VAPOR PRESSURE

TEST SUBSTANCE

Identity: N-Ethylperfluorooctanesulfonamide; may also be referred to as U1464, EtFOSA, EtPOSA, F-6309, or FX-12. (1-Octanesulfonamide, 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-heptadecafluoro-N-ethyl-, CAS # 4151-50-2)

Remarks: Material is a white solid. Report states that sample purity is 99% based on information supplied by Sponsor's representative. The lot number was not recorded.

METHOD

Method: OECD 104, Dynamic Method
GLP (Y/N): Yes
Year completed: 1998
Remarks:
The following quote from the test report describes the only test deviation: "While testing test item, the air condenser was used in place of water condenser because of low melting point of the test item. To the best of our (AScI Corporation) current scientific knowledge and understanding, this deviation should have no effect on the outcome of this test." (Quote taken from the test report).

RESULTS

Vapor Pressure: The vapor pressures at 10, 20, 25, and 30°C were 0.047, 0.142, 0.239, and 0.395 Pa, respectively based on the increasing pressure regression equation.
Decomposition (yes/no/ambiguous): Not noted

Remarks:. The estimated vapor pressures for the increasing and decreasing pressure conditions differ by 20 to 24%.

CONCLUSIONS

The vapor pressure as determined with the dynamic method was assumed to be 0.142 Pa at 20°C based on the increasing pressure regression equation.

DATA QUALITY

Reliability: Klimisch ranking = 2. This study meets all the criteria for quality testing. However, the 99% purity may not be correct. The value was assigned by NMR which is incomplete. Possible non-linear plot (not enough data points).

REFERENCES

Study conducted at the request of 3M Company by, AScI Corporation, Duluth, MN.

OTHER

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

Last changed: 5/16/00

ORIGINAL

FX-12

STUDY TITLE

Determination of Vapor Pressure Curve by Dynamic Method for U1464 (ET POSA)

DATA STANDARD

OECD Guideline 104, "Vapor Pressure Curve, Dynamic Method"

COPY

AUTHORS

Mike Signorelli
Linda Christensen
Phillip Marquis

STUDY COMPLETED

November 6, 1998

TESTING FACILITY

AScI Corporation/AScI-Duluth
Environmental Testing Division
4444 Airpark Boulevard
Duluth, MN 55811-5712

Tel. No. (218) 722-4040

STUDY IDENTIFICATION NUMBERS

AScI Study ID# 5030-045-01[a]

3M Company Environmental Technology and Safety Services
Sponsor Study ID# U1464

[a] This study report contains total of 26 pages. The raw data package (34 pages) supporting this study is kept on file at AScI-DETD.

CERTIFICATION OF GOOD LABORATORY PRACTICE COMPLIANCE

To the best of my knowledge, this study was conducted in accordance with OECD Good Laboratory Practice regulations (1).

ASci Study Director

Name (signed): _____ Date: 11/6/98

Name (typed): Linda Christensen

STATEMENT OF QUALITY ASSURANCE

The study data were reviewed by the AScI Quality Assurance Unit to assure that standard operating procedures and guidelines used to conduct this study were followed, and this report is an accurate reflection of the raw data. The types of audits performed for this study are listed in the following table.

Type of Audit for AScI Study ID# 5030-045-01	Audit Date	Date Reported to Study Director and Management
Test Protocol	3/26/98	3/27/98
In-Life Phase	4/3/98, 4/7/98	4/6/98, 4/8/98
Raw Data and Draft Report	5/27/98	5/27/98
Final Report	11/4/98	11/5/98

AScI's Quality Assurance Unit Auditor

Name (signed): _____ Date: 11/6/98

Name (typed): Alan Mozol

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APPENDIX B. Test Item Information

APPENDIX C. Test Item Receipt and Usage

APPENDIX D. Raw Data Sheets

STUDY SYNOPSIS

Title: Determination of Vapor Pressure Curve by Dynamic Method for U1464 (ET POSA)

Test Method: Dynamic method as described in AScI Protocol# 045-VP.P3M

Key Findings: 1) The estimated vapor pressures of the test item, ET POSA, are 0.142 and 0.176 Pa at 20°C; and 0.239 Pa and 0.293 Pa at 25°C.

Study Sponsor

Company: 3M Environmental Technology & Safety Services
935 Bush Avenue
Building 2-3E-09
St. Paul, Minnesota 55133-3331

Study Representative: Dr. Rich Purdy
Ecotoxicologist

Phone: (612) 778-5379

Fax: (612) 778-6176

Test item

Trade Name: ET POSA. Lot/Batch Number: FC12 from John Eberlin 3/12/98.

Sponsor Study Identification Number: U1464

Molecular Formula: $C_8F_{17}SO_2NCH_2CH_3$

Chemical Abstract Service Registry#/Code#: TradeSecret

Physical Description: White waxy solid

Percent Purity (% active ingredient): 99% Based on information supplied by Sponsor's Representative.

Stability Data: Stable at room temperature.

Storage Conditions: Room temperature in darkness.

Testing Facility

Company: ASci Corporation/ASci-Duluth
Environmental Testing Division (ASci)
4444 Airpark Blvd
Duluth, Minnesota 55811-5712

ASci Study Director: Linda Christensen

Phone: (218) 722-4040

Fax: (218) 722-2592

Reference Item

Trade Name: Toluene. Lot/Batch Number: 8608KTMH.

CAS Registry#/Code#: 108-88-3. Percent Purity: 99.9%.

Source: Mallinckrodt. Date Container Opened: 4/3/98.

Study Initiation Date: 3/30/98.

Experimental Dates

Experiment	Starting Date	Termination Date
Determination of Vapor Pressure	4/3/98	4/8/98

Location Raw Data and Final Report

Certified copies will be archived at testing facility for five years from the date the study is completed, after which, the Sponsor will be contacted to determine further disposition of all records.

1.0 INTRODUCTION

Chemical substances introduced into the environment present potential disposal and reentry hazards. The assessment of environmental fate and effect of these substances is required by various governmental agencies. Accordingly, one of the tests is to determine the vapor pressure of a substance. In this experiment, the test item boiling point is determined at a desired pressure and then vapor pressure and boiling point are correlated.

The vapor pressure of a substance is an environmentally relevant property because (1) it indicates the probability of phase transition, volatilization (liquid/gas) or sublimation (solid/gas), of a chemical, (2) it helps substantiate predictions regarding the environmental behavior of chemicals, (3) chemical vapor pressure and water solubility allow for the calculation of chemical volatility from an aqueous solution, and (4) it is useful in deciding whether photochemically induced degradation can be determined in the homogenous gas phase or in an absorbed phase (OECD 1993).

2.0 TEST METHODS

2.1 Glassware. All glassware employed were cleaned before use by performing the following:

1) Washed in hot detergent water and rinsed three times with warm tap water.

2) Immersed in 10% HNO_3 acid bath and then rinsed with deionized water.

3) Rinsed three times each with acetone followed by hexane.

All glassware used were labeled with the relevant information required for proper identification.

2.2 Test item. The test item, ET POSA, material safety data sheets, and certificate of analysis were received at ASci on March 27, 1998. The test item was in one glass bottle and was stored at room temperature in darkness as received. All relevant information about the test item is described earlier in this report. In this test, 13.39 g of test item was used.

2.3 Reference Item. Reagent grade toluene was used as a reference chemical. All relevant information about the reference item is described earlier in this report. In this test, 35 ml of reference chemical was used.

2.4 Test Apparatus. The test system consisted of:

1) 5-L Ballast bulb to vary pressure in double-neck distillation flask.

2) Closed-tube mercury manometer to measure pressure.

3) 100-ml double-neck distillation flask.

4) Centigrade thermometer, ± 0.2°C, with one hole rubber stopper to fit in the distillation flask.

5) Reflux condenser.

6) Heating element.

7) Vacuum pump.

8) Aspirator.

9) Boiling stones.

10) Nitrogen cylinder.

2.5 Determination of Vapor Pressure. The procedure to determine the vapor pressures of test and reference items was as follows:

1) Placed the item in double-neck distillation flask at a volume about one-third the flask volume, and added three to four boiling stones.

2) Flanged test system together and purged it with nitrogen. The pressure in the system was adjusted using vacuum.

3) Heated the item gradually until temperature equilibrium was reached, that is, when a constant boiling temperature was found.

4) At this point, recorded total pressure (in mm Hg) and boiling point T (in °C).

5) For at least five different pressures, increased pressure and recorded new total pressures and boiling points when equilibria were reached.

6) To obtain the second set of data, recorded total pressure and boiling point as the pressure was lowered.

3.0 TREATMENT OF RESULTS

For each data set with test and reference items, total pressure was converted to Pa unit by multiplying the observed millimeter mercury pressure with 133.3. Similarly, the boiling point (T) was converted to °K by adding 273.15 to the observed °C boiling point. The log of total pressure (Pa) vs. (1/T) from the individual data sets were regressed to develop the vapor pressure curves for each item. Vapor pressures for the test item and the reference item were determined at 10°C, 20°C, 25°C, and 30°C using appropriate linear regression equations.

4.0 RESULTS AND DISCUSSION

Reference Item: Toluene

The observed data obtained for increasing and decreasing the pressure are shown in Tables 1 and 2, respectively for toluene. The calculated regression curves from the plotted curves, Figures 1 and 2, are:

Increasing pressure:

$$\log p = 10.27\text{-}1994\ (1/T) \qquad (1)$$
$$r = -0.999,\ n = 5$$

Decreasing pressure:

$$\log p = 10.35\text{-}2023\ (1/T) \qquad (2)$$
$$r = -0.998,\ n = 5$$

The two data sets are similar and have correlation coefficients greater than 0.995. The estimated vapor pressures at 10, 20, 25, and 30°C are listed in Tables 3 and 4 for the two toluene data sets. Table 5 lists the range of vapor pressure values determined by the dynamic method from interlaboratory comparisons (4).

The toluene vapor pressures determined from equation 1 are within the ranges listed for the interlaboratory comparisons. The values determined from equation 2 are slightly lower at 10 and 20°C. The vapor pressure for toluene at 20°C (2.8×10^3 Pa) is lower than the criterion established in the protocol ($2.9\text{-}3.1 \times 10^3$ Pa). The vapor pressure for toluene at 10°C (1.6×10^{-3} Pa) is lower than the criterion stated in reference 4 ($1.7\text{-}1.8 \times 10^{-3}$ Pa). A problem occurred with the system as the pressure was decreased. To get the toluene to boil as the pressure was decreased, more boiling chips were added to the item. It is not clear whether this would have affected the temperature and pressure measurements.

The experimental design presented here for determining vapor pressure curves appears to provide data comparable to that reported in reference 4. The regression curves are linear for both increasing and decreasing pressures.

Test Item: ET POSA

The observed data obtained for increasing and decreasing the pressure are shown in Tables 6 and 7, respectively. The calculated regression curves from the plotted curves of log p vs (1/T), Figures 3 and 4 are:

Increasing pressure:

$$\log p = 12.64\text{-}3954\ (1/T) \qquad (3)$$
$$r = -0.999,\ n = 5$$

Decreasing pressure:

$$\log p = 12.45\text{-}3871\ (1/T) \qquad (4)$$
$$r = -0.997,\ n = 5$$

The estimated vapor pressures at 10, 20, 25, and 30°C are listed in Tables 8 and 9 for the test item. The estimated vapor pressures for the increasing and decreasing pressure conditions differ by 20 to 24 percent.

5.0 CONCLUSIONS

The placement of the thermometer in the apparatus proved to be pivotal in establishing accurate, steady temperature measurements. The placement of the thermometer for the test item was different than that of the reference item. The thermometer was placed approximately one inch above the bubbling test item in the collection flask. The thermometer for the reference item was placed in the neck of the collection flask.

Based on the increasing pressure regression equation, the vapor pressures at 10°C, 20°C, 25°C, and 30°C were 0.047 Pa, 0.142 Pa, 0.239 Pa, and 0.395 Pa, respectively.

6.0 DEVIATIONS FROM APPROVED TEST PROTOCOL

The deviation which occurred while conducting this study is:

1) While testing test item, the air condenser was used in place of water condenser because of low melting point of the test item.

To the best of our current scientific knowledge and understanding, this deviation should have no effect on the outcome of this test.

7.0 REPORT SIGNATURE

ASci Study Director

Name (signed): Linda Christensen Date: 11/6/98

Name (typed): Linda Christensen

Responsibilities, signatures, initials, and names of key personnel involved in this study are in Table 10.

8.0 REFERENCES

1) Organization for Economic Cooperation and Development (OECD), 1993. OECD Guidelines for Testing of Chemicals. OECD Publication Information Center, Washington, DC.

2) Shoemaker, D.P. and C.W. Garland. 1962. Experiments in Physical chemistry. McGraw-Hill Book Co., pp. 162-163.

3) Organization for Economic Cooperation and Development (OECD), 1997. Environment Directorate, Chemicals Group and Management Committee. OECD Series on Principles of Good Laboratory Practice and Compliance Monitoring. Number 1. OECD Principles on Good Laboratory Practice.

TABLE 1. **Toluene (Reference Item): Boiling Points (T) and Corresponding Vapor Pressures (p) at Increasing Pressure**

Measured Pressure			Observed Temperature		
mmHg	p (Pa)	Log p (Pa)	T (°C)	T (°K)	1/T (°K)
47	6265	3.797	35.0	308.15	0.003245
64	8531	3.931	41.2	314.35	0.003181
77	10264	4.011	45.5	318.65	0.003138
100	13330	4.125	51.2	324.35	0.003083
124	16529	4.218	56.5	329.65	0.003034

TABLE 2. **Toluene (Reference Item): Boiling Points (T) and Corresponding Vapor Pressures (p) at Decreasing Pressure**

Measured Pressure			Observed Temperature		
mmHg	P (Pa)	Log P (Pa)	T (°C)	T (°K)	1/T (°K)
150	19995	4.301	61.2	334.35	0.002991
128	17062	4.232	57.7	330.85	0.003023
110	14663	4.166	53.6	326.75	0.00306
77	10264	4.011	45.0	318.15	0.003143
52	6932	3.841	38.0	311.15	0.003214

TABLE 3. **Toluene (Reference Item): Estimated Vapor Pressures (p) at Different Temperatures (T) Determined from Equation 1**

T (°C)	Vapor Pressure (Pa)	Vapor Pressure (mmHg)
10	1.7×10^3	12.7
20	2.9×10^3	22.0
25	3.8×10^3	28.6
30	4.9×10^3	36.9

TABLE 4. **Toluene (Reference Item): Estimated Vapor Pressures (p) at Different Temperatures (T) Determined from Equation 2**

T (°C)	Vapor Pressure (Pa)	Vapor Pressure (mmHg)
10	1.6×10^3	12.0
20	2.8×10^3	21.1
25	3.7×10^3	27.5
30	4.8×10^3	35.6

TABLE 5. **Range of Vapor Pressure Values for Toluene by the Dynamic Method from OECD and EEC-Laboratory Intercomparison Testing Programs (4)**

Temperature (°C)	Range of Vapor Pressure (Pa)
10	(1.7-1.8) X 10^3
20	(2.9-3.1) X 10^3
30	(4.8-5.0) X 10^3

TABLE 6. **ET POSA (Test Item): Boiling points (T) and Corresponding Vapor Pressures (p) at Rising Pressure**

Observed Pressure			Observed Temperature		
mmHg	p (Pa)	Log p (Pa)	T (°C)	T (°K)	1/T (°K)
42	5599	3.748	171.6	444.75	0.002248
58	7331	3.888	178.8	451.95	0.002213
68	9064	3.957	181.8	454.95	0.002198
83	11064	4.044	186.4	459.55	0.002176
94	12530	4.098	190.2	463.35	0.002158

TABLE 7. ET POSA (Test Item): Boiling points (T) and Corresponding Vapor Pressures (p) at Decreasing Pressure

Observed Pressure			Observed Temperature		
mmHg	p (Pa)	Log p (Pa)	T (°C)	T (°K)	1/T (°K)
106	14130	4.150	193.0	466.15	0.002145
95	12664	4.103	191.0	464.15	0.002154
86	11464	4.059	188.0	461.15	0.002168
75	9998	4.000	183.8	456.95	0.002188
47	6265	3.797	174.4	447.55	0.002234

TABLE 8. ET POSA (Test Item): Estimated Vapor Pressures (p) at Different Temperatures (T) Determined from Equation 3

T (°C)	Vapor Pressure (Pa)	Vapor Pressure (mmHg)
10	0.047	0.36×10^{-3}
20	0.142	1.06×10^{-3}
25	0.239	1.79×10^{-3}
30	0.395	2.97×10^{-3}

Equation 3 -- Log Pa = 12.64-3954 (1/T)

Example Calculation

Log Pa = 12.64-3954 (1/T)

10°C = 283.15°K

Log Pa = 12.64-3954 (1/283.15)

Log Pa = 12.64-13.9643

Log Pa = 1.3243

Pa = Inv Log (-1.3243)

Pa = 0.047

For mm Hg = $\frac{0.047}{133.33} = 0.36 \times 10^{-3}$

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TABLE 9. **ET POSA (Test Item): Estimated Vapor Pressures (p) at Different Temperatures (T) Determined from Equation 4**

T (°C)	Vapor Pressure (Pa)	Vapor Pressure (mmHg)
10	0.060	0.45×10^{-3}
20	0.176	1.32×10^{-3}
25	0.293	2.20×10^{-3}
30	0.479	3.60×10^{-3}

Equation 4 = Log Pa = 12.45-3871 (1/T)

TABLE 10. ET POSA (Test Item): Responsibilities, Signatures, and Initials of Key Personnel Involved in the Study

Personnel	Title/Responsibility	Signature	Initials
Linda Christensen	Study Director		LC
Mike Signorelli	Laboratory assistance		MS
Alan Mozol	Quality Assurance Auditor		AM
Joe Amato	Project Manager		JA
Phillip Marquis	Report Preparation		PM
Nancy Jordan	Archivist		NJJ

Figure 1. A Representative Plot of Log p (Pa) and 1/T (K) for Toluene at Increasing Pressure

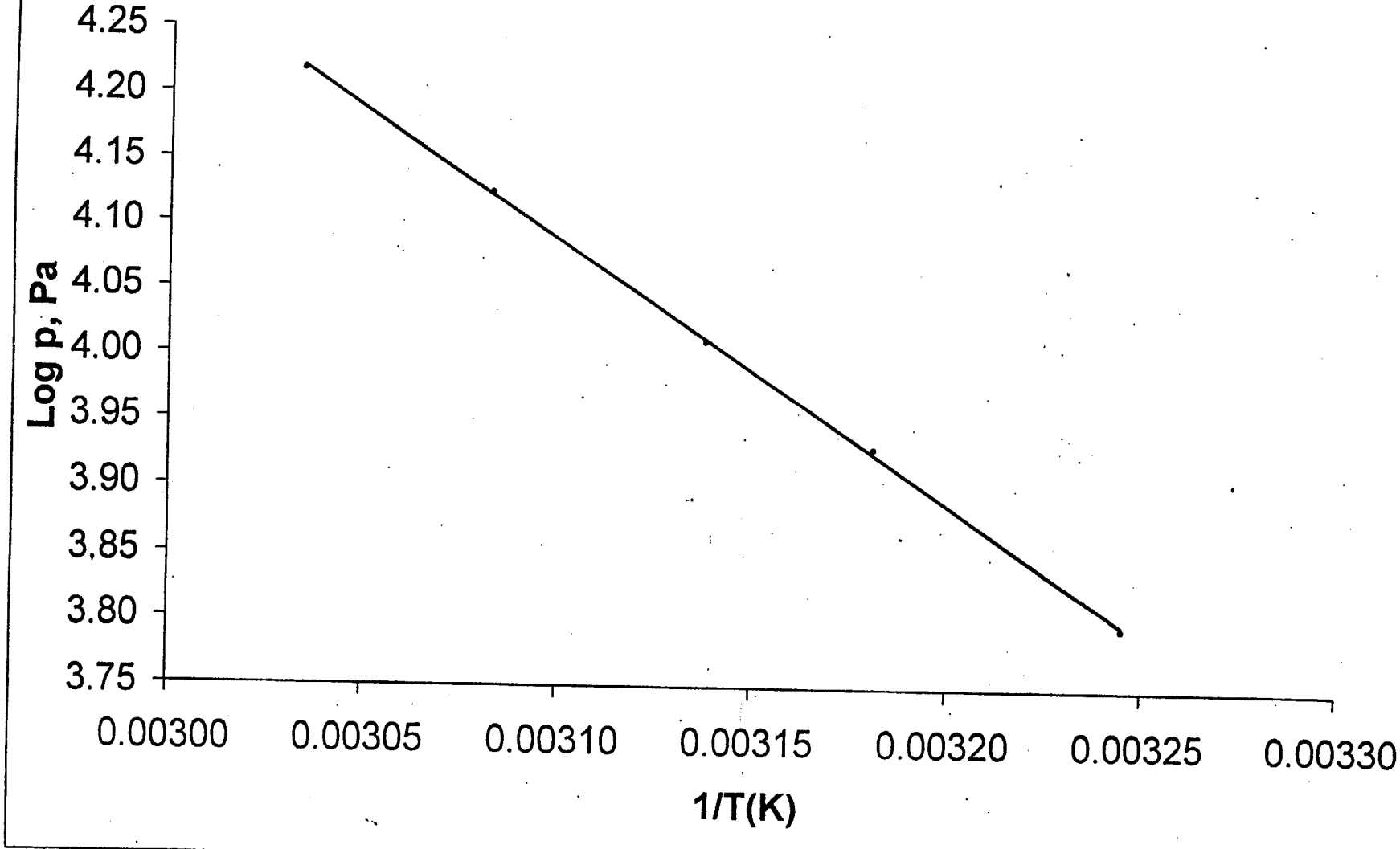

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Figure 2. A Representative Plot of Log p (Pa) and 1/T (K) for Toluene at Decreasing Pressure

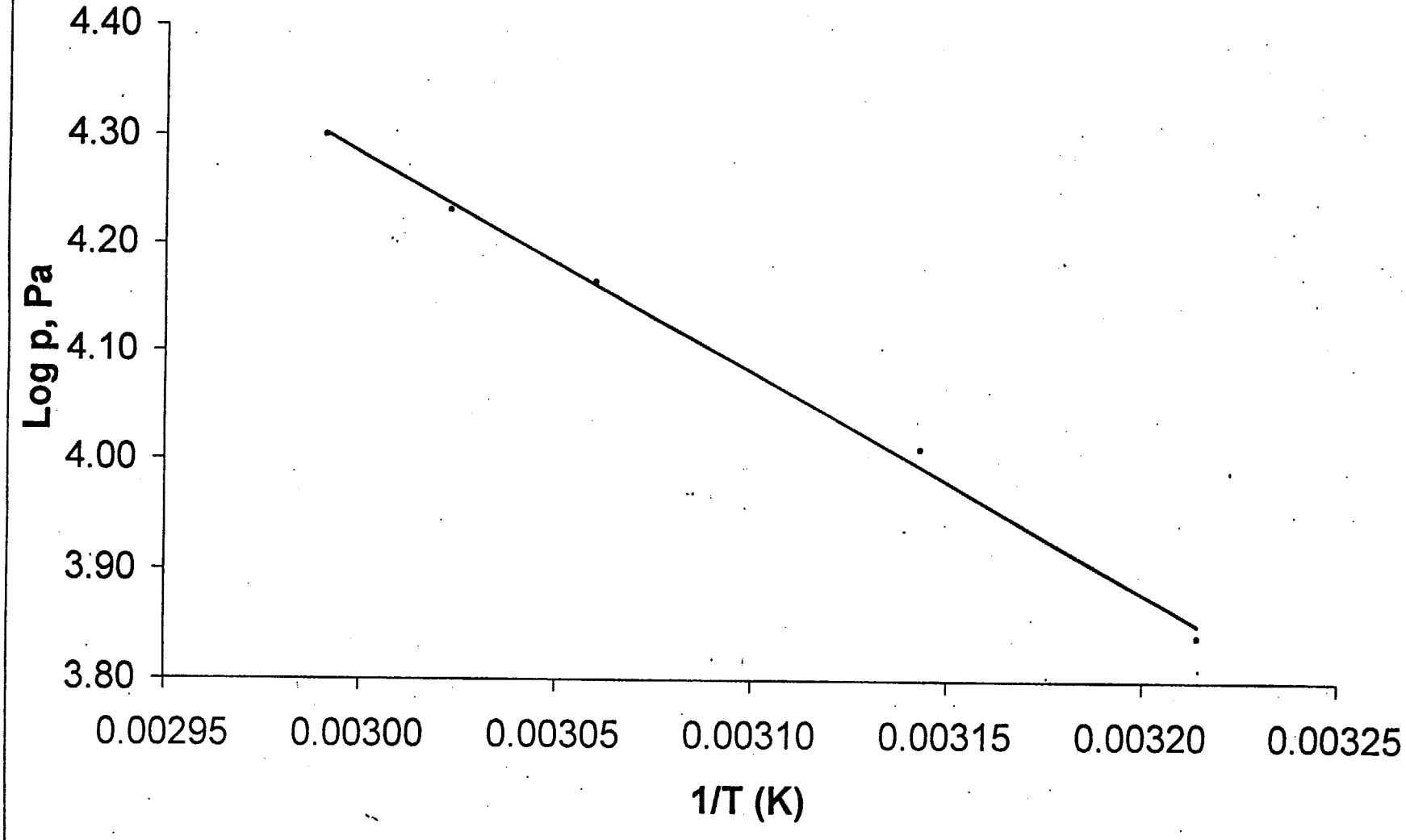

Figure 3. A Representative Plot of Log p (Pa) and 1/T (K) for ET POSA at Increasing Pressure

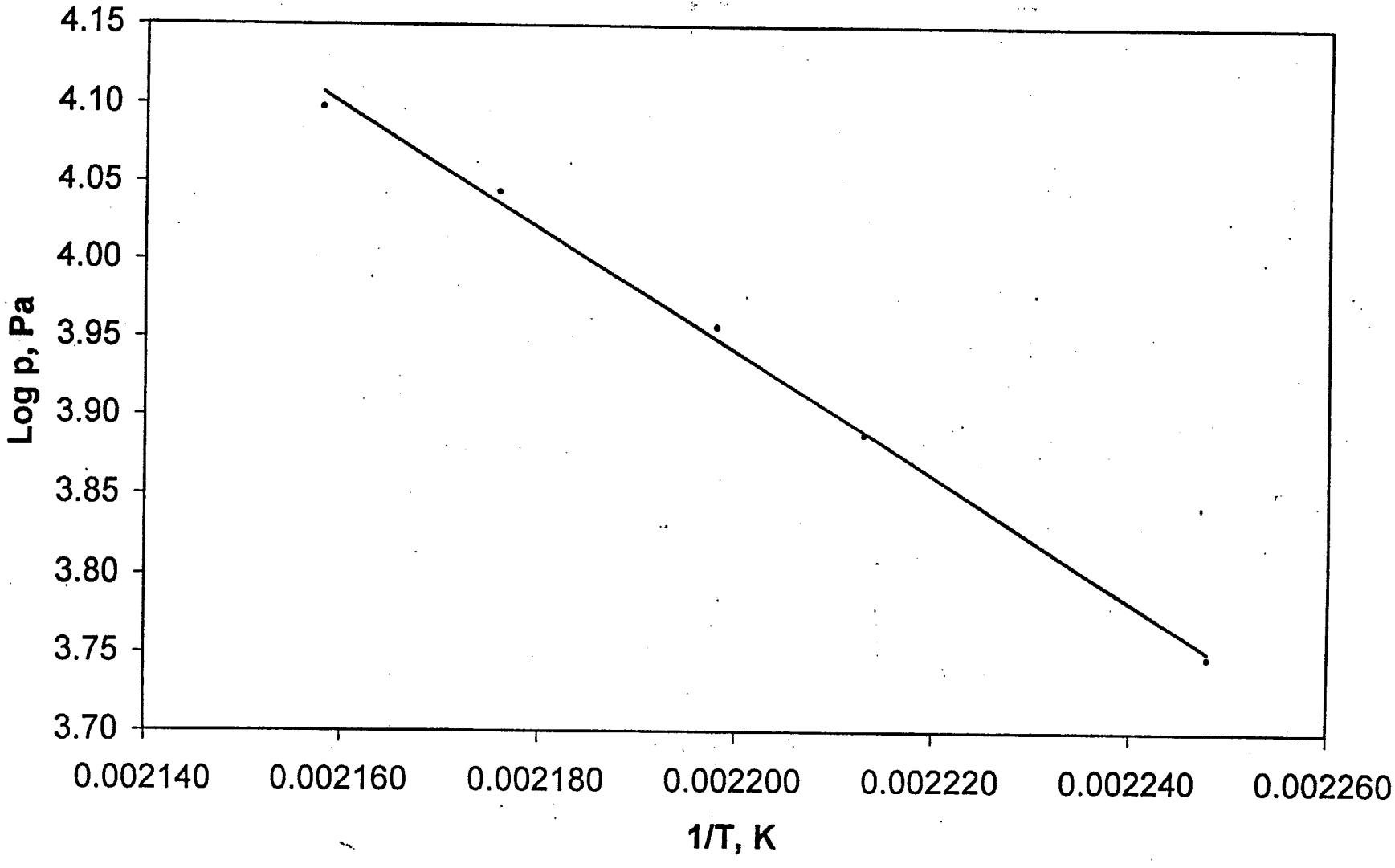

Figure 4. A Representative Plot of Log p (Pa) and 1/T (K) for ET POSA at Decreasing Pressure