28) OECD 105-OPPTS 830.7840, water solubility in nanopure water, 454C-118

DETERMINATION OF THE WATER SOLUBILITY OF PERFLUOROBUTANE SULFONATE, POTASSIUM SALT (PFBS) BY THE SHAKE FLASK METHOD

T-7485 PFBS

WILDLIFE INTERNATIONAL, LTD. PROJECT NUMBER: 454C-118

OECD Guideline for the Testing of Chemicals, 105
Water Solubility

AUTHORS:

Raymond L. VanHoven, Ph.D.
Jon A. MacGregor, B.S.
Willard B. Nixon, Ph.D.

STUDY INITIATION DATE: July 27, 2000

STUDY COMPLETION DATE: August 30, 2000

Submitted to

3M Corporation
Environmental Laboratory
935 Bush Avenue
St. Paul, Minnesota 55144

Wildlife International, Ltd.
8598 Commerce Drive
Easton, Maryland 21601
(410) 822-8600

GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT

SPONSOR: 3M Corporation

TITLE: Determination of the Water Solubility of Perfluorobutane Sulfonate, Potassium Salt (PFBS) by the Shake Flask Method

WILDLIFE INTERNATIONAL, LTD. PROJECT NUMBER: 454C-118

3M ENVIRONMENTAL LAB PROJECT NUMBER: E00-1429

STUDY COMPLETION: August 30, 2000

This study was conducted in compliance with Good Laboratory Practice Standards as described in OECD Principles of Good Laboratory Practice (ENV/MC/CHEM (98) 17), with the following exceptions:

The test substance was not characterized in accordance with Good Laboratory Practice Standards.

The stability of the test substance under storage conditions at the test site was not determined in accordance with Good Laboratory Practice Standards.

STUDY DIRECTOR:

Raymond L. VanHoven, Ph.D.
Scientist
Wildlife International, Ltd.

8-30-00
DATE

SPONSOR APPPROVAL:

9/11/00
DATE

QUALITY ASSURANCE STATEMENT

This study was examined for compliance with Good Laboratory Practice Standards as described in OECD Principles of Good Laboratory Practice (ENV/MC/CHEM (98) 17). The dates of all inspections and audits and the dates that any findings were reported to the Study Director and Laboratory Management were as follows:

		DATE REPORTED TO:	
ACTIVITY:	DATE CONDUCTED:	STUDY DIRECTOR:	MANAGEMENT:
Sample Preparation	July 27, 2000	July 27, 2000	July 27, 2000
Draft Report and Data	August 1, 2000	August 1, 2000	August 3, 2000
Final Report	August 30, 2000	August 30, 2000	August 30, 2000

Susan L. Coleman

Susan L. Coleman, B.A.
Senior Quality Assurance Representative

8-30-00

DATE

REPORT APPROVAL

SPONSOR: 3M Corporation

TITLE: Determination of the Water Solubility of Perfluorobutane Sulfonate, Potassium Salt (PFBS) by the Shake Flask Method

WILDLIFE INTERNATIONAL, LTD. PROJECT NUMBER: 454C-118

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STUDY DIRECTOR:

Raymond L. VanHoven, Ph.D.
Scientist

8/30/00
DATE

MANAGEMENT:

Willard B. Nixon, Ph.D.
Manager, Analytical Chemistry

8/30/00
DATE

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SUMMARY

STUDY TITLE:	Determination of the Water Solubility of Perfluorobutane Sulfonate, Potassium Salt (PFBS) by the Shake Flask Method
WILDLIFE INTERNATIONAL, LTD. PROJECT NUMBER:	454C-118
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:	PFBS
TEST DATES:	Experimental Start – July 27, 2000 Experimental Termination – July 31, 2000

TEST SYSTEM:	NANOpure® Water

SUMMARY:	The overall mean solubility concentration of PFBS in NANOpure® water was 46.2 g PFBS/L (SD = 0.18; CV = 0.38%; N = 6).

INTRODUCTION

This study was conducted by Wildlife International, Ltd. for 3M Corporation at the Wildlife International, Ltd. analytical chemistry facility in Easton, Maryland. A test was conducted to determine the water solubility of PFBS (perfluorobutane sulfonate, potassium salt). The experimental portion of this study was conducted between July 27, 2000 and July 31, 2000. The original raw data generated by Wildlife International, Ltd. and a copy of the final report are filed under Project Number 454C-118 in archives located on the Wildlife International, Ltd. site.

PURPOSE

The purpose of this study was to determine the water solubility of PFBS at 20 ± 0.5°C by the shake flask method. Determination of water solubility by the shake flask method is applicable to test substances with water solubilities equal to or exceeding 0.01 gram/Liter (g/L).

EXPERIMENTAL DESIGN

The water solubility of PFBS was determined at a temperature of 20 ± 0.5°C. A preliminary test was performed to estimate the water solubility. The test consisted of additions of increasingly larger amounts of NANOpure® water to a known weight of test substance at room temperature. The definitive test consisted of equilibration of an excess amount of test substance with NANOpure® water at 30°C followed by equilibration at 20°C and analyzing subsamples by high performance liquid chromatography with mass spectrometric detection (LC/MS).

MATERIALS AND METHODS

This study was conducted according to the procedures outlined in the protocol, "Determination of the Water Solubility of Perfluorobutane Sulfonate, Potassium Salt (PFBS) by the Shake Flask Method." The protocol was based on procedures outlined in the OECD Guideline for Testing of Chemicals, 105: *Water Solubility* (1). This study meets data requirements under this guideline and under U.S. EPA Product Properties Test Guidelines,

SANITIZED

Series:830.7840, *Water Solubility:Column Elution Method; Shake Flask Method* (2) for shake flask methodology and TSCA Title 40 of the Federal Code of Regulations, Part 796, Section 1840: *Water Solubility* (3).

Test Substance

The test substance was received from 3M Environmental Technology and Safety Services on March 27, 2000, assigned Wildlife International, Ltd. Identification number 5216, and stored under ambient conditions. The test substance, a white powder, was identified as: Potassium Perfluorobutane Sulfonate expiration date: March 2010. The test substance was further identified with the 3M Environmental Laboratory test control and reference number . The test substance had a reported purity of 97.90% (Appendix II).

Reagents

All solvents used in the solubility analyses were of ACS reagent grade. NANOpure® water (equivalent to ASTM Type II Designation D1193-91) was used (4).

Preliminary Test Procedure

A preliminary test was conducted to estimate the water solubility of PFBS. The test was performed at room temperature by adding approximately 10 mg of the test substance to a 10-mL volumetric flask. Increasing volumes of NANOpure® water were added (0.1 and 0.4 mL) to the flask. After each addition, the flask was shaken vigorously for approximately 1 minute and visually examined for undissolved test substance. After the addition of a total volume of 0.5-mL of NANOpure® water, no particulates were observed.

Definitive Test Procedure

Approximately 10.0 grams of the test substance was weighed into each of three labeled 8-ounce French square bottles. NANOpure® water, 100 mL equilibrated at 20°C for ~30 minutes, was added to each bottle. The bottles were sealed with screwcaps and wrapped with Parafilm®. The bottles were agitated in a shaker-water bath (Lindberg/Blue, Model No. SWB1122A) maintained at 30 ± 1.0°C. After one day, one bottle was removed and placed in a second constant temperature bath (Lindberg/Blue, Model No. SWB1122A, with agitation switched off) maintained at 20 ± 0.5°C for 24 hours with occasional shaking. Triplicate 10-mL subsamples were removed

from the bottle, placed into 15-mL glass centrifuge tubes, and centrifuged at ~2,000 rpm (845g) for ~5 minutes. A single ~10-mL subsample of the NANOpure® water matrix was centrifuged along with the treated samples for use as a reagent blank. Because undissolved, suspended material was evident upon completion of this centrifugation, a second and more vigorous centrifugation was incorporated into the methodology. Approximately 1-mL aliquots of the supernatant from each subsample in the 15-mL centrifuge tubes were then transferred to plastic microcentrifuge tubes and centrifuged at ~13,000 rpm (14,000g) for ~5 minutes. An appropriate volume from each subsample aliquot was volumetrically removed and diluted into the calibration range of the LC/MS methodology with 50% methanol and 50% NANOpure® water dilution solvent. The dilutions were placed into autosampler vials for analysis by reverse-phase high performance liquid chromatography (HPLC) using a Hewlett-Packard Model 1100 High Performance Liquid Chromatograph with a Perkin-Elmer API 100LC Mass Spectrometer equipped with a Perkin-Elmer Turbo IonSpray ion source. The remaining two bottles were treated in a similar manner following initial equilibration periods of 2 and 3 days at 30.0 ± 1.0°C. Instrumental parameters for the analysis of PFBS are summarized in Table 1 and a method flow chart is provided in Figure 1.

Calibration Curve

Calibration standards of PFBS, ranging in concentration from 1.00 mg PFBS/L to 5.00 mg PFBS/L, were analyzed with each sample set. A linear regression equation was generated using the peak area responses versus the respective concentrations of the calibration standards. A typical calibration curve is presented in Figure 2. The concentration of PFBS in the samples was determined by substituting the peak area response into the applicable linear regression equation. Representative ion chromatograms of low and high calibration standards are presented in Figures 3 and 4, respectively.

Reagent Blanks and Limit of Quantitation

Reagent blanks were analyzed to assess the presence of potential interferences. The reagent blank, consisting of a subsample from the same batch of NANOpure® water as was used for preparation of the 100 g PFBS/L (target nominal concentration) test samples, was processed in an identical manner as for the treated samples. The limit of quantitation (LOQ) for these analyses, calculated as the product of the lowest calibration standard (1.00 mg PFBS/L) and the dilution factor of the reagent blank samples (12500), was 12500 mg PFBS/L = 12.5 g PFBS/L. No chromatographic interferences were observed in the reagent blanks at or above the limit of

quantitation of 12.5 g PFBS/L (Table 2). A representative ion chromatogram of a reagent blank sample is presented in Figure 5.

Example Calculations

Sample number 454C-118-2A, target nominal concentration of 100 g PFBS/L in NANOpure® water.

First Initial Volume: 0.200 mL
First Final Volume: 25.0 mL
Second Initial Volume: 0.100 mL
Second Final Volume: 10.0 mL
Dilution Factor: 12500
PFBS Peak Area: 17031882

Calibration curve equation.
Slope: 3535642.25
Intercept: 4000869
Curve is weighted: (1/x)
Instrument Software: MacQuan, version 1.6.

$$\text{PFBS (g/L) in sample} = \frac{\text{peak area - (y-intercept)}}{\text{slope}} \times \text{dilution factor} \times \text{unit conversion factor}$$

$$= \frac{17031882-4000869}{3535642.25} \times 12500 \times \frac{1\ \text{g}}{1000\ \text{mg}}$$

$$= 46.1$$

RESULTS

Water Solubility

Triplicate subsamples were removed from the appropriate bottles after one, two and three days of shaking in a water bath maintained at 30 ± 1.0°C and following one day of a 20 ± 0.5°C equilibration period (Table 3). Analysis of aqueous subsamples after one day had a mean analytical result of 44.1 g PFBS/L (SD = 0.40,

CV = 0.92%). For subsamples collected after two and three days, the mean concentrations were 46.1 g PFBS/L (SD = 0.15, CV = 0.33%) and 46.3 g PFBS/L (SD = 0.15, CV = 0.33%), respectively. A representative ion chromatogram of a Day 2 solubility sample is presented in Figure 6.

CONCLUSIONS

The Day 2 and Day 3 mean solubility concentrations were well within the 15% agreement criterion of the protocol and were averaged to obtain the overall mean solubility concentration. The overall mean solubility concentration of PFBS in NANOpure® water was 46.2 g PFBS/L (SD = 0.18; CV = 0.38%; N = 6).

REFERENCES

1. **Organisation for Economic Cooperation and Development.** 1995. Guideline for Testing of Chemicals, 105: *Water Solubility.*

2. **Product Properties Test Guidelines.** 1996. OPPTS 830.7840. *Water Solubility: Column Elution Method; Shake Flask Method.*

3. **TSCA Title 40 of the Federal Code of Regulations.** 1994. Part 796, Section 1840: *Water Solubility.*

4. **American Society for Testing and Materials.** 1991. Standard Specification for Reagent Water. D1193-91, ASTM Section II Water and Environmental Technology, Vol. 11.01:45-47.

Table 1

Typical LC/MS Operational Parameters

INSTRUMENT:	Hewlett-Packard Model 1100 High Performance Liquid Chromatograph with a Perkin-Elmer API 100LC Mass Spectrometer operated in Selective Ion Monitoring (SIM) Mode
SOURCE:	Perkin-Elmer TurboIonSpray
ANALYTICAL COLUMN:	Keystone PRISM RP (30 mm x 1.5 mm, 3-µm particle size)
GUARD COLUMN	Keystone Javelin C18 cartridge (20 x 2 mm)
OVEN TEMPERATURE:	40°C
STOP TIME:	3.00 min
FLOW RATE:	200 µL/min
MOBILE PHASE:	25% NANOpure® Water with 0.1% Ammonium Formate: 75% Methanol
INJECTION VOLUME:	5.0 µL
PFBS PEAK RETENTION TIME:	Approximately 1.5 minutes
PFBS MONITORED MASS:	299.0 amu

Table 2

Reagent Blanks Analyzed Concurrently During Sample Analysis

Sample		Measured Concentration of PFBS (g PFBS/L)[1]
Number (454C-118-)	Type	
REB-1	Reagent Blank	< LOQ
REB-2	Reagent Blank	< LOQ
REB-3	Reagent Blank	< LOQ

[1] The limit of quantitation (LOQ) of 12500 mg PFBS/L = 12.5 g PFBS/L was based upon the product of the lowest calibration standard analyzed (1.00 mg PFBS/L) and the dilution factor of the respective blank sample (12500).

Table 3

Solubility of PFBS in NANOpure® Water

Sample Number (454C-118-)	Day of Test	Concentration of PFBS		Mean SD CV
		Fortified (g PFBS/L)	Measured (g PFBS/L)	
1A	1	100	44.3	Mean = 44.1
				SD = 0.40
1B	1	100	43.6	CV = 0.92%
1C	1	100	44.3	
2A	2	100	46.1	Mean = 46.1
				SD = 0.15
2B	2	100	45.9	CV = 0.33%
2C	2	100	46.2	
				Mean = 46.3
3A	3	100	46.3	SD = 0.15
				CV = 0.33%
3B	3	100	46.4	
3C	3	100	46.1	
	Totals:	Mean (Days 2 and 3)=	46.2	
		SD =	0.18	
		CV =	0.38%	
		N =	6	

Note: Results generated using MacQuan version 1.6 software and manual calculations. Values have been rounded for reporting purposes.

METHOD OUTLINE FOR THE PROCESSING OF PFBS IN NANOPURE® WATER

Prepare calibration standards in a solution of 50% methanol and 50% NANOpure® water using volumetric flasks and gas-tight syringes.

↓

Prepare solubility samples by weighing approximately 10 grams of PFBS into three 8-ounce French square bottles and adding 100 mL of NANOpure® water to each.

↓

After the appropriate equilibration periods, centrifuge triplicate subsamples at approximately 2,000 rpm for approximately 5 minutes

↓

Centrifuge an aliquot of each subsample at approximately 13000 rpm for approximately 5 minutes

↓

Dilute subsample aliquots into the range of calibration with a solution of 50% methanol and 50% NANOpure® water

↓

Transfer samples and standards to autosampler vials for analysis by LC/MS.

Figure 1. Analytical method flow chart for the analysis of PFBS in NANOpure® water.

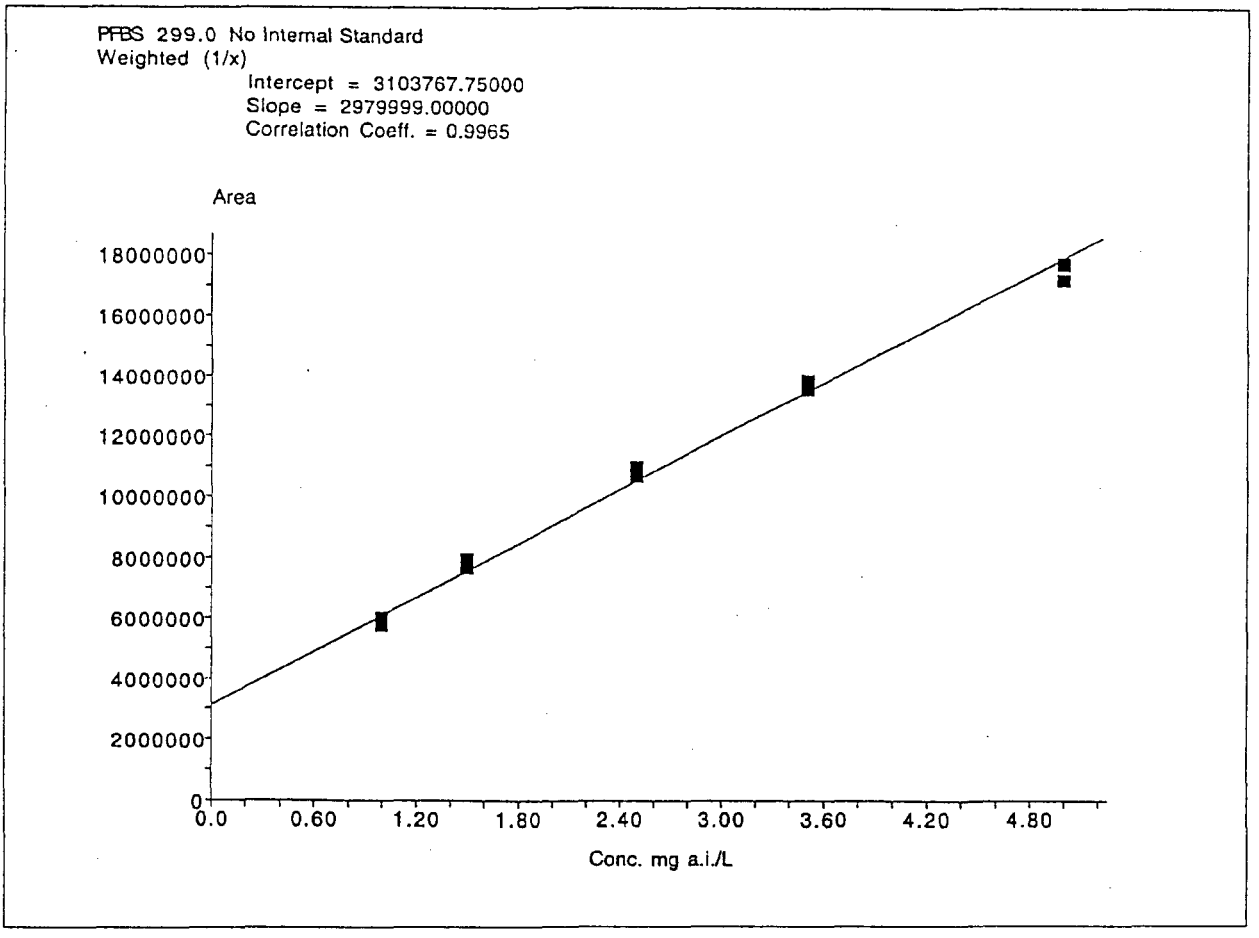

Figure 2. A typical calibration curve for PFBS.

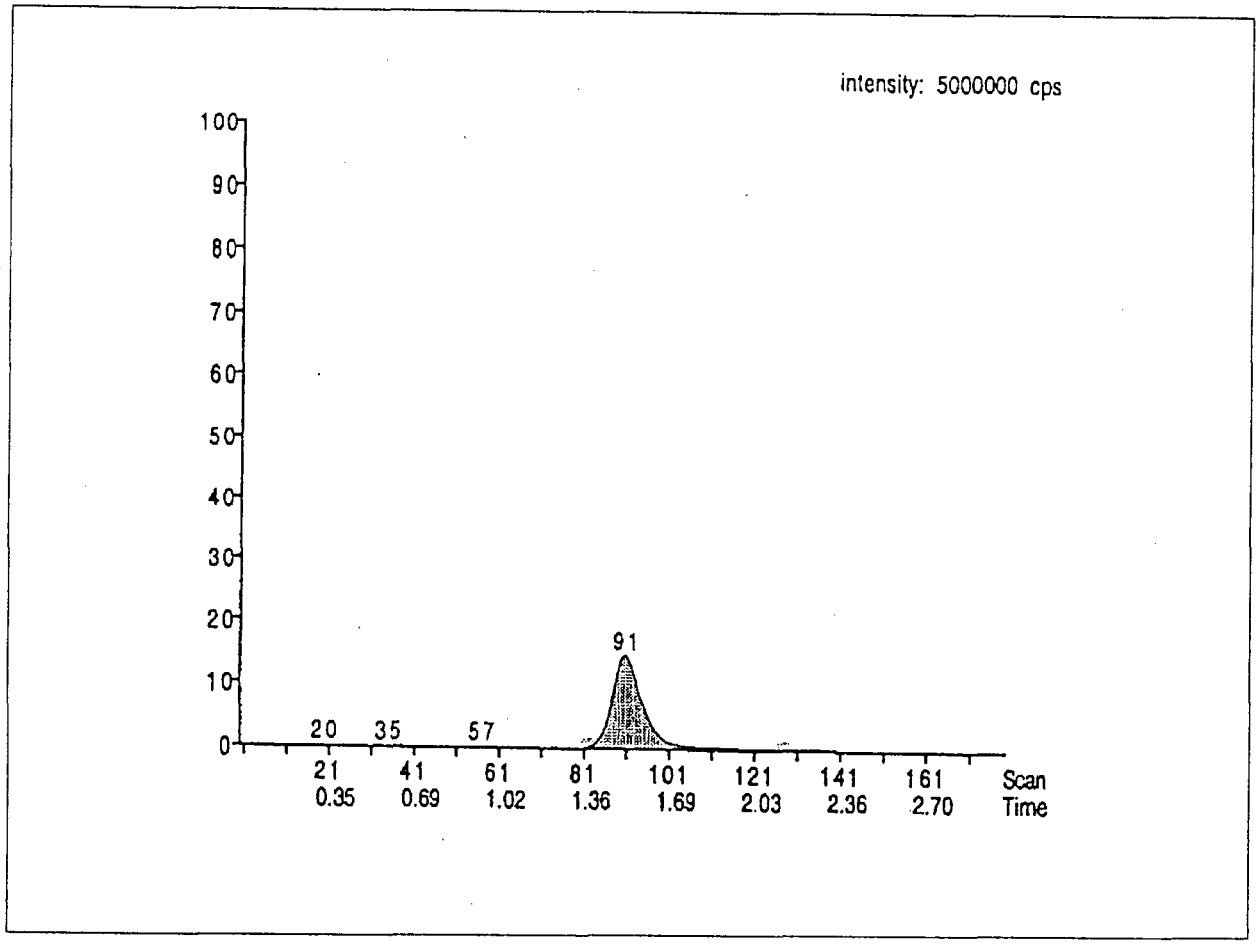

Figure 3. A representative ion chromatogram of a low-level (1.00 mg/L) PFBS standard.

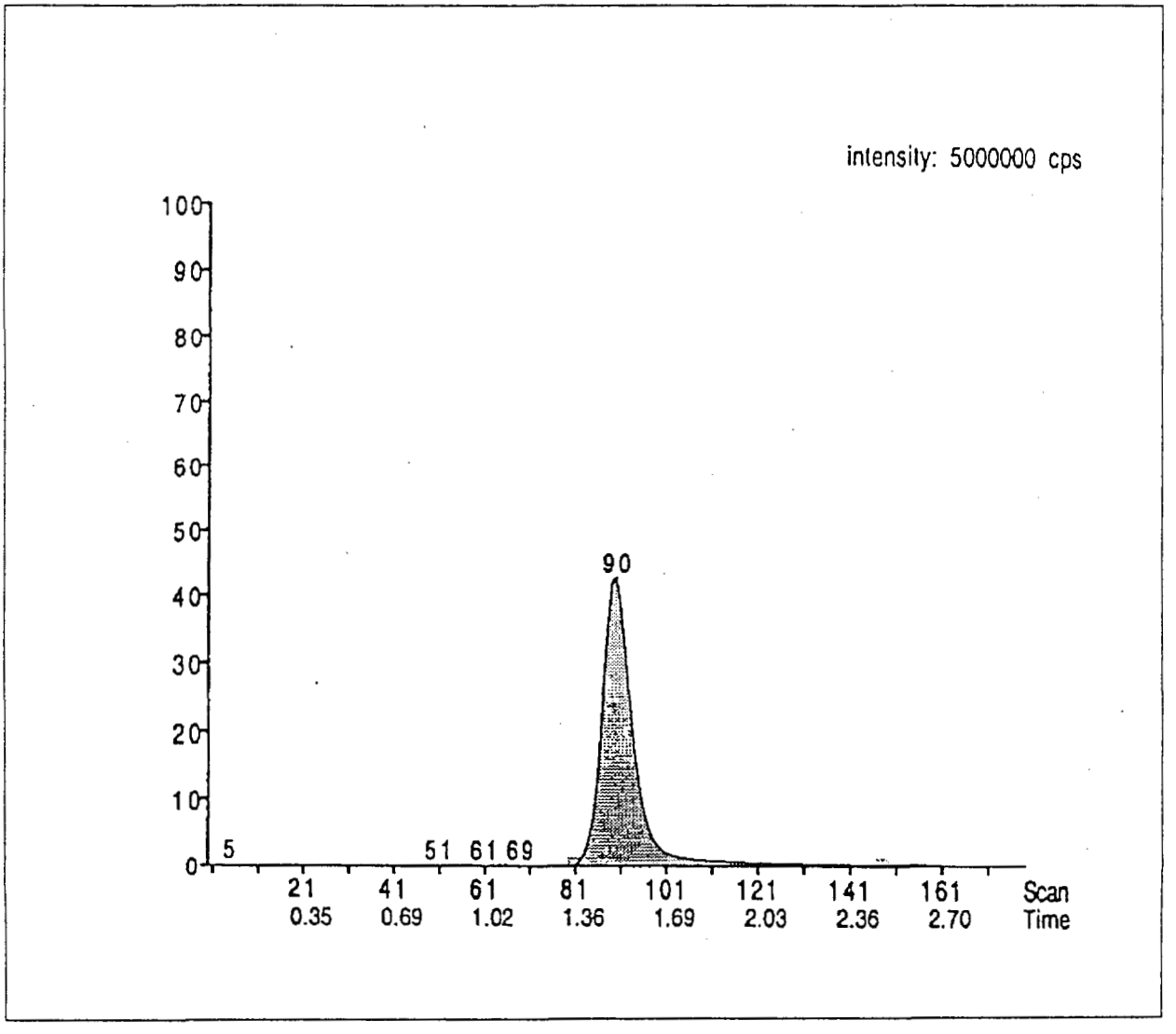

Figure 4. A representative ion chromatogram of a high-level (5.00 mg/L) PFBS standard.

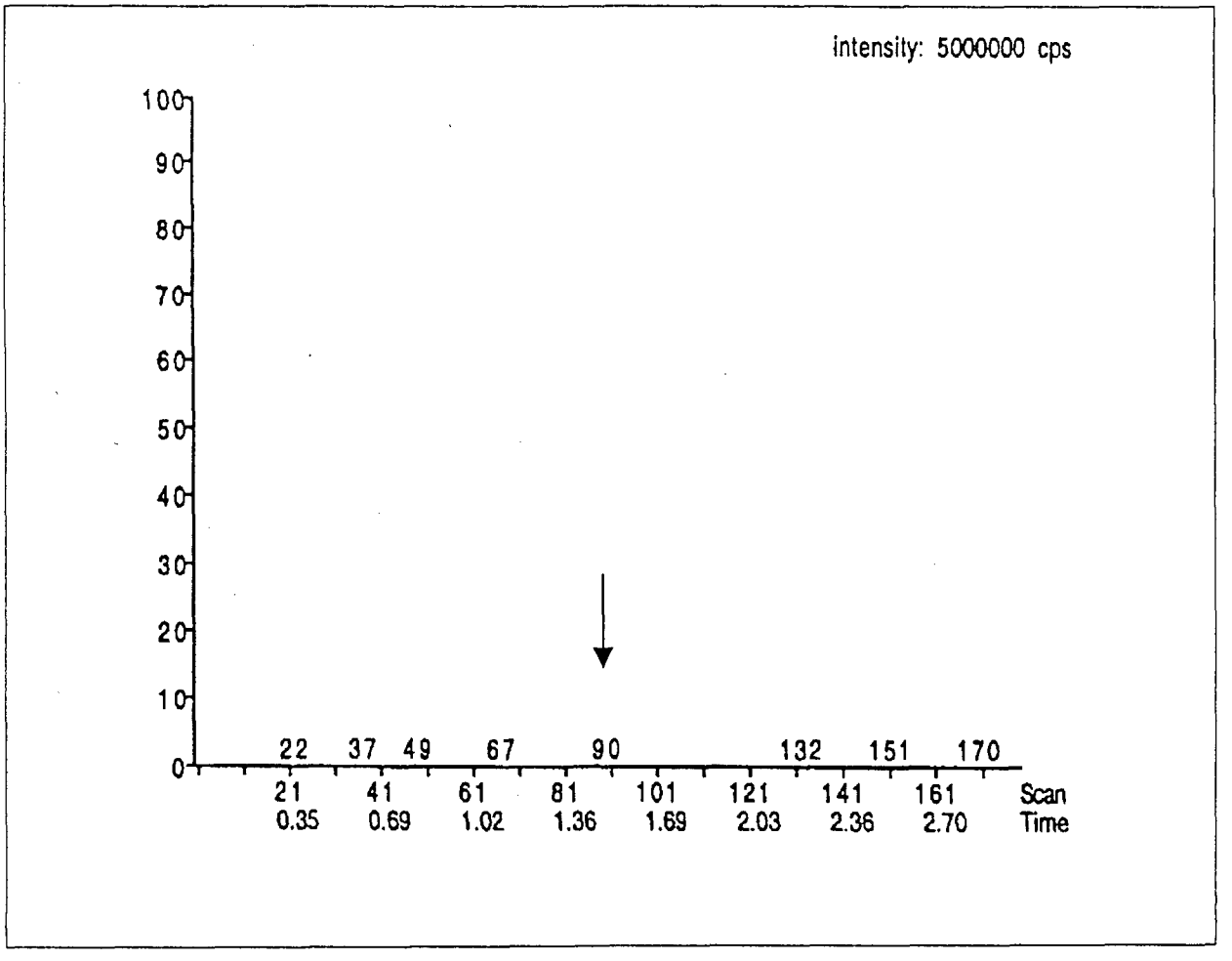

Figure 5. A representative ion chromatogram of a reagent blank sample (454C-118-REB-1). The arrow indicates the retention time of PFBS.

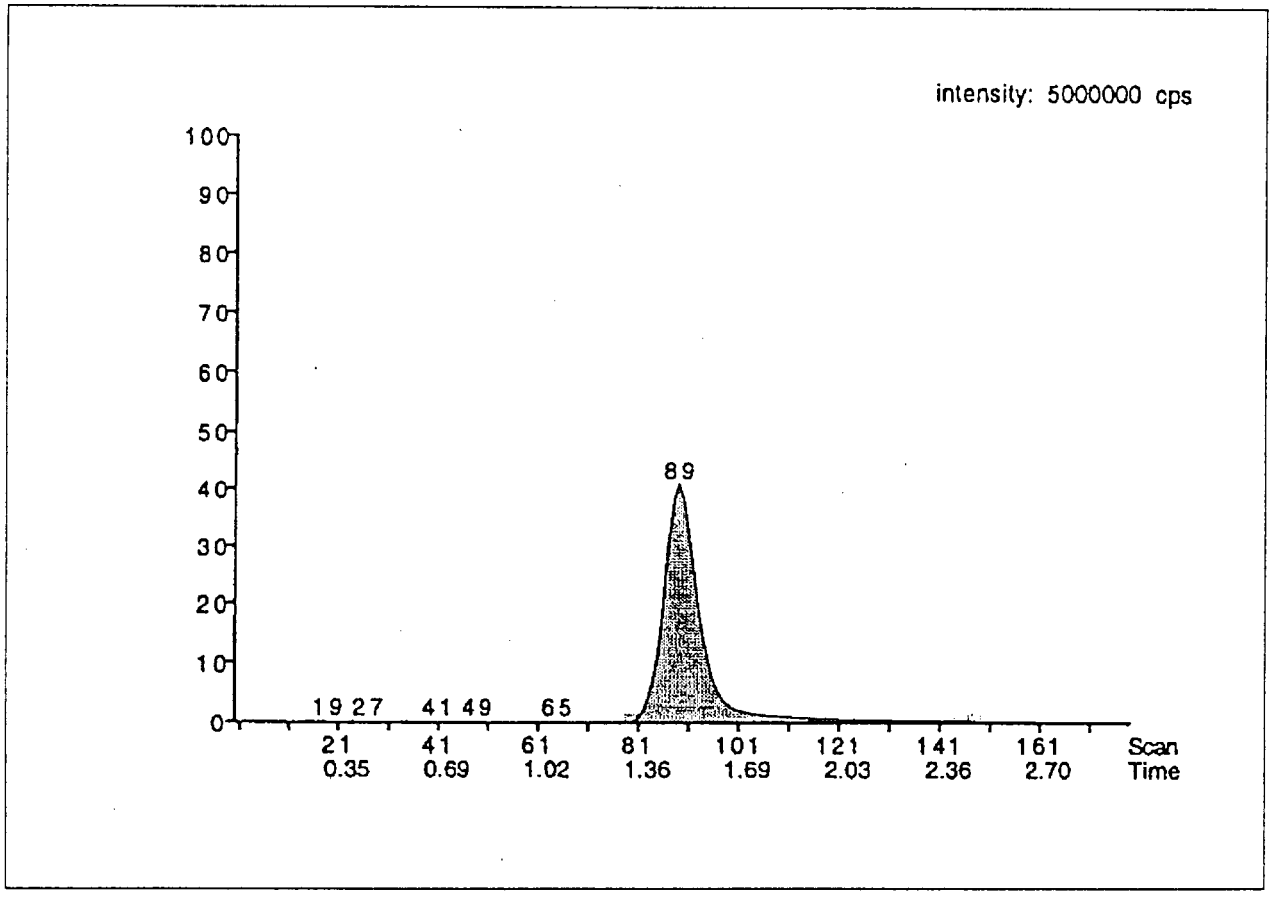

Figure 6. A representative ion chromatogram of a Day 2 solubility sample, 454C-118-2A (nominal concentration of 100 g PFBS/L. Sample diluted 12500x).

APPENDIX I

Protocol and Protocol Amendment

PROTOCOL

DETERMINATION OF THE WATER SOLUBILITY OF
PERFLUOROBUTANE SULFONATE, POTASSIUM SALT (PFBS)
BY THE SHAKE FLASK METHOD

OECD Guideline for the Testing of Chemicals, 105
Water Solubility

3M Lab Project No. E00-1429

Submitted to

3M Corporation
Environmental Laboratory
935 Bush Avenue
St. Paul, Minnesota 55144

Wildlife International, Ltd.

8598 Commerce Drive
Easton, Maryland 21601
(410) 822-8600

July 17, 2000

Wildlife International, Ltd.

DETERMINATION OF THE WATER SOLUBILITY OF
PERFLUOROBUTANE SULFONATE, POTASSIUM SALT (PFBS)
BY THE SHAKE FLASK METHOD

SPONSOR: 3M Corporation
Environmental Laboratory
935 Bush Avenue
St. Paul, Minnesota 55144

SPONSOR'S REPRESENTATIVE:

TESTING FACILITY: Wildlife International, Ltd.
8598 Commerce Drive
Easton, Maryland 21601

STUDY DIRECTOR: Raymond L. Van Hoven, Ph.D.
Scientist

LABORATORY MANAGEMENT: Willard B. Nixon, Ph.D.
Manager of Chemistry

FOR LABORATORY USE ONLY

Proposed Dates:

Experimental Start Date: 7/27/00

Experimental Termination Date: 8/27/00

Project No.: 454C-118

Test Substance No.: 5216

Receipt Date: 3/27/2000

PROTOCOL APPROVAL

STUDY DIRECTOR — DATE: 7/27/00

LABORATORY MANAGEMENT — DATE: 9/27/00

DATE: 7/20/00

PROTOCOL NO.: 454/071700/105/SUB454 3M LAB PROJECT NO. E00-1429

Wildlife International, Ltd.

INTRODUCTION

Wildlife International, Ltd. will experimentally determine the water solubility of Perfluorobutane Sulfonate, Potassium Salt (PFBS). The study will be conducted at the Wildlife International, Ltd. analytical chemistry facility in Easton, Maryland. The study will be performed based on guidance presented in the OECD Guideline for Testing of Chemicals, 105: *Water Solubility* (1) for shake flask methodology. This study is intended to meet data requirements under this guideline and under EPA Product Properties Test Guidelines, OPPTS 830.7840, *Water Solubility: Column Elution Method; Shake Flask Method* (2) and TSCA Title 40 of the Federal Code of Regulations, Part 796, Section 1840: *Water Solubility* (3). Raw data for all work performed at Wildlife International, Ltd. and a copy of the final report will be filed by project number in archives located on the Wildlife International, Ltd. site or at an alternative location to be specified in the final report.

OBJECTIVE

The objective of this study is to determine the water solubility of Perfluorobutane Sulfonate, Potassium Salt, hereafter referred to as PFBS, at 20 ± 0.5°C by the shake flask method.

EXPERIMENTAL DESIGN

The water solubility of PFBS will be determined at a temperature of 20 ± 0.5°C. A preliminary test will be performed to estimate the water solubility at room temperature. The preliminary test consists of additions of increasingly large amounts of water to a known weight of test substance until solubilization is effected. The definitive test consists of equilibration of an excess amount of test substance with water at an elevated temperature, 30°C, followed by equilibration at 20°C.

MATERIALS AND METHODS

Test Substance

The test and reference items (hereinafter referred to as test and reference substances) will be PFBS. Information on the characterization of test and reference substances is required by Good Laboratory Practice Standards (GLP). The Sponsor is responsible for providing Wildlife International, Ltd. written verification that the test and reference substances have been characterized according to GLPs prior to initiation of the study. If written verification of GLP test and reference substance characterization is not provided to Wildlife International, Ltd., it will be noted in the compliance statement of the final report.

Reagents

Water that meets ASTM Type II standards (ASTM D 1193-91) will be used (4). Other solvents may be needed for the analytical method or preparation of stock solutions and analytical standards. All solvents will be ACS reagent grade or better, and will be determined to be free of contaminants that interfere with the quantitation of the test substance.

Preliminary Test Procedure

A preliminary test will be conducted to estimate the solubility of the test substance at room temperature. Approximately 10 mg of the test substance will be placed in a 10-mL glass-stoppered graduated cylinder or flask. Increasing volumes of water will be added stepwise as shown below:

Total volume of water added (mL)	0.1	0.5	1	2	10
Approximate solubility (g/L)	>100	20	10	5	1

After each addition of water to yield the total volume indicated, the container will be shaken vigorously for approximately 10 minutes and visually examined for undissolved test substance. If the test substance is not dissolved after addition of 10 mL of water, the contents of the container will be transferred to a 100-mL cylinder or flask and water added for a final volume of 100 mL. Following shaking, the flask or cylinder will be visually checked for undissolved material, i.e., undissolved material would indicate a water solubility <0.1 g/L. Preliminary testing of the solubility at lower concentrations (<0.1 g/L) may be performed by transferring the contents of the 100-mL cylinder or flask to a 1-L vessel or reinitiating the test with a lesser amount of test substance, e.g., 1 mg.

Definitive Test Procedure

The quantity of test substance necessary to saturate the desired volume of water is estimated from the preliminary test. The volume of water used will depend on the analytical method and solubility range. Greater than (or equal to) five times the quantity of test substance required to achieve water solubility will be transferred to a minimum of three glass or Teflon® test vessels. The desired volume of water will be added to each vessel and the vessels sealed. The closed vessels will be agitated (shaken or stirred) at 30 ± 1.0°C in a water bath. After 1 day, one of the vessels will be removed and re-equilibrated for 24 hours at 20 ± 0.5°C

with occasional shaking or stirring. The contents of the vessel will then be centrifuged. The concentration of the test substance in a minimum of two aliquots of the aqueous phase will be determined. The remaining two vessels are treated similarly following initial equilibration periods of 2 and 3 days at 30 ± 1.0°C. If the concentration results from the last two vessels agree to within 15%, the test is satisfactory. If the results from the three vessels show a trend of increasing values, and/or at the request of the Sponsor, the test may be reinitiated with longer equilibration periods.

Method

The analytical method to be used for quantitation of the test substance will be LC/MS based on procedures developed at Wildlife International, Ltd. The method used was developed at Wildlife International, Ltd. and entitled "Analytical Method Validation for the Determination of Perfluorobutane Sulfonate, Potassium Salt (PFBS) in Freshwater" (Wildlife International, Ltd. Project No. 454C-115).

Calculations

The concentration of the test substance in each vessel will be expressed in g/L, mg/L or µg/L in water as appropriate. The average solubility for test vessels with solubilities within 15% of each other will be averaged and reported.

Sample Handling and Safety

The Sponsor will identify any special handling or safety precautions to be used with the above referenced test substance. All normal precautions with respect to handling and storage will be taken.

Sample and Test Substance Retention

Upon completion of testing, portions of the test substance used as part of this study will be disposed of in accordance with federal, state and local regulations. Any unused portion of the test substance will be returned to the Sponsor.

RECORDS TO BE MAINTAINED

Records to be maintained for data generated by Wildlife International, Ltd. will include, but not be limited to:

1. A copy of the signed protocol.

2. Identification and characterization of the test substance, if provided by the Sponsor.
3. Dates of initiation and completion of the study.
4. Dates of experimental start and termination.
5. Storage conditions of the test substance.
6. Test substance use log.
7. Concentration calculations and records of solution preparation.
8. Instrument operating conditions and chromatograms, if applicable.
9. Statistical calculations.
10. Test conditions.
11. A copy of the final report.

FINAL REPORT

Wildlife International, Ltd will prepare a final report of the results of the study. The report will include, but not be limited to the following, when applicable:

1. Name and address of the facility performing the study.
2. Dates upon which the study was initiated and completed.
3. A statement of compliance signed by the Study Director addressing any exceptions to Good Laboratory Practice Standards.
4. Purpose and procedure, as stated in the approved protocol, including all amendments and deviations to the protocol.
5. The test substance identification, including name or code number, purity, empirical formula, molecular formula, lot or batch number, method of analysis, and any other information provided by the Sponsor.
6. Description of the test method or reference to the method used along with any modifications made.
7. The individual concentrations of each sample.
8. The means and standard deviations of solubility determinations at each interval for each temperature tested.
9. Description of any problems experienced and how they were resolved.
10. A statement prepared by the Quality Assurance Unit listing the dates that study inspections and audits were made and findings reported to the Study Director and Management.

- 7 -

CHANGING OF PROTOCOL

Planned changes to the protocol will be in the form of written amendments signed by the Study Director and the Sponsor. Amendments will be considered as part of the protocol and will be attached to the final protocol. Any other changes will be in the form of written deviations filed with the raw data. All changes to the protocol will be indicated in the final report.

GOOD LABORATORY PRACTICES

This study will be conducted according to the Good Laboratory Practices described in OECD (ENV/MC/CHEM (98) 17). Each study conducted by Wildlife International, Ltd. is routinely examined by the Wildlife International, Ltd. Quality Assurance Unit for compliance with Good Laboratory Practices, Standard Operating Procedures and the specified protocol. A statement of compliance with Good Laboratory Practices will be prepared for all portions of the study conducted by Wildlife International, Ltd. The Sponsor will be responsible for compliance with Good Laboratory Practices for procedures performed by other laboratories. Raw data for all work performed at Wildlife International, Ltd. and a copy of the final report will be filed by project number in archives located on the Wildlife International, Ltd. site or at an alternative location to be specified in the final report

REFERENCES

1 **Organisation for Economic Cooperation and Development.** 1995. Guideline for Testing of Chemicals, 105: *Water Solubility.*

2 **Product Properties Test Guidelines.** 1996. OPPTS 830.7840. *Water Solubility: Column Elution Method; Shake Flask Method.*

3 **TSCA Title 40 of the Federal Code of Regulations.** 1994. Part 796, Section 1840. *Water Solubility.*

4 **American Society for Testing and Materials.** 1991. Standard Specification for Reagent Water. D1193-91, ASTM Section II Water and Environmental Technology, Vol. 11.01: 45-47.

Wildlife International, Ltd.

DEVIATION TO STUDY PROTOCOL

STUDY TITLE: Determination of the Water Solubility of Perfluorobutane Sulfonate, Potassium Salt (PFBS) by the Shake Flask Method

PROTOCOL NO.: 454/071700/105/SUB454 DEVIATION NO.: 1

SPONSOR: 3M Corporation PROJECT NO.: 454C-118

DATE OF DEFACTO DEVIATION: July 27, 2000

DEVIATION: Page 4, Preliminary Test Procedure

The protocol states that the container will be shaken vigorously for approximately 10 minutes and visually examined for undissolved test substance upon each addition of water. In the experiment, the container was shaken for only approximately one minute.

REASON: Analyst oversight.

IMPACT: In the opinion of the Study Director, the cited deviation during the preliminary test had no impact on the design and conduct of the definitive test.

STUDY DIRECTOR
Raymond L. Van Hoven, Ph.D.

7-27-00
DATE

LABORATORY MANAGEMENT
Willard B. Nixon, Ph.D.

7/27/00
DATE

APPENDIX II

Certificate of Analysis

$C_4F_9SO_3^-K^+$ Lot 1; PFBS)
Lot 2 (1999)

April 17, 2000
(Supercedes Certificate of Analysis dated April 11, 2000)

This sample was analyzed using and elemental analyses techniques. The results of these tests show the sample to contain the following weight percent composition:

	0.13 %
$C_4F_9SO_3^-K^+$	97.90%
	1.96 %

Additionally, the isomer distribution of the sample was determined using ^{19}F-NMR techniques and found to contain the following weight percent composition:

$CF_3(CF_2)_3$-$SO_3^-K^+$ (Normal chain)	97.86 %
	0.04 %

Analytical Study Number: 60488

APPENDIX III

Personnel Involved in the Study

The following key Wildlife International, Ltd. personnel were involved in the conduct or management of this study:

1. Willard B. Nixon, Ph.D., Manager, Analytical Chemistry
2. Raymond L. VanHoven, Ph.D., Scientist
3. Jon A. MacGregor, B.S., Scientist
4. Frank J. Lezotte, B.S., Chemist