OCTANOL/WATER PARTITION COEFFICIENT

TEST SUBSTANCE

Identity: N-ethylperfluorooctane sulfonamidoethanol; may also be referred to as N-EtFOSE Alcohol or FM-3422. (1-Octanesulfonamide, N-ethyl-1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-heptadecafluoro-N-(2-hydroxyethyl)-, CAS # 1691-99-2)

Remarks: Material is an off-white, waxy solid. The test laboratory identifies the compound as "T-5874" Batch 2334.

METHOD

Method: OECD 117 & EEC-Directive 92/69 EEC, Annex V, Part A.8
GLP (Y/N): Yes
Year completed: 1994
Analytical: HPLC with Spectrophotometric detector

Remarks: Testing used six substances with known log P_{ow} values as references. These substances were used to calibrate the elution time in units of log P_{ow}.

Reference substance	**log P_{ow}**
Benzylalcohol	1.1
Toluene	2.7
1,4-dichlorobenzene	3.4
Fluoranthene (98%)	4.7
Dibenzyl (99%)	4.8
2,4-DDT (99%)	6.2

A stock solution of the test substance was prepared by dissolving 165 mg in 50.0 mL acetonitrile and sonicating for 3 minutes. The stock solution was diluted 10 times with mobile phase, resulting in the test solution.

Mobile phase is a 75/25 (v/v) solution of acetonitrile / Milli-Q water respectively.

RESULTS

Substance	log P_{ow}
Reference chemicals	
Benzylalcohol	1.1
Toluene	2.7
1,4-dichlorobenzene	3.4
Fluoranthene	4.7
Dibenzyl	4.8

2,4-DDT	6.2
Test substance	
Peak 1	5.6*
Peak 2 (major comp.)	4.4*
Several small peaks	2.2/4.4*

* Interpolated from the regression line: y = 0.288x – 0.730 (r=0.972, n=6)

CONCLUSIONS

This study estimates the log P_{ow} for N-EtFOSE alcohol.

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

DATA QUALITY

Reliability: Klimisch ranking 2

-per OECD. HPLC methods enables partition coefficients to be estimated in the log P_{ow} range between 0 and 6.

-Notox states impurities are of minor importance in HPLC method, however OECD states, HPLC method is less sensitive to the presence of impurities in the test substance than the Shake Flask method. Nevertheless, in some cases impurities can make the interpretation of the results difficult due to uncertainty in peak assignments. Mixtures which result in an unresolved band, upper and lower limits of log P_{ow} should be stated. Duplicate analysis should be reported in order to show confidence in the measurement (log P_{ow} values should fall within a range of +/- 0.1 log units) – data provided mean value therefore can't show confidence. pH of eluant is critical of ionizable compounds.
The study did not include any fluorochemicals among the reference substances. Without fluorochemical reference compounds, it is not known whether the same relationship between the capacity factor k" and Kow exists for compounds with perfluoroalkyl portions as exits for nonfluorinated reference materials such as those used in this study. The reference compounds should have been also injected independently to insure appropriate retention time assigned to each reference compound. Reference substances were injected twice, however only the mean retention time data is given for the reference substances thus repeatability of the retention times is not clear. It was not demonstrated that the test material was soluble in the acetonitrile stock solution or in the mobile phase. No demonstration that the major components of the test substance are detectable at UV λ = 210 nm (Generally, UV absorbance detectors poor choice for N-EtFOSE alcohol and related fluorochemicals). UV adsorption spectra is needed of the purified main components of the test substance. Assumptions are made that the major peak from the HPLC of the test substance is the major component. No attempt was made to confirm this (e.g. Mass spec analysis to show the material detected has the appropriate mass). It is not known whether

the major components even come off the column. Some k' values reported are calculated incorrectly.

REFERENCES

Study conducted at the request of 3M Company by NOTOX Safety & Environmental Research B.V., The Netherlands, April 7, 1994.

REPORT

DETERMINATION OF THE PARTITION COEFFICIENT (N-OCTANOL/WATER) OF T-5874 BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

NOTOX Project 121275
NOTOX substance 38187

STATEMENT OF GLP COMPLIANCE

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

The study described in this report 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: Drs. R. de Vries

i/a .....

Date: June 3, 1994

QUALITY ASSURANCE STATEMENT

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

Study procedures were subject to periodic inspections and general non study specific processes were also inspected at periodic intervals.

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

DATES OF QAU INSPECTIONS/ AUDITS	REPORTING DATES
25 March 1994	25 March 1994
28 March 1994	28 March 1994
21 April 1994	21 April 1994

Quality Assurance Manager C.J. Mitchell B.Sc.

Date: 09-06-94

REPORT APPROVAL

STUDY DIRECTOR: Drs. R. de Vries

c/a

.....

Date: June 8, 1994

MANAGEMENT: J.A.M.W. van Helvoirt
Section head Physico Chemistry

pp.

Dr. Ilona C. Enninga
Technical Director

.....

Date: 09/06/1994

PREFACE

Sponsor	3M Belgium - Chemical EBC Canadastraat 11 B-2070 ZWIJNDRECHT Belgium
Study Monitor	Mr. R.H. Cox
Testing Facility	NOTOX B.V. Hambakenwetering 3 5231 DD 's-Hertogenbosch The Netherlands
Study Director	Drs. R. de Vries
Study plan	Start: 30 March 1994 Completed: 07 April 1994

TEST SUBSTANCE

Identification	T-5874
Description	Cream solid
Batch	2334
Purity	100%
Storage conditions	At room temperature in the dark
Stability under storage conditions	Stable
Expiry date	January 01, 1996

PURPOSE AND PRINCIPLE

The purpose of the study was to determine the partition coefficient of the test substance between n-octanol and water.

The partition coefficient (n-octanol/water) (P_{ow}) is defined as the ratio of the equilibrium concentrations in a two phase system consisting of n-octanol and water.

The retention time of a substance in a reversed-phase High Performance liquid Chromatography (HPLC) chromatographic system is related to its partition coefficient (n-octanol/water).
The HPLC method is useful for log P_{ow} values between 0 and 6.
Using the HPLC method, impurities are of minor importance. The HPLC method must also be applied if the test substance is a mixture of compounds but is not applicable to strong acids and bases, metal complexes, substances which react with the eluent or surface-active agents.

GUIDELINES

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

Organization for Economic Co-operation and Development (OECD), OECD Guideline for Testing of Chemicals, guideline no. 117: "Partition coefficient (n-octanol/water) High Performance Liquid Chromatography (HPLC) method" (adopted March 30, 1989).

European Economic Community (EEC), EEC-Directive 92/69 EEC, Annex V, Part A, Methods for the determination of physico-chemical properties, A.8: "Partition coefficient". EEC Publication no. L383, December 1992.

ARCHIVING

NOTOX B.V. will archive the following data for at least 10 years: protocol, report, test substance reference sample and raw data.

TEST SYSTEM AND RATIONALE

A High Performance Liquid Chromatograph with a spectrophotometric detector. The stationary phase was bonded silica C_{18}. The mobile phase was 75/25 (v/v) acetonitrile/water. Both HPLC instrumentation and conditions are described in the section "method of chemical analysis".

The test system was recognized by the international guidelines (OECD, EEC).

REFERENCE SUBSTANCES

Six chemicals for which log P_{ow} has been reported were used to calibrate the elution time in units of log P_{ow}. The mixture of reference substances consisted of:

Reference substance	log P_{ow}[1]
- benzylalcohol (p.a., Merck)	1.1
- toluene (p.a., Merck)	2.7
- 1,4-dichlorobenzene (z.s., Merck)	3.4
- fluoranthene (98%, GC, Aldrich Chemie)	4.7
- dibenzyl (99%, GC, Aldrich Chemie)	4.8
- 2,4-DDT (99%, HPLC, Riedel de Haën, Seelze, FRG)	6.2

[1] log P_{ow} values according to the OECD guideline.

PERFORMANCE OF THE TEST

Preparation of the solutions

Solutions of the reference substances (except for ethylmethylketone) were prepared in methanol (HPLC-grade, Labscan Limited Co., Dublin, Ireland) at a concentration of 1.5-2 g/l. A mixture of the reference substances was prepared by adding 125 µl of each solution to a volumetric flask. Subsequently, this flask was made up with mobile phase to a final volume of 25.0 ml.
For the determination of t_0 (retention time of the unretarded component), a solution of formamide (p.a., Merck, Darmstadt, FRG) in methanol was prepared at a concentration of 1.7 g/l and thereafter diluted 10 times with mobile phase.
A stock solution of T-5874 was prepared by dissolving 165 mg test substance in 50.0 ml acetonitrile (3 minutes sonication). The stock solution was diluted 10 times with mobile phase, resulting in the test solution.

Performance of the test

The solutions were injected in the following sequence: the mixture of reference substances, mobile phase, the formamide solution, the test solution (in duplicate), mobile phase, the mixture of reference substances and the formamide solution.

Temperature of measurement

The temperature of the mobile phase was recorded several times during the measurements.

METHOD OF CHEMICAL ANALYSIS

The conditions used for the High Performance Liquid Chromatographic method are described below:

Analysis

Column	LiChrospher 100 RP-18; 125 x 4 (I.D.) mm; d_p= 5 µm (Merck, Darmstadt, FRG)
Mobile phase	75/25 (v/v) acetonitrile (HPLC-grade, Labscan Limited Co., Dublin, Ireland)/Milli-Q water (Millipore Corp., Bedford, MA, USA)
Flow	1 ml/min
Detection	UV at λ= 210 nm
Injection volume	10 µl

Instrumentation

HPLC Pump	Series 410 LC (Perkin Elmer, Norwalk, CT, USA)
Autosampler	ISS-200 (Perkin Elmer)
Detector	SpectroMonitor 3100 (LDC Analytical, Riviera Beach, FL, USA)
Integrator	SP 4290 (Spectra Physics, San Jose, CA, USA)

DATA HANDLING

Using High Performance Liquid Chromatography, especially large log P_{ow} values can be accurately determined from the chromatographic retention data. To this end, the capacity factor (k') was used, since it is proportional to the partition coefficient. The capacity factor was calculated from the retention of the substance concerned (t_r) and the unretarded component (t_o):

$$k' = (t_r - t_o)/t_o$$

From the results of the reference substances, a plot of log P_{ow} (x-value) versus log k' (y-value) was constructed, using linear regression analysis. The log k' value of each component of the test substance was compared with the log k' values of the reference substances with known log P_{ow} values.

RESULTS

Representative HPLC chromatograms of the test solution, the blank (mobile phase), the mixture of reference substances and the formamide solution are shown in Figures 1 to 4.

In the HPLC chromatograms of T-5874, using detection at 210 nm, one large peak at 4.1 minutes and a small peak at 7.7 minutes were observed. Between 1.6 and 4 minutes, several small peaks were observed. It was assumed that the large peak derived from the major component of test substance whereas the small(er) peaks derived from impurities.

The t_0 was determined to be 0.90 minutes as a mean value of both measurements (i.e 0.89 and 0.90 minutes).

The mean values of the retention times, k' values, log k' values, log P_{ow} and P_{ow} values, are summarized in Table 1. The plot of log P_{ow} (x-value) versus log k' (y-value) from the reference substances is shown in Figure 5.

Table 1 Results of the test.

Substance	t_r [1]	k'	log k'	log P_{ow}	P_{ow}
benzylalcohol	1.14	0.274	-0.563	1.1	
toluene	2.24	1.503	0.177	2.7	
1,4-dichlorobenzene	2.89	2.229	0.348	3.4	
fluoranthene	5.67	5.330	0.727	4.7	
dibenzyl	4.07	3.547	0.550	4.8	
2,4-DDT	9.12	9.190	0.963	6.2	
test substance					
peak 1	7.70	7.598	0.881	5.6 [2]	39.8×10^4
peak 2 (major comp.)	4.05	3.525	0.547	4.4 [2]	2.51×10^4
several small peaks	1.6 / 4	0.79/ 3.47	-0.10/0.54	2.2/4.4 [2]	1.50×10^2 / 2.51×10^4

[1] Mean value of the retention times of both chromatograms.
[2] Interpolated from the regression line: y = 0.288x - 0.730 (r=0.972, n=6)
Note : The calculations were performed using not rounded values.

The temperature of the mobile phase was 20.2-20.6°C during the measurements.

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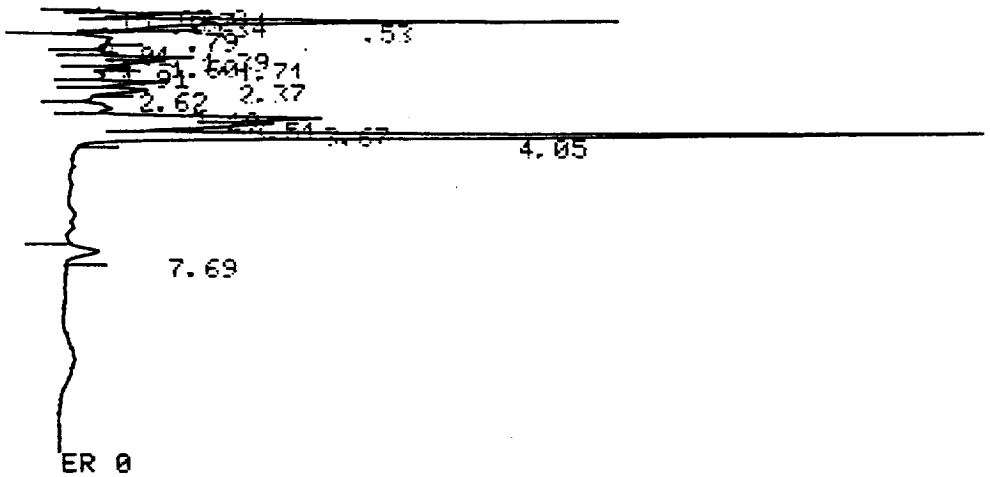

ER 0
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38196/121286 07-04-94 08:44:55 CH= "A" PS= 1.

FILE 1. METHOD 0. RUN 10 INDEX 10 BIN 53

ANALYST: KRH

PEAK#	AREA%	RT	AREA	BC
1	0.495	0.15	1399	02
2	2.459	0.25	6951	02
3	1.083	0.34	3061	02
4	18.792	0.53	53119	08
5	1.288	0.79	3640	05
6	2.538	1.01	7175	05
7	0.468	1.39	1322	02
8	2.476	1.6	7000	02
9	1.977	1.71	5588	02
10	0.636	1.91	1797	03
11	1.864	2.37	5268	02
12	2.067	2.62	5842	03
13	1.718	3.19	4857	02
14	11.232	3.51	31750	02
15	10.435	3.67	29495	02
16	38.458	4.05	108707	03
17	2.014	7.69	5694	01
TOTAL	100.		282665	

Figure 1 HPLC chromatogram of the test solution of 330 mg/l T-5874 in mobile phase.

Column	LiChrospher 100 RP-18; 125x4 (I.D.) mm; d_p=5 µm
Mobile phase	75/25 (v/v) acetonitrile/Milli-Q water
Flow	1 ml/min
Detection	UV, at λ = 210 nm
Injection volume	10 µl

Note: The peaks between 0 and 1.6 minutes were also observed in the chromatograms of the blank (see Figure 2). Therefore it was assumed that these peaks did not derive from the test substance.

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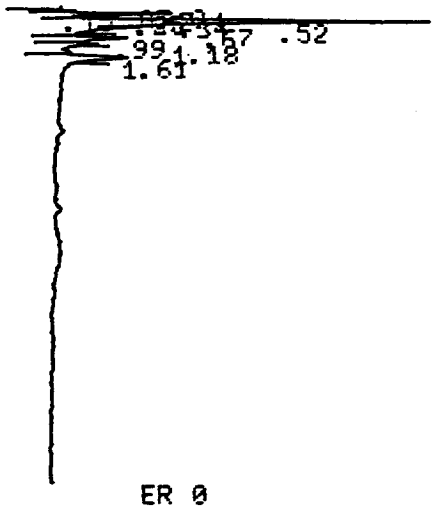

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DATA SAVED TO BIN # 56

38196/121286 07-04-94 09:34:04 CH= "A" PS= 1.

FILE 1. METHOD 0. RUN 13 INDEX 13 BIN 56

ANALYST: KRH

PEAK#	AREA%	RT	AREA BC
1	1.543	0.15	877 02
2	10.515	0.25	5976 02
3	3.853	0.34	2190 02
4	55.335	0.52	31449 02
5	7.768	0.67	4415 03
6	6.901	0.99	3922 02
7	2.509	1.18	1426 03
8	11.576	1.61	6579 01
TOTAL	100.		56834

Figure 2 HPLC chromatogram of a blank (mobile phase).

Column	LiChrospher 100 RP-18; 125x4 (I.D.) mm; d_p=5 µm
Mobile phase	75/25 (v/v) acetonitrile/Milli-Q water
Flow	1 ml/min
Detection	UV, at λ = 210 nm
Injection volume	10 µl

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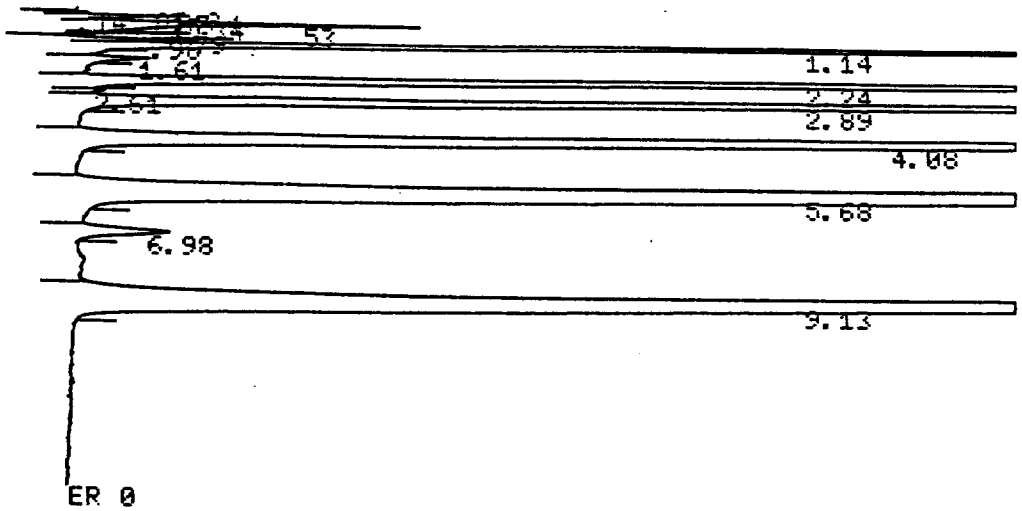

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38196/121286 07-04-94 09:50:17 CH= "A" PS= 1.

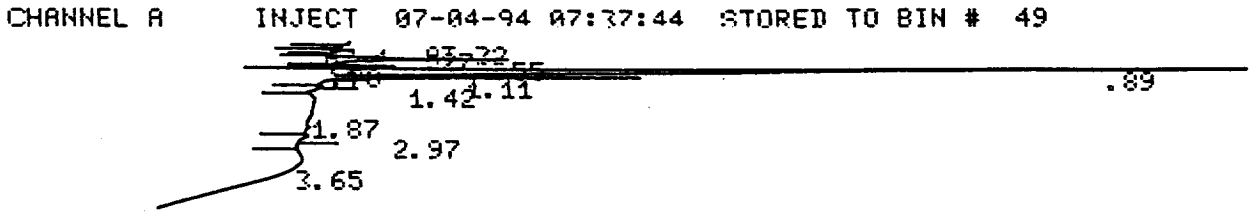
FILE 1. METHOD 0. RUN 14 INDEX 14 BIN 57

ANALYST: KRH

PEAK#	AREA%	RT	AREA BC
1	0.023	0.14	1131 02
2	0.145	0.25	7039 02
3	0.06	0.34	2917 02
4	0.879	0.53	42751 08
5	0.073	0.79	3542 05
6	0.139	0.96	6771 06
7	8.977	1.14	436774 03
8	0.112	1.61	5460 01
9	11.317	2.24	550640 01
10	0.013	2.61	611 02
11	10.567	2.89	514173 03
12	15.524	4.08	755334 01
13	38.584	5.68	1877339 01
14	0.316	6.98	15361 01
15	13.272	9.13	645770 01
TOTAL	100.		4865613

Figure 3 HPLC chromatogram of the mixture of reference substances in mobile phase.

Column	LiChrospher 100 RP-18; 125x4 (I.D.) mm; d_p=5 µm
Mobile phase	75/25 (v/v) acetonitrile/Milli-Q water
Flow	1 ml/min
Detection	UV, at λ = 210 nm
Injection volume	10 µl



38196/121286 07-04-94 07:37:44 CH= "A" PS= 1.

FILE 1. METHOD 0. RUN 6 INDEX 6 BIN 49

ANALYST: KRH

PEAK#	AREA%	RT	AREA BC
1	0.35	0.16	2624 02
2	1.189	0.27	8926 02
3	0.79	.3	5930 02
4	10.44	.35	78363 08
5	1.884	0.78	14143 06
6	54.548	0.89	409437 08
7	6.246	1.11	46879 05
8	0.12	1.42	899 05
9	3.094	1.87	23224 02
10	0.424	2.97	3185 03
11	20.915	3.65	156989 01
TOTAL	100.		750599

Figure 4 HPLC chromatogram of a formamide solution of 0.2 g/l in mobile phase.

Column	LiChrospher 100 RP-18; 125x4 (I.D.) mm; d_p=5 µm
Mobile phase	75/25 (v/v) acetonitrile/Milli-Q water
Flow	1 ml/min
Detection	UV, at λ = 210 nm
Injection volume	10 µl

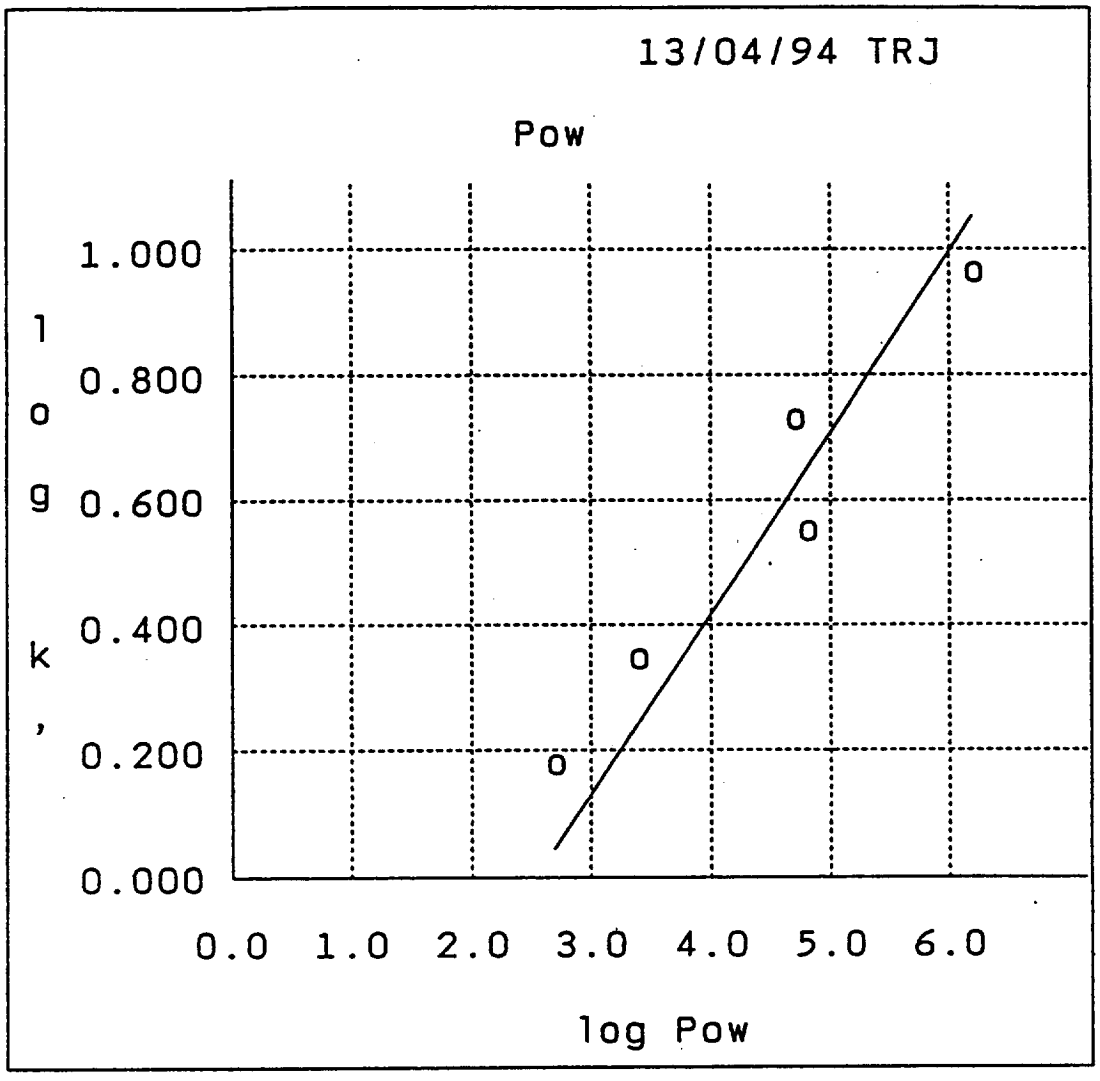


Figure 5 Plot of log P_{OW} (x-value) versus log k' (y-value) from the reference substances.