratio of the two purities or a factor of 0.9603. No attempt was made to recalculate analytical standard concentrations or measured concentrations from original raw data. As such, minor errors due to rounding may be present. Attached is the revised summary page from the final report and the most recent certificate of analysis.

RECEIVED  
OPPT NCIS  
2004 MAY 12 AM 9:12

- 2 -

**REVISED STUDY SUMMARY BASED ON A PURITY OF 86.9%**

|                                                             |                                                       |
|-------------------------------------------------------------|-------------------------------------------------------|
| SPONSOR:                                                    | 3M Corporation                                        |
| SPONSOR'S REPRESENTATIVE:                                   | Susan A. Beach                                        |
| LOCATION OF STUDY, RAW DATA AND A COPY OF THE FINAL REPORT: | Wildlife International Ltd.<br>Easton, Maryland 21601 |

  

|                                             |                                                                                                                            |
|---------------------------------------------|----------------------------------------------------------------------------------------------------------------------------|
| WILDLIFE INTERNATIONAL LTD. PROJECT NUMBER: | 454A-109                                                                                                                   |
| TEST SUBSTANCE:                             | PFOS (Perfluorooctane Sulfonic Acid Potassium Salt)                                                                        |
| STUDY:                                      | PFOS: A Semi-Static Life-Cycle Toxicity Test with the Cladoceran ( <i>Daphnia magna</i> )                                  |
| NOMINAL TEST CONCENTRATIONS                 | Negative Control, 1.3, 2.8, 5.5, 11, 22 and 44 mg a.i./L                                                                   |
| MEAN MEASURED TEST CONCENTRATIONS:          | Negative Control, 1.4, 2.8, 5.4, 12, 23 and 46 mg a.i./L                                                                   |
| TEST DATES:                                 | Experimental Start – July 12, 1999<br>Biological Termination – August 2, 1999<br>Experimental Termination – August 4, 1999 |
| LENGTH OF TEST:                             | 21 Days                                                                                                                    |

  

|                           |                                                                |
|---------------------------|----------------------------------------------------------------|
| TEST ORGANISM:            | Neonate Cladocerans ( <i>Daphnia magna</i> )                   |
| SOURCE OF TEST ORGANISMS: | Wildlife International Ltd. cultures<br>Easton, Maryland 21601 |
| AGE OF TEST ORGANISMS:    | Juveniles < 24 hours old                                       |

  

|       |              |
|-------|--------------|
| NOEC: | 12 mg a.i./L |
| LOEC: | 23 mg a.i./L |
| MATC: | 17 mg a.i./L |

**CERTIFICATE OF ANALYSIS**

# INTERIM CERTIFICATE OF ANALYSIS

Revision 1(9/7/00)

Centre Analytical Laboratories COA Reference #: 023-018A

3M Product: PFOS, Lot 217

Reference #: SD-018

Purity: 86.9%

| Test Name                         | Specifications               | Result                      |
|-----------------------------------|------------------------------|-----------------------------|
| Purity <sup>1</sup>               |                              | 86.9%                       |
| Appearance                        | White Crystalline Powder     | Conforms                    |
| Identification<br>NMR             |                              | Positive                    |
| Metals (ICP/MS)                   |                              |                             |
| 1. Calcium                        |                              | 1. 0.005 wt./wt. %          |
| 2. Magnesium                      |                              | 2. 0.001 wt./wt. %          |
| 3. Sodium                         |                              | 3. 1.439 wt./wt. %          |
| 4. Potassium <sup>2</sup>         |                              | 4. 6.849 wt./wt. %          |
| 5. Nickel                         |                              | 5. <0.001 wt./wt. %         |
| 6. Iron                           |                              | 6. 0.005 wt./wt. %          |
| 7. Manganese                      |                              | 7. <0.001 wt./wt. %         |
| Total % Impurity (NMR)            |                              | 1.93 wt./wt. %              |
| Total % Impurity<br>(LC/MS)       |                              | 8.41 wt./wt. %              |
| Total % Impurity<br>(GC/MS)       |                              | None Detected               |
| Related Compounds –<br>POAA       |                              | 0.33 wt./wt. %              |
| Residual Solvents (TGA)           |                              | None Detected               |
| Purity by DSC                     |                              | Not Applicable <sup>3</sup> |
| Inorganic Anions (IC)             |                              |                             |
| 1. Chloride                       |                              | 1. <0.015 wt./wt. %         |
| 2. Fluoride                       |                              | 2. 0.59 wt./wt. %           |
| 3. Bromide                        |                              | 3. <0.040 wt./wt. %         |
| 4. Nitrate                        |                              | 4. <0.009 wt./wt. %         |
| 5. Nitrite                        |                              | 5. <0.006 wt./wt. %         |
| 6. Phosphate                      |                              | 6. <0.007 wt./wt. %         |
| 7. Sulfate <sup>4</sup>           |                              | 7. 8.76 wt./wt. %           |
| Organic Acids <sup>5</sup> (IC)   |                              |                             |
| 1. TFA                            |                              | 1. <0.1 wt./wt. %           |
| 2. PFPA                           |                              | 2. <0.1 wt./wt. %           |
| 3. HFBA                           |                              | 3. 0.10 wt./wt. %           |
| 4. NFPA                           |                              | 4. 0.28 wt./wt. %           |
| Elemental Analysis <sup>6</sup> : |                              |                             |
| 1. Carbon                         | 1. Theoretical Value = 17.8% | 1. 12.48 wt./wt. %          |
| 2. Hydrogen                       | 2. Theoretical Value = 0%    | 2. 0.244 wt./wt. %          |
| 3. Nitrogen                       | 3. Theoretical Value = 0%    | 3. 1.74 wt./wt. %           |
| 4. Sulfur                         | 4. Theoretical Value = 5.95% | 4. 8.84 wt./wt. %           |
| 5. Fluorine                       | 5. Theoretical Value = 60%   | 5. 54.1 wt./wt. %           |

**INTERIM CERTIFICATE OF ANALYSIS**  
**Centre Analytical Laboratories COA Reference #: 023-018A**

Date of Last Analysis: 08/31/00

Expiration Date: 08/31/01

Storage Conditions: Frozen  $\square$ -10°C

Re-assessment Date: 08/31/01

<sup>1</sup>Purity = 100% - (sum of metal impurities, 1.45% +LC/MS impurities, 8.41%+Inorganic Fluoride, 0.59%+NMR impurities, 1.93%+organic acid impurities, 0.38%+POAA, 0.33%)

Total impurity from all tests = 13.09%

Purity = 100% - 13.09% = 86.9%

<sup>2</sup>Potassium is expected in this salt form and is therefore not considered an impurity.

<sup>3</sup>Purity by DSC is generally not applicable to materials of low purity. No endotherm was observed for this sample.

<sup>4</sup>Sulfur in the sample appears to be converted to SO<sub>4</sub> and hence detected using the inorganic anion method conditions. The anion result agrees well with the sulfur determination in the elemental analysis, lending confidence to this interpretation. Based on the results, the SO<sub>4</sub> is not considered an impurity.

|                  |                           |
|------------------|---------------------------|
| <sup>5</sup> TFA | Trifluoroacetic acid      |
| HFBA             | Heptafluorobutyric acid   |
| NFPA             | Nonofluoropentanoic acid  |
| PFPA             | Pentafluoropropanoic acid |

<sup>6</sup>Theoretical value calculations based on the empirical formula, C<sub>8</sub>F<sub>17</sub>SO<sub>3</sub> K<sup>+</sup> (MW=538)

This work was conducted under EPA Good Laboratory Practice Standards (40 CFR 160).

***INTERIM CERTIFICATE OF ANALYSIS***  
**Centre Analytical Laboratories COA Reference #: 023-018A**

LC/MS Purity Profile:

| <b>Impurity</b> | <b>wt./wt. %</b> |
|-----------------|------------------|
| C4              | 1.22             |
| C5              | 1.33             |
| C6              | 4.72             |
| C7              | 1.14             |
| <b>Total</b>    | <b>8.41</b>      |

Note: The C4 and C6 values were calculated using the C4 and C6 standard calibration curves, respectively. The C5 value was calculated using the average response factors from the C4 and C6 standard curves. Likewise, the C7 value was calculated using the average response factors from the C6 and C8 standard curves.

Prepared By: \_\_\_\_\_  
David S. Bell  
Scientist, Centre Analytical Laboratories

\_\_\_\_\_  
Date

Reviewed By: \_\_\_\_\_  
John Flaherty  
Laboratory Manager, Centre Analytical Laboratories

\_\_\_\_\_  
Date



September 27, 2000

Ms. Susan A. Beach  
3M Corporation  
Environmental Laboratory  
935 Bush Avenue  
3M Building 2-3E-09  
P.O. Box 33331  
St. Paul, Minnesota 55133

Dear Susan:

Enclosed please find Addendums for Wildlife International, Ltd. Project Numbers 454A-101, 454A-102, 454A-103A, 54A-104, 454A-105, 454A-106, 454A-107, 454A-108 and 454A-109

If you have any questions, please let me know.

Sincerely,

Kurt R. Drott  
Senior Biologist

KRD/cyw8/3m  
Enclosures