PHYSICAL/CHEMICAL PROPERTIES

TEST SUBSTANCE

Identity: [2-(N-Ethylperfluorooctanesulfonamido) ethyl acrylate; may also be referred to as B1228, D-1, EtFOSEA, or FX-13. (2-Propenoic acid, 2-[ethyl[heptadecafluorooctyl)sulfonyl]amino]ethyl ester, CAS # 423-82-5)

Remarks: Material is an amber solid.

Remarks: Report states that sample purity is 99% or more based on information supplied by Sponsor. The purity/identity of the test substance cannot be substantiated. As presented in the report, the structural formula indicates that "purity" cannot be assigned as "99% or more". The lot number was 101.

METHOD

Water Solubility: OECD 105, Shake Flask Method
Hydrolysis as a Function of pH: OECD 111
Dissociation Constants in Water: OECD 112, Spectrophotometric Method
Partition Coefficient (1-octanol/water) by HPLC: OECD 117
Vapor Pressure: OECD 104, Gas Saturation Method

GLP (Y/N): Yes
Year completed: 1994
Remarks:
Details from these studies can be found in one report entitled: "Determination of Physico-chemical Properties of Sample D-1".

RESULTS

Water Solubility: 0.89 mg/L at 25°C
Hydrolysis as a Function of pH: At 25°C the reaction rate constants of the test substance at pH 4, 7, and 9 were 0.017, 0.020, and 0.046 day^{-1} with half life times of 42, 35, and 15 days, respectively.
Dissociation Constants in Water: No dissociation constants of the test substance were determined because the spectra of the test substance at different pH values did not significantly differ from one another.
Partition Coefficient: $K_{ow} > 6$
Vapor Pressure: 6.0×10^{-3} Pa at 25°C

Remarks: The study results indicated that the test substance was composed of different components which, in some cases, yielded results different from each other.

Water solubility: It is suggested that the water solubility of each component of the test substance is different from the others.
Hydrolysis as a function of pH: The chemical structure of the test substance suggests that the reaction rate of each component is similar to each other.
Dissociation constants in water: The water solubility of the test substance is too low to find ionized and unionized forms of the test substance. The laboratory did not investigate its constants further by either the titration method or the conductometric method in the OECD Guideline because both methods are considered not suitable for substances with such a low solubility. However, the chemical structure of the test substance suggests that its dissociation potential is very low.
Parition coefficient: The test substance was detected as ten peaks or more by HPLC.
Vapor Pressure: The chemical structure of the test substance suggests that the vapor pressure of each component is different from the others.

DATA QUALITY

Water Solubility
Reliability: Klimisch ranking 2.
The report states a purity of the substance of "99% or more". Since the purity/identity of the test substance cannot be substantiated, the stated average solubility of the test substance is not defensible. As presented, the structural formula indicates that "purity" cannot be assigned as "99 % or more". Since the method of detection is not compound specific, and was not validated, it cannot be determined if the peaks detected in the sample chromatograms do indeed arise from the test substance.

Hydrolysis
Reliability: Klimisch ranking 2.
Without the purity/identity information on the test substance and compound specific analytical techniques, the experimentally determined rate constant cannot be substantiated.

Dissociation Constants in Water
Reliability: Klimisch ranking 1.
Potential for a compound of the given structure to ionize at any pH is low.

Octanol/Water Partition Coefficient

Reliability: Klimisch ranking 3.
Since the purity/identity of the test substance cannot be substantiated, the stated partition coefficient is not defensible. It was not established in this study what the retention time(s) of the test substance(s) is. The UV detector is not compound specific and it has not been definitively established that the >10 peaks assigned as belonging to the test compound are actually related to the test material. Furthermore, it has not been established that this method for determining the octanol/water partition coefficient applies to fluorochemical compounds similar in structure to the test material.

Vapor Pressure
Reliability: Klimisch ranking 3.
The purity/identity of the test substance cannot be substantiated. The chromatogram of the test substance has a distinctly different profile that that of the standard. It has not been established that under the conditions of the experiment the nitrogen passed over the substance has become saturated with gas phase test substance. The reference compound (benzoic acid) has a measured vapor pressure ~ 16 times that determined for the test substance. It would be appropriate to choose a reference compound with a vapor pressure closer to that determined for the test compound The lower molecular weight constituents will have a higher vapor pressure than the n=8 component.

REFERENCES
Study conducted at the request of Sumitomo 3M LTD by Mitsubishi Chemical Safety Institute Ltd., Yokohama, Japan

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

Last changed: 5/22/00

M.S.I. Report No. 4B223-4B227

Determination of Physico-chemical Properties of Sample D-1

Submitted to:

SUMITOMO 3M LTD.

Prepared by:

Mitsubishi Chemical Safety Institute Ltd.

February 14, 1996
(The original report was submitted on August 8, 1994)

Determination of Physico-chemical Properties of Sample D-1

(English version)

Study No.: 4B223-4B227
Study Title: Determination of Physico-chemical Properties of Sample D-1
Sponsor: SUMITOMO 3M LTD.

This test was conducted in Yokohama Laboratory of Mitsubishi Chemical Safety Institute Ltd., 1000 Kamoshida-cho, Aoba-ku, Yokohama 227, Japan.

This report is the English version of the original, which was written in Japanese. The undersigned hereby declare that this version faithfully reflects the original report to the best of our knowledge.

Translated by

Shiro Iwami Date: Feb. 14, 1996

Shiro IWAMI, M.Sc.
Environmental Science Division of the Yokohama Laboratory

Approved by

Jun Toriya Date: Feb. 14, 1996

Jun TORIYA, B.Sc.
Head of the Yokohama Laboratory,

Testing Facility

Study No.: 4B223-4B227
Study Title: Determination of Physico-chemical Properties of Sample D-1
Sponsor: SUMITOMO 3M LTD.

Testing Facility:

Yokohama Laboratory, Mitsubishi Chemical Safety Institute Ltd.† (M.S.I.),
1000 Kamoshida-cho, Aoba-ku, Yokohama 227, Japan (telephone: 045-963-3541)

† *The company's name was changed on October 1, 1994.*
(The former name: Mitsubishi-kasei Institute of Toxicological and Environmental Sciences)

Facility Management:

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Head of the Yokohama Laboratory

Study Management:

Tadayoshi SHIGEOKA, Ph.D. Sealed date: August 8, 1994
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Study Director:

Takemi NAKANOME, B.Sc. Sealed date: August 8, 1994
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Environmental Science Division of the Yokohama Laboratory

Contents

(59 pages in all)

Abstract

Study No.: 4B223-4B227

Study Title: Determination of Physico-chemical Properties of Sample D-1

Sponsor: SUMITOMO 3M LTD.

1 Test Substance

Name: Sample D-1

Chemical name: 2-[N-ethyl-N-perfluoroalkyl (C=1-8) sulfonylamino] ethyl acrylate

Structural formula: $C_nF_{2n+1}SO_2N(C_2H_5)CH_2CH_2OC(=O)CH=CH_2$
n=1-8 (n=8: approx. 78 %; n=1-7: approx. 21 %)

2 Water Solubility [Study No. 4B223]

2.1 Methods: No.105† "Water Solubility" (Flask Method)

2.2 Results: Water solubility of the test substance was 0.89 mg/L at 25°C.

3 Hydrolysis as a function of pH [Study No. 4B224]

3.1 Methods: No.111† "Hydrolysis as a function of pH", (testing at pH 4, 7, and 9 at 25°C)

3.2 Results: At 25°C reaction rate constants of the test substance at pH 4, 7, and 9 were 0.017, 0.020, and 0.046 day^{-1} with half life time of 42, 35, and 15 days, respectively.

4 Dissociation Constants in Water [Study No. 4B225]

4.1 Methods: No.112† "Dissociation Constants in Water" (Spectrophotometric Method)

3.2 Results: No dissociation constants of the test substance were determined because the spectra of the test substance at different pH did not significantly differ from one another. (But the chemical structure of the test substance suggests that its dissociation potential is very low.)

5 Partition Coefficient (1-octanol/water) by HPLC Method [Study No. 4B226]

5.1 Methods: No.117† "Partition Coefficient (n-octanol/water), High Performance Liquid Chromatography (HPLC) Method"

5.2 Results: The test substance was detected as ten peaks or more by HPLC. The log Pow value of the main component was more than 6.

6 Vapor Pressure [Study No. 4B227]

6.1 Methods: No.104† "Vapor Pressure Curve" (Gas Saturation Method)

6.2 Results: Vapor pressure of the test substance was 6.0×10^{-3} Pa at 25°C.

† *Number in Organization for Economic Cooperation and Development (OECD) Guidelines for Testing of Chemicals*

1 Test Substance

1.1 Identification

1)Name†: Sample D-1

Chemical name†: 2-[N-ethyl-N-perfluoroalkyl(C=1-8) sulfonylamino] ethyl acrylate

2)Structural formula†: $C_nF_{2n+1}SO_2N(C_2H_5)CH_2CH_2OC(=O)CH=CH_2$
n=1-8
(n=8: approx. 78 %; n=1-7: approx. 21 %)

3)Physico-chemical properties:

Solubility†:	water:	insoluble
	acetone:	50 % or more
	DMSO:	insoluble
Melting point†:	27-42°C	
Boiling point†:	approx. 150°C (1 mm Hg)	

† *provided by the sponsor*

1.2 Source

1)Batch‡: Lot No. 101
2)Supplier: Misao SHIBA, SUMITOMO 3M LTD.
3)Supplied quantity‡: approx. 100 g
4)Purity‡: 99 % or more
5)Appearance: amber like wax

‡ *provided by the sponsor*

2 Water Solubility [Study No. 4B223]

A solution is a homogeneous mixture of different substances in a solvent. The particle sizes of the dispersed substances are of the same magnitude as molecules and ions. Therefore, the smallest volumes which can be obtained from a solution are always of uniform composition.

Solubility in water is a significant parameter because

- the spatial and temporal movement (mobility) of a substance is largely determined by its solubility in water;
- water soluble substances gain ready access to humans and other living organisms;
- the knowledge of the solubility in water is a prerequisite for testing biological degradation and bio-accumulation in water and for other tests.

2.1 Materials and Methods

This study was conducted in accordance with the standard procedure "Water Solubility" (Flask Method) in the OECD† Guidelines for Testing of Chemicals No.105 (1981). This method is summarized as follows:

The test substance (Solids must be pulverized.) is dissolved in water at a temperature somewhat above the test temperature. When saturation is achieved the mixture is cooled and kept at the test temperature, stirring as long as necessary to reach equilibrium. Subsequently, mass concentration of the test substance in the aqueous solution, which must not contain any undissolved particles, is determined by a suitable analytical method.

† *Organization for Economic Cooperation and Development*

2.1.1 Reagents

n-hexane:	Wako Pure Chemical Industries, Ltd.,	guaranteed reagent
acetone:	Wako Pure Chemical Industries, Ltd.,	guaranteed reagent

2.1.2 Apparatus

incubator:	Taitec Corporation,	model M-100
centrifugal separator:	Hitachi Ltd.,	model SCT 15B
gas chromatograph(GC):	Shimadzu Corporation,	model GC-14A
integrator:	Shimadzu Corporation,	model C-R3A

2.1.3 Test procedure

Two hundred milligrams of the test substance was dissolved in 10 mL of acetone in an Erlenmeyer glass flask. The solvent was evaporated with stirring to deposit a thin layer of the test substance onto inner surface of the flask. The residual solvent was thoroughly removed by stream of nitrogen gas . Four hundred milliliters of distilled water was added to the flask, which was then shaken at 40±0.2°C over a 3-day period. A 50-mL aliquot of the water phase was sampled after 1, 2, and 3 days. The aliquot was subsequently shaken at 25±0.2°C for 1 day or more to reach equilibrium of dissolution of the test substance.

The operation by the above procedure was repeated once more.

2.1.4 Analytical methods

The concentration of the test substance was measured by gas chromatography (GC). Prior to GC analysis, each equilibrated aliquot was treated as follows:

The aliquot was centrifuged at 4000 rpm (3000 × g) at 25°C. Sodium chloride, 1.2 g, was dissolved in the supernatant, to which 4 mL of *n*-hexane was then added. They were shaken for 1 minutes and then centrifuged at 4000 rpm (3000 × g) at 25°C. The *n*-hexane phase (supernatant) was analyzed by GC under the following conditions:

GC conditions

column:	Shimadzu Corporation, wide bore column CBP20-W25-100, 0.53 mm i.d., 25 m in length
temperature:	column: 120°C; injector: 200°C; detector: 240°C
carrier:	nitrogen gas (flow rate: 20 mL/min)
detector:	electron capture detector (ECD)
injection volume:	3 µL

As informed by the sponsor, the components of the test substance differ in length of the perfluoroalkyl chain (see §1.1). In fact, the test substance was detected as four major peaks with retention time of 3.8, 4.5, 5.1, and 6.0 minutes by GC under the conditions described above. Therefore, the concentration of the test substance was based on total area of the four peaks.

2.1.5 Calibration curve

Standard solutions of the test substance were prepared to make concentrations of 0, 0.2, 1.0, and 5.0 mg/L in *n*-hexane. These standard solutions were analyzed by GC under the conditions described in §2.1.4. The total peak area of the four peaks

was calculated and plotted against the concentration of the test substance. The calibration curve yielded a straight line passing through the origin and its correlation coefficient was calculated to be 0.999 by the least square method described in Japanese Industrial Standards (JIS) Z 9041-1968.

[Figure 2.1 and Figure 2.2]

At measurement of the test substance concentration, the standard solution with 1.0 mg/L was analyzed. Concentration in each test sample was calculated from ratio of total peak area for the sample to that for the standard solution.

2.1.6 Recovery

An acetone solution of the test substance was prepared to make a concentration of 483 mg/L. Two milliliters of the solution was dissolved in 1000 mL of distilled water (final concentration of the test substance: 0.97 mg/L).

Sodium chloride, 1.2 g, was dissolved in 4 mL of the water solution, to which 4 mL of *n*-hexane was then added. They were shaken for 1 minutes and then centrifuged at 4000 rpm (3000 × g) at 25 °C. The concentration of the test substance in the *n*-hexane phase (supernatant) was measured by GC under the conditions described in §2.1.4 to evaluate recovery.

[Table 2.2 and Figure 2.3]

The recovery of the test substance was 94 %. The concentrations of the test substance reported below were corrected by this factor.

2.2 Results and Discussion

Average water solubility of the test substance was 0.89 mg/L at 25 °C.

[Table 2.1 and Figure 2.4.1 to Figure 2.4.3]

The component with GC retention time of 4.5 minutes was rather soluble in water compared with the others. Dissolution of this component at 40 °C reached equilibrium within 1 day.

It is suggested that water solubility of each component of the test substance differs from one another.

3 Hydrolysis as a Function of pH [Study No. 4B224]

The testing of substance for hydrolysis is relevant to their persistence. Hydrolysis is one of the most common reactions controlling abiotic degradation and is therefore one of the main degradation paths of substances in the environment.

A procedure to determine hydrolysis is important also in indicating whether other testing should be carried out on a parent compound or its hydrolysis product. It is the degradation products that are crucial.

Hydrolysis behavior needs to be examined at pH values normally found in the environment (pH 4-9) and under more acidic conditions (pH 1-2) for physiological purpose.

Surface-controlled reactions can sometimes predominate over bulk solution hydrolysis, especially in the soil environment. This may result in different degradation rates than would be predicted from this methods based upon rates in homogeneous solutions.

3.1 Materials and Methods

This study was conducted in accordance with the standard procedure "Hydrolysis as a function of pH" in the OECD Guidelines for Testing of Chemicals No.111 (1981).

In the environment, chemicals usually occur in dilute solution, which means that water is present in large excess, and therefore, the kinetics of hydrolysis are generally pseudo-first order at fixed pH and temperature.

The hydrolysis reaction may be influenced by acidic or basic species H_3O^+ (H^+) and OH^-, in which case it is referred to as specific acid or specific base catalysis.

The concentration of the test substance is determined as a function of time. The logarithms of the concentrations are plotted against time and the slope of the resulting straight line (assuming first-order or pseudo-first order behavior) gives the rate constant.

3.1.1 Reagents and water

acetone:	Wako Pure Chemical Industries, Ltd.,	guaranteed reagent
n-hexane:	Kishida Chemical Co., Ltd.	guaranteed reagent
sodium chloride:	Junsei Chemical Co., Ltd.,	guaranteed reagent
sodium hydroxide:	Junsei Chemical Co., Ltd.,	guaranteed reagent
monopotassium phosphate:	Wako Pure Chemical Industries, Ltd.,	guaranteed reagent
monopotassium citrate:	Nacalai Tesque Inc.,	extra pure reagent
boric acid:	Wako Pure Chemical Industries, Ltd.,	guaranteed reagent
potassium chloride:	Junsei Chemical Co., Ltd.	guaranteed reagent
water:	deionized water purified by Milli-Q®	

3.1.2 Apparatus

incubator:	Taitec Corporation,	model M-100
pH meter:	Toa Electronics Ltd.,	model HM-50S
gas chromatograph(GC):	Shimadzu Corporation,	model GC-14A
	[equipped with an electron capture detector (ECD)]	
integrator:	Shimadzu Corporation,	model C-R3A

3.1.3 Test procedure

3.1.3.1 Preparation of buffer solutions

Buffer solutions were prepared by the following two methods described in the annex of the Guideline No. 111:

- Buffer mixtures of Clark and Lubs
- Citrate buffer of Kolthoff and Vleeschhouwer

Each buffer solution of pH values 4, 7, and 9 was prepared to be 1000 mL using the following reagents:

1) pH 4: 0.1 M monopotassium citrate and 0.1 *N* sodium hydroxide
2) pH 7: 0.1 M monopotassium phosphate and 0.1 *N* sodium hydroxide
3) pH 9: 0.1 M potassium chloride, 0.1 M boric acid and 0.1 *N* sodium hydroxide

The buffer solutions obtained were passed through Millipore® filter of pore size 0.25 μm. The pH values of the filtrates were determined to be 4.05, 7.02, and 9.05 for nominal values of 4, 7, and 9, respectively.

3.1.3.2 Preparation of stock solution of the test substance

The test substance, 80 mg, was dissolved and diluted with acetone to prepare a stock solution with 40 mg/L.

3.1.3.3 Preliminary test (at 50 °C)

Four milliliters of the stock solution (prepared in §3.1.3.2) was added to 400 mL each of the three buffer solutions to prepare test solutions of pH values 4, 7, and 9. The solutions thus prepared were tested under the following conditions:

I) Conditions

pH:	4, 7, and 9
concentration:	0.40 mg/L
temperature:	50.0±0.1 °C
testing period:	5 days with continuous shaking
test volume:	400 mL (containing 1 % acetone)
test vessel:	Erlenmeyer glass flask

II) Analytical methods

The concentration of the test substance was measured by GC. Prior to GC analysis, the test solutions were treated by the following procedure:

Sodium chloride, 128 g, was dissolved in each of the three 400-mL solutions, from which the test substance was then extracted with 100 mL of *n*-hexane. The extraction was repeated twice more. The three extracts were combined and concentrated to 50 mL, which was then transferred into a 200-mL glass volumetric flask and filled to 200 mL with *n*-hexane. The hexane solution was analyzed by GC under the following conditions:

GC conditions

column:	Shimadzu Corporation, wide bore column CBP20-W25-100, 0.53 mm i.d., 25 m in length
temperature:	column: 120 °C; injector: 200 °C; detector: 240 °C
carrier:	nitrogen gas (flow rate: 20 mL/min)
detector:	ECD
injection volume:	3 µL

As described in §2.1.4, the concentration of the test substance was based on total area of the four peaks detected by GC (retention time: 3.6, 4.3, 4.9, and 5.8 minutes).

III) Recovery

Four milliliters of the stock solution (prepared in §3.1.3.2) was added to 400 mL of water in an Erlenmeyer glass flask (final concentration: 0.4 mg/L). Sodium chloride, 128 g, was dissolved in this solution, from which the test substance was then extracted with 100 mL of *n*-hexane. The extraction was repeated twice more.

The three extracts were combined and concentrated to 50 mL, which was then transferred into a 200-mL glass volumetric flask and filled to 200 mL with *n*-hexane. The concentration of the test substance in the final solution was measured by GC under the conditions described in §3.1.3.3.II.

Recovery was determined to be 101 %. The concentrations of the test substance reported for this preliminary test were corrected by this factor.

[Table 3.6 and Figure 3.1]

3.1.3.4 Further investigation (at 25 °C)

Four milliliters of the stock solution (prepared in §3.1.3.2) was added to 400 mL each of the three buffer solutions to prepare test solutions of pH values 4, 7, and 9. Six 5-mL aliquots from each of the test solutions were transferred into glass tubes. These solutions were examined as a function of time under the following conditions:

I) Conditions

pH:	4, 7, and 9
concentration:	0.40 mg/L
temperature:	25.0±0.2 °C
testing period:	33 days with no shaking
test volume:	5 mL (containing 1 % acetone)
test vessel:	glass tube

II) Analytical methods

The concentration of the test substance was measured by GC. Prior to GC analysis, the test solutions were treated by the following procedure:

Sodium chloride, 1.6 g, was dissolved in the 5-mL aliquot treated in the tube. Two milliliters of *n*-hexane was added to this solution. They were shaken for 1 minutes and then centrifuged at 3000 rpm. The *n*-hexane phase (supernatant) was analyzed by GC as described in §3.1.3.3.

As described in §2.1.4, the concentration of the test substance was based on total area of the four peaks detected by GC (retention time: 3.6, 4.3, 4.9, and 5.8 minutes).

III) Recovery

Four milliliters of the stock solution (prepared in §3.1.3.2) was added to 400 mL of water in an Erlenmeyer glass flask (the final concentration: 0.4 mg/L). Sodium

chloride, 1.6 g, was dissolved in a 5-mL sample of this solution, to which two milliliters of *n*-hexane was then added. They were shaken for 1 minutes and then centrifuged at 3000 rpm. The *n*-hexane phase (supernatant) was analyzed by GC under the conditions described in §3.1.3.3.

Recovery was determined to be 95 %. The concentrations of the test substance reported for this further investigation were corrected by this factor.

[Table 3.7 and Figure 3.3]

3.1.3.5 Calibration curve

The calibration curve prepared in §2.1.5 (Determination of Water Solubility) was used.

[Figure 2.1 and Figure 2.2]

At measurement of the test substance concentration, the standard solution of the test substance with 1.0 mg/L was analyzed. Concentration in each test sample was calculated from ratio of total peak area for the sample to that for the standard solution.

3.1.3.6 Calculations

Residual percent of the test substance was calculated as follows:

$$r_t = 100\,(C_t / C_{t_0})$$

where

r_t: residual percent of the test substance after "t" days
C_t: concentration of the test substance after "t" days (mg/L)
C_{t_0}: initial concentration of the test substance (0.4 mg/L)

Assuming that the logarithms of the residual concentrations are first order as a function of time, their reaction rate constants were calculated as follows:

$$k = t^{-1}\ ln\,(100/r_t)$$

where

k: reaction rate constant of the test substance (day^{-1})

Using the average of these rate constants, half life time of the test substance was calculated as follows:

$$t_{1/2} = k^{-1}\ ln\ 2$$

where

$t_{1/2}$: half life time of the test substance (days)

3.2 Results and Discussion

1) Preliminary test

After 5 days at 50°C the residual percents of the test substance at pH 4, 7, and 9 were 76, 76, and 52, respectively.

[Table 3.5 and Figure 3.2]

On the basis of the results, it is suggested that the test substance is transformed at least by 24 %.

Relating to the preliminary test result, there is the following description in the OECD Guideline:

If less than 10 per cent of the reaction is observed after 5 days ($t_{1/2}$ > 1 year), the chemical is considered hydrolytically stable and no additional testing is required.

Therefore, we decided to perform the further investigation described below.

2) Further investigation

At 25°C the reaction rate constants of the test substance at pH 4, 7, and 9 were 0.017, 0.020, and 0.046 day^{-1} with half life time of 42, 35, and 15 days, respectively.

[Table 3.1 to Table 3.4 and Figure 3.4]

The chemical structure of the test substance (shown in §2.2) suggests that the reaction rate of each component is similar to each other.

4 Dissociation Constants in Water [Study No. 4B225]

The dissociation of a chemical in water is of importance in assessing its impact upon the environment. It governs the form of the substance which in turn determines its behavior and transport. It may affect the adsorption of the chemical on soils and sediments and adsorption into biological cells.

4.1 Materials and Methods

This study was conducted in accordance with the standard procedure "Dissociation Constants in Water" (Spectrophotometric Method) in the OECD Guidelines for Testing of Chemicals No.112 (1981). General summary of this method is as follows:

A wavelength is found where the ionized and unionized forms of the compound have appreciably different extinction coefficients. The UV-visible absorption spectrum is obtained from solutions of constant concentration under a pH condition where the substance is essentially unionized and fully ionized and at several intermediate pH's. This may be done, either by adding increments of concentrated acid (base) to a relatively large volume of a solution of the compound in a multicomponent buffer, initially at high (low) pH, or by adding equal volumes of a stock solution of the compound in e.g. water, methanol, to constant volumes of various buffer solutions covering the desired pH range. From the pH and absorbance values at the chosen wavelength, a sufficient number of values for the pKa is calculated using data from at least 5 pH's where the compound is at least 10 per cent and less than 90 per cent ionized.

4.1.1 Reagents and water

methanol:	Junsei Chemical Co., Ltd., guaranteed reagent
0.1 *N* hydrochloric acid:	Kishida Chemical Co., Ltd., reagent for titration (diluted to 0.01 *N* with water prior to use)
0.1 *N* sodium hydroxide:	Kishida Chemical Co., Ltd., reagent for titration (diluted to 0.01 *N* with water prior to use)
water:	Deionized water was distilled.

4.1.2 Apparatus

UV-visible spectrophotometer: Shimadzu Corporation, Model UV-260

4.1.3 Test procedure

4.1.3.1 Preparation of stock solution of the test substance

A stock solution of the test substance with 80 mg/L in methanol was prepared using 20.0 mg of the substance.

4.1.3.2 Preparation of test solutions

For measurement of UV-visible spectra of the test substance, three solutions of the test substance were prepared as follows:

1) Acid solution of the test substance

Two milliliters of the stock solution (prepared in §4.1.3.1.) and 2.4 mL of 0.01 *N* hydrochloric acid were transferred into a 200-mL glass volumetric flask, which was then filled to 200 mL with water. (final concentration of the test substance: 0.8 mg/L)

2) Alkaline solution of the test substance

Two milliliters of the stock solution and 2.4 mL of 0.01 *N* sodium hydroxide were transferred into a 200-mL glass volumetric flask, which was then filled to 200 mL with water. (final concentration of the test substance: 0.8 mg/L)

3) Neutral solution of the test substance

Two milliliters of the stock solution was transferred into a 200-mL glass volumetric flask, which was then filled to 200 mL with water. (final concentration of the test substance: 0.8 mg/L)

4.1.3.3 Measurement of UV-visible spectrum

The UV-visible spectra of the test substance in the solutions of three different pH values were measured with the apparatus shown in §4.1.2.

4.2 Results and Discussion

No dissociation constants of the test substance were determined because the spectra of the test substance at different pH did not significantly differ from one another.

[Figure 4.1]

The water solubility of the test substance is too low to find an ionized and unionized forms of the test substance. We did not investigate its constants further by either "Titration Method" or "Conductometric Method" in the OECD Guideline because the both methods are considered not suitable for the substance having such a low solubility (0.89 mg/L, reported in §2.2).

But the chemical structure of the test substance (shown in §1.1.2) suggests that its dissociation potential is very low.

5 Partition Coefficient (1-octanol/water) by HPLC Method [Study No. 4B226]

The partition coefficient (P) is defined as the ratio of the equilibrium concentrations of a dissolved substance in a two-phase system consisting of two largely immiscible solvents. In case of 1-octanol and water,

$$P_{ow} = C_o / C_w$$

where

P_{ow}: 1-octanol/water partition coefficient
C_o: concentration in 1-octanol phase
C_w: concentration in water phase

The partition coefficient being the quotient of two concentrations is usually given in the form of its logarithm to base ten (log Pow).

Pow is a key parameter in studies of the environmental fate of chemical substances. A highly-significant relationship between the Pow of substances and their bioaccumulation in fish has been shown. It has also been shown that Pow is a useful parameter in the prediction of adsorption on soil and sediments and for establishing quantitative structure-activity relationships for a wide range of biological effects.

5.1 Materials and Methods

This study was conducted in accordance with the standard procedure "Partition Coefficient (n-octanol/water), High Performance Liquid Chromatography (HPLC) Method" in the OECD Guidelines for Testing of Chemicals No.117(1989). Principle of this method is as follows:

HPLC is performed on analytical columns packed with a commercially available solid phase containing long hydrocarbon chains (e.g. C_8, C_{18}) chemically bound onto silica.

Chemicals injected onto such a column move along it by partitioning between the mobile solvent phase and the hydrocarbon stationary phase. The chemicals are retained in proportion to their hydrocarbon-water partition coefficient, with water soluble chemicals eluted first and oil-soluble chemicals last. This enables the relationship between the retention time on a reverse-phase column and the 1-octanol/water partition coefficient to be established.

5.1.1 Reference compounds

As reference compounds whose Pow values are well known, we selected five substances: methyl benzoate, bromobenzene, diphenyl, dibenzyl, and DDT. In addition, thiourea was used for determination of the dead time. A mixture of the six substances was prepared in acetonitrile.

5.1.2 Correlation between retention time and Pow

The mixture of the reference compounds (shown in §5.1.1) was analyzed by high performance liquid chromatography (HPLC) under the conditions described below:

HPLC conditions

column:	GL Sciences Inc., Inertsil ODS-2 4.6 mm i.d., 250 mm in length (C_{18} chemically bound onto silica)
mobile phase:	acetonitrile/water = 75/25 (v/v)
flow rate:	1.0 mL/min.
wavelength:	210 nm
temperature:	25 °C

The HPLC retention time of the reference compounds were corrected as follows:

$$Rt = Rt' - Rt_0$$

where

Rt:	corrected retention time of the compound (minutes)
Rt':	retention time of the compound (minutes)
Rt_0:	retention time of thiourea (minutes)

Correlation equation between the corrected retention time of the five compounds and their corresponding Pow was computed by the least square method, resulting in the following equation:

$$log\ P_{ow} = 5.274\ log\ Rt - 0.0219 \quad (r = 0.973)$$

[Figure 5.1 and Figure 5.2]

5.1.3 Retention time of the test substance

A mixture of the test substance and thiourea was prepared in acetonitrile. The mixture was analyzed by HPLC under the conditions described in §5.1.2. Based on the chromatogram, retention time (Rt', minutes) were determined for components of the test substance.

5.2 Results and Discussion

The test substance was detected as ten peaks or more by HPLC.

The corrected retention time (Rt) of the main component was determined to be 29.245 minutes, while that of DDT (Log Pow is reported to be 6.20.) was 13.263 minutes.

Based on the correlation equation shown in §5.1.2, the log Pow value of the main component was more than 6.

[Figure 5.2]

6 Vapor pressure [Study No. 4B227]

The environmental relevance of vapor pressure is accounted for by the following reasons:

- The vapor pressure gives an indication of the probability of the phase transitions, liquid/gas and solid/gas.
- The vapor pressure, together with the solubility in water, is the major auxiliary variable for calculating the volatility of a substance from an aqueous solution.
- Vapor pressure is thus a significant factor for predicting atmospheric concentrations.
- The vapor pressure of a substance can furthermore be useful as a basis for deciding whether or not a photochemically induced degradation study (in the homogeneous gas phase or in an absorbed phase) is necessary.

6.1 Materials and Methods

This study was conducted in accordance with the standard procedure "Vapor Pressure Curve" (Gas Saturation Method) in the OECD Guidelines for Testing of Chemicals No.104 (1981). This method is summarized as follows:

A stream of inert carrier gas (nitrogen gas) is passed over the substance in such a way that it becomes saturated with vapor of the substance and the vapor is then collected in a trap adsorbent. Measurement of the amount of material transported by a known amount of carrier gas is used to calculate the vapor pressure at a given temperature.

6.1.1 Reagents

acetonitrile:	Wako Pure Chemical Industries, Ltd.,	reagent for HPLC
adsorbent:	GL Sciences Inc.,	Tenax GC®, 60-80 mesh

6.1.2 Apparatus

saturator column:	Sibata Scientific Technology Ltd., 12 mm i.d., 150 mm in length	
adsorbent column:	Sibata Scientific Technology Ltd., 12 mm i.d., 150 mm in length	
glass bead:	Iuchi Seieido Co., Ltd.,	1 mm in diameter
flow meter:	Shinagawa Corporation,	model NWK-IC
gas chromatograph:	Shimadzu Corporation,	model GC-14A
data processor:	Shimadzu Corporation,	model C-R3A

6.1.3 Vapor pressure measuring apparatus

For measurement of vapor pressure of the test substance, an apparatus shown in Figure 6.5 was assembled and set in a room air-conditioned at 25±1°C.

[Figure 6.5]

The glass beads coated with the test substance were packed into the saturator column up to 7 cm in length. Nitrogen gas, kept at a constant flow rate by the pressure controller and the flow gauge, was introduced to this column.

The nitrogen gas saturated with vapor of the test substance was delivered to the Tenax GC®-packed column to trap the test substance. Total amount of the nitrogen gas which passed through this trap column was measured by the flow meter.

Preparation of saturator column

1) Ten milligrams of the test substance was dissolved in 10 mL of acetonitrile in a 100-mL round bottom glass flask.

2) Eight milliliters of glass beads was added into the flask. The solvent was removed by rotary evaporation. The residual solvent was thoroughly eliminated with a vacuum pump at room temperature. (The test substance was thus coated onto the glass beads.)

3) The glass beads coated with the test substance was packed into the saturator column up to 7 cm in length. Both open sides of the column were held by quartz glass wool, which was then incorporated in the vapor pressure measuring apparatus.

nitrogen gas

1) Nitrogen gas was delivered into the vapor pressure measuring apparatus for 70 hours. Total volume of the carrier gas was measured with the flow meter and determined to be 1.017 m^3. (average flow rate: 242 mL/min)

2) The total volume of the gas was corrected as follows:

$$V = Vr\,[(273/T)(760-P) / 760]^{1/2}$$

where

V: total gas volume corrected (m^3)
Vr: amount of the gas measured by the flow meter (1.017 m^3)
T: temperature of the nitrogen gas (298 °K)
P: pressure difference between at the flow meter and at the absorber-packed column (0.4 mm Hg)

The total volume of the carrier was thus corrected by factors of the temperature and pressure difference, resulting in 0.973 m^3.

Adsorbent column

1) Eight milliliters of the adsorbent (Tenax GC®) was packed into the absorbent column. Both open sides of the column were retained with quartz glass wool. The column thus prepared was connected to the saturator column.

2) The test substance trapped by the Tenax GC® was desorbed by the procedure described in §6.1.4.

6.1.4 Analytical methods

The test substance trapped by the adsorbent was desorbed as follows:

1) The adsorbent packed in the column was transferred into a glass filter. The inside of the vacant column was rinsed with 20 mL of acetonitrile, which was then transferred in the glass filter. The rinse of the Tenax GC® was repeated twice more with 20 mL and 10 mL each of acetonitrile.

2) The three filtrates were combined and transferred into a 50-mL volumetric glass flask, which was then filled to 50 mL with acetonitrile. An aliquot of the solution was diluted 50 fold in volume.

3) The concentration of the test substance in the final solution was measured by GC under the following conditions:

GC conditions

column:	Shimadzu Corporation, wide bore column CBP20-W25-100, 0.53 mm i.d., 25 m in length
temperature:	column 120 °C, injector 200 °C, detector 240 °C
carrier:	nitrogen gas (flow rate: 20 mL/min)
detector:	ECD
injection volume:	3 μL

Calibration curve

Standard solutions of the test substance were prepared to make concentrations of 0, 0.25, 0.5, and 1.0 mg/L in acetonitrile. They were analyzed by GC under the above conditions. A calibration curve prepared by the method described in §2.1.5 generated a straight line which crosses the origin and its correlation coefficient was calculated to be 0.999.

[Figure 6.1 and Figure 6.2]

At measurement of the test substance concentration, the standard solution with 1.0 mg/L was analyzed. Concentration in each test sample was calculated from ratio of total peak area for the sample to that for the standard solution.

Desorption efficiency (Recovery)

Desorption efficiency of the test substance from Tenax GC® was determined according to the following procedure:

1) Twenty five milliliters of the solution of the test substance with 1 mg/L in acetonitrile (mass of the test substance: 25 μg) was added to 4 mL of the adsorbent in a 50-mL round bottom glass flask. The solvent was removed by rotary evaporation to adsorb the test substance onto the Tenax GC®.

2) The test substance adsorbed on the Tenax GC® was recovered by the desorption procedure described above (except that the substance was extracted with 10, 10, and 5 mL of acetonitrile and the filtrates were transferred into 25-mL volumetric glass flask, which was then filled to 25 mL). The concentration of the test substance in the final solution was measured by GC to evaluate desorption efficiency.

The desorption efficiency was determined to be 97 %. For measurement of vapor pressure, the concentration of the test substance was corrected by this factor.

[Table 6.3 and Figure 6.3]

6.1.5 Calculation of vapor pressure

Vapor pressure of the test substance was calculated as follows:

$$P = (W/M)(RT/V)$$

where

P: vapor pressure (Pa)
W: amount of the test substance trapped (g)
M: molecular weight of the test substance (g $mole^{-1}$)
R: gas constant (8.31 Pa m^3 $mole^{-1}$ K^{-1})
V: total volume of the carrier gas corrected (m^3)
T: temperature (°K)

6.2 Results and Discussion

Vapor pressure of the test substance was 6.0×10^{-3} Pa.

[Table 6.1 to Table 6.2, and Figure 6.4]

But the chemical structure of the test substance (shown in §1.1.2) suggests that vapor pressure of each component is different from one another.

Vapor pressure of benzoic acid (a reference substance in the OECD Guideline) was 0.10 Pa (24 °C) under the conditions employed in this study.

Table 2.1 Calculation of concentration of the test substance dissolved in water

Measurement No. 1

time at 40 °C, days		1	2	3
conc. in Std., mg/L	A	1.01	1.01	1.01
peak area, μV·sec				
Std.	B	2589644	2717449	2732276
test solution	C	2030738	2414771	2230296
concentration factor	D	1	1	1
conc. of the substance, mg/L	E	0.84	0.95	0.88

Calculation equation

$$E = A(C/B)(1/D)(1/0.94)$$

Measurement No. 2

time at 40 °C, days		1	2	3
conc. in Std., mg/L	A	1.01	1.01	1.01
peak area, μV·sec				
Std.	B	2589644	2717449	2732276
test solution	C	2115067	2319263	2293546
concentration factor	D	1	1	1
conc. of the substance, mg/L	E	0.88	0.92	0.90

Calculation equation

$$E = A(C/B)(1/D)(1/0.94)$$

average water solubility: 0.89mg/l

Table 2.2 Calculation of recovery

conc. of the substance added, mg/L	A	0.97
conc. in Std., mg/L	B	1.01
peak area, $\mu V \cdot sec$		
Std.	C	2723221
test solution	D	2456907
concentration factor	E	1
conc. of the substance recovered, mg/L		0.91
recovery (%)	F	94

Calculation equation

$$F=B(D/C)(1/E)(100/A)$$

Figure 2.1 Calibration curve of the test substance

Curve Fitting [Least Square Method]

Input Data

No.	Concentration X(mg/l)	Peak Area Y(μV·sec)
1	0	0
2	0.2016	550784
3	1.008	2758732
4	5.04	13119948

$Y = 5.0483 \times 10^4 + 2.5966 \times 10^6 \times X^1$

$r = 0.99995$

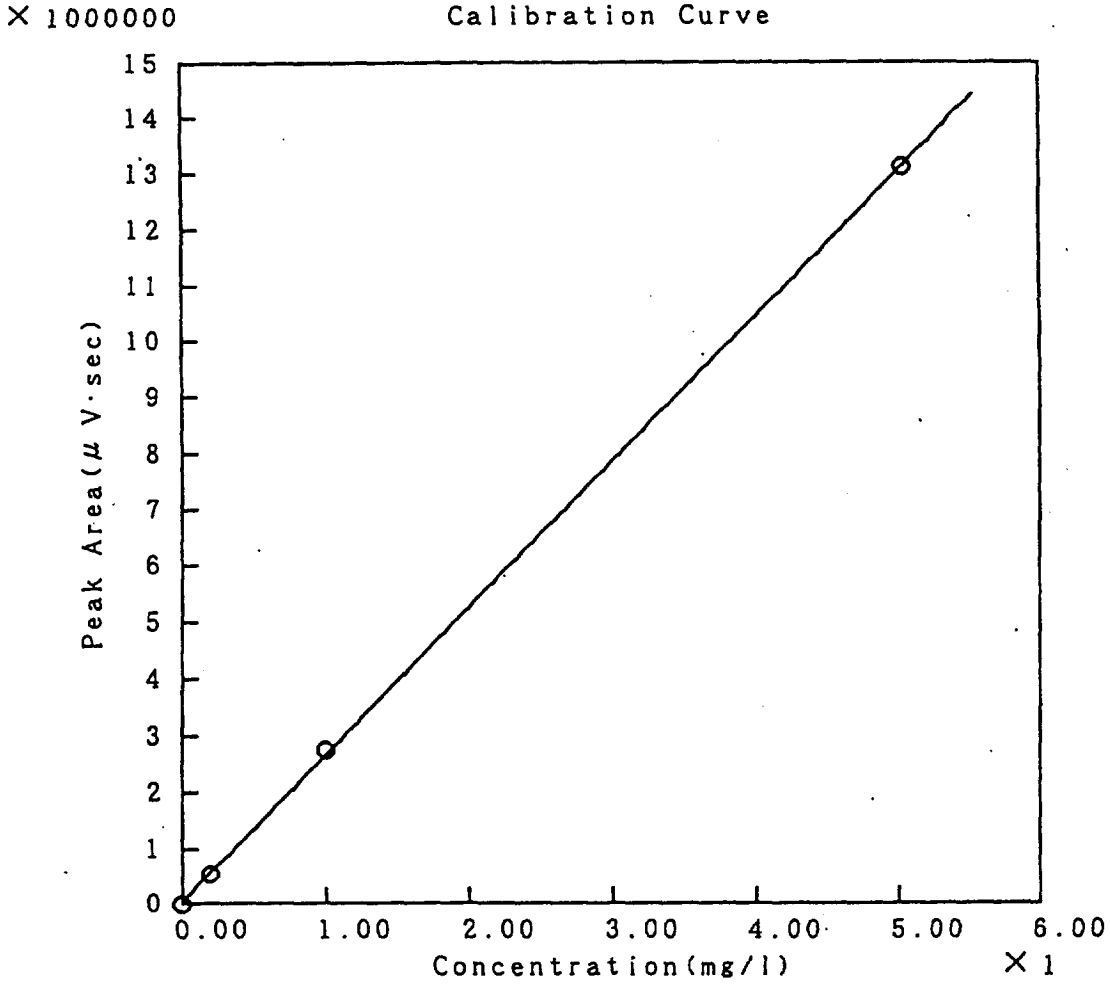
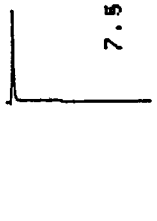


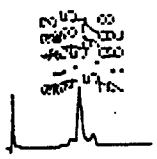
Figure 2.2 GC chromatograms of the test substance
--- for calibration curve ---

Std. 0mg/l



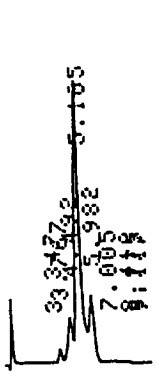
WARNING NO PEAK

Std. 0.202mg/l



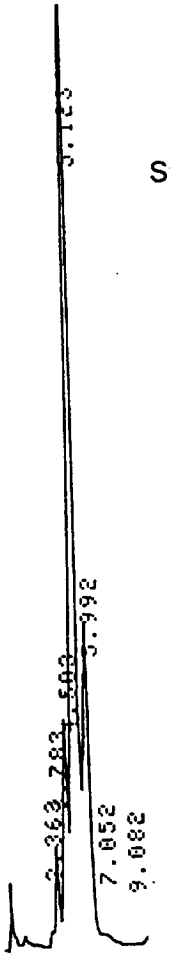
PKNO	TIME	AREA	MK
1	3.76	15993	V
2	4.485	64568	V
3	5.1	373677	V
4	5.97	96547	V
TOTAL		550784	

Std. 1.008mg/l



PKNO	TIME	AREA	MK
1	3.047	11073	
2	3.767	80396	V
3	4.492	317962	V
4	5.105	1821019	V
5	5.982	539354	SV
TOTAL		2769805	

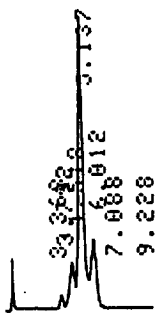
Std. 5.04mg/l



PKNO	TIME	AREA	MK	IDNO	CONC
1	3.063	53734			0.4073
2	3.783	467507	V		3.5436
3	4.503	1545826	V		11.7168
4	5.123	8586993	V		65.0866
5	5.992	2519622	SV		19.0979
6	7.052	19504	T		0.1478
TOTAL		13193186			100

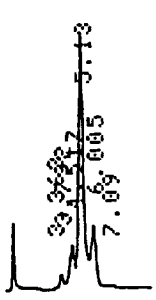
Figure 2.3 GC chromatograms of the test substance
. --- for recovery---

Std. 1.008mg/l



PKNO	TIME	AREA	MK
1	3.368	11920	
2	3.792	74532	V
3	4.518	308316	V
4	5.137	1812582	V
5	6.012	527793	SV
	TOTAL	2735141	

test solution



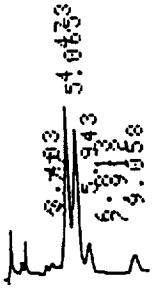
PKNO	TIME	AREA	MK
1	3.368	11050	
2	3.792	69258	V
3	4.517	280909	V
4	5.13	1628500	V
5	6.005	478240	SV
	TOTAL	2467957	

Figure 2.4.1 GC chromatograms of the test substance
--- for measurement of concentration in water
(after 1 day at 40°C)---

Std. 1.008mg/l

PKNO	TIME	AREA	MK
1	3.005	11000	
2	3.757	67922	V
3	4.49	293103	V
4	5.087	1732292	V
5	5.955	496327	SV
	TOTAL	2600681	

test solution
n=1



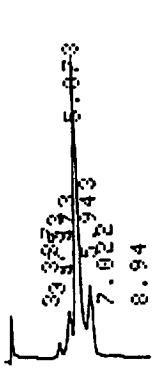
PKNO	TIME	AREA	MK
1	3.403	4725?	
2	3.77	24910	V
3	4.473	927788	V
4	5.065	879701	V
5	5.943	198339	V
6	7.912	21530	
7	9.058	150040	
	TOTAL	2253382	

test solution
n=2

PKNO	TIME	AREA	MK
1	3.268	39743	
2	3.763	16505	V
3	4.477	1039350	V
4	5.077	859751	V
5	5.953	199461	V
6	7.947	17090	
7	9.05	131063	
	TOTAL	2303814	

Figure 2.4.2 GC chromatograms of the test substance
--- for measurement of concentration in water
(after 2 days at 40°C)---

Std. 1.008mg/l

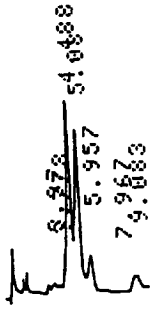


PKNO	TIME	AREA	MK
1	3.037	11413	
2	3.753	74675	V
3	4.473	310527	V
4	5.073	1802535	V
5	5.943	529712	SV
	TOTAL	2728862	

test solution
n=1

PKNO	TIME	AREA	MK
1	3.41	51435	
2	3.77	35983	V
3	4.475	1004015	V
4	5.07	1119855	V
5	5.948	254918	V
6	7.998	10107	
7	9.05	120671	
	TOTAL	2613016	

test solution
n=2



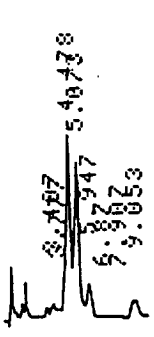
PKNO	TIME	AREA	MK
1	3.37	10013	
2	3.778	23320	V
3	4.488	1053734	V
4	5.08	1002276	V
5	5.957	239933	V
6	7.967	20401	
7	9.003	174404	
	TOTAL	2523247	

Figure 2.4.3 GC chromatograms of the test substance
--- for measurement of concentration in water
(after 3 days at 40°C)---

Std. 1.008mg/l

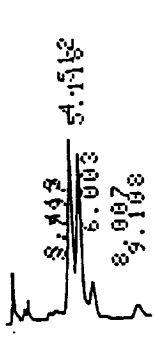
PKNO	TIME	AREA	MK
1	3.32	11824	
2	3.737	76145	V
3	4.447	311144	V
4	5.053	1819770	V
5	5.917	525217	SV
	TOTAL	2744109	

test solution
n=1



PKNO	TIME	AREA	MK
1	3.407	52904	
2	3.777	29460	V
3	4.478	1015002	V
4	5.073	965788	V
5	5.947	220046	V
6	6.87	17402	
7	7.907	29211	V
8	9.053	166957	
	TOTAL	2497030	

test solution
n=2



PKNO	TIME	AREA	MK
1	3.413	38661	
2	3.797	24657	V
3	4.512	1008133	V
4	5.118	1020090	V
5	6.003	240666	V
6	8.007	14323	
7	9.108	116910	
	TOTAL	2463445	

Table 3 Calculations of reaction rate constant and half life time of the test substance (Table 3.1-Table 3.3)

Table 3.1 (pH 4 at 25°C)

time, days (t)	residual percent of the substance, mg/L (C)	rate constant, day^{-1} (k)
5	89.7	0.022
8	87.3	0.017
15	77.0	0.017
19	76.8	0.014
26	68.3	0.015
33	62.1	0.014

average rate constant $0.017day^{-1}$
half life time ($t_{1/2}$) 42 days

calculation equations

$$k=1/t \times \ln 100/c$$
$$t_{1/2}=1/k \times \ln 2$$

Table 3.2 (pH 7 at 25°C)

time, days (t)	residual percent of the substance, mg/L (C)	rate constant, day^{-1} (k)
5	88.6	0.024
8	86.9	0.018
15	74.1	0.020
19	69.5	0.019
26	59.9	0.020
33	53.8	0.019

average rate constant $0.020day^{-1}$
$t_{1/2}$ 35 days

Table 3. (pH 9 at 25°C)

time, days (t)	residual percent of the substance, mg/L (C)	rate constant, day^{-1} (k)
2	92.4	0.040
5	78.4	0.049
8	69.0	0.046
12	57.1	0.047
15	50.2	0.046
19	37.7	0.051

average rate constant $0.046day^{-1}$
$t_{1/2}$ 15 days

Table 3.4 Calculations of residual concentration of the test substance
--- for *Further investigation* (at 25.0 ± 0.2°C) ---

time, days	pH	peak area, μV•sec: Std B	peak area, μV•sec: test solution C	conc. in test soln.	residual percent D
2	9	2889193	2523765	0.371	92.4
5	4	2951466	2502799	0.361	89.7
	7	2951466	2473261	0.356	88.6
	9	2951466	2186759	0.315	78.4
8	4	2968192	2449526	0.351	87.3
	7	2968192	2439536	0.350	86.9
	9	2968192	1936885	0.278	69.0
12	9	2939623	1585436	0.229	57.1
15	4	2871668	2089210	0.309	77.0
	7	2871668	2011015	0.298	74.1
	9	2871668	1361482	0.202	50.2
19	4	2871088	2084603	0.309	76.8
	7	2871088	1886031	0.279	69.5
	9	2871088	1022942	0.152	37.7
26	4	2962788	1912723	0.275	68.3
	7	2962788	1676378	0.241	59.9
33	4	2928913	1718699	0.250	62.1
	7	2928913	1490039	0.216	53.8

concentration of the standard solution (A): 1.01 mg/L
concentration factor (E): 2.5
initial concentration of the test substance(F): 0.402 mg/L
recovery(G): 95 %

calculation equation: $D = A(C/D) \cdot E^{-1} \cdot F^{-1}(100/G) \cdot 100$

Table 3.5 Calculations of residual concentration of the test substance
--- for *Preliminary test* (after 5 days at 50.0±0.1°C) ---

		pH 4	pH 7	pH 9
initial concentration of the test substance, mg/L	A	0.402	0.402	0.402
after 5 days				
conc. in Std., mg/L	B	1.00	1.00	1.00
peak area, μV*sec				
Std.	C	3022269	3022269	3022269
test solution	D	1867539	I876183	1264465
concentration factor	E	2	2	2
conc. in the test solution		0.306	0.307	0.207
residual percent of the test substance	F	76	76	52

calculation equation

$$F = B(D/C)(1/E)(1/A)(1/1.01)100$$

Table 3.6 Calculation of recovery
--- for *Preliminary test* ---

conc. of the test substance added, mg/L	A	0.402
conc. in Std., mg/L	B	1.00
peak area, μV*sec		
Std.	C	2977220
test solution	D	2420272
concentration factor	E	2
conc. of the test substance recovered, mg/L		0.406
recovery, %	F	101

calculation equation

$$F = B(D/C)(1/E)(1/A)100$$

Table 3.7 Calculation of recovery
--- for *Further investigation* ---

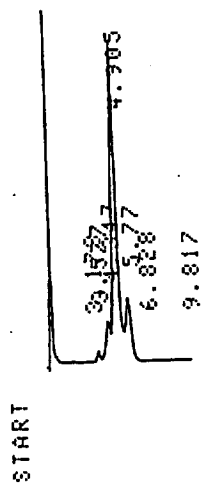
conc. of the test substance added, mg/L	A	0.402
conc. in Std., mg/L	B	1.01
peak area, μV*sec		
Std.	C	2782217
test solution	D	2639636
concentration factor	E	2.5
conc. of the test substance recovered, mg/L		0.383
recovery, %	F	95

calculation equation

$$F = B(D/C)(1/E)(1/A)100$$

Figure 3.1 GC chromatograms of the test substance
--- for recovery in *Preliminary test* ---

Std. (1.0mg/ℓ)



PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.173	10447			0.3497	
2	3.577	68147	V		2.2809	
3	4.347	276040	V		9.2393	
4	4.905	2100565	V		70.3078	
5	5.77	532469	SV		17.8222	
	TOTAL	2987667			100	

test solution

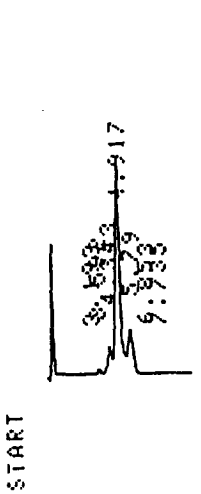
PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.592	55151	V		2.2787	
2	4.342	229949	V		9.5009	
3	4.9	1717273	V		70.9537	
4	5.773	417900	SV		17.2667	
	TOTAL	2420272			100	

Figure 3.2 GC chromatograms of the test substance
--- for measurement of concentration in water in *Preliminary test* ---

Std. (1.0mg/ℓ)

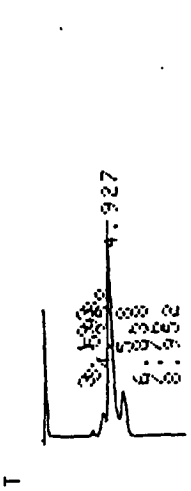
PKNO	TIME	AREA	MK
1	3.100	12332	
2	3.595	69736	V
3	4.342	302001	V
4	4.913	2118252	V
5	5.788	532279	SV
TOTAL		3034501	

PH 4



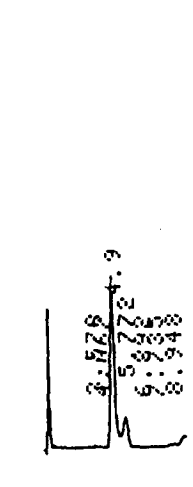
PKNO	TIME	AREA	MK
1	3.592	36826	V
2	4.343	186550	V
3	4.917	1308749	V
4	5.79	335415	V
5	6.850	15309	V
TOTAL		1882847	

PH 7



PKNO	TIME	AREA	MK
1	3.598	39049	V
2	4.358	176830	V
3	4.927	1311004	V
4	5.8	349300	SV
TOTAL		1876183	

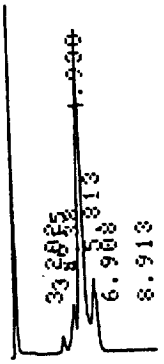
PH 9



PKNO	TIME	AREA	MK
1	3.573	20173	
2	4.067	15877	V
3	4.9	997747	V
4	5.772	230668	SV
5	8.948	20385	V
TOTAL		1284850	

Figure 3.3 GC chromatograms of the test substance
--- for recovery in *Further investigation* ---

Std. 1.01mg/l



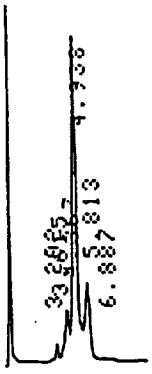
PKNO	TIME	AREA	MK
1	3.202	11582	
2	3.615	78688	V
3	4.38	307633	V
4	4.938	1888329	V
5	5.813	507568	V
6	6.908	22736	V
	TOTAL	2816536	

test solution

PKNO	TIME	AREA	MK
1	3.163	10263	
2	3.572	71301	V
3	4.322	270242	V
4	4.882	1790986	V
5	5.757	507107	V
6	6.81	27486	V
	TOTAL	2677385	

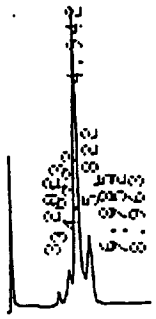
Figure 3.4 GC chromatograms of the test substance
--- for measurement of concentration in water
in *Further investigation* --- [1] *after 2 days*

Std. 1.01mg/l



PKNO	TIME	AREA	MK
1	3.202	1229	V
2	3.615	81198	V
3	4.367	331785	V
4	4.938	1928231	V
5	5.813	547979	V
6	6.887	26503	V
	TOTAL	2928667	

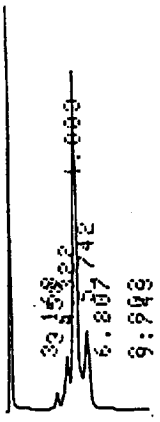
pH 9



PKNO	TIME	AREA	MK
1	3.613	69455	V
2	4.382	241338	V
3	4.942	1738036	V
4	5.822	474936	V
5	6.905	19004	V
	TOTAL	2542849	

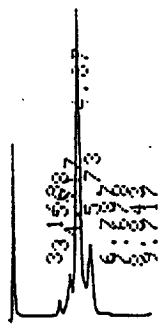
Figure 3.4 (continued) [2] *after 5 days*

Std. 1.01mg/l



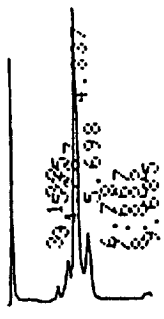
PKNO	TIME	AREA	MK	IDNO
1	3.168	11917		
2	3.58	84103	V	
3	4.322	332077	V	
4	4.883	1973856	V	
5	5.742	561432	SV	
	TOTAL	2963383		

pH 4



PKNO	TIME	AREA	MK	IDNO
1	3.168	10210		
2	3.568	72548	V	
3	4.317	264864	V	
4	4.87	1763114	V	
5	5.73	402272	SV	
	TOTAL	2513109		

pH 7



PKNO	TIME	AREA	MK	IDNO
1	3.535	69921	V	
2	4.287	265288	V	
3	4.837	1672584	V	
4	5.698	465468	SV	
5	9.685	18761		
	TOTAL	2492022		

pH 9

PKNO	TIME	AREA	MK	IDNO
1	3.557	61419	V	
2	4.313	171877	V	
3	4.857	1626920	V	
4	5.717	326543	SV	
5	9.707	10937		
	TOTAL	2197696		

Figure 3.4 (continued) [3] *after 8 days*

Std. 1.01mg/l

pH 7

PKNO	TIME	AREA	MK	IDNO
1	3.568	72575	V	
2	4.318	276869	V	
3	4.87	1764394	V	
4	5.743	325699	SV	
	TOTAL	2439536		

pH 4

PKNO	TIME	AREA	MK	IDNO
~~1~~	~~3.193~~	~~13035~~		
2	3.54	84865	V	
3	4.283	342423	V	
4	4.837	1976176	V	
5	5.703	564727	V	
~~6~~	~~6.78~~	~~27643~~	V	
	TOTAL	3008870		

pH 9

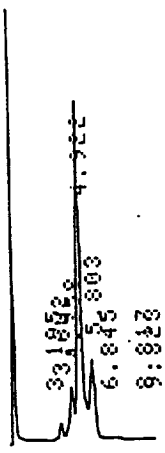
PKNO	TIME	AREA	MK	IDNO
1	3.537	47846	V	
2	4.035	34361	V	
3	4.303	56378	V	
4	4.838	1442243	V	
5	5.705	356058	SV	
6	~~9.200~~	~~12215~~	T	
7	~~9.708~~	~~17949~~		
	TOTAL	1968049		

pH 4

PKNO	TIME	AREA	MK	IDNO
~~1~~	~~3.193~~	~~11039~~		
2	3.607	70742	V	
3	4.36	225643	V	
4	4.92	1689464	V	
5	5.782	463678	SV	
	TOTAL	2460565		

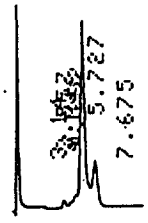
Figure 3.4 (continued) [4] *after 12 days*

Std. 1.01mg/l



PKNO	TIME	AREA	MK	IDNO
1	~~3.195~~	~~12673~~		
2	3.602	85290	V	
3	4.358	341391	V	
4	4.922	2006286	V	
5	5.803	506656	SV	
	TOTAL	2952296		

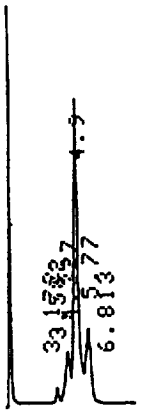
pH 9



PKNO	TIME-	AREA	MK	IDNO
1	3.557	41384	V	
2	4.048	27159	V	
3	4.332	70038	V	
4	4.86	1115007	V	
5	5.727	331848	V	
	TOTAL	1585436		

Figure 3.4 (continued) [5] *after 15 days*

Std. 1.0lmg/l



PKNO	TIME	AREA	MK	IDNO
1	3.172	10294		
2	3.582	78196	V	
3	4.327	329297	V	
4	4.9	1883392	V	
5	5.77	580784	SV	
	TOTAL	2884962		

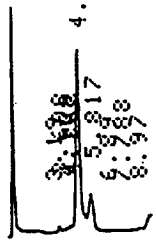
pH 4

PKNO	TIME	AREA	MK	IDNO
1	3.117	10243		
2	3.525	59779	V	
3	4.267	169135	V	
4	4.812	1444165	V	
5	5.65	416131	SV	
	TOTAL	2099453		

pH 7

PKNO	TIME	AREA	MK	IDNO
1	3.54	54941	V	
2	4.275	176806	V	
3	4.833	1362655	V	
4	5.687	416614	SV	
	TOTAL	2011015		

pH 9



PKNO	TIME	AREA	MK	IDNO
1	3.608	32900	V	
2	4.108	22387	V	
3	4.368	35866	V	
4	4.93	1010484	V	
5	5.817	259845	SV	
6	8.97	16905		
	TOTAL	1378387		

Figure 3.4 (continued) **[6] *after 19 days***

Std: 1.01mg/l

pH 7

PKNO	TIME	AREA	MK	IDNO
1	3.578	52284	V	
2	4.322	155916	V	
3	4.895	1248817	V	
4	5.752	429014	SV	
	TOTAL	1886031		

pH 9

PKNO	TIME	AREA	MK	IDNO
1	3.62	22481		
2	4.108	14349	V	
3	4.417	40911	V	
4	4.953	723651	V	
5	5.83	221549	V	
	TOTAL	1022942		

PKNO	TIME	AREA	MK	IDNO
~~1~~	~~3.122~~	~~13139~~		
2	3.53	80003	V	
3	4.252	328533	V	
4	4.827	1879027	V	
5	5.68	583525	SV	
	TOTAL	2884227		

pH 4

PKNO	TIME	AREA	MK	IDNO
1	3.597	55724	V	
2	4.347	149067	V	
3	4.917	1414172	V	
4	5.783	465641	SV	
	TOTAL	2084603		

Figure 3.4 (continued) [7] *after 26 days*

Std. 1.01mg/l

PKNO	TIME	AREA	MK	IDNO
1	3.54	54270	V	
2	4.308	130219	V	
3	4.842	1100240	V	
4	5.692	391650	V	
	TOTAL	1676378		

pH 7

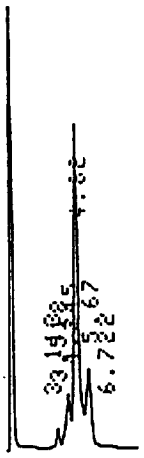
PKNO	TIME	AREA	MK	IDNO
~~1~~	~~3.12~~	~~11819~~		
2	3.52	86807	V	
3	4.245	338231	V	
4	4.803	1970817	V	
5	5.657	566933	SV	
	TOTAL	2974607		

pH 4

PKNO	TIME	AREA	MK	IDNO
1	3.527	58084	V	
2	4.28	152256	V	
3	4.823	1349836	V	
4	5.667	352547	SV	
	TOTAL	1912723		

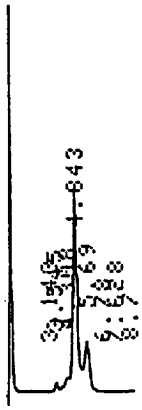
Figure 3.4 (continued) [8] *after 33 days*

Std. 1.01mg/l



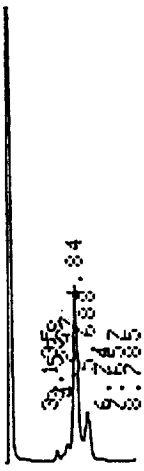
PKNO	TIME	AREA	MK	IDNO
1	3.113	10906		
2	3.518	86430	V	
3	4.245	341379	V	
4	4.82	1937852	V	
5	5.67	563253	V	
6	6.722	27141	V	
TOTAL		2966930		

pH 4



PKNO	TIME	AREA	MK	IDNO
1	3.545	48236	V	
2	4.308	114485	V	
3	4.843	1193570	V	
4	5.69	362408	SV	
TOTAL		1718699		

pH 7



PKNO	TIME	AREA	MK	IDNO
1	3.538	46752	V	
2	4.297	116978	V	
3	4.84	1032199	V	
4	5.688	294110	SV	
TOTAL		1490039		

Figure 4.1 UV spectrum of the test substance
in acidic, neutral, and alkaline solutions

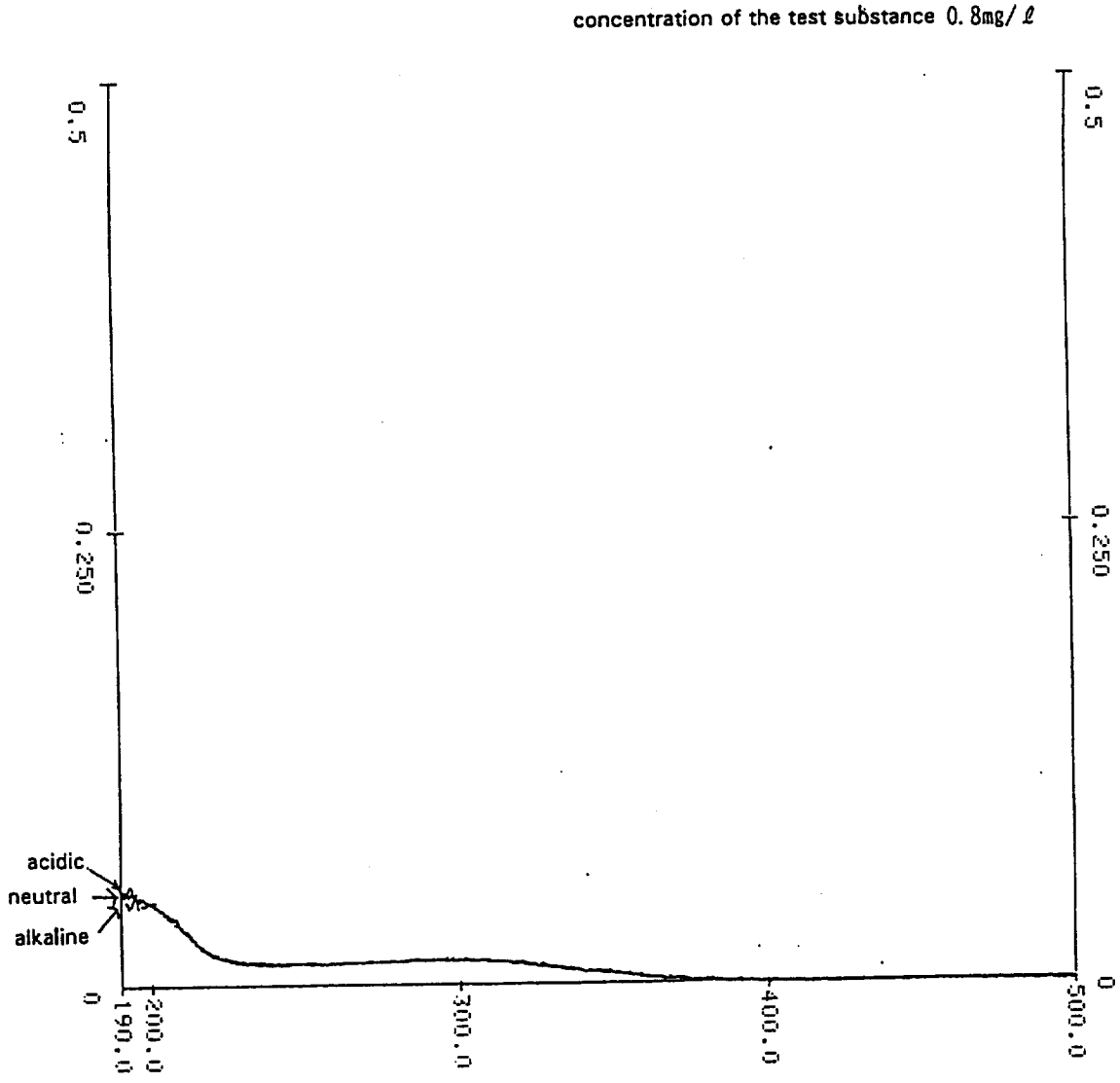


Figure 5.1 Correlation between HPLC retention time and Pow of the reference compounds

Curve Fitting [Least Square Method]

Input Data

No.	log Rt	log Pow
1	0.355	2.12
2	0.626	2.99
3	0.813	3.76
4	0.981	4.81
5	1.123	6.20

$Y = -2.1919 \times 10^{-2} + 5.1282 \times X1$

$r = 0.97258$

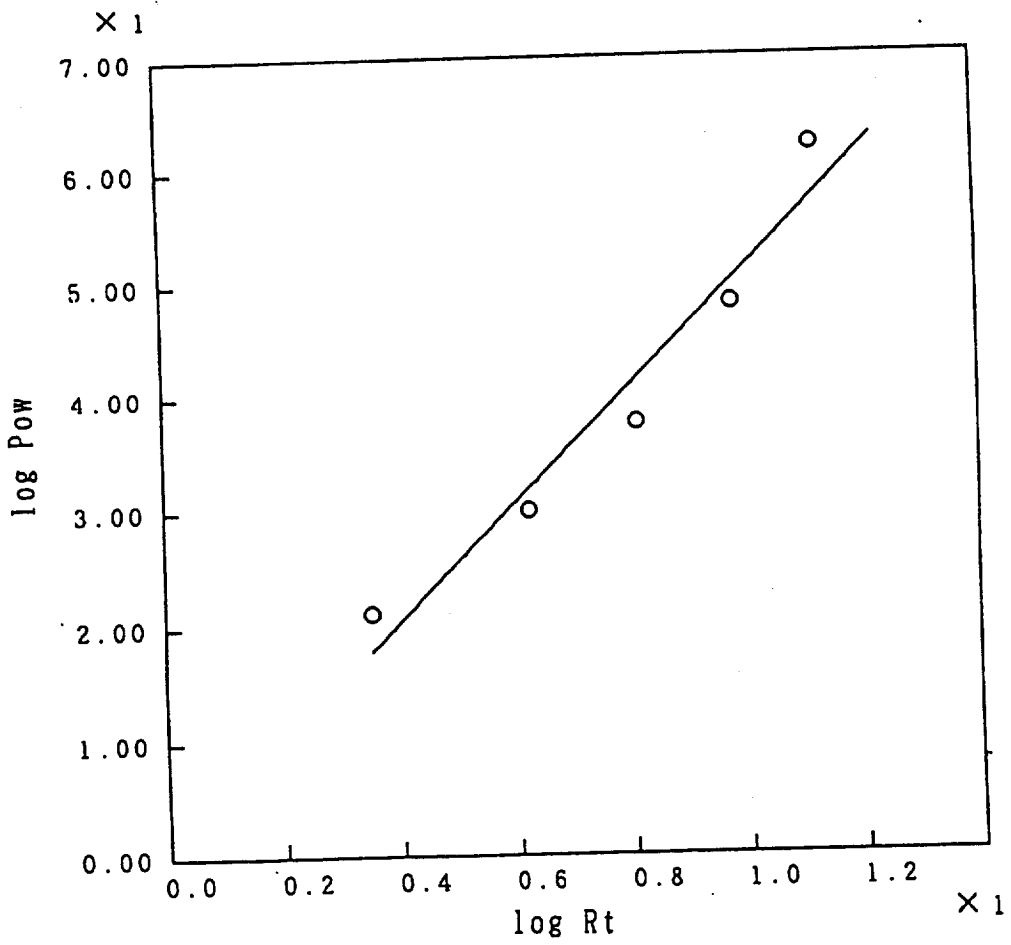
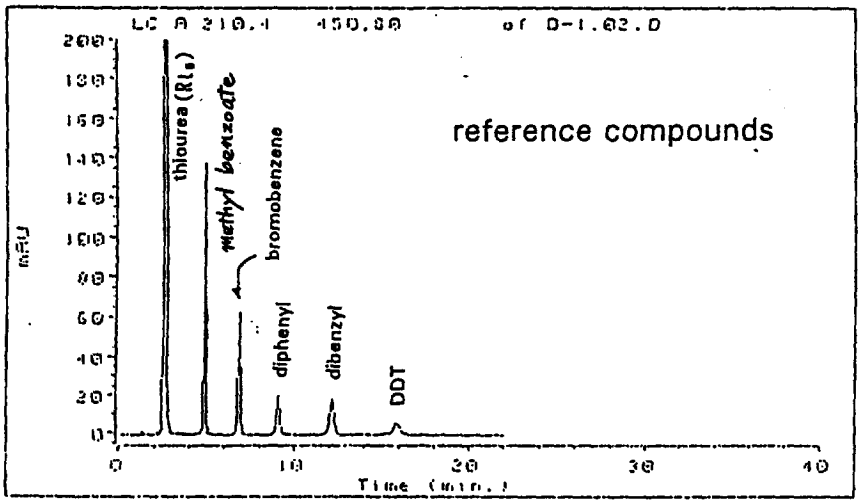
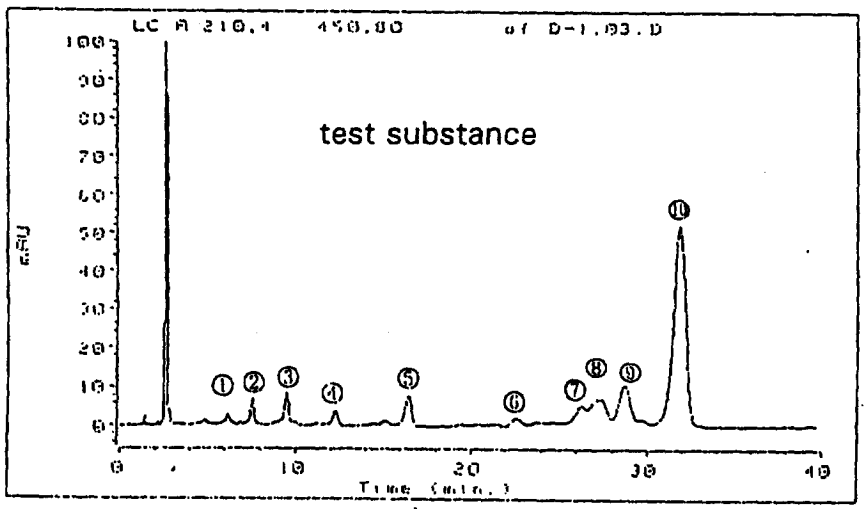


Figure 5.2 HPLC chromatograms of the reference compounds and the test substance



Peak#	Ret Time	Type	Width	Area		Rt-Rt₀	log Rt	log Pow
1	2.637	PV	0.070	11214	thiourea			
2	4.903	BV	0.116	1042	methyl benzoate	2.266	0.355	2.12
3	6.867	BV	0.157	633.02	bromobenzene	4.230	0.626	2.99
4	9.139	BV	0.208	278.54	diphenyl	6.502	0.813	3.76
5	12.204	BV	0.274	353.74	dibenzyl	9.567	0.981	4.81
6	15.900	VV	0.325	169.83	DDT	13.263	1.123	6.20



Peak#	Ret Time	Type	Width	Area	thiourea	Rt-Rt₀	log Rt	log Pow
1	2.633	PV	0.069	2559				
2	6.144	VV	0.236	53.99	①	3.511	0.545	2.77
3	7.555	VV	0.199	90.09	②	4.922	0.692	3.75
4	9.502	BV	0.261	138.64	③	6.869	0.837	4.27
5	12.304	BV	0.255	79.43	④	9.671	0.985	5.03
6	16.445	VV	0.377	214.84	⑤	13.812	1.140	5.82
7	22.562	BV	0.416	67.83	⑥	19.929	1.299	6.64
8	26.308	BV	0.462	168.18	⑦	23.675	1.374	7.02
9	27.359	VV	0.751	359.48	⑧	24.726	1.393	7.12
10	28.722	VV	0.482	371.23	⑨	26.089	1.416	7.24
11	31.878	BV	0.669	2576	⑩	29.245	1.466	7.50

Table 6.1 Calculation of vapor pressure

trapped test substance (*corrected* mass), g	W	1.47×10^{-3}
molecular weight of the test substance	M	625
gas constant, $Pa*m^3*mole^{-1}*K^{-1}$	R	8.31
corrected total volume of the carrier gas, m^3	V	0.973
temperature, °K	T	298
vapor pressure, Pa	P	6.0×10^{-3}

calculation equations

$$P = (W/M)(RT/V)$$

Table 6.2 Calculation of mass of the trapped test substance

conc. in Std., mg/L	A	1.006
peak area, $\mu V*sec$		
Std.	B	2489067
test solution	C	1412087
conc. in the test solution, mg/L		0.571
final volume, mL	D	50
dilution factor	E	50
trapped test substance as *measured* mass, g		1.43×10^{-3}
as *corrected* mass, g	W	1.47×10^{-3}

calculation equation

$$W = A(C/B)E(D/1000)(1/0.97)(1/1000)$$

Table 6.3 Calculation of desorption efficiency

conc. of the test substance added, mg/L	A	0.02515
conc. in Std., mg/L	B	1.006
peak area, μV*sec		
Std.	C	2451409
test solution	D	2372240
final volume, mL		25
conc. of the test substance recovered, mg/L	E	0.02434
desorption efficiency, %	F	97

calculation equations

$E = B(D/C)(25/1000)$ $F = (E/A)100$

Figure 6.1 Calibration curve of the test substance

Curve Fitting [Least Square Method]

Input Data

No.	Concentration X(mg/l)	Peak Area Y(μV·sec)
1	0	0
2	0.252	602790
3	0.503	1234186
4	1.003	2475687

$Y = -9.1506 \times 10^{3} + 2.4740 \times 10^{6} \times X^{1}$

$r = 0.99997$

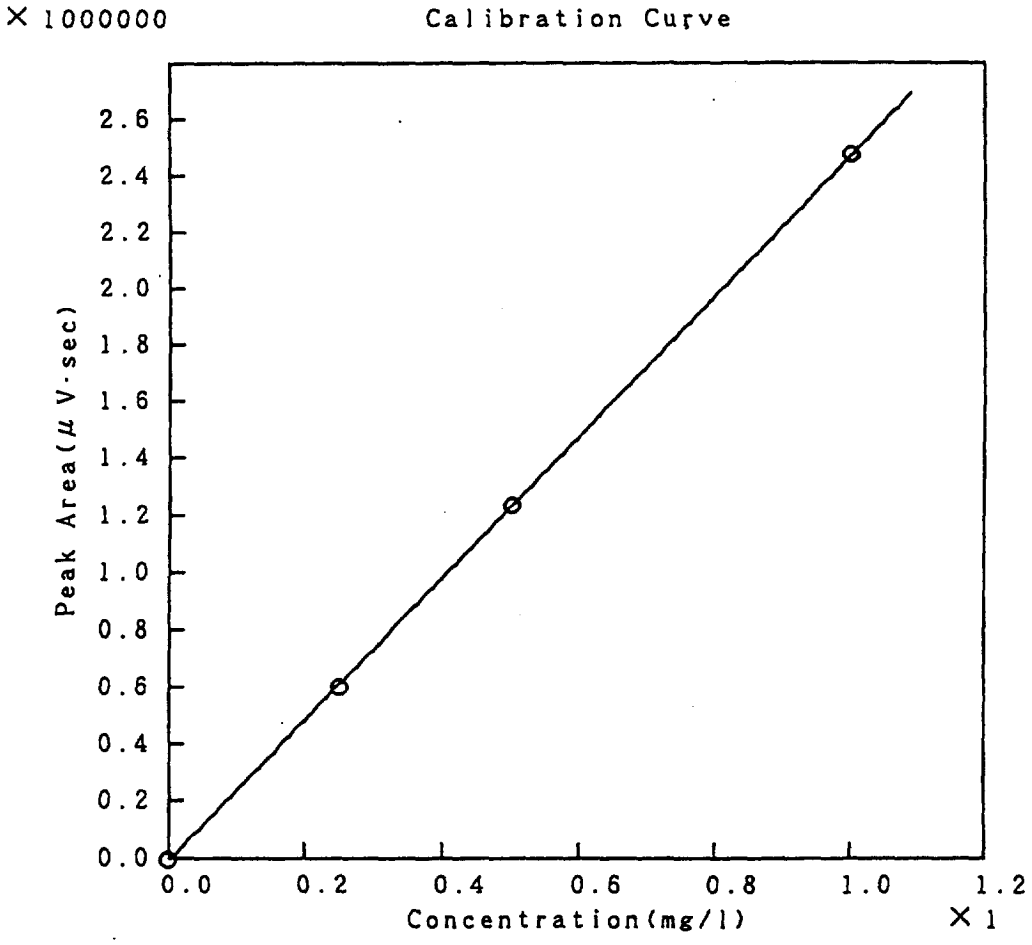
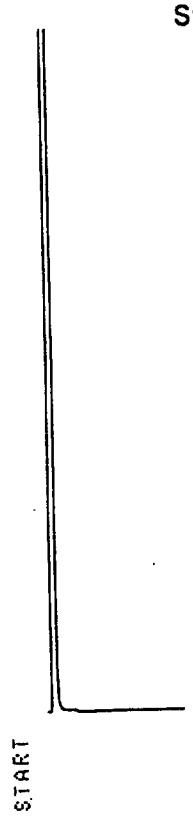
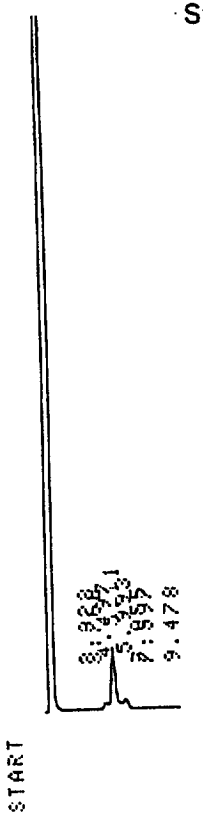


Figure 6.2 GC chromatograms of the test substance
--- for calibration curve ---

Std. 0mg/ℓ

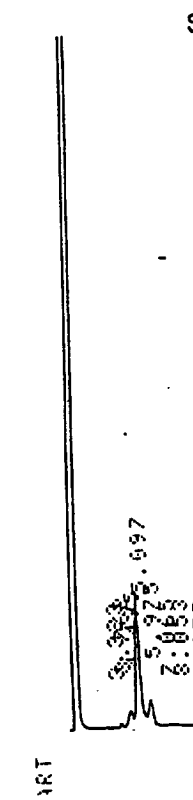


Std. 0.25mg/ℓ



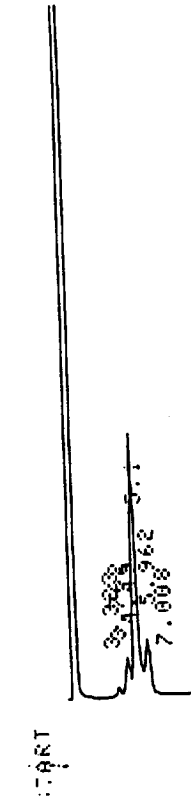
PKNO	TIME	AREA	MK	IDNO	CONC
1	3.762	13433	V		2.2284
2	4.497	63505	V		10.5351
3	5.11	417290	V		69.2264
4	5.993	108563	SV		18.0101
TOTAL		602790			100

Std. 0.50mg/ℓ



PKNO	TIME	AREA	MK	IDNO	CONC
1	3.762	28988	V		2.3488
2	4.495	131742	V		10.6744
3	5.097	843023	V		68.306
4	5.975	230432	SV		18.6708
TOTAL		1234186			100

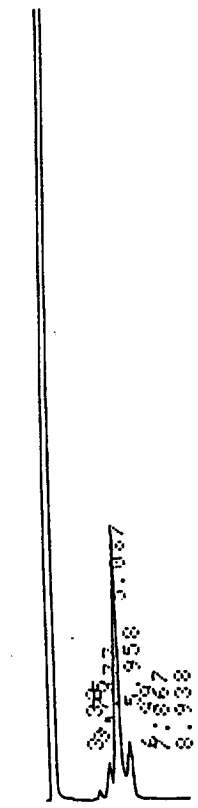
Std. 1.0 mg/ℓ



PKNO	TIME	AREA	MK	IDNO	CONC
1	3.920	10210			0.411
2	3.762	62275	V		2.5051
3	4.49	266548	V		10.7224
4	5.1	1674132	V		67.3449
5	5.962	472732	SV		19.0165
TOTAL		2485905			106

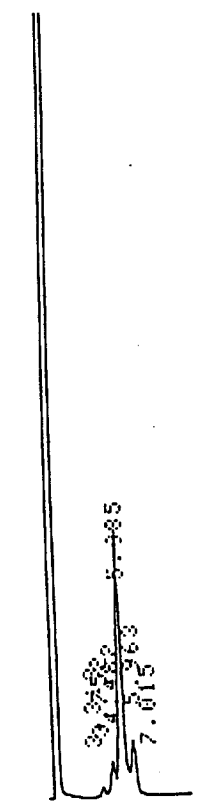
Figure 6.3 GC chromatograms of the test substance
--- for desorption efficiency ---

Std. (1.0mg/ℓ)



PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.02	10309			0.4188	
2	3.75	60986	V		2.4774	
3	4.477	262684	V		10.6708	
4	5.087	1662992	V		67.5541	
5	5.958	464747	SV		18.879	
TOTAL		2461717			100	

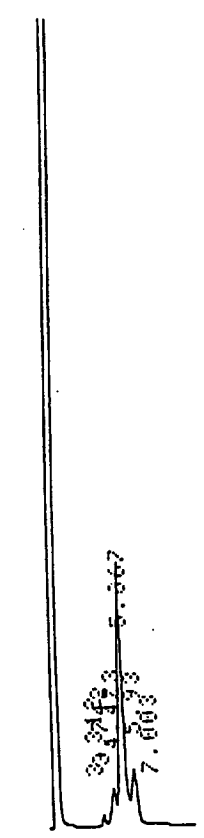
test solution



PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.752	59840	V		2.5225	
2	4.482	253092	V		10.6651	
3	5.085	1612761	V		67.9847	
4	5.963	446637	SV		18.8276	
TOTAL		2372240			100	

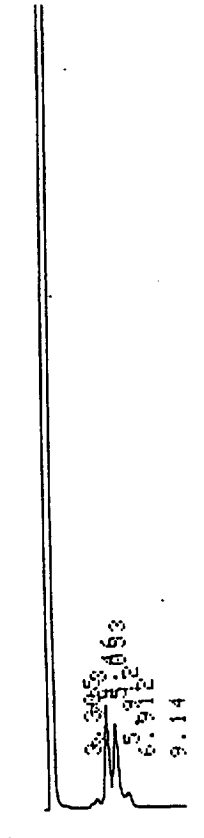
Figure 6.4 GC chromatograms of the test substance
--- for measurement of mass of the trapped test substance ---

Std. (1.0mg/ℓ)



PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.742	58593	V		2.354	
2	4.473	262690	V		10.5538	
3	5.067	1683711	V		67.6442	
4	5.93	484073	SV		19.448	
TOTAL		2489067			100	

test solution



PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.305	21602			1.5067	
2	3.723	66097	V		4.6103	
3	4.46	623477	V		43.4876	
4	5.053	588125	V		41.0218	
5	5.912	134388	V		9.3736	
TOTAL		1433689			100	

Figure 6.5 Vapor pressure measuring apparatus