PROTOCOL

DETERMINATION OF THE VAPOR PRESSURE OF PFOS USING THE SPINNING ROTOR GAUGE METHOD

OECD Guideline for Testing of Chemicals, 104
Vapor Pressure Curve

3M Lab Request No. U2723

Submitted to

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Environmental Laboratory
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St. Paul, Minnesota 55133

WILDLIFE INTERNATIONAL LTD.

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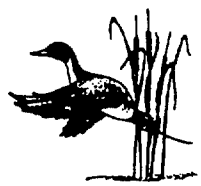

November 24, 1998

DETERMINATION OF THE VAPOR PRESSURE OF PFOS
USING THE SPINNING ROTOR GAUGE METHOD

SPONSOR:	3M Corporation Environmental Laboratory P.O. Box 33331 St. Paul, Minnesota 55133
SPONSOR'S REPRESENTATIVE:	Ms. Susan A. Beach
TESTING FACILITY:	Wildlife International Ltd. 8598 Commerce Drive Easton, Maryland 21601
STUDY DIRECTOR:	~~Joel Stenzel~~ Raymond L. VanHoven, Ph.D.
LABORATORY MANAGEMENT:	Willard B. Nixon, Ph.D. Manager of Chemistry

FOR LABORATORY USE ONLY

Proposed Dates:

Experimental Start Date: 01-12-99 Experimental Termination Date: 02-10-99

Project No.: 454C-105

Test Concentrations: NA

Test Substance No.: 1675A Reference Substance No. (if applicable): NA

PROTOCOL APPROVAL

Raymond L. VanHoven — 01/12/99
STUDY DIRECTOR — DATE

Willard B. Nixon — 1/12/99
LABORATORY MANAGEMENT — DATE

Susan A. Beach — 11/30/98
SPONSOR'S REPRESENTATIVE — DATE

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INTRODUCTION

Wildlife International Ltd. will determine the vapor pressure of PFOS (perfluorooctane sulfonic acid, potassium salt) using the spinning rotor gauge method. The study will be conducted at the Wildlife International Ltd. analytical chemistry facility in Easton, Maryland. The study will be performed following procedures in the OECD Guideline for Testing of Chemicals, 104, *Vapor Pressure Curve* (1) and Product Properties Test Guidelines, OPPTS 830.7950, *Vapor Pressure* (2). 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 at Wildlife International Ltd. or at an alternative location to be specified in the final report.

PURPOSE

The purpose of this study is to determine the vapor pressure of PFOS at approximately 20°C using the spinning rotor gauge method.

EXPERIMENTAL DESIGN

The spinning rotor gauge (SRG) method described in this protocol is applicable to both liquids and solids with vapor pressures in the approximate range of 0.5 to 10^{-4} Pascal (Pa). The vapor pressure of a control sample (empty sample chamber) and a single sample of the test substance will be determined three times each. The temperature of the sample chambers will be maintained at 20.0 ± 0.5°C.

MATERIALS AND METHODS

Test Substance

The Sponsor will be asked to provide the following information concerning the test substance, if available:

Chemical Name (or designation)
Lot or Batch Number
Purity
Molecular Weight
Composition (for multi-component mixtures)
Empirical formula(s)
Expiration Date

The attached form **IDENTIFICATION OF TEST SUBSTANCE BY SPONSOR** (Appendix I) is to be used to provide available information. The Sponsor is responsible for information on the purity and composition of the test substance, as well as all information related to the safe handling of the test substance. The Sponsor also agrees to accept any unused test substance or test substance containers remaining at the end of the study.

Apparatus

The high vacuum system consists of a turbomolecular pump (Leybold TURBOVAC 50) and rough pump (Leybold TRIVAC S 1,6B) fitted with an ion gauge and controller (Metra 731 Digital Ion Gauge Controller), a spinning rotor gauge (Leybold VR200), and sample introduction system. The sample introduction system consists of two high vacuum glass sample chambers with valves and a glass to metal transition flange that connects to the vacuum system. The apparatus is presented schematically in Figure 1.

Test Procedure

The high vacuum system, with attached sample introduction system and SRG, will be evacuated to high vacuum prior to taking measurements. The system will be heated to at least 50°C for twelve hours or more, then allowed to cool to room temperature. The SRG will be used to measure the deceleration rate at the system's ultimate vacuum (offset). The offset value will be entered and the SRG used to measure vapor pressures.

A sample of the test substance will be placed in one of the sample chambers; the other sample chamber will remain empty. The chamber containing test substance and the blank sample chamber will be degassed thoroughly, typically several hours, to remove entrapped air and water vapor. The sample chamber temperatures will be controlled at 20±0.5°C. The vapor pressure of each sample chamber will be measured at least three times as follows: The sample chamber will be opened to the vacuum system. The SRG will monitor and record pressure at 30-second intervals. The system will be allowed to achieve a steady-state pressure, then the valves to the vacuum pumps will be closed. The SRG will monitor and record changes in pressure at 30-second intervals for at least 10 minutes. The system will be allowed to achieve equilibrium with the sample. The valves will be opened again and the measurements repeated.

Calculations

The mean and standard deviation of the steady-state pressure will be calculated from the pressure measurements made just prior to closing the valves to the vacuum pumps. A linear regression (least squares fit) will be performed on the vapor pressure measurements collected after the valves to the vacuum pumps are closed. The out-gassing rate of the system will be equal to the slope of the regression equation for the blank sample. The uncorrected vapor pressure of the sample will be equal to the intercept of the regression equation, i.e. the vapor pressure at the moment the valves were closed. If significant, the vapor pressure determined for the blank sample chamber will be considered the baseline or background pressure and will be subtracted from each vapor pressure measurement for the test substance chamber. The means and standard deviations of the vapor pressures and out-gassing rates will be calculated.

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. Spinning rotor gauge pressure readings.
8. Statistical calculations.
9. Test conditions.
10. A copy of the final report.

FINAL REPORT

A final report of the results of the study will be prepared by Wildlife International Ltd. 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, chemical abstract number or code number, purity, composition, empirical formula, molecular formula, manufacturer's lot/batch number, dissociation in water, method of analysis, or other information provided by the Sponsor.
6. Description of the test method or reference to the method used along with any modifications made.

7. Individual and mean values obtained for out-gassing rates and vapor pressure.
8. Description of any problems experienced and how they were resolved.
9. A statement prepared by the Quality Assurance Unit listing the dates that study inspections and audits were made and any findings that were recorded.

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 (ISBN 92-84-12367-9) and EPA (40 CFR Parts 160 and/or 792). 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, 104: *Vapor Pressure Curve.*

2 **Product Properties Test Guidelines** 1996. OPPTS 830.7950. *Vapor Pressure.*

APPENDIX I
IDENTIFICATION OF TEST SUBSTANCE BY SPONSOR

To be Completed by Sponsor

I. Test Substance Identity (name to be used in the report): Perfluorooctane Sulfonic Acid, Potassium Salt (PFOS)

Sample Code or Batch Number: Lot 217

Purity (% Active Ingredient): 98.9% Expiration Date: 2008

II. Test Substance Characterization

Have the identity, strength, purity and composition or other characteristics which appropriately define the test substance been determined prior to the start of this study in accordance with GLP Standards? Yes ___ No X

III. Test Substance Storage Conditions

Please indicate the recommended storage conditions at Wildlife International Ltd.

Ambient room temperature.

Has the stability of the test substance under these storage conditions been determined in accordance with GLP Standards? Yes ___ No X

Other pertinent stability information:

IV. Toxicity Information:

Mammalian: Rat LD50 251 mg/kg Mouse LD50 N/A

Aquatic:	Invertebrate Toxicity (EC/LC50)	Fish Toxicity (LC50)
	Daphnia magna: 27 mg/L	Rainbow trout: 11 mg/L
	Daphnia magna: 50 mg/L	Fathead minnow: 38 mg/L

Other Toxicity Information (including findings of chronic and subchronic tests):

See MSDS.

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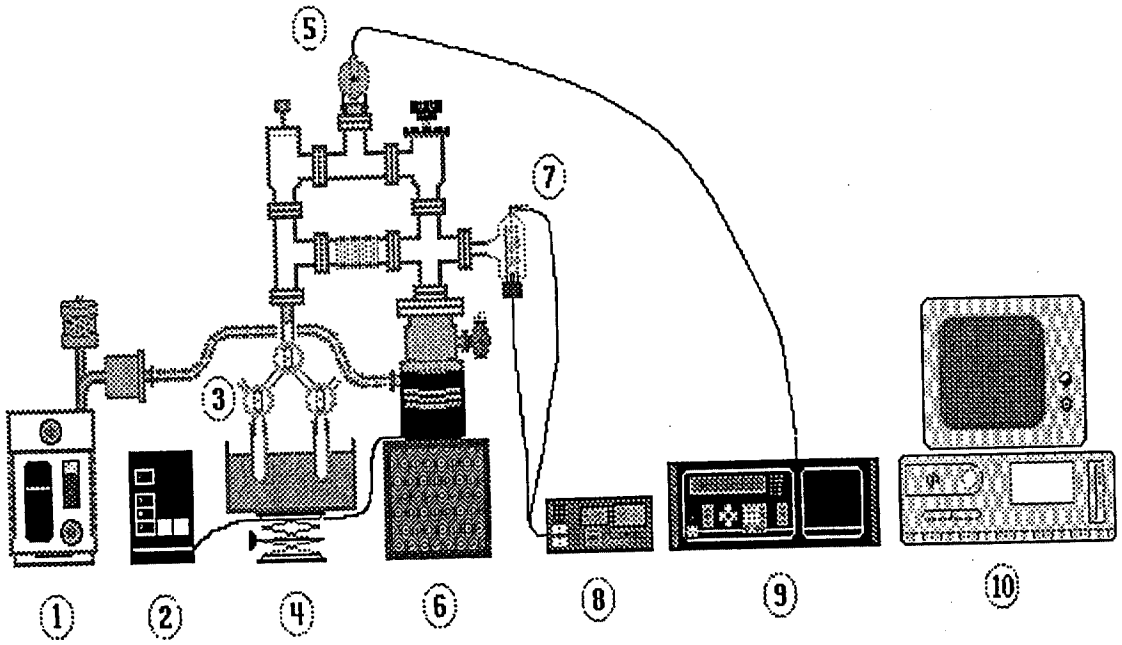
Figure 1. Schematic of Spinning Rotor Gauge Apparatus.



1. vacuum pump
2. turbovac controller
3. sample chambers
4. water bath
5. spinning rotor gauge head
6. turbomolecular pump
7. ion gauge
8. ion gauge controller
9. spinning rotor gauge
10. computer

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