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.

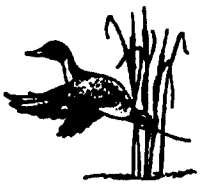
STUDY INITIATION DATE: January 12, 1999

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

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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 L. Van Hoven, Ph.D.
Scientist

5-5-99
DATE

SPONSOR APPROVAL:

Sponsor

5/10/99
DATE

QUALITY ASSURANCE STATEMENT

SPONSOR: 3M Corporation

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		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

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STUDY DIRECTOR:

Raymond L. Van Hoven, Ph.D.
Scientist

5-5-99
DATE

MANAGEMENT:

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

5/5/99
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		

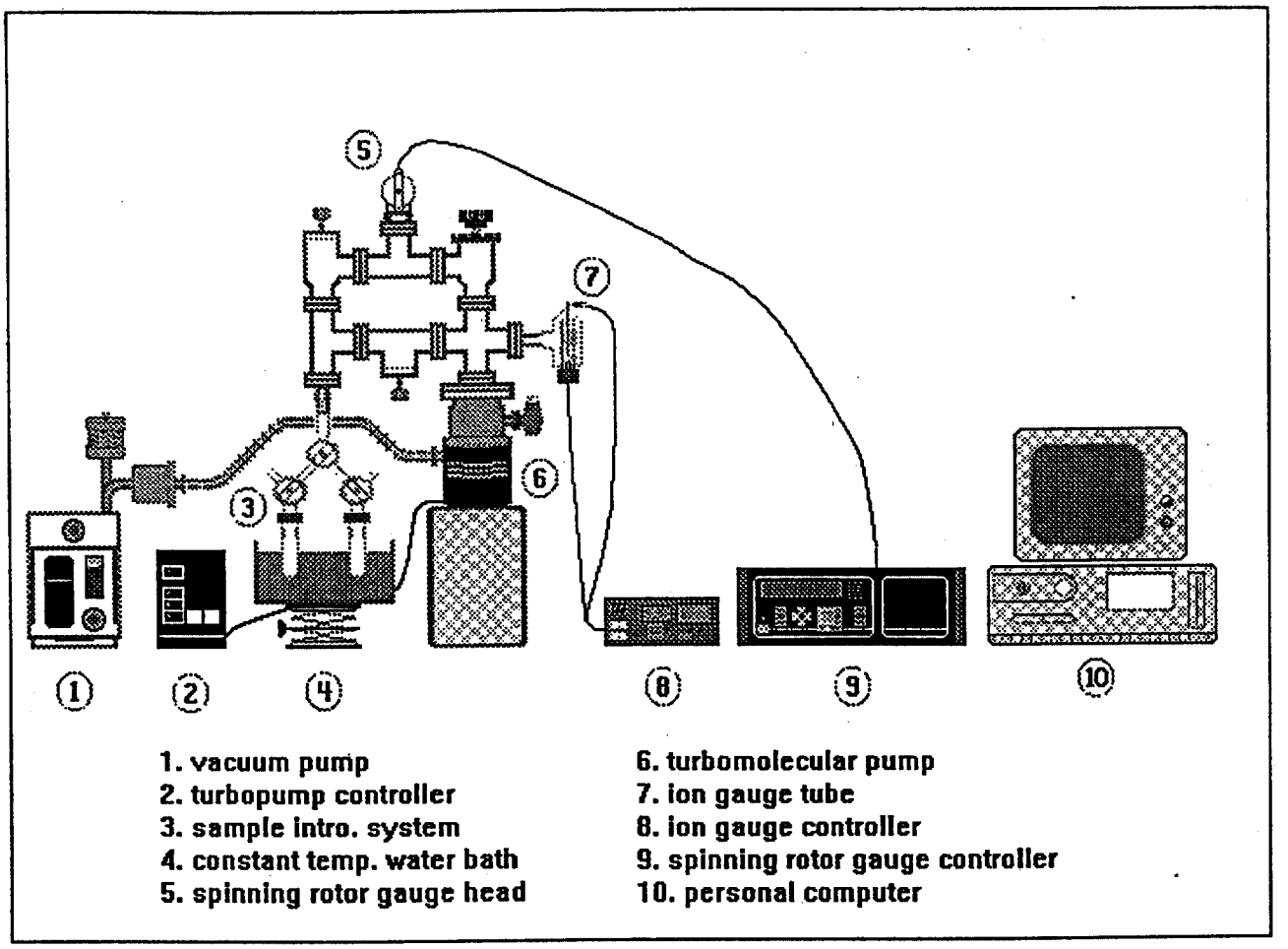

Figure 1. Diagram of high vacuum system configuration.

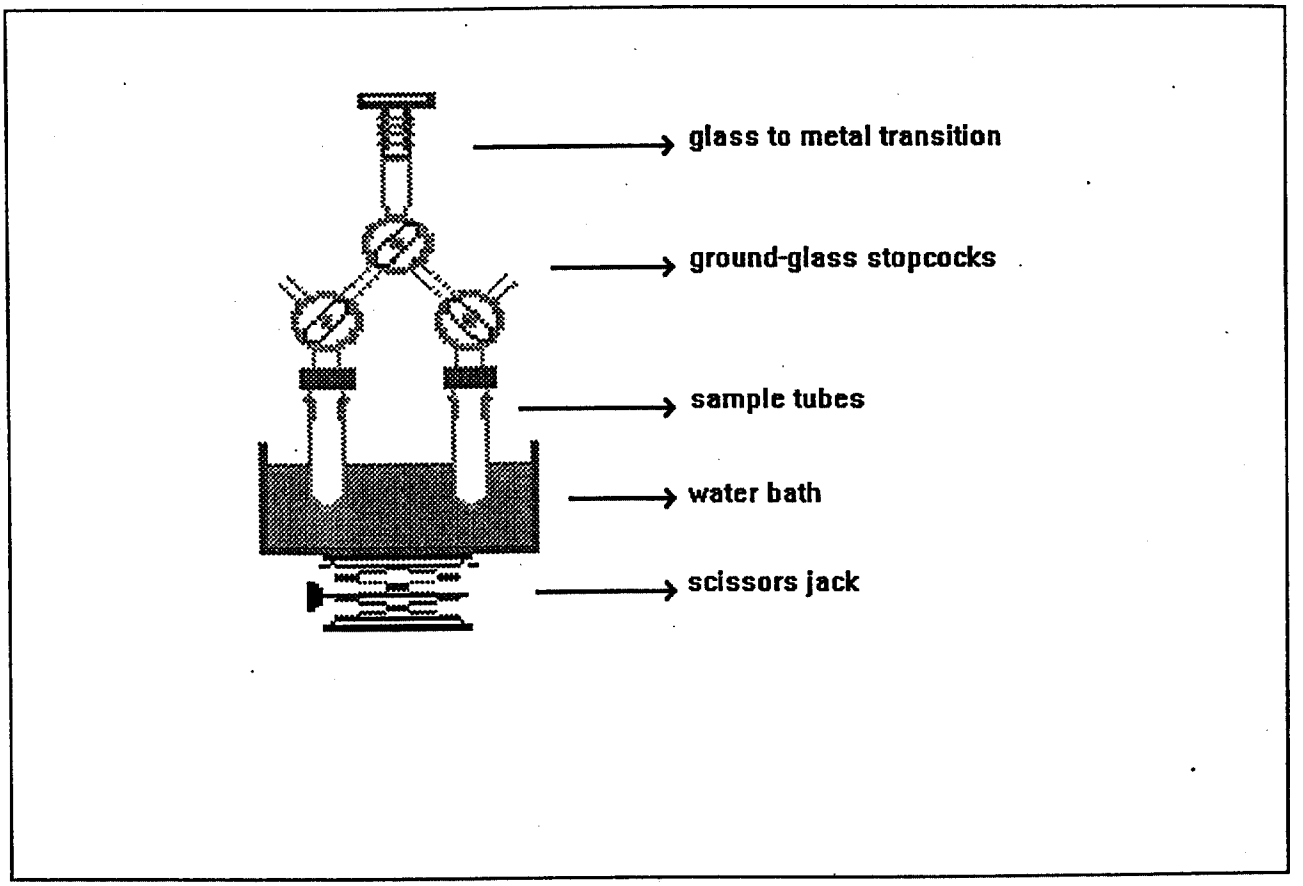

Figure 2. Diagram of sample introduction system.

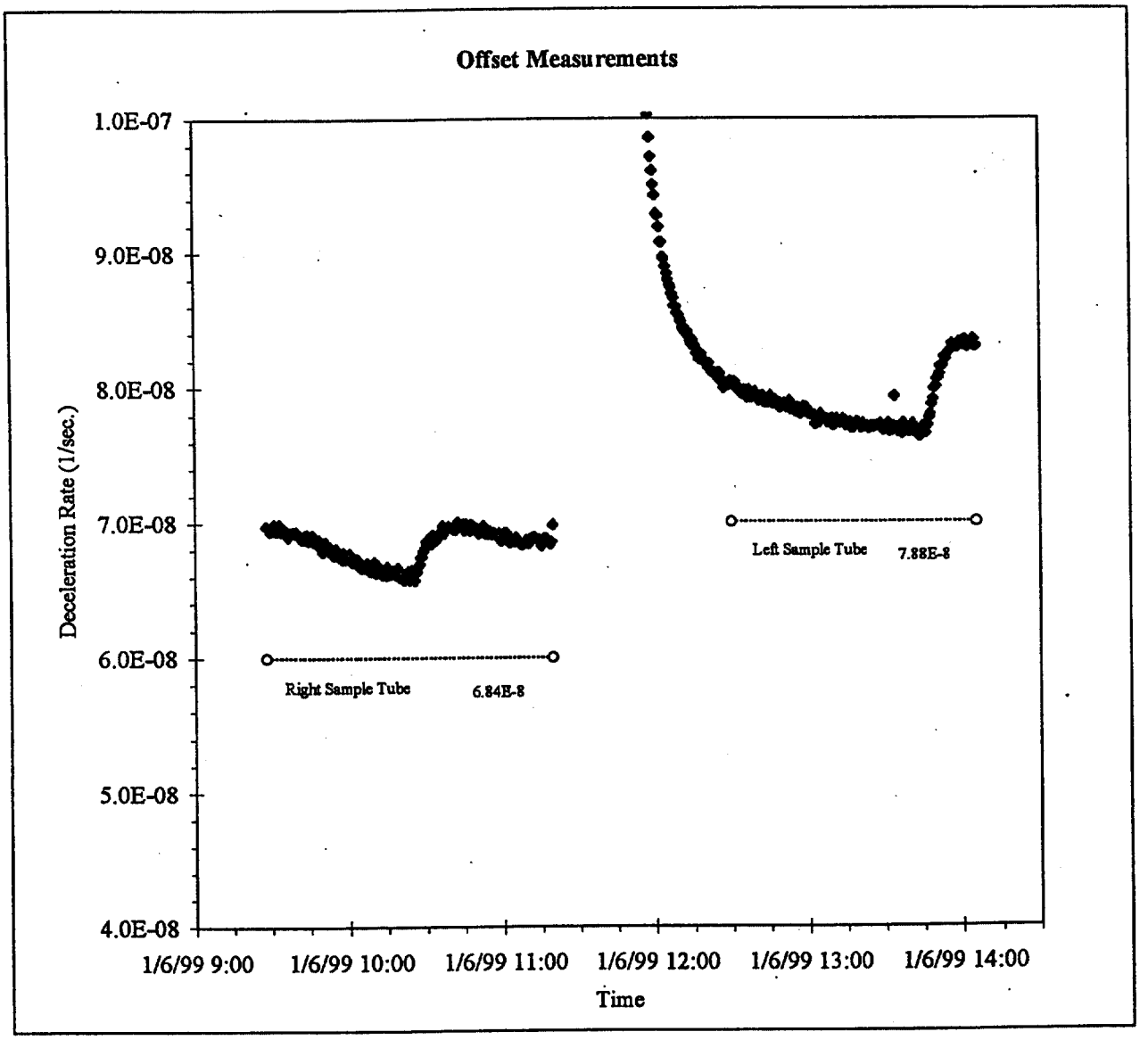

Figure 3. Graphical presentation of offset measurement.

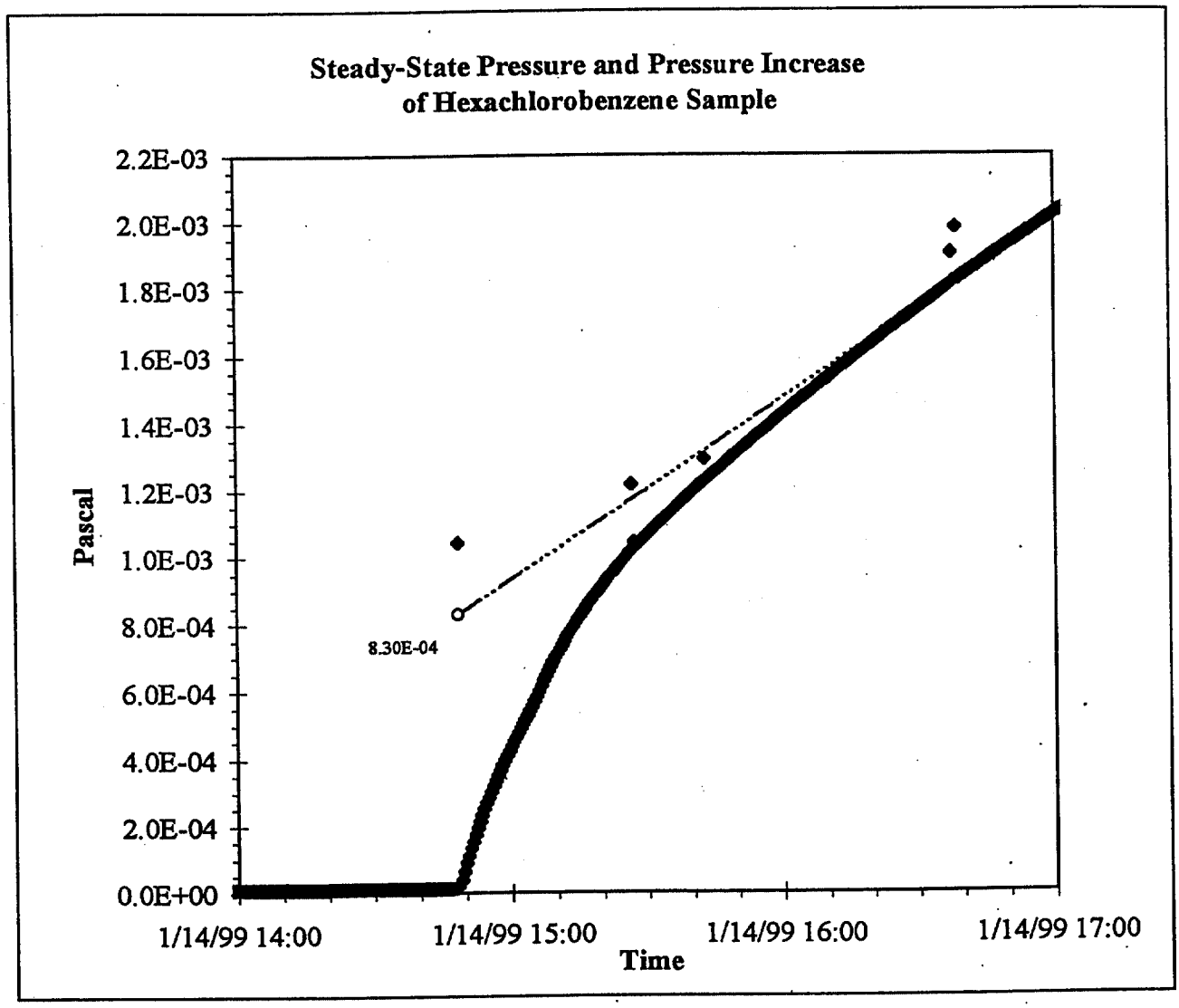

Figure 4. Steady-state pressure and pressure increase of Hexachlorobenzene sample.

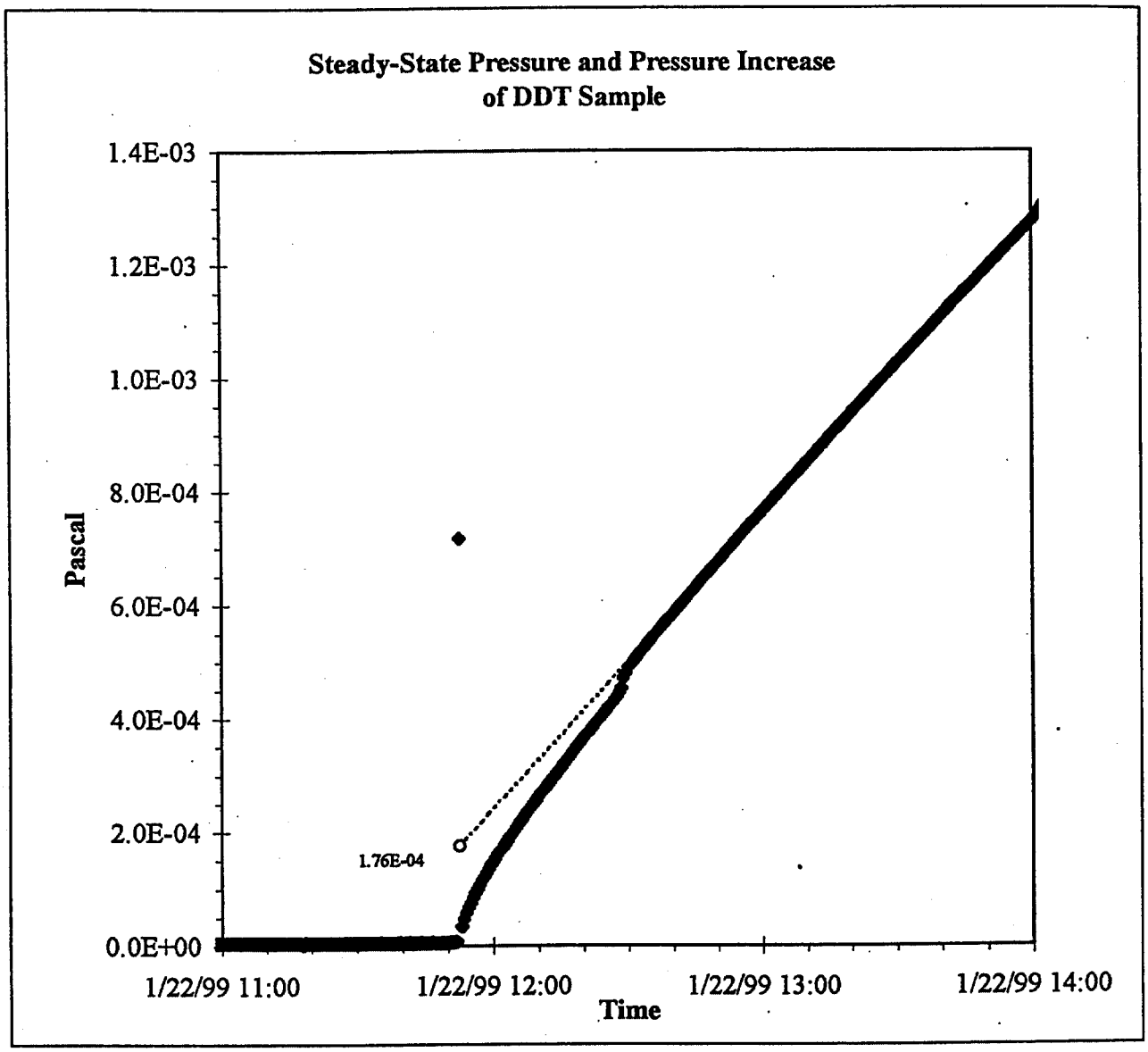

Figure 5. Steady-state pressure and pressure increase of DDT sample.

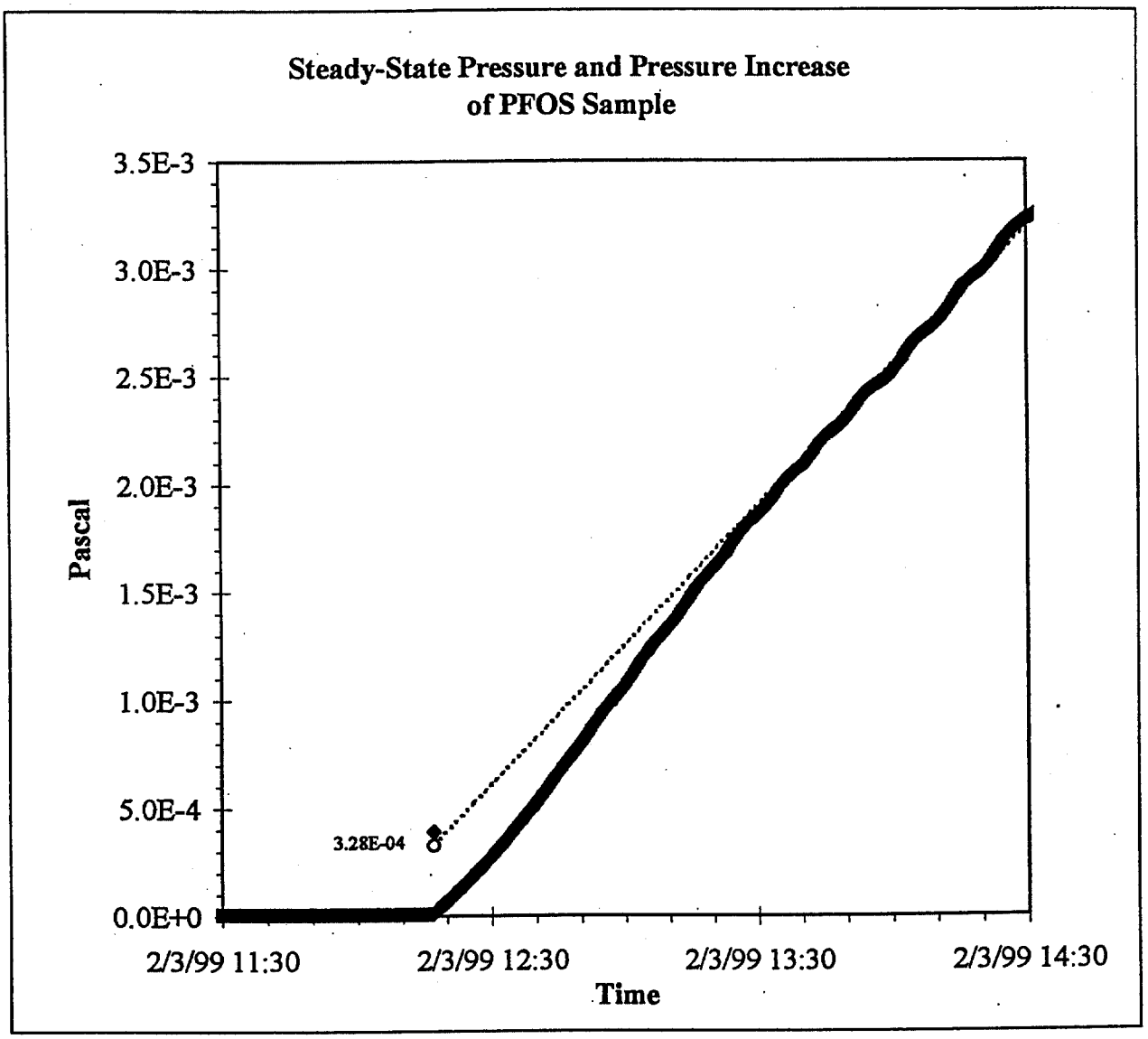

Figure 6. Steady-state pressure and pressure increase of PFOS sample.

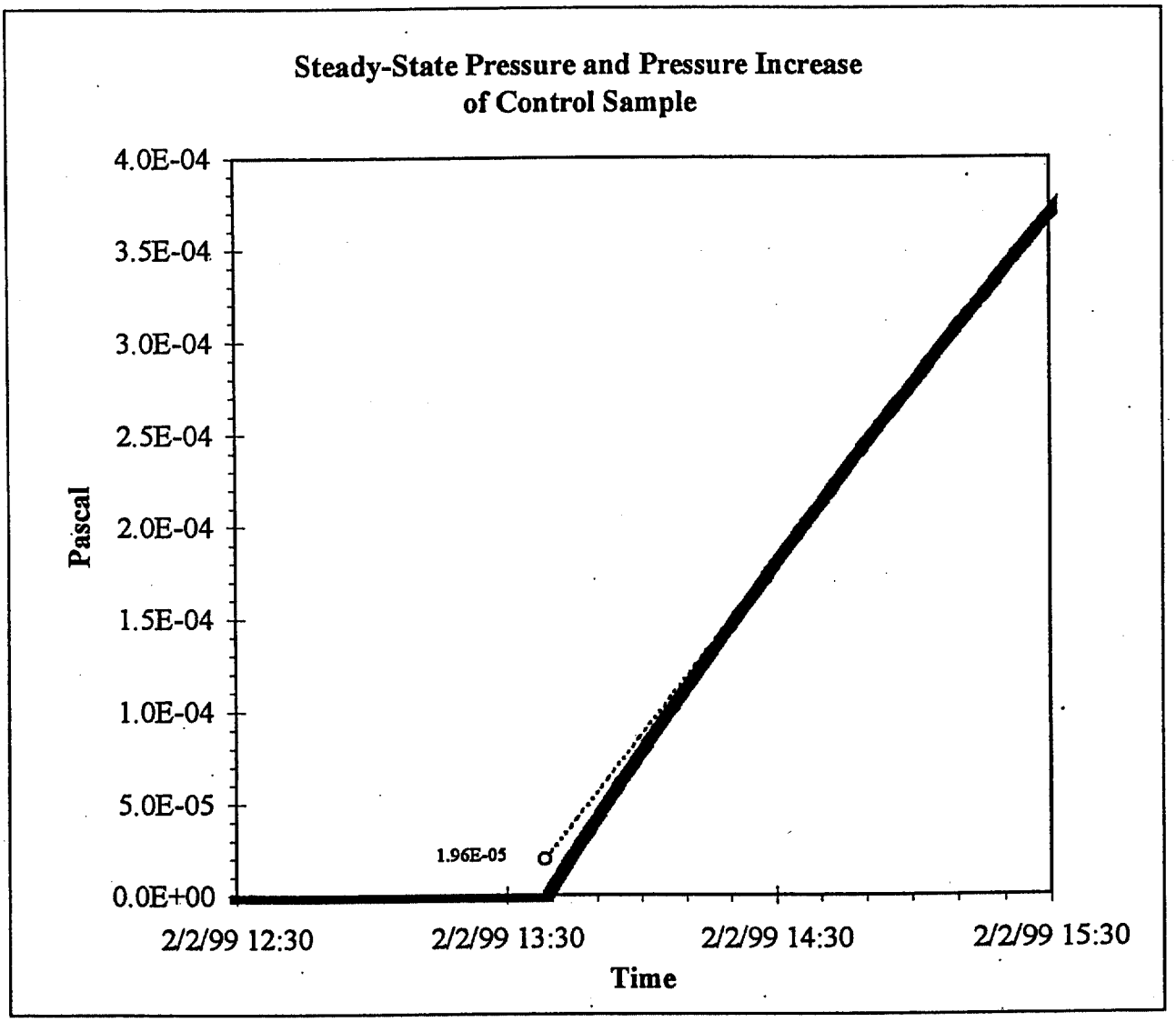

Figure 7. Steady-state pressure and pressure increase of Control sample.

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