

AR226-1768

Wildlife International, Ltd.

Project Number 454C-107

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DETERMINATION OF THE WATER SOLUBILITY OF PFOS
BY THE SHAKE FLASK METHOD

ADDENDUM

WILDLIFE INTERNATIONAL, LTD. PROJECT NUMBER: 454C-107
3M LAB REQUEST NO. U2723

OECD Guideline for the Testing of Chemicals, 105
Water Solubility

This study was originally reported using a test substance purity of 98.9%. 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 measured concentrations originally reported were simply corrected by the ratio of the two purities, or a factor of 0.8787. No attempt was made to recalculate analytical standard concentrations. 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.

REVISED STUDY SUMMARY BASED ON A PURITY OF 86.9%

STUDY TITLE:	Determination of the Water Solubility of PFOS by the Shake Flask Method
WILDLIFE INTERNATIONAL LTD.	
PROJECT NUMBER:	454C-107
SPONSOR:	3M Corporation
TESTING FACILITY:	Wildlife International Ltd. Easton, Maryland 21601
LOCATION OF STUDY, RAW DATA AND A COPY OF THE FINAL REPORT:	Wildlife International Ltd. Easton, Maryland 21601

TEST SUBSTANCE:	PFOS
TEST DATES:	Experimental Start - February 11, 1999 Experimental Termination - February 20, 1999

TEST SYSTEM:	NANOpure® Water
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SUMMARY:	The overall mean solubility concentration of PFOS in NANOpure® water was 498 mg a.i./L (SD = 46.4; CV = 9.32%; N = 6).
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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 ¹		86.9%
Appearance	White Crystalline Powder	Conforms
Identification 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 ²		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 (LC/MS)		8.41 wt./wt. %
Total % Impurity (GC/MS)		None Detected
Related Compounds - POAA		0.33 wt./wt. %
Residual Solvents (TGA)		None Detected
Purity by DSC		Not Applicable ³
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 ⁴		7. 8.76 wt./wt. %
Organic Acids ⁵ (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 ⁶ :		
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. %

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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 $\leq 10^{\circ}\text{C}$

Re-assessment Date: 08/31/01

¹Purity = 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%

²Potassium is expected in this salt form and is therefore not considered an impurity.

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

⁴Sulfur in the sample appears to be converted to SO_4 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_4 is not considered an impurity.

⁵ TFA	Trifluoroacetic acid
HFBA	Heptafluorobutyric acid
NFPA	Nonofluoropentanoic acid
PFPA	Pentafluoropropanoic acid

⁶Theoretical value calculations based on the empirical formula, $\text{C}_2\text{F}_{17}\text{SO}_3\text{K}^+$ (MW=538)

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

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

LC/MS Purity Profile:

Impurity	wt./wt. %
C4	1.22
C5	1.33
C6	4.72
C7	1.14
Total	8.41

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



November 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 the following studies:

1. **NON-GLP STUDY: DETERMINATION OF THE SOLUBILITY OF PFOS IN WILDLIFE INTERNATIONAL, LTD. FRESHWATER, FRESHWATER ALGAL MEDIUM, AND SALTWATER**
WILDLIFE INTERNATIONAL, LTD. PROJECT No.: 454C-101-1
3M LAB REQUEST No. U2723
2. **DETERMINATION OF THE WATER SOLUBILITY OF PFOS BY THE SHAKE FLASK METHOD**
WILDLIFE INTERNATIONAL, LTD. PROJECT No.: 454C-107
3M LAB REQUEST No. U2723

If you have any questions, please let me know.

Sincerely,

Raymond L. Van Hoven, Ph.D.
Scientist

RLV/cyw8/3m
Enclosures