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REPORT

DETERMINATION OF THE VAPOUR PRESSURE OF

[redacted]

NOTOX Project 338581
NOTOX Substance 111834/C

CONFIDENTIALITY STATEMENT

This report contains the unpublished results of research sponsored by [redacted] Reproduction, issue or disclosure to third parties in any form is not permitted without prior written authorisation from the sponsor.

STATEMENT OF GLP COMPLIANCE

NOTOX B.V., 's-Hertogenbosch, The Netherlands

The study described in this report has been correctly reported and was conducted in compliance with the most recent edition of:

The OECD Principles of Good Laboratory Practice

which are essentially in conformity with:

The United States Food and Drug Administration. Title 21 Code of Federal Regulations Part 58.

The United States Environmental Protection Agency (FIFRA). Title 40 Code of Federal Regulations Part 160.

The United States Environmental Protection Agency (TSCA). Title 40 Code of Federal Regulations Part 792.

Study Director

Ing. H.J. Krips

.....

Date: June 19, 2002

Management

Dr. Ir. H. Willems
Section Head Analytical & Physical Chemistry

.....

Date: June 19, 2002

Principal Scientist

Drs. A.C.G. van Genderen

.....

Date:
29/4/2002

QUALITY ASSURANCE STATEMENT

NOTOX B.V., 's-Hertogenbosch, The Netherlands

This report was audited by the NOTOX Quality Assurance Unit to ensure that the methods and results accurately reflect the raw data.

The dates of Quality Assurance inspections and audits are given below.
During the on-site inspections procedures applicable to this type of study were inspected.

DATES OF QAU INSPECTIONS/AUDITS	REPORTING DATES
on-site inspection (s)	
11-Dec-2001 (Process, DSC and vapour pressure)	13-Dec-2001
protocol inspection (s)	
13-Feb-2002 (Study)	13-Feb-2002
report audit (s)	
22-Apr-2002 (Study)	22-Apr-2002

Head of Quality Assurance
C.J. Mitchell B.Sc.
pp

..... J.T.G. Wiltschek

Date: June 24 2002

SUMMARY

Guidelines:

The determination of the vapour pressure of Trigonox R-938 was based on the following guidelines:

European Economic Community (EEC), EEC directive 92/69 EEC, Part A, Methods for the determination of physico-chemical properties, A.4 "Vapour pressure", EEC Publication No. L383, December 1992.

Organisation for Economic Co-operation and Development (OECD), OECD guidelines for testing chemicals, Guideline No.104: "Vapour pressure curve", July 27, 1995.

Result:

Using the Static Technique, the vapour pressure of [redacted]t 20°C was determined to be:

p (20°C) = 761 ± 30 Pa = 5.71 ± 0.23 mm Hg

Discussion:

Based on the structural formula of [redacted], a lower vapour pressure was expected. Probably the relative high vapour pressure was caused by the presence of water and [redacted]

PREFACE

Sponsor	[redacted]
Study Monitor	[redacted]
Testing Facilities	NOTOX B.V. Hambakenwetering 7 5231 DD 's-Hertogenbosch, The Netherlands The test was performed at the Thermodynamic Centre Utrecht Utrecht University Padualaan 8 3584 CH Utrecht, The Netherlands
Study Director	Ing. H.J. Krips (NOTOX B.V.)
Principal Scientist	Drs. A.C.G. van Genderen (Utrecht University)
Study plan	Start : 20 March 2002 Completed : 21 March 2002

TEST SUBSTANCE

Identification	[redacted]
Chemical name	[redacted]
CAS RN	[redacted]
Description	Clear colourless liquid
Batch	[redacted]
Purity	See Certificate of Analysis
Test substance storage	In refrigerator in the dark
Stability under storage conditions	Stable
Expiry date	01 January 2003

The sponsor is responsible for all test substance data unless determined by NOTOX.

Note: **Don't heat up the test substance above 50°C**

PURPOSE AND PRINCIPLE

The purpose of the study was to evaluate the vapour pressure of the test substance at 20°C. The vapour pressure is a function of the temperature and is specified in Pascal (Pa) or in mm Hg. One mm Hg equals 133.322 Pa.

The Static Technique was used to determine the vapour pressure at 20°C. In the static process, at thermodynamic equilibrium, the vapour pressures established in a closed system are determined at three specific temperatures. The measured results (ln p) are plotted versus the reciprocal temperature (in kelvin) and yield a rectilinear curve for the limited temperature range.

GUIDELINES

The study procedure described in this report is based on the following guidelines:

European Economic Community (EEC), EEC directive 92/69 EEC, Part A, Methods for the determination of physico-chemical properties, A.4 "Vapour pressure", EEC Publication No. L383, December 1992.

Organisation for Economic Co-operation and Development (OECD), OECD guidelines for testing chemicals, Guideline No.104: "Vapour pressure curve", July 27, 1995.

ARCHIVING

NOTOX B.V. will archive the following data for at least 10 years:
protocol, report, test substance reference sample and raw data. Thereafter, no data will be withdrawn without the sponsor's written consent.

TEST SYSTEM AND RATIONALE

The Static Technique.

Recognised by the international guidelines as recommended test system (EEC, OECD).

CALIBRATION OF THE TEST SYSTEM

The Static Technique is calibrated periodically using the primary pressure standard type 6-201-0001 (CEC Division of Bell & Howell company, California, USA).

PERFORMANCE OF THE TEST

Static vapour pressure measurements were made with a capacitance manometer fitted with a 133 kPa capacitive sensor. The reference pressure at the right-hand side of the diaphragm of the pressure sensor was kept below 10^{-4} Pa. The temperature of the sample was measured with a platinum resistance thermometer.

A clean and dry sample vessel was filled with 7.3 g of the test substance. After attaching the vessel to the measuring set-up, it was evacuated during about 2 minutes. The vessel was thereafter immersed in a thermostatic water bath of 9.31°C and the measurements were started. After measurement 17, the temperature of the thermostatic bath was raised to 15.48°C, and finally after measurement 28 to 20.86°C. A total of 44 measurements was performed.

DATA HANDLING

Because the measured vapour pressures were > 0.1 Pa, no correction for thermal transpiration was made.

The test substance was considered to show ideal behaviour. Thus the vapour pressure curve was derived by using a least squares method to calculate a straight line that best fits the measured data (Clapeyron).

The vapour pressure at 20°C was interpolated from the vapour pressure curve.

RESULTS

The Static Technique was used for the determination of the vapour pressure at 20°C.

At the beginning of the test, the vapour pressure of the test substance decreased slightly every next measurement, due to the removal of volatile impurities. After measurement 8, this decrease became negligible and the vapour pressure became stable. So from that moment on, the collected data were used for the determination of the vapour pressure of [redacted].

The results of the vapour pressure measurements are shown in the appendix added to the report and plotted in Figure 1. The results of the calculations are summarised in Table 1.

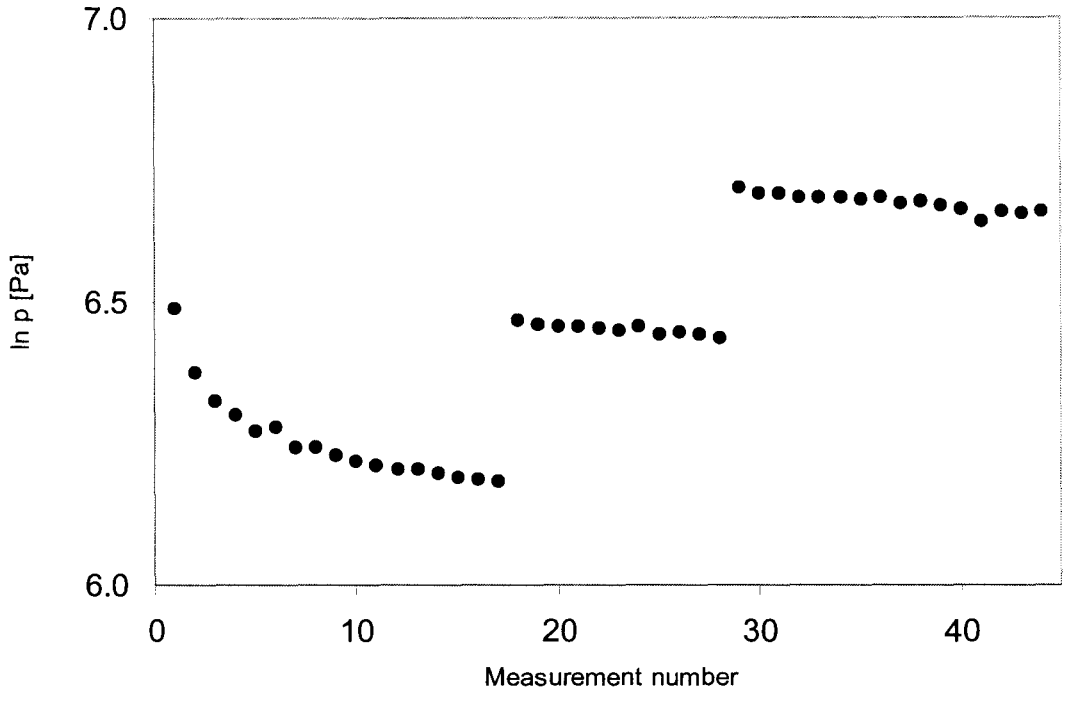

Figure 1 Graphical representation of the vapour pressure measurements (ln p versus measurement number) for [redacted].

Table 1 Determination of the vapour pressure of [redacted] at 3 different temperatures, using the Static Technique.

Measurement	Temperature		Mean vapour pressure $\pm 2\sigma_{n-1}$ [pa]	
	[°C]	[K]	p	ln p
9 – 19 [1]	9.31	282.46	495 ± 12	6.20 ± 0.024
18 – 28 [2]	15.48	288.63	632 ± 8	6.45 ± 0.012
29 – 44 [2]	20.86	294.01	790 ± 19	6.67 ± 0.023

[1] The first eight measurements were not used for calculations (see 'appendix').

[2] The first measurement was not used for calculations (see 'appendix').

Figure 2 shows the plot of the ln p values from Table 1 against the reciprocal temperature 1000/T (with T in Kelvin), taking into account the calculated error.

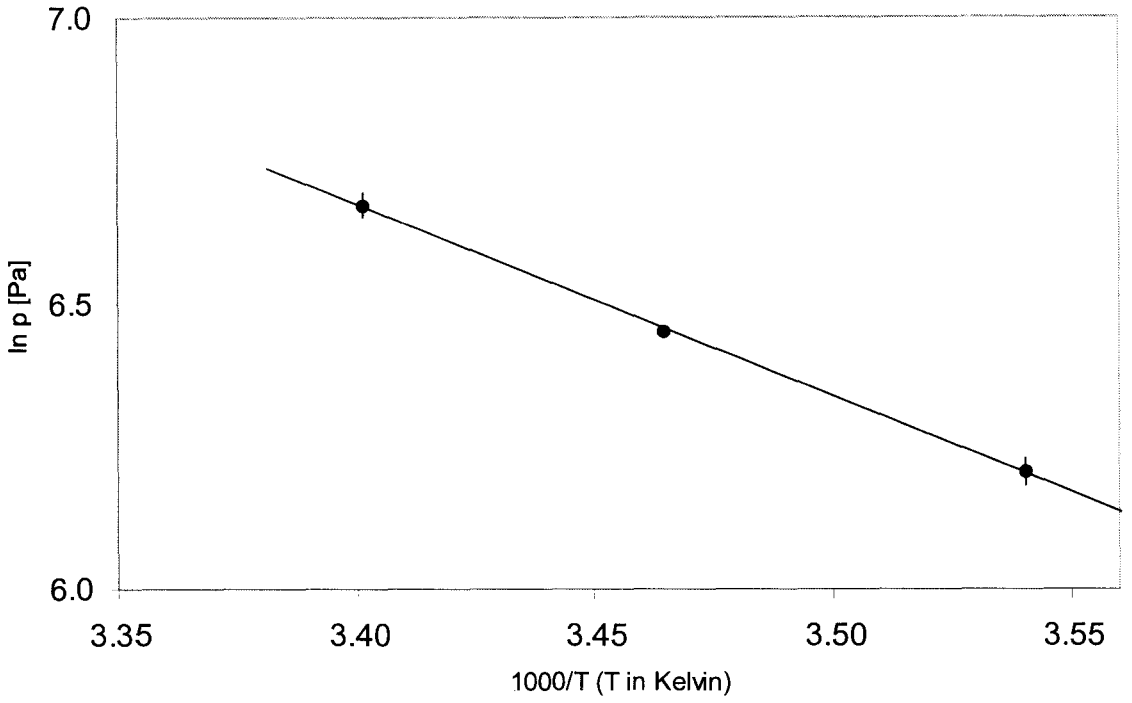

Figure 2 Vapour pressure curve (ln p versus 1000/T) for [redacted]

Fitting these data using the least squares method gives a value of 761 Pa for the vapour pressure of the test substance at 20°C with 23 Pa for $2\sigma_{n-1}$ (i.e. 3.0%). The maximum deviation between the fit and the data points is always less than about 0.6%. Errors made in estimating the mean value per temperature series are up to 1.2%. So a value of 30 Pa (i.e. 3.9%) is a reasonable value for the uncertainty in the final result.

Conclusion:

The vapour pressure of [redacted] at 20°C was determined to be:

p (20°C) = 761 ± 30 Pa = 5.71 ± 0.23 mm Hg

Discussion:

Based on the structural formula of [redacted] a lower vapour pressure was expected. Probably the relative high vapour pressure was caused by the presence of water and 3-methyl-2-butanone.

APPENDIX

The measured vapour pressures are summarised in the table below and are expressed in Pa. The values were rounded off before printing.

nr	p	ln p	nr	p	ln p	nr	p	ln p
1 *	657	6.49	18 *	643	6.47	29 *	811	6.70
2 *	587	6.37	19	640	6.46	30	802	6.69
3 *	559	6.33	20	636	6.45	31	804	6.69
4 *	544	6.30	21	636	6.45	32	799	6.68
5 *	529	6.27	22	634	6.45	33	797	6.68
6 *	533	6.28	23	632	6.45	34	799	6.68
7 *	515	6.24	24	635	6.45	35	796	6.68
8 *	515	6.24	25	629	6.44	36	798	6.68
9	508	6.23	26	631	6.45	37	791	6.67
10	503	6.22	27	627	6.44	38	792	6.67
11	499	6.21	28	624	6.44	39	788	6.67
12	496	6.21				40	781	6.66
13	496	6.21				41	766	6.64
14	492	6.20				42	778	6.66
15	489	6.19				43	777	6.66
16	486	6.19				44	777	6.66
17	484	6.18						

* System instability. (ln) p values obtained during this measurement were not used for calculations.

CERTIFICATE OF ANALYSIS

[redacted]

Certificate of Analysis

[redacted]

[redacted]
page 1 of 2

ICS-331

Product name : [redacted]

Chemical name : [redacted]

Batch number : [redacted]

Test results:

Method	Analysis of	Unit	Result *[1]
Jo/72.11,	[redacted]		
[redacted]			
[redacted]			
[redacted]			
[redacted]			

*[1] bracketed values are estimated 95% confidence intervals

File code : [redacted]

Analytical documentation : [redacted]

Authorized by

Name : [redacted]

[redacted] :

[redacted]

Certificate of Analysis

: specification of the peroxidic compounds

structure	% m/m