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CHEMICAL MANUFACTURERS ASSOCIATION

June 27, 1997

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Dr. Lynn Goldman
Assistant Administrator
Office of Prevention, Pesticides and Toxic Substances TS-7101
Environmental Protection Agency
401 M Street, SW, Room 637, East Tower
Washington, DC 20460

Dear Dr. Goldman:

The Chemical Manufacturers Association makes available to the public and appropriate government agencies final reports of the environmental, health and safety research that it manages. In keeping with this policy, the following recently completed reports are enclosed:

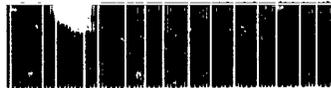
1. DECABROMODIPHENYL OXIDE (DBDPO): Determination of *n*-Octanol/Water Partition Coefficient; and
2. DECABROMODIPHENYL OXIDE (DBDPO): Determination of the Water Solubility.

These reports do not include confidential information.

If you have any questions, please call Has Shah of my staff at 703-741-5637.

Sincerely Yours,

Courtney M. Price
Vice President, CHEMSTAR



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**DECABROMODIPHENYL OXIDE (DBDPO):
DETERMINATION OF THE WATER SOLUBILITY**

WILDLIFE INTERNATIONAL LTD. PROJECT NUMBER: 439C-102

**U.S. EPA 40 CFR Ch. 1 § 796.1860 Water Solubility (Generator Column Method)
OECD Guideline 105 Water Solubility (Column Elution Method)**

AUTHORS:

**Joel I. Stein
Barbara J. Markley, Ph.D.**

STUDY INITIATION DATE: February 14, 1996

STUDY COMPLETION DATE: June 10, 1997

Submitted to:

**Chemical Manufacturers Association's
Brominated Flame Retardant Industry Panel
1300 Wilson Boulevard
Arlington, Virginia 22209**



WILDLIFE INTERNATIONAL LTD.

**8598 Commerce Drive
Easton, Maryland 21601
(410) 822-8600**

Page 1 of 54

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GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT

SPONSOR: Chemical Manufacturers Association's Brominated Flame Retardant Industry Panel

TITLE: Decabromodiphenyl Oxide (DBDPO): Determination of the Water Solubility

WILDLIFE INTERNATIONAL LTD. PROJECT NUMBER: 439C-102

STUDY COMPLETION: June 10, 1997

This study was conducted in compliance with Good Laboratory Practice Standards as published by the U.S. Environmental Protection Agency in 40 CFR Part 792, 17 August 1989; OECD Principles of Good Laboratory Practices, (OECD)C(81)30 (Final)Annex 2; and Japan MHW/MITI 59 Kikyoku No. 85; EA, Kankiken No. 233: MHW, Eisei No. 38 and MITI, 63 Kikyoku No. 823.

STUDY DIRECTOR:

Joel I. Stenzel
Joel I. Stenzel, B.S.
Senior Chemist

6/10/97
DATE

SPONSOR APPROVAL:

Harmukh Shah
Sponsor

6/10/97
DATE

QUALITY ASSURANCE

SPONSOR: Chemical Manufacturers Association's Brominated Flame Retardant Industry Panel

TITLE: Decabromodiphenyl Oxide (DBDPO): Determination of the Water Solubility

WILDLIFE INTERNATIONAL LTD. PROJECT NO.: 439C-102

This study was examined for compliance with Good Laboratory Practice Standards as published by the U.S. Environmental Protection Agency in 40 CFR Part 792, 17 August 1989; OECD Principles of Good Laboratory Practices, (OECD)C(81)30 (Final)Annex 2; and Japan MHW/MITI 59 Kikyoku No. 85; EA, Kankiken No. 233; MHW, Eisei No. 38 and MITI, 63 Kikyoku No. 823. The dates of all inspections and audits and the dates that any findings were reported to the Study Director and Laboratory Management were as follows:

ACTIVITY:	DATE CONDUCTED:	DATE REPORTED TO:	
		STUDY DIRECTOR:	MANAGEMENT:
Test Substance Composite Preparation	February 19, 1996	February 19, 1996	February 21, 1996
Stock Solution Preparation	August 5, 1996	August 5, 1996	August 14, 1996
Generator Column Preparation	August 28, 1996	September 3, 1996	September 4, 1996
Stock Preparation	August 29, 1996	September 3, 1996	September 4, 1996
Matrix Fortification Preparation	August 30, 1996	August 30, 1996	September 3, 1996
Draft Report and Data	January 30, 31, February 3, 4, 1997	February 5, 1997	February 10, 1997
Final Report	June 9, 1997	June 9, 1997	June 10, 1997

Susan L. Hopper

Susan L. Hopper
Senior Quality Assurance Representative

6-10-97

DATE

REPORT APPROVAL

SPONSOR: Chemical Manufacturers Association's Brominated Flame Retardant Industry Panel

TITLE: Decabromodiphenyl Oxide (DBDPO): Determination of the Water Solubility

WILDLIFE INTERNATIONAL LTD. PROJECT NO.: 439C-102

This report was reviewed by the individuals involved in the conduct and management of the study, and was found to be an accurate reflection of the methods used, data collected and results of the study.

STUDY DIRECTOR:

Joel I. Stenzel
Joel I. Stenzel, B.S.
Senior Chemist

6/10/97
DATE

MANAGEMENT:

Willard B. Nixon
Willard B. Nixon, Ph.D.
Manager, Analytical Chemistry

6/10/97
DATE

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SUMMARY

SPONSOR:	Chemical Manufacturers Association's Brominated Flame Retardant Industry Panel
SPONSOR'S REPRESENTATIVE:	Hasmukh Shah, Ph.D.
LOCATION OF STUDY, RAW DATA AND A COPY OF THE FINAL REPORT:	Wildlife International Ltd. Easton, Maryland 21601

WILDLIFE INTERNATIONAL LTD.	
PROJECT NUMBER:	439C-102
TEST SUBSTANCE:	Decabromodiphenyl oxide (DBDPO)
STUDY:	Decabromodiphenyl oxide (DBDPO): Determination of the Water Solubility
TEST DATES:	Experimental Start - May 3, 1996 Experimental Termination - September 16, 1996

SUMMARY:	The solubility of DBDPO in water at 25.0°C was determined to be less than 0.1 µg/L (ppb) using a column elution method.
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INTRODUCTION

This study was conducted by Wildlife International Ltd. for Chemical Manufacturers Association's (CMA) Brominated Flame Retardant Industry Panel at the Wildlife International Ltd. analytical chemistry facility in Easton, Maryland. Tests were performed using a column elution method. Samples were eluted from a generator column and analyzed from August 28 to September 13, 1996. Raw data generated by Wildlife International Ltd. and a copy of the final report are filed under Project Number 439C-102 in archives located on the Wildlife International Ltd. site.

OBJECTIVE

The objective of this study was to determine the solubility limit of the test substance, decabromodiphenyl oxide (DBDPO), in water at 25.0°C using a column elution method.

EXPERIMENTAL DESIGN

A preliminary test was conducted to estimate the solubility of the test substance in reagent water at room temperature. A column elution method was used for the definitive test. The column temperature was maintained at 25.0 °C and reagent water was pumped through it at approximately 0.5 mL per minute to elute the test substance. Samples of the eluate were collected and analyzed to determine the saturation concentration of two major components of the test substance. The flow rate of reagent water through the column was reduced to approximately half the original flow rate and the saturation concentration determined again.

MATERIALS AND METHODS

This study was conducted according to procedures outlined in the protocol, "Decabromodiphenyl Oxide (DBDPO): Determination of the Water Solubility" (Appendix I). The

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protocol was based on procedures outlined in OECD Guidelines, Method 105 (1); and EPA 40 CFR Ch. 1 § 796.1860 (2). The column elution method was used to determine the water solubility limit of the test substance.

Test Substance

The test substance consisted of a composite of decabromodiphenyl oxide (DBDPO) samples received from three manufacturers. The materials' identity and date received from each of the manufacturers are given below:

<u>Manufacturer</u>	<u>Lot/Batch</u>	<u>Date Received</u>	Wildlife International Ltd. <u>ID No.</u>
Great Lakes Chemical Corp.	5480DH24A	October 26, 1995	3517
Albemarle Corp.	4449-1N	December 20, 1995	3518
Bromine Compound Co.	950289	January 30, 1996	3547

An equal part (300 g) of each of the manufacturer's products was placed in a 2-L, high density polyethylene (HDPE) bottle. The bottle was placed on a reciprocating shaker for two hours. The composite test substance was assigned Wildlife International Ltd. identification number 3578. Subsamples of the composite test substance were shipped to Albemarle Corporation for characterization and homogeneity analyses. The analyses were performed on March 13, 1996. The results of the analyses indicated the composite test substance was homogeneous and contained the following components:

Octabromodiphenyl oxide	0.04%
Nonabromodiphenyl oxide	2.5%
Decabromodiphenyl oxide	97.4%

The composite test substance was stored under ambient conditions.

Reagent Water

The reagent water used in this study exceeded the requirements for ASTM Type II water. The water was obtained from a well located on the Wildlife International Ltd. site. The well water was pumped through a series of filters to remove microorganisms and particles greater than 0.2 μm . The water was further purified using a Culligan® Hi-Flo 1 Water Softener, a Culligan® S-Series Reverse Osmosis System, and a Barnstead NANOpure ultrapure water system. The resistivity of the purified reagent water used for this study was greater than 17.8 megohm-cm.

Preliminary Test

A preliminary test was performed to estimate the solubility of the test substance in reagent water. Approximately 100 mg of the composite test substance was placed in a vessel. A pre-determined volume of reagent water was added, and the vessel shaken. The solubility of the test substance was determined by visual observation. The process was repeated twice by adding successively larger volumes of reagent water.

Solvents

Analytical grade ethyl acetate (Fisher Chemical, Fairlawn, NJ, catalog number E196-4) was used as an extraction solvent for this study. Phenyl ether, with a purity of >98%, was used for preparation of calibration standards and samples. It was purchased from Fluka Chemika (Switzerland, catalog number 42730). Analytical grade dimethyl formamide (Burdick and Jackson, catalog number 076-4) was used for the preparation of fortification standards.

Preparation of Generator Column

The inert carrier material used to charge the generator column was glass beads. The glass beads were supplied from J.T. Baker (Phillipsburg, NJ, cat. number 7467-01), and identified on the label as Empore, Filter Aid 400, high density glass beads. Approximately 25 mL (~65.6 g) of the glass beads were rinsed with ethyl acetate to remove any potential contaminants, then transferred to a round-bottom flask.

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A subsample of the composite test substance (323.4 mg) was weighed onto a glass cover slip and placed into a 500 mL Erlenmeyer flask. Ethyl acetate was added to the 300 mL mark. The flask was sonicated for about 10 minutes, but the test substance did not completely dissolve. Another 100 mL of ethyl acetate was added, and the flask was sonicated for about 5 minutes more. Most of the test substance was dissolved, but some very fine material was uniformly suspended. The test substance solution was transferred to the round-bottom flask containing the glass beads.

A rotary evaporator was used to remove the solvent and coat the test substance onto the glass beads. The water bath of the rotary evaporator was set at 40 °C. The trap was emptied after the solvent had evaporated, and the flask was replaced on the rotary evaporator for another 35 minutes, to ensure all the solvent had been removed. The contents of the round-bottom flask then were transferred to a 150 mL beaker. Approximately 25 mL of reagent water were added, and the beaker was swirled and sonicated to remove air bubbles and homogenize the slurry.

The generator column was an Adjusta-Chrom[®], jacketed, recycling column supplied by Ace Glass Inc. (Vineland, NJ, cat. number 5819). The glass column, 300 mm long with an internal diameter of 10 mm, was equipped with Teflon[®] plungers and glass filter discs at both ends. A small plug of silanized glass wool was placed in the bottom of the column, the plunger inserted, and end fittings fastened to the bottom. The slurry of glass beads coated with test substance was poured into the top of the column. Another small plug of silanized glass wool was placed on the top of the column, the plunger inserted, and end fittings attached. The height of the material packed in the column was approximately 24 cm. A diagram of the generator column is presented in Figure 1.

Apparatus Configuration

A Cannon CT-500 Constant Temperature Bath (Cannon Instrument Company, State College, PA) was used to maintain the test temperature (25.0 ± 0.1 °C) throughout the experiment. The chiller coil of the constant temperature bath was connected to a Nessler Coolflow CFT-25 Refrigerated Recirculator (Nessler Instruments, Inc., Portsmouth, NH). Both the constant temperature bath and recirculating chiller were filled with water. The temperature of the water bath

was monitored using an ASTM #45C kinematic viscosity thermometer (Ever Ready Thermometer Company, West Paterson, N.J.).

A Teel Model PP650A submersible pump (Dayton Electric Mfg. Co., Chicago, IL) was placed in the constant temperature bath. The submersible pump was used to pump a continuous stream of water through the jacket surrounding the generator column, to maintain a constant temperature.

A 2-liter Erlenmeyer flask was used as a reservoir for the reagent water being pumped through the generator column. The flask was filled with reagent water and submerged in the constant temperature bath so that the top of the flask remained above the surface of the water in the constant temperature bath. The inlet line of a Milton Roy miniPump® (P/N 920-4901) was placed in the 2-L flask, and the top of the flask was covered with aluminum foil. The pump was used to control the flow rate of the reagent water through the generator column. The supply of reagent water in the 2-L flask was replenished intermittently during the experiment.

The eluate from the generator column was directed to a Fozzy 200[®] fraction collector (Isco, Inc., Lincoln, NE). The fraction collector was programmed to collect individual samples of the eluate into borosilicate glass bottles. A diagram of the apparatus configuration is presented in Figure 2.

Sample Collection

The pump was initially set to deliver approximately 0.5 mL of reagent water per minute through the generator column. The fraction collector was programmed to collect eluate samples every 98.25 minutes (~ 50 mL). The eluate was collected dropwise into 4-ounce square bottles containing 25 mL of the extraction solvent (ethyl acetate). The sample bottles were labelled with a sequential sample number. After 130 consecutive samples were collected, the flow rate was changed to approximately half the original flow rate (~ 0.25 mL/min.). The fraction collector was reprogrammed to collect samples every 487.8 minutes (~100 mL), and sample collection was

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resumed. The following work day, the volume of the samples was found to be much larger than expected, and the flow rate was readjusted. The fraction collector was reprogrammed to collect samples every 204 minutes (~50 mL), and sample collection resumed with sample number 140. The sample bottles were capped and stored on a laboratory bench under ambient conditions prior to extraction and analysis.

Analytical Method

The analytical method consisted of extracting the samples with ethyl acetate, evaporating the ethyl acetate, and reconstituting the samples in phenyl ether. A flow chart of the method is presented in Figure 3. Ethyl acetate was added to each sample bottle to adjust the volume to approximately 25 mL prior to the first extraction to make up for any minor evaporative losses during sample collection. The sample bottle was capped and shaken for approximately 30 seconds. The ethyl acetate layer was transferred to a round-bottom flask using a disposable pipet. The sample was extracted with another 20 mL portion of ethyl acetate. The extracts were combined in the round-bottom flask, and 200 μ L of phenyl ether was added. The extract was placed on a rotary evaporator to remove the ethyl acetate. The remaining residue was transferred to a culture tube. The round-bottom flask was rinsed three times with ethyl acetate and the rinsates were combined in the culture tube. The ethyl acetate was evaporated from the tube under a stream of nitrogen at 40 to 50°C. The sample was brought to a final volume of 1.0 mL using phenyl ether and placed in a vial for analysis.

Concentrations of DBDPO in the samples were determined using a Hewlett-Packard Model 5890 Gas Chromatograph (GC). The gas chromatograph was equipped with a cyclo-splitter liner in the injection port and an electron capture detector (ECD). Chromatographic separations were achieved using an Rtx-1 capillary column (15 m \times 0.53 mm, 0.1 μ m film thickness) supplied by Restek Corporation (Bellefonte, PA). Chromatographic grade helium was used as the carrier gas, and argon (95%)/methane (5%) was used as the auxiliary gas. The instrument parameters are summarized in Table 1.

Preparation of Quality Control Samples

A fortification standard of DBDPO in dimethyl formamide (DMF) was prepared at a concentration of 1.0 mg/L. This standard was used to fortify reagent water (matrix spikes) and ethyl acetate (reagent spikes). A reagent blank and reagent fortification (5 ppb) were prepared and analyzed along with each set of samples from the generator column. A matrix blank and two matrix fortifications (1 ppb and 5 ppb) were also prepared and analyzed with each set. A sample of the phenyl ether was also analyzed with each sample set.

Calibration and Quantitation

Calibration standards of DBDPO were prepared in phenyl ether. The standards were prepared using the test substance, and ranged in concentration from 5 to 500 μg DBDPO/L. A complete set of calibration standards was analyzed before and after each set of samples, and a standard was injected at least after every five samples. Representative chromatograms of low and high calibration standards are shown in Figures 4 and 5, respectively. The sum of the peak areas for the two major peaks (23.5 and 25.1 minutes) was used to determine the instrument response. The instrument limit of detection (LOD) for this study was not experimentally determined, but was set based upon the injection volume (2 μL) and the lowest calibration standard concentration (5 $\mu\text{g}/\text{L}$). The LOD thus was set at 10 pg of DBDPO injected.

Calibration curves were calculated based on linear regression equations using the instrument responses versus the respective standard concentrations. Calibration curves were segmented by concentration to achieve better quantitation of samples in the lower concentration ranges. Separate calibration curves were generated for the low-level standards (5 to 25 $\mu\text{g}/\text{L}$) and for the high-level standards. Representative low-level and high-level linear calibration curves are presented in Figures 6 and 7, respectively.

RESULTS AND DISCUSSION

Preliminary Test

The test substance at approximately 100 mg did not appear to be soluble in 10 mL, 100 mL or 1 L of reagent water. Most of the test substance was observed to settle to the bottom of the bottle. The solubility was estimated to be much less than 100 mg/L, based on visual observations.

Quality Control Samples

No interferences were observed at or above the limit of detection ($0.1 \mu\text{g DBDPO/L}$) in any of the matrix blank samples. A representative chromatogram of a matrix blank is shown in Figure 8. The instrument response for the matrix blanks (sum of peak areas) was always well below the response of the lowest calibration standard. There were no interferences observed in the reagent blanks (ethyl acetate) as well, with the exception of two samples. The reagent blank samples that did show interferences were attributed to contamination, due to the magnitude of the interference. A representative chromatogram of a reagent blank is shown in Figure 9. There were no interferences observed in the phenyl ether samples. A representative chromatogram of a phenyl ether sample is shown in Figure 10.

The mean recovery from six matrix samples fortified at 5 ppb was 88%, and ranged from 74% to 106%. A representative chromatogram of a 5 ppb matrix fortification is shown in Figure 11. Most of the matrix samples fortified at 1 ppb appeared to have been contaminated, and had recoveries much greater than 100%. Only two of the matrix samples fortified at 1 ppb appeared to have reasonable recoveries of 60%. A representative chromatogram of a 1 ppb matrix fortification is shown in Figure 12. Although the low-level matrix fortifications did not always yield acceptable recoveries, none had less than 60% recovery; therefore, the 1 ppb concentration was considered the limit of quantitation (LOQ). The mean recovery from six reagent fortifications at 5 ppb was 74%, and ranged from 51% to 83%. A representative chromatogram of a reagent fortification is shown in Figure 13.

Column Elution

The temperature of the water bath ranged from 24.95 to 25.00°C during the experiment (Table 2). The flow rate of reagent water through the generator column was measured at the start of sample collection for each pump setting. Flow rates were also calculated based on the volume and collection time of each sample that was analyzed. The initial pump setting was 22 and the flow rate was measured at 0.51 mL/min. The calculated flow rates for samples collected at the initial setting averaged 0.44 mL/min. and ranged from 0.31 to 0.56 mL/min (Table 3). After collection of sample number 139, the flow rate was reduced to approximately half the initial flow rate. The pump setting was changed to 8 and the flow rate was measured at 0.25 mL/min. The calculated flow rates for samples collected at this pump setting were constant at 0.19 mL/min (Table 4).

The results from the analyses of samples eluted from the generator column at a nominal flow rate of 0.5 mL/min. are presented in Table 3. Not all of the samples were analyzed. There were some erratic results from samples collected early in the experiment. This might have been due to contamination of the sample bottles. Results from later samples were much more consistent. The concentration of DBDPO in many of the samples was below the limit of quantitation (1 ppb) and some results were below the limit of detection (0.1 ppb). The solubility limit was considered to have been achieved when at least five consecutive samples gave similar results. The results from samples 57 through 67 met these criteria and were considered to have reached the solubility plateau. The mean concentration measured in these samples was less than 0.1 ppb, and the standard deviation could not be calculated. A representative chromatogram of a sample is shown in Figure 14. At a flow rate of 0.5 mL/min., the solubility limit of DBDPO in water was determined to be less than 0.1 ppb.

The results from the analyses of samples eluted from the generator column at a nominal flow rate of 0.25 mL/min. are presented in Table 4. The concentration of DBDPO in most of the samples was below the limit of quantitation (1 ppb) and many results were below the limit of detection (0.1 ppb). The results from samples 153 through 160 were considered to have reached the solubility plateau. The mean concentration measured in these samples was less than 0.1 ppb, and the standard

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deviation could not be calculated. A representative chromatogram of a sample is shown in Figure 15. At a flow rate of 0.25 mL/min., the solubility limit of DBDPO in water was determined to be less than 0.1 ppb.

CONCLUSIONS

Based on the results from samples collected at both flow rates from the generator column, the solubility of DBDPO in water was determined to be less than 0.1 μg DBDPO/L (ppb) at 25.0°C.

REFERENCES

1. **Organisation for Economic Cooperation and Development.** 1981. Guideline for Testing of Chemicals, Method 105, "Water Solubility" (Column Elution Method - Flask Method).
2. **U.S. Environmental Protection Agency.** 1991. 40 CFR § 796.1860, Water Solubility (generator column method). Washington, D.C.

Table 1

Typical Gas Chromatographic Operational Parameters

INSTRUMENT:	Hewlett-Packard Model 5890 Gas Chromatograph (GC) Equipped with a Model G1030A Chemstation
DETECTOR:	Hewlett-Packard Electron-Capture Detector (ECD)
ANALYTICAL COLUMN:	Restek-Rtx-1 Column (15 m x 0.53 mm, 0.1 μ m film thickness)
INJECTOR TEMPERATURE:	300°C
OVEN:	Initial temperature: 150°C Initial hold time: 2 minutes Ramp: 5°C/minute Final temperature: 300°C Final hold time: 3 minutes
DETECTOR TEMPERATURE:	320°C
INJECTION VOLUME:	2 μ L
CARRIER GAS:	Helium at 50 mL/minute from split vent
MAKE-UP GAS:	Argon/Methane at 60 mL/minute from detector
DECABROMODIPHENYL OXIDE (DBDPO) PEAK RETENTION TIMES:	23.7 and 25.1 minutes

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Table 2

Water Bath Test Temperatures

Date & Time of Observation	Temperature (°C)
8/28/96 - 18:40	25.00
8/29/96 - 8:00	25.00
8/29/96 - 16:50	25.00
8/30/96 - 7:45	25.00
8/30/96 - 17:00	25.00
8/31/96 - 12:30	25.00
8/31/96 - 14:13	24.95
9/1/96 - 11:40	25.00
9/1/96 - 12:45	25.00
9/2/96 - 12:12	25.00
9/2/96 - 13:23	25.00
9/3/96 - 7:31	25.00
9/3/96 - 13:02	24.95
9/3/96 - 16:33	25.00
9/4/96 - 8:10	25.00
9/4/96 - 17:26	25.00
9/5/96 - 7:38	25.00
9/5/96 - 17:14	25.00
9/6/96 - 8:28	25.00
9/6/96 - 14:54	25.00
9/6/96 - 16:58	25.00
9/9/96 - 8:11	25.00
9/9/96 - 16:21	25.00
9/10/96 - 8:08	25.00
9/10/96 - 12:07	25.00
9/10/96 - 17:03	25.00
9/11/96 - 9:43	25.00
9/11/96 - 16:28	25.00
9/12/96 - 8:14	25.00
9/12/96 - 16:4	25.00

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Table 3

Results for Samples Collected at a Nominal Flow Rate of 0.5 mL/min.

Sample ID (439C-102-)	Sample Volume (mL)	Collection Time (min.)	Flow Rate (mL/min.)	Measured Concentration (μg DBDPO/L)
1	50	98.25	0.509	0.1
2	51	98.25	0.519	0.1
3	49	98.25	0.499	<0.1
4	49	98.25	0.499	[823] ¹
5	49	98.25	0.499	1.0
6	49	98.25	0.499	0.5
7	49	98.25	0.499	0.6
8	49	98.25	0.499	0.6
9	52	98.25	0.528	0.5
10	52	98.25	0.528	[50.1] ¹
11	52	98.25	0.528	0.2
12	51	98.25	0.519	2.1
13	55	98.25	0.557	1.1
14	51	98.25	0.519	2.3
15	50	98.25	0.509	1.1
16	46	98.25	0.470	0.1
17	44	98.25	0.451	0.6
18	44	98.25	0.451	0.2
19	48	98.25	0.490	0.5
20	45	98.25	0.461	4.1
21	44	98.25	0.451	<0.1
22	44	98.25	0.451	0.4
23	45	98.25	0.461	0.2
24	44	98.25	0.451	4.4
25	44	98.25	0.451	<0.1
26	44	98.25	0.451	<0.1
27	45	98.25	0.461	<0.1
28	45	98.25	0.461	<0.1
29	44	98.25	0.451	3.4
30	45	98.25	0.461	7.4
31	45	98.25	0.461	<0.1
32	45	98.25	0.461	<0.1
33	44	98.25	0.451	0.5
34	46	98.25	0.470	0.3
35	44	98.25	0.451	<0.1
36	44	98.25	0.451	<0.1
37	44	98.25	0.451	5.9
38	42	98.25	0.432	0.3
39	42	98.25	0.432	0.2
40	43	98.25	0.442	<0.1
41	43	98.25	0.442	<0.1
42	43	98.25	0.442	<0.1
43	42	98.25	0.422	<0.1
44	39	98.25	0.394	0.2
45	38	98.25	0.384	<0.1

¹Numbers in brackets [] are extrapolated values.

Table 3 (Continued)

Results for Samples Collected at a Nominal Flow Rate of 0.5 mL/min.

Sample ID (439C-102-)	Sample Volume (mL)	Collection Time (min.)	Flow Rate (mL/min.)	Measured Concentration (μ g DBDPO/L)
46	38	98.25	0.384	< 0.1
47	38	98.25	0.384	< 0.1
48	38	98.25	0.384	< 0.1
49	36	98.25	0.365	< 0.1
50	35	98.25	0.355	0.2
51	35	98.25	0.355	< 0.1
52	36	98.25	0.365	< 0.1
53	36	98.25	0.365	< 0.1
54	36	98.25	0.365	< 0.1
55	36	98.25	0.365	< 0.1
56	36	98.25	0.365	[32.4] ¹
57	39	98.25	0.394	< 0.1
58	40	98.25	0.403	< 0.1
59	43	98.25	0.442	< 0.1
60	44	98.25	0.451	< 0.1
61	43	98.25	0.442	< 0.1
62	43	98.25	0.442	< 0.1
63	43	98.25	0.442	< 0.1
64	40	98.25	0.403	< 0.1
65	32	98.25	0.326	< 0.1
66	30	98.25	0.307	< 0.1
67	30	98.25	0.307	< 0.1
97	53	98.25	0.538	< 0.1

¹Numbers in brackets [] are extrapolated values.

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Table 4

Results for Samples Collected at a Nominal Flow rate of 0.25 mL/min.

Sample ID (439C-102-)	Sample Volume (mL)	Collection Time (min.)	Flow Rate (mL/min.)	Measured Concentration (μg DBDPO/L)
140	40	204	0.194	< 0.1
141	38	204	0.185	< 0.1
142	39	204	0.190	< 0.1
143	39	204	0.190	14.1
144	38	204	0.185	< 0.1
145	39	204	0.190	< 0.1
146	40	204	0.194	< 0.1
148	40	204	0.194	< 0.1
149	39	204	0.190	< 0.1
150	38	204	0.185	< 0.1
151	38	204	0.185	< 0.1
152	39	204	0.190	0.8
153	40	204	0.194	< 0.1
154	39	204	0.190	< 0.1
155	40	204	0.194	< 0.1
156	40	204	0.194	< 0.1
157	40	204	0.194	< 0.1
158	39	204	0.190	< 0.1
159	39	204	0.190	< 0.1
160	39	204	0.185	< 0.1

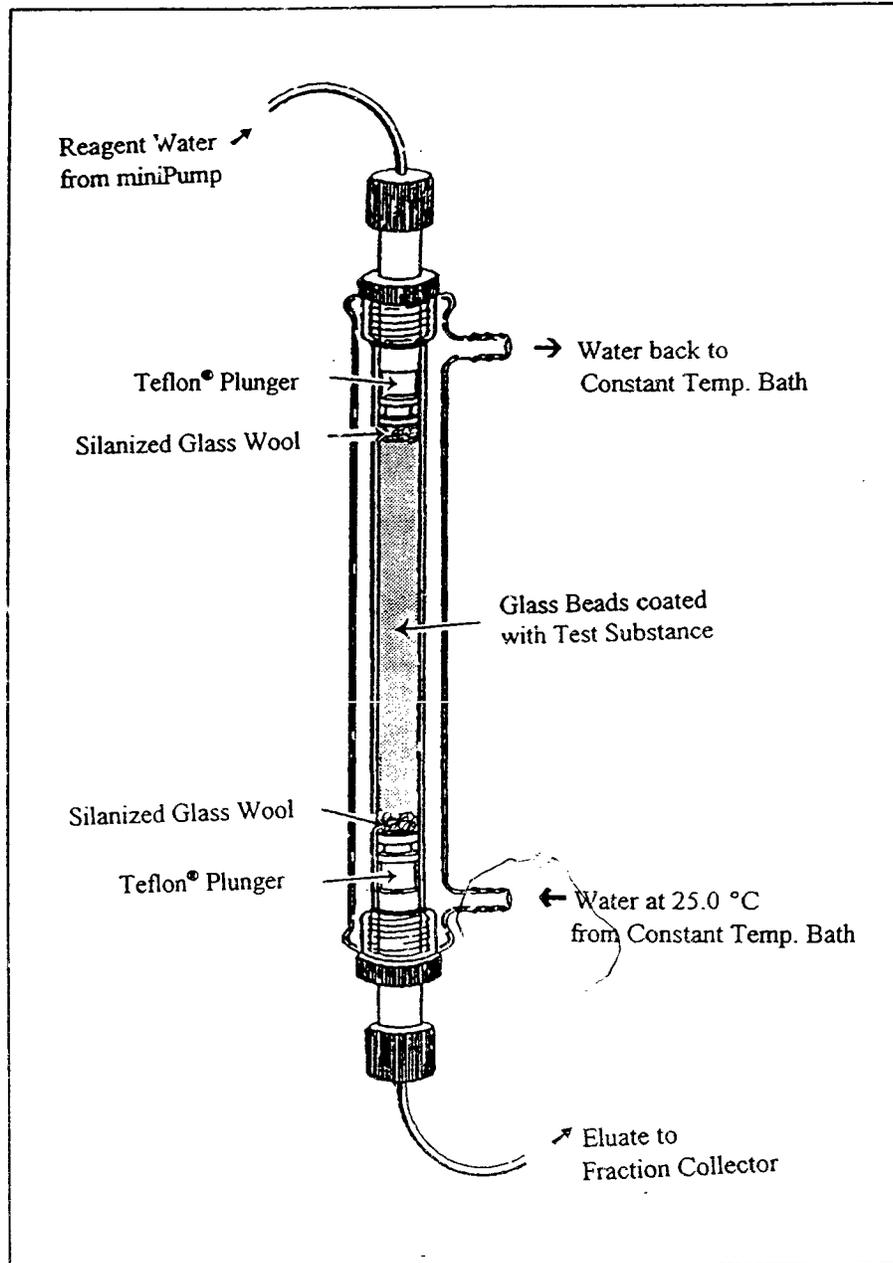


Figure 1. Diagram of generator column.

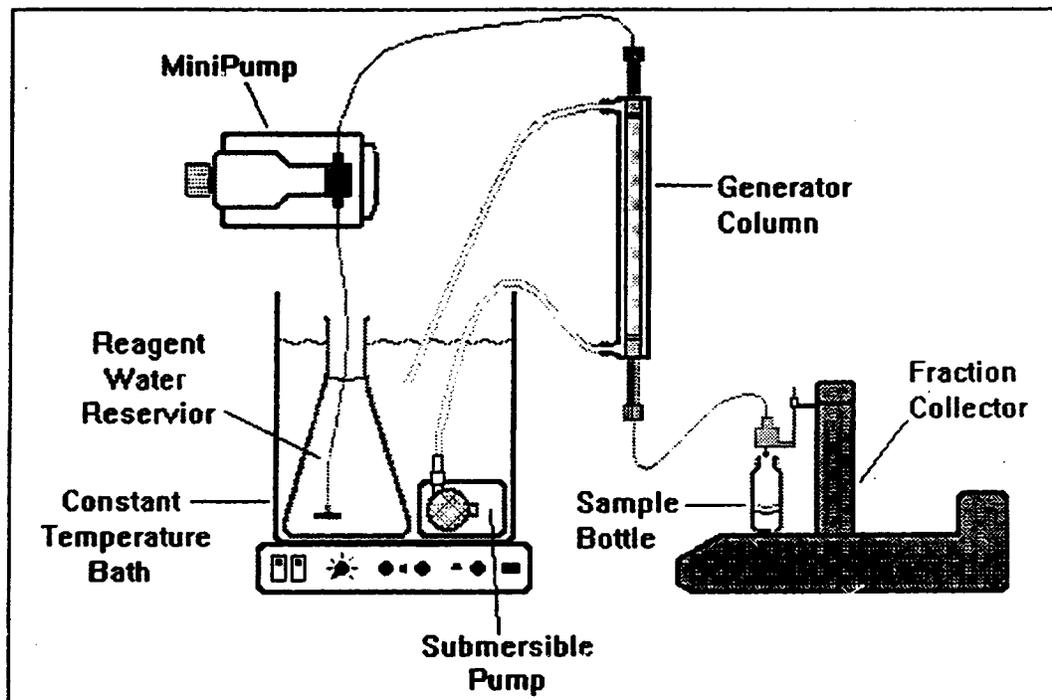


Figure 2. Diagram of apparatus configuration.

0.029

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ANALYTICAL METHOD FLOW CHART

Add 25 mL of ethyl acetate to 4 oz. French square bottles. Label the bottles and place on fraction collector. Collect the eluate fractions from the generator column into the bottles. Cap the bottles as soon as possible after collection and store on lab bench.

↓

Prepare reagent and matrix blanks and fortifications as needed.

↓

Add ethyl acetate to the samples to adjust the volume to ~25 mL. Cap the bottles and shake for about 30 seconds. Pipet the ethyl acetate layer into a round bottom flask.

↓

Repeat the extraction with an additional 20 mL of ethyl acetate.

↓

Combine the extracts and add 200 μ L of phenyl ether (diphenyl oxide, DPO). Measure the volume of water in each sample using a graduated cylinder. Evaporate the extract on a rotovap at 30 to 50°C to less than 0.5 mL.

↓

Pipet the extract from the round bottom flask to a sample tube. Rinse the round bottom flask with 1 to 1.5 mL of ethyl acetate.

↓

Transfer the ethyl acetate to the sample tube.

↓

Repeat the rinsing steps two more times.

↓

Evaporate the sample on a nitrogen evaporator at 30 to 50°C to less than 0.5 mL. Add phenyl ether to bring the sample volume to 1.0 mL.

↓

Transfer the phenyl ether extract into a vial for analysis using GC/ECD.

Figure 3. Analytical method flow chart.

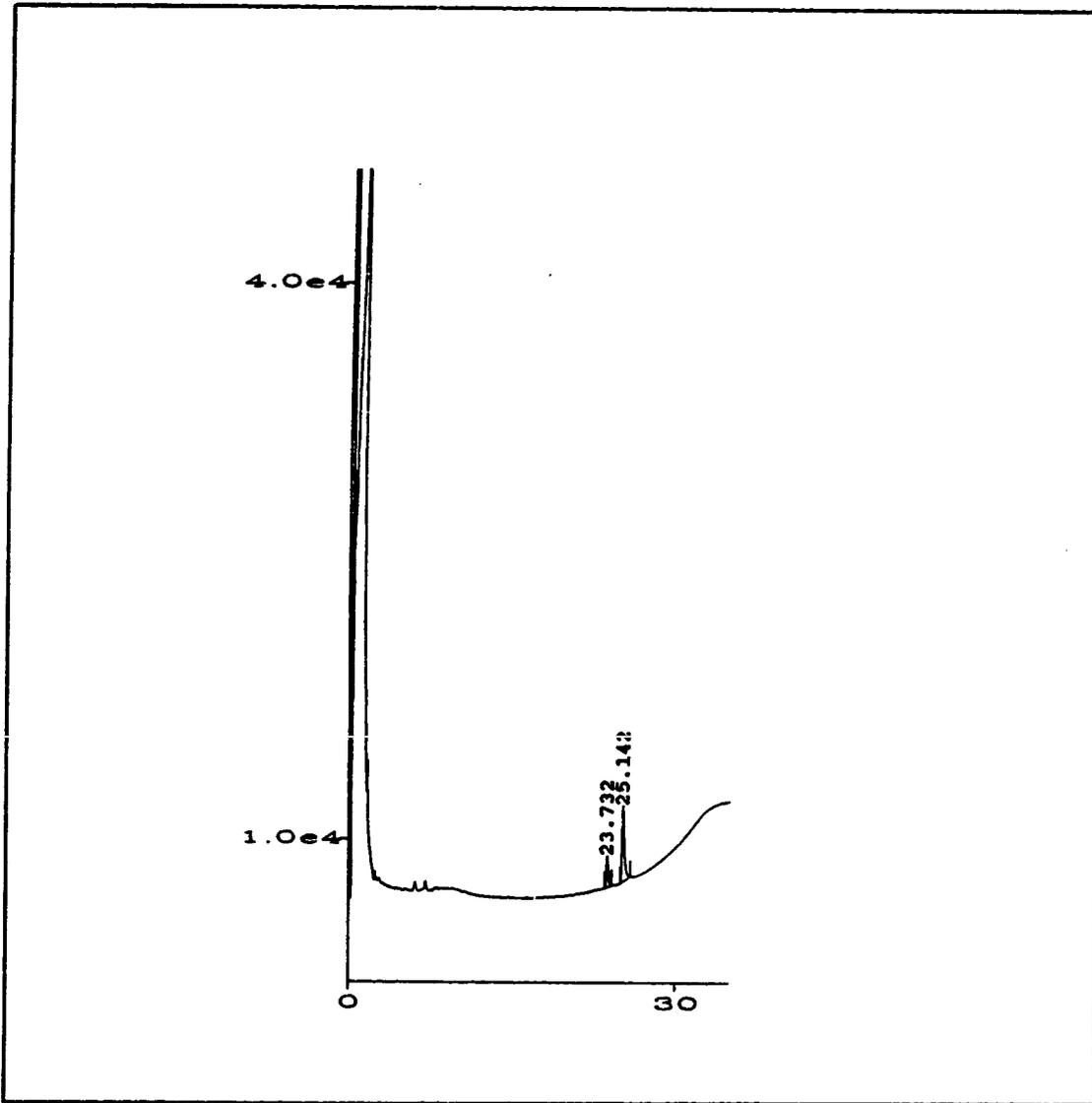


Figure 4. Representative chromatogram of a low-level calibration standard.

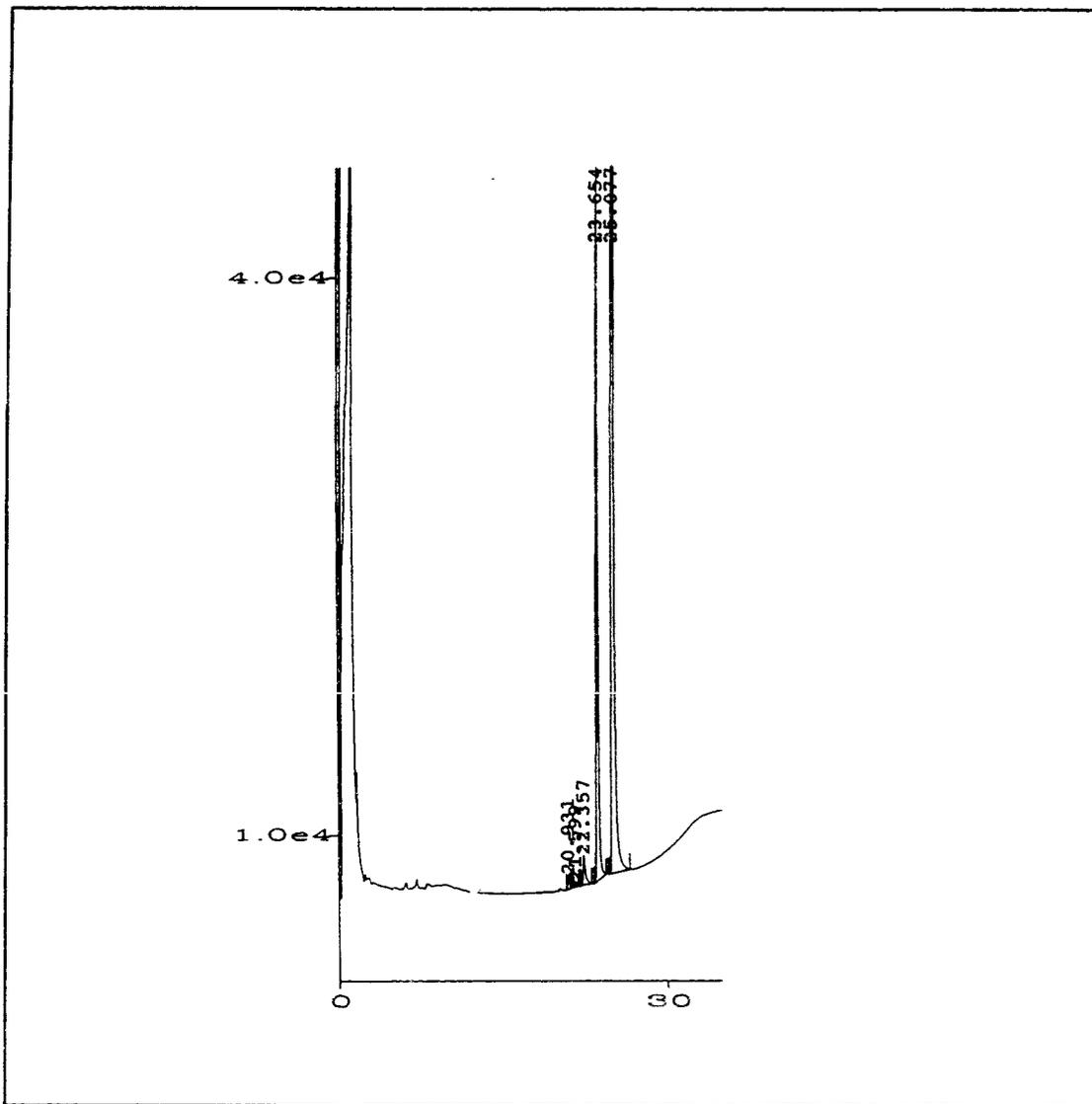


Figure 5. Representative chromatogram of a high-level calibration standard.

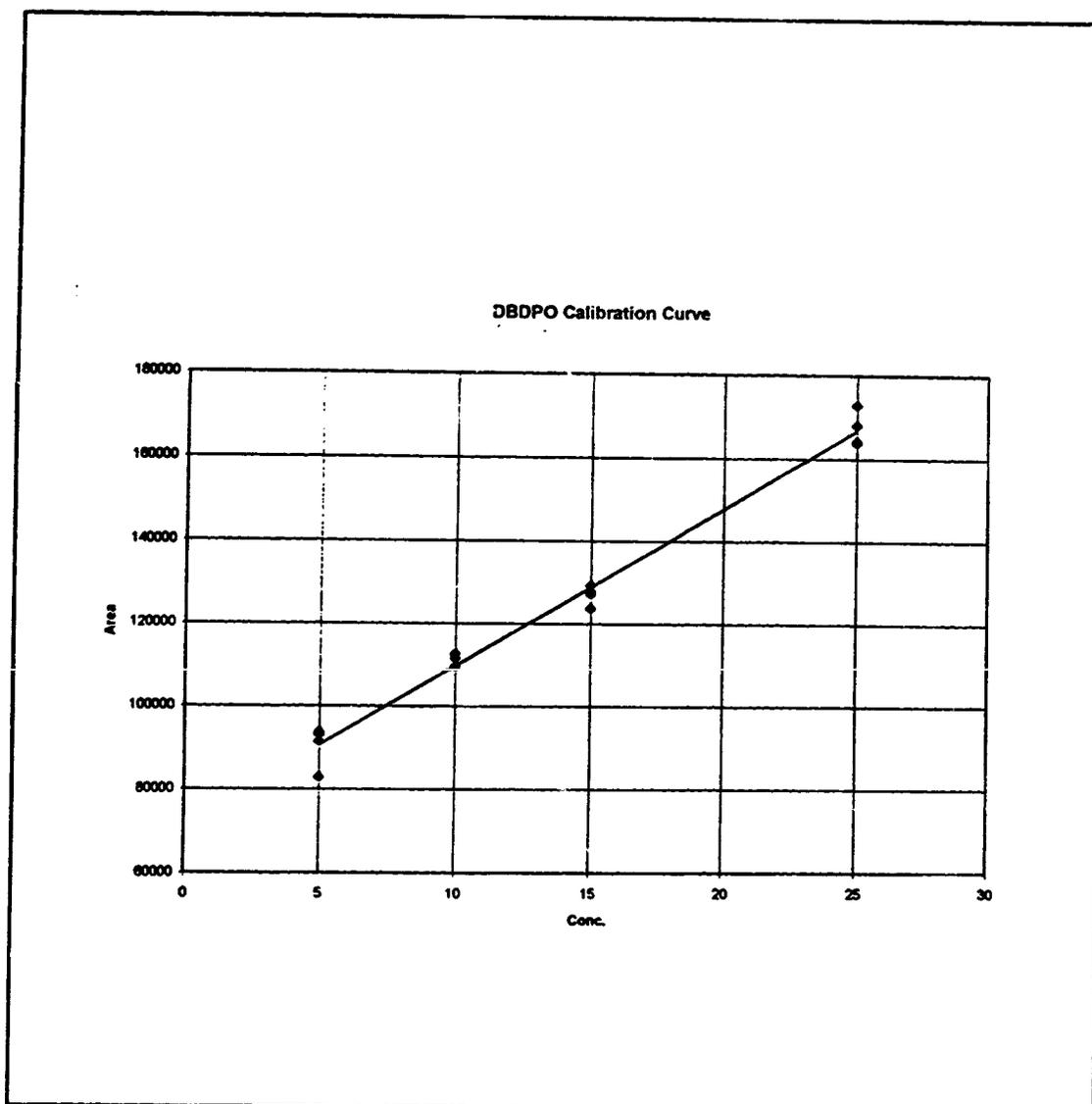


Figure 6. Representative low-level calibration curve.

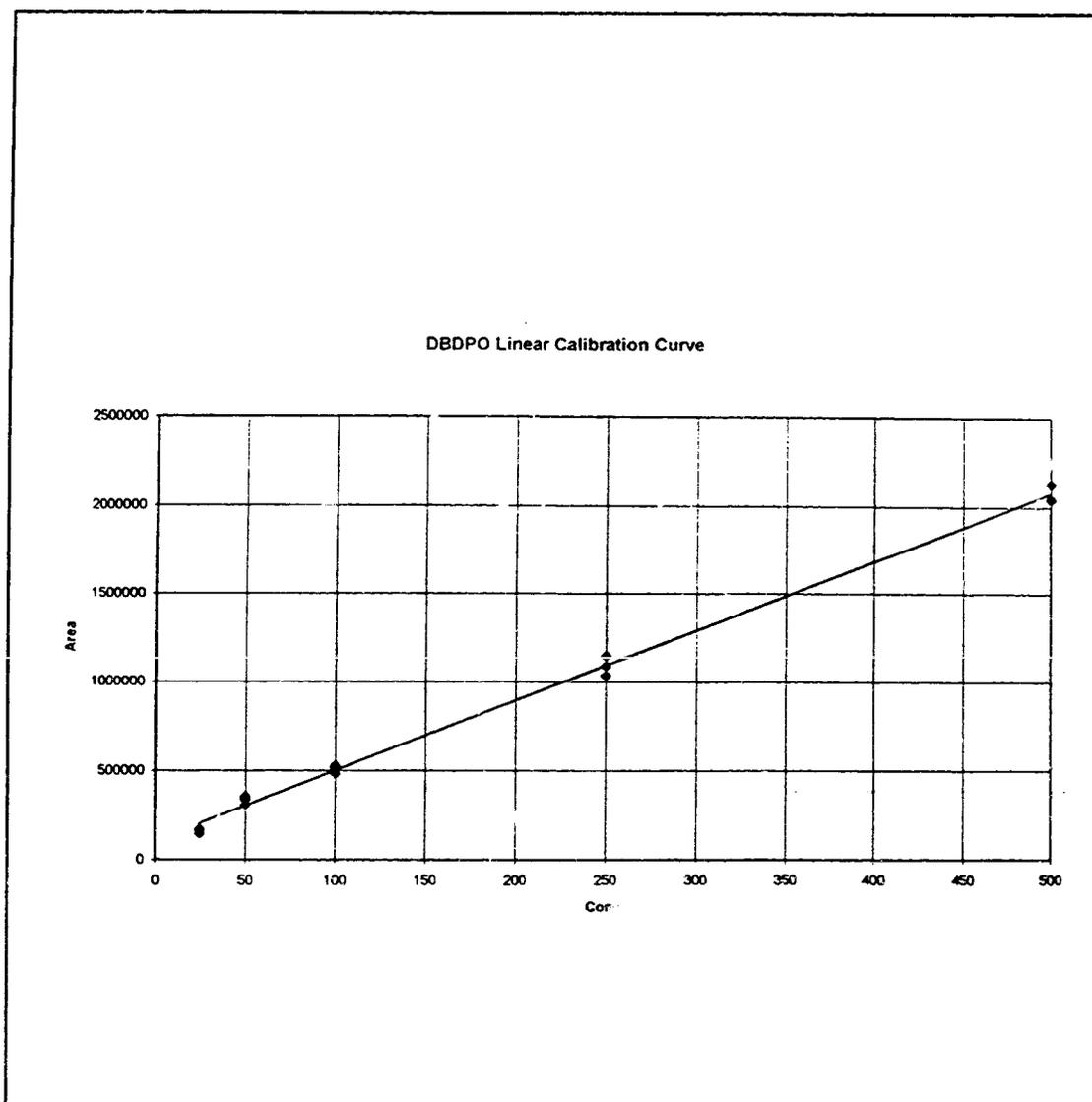


Figure 7. Representative high-level calibration curve.

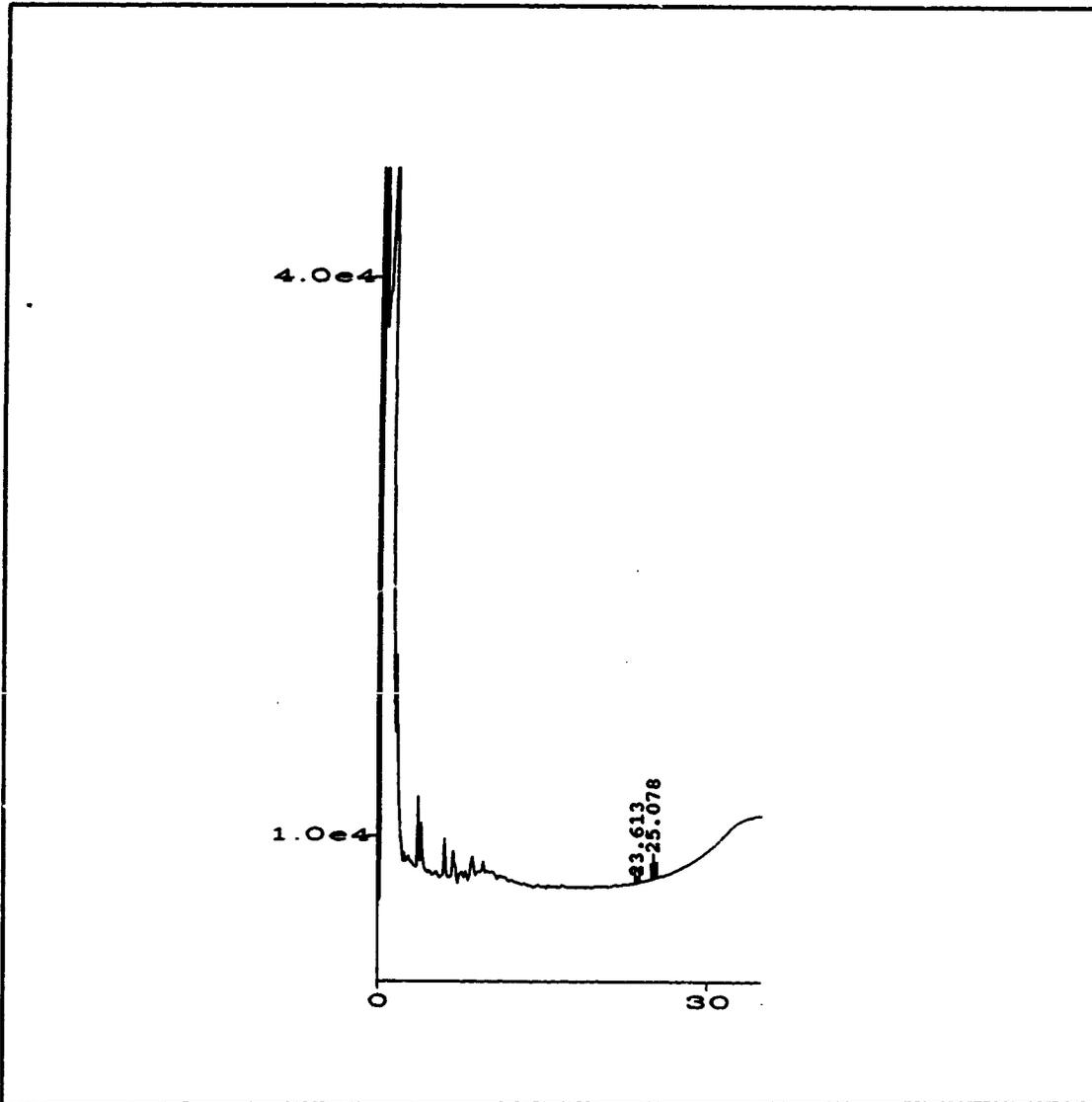


Figure 8. Representative chromatogram of a matrix blank.

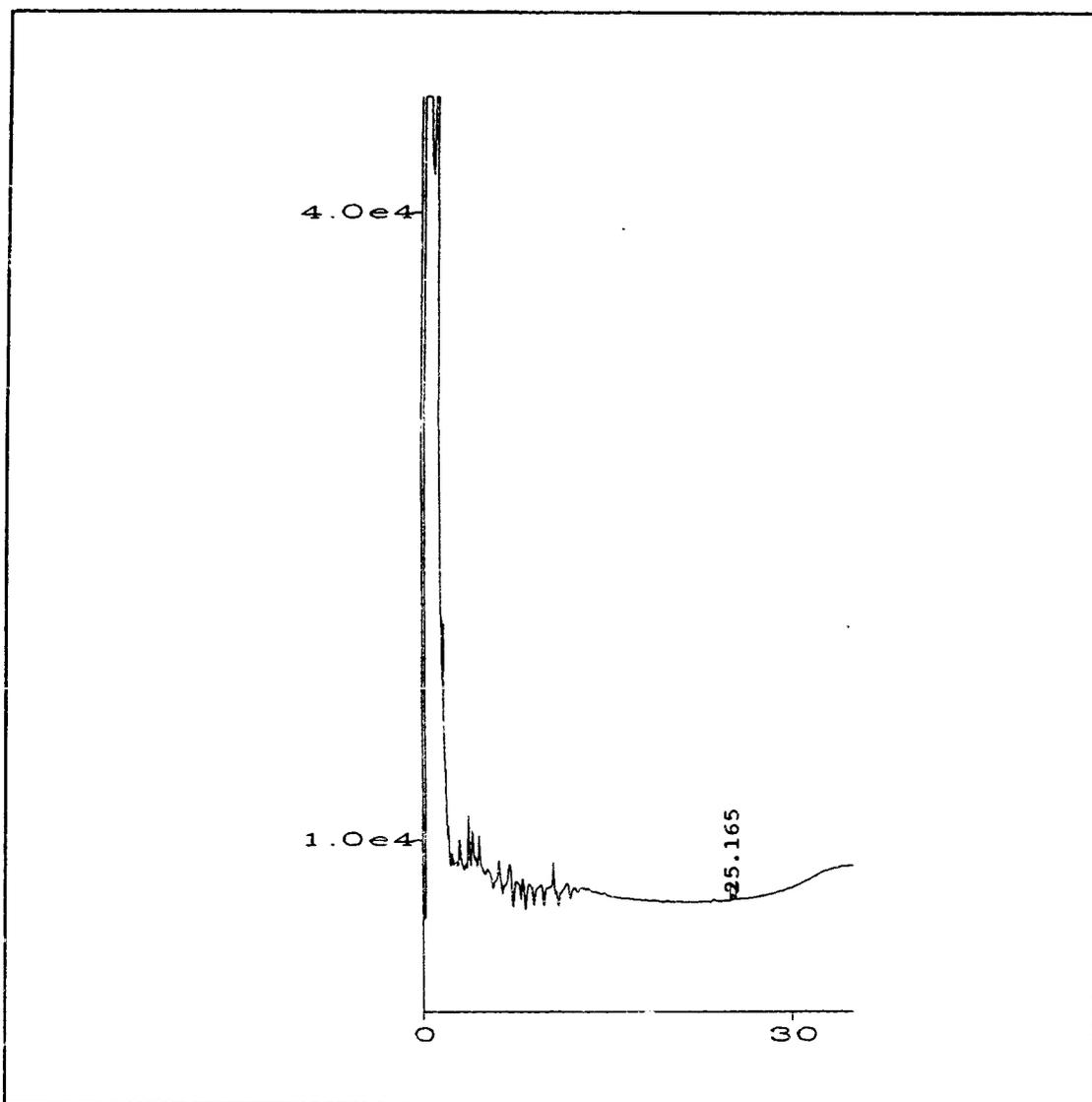


Figure 9. Representative chromatogram of a reagent blank.

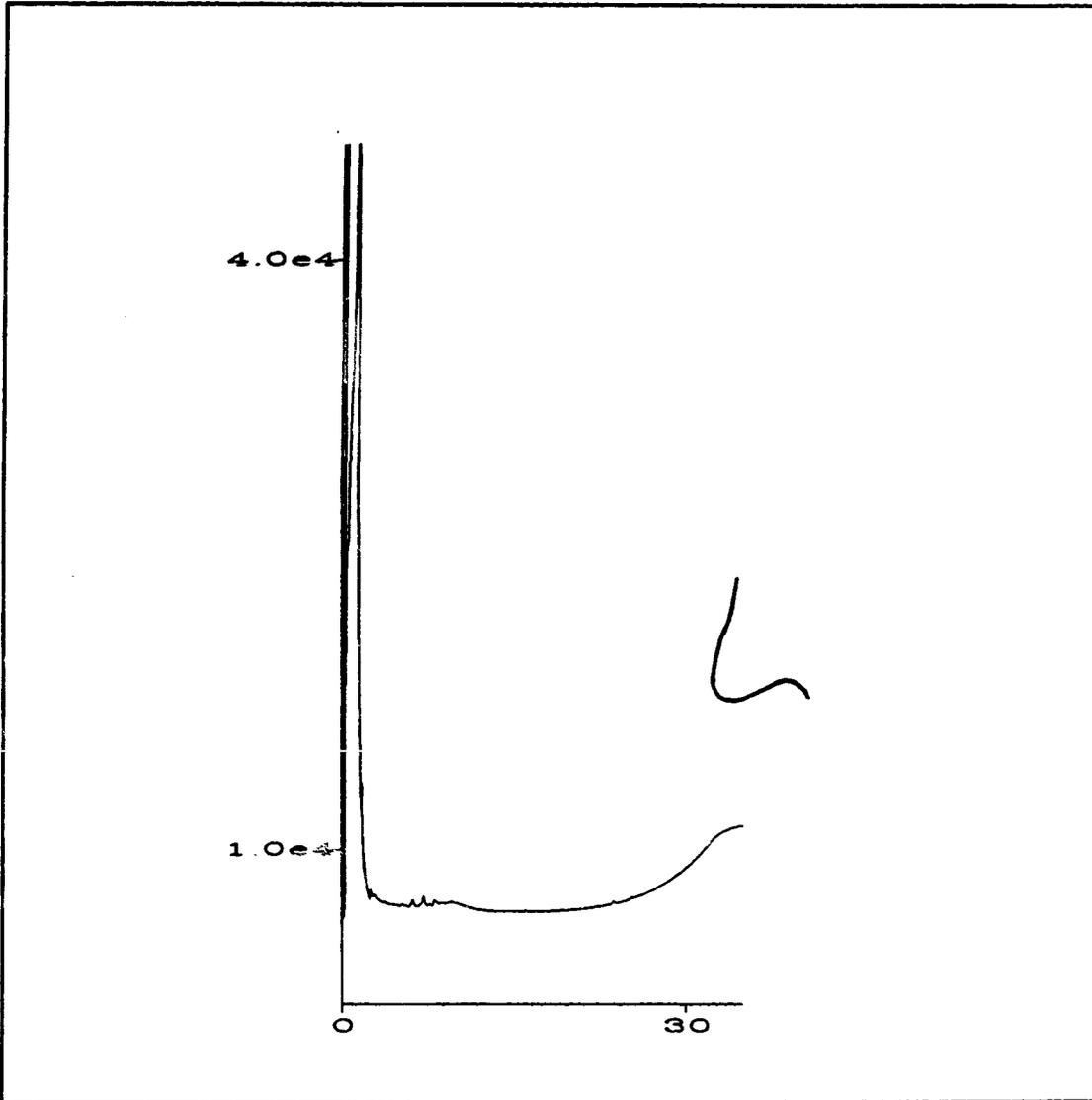


Figure 10. Representative chromatogram of a phenyl ether sample.

0-0-3-5

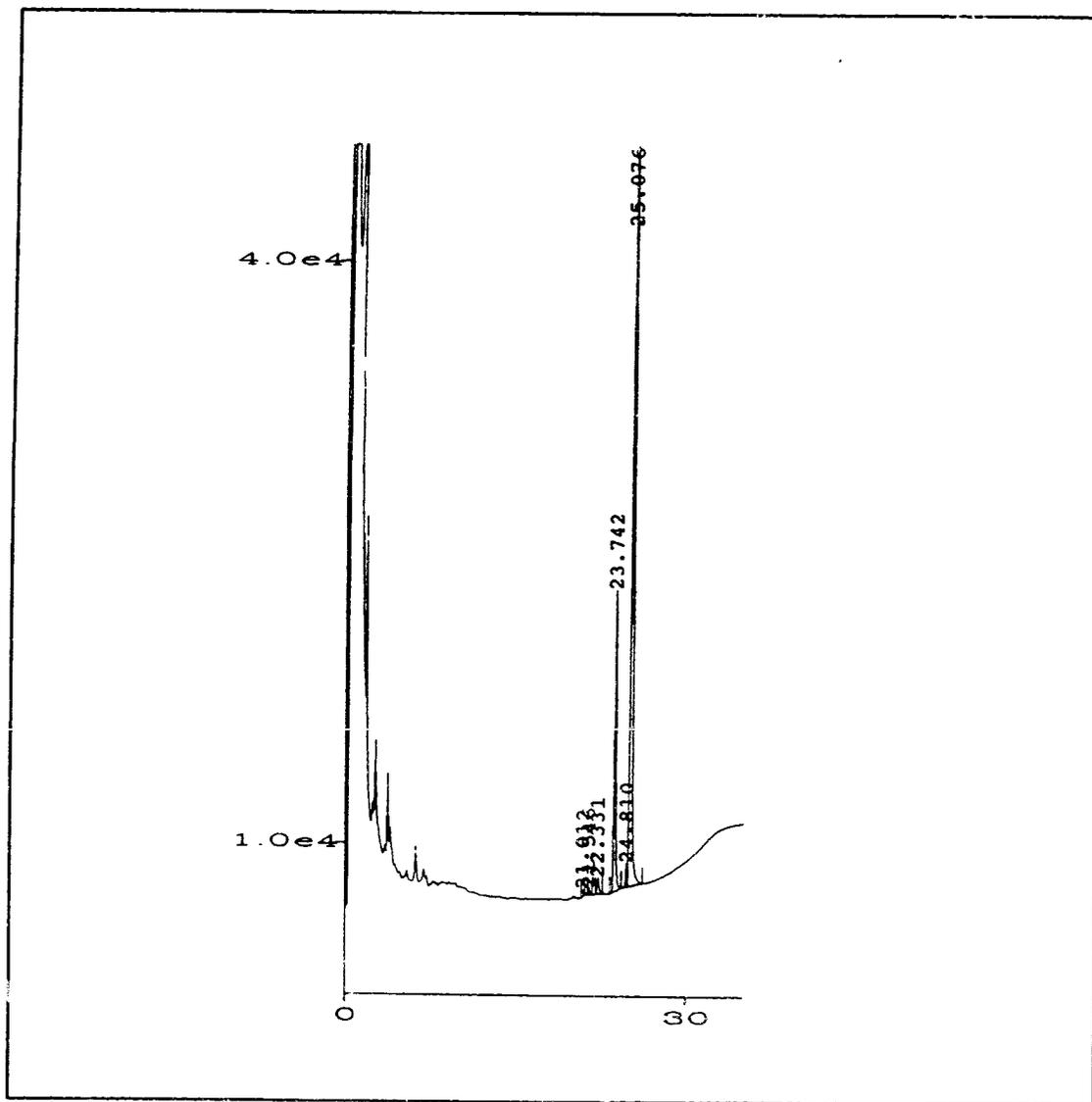


Figure 11. Representative chromatogram of a 5-ppb matrix fortification.

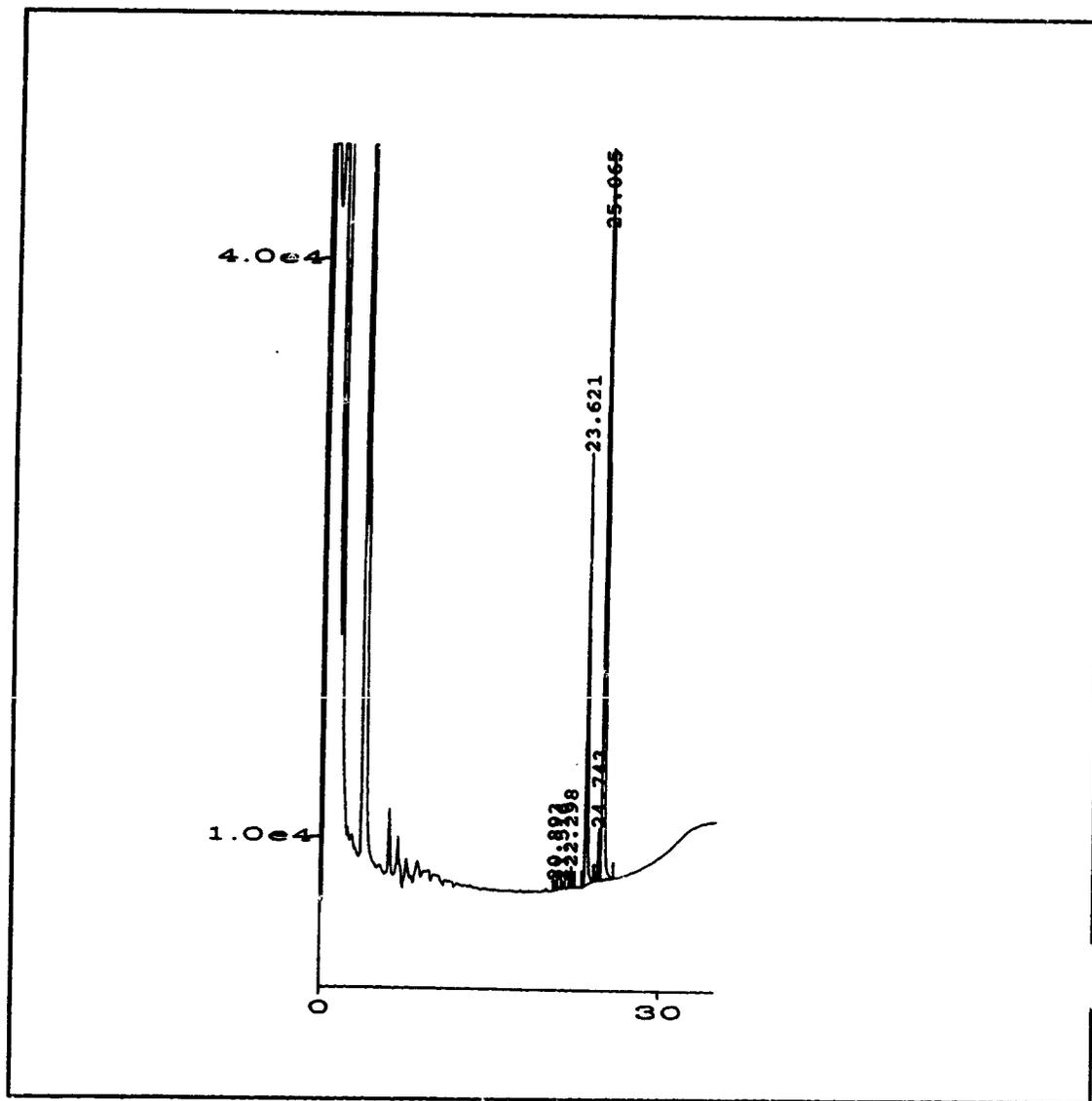


Figure 12. Representative chromatogram of a 1-ppb matrix fortification.

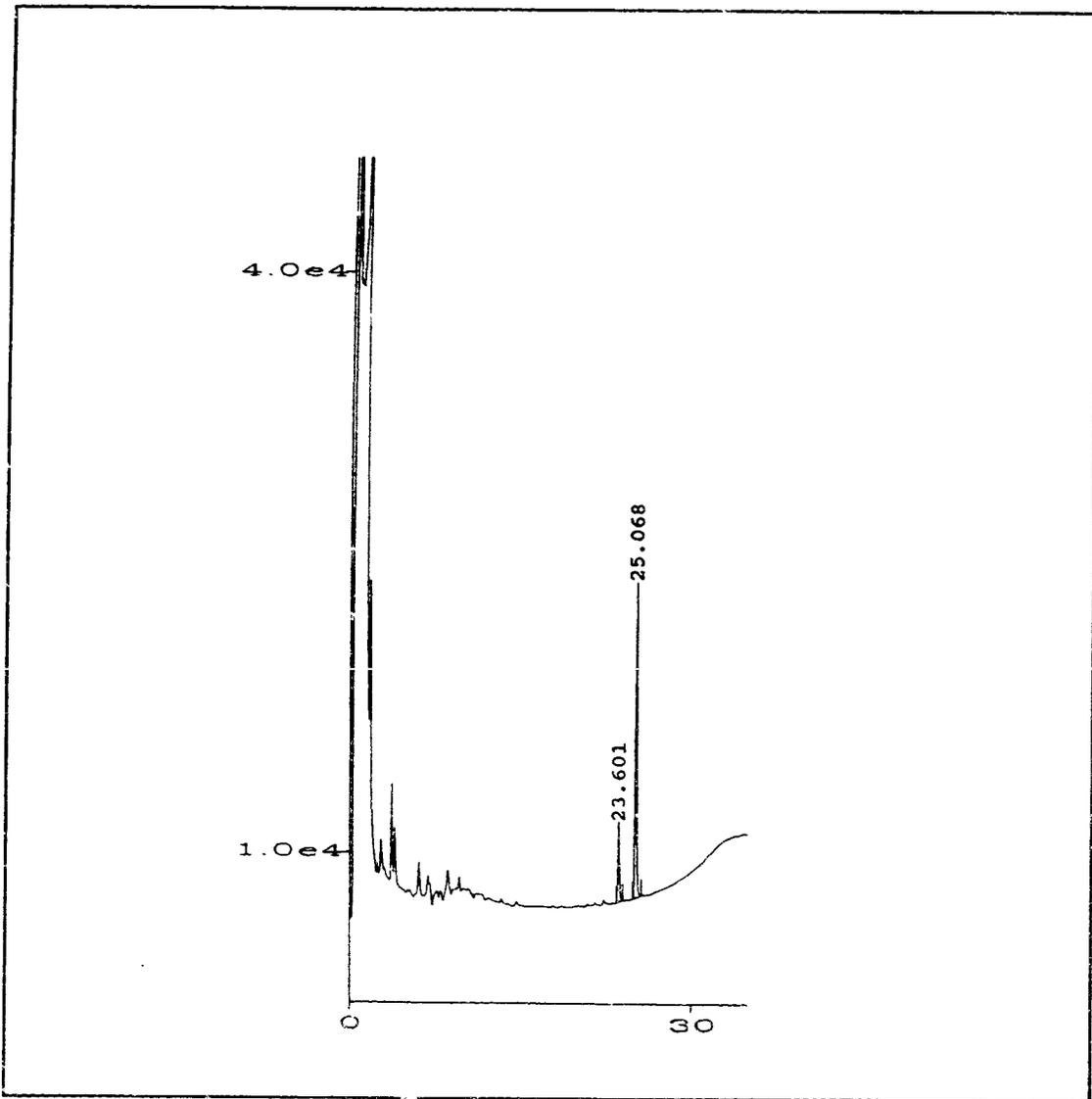


Figure 13. Representative chromatogram of a reagent fortification.

**DECABROMODIPHENYL OXIDE (DBDPO):
DETERMINATION OF n-OCTANOL/WATER PARTITION COEFFICIENT**

WILDLIFE INTERNATIONAL LTD. PROJECT NUMBER: 439C-101

**OPPTS 830.7560 Partition Coefficient (n-Octanol/Water),
Generator Column Method**

AUTHORS:

**Jon A. MacGregor
Willard B. Nixon, Ph.D.**

STUDY INITIATION DATE: March 26, 1997

STUDY COMPLETION DATE: June 16, 1997

Submitted to:

**Chemical Manufacturers Association's
Brominated Flame Retardant Industry Panel
1300 Wilson Boulevard
Arlington, Virginia 22209**



WILDLIFE INTERNATIONAL LTD.

**8598 Commerce Drive
Easton, Maryland 21601
(410) 822-8600**



Page 1 of 70

CYR13

GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT

SPONSOR: Chemical Manufacturers Association's Brominated Flame Retardant Industry Panel

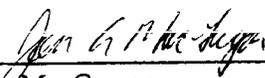
TITLE: Decabromodiphenyl Oxide (DBDPO): Determination of n-Octanol/Water Partition Coefficient

WILDLIFE INTERNATIONAL LTD. PROJECT NUMBER: 439C-101

STUDY COMPLETION: June 16, 1997

This study was conducted in compliance with Good Laboratory Practice Standards as published by the U.S. Environmental Protection Agency in 40 CFR Part 792, 17 August 1989; OECD Principles of Good Laboratory Practices, (OECD)C(81)30 (Final)Annex 2; and Japan MHW/MITI 59 Kikyoku No. 85; EA, Kankiken No. 233; MHW, Eisei No. 38 and MITI, 63 Kikyoku No. 823.

STUDY DIRECTOR:

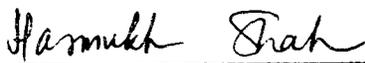


Jon A. MacGregor
Senior Chemist

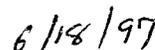


DATE

SPONSOR APPROVAL:



Sponsor



DATE

QUALITY ASSURANCE

SPONSOR: Chemical Manufacturers Association's Brominated Flame Retardant Industry Panel

TITLE: Decabromodiphenyl Oxide (DBDPO): Determination of n-Octanol/Water Partition Coefficient

WILDLIFE INTERNATIONAL LTD. PROJECT NO. 439C-101

This study was examined for compliance with Good Laboratory Practice Standards as published by the U.S. Environmental Protection Agency in 40 CFR Part 792, 17 August 1989; OECD Principles of Good Laboratory Practices, (OECD)C(81)30 (Final)Annex 2; and Japan MHW/MITI 59 Kikyoku No. 85 EA, Kankiken No. 233: MHW, Eisei No. 38 and MITI, 63 Kikyoku No. 823. The dates of all inspections and audits and the dates that any findings were reported to the Study Director and Laboratory Management were as follows:

ACTIVITY:	DATE CONDUCTED:	DATE REPORTED TO:	
		STUDY DIRECTOR:	MANAGEMENT:
Standard Stock Preparation	March 26, 1997	March 26, 1997	March 31, 1997
Matrix Fortifications Preparation and Sample Collection	March 31, 1997	March 31, 1997	April 1, 1997
Matrix Fortifications Preparation and Sample Extraction	April 3, 1997	April 3, 1997	April 3, 1997
Matrix Fortifications	April 22, 1997	April 22, 1997	April 23, 1997
Test Substance Preparation	April 29, 1997	April 29, 1997	April 30, 1997
Matrix Fortifications Preparation	April 30, 1997	April 30, 1997	May 1, 1997
Draft Report and Data	May 21 & 22, 1997	May 22, 1997	May 23, 1997
Final Report	June 16, 1997	June 16, 1997	June 16, 1997

Susan L. Hopper
 Susan L. Hopper
 Senior Quality Assurance Representative

6-16-97
 DATE

REPORT APPROVAL

SPONSOR: Chemical Manufacturers Association's Brominated Flame Retardant Industry Panel

TITLE: Decabromodiphenyl Oxide (DBDPO): Determination of n-Octanol/Water Partition Coefficient

WILDLIFE INTERNATIONAL LTD. PROJECT NO.: 439C-101

This report was reviewed by the individuals involved in the conduct and management of the study, and was found to be an accurate reflection of the methods used, data collected and results of the study.

STUDY DIRECTOR:

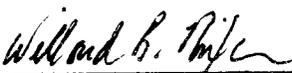


Jon A. MacGregor
Senior Chemist

6/16/97

DATE

MANAGEMENT:



Willard B. Nixon, Ph.D.
Manager, Analytical Chemistry

6/16/97

DATE

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SUMMARY

SPONSOR:	Chemical Manufacturers Association's Brominated Flame Retardant Industry Panel
SPONSOR'S REPRESENTATIVE:	Hasmukh Shah, Ph.D.
LOCATION OF STUDY, RAW DATA AND A COPY OF THE FINAL REPORT:	Wildlife International Ltd. Easton, Maryland 21601

WILDLIFE INTERNATIONAL LTD.	
PROJECT NUMBER:	439C-101
TEST SUBSTANCE:	Decabromodiphenyl Oxide (DBDPO)
STUDY:	Decabromodiphenyl Oxide (DBDPO): Determination of n-Octanol/Water Partition Coefficient
TEST DATES:	Experimental Start - March 31, 1997 Experimental Termination - May 5, 1997

SUMMARY:	The \log_{10} octanol/water partition coefficient (K_{ow}) of DBDPO was determined to be 6.265 at $25 \pm 0.05^\circ\text{C}$ using the generator column method.
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INTRODUCTION

This study was conducted by Wildlife International Ltd. for Chemical Manufacturers Association's (CMA) Brominated Flame Retardant Industry Panel at the Wildlife International Ltd. analytical chemistry facility in Easton, Maryland. Tests were performed using the generator column method. Samples were eluted from a generator column and analyzed from March 31 to May 5, 1997. Raw data generated by Wildlife International Ltd. and a copy of the final report are filed under Project Number 439C-101 in archives located on the Wildlife International Ltd. site.

OBJECTIVE

The objective of this study was to determine the n-octanol/water partition coefficient of decabromodiphenyl oxide (DBDPO) at 25.0°C using a generator column method.

EXPERIMENTAL DESIGN

A single generator column was prepared for the definitive test. The column was packed with Chromosorb WHP support and loaded with a nominal 0.1% solution of the test substance in octanol. Dilutions of the final test substance solution in octanol were analyzed. The column temperature was maintained at $25 \pm 0.05^\circ\text{C}$ and reagent water saturated with octanol was pumped through it at approximately 1.0 mL per minute to elute the test substance. Samples of the eluate were collected and analyzed to determine the concentration of the test substance in the aqueous fractions.

MATERIALS AND METHODS

This study was conducted according to procedures outlined in the protocol, "Decabromodiphenyl Oxide (DBDPO): Determination of n-Octanol/Water Partition Coefficient" (Appendix I). The protocol was based on procedures outlined in OPPTS 830.7560 (1). The generator column method was used to determine the partition coefficient of the test substance.

Test Substance

The test substance consisted of a composite of decabromodiphenyl oxide (DBDPO) samples received from three manufacturers. The material's identity and date received from each of the manufacturers is given below:

<u>Manufacturer</u>	<u>Lot/Batch</u>	<u>Date Received</u>	Wildlife International Ltd. <u> ID No. </u>
Great Lakes Chemical Corp.	5480DH24A	October 26, 1995	3460
Albemarle Corp.	4449-1N	December 20, 1995	3518
Bromine Compounds Ltd.	950289	January 30, 1996	3547

An equal part (300 g) of each of the manufacturer's DBDPO material was placed in a 2-L, high density polyethylene (HDPE) bottle. The bottle was placed on a reciprocating shaker for two hours. The composite test substance was assigned Wildlife International Ltd. identification number 3578. Subsamples of the composite test substance were shipped to Albemarle Corporation for characterization and homogeneity analyses. The analyses were performed on March 13, 1996. The results of the analyses indicated the composite test substance was homogeneous and contained the following components:

Octabromodiphenyl oxide	0.04%
Nonabromodiphenyl oxide	2.5%
Decabromodiphenyl oxide	97.4%

The composite test substance was stored under ambient conditions.

Reagent Water

The reagent water used in this study met the specifications for ASTM Type II water. The water was obtained from a well located on the Wildlife International Ltd. site. The well water was

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pumped through a series of filters to remove microorganisms and particles greater than 0.2 μm . The water was further purified using a Culligan® Hi-Flo 1 Water Softener, a Culligan® S-Series Reverse Osmosis System, and a Barnstead NANOpure® ultrapure water system. The resistivity of the purified reagent water used for this study was at least 17.3 megohm-cm. The reagent water was saturated with octanol prior to use in this study.

Solvents

1-Octanol (certified), Fisher Chemical (Fairlawn, NJ), catalog number A 402-4, was used to prepare a stock solution of the test substance. Dimethylformamide (DMF), Burdick and Jackson (Muskegon, MI, catalog number 076-1), was used to prepare stock solutions of the test substance for matrix fortifications. Diphenyl ether (DPO), purchased from Fluka Chemical (Switzerland, catalog number 42730), was used to prepare calibration standards and final sample volumes. Ethyl Acetate (EtoAc), Burdick and Jackson (catalog number 300-4), was used as an extraction solvent for this study. A solution of 5% KOH, Mallinkrodt (catalog number 6984) in methanol, Burdick and Jackson (catalog number 230-4) along with a 10% phosphoric acid (85%) solution, Fisher (catalog number A242-212) and tetrahydrofuran (THF), Fisher (catalog number T425-1) were used to rinse all glassware prior to use. All solvents used for this study were either suitable for HPLC and residue analysis or certified reagents.

Preparation of Generator Column

The generator column was supplied by At-Mar Glass Co. (Kennett Square, PA). The glass column was ~20 cm long with an internal diameter of ~6 mm, and was joined to a section with an internal diameter of ~9 mm. The entire column was enclosed in a water jacket for temperature control. A diagram of the generator column is presented in Figure 1.

The inert support material used in the generator column was Chromosorb WHP (100 - 120 mesh), and was purchased from Supelco Inc. (Bellefonte, PA, catalog number 2-0159). A small plug of silanized glass wool was placed in the column just above the enlarged section, the column

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was filled with the support material, and another plug of glass wool was added to the top of the column. Gentle tapping and vibration were used to facilitate packing of the column.

A stock solution of the composite test substance was prepared by combining 25 mg of test substance with 25 g of octanol and sonicating for approximately 10 minutes to dissolve the test substance. The nominal concentration of the stock solution was 0.1% test substance by weight or 0.830 mg/mL, based on the density of octanol (0.827 g/mL). The stock solution was centrifuged at approximately 2000 rpms for approximately five minutes and then filtered through a 0.20 μ m acrodisc to remove any undissolved test substance. Three subsamples of the final filtered stock solution were diluted in diphenyl ether (DPO) and analyzed to determine the concentration of test substance in the octanol. An aliquot of the stock solution (15 mL) was also used to charge the generator column. The stock solution was loaded onto the support material by applying gentle suction at the bottom of the column with the aid of a vacuum pump apparatus.

The column was then back-flushed with reagent water saturated with octanol by applying gentle suction at the top of the column to remove any entrapped air. End fittings were attached to the column after it had been flushed.

Apparatus Configuration

A Nesslab Model IC-515 Constant Temperature Water Bath (Nesslab Instruments, Inc., Portsmouth, NH) was used to maintain the test temperature ($25.0 \pm 0.05^\circ\text{C}$) throughout the experiment. The constant temperature bath was filled with water. The temperature of the water bath was monitored using an ASTM 45C kinematic viscosity thermometer (Ever Ready Thermometer Company, West Paterson, NJ).

A Teel Model 1P680A submersible pump (Dayton Electric Mfg. Co., Chicago, IL) was placed in the constant temperature bath, and was used to pump a continuous stream of water through the jacket surrounding the generator column in order to maintain a constant temperature.

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A 2-L Erlenmeyer flask was used as a reservoir for the reagent water saturated with octanol that was being pumped through the generator column. The flask was submerged in the constant temperature bath. The inlet line of a Waters Model 510 solvent delivery system (Waters Associates, Milford, MA) was placed in the 2-L flask, and the top of the flask covered with aluminum foil. The pump was used to control the flow rate of the reagent water through the generator column.

The eluate from the generator column was directed to 500 mL separatory funnels containing appropriate volumes of extraction solvent. The individual samples were collected manually. A diagram of the apparatus configuration is presented in Figure 2.

Aqueous Sample Collection

Prior to the collection of aqueous samples, the pump was set to deliver approximately 0.5 mL reagent water saturated with octanol per minute overnight to equilibrate the system. Following the equilibration period, the pump was set to deliver approximately 1.0 mL of reagent water saturated with octanol per minute through the generator column. The eluate was collected dropwise into 500 mL separatory funnels containing 100 mL of the extraction solvent. Three consecutive individual aqueous samples were collected at approximately four hour intervals into three separate 500 mL separatory funnels. The volume of sample collected ranged from 255 to 270 mL.

Analytical Method

The analytical method consisted of extracting the aqueous samples with ethyl acetate, evaporating the solvent, and reconstituting the sample residues in diphenyl ether (DPO). Prior to use, all glassware was base-washed using a 5% by weight potassium hydroxide in methanol solution, acid-washed using a 10% aqueous phosphoric acid solution, and then rinsed with reagent water. Acetone was then used to dry the glassware, followed by successive rinses with tetrahydrofuran and ethyl acetate solvents. Ethyl acetate (100 mL) was added to each separatory funnel prior to sample collection. After collection, each separatory funnel was stoppered and shaken for approximately one minute. The layers were allowed to separate and several crystals of sodium chloride were added, as necessary, to break any emulsions. The lower aqueous layer was drained into an appropriately

sized Erlenmeyer flask and the ethyl acetate layer was then drained into a 250 mL round-bottom flask. The solvent volume was reduced to approximately 10 mL using rotary evaporation at 30 - 50°C. The aqueous portion was carefully poured back into the separatory funnel and 100 mL of ethyl acetate extraction solvent was added to each flask as a rinse, prior to adding it to the separatory funnels. This extraction procedure was repeated two additional times. The extracts were combined each time in the round-bottom flask. Following extraction, the volume of the aqueous sample was measured using an appropriately sized graduated cylinder. The final combined ethyl acetate extracts were reduced to approximately 2 mL using rotary evaporation at 30 - 50°C and then quantitatively transferred to individual test tubes. The extracts were then reduced to the residual octanol using a nitrogen evaporator at 30 - 50°C. The final residues were reconstituted by additions of diphenyl ether (DPO) to a final volume of 1.0 mL. The final volumes were based on visual comparison to a blank test tube containing 1.0 mL DPO (measured using a class A volumetric pipet). The final DPO extracts were transferred to labelled autosampler vials for analysis.

The analytical preparation procedure used for the analysis of samples of the octanol stock solution consisted of diluting the samples using DPO. Subsamples of 0.200 mL were diluted to a final volume of 100 mL and mixed. Aliquots of the diluted solutions were placed in autosampler vials for analysis.

Concentrations of DBDPO in the samples were determined using a Hewlett-Packard Model 5890 Gas Chromatograph (GC). The gas chromatograph was equipped with an electron capture detector (ECD). Chromatographic separations were achieved using an Rtx-1 capillary column (15 m X 0.53 mm, 0.1 μ m film thickness) manufactured by Restek Corporation (Bellefonte, PA). Chromatographic grade helium was used as the carrier gas, and argon/methane was used as the auxiliary gas. The instrument parameters are summarized in Table 1. A method flow chart is provided in Figure 3.

Preparation of Quality Control Samples

Fortification solutions of DBDPO were prepared in dimethylformamide (DMF) at concentrations of 1.00 mg/L and 100 mg/L. These standards were used to fortify matrix samples at 0.200 and 5.00 μg DBDPO/L. One reagent (method) blank, two matrix blanks and two matrix fortifications (0.200 ppb and 5.00 ppb) were prepared and analyzed along with the aqueous samples collected from the generator column.

Calibration Curve and Quantitation

Calibration standards of DBDPO were prepared in diphenyl ether (DPO). The standards were prepared using the test substance, and ranged in concentration from 10 to 100 μg DBDPO/L. A set of calibration standards was analyzed before and after the set of samples, and a standard was injected a minimum of every five samples during the analytical run. The sum of the peak areas for the two major peaks (25.6 and 26.9 minutes) was used to determine the instrument response. A calibration curve was constructed from the linear regression equation using the respective concentration versus summed peak area responses of the calibration standards (Figure 4). Representative chromatograms of low and high calibration standards are shown in Figures 5 and 6, respectively. The concentration of DBDPO in the samples was determined by substituting the summed peak area responses into the applicable linear regression equation generated from the calibration curve as follows:

$$\text{DBDPO in Sample } (\mu\text{g/L}) = [(\text{Summed Peak Area} - \text{Y-Intercept}) / \text{Slope}] \times \text{Dilution Factor}$$

$$\text{Molar Concentration (M)} = \frac{\text{Measured DBDPO Concentration (g/L)}}{\text{Molecular Weight (DBDPO)}} \times 1$$

$$\% \text{ Recovery} = \frac{\text{Measured DBDPO Concentration } (\mu\text{g/L})}{\text{Nominal DBDPO Concentration } (\mu\text{g/L})}$$

The instrument limit of detection (LOD) for this study was set based upon the injection volume (2.0 μL) and the lowest calibration standard concentration (10.0 μg DBDPO/L). The LOD was set at 20 ng of DBDPO injected.

The limit of quantitation (LOQ) was $0.0400 \mu\text{g DBDPO/L}$ calculated as the product of the lowest calibration standard ($10.0 \mu\text{g DBDPO/L}$) and the dilution factor of the matrix blank sample (0.0040).

RESULTS AND DISCUSSION

Quality Control Samples

No interferences were observed at or above the LOQ in the one reagent and two matrix blank samples. A chromatogram of the matrix blank is shown in Figure 7.

The percent recovery of the 0.200 and $5.00 \mu\text{g DBDPO/L}$ matrix fortifications were 118 and 99%, respectively, of nominal concentrations. The mean recovery was calculated as 109% of nominal concentration. Representative chromatograms of the 0.200 and $5.00 \mu\text{g DBDPO/L}$ matrix fortification samples are shown in Figures 8 and 9, respectively.

Column Elution

The temperature of the water bath remained constant at 25.0°C throughout the experiment (Table 2).

The nominal flow rate of reagent water through the generator column was measured prior to the start of sample collection. Flow rates were also calculated based on the volume and collection time of each sample that was analyzed. The pump setting was 1.0 mL/min. and the flow rate was measured at 1.0 mL/min. The calculated flow rates for samples averaged 0.98 mL/min. and ranged from 0.94 to 1.0 mL/min. (Table 3).

The results from the analyses of aqueous samples eluted from the generator column are presented in Table 3. A representative chromatogram is shown in Figure 10. The mean concentration of DBDPO measured in these samples was $0.0400 \mu\text{g DBDPO/L}$, or $4.17 \times 10^{-11} \text{ M}$ (molecular weight of DBDPO is 959 g/mole).

The results from the analyses of the octanol stock solution are presented in Table 4. A representative chromatogram is shown in Figure 11. The mean concentration of DBDPO measured in these samples was 0.0738 g DBDPO/L, or 7.69×10^{-5} M (molecular weight of DBDPO is 959 g/mole).

CONCLUSIONS

The octanol/water partition (K_{ow}) coefficient was calculated from the following equation:

$$K_{ow} = \frac{\text{Measured Concentration in Octanol (M)}}{\text{Measured Concentration in Aqueous Samples (M)}}$$

Based on the results from octanol samples collected from the stock solution and aqueous samples collected from the generator column, the mean octanol/water partition coefficient (K_{ow}) for DBDPO was determined to be 1.84×10^6 ($\log_{10} K_{ow}$ is 6.265).

REFERENCES

1. **U.S. Environmental Protection Agency.** 1996. Product Properties Test Guidelines, OPPTS 830.7560, Partition Coefficient (n-Octanol/Water), Generator Column Method. Washington, D.C.

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Table 1

Typical Gas Chromatographic Operational Parameters

INSTRUMENT:	Hewlett-Packard Model 5890 Gas chromatograph (GC) Equipped with a Model G1030A Chemstation
DETECTOR:	Hewlett-Packard Electron-Capture Detector (ECD)
ANALYTICAL COLUMN:	Restek-Rtx-1 Column (15 m x 0.53 mm, 0.1 μ m film thickness)
INJECTOR TEMPERATURE:	300°C
OVEN:	Initial temperature: 150°C Initial hold time: 2 minutes Ramp: 5°C/minute Final temperature: 300°C Final hold time: 3 minutes
DETECTOR TEMPERATURE:	320 °C
INJECTION VOLUME:	2 μ L; splitless
CARRIER GAS:	Helium, ~4-6 mL/minute (~ 4 p.s.i., CHP)
MAKE-UP GAS:	Argon/Methane, ~45 mL/minute
DECABROMODIPHENYL OXIDE (DBDPO) PEAK RETENTION TIME:	25.6 and 26.9 minutes

Table 2

Water Bath Test Temperatures

Date & Time of Observation	Water Bath Temperature (°C)
4/30/97 6:30	25.00
4/30/97 11:00	25.00
4/30/97 15:15	25.00
4/30/97 19:43	25.00

0.075

Table 3

Results for Aqueous Samples Collected from Generator Column

Sample ID (439C-101-)	Peak Area (uV*sec)	Sample Volume (mL)	Final Volume (mL)	Collection Time (min.)	Calculated Flow Rate (mL/min.)	Measured Concentration (µg DBDPO/L)	Measured Concentration (M)
10	15,661	255	1.0	270	0.94	0.0417	4.35×10^{-11}
11	16,424	265	1.0	255	1.0	0.0420	4.38×10^{-11}
12	14,751 ¹	270	1.0	268	1.0	0.0363	3.79×10^{-11}
						$\bar{x} = 0.0400$	$\bar{x} = 4.17 \times 10^{-11}$
						SD = 0.0032	SD = 3.32×10^{-12}

¹Peak area value represents mean of duplicate reinjections, (14372 and 15130) due to an instrument injection error.

Table 4
Results for Octanol Stock Solution Samples

Sample ID (439C-101-)	Peak Area (uV*sec)	Sample Volume (mL)	Final Volume (mL)	Dilution Factor	Measured Concentration ($\mu\text{g DBDPO/L}$)	Measured Concentration (M)
0-7	117579	0.200	100	2.0	77470	8.08×10^{-5}
0-8	102392	0.200	100	2.0	67470	7.04×10^{-5}
0-9	115902	0.200	100	2.0	76360	7.96×10^{-5}
					$\bar{x} = 73770$	$\bar{x} = 7.69 \times 10^{-5}$
					SD = 5480	SD = 5.69×10^{-6}

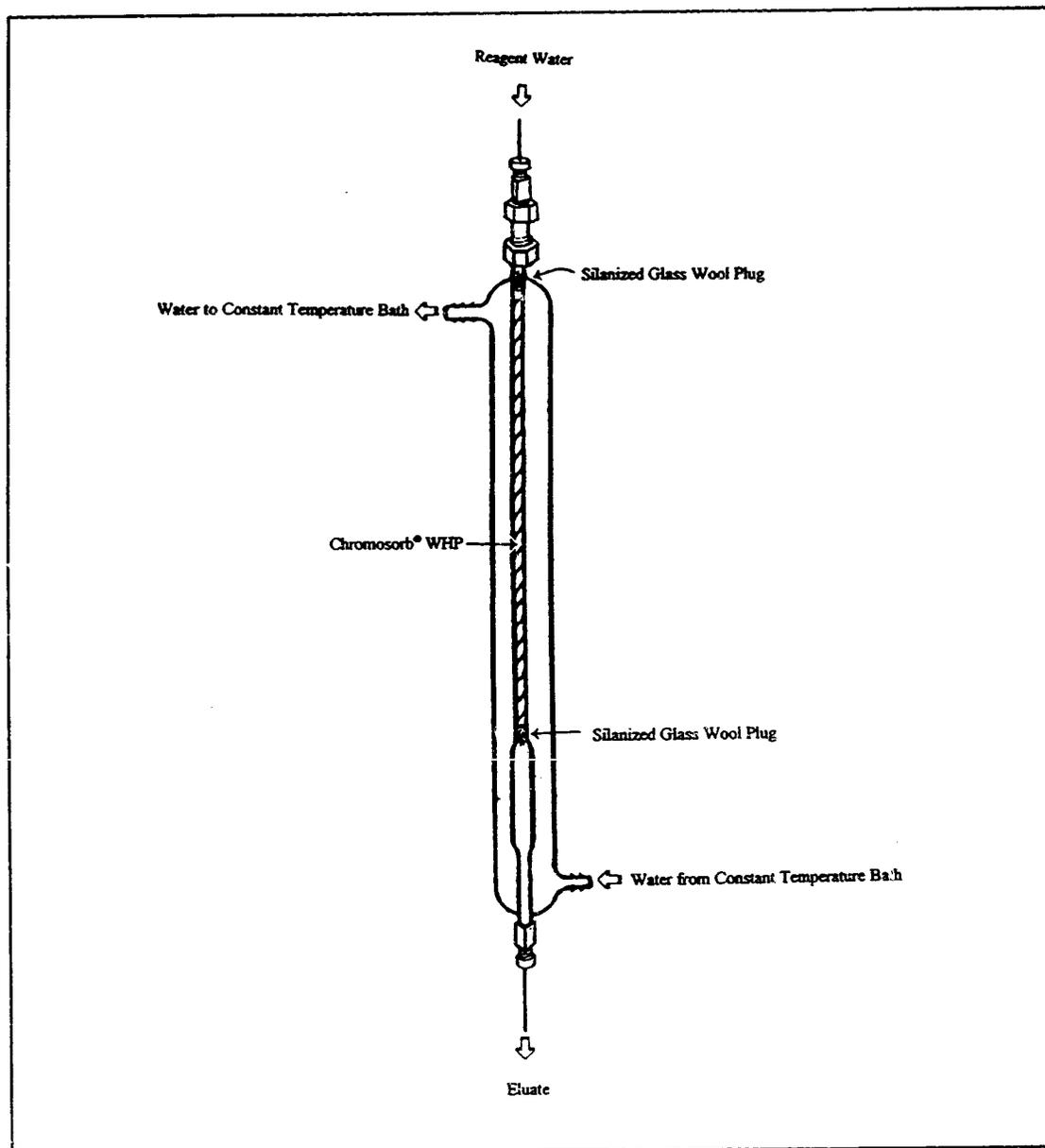


Figure 1. Diagram of generator column.

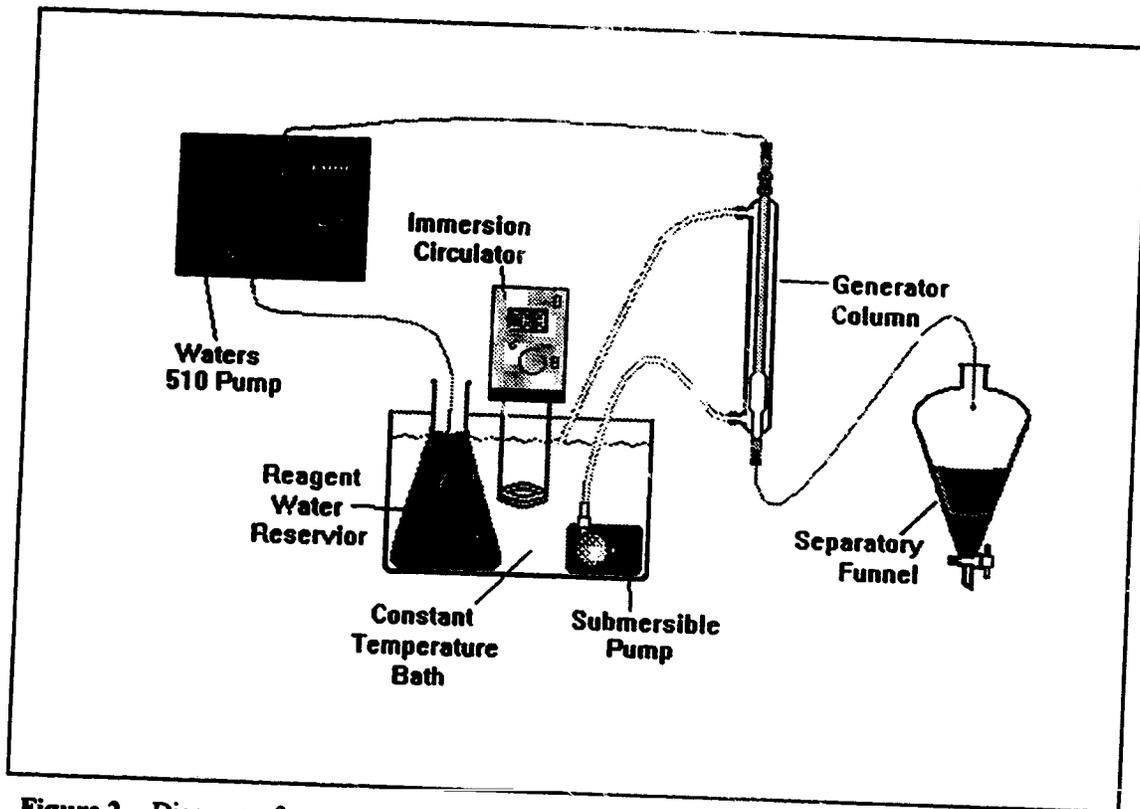


Figure 2. Diagram of apparatus configuration.

ANALYTICAL METHOD FLOW CHART

Measure 250 mL samples of octanol saturated NANOpure® water into 500 mL separatory funnels using 250 mL graduated cylinders, or equivalent

↓

Fortify with the appropriate standard solution; swirl to mix. Add 100 mL of ethyl acetate to each bottle.

↓

Shake each sample for approximately one minute. Allow layers to separate. Add several crystals of NaCl to break emulsions, if necessary. Drain aqueous layer into appropriately sized flask. Drain ethyl acetate into 250 mL round-bottom flasks. Reduce volume to approximately 10 mL by rotary evaporation.

↓

Pour aqueous sample back into separatory funnel. Add 100 mL of ethyl acetate extraction solvent to aqueous flask to rinse. Transfer to separatory funnel.

↓

Repeat the extraction procedure above two additional times.

↓

Combine the extracts into the same flask after reducing solvent volume each time.

↓

Measure the volume of water in each aqueous generator column sample using an appropriately sized graduated cylinder.

↓

Evaporate the extract to approximately 2 mL on a rotovap at 30 to 50°C. Quantitatively transfer to test tubes.

↓

Reduce to residual octanol on nitrogen evaporator at 30 to 50°C.

↓

Reconstitute the extracts using additions of diphenyl ether (DPO) to bring the final sample volume to 1.0 mL. Volume based on visual comparisons to a blank tube containing 1.0 mL of diphenyl ether measured using a class A volumetric pipet.

↓

Transfer the phenyl ether extract into a vial for analysis using GC/ECD.

Figure 3. An analytical method flow chart.