Determination of the Vapor Pressure of PFOS Using the Spinning Rotor Guage Method

VAPOR PRESSURE

TEST SUBSTANCE

Identity: Perfluorooctanesulfonate; may also be referred to as PFOS or FC-95. (1-Octanesulfonic acid, 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-heptadecafluoro-, potassium salt, CAS # 2795-39-3)

Remarks: The test substance is a white powder. Sample was taken from 3M lot number 217. Sample was stored under ambient conditions prior to testing. Purity determined to be 90.49% by LC/MS, ^{1}H-HMR, ^{19}F-NMR and elemental analyses techniques.

METHOD

Method: OECD 104, U.S. EPA OPPTS 830.7950

GLP (Y/N): Yes
Year completed: 1999

Remarks: Determination of the vapour pressure was done by using the Spinning Rotor Gauge method.

RESULTS

Vapor Pressure: $3.31X10^{-4}$ Pa
Temperature °C: 20°C
Decomposition (yes/no/ambiguous): No

Remarks: The measured vapour pressure was repeatable.

CONCLUSIONS

Remarks: The vapor pressure of the test substance was determined to be $3.31X10^{-4}$ Pa at 20°C using the spinning rotor gauge method.

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

DATA QUALITY

Reliability: Klimisch ranking 1.

REFERENCES

Study conducted at the request of 3M Company by Wildlife International, Ltd. of Easton, Maryland.

OTHER

Last changed: 5/3/00

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

WILDLIFE INTERNATIONAL LTD. PROJECT NUMBER: 454C-105

OECD Guideline 104 Vapour Pressure Curve
U.S. EPA OPPTS 830.7950 Vapor Pressure

AUTHORS:

Raymond L. Van Hoven, Ph.D.
Joel I. Stenzel
Willard B. Nixon, Ph.D.

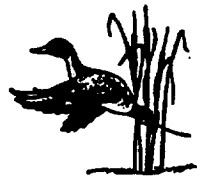
STUDY INITIATION DATE: January 12, 1999

STUDY COMPLETION DATE: May 5, 1999

Submitted to

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

WILDLIFE INTERNATIONAL LTD.



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

GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT

SPONSOR: 3M Corporation

TITLE: Determination of the Vapor Pressure of PFOS Using the Spinning Rotor Gauge Method

WILDLIFE INTERNATIONAL LTD. PROJECT NUMBER: 454C-105

STUDY COMPLETION: May 5, 1999

This study was conducted in compliance with Good Laboratory Practice Standards as published by the U.S. Environmental Protection Agency in 40 CFR Part 792, 17 August 1989; OECD Principles of Good Laboratory Practice, OCDE/GD (92) 32, Environment Monograph No. 45, Paris 1992; and Japan MAFF, 59 NohSan, Notification No. 3850, Agricultural Production Bureau, 10 August 1984, with the following exceptions:

The test and reference substances were not characterized in compliance with Good Laboratory Practices Standards.

The stability of the test substance and reference standards under the conditions at the test site was not tested in compliance with Good Laboratory Practice Standards.

STUDY DIRECTOR:

Raymond E. Van Hoven, Ph.D.
Scientist

5-5-99
DATE

SPONSOR APPROVAL:

Susan A. Beach
Sponsor

5/10/99
DATE

QUALITY ASSURANCE STATEMENT

SPONSOR: 3M Corporation

TITLE: Determination of the Vapor Pressure of PFOS Using the Spinning Rotor Gauge Method

WILDLIFE INTERNATIONAL LTD. PROJECT NUMBER: 454C-105

This study was examined for compliance with Good Laboratory Practice Standards as published by the U.S. Environmental Protection Agency in 40 CFR Part 792, 17 August 1989; OECD Principles of Good Laboratory Practice, OCDE/GD (92) 32, Environment Monograph No. 45, Paris 1992; and Japan MAFF, 59 NohSan, Notification No. 3850, Agricultural Production Bureau, 10 August 1984. 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:
Test Substance Preparation and Test Initiation	January 27, 1999	January 27, 1999	January 27, 1999
Measurement One	January 29, 1999	January 29, 1999	January 29, 1999
Draft Report and Data	February 24, 1999	February 24, 1999	February 25, 1999
Final Report	May 5, 1999	May 5, 1999	May 5, 1999

Lisa T. Drottar
Quality Assurance Representative

5599
DATE

REPORT APPROVAL

SPONSOR: 3M Corporation

TITLE: Determination of the Vapor Pressure of PFOS Using the Spinning Rotor Gauge Method

WILDLIFE INTERNATIONAL LTD. PROJECT NUMBER: 454C-105

STUDY DIRECTOR:

Raymond L. Van Hoven, Ph.D.
Scientist

5-5-99
DATE

MANAGEMENT:

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

5/6/99
DATE

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SUMMARY

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

WILDLIFE INTERNATIONAL LTD. PROJECT NUMBER:	454C-105
TEST SUBSTANCE:	PFOS (Perfluorooctane Sulfonic Acid, Potassium Salt)
STUDY:	Determination of the Vapor Pressure of PFOS Using the Spinning Rotor Gauge Method
TEST DATES:	Experimental Start - January 27, 1999 Experimental Termination - February 12, 1999

SUMMARY:	The vapor pressure of PFOS was determined to be 3.31×10^{-4} Pa at 20°C using the spinning rotor gauge method.

INTRODUCTION

This study was conducted by Wildlife International Ltd. for 3M Corporation at the Wildlife International Ltd. analytical chemistry facility in Easton, Maryland. Tests were performed using the spinning rotor gauge method. The vapor pressure of the test substance was analyzed from January 27 to February 12, 1999. Raw data generated by Wildlife International Ltd. and a copy of the final report are filed under Project Number 454C-105 in archives located on the Wildlife International Ltd. site.

OBJECTIVE

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

EXPERIMENTAL DESIGN

The spinning rotor gauge (SRG) system was used to determine vapor pressure. The SRG measured the rotational frequency of a stainless steel ball that was magnetically suspended within a vacuum chamber. The deceleration rate of the ball was used to determine the gas pressure. The system was configured with two sample chambers. One of the sample chambers contained a sample of the test substance, while the other remained empty to make control measurements. The system steady-state pressure and pressure increase of the test substance and control samples were measured three times.

MATERIALS AND METHODS

This study was conducted based on procedures outlined in OECD Guidelines, Method 104 (1); and EPA OPPTS 830.7950 (2). The spinning rotor gauge method was used to determine the vapor pressure of the test substance.

Test Substance

The test substance was received from 3M Corporation on October 29, 1998, and was assigned Wildlife International Ltd. identification number 4675 upon receipt. The test substance, a white powder, was identified

as: FC-95, Lot 217, 3M ID#: 98-0211-3916-1. Based on information supplied by the Sponsor, the test substance had a purity of 98.9% and an average molecular weight of 538 AMU. The test substance was stored under ambient conditions.

Reference Substances

The hexachlorobenzene reference substance was received from Aldrich Chemical Company on February 10, 1997, and was assigned Wildlife International Ltd. identification number 3948 upon receipt. The reference substance, a white powder, was identified as: Hexachlorobenzene; catalog no. 17105-0, CAS Number 118-74-1. The reference substance had a reported purity of 99%, and was stored under ambient conditions.

The DDT reference substance was received from Aldrich Chemical Company on January 20, 1999, and was assigned Wildlife International Ltd. identification number 4749 upon receipt. The reference substance, a white powder, was identified as: 1,1-bis(p-Chlorophenyl)-2,2,2-Trichloroethane; lot number 77 H3614; catalog no. C8894, CAS Number 50-29-3. The reference substance had a reported purity of 99.3%, and was stored in a refrigerator.

Apparatus Configuration

The high vacuum system consisted of a vacuum pump (Leybold TRIVAC S 1,6 B), a turbomolecular pump (Leybold TURBOVAC 50), an ion gauge and controller (Metra 731 Digital Ion Gauge Controller), a spinning rotor gauge (Leybold VISCOVAC VM 212), a sample introduction system, and various stainless-steel con-flat fittings, including three valves. The con-flat fittings were assembled using copper gaskets. The valves were used to isolate the sample chambers, the spinning rotor gauge and/or the vacuum pumps. The spinning rotor gauge controller was connected to a computer via the serial interface (RS-232 port). A diagram of the apparatus configuration is presented in Figure 1.

The sample introduction system was custom designed and manufactured by At-Mar Glass Co. (Kennett Square, PA). The system consisted of two high vacuum glass sample tubes. During the study, one tube contained a sample of the test or reference substance while the other tube remained empty (control). The temperature of the samples were maintained by immersing the sample tubes in a water bath. The temperature of the water bath was periodically monitored using an ASTM 44C mercury thermometer. The tubes were independently vented and connected to the vacuum system using three ground-glass stopcocks. A glass to metal transition flange

connected the sample introduction system to the vacuum system. A diagram of the sample introduction system is presented in Figure 2.

A steel ball was inserted into the spinning rotor gauge tube before the tube was connected to the high vacuum system. The con-flat fittings and valves of the high vacuum system were wrapped in heat tape. The temperature of the heat tape was controlled using a rheostat. The surface temperature of the vacuum system was periodically monitored using an Omega Model HH23 digital thermometer (Omega Engineering, Inc., Stamford, CT) with a thermocouple placed between the heat tape and the outer surface of the fittings. The air temperature also was periodically monitored using the digital thermometer.

Procedures

The high vacuum system was heated to approximately 80°C and evacuated for at least 18 hours prior to making any measurements. After heating, the system was allowed to cool until reaching room temperature. The steel ball inside the spinning rotor gauge tube was suspended in a magnetic field and driven to a rotational frequency of approximately 415 Hz. The deceleration rate of the ball was monitored using the spinning rotor gauge. The parameters of the spinning rotor gauge were set to record a deceleration measurement every 30 seconds based on the molecular weight (28.96 g/mole) and viscosity (18.2×10^{-6} Pa·s) of air. The offset value was determined for each sample tube when the deceleration rate reached a plateau. The offset value (mean deceleration rate) was entered into the spinning rotor gauge controller.

A sample of hexachlorobenzene was placed in one of the sample chambers, while the other chamber remained empty (control). The parameters of the spinning rotor gauge were changed to record pressure, in Pascal (Pa), every 30 seconds. This setting was used for all subsequent measurements. The molecular weight parameter was set to 284.78 and viscosity was set to zero. The sample chamber was evacuated to achieve a steady-state pressure. Temperatures were periodically recorded. After the system reached temperature and pressure equilibrium, the steady-state pressure of the sample was measured with the spinning rotor gauge. The valves to the vacuum pumps were then closed, and the pressure increase was monitored for at least two hours.

A sample of DDT was placed in one of the sample chambers, while the other chamber remained empty (control). The molecular weight parameter was set to 354.5 and viscosity was set to zero. The sample chamber was evacuated to achieve a steady-state pressure. Temperatures were periodically recorded. After the system

reached temperature and pressure equilibrium, the steady-state pressure of the sample was measured with the spinning rotor gauge. The valves to the vacuum pumps were then closed, and the pressure increase was monitored for at least two hours.

A small sample of the test substance was placed in one of the sample chambers, while the other chamber remained empty (control). The molecular weight parameter was set to 538, and viscosity was set to zero. The sample chamber was opened to the vacuum system and pumped-down to a steady-state pressure. Temperatures were periodically recorded. After the system reached temperature and pressure equilibrium, the steady-state pressure of the sample was measured with the spinning rotor gauge. The valves to the vacuum pumps were then closed, and the pressure increase was monitored for at least at least two hours. Measurements then alternated between the PFOS sample and the control sample (empty sample chamber) for a total of three determinations of each.

Calculations

The steady-state pressures were determined by averaging the pressure values recorded prior to closing the valves to the vacuum pumps during each measurement. Standard deviations and 95% confidence intervals were also calculated.

The pressure increase data were analyzed using least-squares-fit linear regression analysis. A line was fit to the portion of the data that was determined to have achieved equilibrium pressure with the sample. The slope and intercept were calculated for each measurement. Correlation coefficients and 95% confidence intervals for each parameter were also calculated. The out-gassing rate was determined from the slope of the line and the vapor pressure was determined from the intercept value.

The means and standard deviations of the replicate measurements of the control and PFOS sample were also calculated.

RESULTS AND DISCUSSION

A graphical presentation of the offset measurement is shown in Figure 3. The graphs show data collected for approximately 2 hours from each sample tube. The mean offset value was based on the average deceleration rate measured from left (7.877×10^{-8} s^{-1}) and right (6.838×10^{-8} s^{-1}) sample chambers on January 6, 1999. The offset value was determined to be 7.357×10^{-8} s^{-1}.

The steady-state pressure and pressure increase of hexchlorobenzene were determined on January 14, 1999, and are presented in Figure 4. The steady-state pressure was 1.24×10^{-5} Pa and the vapor pressure was determined to be 8.30×10^{-4} Pa. The vapor pressure was consistent with the range found in the literature (2).

The steady-state pressure and pressure increase of DDT were determined on January 22, 1999, and are presented in Figure 5. The steady-state pressure was 4.81×10^{-6} Pa and the vapor pressure was determined to be 1.76×10^{-4} Pa. The vapor pressure was consistent with the range found in the literature (3).

The steady-state pressure and pressure increase of PFOS were determined on February 3, 5 and 12, 1999. The mean steady-state pressure was 4.51×10^{-6} Pa (Table 1). For each associated pressure increase measurement, the slope of the pressure increase data became constant after approximately ninety minutes. This indicated the system had achieved saturation of the gas from the PFOS sample. A line was fit to this region of the data, and the slope and intercept were determined. The intercept values represented the vapor pressure of the sample. The mean vapor pressure of PFOS was 3.31×10^{-4} Pa (Table 2). A representative graphical presentation of the data is shown in Figure 6.

The steady-state pressure and pressure increase of the control sample were determined on February 2, 4, and 8, 1999. The mean baseline pressure was -1.22×10^{-6} Pa (Table 3). For each measurement, the slope of the pressure increase data became constant after approximately thirty minutes, indicating the system had achieved equilibrium. A line was fit to this region of the data, and the slope and intercept were determined. The slope values represented the out-gassing rate of the system. The mean out-gassing rate was 4.78×10^{-8} Pa/sec. (Table 4). The intercept values represented the background pressure of the system. The mean background pressure was 1.51×10^{-5} Pa. The background pressure was considered insignificant compared to the vapor

pressure of PFOS and was not subtracted from those values. A representative graphical presentation of the data is shown in Figure 7.

Occasionally during the study, a leak developed at one of the ground glass joints of the sample introduction system. This was corrected by adjusting the stopcocks and/or repositioning the sample tubes. The spinning rotor gauge displayed either "MTIME TOO LONG" or "BAD SIGNAL" messages during the study, but was restarted without any difficulties each time.

The mean temperature of the water bath, during measurements of the PFOS sample, was 20.1°C and ranged from 19.50 to 20.50°C. The air temperature ranged from 19.0 to 22.3°C, and the surface temperature of the vacuum system ranged from 19.5 to 22.9°C.

CONCLUSION

Based on the results from three sets of measurements collected from the spinning rotor gauge, the vapor pressure of PFOS was determined to be 3.31×10^{-4} Pa at 20°C.

REFERENCES

1 **Organisation for Economic Cooperation and Development.** 1995. Guideline for Testing of Chemicals, Method 104, "Vapour Pressure Curve".

2 **U.S. Environmental Protection Agency.** 1996. OPPTS 830.7950, Vapor Pressure. Washington, D.C.

3 **Tinsley, I.J.** 1979. Chemical Concepts in Pollution Behavior. Wiley Interscience, New York.

Table 1

Steady-State Pressure of PFOS Sample

Trial Number	Date	Steady-State Pressure (Pascal)	Standard Deviation	95% Confidence Interval
1	2/3/99	7.1440 E-6	5.8716 E-7	±6.1426 E-8
2	2/5/99	5.6664 E-6	4.8038 E-7	±6.1158 E-8
3	2/12/99	7.2968 E-7	5.2793 E-7	±6.0657 E-8
	Mean =	4.513 E-6		
	Std.Dev. =	3.359 E-6		

Table 2

Pressure Increase of PFOS Sample

Trial Number	Slope (Pa/sec.)	95% Confidence Interval	Intercept (Pascal)	95% Confidence Interval	Correlation Coefficient
1	3.5926 E-7	3.5808 to 3.6044 E-7	3.2796 E-4	3.1615 to 3.3977 E-4	0.99904
2	2.3696 E-7	2.3607 to 2.3785 E-7	4.6090 E-4	4.5107 to 4.7072 E-4	0.99875
3	1.1970 E-7	1.1949 to 1.1992 E-7	2.0452 E-4	2.0190 to 2.0715 E-4	0.99978
Means	2.386 E-7		3.311 E-4		
Std.Dev.	1.198 E-7		1.282 E-4		

Table 3

Steady-State Pressure of Control Sample

Trial Number	Date	Baseline Pressure (pascal)	Standard Deviation	95% Confidence Interval
1	2/2/99	-3.4529 E-6	5.2286 E-7	±7.3013 E-8
2	2/4/99	3.1513 E-7	4.0515 E-7	±4.1621 E-8
3	2/8/99	-5.2957 E-7	1.8258 E-7	±2.2633 E-8
	Mean =	-1.222 E-6		
	Std.Dev. =	1.977 E-6		

Table 4

Pressure Increase of Control Sample

Trial Number	Slope (Pa/sec.)	95% Confidence Interval	Intercept (Pascal)	95% Confidence Interval	Correlation Coefficient
1	5.2256 E-8	5.217 to 5.234 E-8	1.9635 E-5	1.917 to 2.010 E-5	0.99990
2	5.1886 E-8	5.186 to 5.191 E-8	1.7799 E-5	1.762 to 1.798 E-5	0.99998
3	3.9377 E-8	3.936 to 3.939 E-8	7.8875 E-6	7.781 to 7.994 E-6	0.99999
Means	4.7840 E-8		1.5107 E-5		
Std.Dev.	0.7331 E-8		0.6319 E-5		

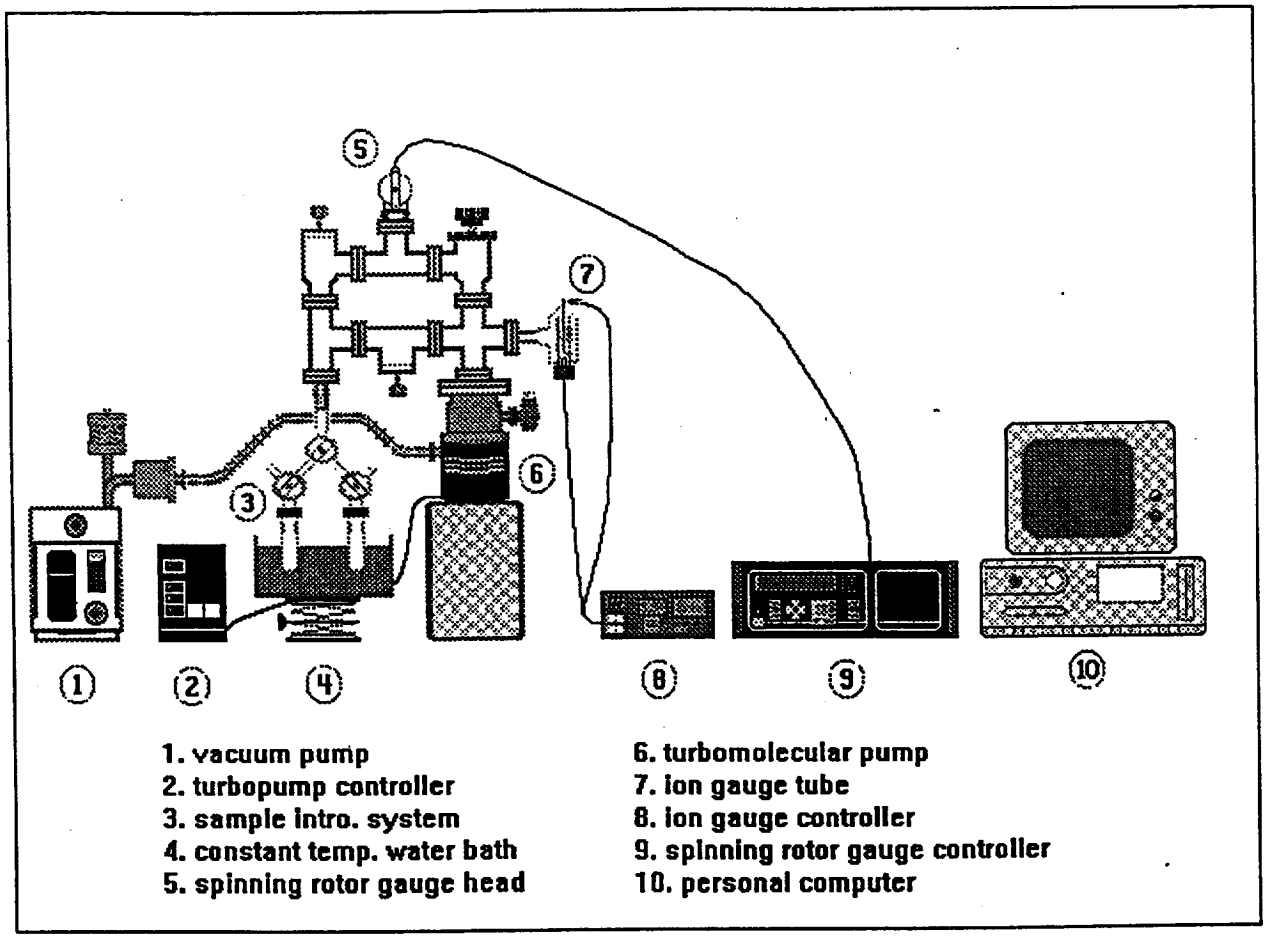


Figure 1. Diagram of high vacuum system configuration.

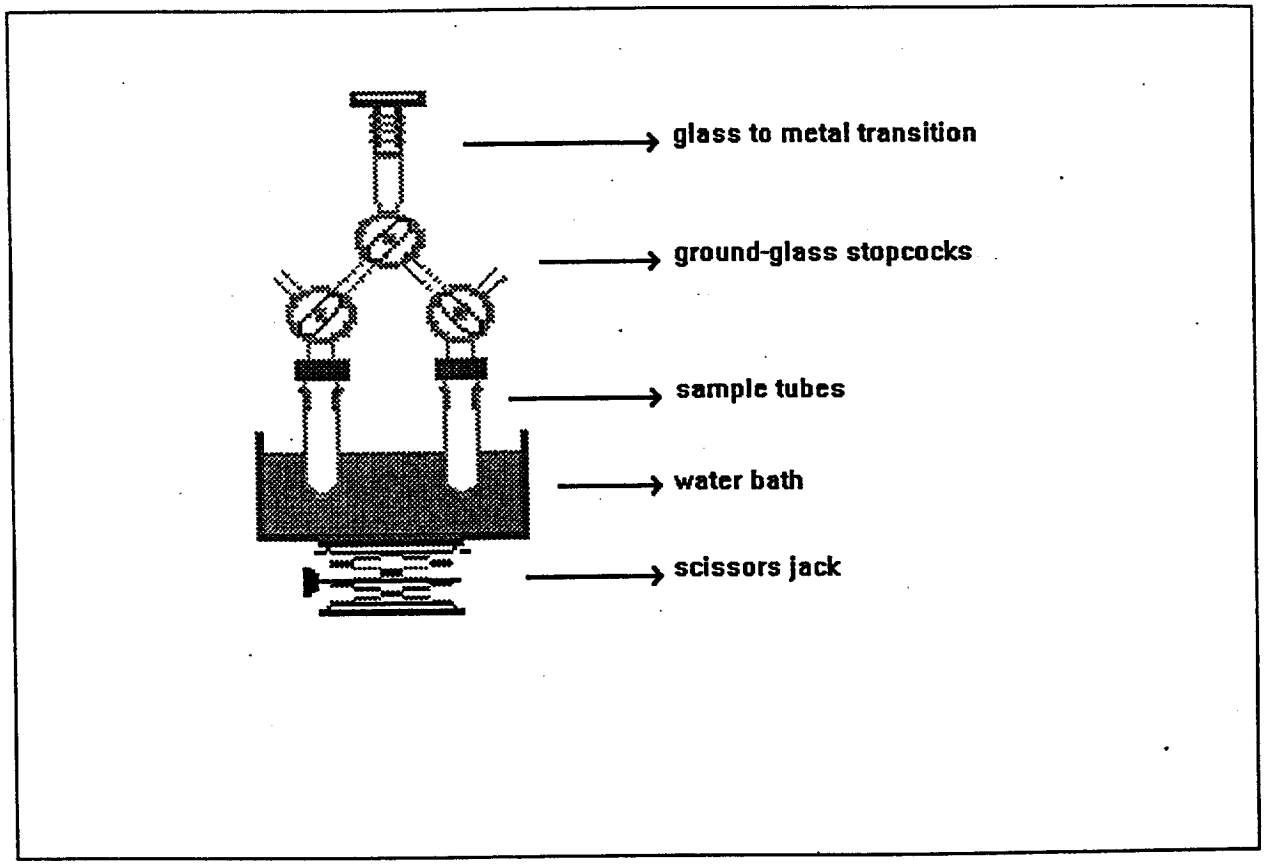


Figure 2. Diagram of sample introduction system.

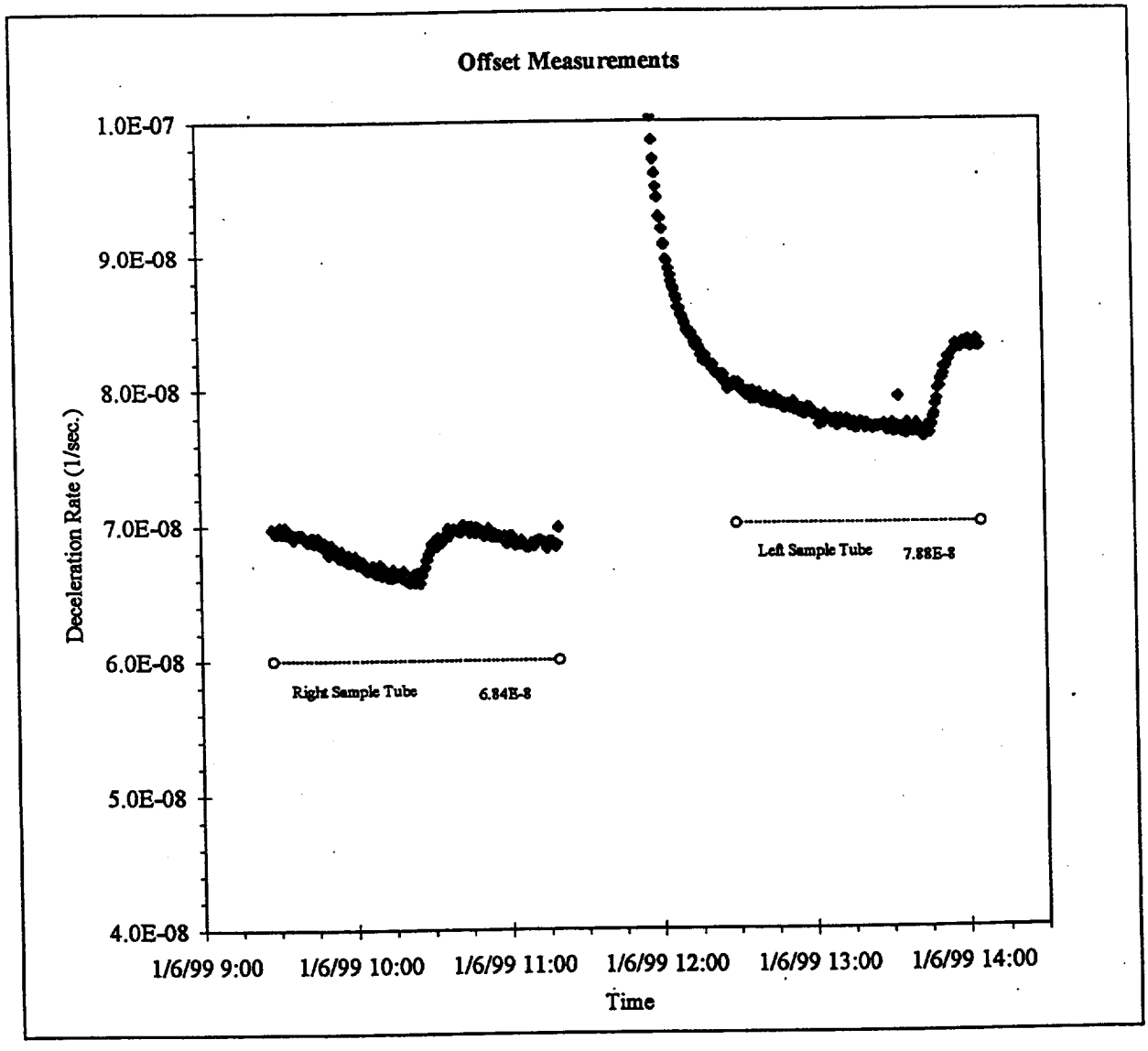


Figure 3. Graphical presentation of offset measurement.

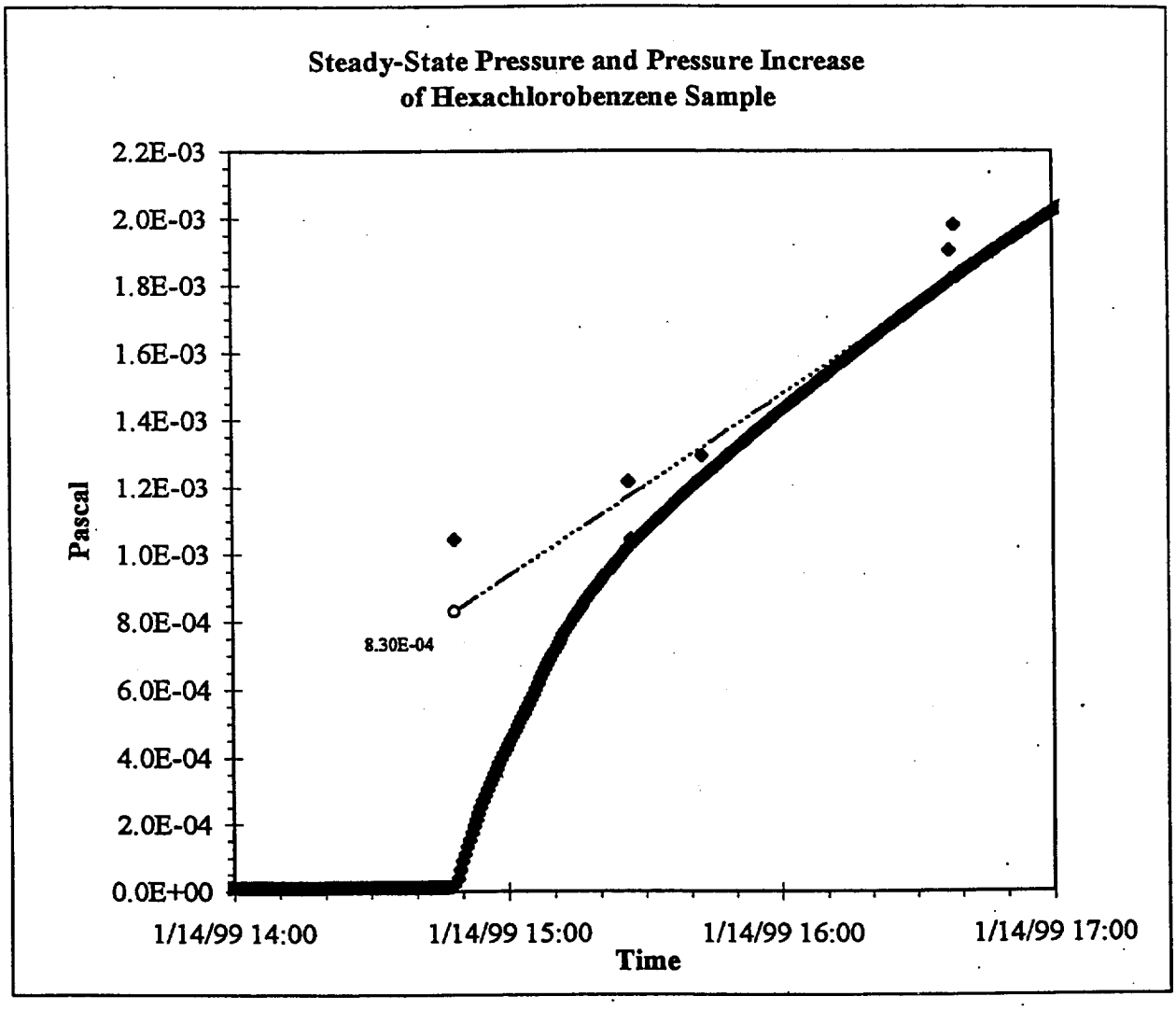


Figure 4. Steady-state pressure and pressure increase of Hexachlorobenzene sample.

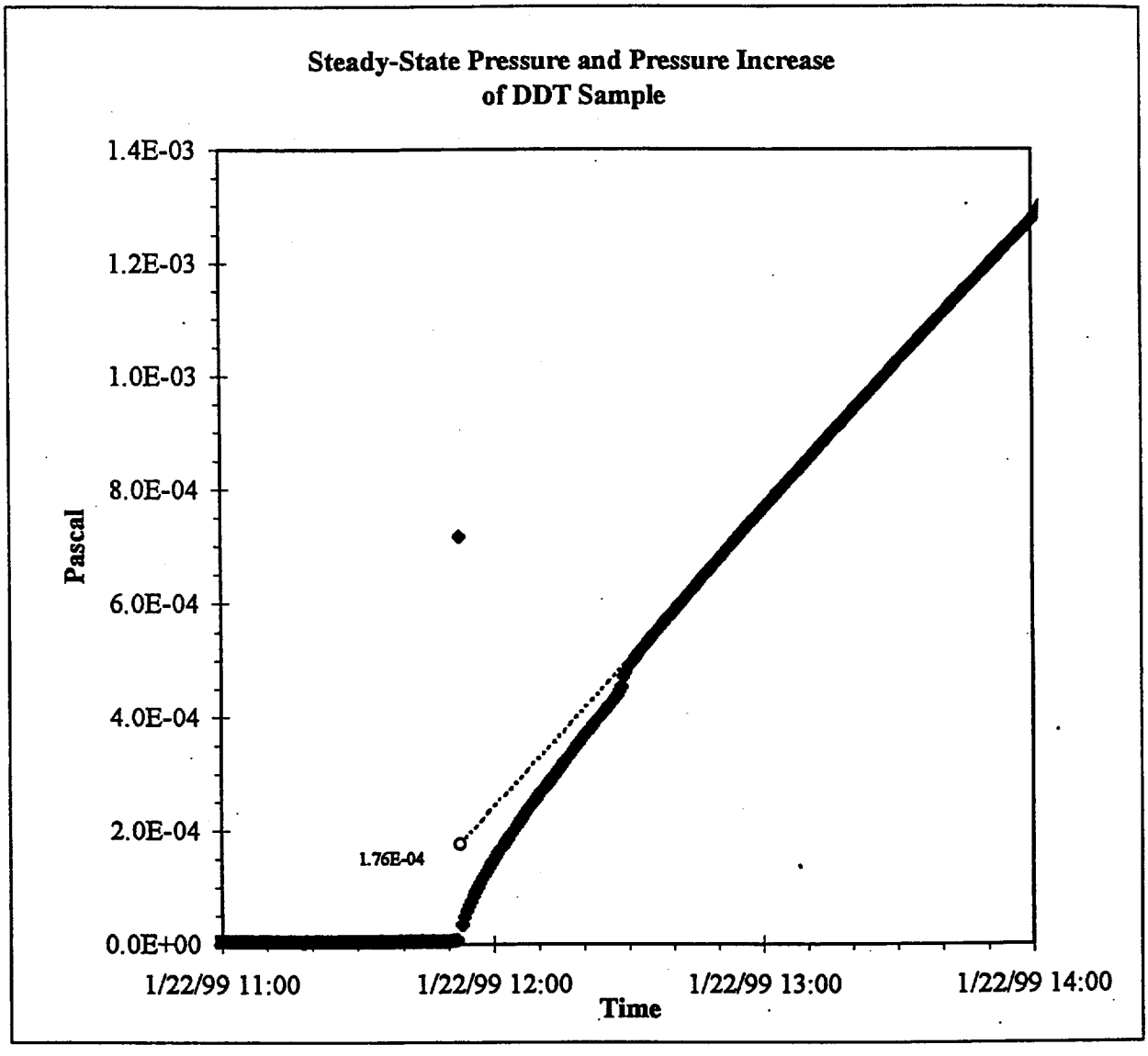


Figure 5. Steady-state pressure and pressure increase of DDT sample.

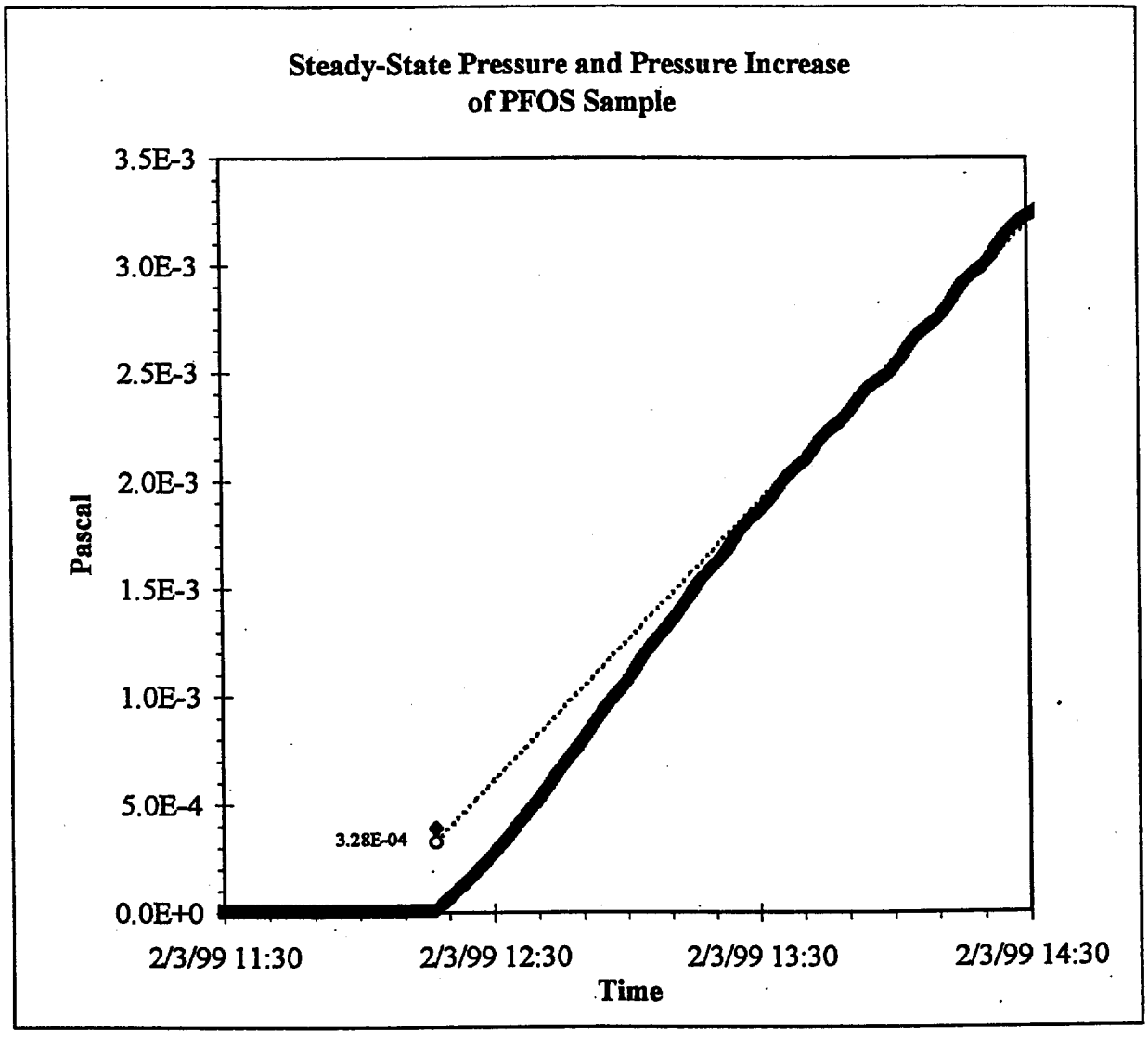


Figure 6. Steady-state pressure and pressure increase of PFOS sample.

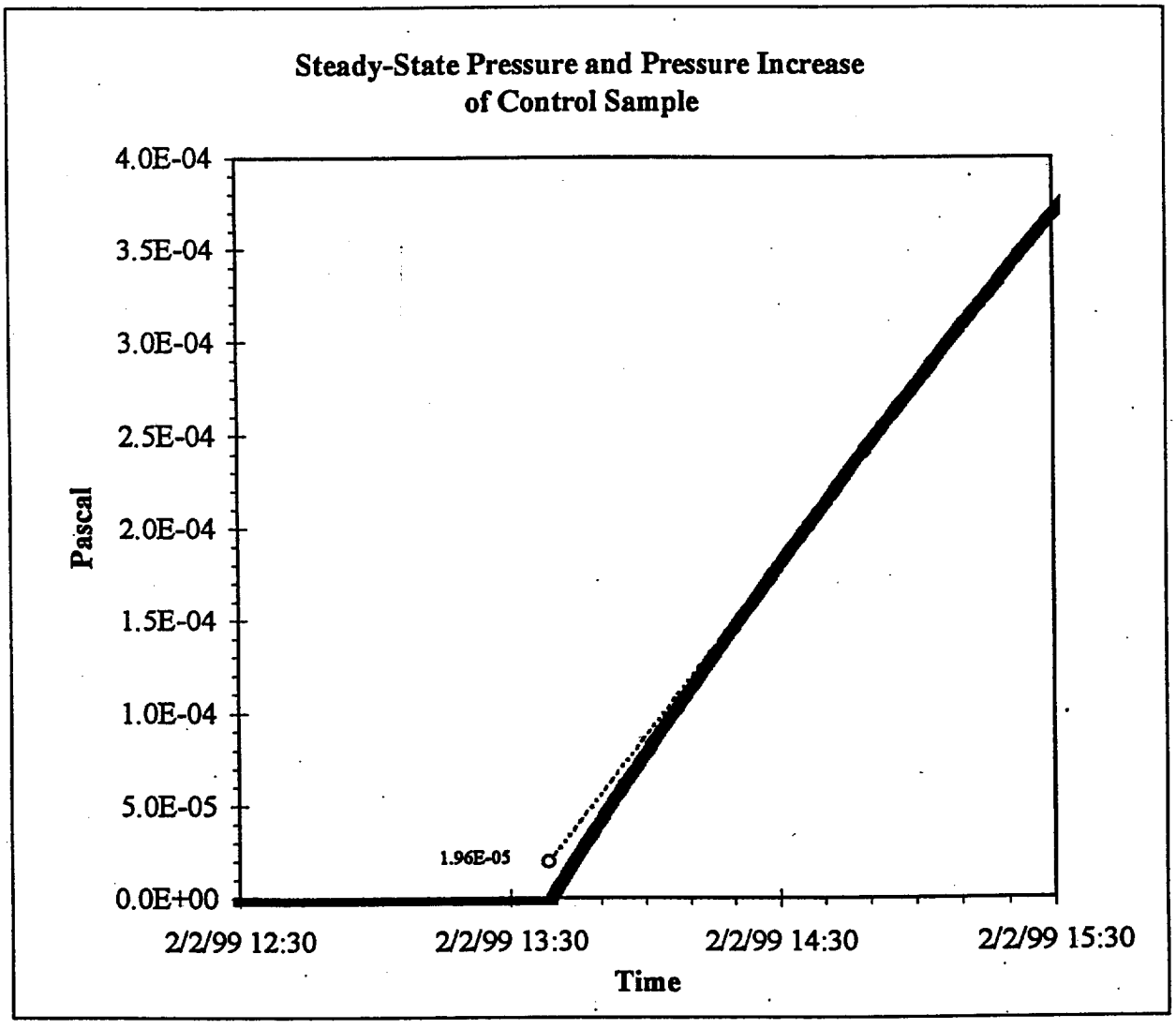


Figure 7. Steady-state pressure and pressure increase of Control sample.

APPENDIX I

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. Joel I. Stenzel, B.S., Systems Analyst
4. Keri Leach, B.S., Laboratory Technician

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

3M Corporation
Environmental Laboratory
P.O. Box 33331
St. Paul, Minnesota 55133

WILDLIFE INTERNATIONAL LTD.

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

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.: 3675A Reference Substance No. (if applicable): NA

PROTOCOL APPROVAL

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

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

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

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

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:

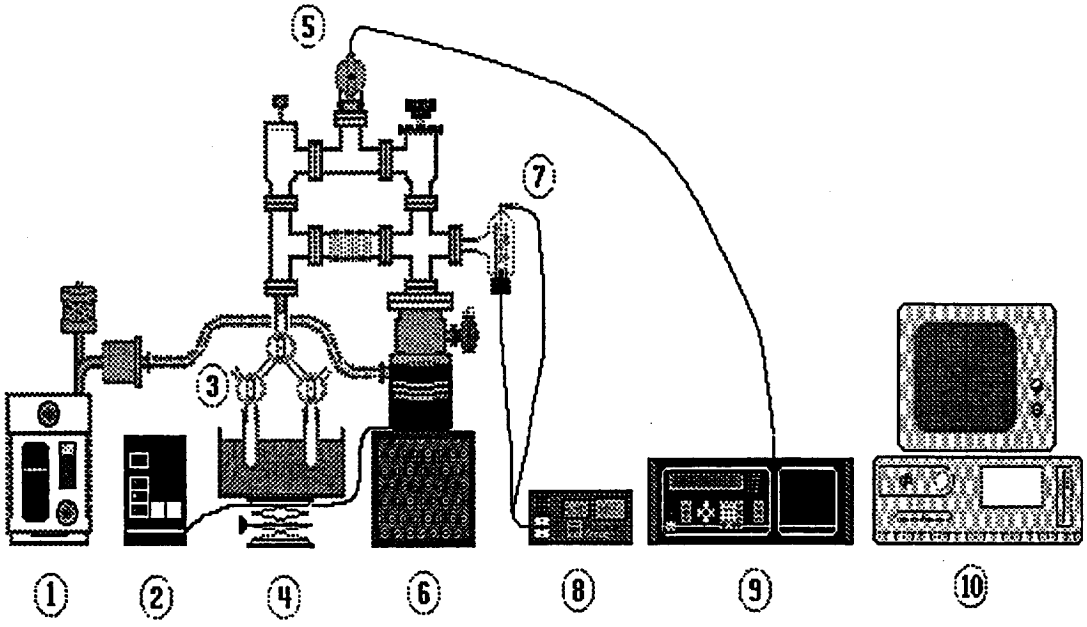
Mammalian: Rat LD50 251 mg/kg Mouse LD50 N/A

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.

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