

## OCTANOL/WATER PARTITION COEFFICIENT

### TEST SUBSTANCE

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**Identity:** N2-(N-methylperfluorooctanesulfonamido)-ethyl acrylate; may also be referred to as N-MeFOSEA, or MeFOSEA. (2-Propenoic acid, 2-[[[(heptadecafluorooctyl)sulfonyl]methylamino]ethyl ester, CAS # 25268-77-3)

**Remarks:** Material is an amber, waxy solid. The test laboratory identifies the compound as "T-5869" (Batch 2408). Sample purity is given as "95%". The remaining 5% of the product was not characterized.

### METHOD

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**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 287 mg in 50.0 mL acetonitrile and sonicating for 2 minutes. The stock solution was diluted 10 times with mobile phase, resulting in the test solution (574 mg/L).

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

### RESULTS

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| 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 (major comp.) | 5.6*      |
| Peak 2               | 5.4*      |
| Peak 3               | 5.3*      |
| Several small peaks  | -0.7/5.2* |

• Interpolated from the regression line:  $y = 0.288x - 0.730$  ( $r=0.972$ ,  $n=6$ )

\*\*  $\log P_{ow}$  values according to the OECD guidelines.

## CONCLUSIONS

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This study estimates the  $\log P_{ow}$  for MeFOSEA.

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

## DATA QUALITY

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**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  $\pm 0.1$  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  $K_{ow}$  exists for compounds with perfluoroalkyl portions as exists 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  $\lambda = 210$  nm. 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

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

#### OTHER

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**Last changed: 5/17/00**

## REPORT

DETERMINATION OF THE PARTITION COEFFICIENT (N-OCTANOL/WATER) OF  
T-5869.4  
BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

Summary  
Created  
5/17/00  
McFOSSE

RECEIVED

NOV - 9 1994

3M

TOXICOLOGY

STATEMENT OF GLP COMPLIANCE

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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:

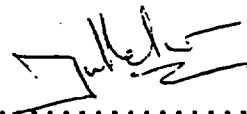
The United States Food and Drug Administration. Title 21 Code of Federal Regulations Part 58.

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

cla   
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Date: June 3, 1991

**QUALITY ASSURANCE STATEMENT**

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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/<br>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.

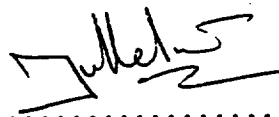
  
Date: 09-06-94.

REPORT APPROVAL

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STUDY DIRECTOR:

Drs. R. de Vries

*cla* 

Date: *June 9, 1994*

MANAGEMENT:

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

PP.



Dr. Wona C. Enninga  
Technical Director

Date: *09/06/1994*

PREFACE

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

TEST SUBSTANCE

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|                                    |                                 |
|------------------------------------|---------------------------------|
| Identification                     | T-5869                          |
| Description                        | Cream solid                     |
| Batch                              | 2408                            |
| Purity                             | 95%                             |
| Storage conditions                 | At room temperature in the dark |
| Stability under storage conditions | Stable                          |
| Expiry date                        | January 01, 1996                |

## PURPOSE AND PRINCIPLE

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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

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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

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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<sub>18</sub>. 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<sub>ow</sub> has been reported were used to calibrate the elution time in units of log P<sub>ow</sub>. The mixture of reference substances consisted of:

| Reference substance                                | log P <sub>ow</sub> <sup>1</sup> |
|----------------------------------------------------|----------------------------------|
| - 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                              |

<sup>1</sup> log P<sub>ow</sub> values according to the OECD guideline.

## PERFORMANCE OF THE TEST

### Preparation of the solutions

Solutions of the reference substances 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<sub>0</sub> (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-5869 was prepared by dissolving 287 mg test substance in 50.0 ml acetonitrile (2 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

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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;<br>$d_p = 5 \mu\text{m}$ (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 $\lambda = 210 \text{ nm}$                                                                                              |
| Injection volume | 10 $\mu\text{l}$                                                                                                              |

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

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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 ( $t_0$ ):

$$k' = (t_r - t_0)/t_0$$

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-5869, using detection at 210 nm, one large and 2 small test substance peaks were observed. In addition, many small test substance peaks were observed between approximately 1 and 6 minutes. It was assumed that the large peak derived from the major component of test substance whereas the small 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 (major comp.) | 7.75    | 7.659      | 0.884      | 5.6 <sup>2</sup>      | $3.98 \times 10^5$       |
| peak 2               | 6.96    | 6.777      | 0.831      | 5.4 <sup>2</sup>      | $2.51 \times 10^5$       |
| peak 3               | 6.60    | 6.374      | 0.804      | 5.3 <sup>2</sup>      | $2.00 \times 10^5$       |
| several small peaks  | 1 - 6   | 0.12/ 5.67 | -0.93/0.75 | -0.7/5.2 <sup>2</sup> | 0.2/1.58x10 <sup>5</sup> |

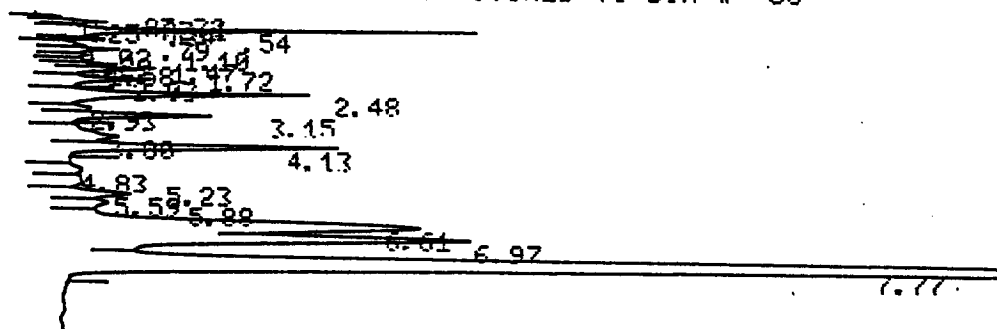
<sup>1</sup> Mean value of the retention times of both chromatograms.

<sup>2</sup> 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.

CHANNEL A INJECT 07-04-94 08:03:58 STORED TO BIN # 50



DATA SAVED TO BIN # 50

38196/121286

07-04-94 08:03:58

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FILE 1. METHOD 0. RUN 7 INDEX 7 BIN 50

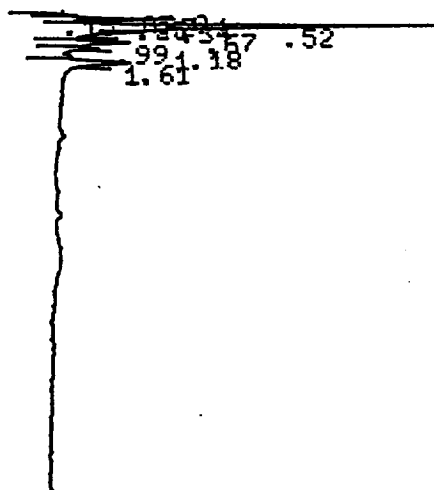
| PEAK# | AREA%  | RT   | AREA BC    |
|-------|--------|------|------------|
| 1     | 0.041  | 0.15 | 1321 02    |
| 2     | 0.209  | 0.25 | 6658 02    |
| 3     | 0.098  | 0.34 | 3122 02    |
| 4     | 3.425  | 0.54 | 109239 08  |
| 5     | 0.121  | 0.79 | 3874 05    |
| 6     | 0.251  | 1.02 | 8011 06    |
| 7     | 0.498  | 1.1  | 15893 06   |
| 8     | 0.189  | 1.29 | 6033 06    |
| 9     | 0.25   | 1.47 | 7978 06    |
| 10    | 0.428  | 1.58 | 13648 06   |
| 11    | 0.839  | 1.72 | 26774 06   |
| 12    | 0.899  | 2.03 | 28669 07   |
| 13    | 3.097  | 2.48 | 98772 08   |
| 14    | 0.092  | 2.93 | 2936 06    |
| 15    | 1.652  | 3.15 | 52708 03   |
| 16    | 1.125  | 3.8  | 35886 02   |
| 17    | 4.167  | 4.13 | 132925 03  |
| 18    | 0.248  | 4.83 | 7895 02    |
| 19    | 0.432  | 5.23 | 13795 02   |
| 20    | 1.217  | 5.59 | 38811 02   |
| 21    | 0.89   | 5.88 | 28393 02   |
| 22    | 13.076 | 6.61 | 417083 02  |
| 23    | 11.364 | 6.97 | 362494 02  |
| 24    | 55.39  | 7.77 | 1766796 03 |

Figure 1 HPLC chromatogram of the test solution of 574 mg/l T-5869 in mobile phase.

Column LiChrospher 100 RP-18; 125x4 (I.D.) mm;  $d_p=5\ \mu\text{m}$   
 Mobile phase 75/25 (v/v) acetonitrile/Milli-Q water  
 Flow 1 ml/min  
 Detection UV, at  $\lambda = 210\ \text{nm}$   
 Injection volume 10  $\mu\text{l}$

Note: All the peaks between 0 and approximately 1 minute and some of the peaks between approximately 1 and 2 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.

CHANNEL A INJECT 07-04-94 09:34:04 STORED TO BIN # 56

ER 0  
DATA SAVED TO BIN # 56

38196/121286

07-04-94 09:34:04

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

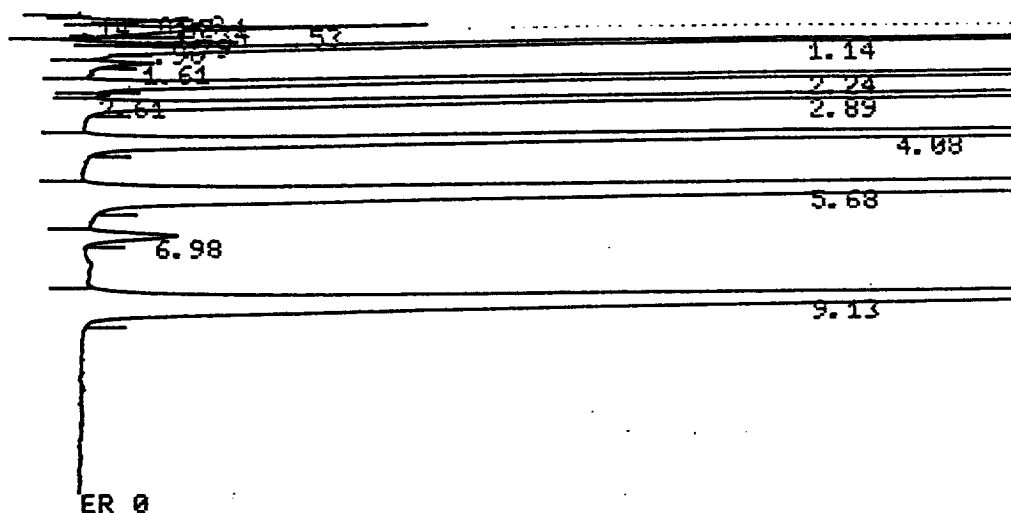
ANALYST: KRH

| 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; $d_p=5\ \mu\text{m}$ |
| Mobile phase     | 75/25 (v/v) acetonitrile/Milli-Q water                       |
| Flow             | 1 ml/min                                                     |
| Detection        | UV, at $\lambda = 210\ \text{nm}$                            |
| Injection volume | 10 $\mu\text{l}$                                             |

CHANNEL A INJECT 07-04-94 09:50:17 STORED TO BIN # 57

ER 0  
DATA SAVED TO BIN # 57

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 |        |      | 4865613 |    |

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

|                  |                                                              |
|------------------|--------------------------------------------------------------|
| Column           | LiChrospher 100 RP-18; 125x4 (I.D.) mm; $d_p=5\ \mu\text{m}$ |
| Mobile phase     | 75/25 (v/v) acetonitrile/Milli-Q water                       |
| Flow             | 1 ml/min                                                     |
| Detection        | UV, at $\lambda = 210\ \text{nm}$                            |
| Injection volume | 10 $\mu\text{l}$                                             |

CHANNEL A INJECT 07-04-94 07:37:44 STORED TO BIN # 49



DATA SAVED TO BIN # 49

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   | 0.79 | 5930   | 02 |
| 4     | 10.44  | 1.11 | 78363  | 08 |
| 5     | 1.884  | 0.78 | 14143  | 06 |
| 6     | 54.548 | 0.89 | 409437 | 08 |
| 7     | 6.246  | 1.11 | 179    | 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; $d_p=5 \mu m$ |
| Mobile phase     | 75/25 (v/v) acetonitrile/Milli-Q water                |
| Flow             | 1 ml/min                                              |
| Detection        | UV, at $\lambda = 210 \text{ nm}$                     |
| Injection volume | 10 $\mu l$                                            |

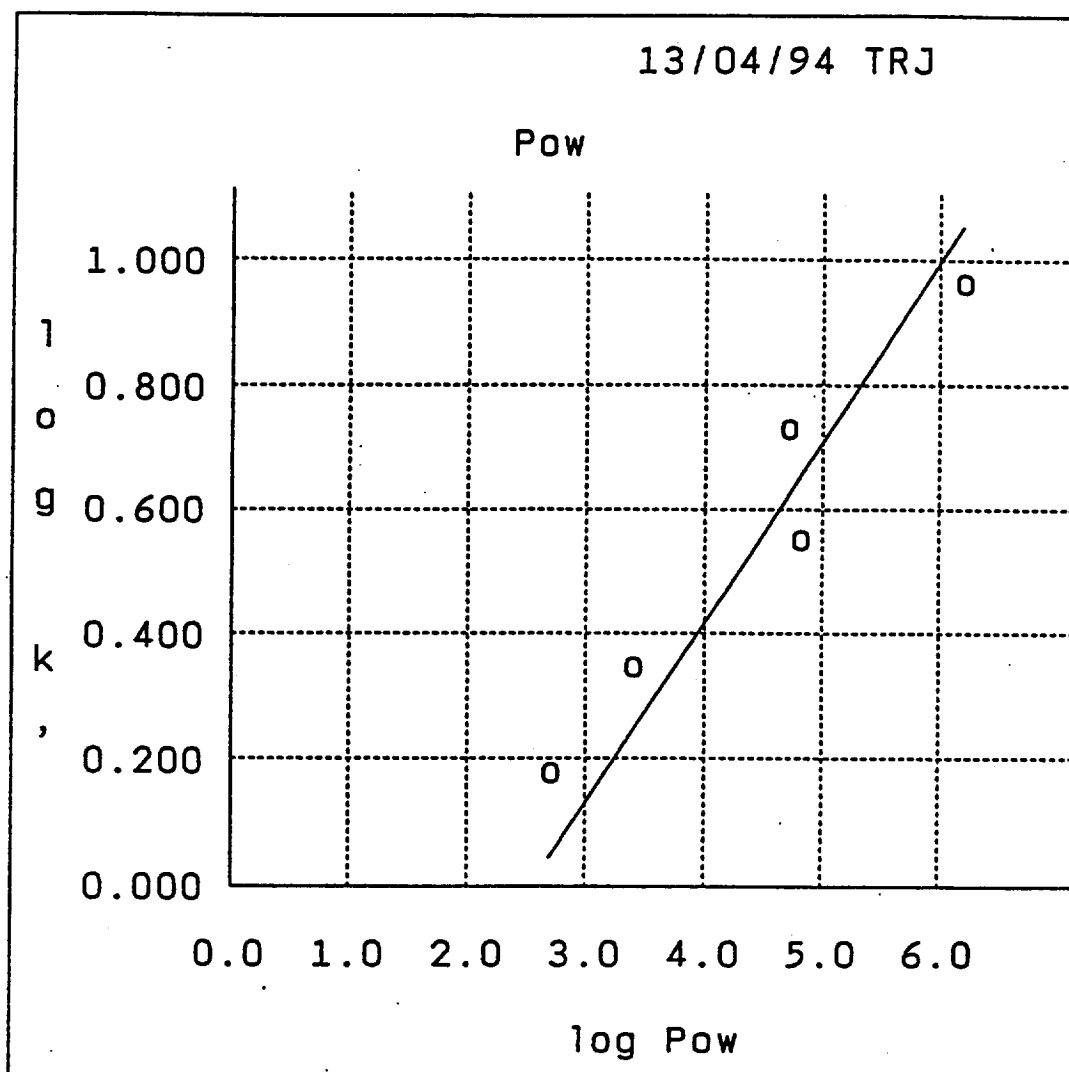


Figure 5 Plot of log  $P_{ow}$  (x-value) versus log  $k'$  (y-value) from the reference substances.