Method 505 Analysis of Organohalide Pesticides and Aroclors in Drinking Water by Microextraction and Gas Chromatography

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Physical and Chemical Methods Branch
Environmental Monitoring and Support Laboratory
U. S. Environmental Protection Agency
Cincinnati, Ohio 45268

METHOD 505. ANALYSIS OF ORGANOHALIDE PESTICIDES AND AROCLORS IN DRINKING WATER BY MICROEXTRACTION AND GAS CHROMATOGRAPHY

1. SCOPE AND APPLICATION

1.1 This method (1,2,3) is applicable to the determination of the following analytes in finished drinking water, drinking water during intermediate stages of treatment, and the raw source water:

Analyte	CAS No.
Alachlor	15972-60-8
Aldrin	309-00-2
Chlordane	57-74-9
Dieldrin	60-57-1
Endrin	72-20-8
Heptachlor	76-44-8
Heptachlor Epoxide	1024-57-3
Hexachlorobenzene	118-74-1
Lindane	58-89-9
Methoxychlor	72-43-5
Toxaphene	8001-35-2
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5

1.2 PCBs are determined as Aroclors by this method.

1.3 For compounds other than the above mentioned analytes or for other sample sources, the analyst must demonstrate the applicability of the method by collecting precision and accuracy data on actual samples (4) and provide qualitative confirmation of results by Gas Chromatography/Mass Spectrometry (GC/MS) (5), or by GC analysis using dissimilar columns.

1.4 The experimentally determined method detection limits (MDL) (6) for the above organohalides and Aroclors are shown in Table 1. Actual detection limits are highly dependent upon the characteristics of the gas chromatographic system used (e.g. column type, age, and proper conditioning; detector condition; and injector mode and condition).

2. SUMMARY OF METHOD

2.1 Thirty-five mL of sample are extracted with 2 mL of hexane. Two µL of the extract are then injected into a gas chromatograph equipped with a linearized electron capture detector for separation and

analysis. Aqueous calibration standards are extracted and analyzed in an identical manner in order to compensate for possible extraction losses.

2.2 The extraction and analysis time is 30 to 50 minutes per sample depending upon the analytes and the analytical conditions chosen. (See Tables 1-2 and Figures 1-10.)

2.3 Confirmatory evidence can be obtained using a dissimilar column. When component concentrations in water samples are sufficiently high, GC/MS may be employed.

3. INTERFERENCES

3.1 Impurities contained in the extracting solvent usually account for the majority of the analytical problems. Solvent blanks should be analyzed on each new bottle of solvent before use. Indirect daily checks on the extracting solvent are obtained by monitoring the laboratory reagent blanks (9.1.1). Whenever an interference is noted in the sample blank, the analyst should analyze another solvent blank. Low level interferences generally can be removed by distillation or column chromatography (3); however, it is generally more economical to obtain a new source solvent. An interference-free solvent is a solvent containing non-detectable peaks at the retention times of the analytes of interest.

3.2 Caution must be taken in the determination of endrin since it has been reported that the splitless injector may cause endrin degradation (7). The analyst should be alerted to this possible interference resulting in an erratic response for endrin.

3.3 Variable amounts of pesticides and Aroclors from aqueous solutions adhere to glass surfaces. It is recommended that sample transfers and glass surface contacts be minimized.

3.4 Aldrin and methoxychlor are rapidly oxidized by chlorine. Dechlorination with sodium thiosulfate at time of collection will retard further oxidation of these compounds.

4. SAFETY

4.1 The toxicity and carcinogenicity of chemicals used in this method have not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized. Each laboratory is responsible for maintaining awareness of OSHA regulations regarding safe handling of chemicals used in this method. Additional references to laboratory safety are available (9-10) for the information of the analyst.

4.2 The following organohalides have been tentatively classified as known or suspected human or mammalian carcinogens: aldrin,

Aroclors, chlordane, dieldrin, heptachlor, hexachlorobenzene, and toxaphene. Pure standard materials and stock standard solutions of these compounds should be handled in a hood or glovebox. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5. APPARATUS AND EQUIPMENT

5.1 SAMPLE CONTAINERS - 40-mL screw cap vials (Pierce #13075 or equivalent) each equipped with a PTFE-faced silicone septum (Pierce #12722 or equivalent). Prior to use, wash vials and septa with detergent and rinse with tap and distilled water. Allow the vials and septa to air dry at room temperature, place the vials in a 400°C oven for one hour, then remove and allow to cool in an area known to be free of organics.

5.2 VIALS - auto sampler, screw cap with septa, 1.8 mL, Varian #96-000099-00 or equivalent or any other autosampler vials not requiring more than 1.8 mL sample volumes.

5.3 AUTO SAMPLER - Hewlett-Packard 7671A, or equivalent.

5.4 MICRO SYRINGES - 10 and 100 µL.

5.5 MICRO SYRINGE - 25 µL with a 2-inch by 0.006-inch needle - Hamilton 702N or equivalent.

5.6 PIPETTES - 2.0 and 5.0 mL transfer.

5.7 VOLUMETRIC FLASKS - 10 and 100 mL, glass stoppered.

5.8 STANDARD SOLUTION STORAGE CONTAINERS - 15-mL bottles with PTFE-lined screw caps.

5.9 GAS CHROMATOGRAPHY SYSTEM

5.9.1 The GC must be capable of temperature programming and should be equipped with a linearized electron capture detector, capillary column, and splitless injector (0.5 min. splitless mode). Alternately, an on-column injector may be employed.

5.9.2 Three gas chromatographic columns are recommended. Column A should be used as the primary analytical column unless routinely occurring analytes are not adequately resolved. Columns B and C are recommended for use as confirmatory columns when GC/MS confirmation is not available.

5.9.2.1 Column A - 0.32mm ID x 30 M long fused silica capillary with chemically bonded methyl polysiloxane phase (DB-1, 1.0µm film, or equivalent). The linear velocity of the helium carrier gas is established at 25 cm/sec. The column temperature is programmed

from 180°C to 260°C at 4°C/min and held at 260°C until all expected compounds have eluted. Injector temperature: 200°C. Detector temperature: 290°C. (See Table 1 for retention data.)

5.9.2.2 Column B (alternate confirmation column) - 0.32mm ID x 30 M long fused silica capillary with a 1:1 mixed phase of dimethyl silicone and polyethylene glycol (Durawax-DX3, 0.25um film, or equivalent). The column temperature is programmed from 100°C to 210°C at 8°C/min, and held at 210°C until all expected compounds have eluted. Then the post temperature is programmed to 240°C at 8°C/min for 5 min. (See Table 2 for retention data).

5.9.2.3 Column C (alternate confirmation column) - 0.32mm ID x 25 M long fused silica capillary with chemically bonded 50:50 Methyl-Phenyl silicone (OV-17, 1.5u film thickness, or equivalent). The linear velocity of the helium carrier gas is established at 39 cm/sec. The column temperature is programmed from 100°C to 260°C at 4°C/min and held at 260°C until all expected compounds have eluted.

6. REAGENTS AND CONSUMABLE MATERIALS

6.1 REAGENTS

6.1.1 Hexane extraction solvent - UV Grade, Burdick and Jackson #216 or equivalent.

6.1.2 Methyl alcohol - ACS Reagent Grade, demonstrated to be free of analytes.

6.1.3 Sodium chloride, NaCl - ACS Reagent Grade - For pretreatment before use, pulverize a batch of NaCl and place in a muffle furnace at room temperature. Increase the temperature to 400°C and hold for 30 min. Place in a bottle and cap.

6.1.4 Sodium thiosulfate, $Na_2S_2O_3$—ACS Reagent Grade—For preparation of solution (0.04 g/mL), mix 1 g of $Na_2S_2O_3$ with reagent water and bring to 25-mL volume in a volumetric flask.

6.2 REAGENT WATER - Reagent water is defined as water free of interference when employed in the procedure described herein.

6.2.1 A Millipore Super-Q Water System or its equivalent may be used to generate deionized reagent water.

6.2.2 Test reagent water each day it is used by analyzing it according to Sect. 10.

6.3 STANDARD STOCK SOLUTIONS - These solutions may be obtained as certified solutions or prepared from pure standard materials using the following procedures:

6.3.1 Prepare stock standard solutions (5000 ug/mL) by accurately weighing about 0.0500 g of pure material. Dissolve the material in methanol and dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.3.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4°C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.3.3 Stock standard solutions must be replaced after six months, or sooner if comparison with check standards indicates a problem.

6.4 SECONDARY DILUTION STANDARDS — Use standard stock solutions to prepare secondary dilution standard solutions that contain the analytes in methanol. The secondary dilution standards should be prepared at concentrations that can be easily diluted to prepare aqueous calibration standards (Sect. 8.1.1) that will bracket the working concentration range. Store the secondary dilution standard solutions with minimal headspace and check frequently for signs of deterioration or evaporation, especially just before preparing calibration standards. The storage time described for stock standard solutions in Sect. 6.4.3 also applies to secondary dilution standard solutions.

7. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

7.1 SAMPLE COLLECTION

7.1.1 Collect all samples in duplicate 40-mL bottles into which 3 mg of sodium thiosulfate crystals have been added to the empty bottles just prior to shipping to the sampling site. Alternately, 75 µL of freshly prepared sodium thiosulfate solution (0.04 g/mL) may be added to empty 40-mL bottles just prior to sample collection. In collecting field samples, it is recommended to add sodium thiosulfate solution at the sampling site.

7.1.2 When sampling from a water tap, open the tap and allow the system to flush until the water temperature has stabilized (usually about 10 min). Adjust the flow to about 500 mL/min and collect duplicate samples from the flowing stream.

7.1.3 When sampling from a well, fill a wide-mouth bottle or beaker with sample, and carefully fill duplicate 40-mL sample bottles.

7.2 SAMPLE PRESERVATION

7.2.1 The samples must be chilled to 4°C at the time of collection and maintained at that temperature until the analyst is prepared for the extraction process. Field samples that will not be received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to insure that they will be maintained at 4°C until arrival at the laboratory.

7.3 SAMPLE STORAGE

7.3.1 Store samples and extracts at 4°C until extraction and analysis.

7.3.2 Extract all samples as soon as possible after collection. Results of holding time studies suggest that all analytes (aldrin, dieldrin, endrin, heptachlor epoxide, hexachlorobenzene, lindane, and the aroclors) with the possible exception of heptachlor may be extracted within 14 days after collection. In general, heptachlor showed inconsistent results. If heptachlor is to be determined, samples must be extracted within 7 days of collection. Samples from which analytes have not been extracted within these prescribed periods of time must be discarded and replaced.

8. CALIBRATION AND STANDARDIZATION

8.1 CALIBRATION

8.1.1 At least three calibration standards are needed. One should contain analytes at a concentration near but greater than the method detection limit for each compound; the other two should be at concentrations that bracket the range expected in samples. For example, if the MDL is 0.01 µg/L, and a sample expected to contain approximately 0.10 µg/L is to be analyzed, aqueous standards should be prepared at concentrations of 0.02 µg/L, 0.10 µg/L, and 0.20 µg/L.

8.1.2 To prepare a calibration standard, add an appropriate volume of a secondary dilution standard to a 35-mL aliquot of

reagent water in a 40-mL bottle. Do not add less than 20 µL of an alcoholic standard to the reagent water. Use a 25-µL micro syringe and rapidly inject the alcoholic standard into the middle point of the water volume. Remove the needle as quickly as possible after injection. Mix by inverting and shaking the capped bottle several times. Aqueous standards must be prepared fresh daily.

8.1.3 Starting with the standard of lowest concentration, prepare, extract, and analyze each calibration standard beginning with Sect. 10.1.4 and tabulate peak height or area response versus the concentration in the standard. The results are to be used to prepare a calibration curve for each compound by plotting the peak height or area response versus the concentration. Alternatively, if the ratio of response to concentration (calibration factor) is a constant over the working range (<10% relative standard deviation), linearity to the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

8.1.4 The working calibration curve or calibration factor must be verified on each working day by the measurement of one or more calibration standards. If the response for an analyte varies from the predicted response by more than ±15%, the test must be repeated using a fresh calibration standard. If the results still do not agree, generate a new calibration curve or use a single point calibration standard as described in Sect. 8.1.5.

8.1.5 Single point calibration is an acceptable alternative to a calibration curve. Prepare single point standards from the secondary dilution standard solutions. The single point calibration standard should be prepared at a concentration that produces a response close (±20%) to that of the unknowns. Do not use less than 20 µL of the secondary dilution standard solution to produce a single point calibration standard in reagent water.

8.2 INSTRUMENT PERFORMANCE - Check the performance of the entire analytical system daily using data gathered from analyses of reagent blanks, standards, duplicate samples, and the laboratory control standard (Sect. 9.2.2).

8.2.1 Significant peak tailing in excess of that shown for the target compounds in the method chromatograms (Figures 1-10) must be corrected. Tailing problems are generally traceable to active sites on the GC column, improper column installation, or the detector operation.

8.2.2 Check the precision between replicate analyses. A properly operating system should perform with an average relative

standard deviation of less than 10%. Poor precision is generally traceable to pneumatic leaks, especially at the injection port. If the GC system is apparently performing acceptably but with decreased sensitivity, it may be necessary to generate a new curve or set of calibration factors to verify the decreased responses.

8.2.3 Observed relative area responses of endrin must meet the following criteria: endrin $\geq$ 50% of total area.

9. QUALITY CONTROL

9.1 MONITORING FOR INTERFERENCES

9.1.1 Laboratory Reagent Blanks - A laboratory reagent blank is an aliquot of reagent water analyzed as if it were a sample. Add $N_2S_2O_3$ to laboratory reagent blanks. Analyze a laboratory reagent blank each day and as necessary to identify sources of contamination. The laboratory reagent blank should contain less than the MDL response of each analyte.

9.2 ASSESSING ACCURACY

9.2.1 Each quarter, it is essential that the laboratory analyze quality control check samples for each contaminant. If any criteria established by USEPA are not met, corrective action needs to be taken and documented.

9.2.2 After every 10 samples, and preferably in the middle of each day, analyze a laboratory control standard. Calibration standards may not be used for accuracy assessments and the laboratory control standard may not be used for calibration of the analytical system.

9.2.2.1 Laboratory Control Standard Concentrate - If internally prepared laboratory control standards are used to provide the routine assessment of accuracy, they should be prepared from a separate set of stock standards. From stock standards prepared as described in Sect. 6.4, add a sufficient volume of each stock standard to methanol in a 10-mL volumetric flask to yield a concentration of 2.5 µg/mL and adjust to volume.

9.2.2.2 Laboratory Control Standard — Add 20 µL of the control concentrate to a 35-mL aliquot of reagent water poured into a 40-mL bottle containing 75 µL of 0.04 g/mL sodium thiosulfate.

9.2.2.3 Analyze the 35-mL aliquot as described in Sect. 10. For each analyte in the laboratory control

standard, calculate the percent recovery (P_i) with the equation:

$$P_i = \frac{100\ S_i}{T_i}$$

where S_i = the analytical result from the laboratory control standard, in µg/L; and

T_i = the known concentration of the spike, in µg/L.

9.2.3 It is essential that the laboratory analyze an unknown performance evaluation sample (when available) once per year for all regulated contaminants measured. Results need to be within acceptance limits established by USEPA for each analyte.

10. PROCEDURE

10.1 SAMPLE PREPARATION

10.1.1 Remove samples from storage and allow them to equilibrate to room temperature.

10.1.2 Remove the container caps. Withdraw and discard a 5-mL volume using a transfer pipet. Replace the container caps and weigh the containers with contents to the nearest 0.1 g and record these weights for subsequent sample volume determinations (Sect. 10.3).

10.1.3 Remove the container cap of each sample, and add 6 g NaCl (Sect. 6.1.3) to the sample bottle. Recap and dissolve the NaCl by inverting and shaking the bottles several times (approx. 20 sec).

10.2 EXTRACTION AND ANALYSIS

10.2.1 Remove the cap, and using a transfer or automatic dispensing pipet, add 2.0 mL of hexane. Recap and shake vigorously by hand for 1 min. Invert the bottle and allow the water and hexane phases to separate.

10.2.2 Remove the cap and carefully transfer approximately 0.5 mL of hexane layer into an autosampler vial using a disposable glass pipet.

10.2.3 Transfer the remaining hexane phase, being careful not to include any of the water phase, into a second autosampler vial. Reserve this second vial at 4°C for an immediate reanalysis if necessary.

10.2.4 Transfer the first sample vial to an autosampler set up to inject 1-2 µL portions into the gas chromatograph for analysis (See Sect. 5.9 for GC conditions). Alternately, 1-2 µL portions of samples, blanks, and standards may be manually injected, although an autosampler is strongly recommended.

10.3 DETERMINATION OF SAMPLE VOLUME IN BOTTLES NOT CALIBRATED

10.3.1 Discard the remaining sample/hexane mixture from the sample bottle. Shake off the remaining few drops using short, brisk wrist movements.

10.3.2 Reweigh the empty container with original cap and calculate the net weight of sample by difference to the nearest 0.1 g (Sect. 10.1.2 minus Section 10.3.2). This net weight (in grams) is equivalent to the volume of water (in mL) extracted (Sect. 11.3).

11. CALCULATIONS

11.1 Identify the organohalides in the sample chromatogram by comparing the retention time of the suspect peak to retention times generated by the calibration standards and the laboratory control standard. Identify the multicomponent compounds using all peaks that are characteristic of the specific compound from chromatograms generated with individual standards. Select the most sensitive and reproducible peaks for calculation purposes.

11.2 Use the calibration curve or calibration factor (Sect. 8.1.3) to directly calculate the uncorrected concentration (C_i) of each analyte in the sample (e.g., calibration factor x response).

11.3 Calculate the sample volume (V_s) as equal to the net sample weight:

$$V_s = \text{gross weight (Sect. 10.1.2) - bottle tare (Sect. 10.3.2).}$$

11.4 Calculate the corrected sample concentration as:

$$\text{Concentration, } \mu g/L = C_i \times \frac{35}{V_s}$$

11.5 Report the results for the unknown samples in µg/L. Round off the results to the nearest 0.1 µg/L or two significant figures.

12. ACCURACY AND PRECISION

12.1 Single laboratory (EMSL-Cincinnati) accuracy and precision at several concentrations in reagent, ground, and tap water matrices are presented in Table 3 (11). These results were obtained from data generated with a DB-1 column.

Table 2. Chromatographic Conditions and Rentention Data of Organohalide Pesticides and Aroclors

Analyte	Column B Retention Time[1], Min	Column C Retention Time[1], Min
Alachlor	19.71	21.09
Aldrin	18.37	21.39
Butachlor	25.16	26.75
Chlorpyrifos	21.52	24.36
Chlorpyrifos-Methyl	10.46, 21.02	22.35
Dichlobenil	11.20	8.09
Dichlorvos	8.91	1.18, 5.48
Dieldrin	45.08	27.76
Endrin	33.30	29.17
Heptachlor	17.53	19.99
Heptachlor Epoxide	24.61	24.63
Hexachlorobenzene	13.35	15.62
Lindane	18.35	18.67
Metolachlor	21.42	23.50
Methoxychlor	-	36.44
Propachlor	14.43	14.70
Aroclor 1016	15.11, 16.62, 19.45, 20.43 20.64	10.27, 18.52, 20.75, 22.63, 23.84
Aroclor 1221	10.46, 11.62, 13.19	8.36, 8.54, 10.27, 10.73, 12.78
Aroclor 1232	15.10, 16.61, 19.43, 20.42 20.62, 22.68, 25.61	16.23, 18.49, 20.71 22.61, 23.83, 25.63
Aroclor 1242	15.10, 16.61, 19.44, 20.42 20.63, 22.66, 25.64	16.24, 18.48, 20.66 22.59, 23.80
Aroclor 1248	16.60, 19.42, 20.41, 20.61 22.66, 23.48, 24.20, 25.62	20.69, 22.59, 23.79, 25.09, 25.56, 26.75
Aroclor 1254	20.55, 25.63, 26.62, 32.99 35.72, 36.38, 44.80	25.84, 28.73, 29.67 30.42, 32.14
Aroclor 1260	26.56, 31.21, 33.72, 36.29 42.33, 43.25, 44.68	10.92, 11.53, 12.29, 12.98, 14.55, 15.48
Chlordane	16.28, 17.51, 24.57, 25.60 26.28	19.03, 20.00, 22.84, 25.67, 25.96, 26.32
Toxaphene	27.68, 35.56, 40.67, 43.29	28.80, 29.25, 30.83 32.16, 34.40

[1]More than one peak listed does not implicate the total number of peaks characteristic of the multi-component pesticide. Listed peaks indicate only the ones chosen for quantification.

13. REFERENCES

1. Glaze, W.W., Lin, C.C., Optimization of Liquid-Liquid Extraction Methods for Analysis of Organics in Water, EPA-600/S4-83-052, January 1984.

2. Henderson, J.E., Peyton, G.R. and Glaze, W.H. (1976). In "Identification and Analysis of Organic Pollutants in Water" (L.H. Keith ed.), pp. 105-111. Ann Arbor Sci. Publ., Ann Arbor, Michigan.

3. Richard, J.J., Junk, G.A., "Liquid Extraction for Rapid Determination of Halomethanes in Water," Journal AWWA, 69, 62, January 1977.

4. "Handbook for Analytical Quality Control in Water and Wastewater Laboratories," EPA-600/4-79-019, U. S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, 45268, March 1979.

5. Budde, W.L., Eichelberger, J.W., "Organic Analyses Using Gas Chromatography-Mass Spectrometry," Ann Arbor Science, Ann Arbor, Michigan 1979.

6. Glaser, J.A. et al., "Trace Analyses for Wastewaters," Environmental Science and Technology, 15, 1426 (1981).

7. Bellar, T.A., Stemmer, P., Lichtenberg, J.J., "Evaluation of Capillary Systems for the Analysis of Environmental Extracts," EPA-600/S4-84-004, March 1984.

8. "Carcinogens-Working with Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute of Occupational Safety and Health, Publication No. 77-206, August, 1977.

9. "OSHA Safety and Health Standards, General Industry," (29CFR1910), Occupational Safety and Health Administration, OSHA 2206, (Revised, January 1976).

10. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

11. Winfield, T., et al. "Analysis of Organohalide Pesticides in Drinking Water by Microextraction and Gas Chromatography." In preparation.

Table 1. Chromatographic Conditions and Method Detection Limits of Organohalide Pesticides and Aroclors

Analyte	Column A Retention Time[1], Min	MDL, ug/L
Hexachlorobenzene	11.9	0.002
Lindane	12.3	0.003
Alachlor	15.1	0.225
Heptachlor	15.9	0.003
Aldrin	17.6	0.007
Heptachlor Epoxide	19.0	0.004
Dieldrin	22.1	0.012
Endrin	24.2	0.063
Methoxychlor	30.0	0.956
Aroclor 1016	13.6, 14.8, 15.2 16.2, 17.7	0.08
Aroclor 1221	7.7, 9.0, 15.9, 19.1, 24.7	15.0
Aroclor 1232	11.2, 14.7, 13.6, 15.2 17.7	0.48
Aroclor 1242	11.2, 13.6, 14.7, 15.2 17.7, 19.8	0.31
Aroclor 1248	14.8, 16.2, 17.1, 17.7 19.8, 22.0	0.102
Aroclor 1254	19.1, 21.9, 23.4, 24.9 26.7	0.102
Aroclor 1260	23.4, 24.9, 26.7, 28.2 29.9, 32.6	0.189
Chlordane	15.1, 15.9, 20.1, 20.9 21.3	0.14
Toxaphene	21.7, 22.5, 26.7, 27.2	1.0

[1]More than one peak listed does not implicate the total number of peaks characteristic of the multi-component pesticide. Listed peaks indicate only the ones chosen for quantification.

Column A conditions: Column A - 0.32mm ID x 30 M long fused silica capillary with methyl polysiloxane phase (DB-1, 0.25 µm film, or equivalent). The linear velocity of the helium carrier gas is established at 25 cm/sec. The column temperature is programmed from 180°C to 260°C at 4°C/min and held at 260°C until all expected compounds have eluted. Injector temperature: 200°C; detector temperture: 290°C.

Table 2. Chromatographic Conditions and Rentention Data of Organohalide Pesticides and Aroclors

Analyte	Column B Retention Time[1], Min	Column C Retention Time[1], Min
Alachlor	19.71	21.09
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Butachlor	25.16	26.75
Chlorpyrifos	21.52	24.36
Chlorpyrifos-Methyl	10.46, 21.02	22.35
Dichlobenil	11.20	8.09
Dichlorvos	8.91	1.18, 5.48
Dieldrin	45.08	27.76
Endrin	33.30	29.17
Heptachlor	17.53	19.99
Heptachlor Epoxide	24.61	24.63
Hexachlorobenzene	13.35	15.62
Lindane	18.35	18.67
Metolachlor	21.42	23.50
Methoxychlor	-	36.44
Propachlor	14.43	14.70
Aroclor 1016	15.11, 16.62, 19.45, 20.43 20.64	10.27, 18.52, 20.75, 22.63, 23.84
Aroclor 1221	10.46, 11.62, 13.19	8.36, 8.54, 10.27, 10.73, 12.78
Aroclor 1232	15.10, 16.61, 19.43, 20.42 20.62, 22.68, 25.61	16.23, 18.49, 20.71 22.61, 23.83, 25.63
Aroclor 1242	15.10, 16.61, 19.44, 20.42 20.63, 22.66, 25.64	16.24, 18.48, 20.66 22.59, 23.80
Aroclor 1248	16.60, 19.42, 20.41, 20.61 22.66, 23.48, 24.20, 25.62	20.69, 22.59, 23.79, 25.09, 25.56, 26.75
Aroclor 1254	20.55, 25.63, 26.62, 32.99 35.72, 36.38, 44.80	25.84, 28.73, 29.67 30.42, 32.14
Aroclor 1260	26.56, 31.21, 33.72, 36.29 42.33, 43.25, 44.68	10.92, 11.53, 12.29, 12.98, 14.55, 15.48
Chlordane	16.28, 17.51, 24.57, 25.60 26.28	19.03, 20.00, 22.84, 25.67, 25.96, 26.32
Toxaphene	27.68, 35.56, 40.67, 43.29	28.80, 29.25, 30.83 32.16, 34.40

[1]More than one peak listed does not implicate the total number of peaks characteristic of the multi-component pesticide. Listed peaks indicate only the ones chosen for quantification.

Table 3. Matrix Effect on Single Laboratory Recovery and Precision of Organohalide Pesticides and Aroclors

Compound	Matrix[1]	Number of Samples	Spike Level (µg/L)	Average Accuracy (% Recovery)	Relative Standard Deviation(%)
Alachlor	RW	6	0.50	102	13.1
Aldrin	RW	7	0.05	106	18.9
		3	1.8	105	4.9
	GW	7	0.05	86	18.9
		3	1.2	92	15.7
	TW	3	1.2	94	5.6
Chlordane	RW	8	0.17	NA	8.0
		7	3.4	NA	3.6
	TW	8	0.17	104	11.8
		8	3.4	95	10.1
Dieldrin	RW	7	0.10	87	19.6
		7	3.6	114	8.0
	GW	7	0.10	67	15.0
		6	3.6	94	9.1
	TW	8	0.10	92	17.1
		6	3.6	81	17.3
Endrin	RW	6	0.10	119	25.0
		6	3.6	99	6.6
	GW	6	0.10	94	21.5
		6	3.6	100	11.3
	TW	7	0.10	106	13.2
		6	3.6	85	14.6
Heptachlor	RW	6	0.032	77	13.3
		6	1.2	80	9.3
	GW	7	0.032	37	18.3
		6	1.2	71	13.8
	TW	8	0.032	200	11.3
		8	1.2	106	15.8

Table 3. (Continued)

Compound	Matrix[1]	Number of Samples	Spike Level (µg/L)	Average Accuracy (% Recovery)	Relative Standard Deviation(%)
Heptachlor Epoxide	RW	8	0.04	100	15.6
		8	1.4	115	5.7
	GW	7	0.04	90	15.8
		6	1.4	103	6.7
	TW	6	0.04	112	6.7
		5	1.4	81	7.3
Hexachlorobenzene	RW	7	0.0025	104	13.0
		8	0.09	103	6.4
	GW	7	0.002	91	12.0
		7	0.09	101	4.4
	TW	7	0.0027	100	15.6
		6	0.09	88	15.2
Lindane	RW	7	0.03	91	7.1
		7	1.2	111	4.5
	GW	7	0.03	88	8.8
		7	1.2	109	3.1
	TW	8	0.03	103	7.9
		6	1.2	93	19.8
Methoxychlor	RW	5	2.10	100	21.0
		6	7.03	98	11.1
Toxaphene	RW	8	10	NA	12.6
		8	80	NA	15.3
	TW	8	10	110	8.6
		8	80	114	11.8
Aroclor 1016	RW	8	1.0	NA	6.6
	TW	8	1.0	97	7.7
Aroclor 1221	RW	7	180	NA	8.3
	TW	7	180	92	10.4

Table 3. (Continued)

Compound	Matrix[1]	Number of Samples	Spike Level (µg/L)	Average Accuracy (% Recovery)	Relative Standard Deviation(%)
Aroclor 1232	RW	8	3.9	NA	13.5
	TW	8	4.3	86	8.5
Aroclor 1242	RW	6	4.7	NA	6.0
	TW	7	4.8	96	7.7
Aroclor 1248	RW	7	3.6	NA	11.5
	TW	8	3.4	84	11.8
Aroclor 1254	RW	8	1.8	NA	10.4
	TW	8	1.7	85	13.9
Aroclor 1260	RW	7	2.0	NA	20.7
	TW	6	1.8	88	18.0

[1]Matrix Identities

RW = Reagent water
GW = Ground water
TW = Tap Water

NA = Not applicable. A separate set of aqueous standards was not analyzed, and the values shown for RW were used to calculate % recovery for the TW matrix.

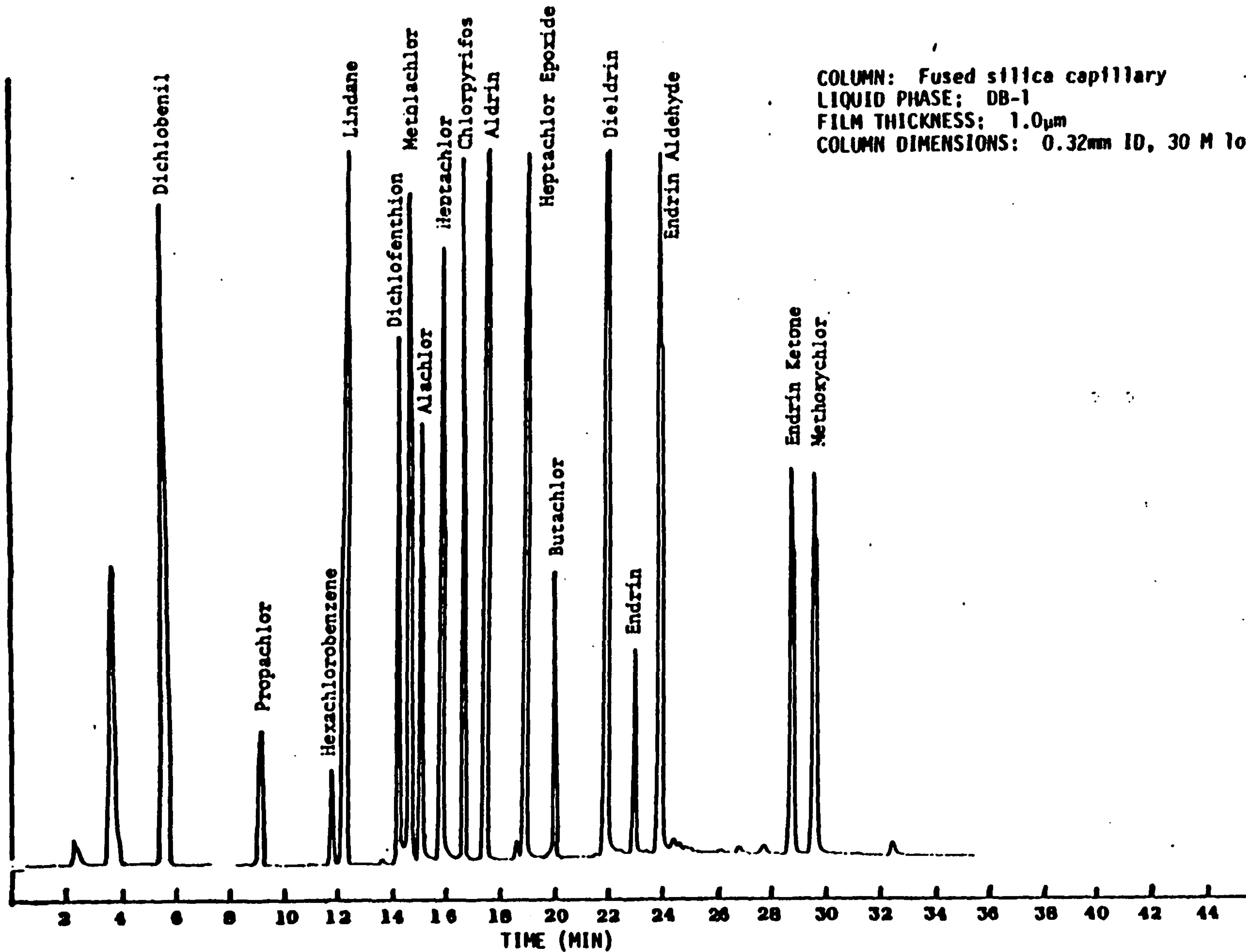

Figure 1. Hexane spiked at 7.71 ug/L with heptachlor and lindane; 9.14 ug/L with heptachlor epoxide; 11.4 ug/L with aldrin and hexachlorobenzene; 23 ug/L with butachlor, chlorpyrifos, chlorpyrifos-methyl, diclobenil, dieldrin, endrin, metolochlor, and propachlor; and 44.9 ug/L with methoxychlor.

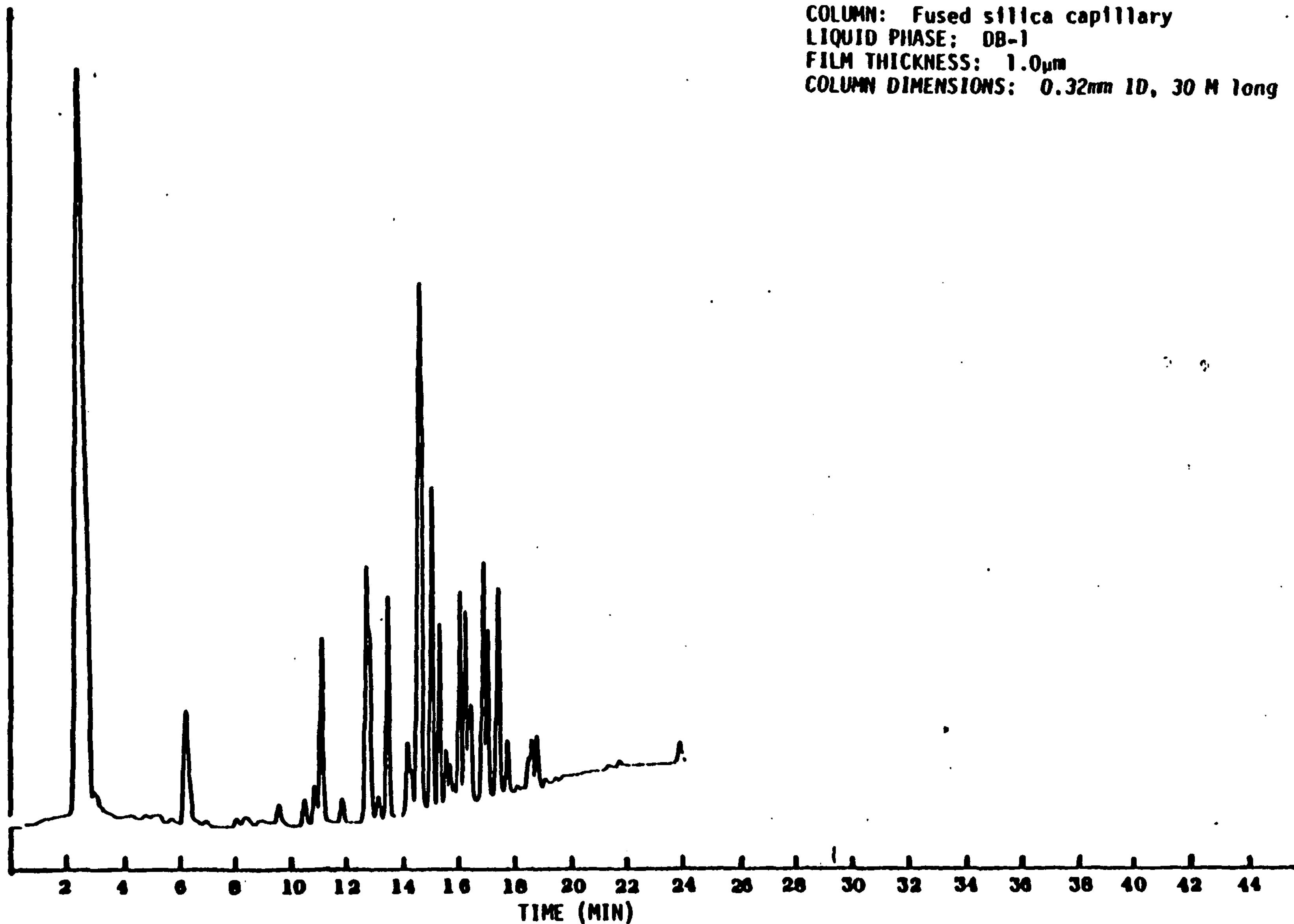

Figure 2. Hexane spiked at 11.4 ug/L with Aroclor 1016.

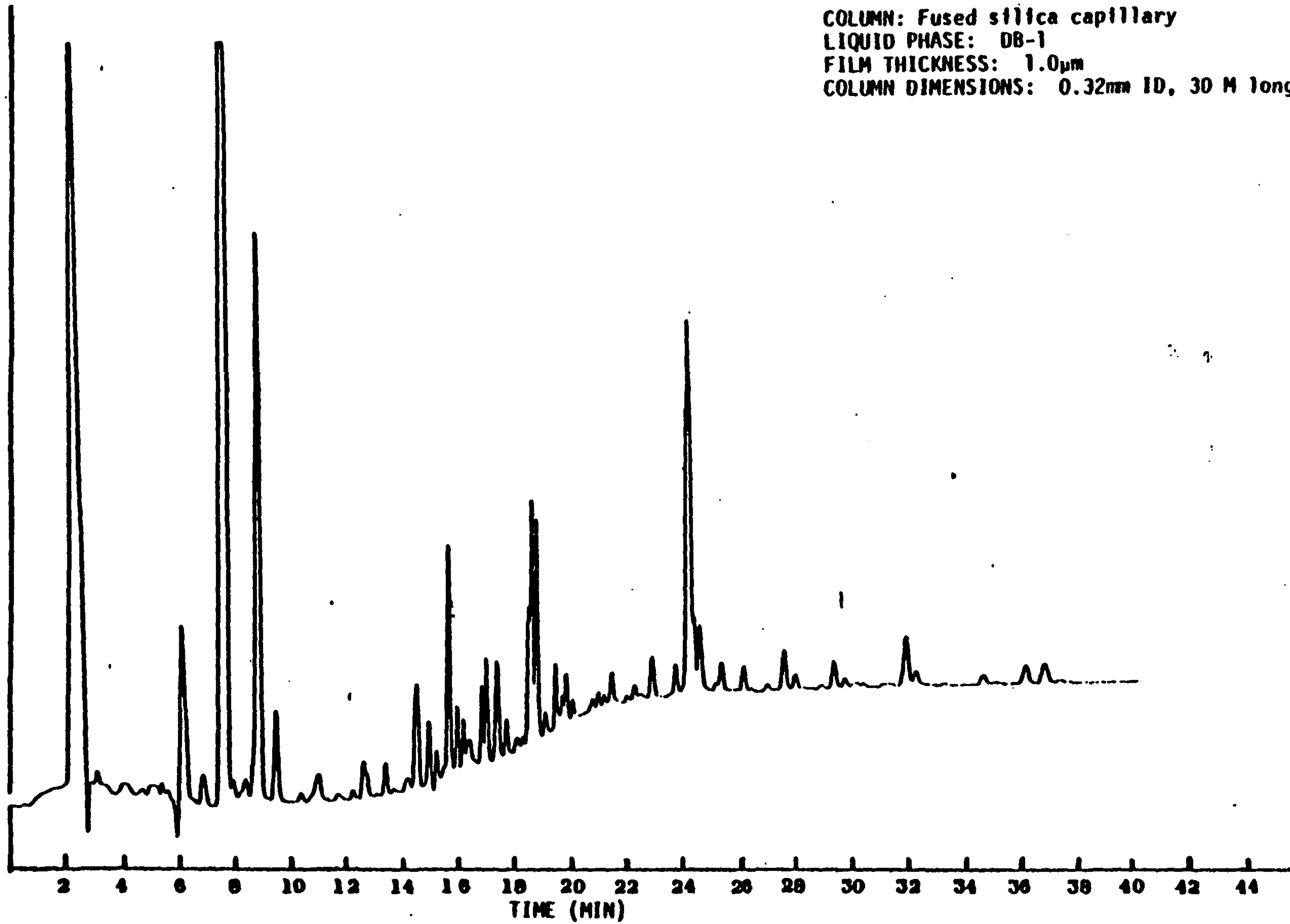

Figure 3. Hexane spiked at 171.4 ug/L with Aroclor 1221.

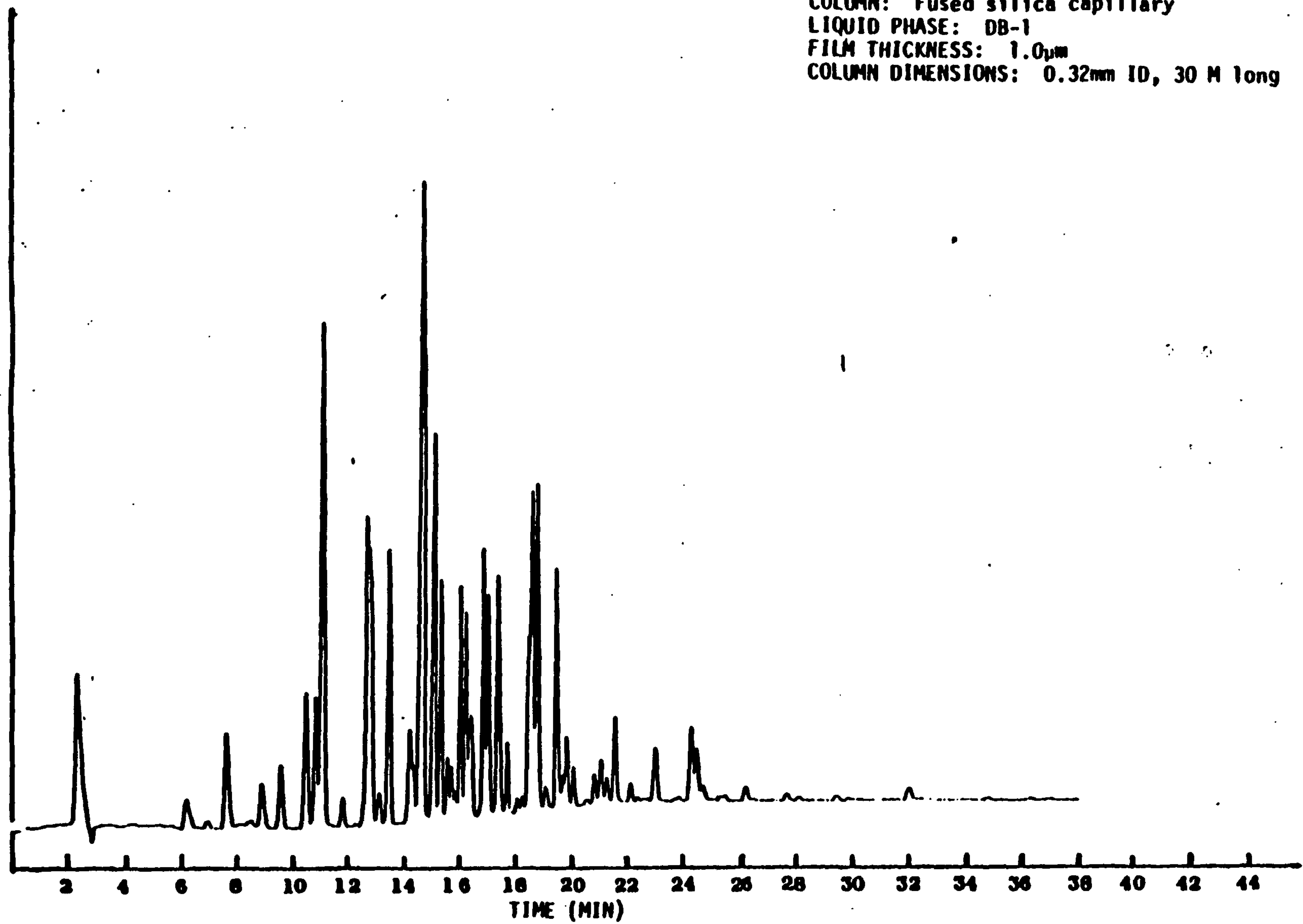

Figure 4. Hexane spiked at 57.1 ug/L with Aroclor 1232.

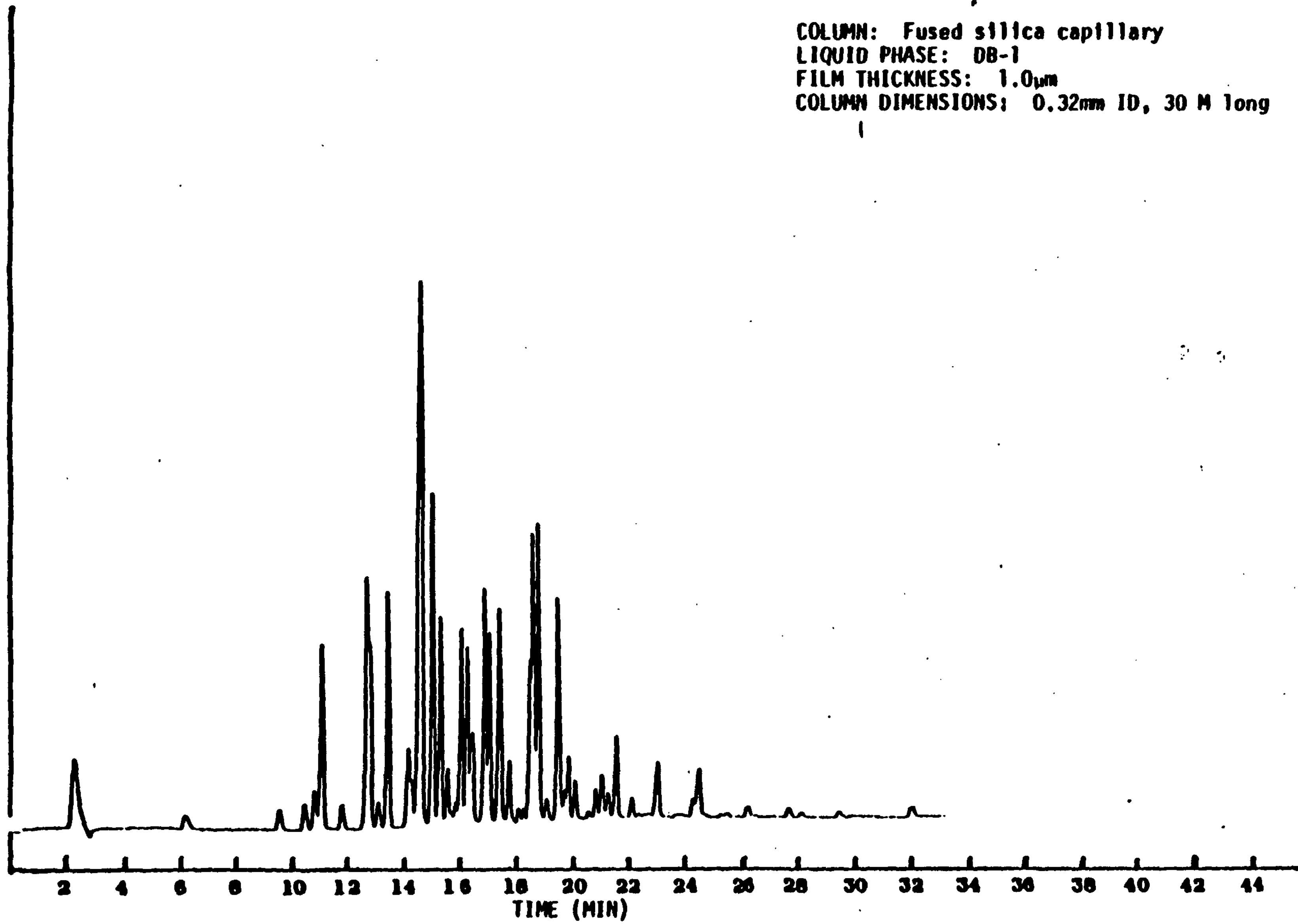

Figure 5. Hexane spiked at 57.1 ug/L with Aroclor 1242.

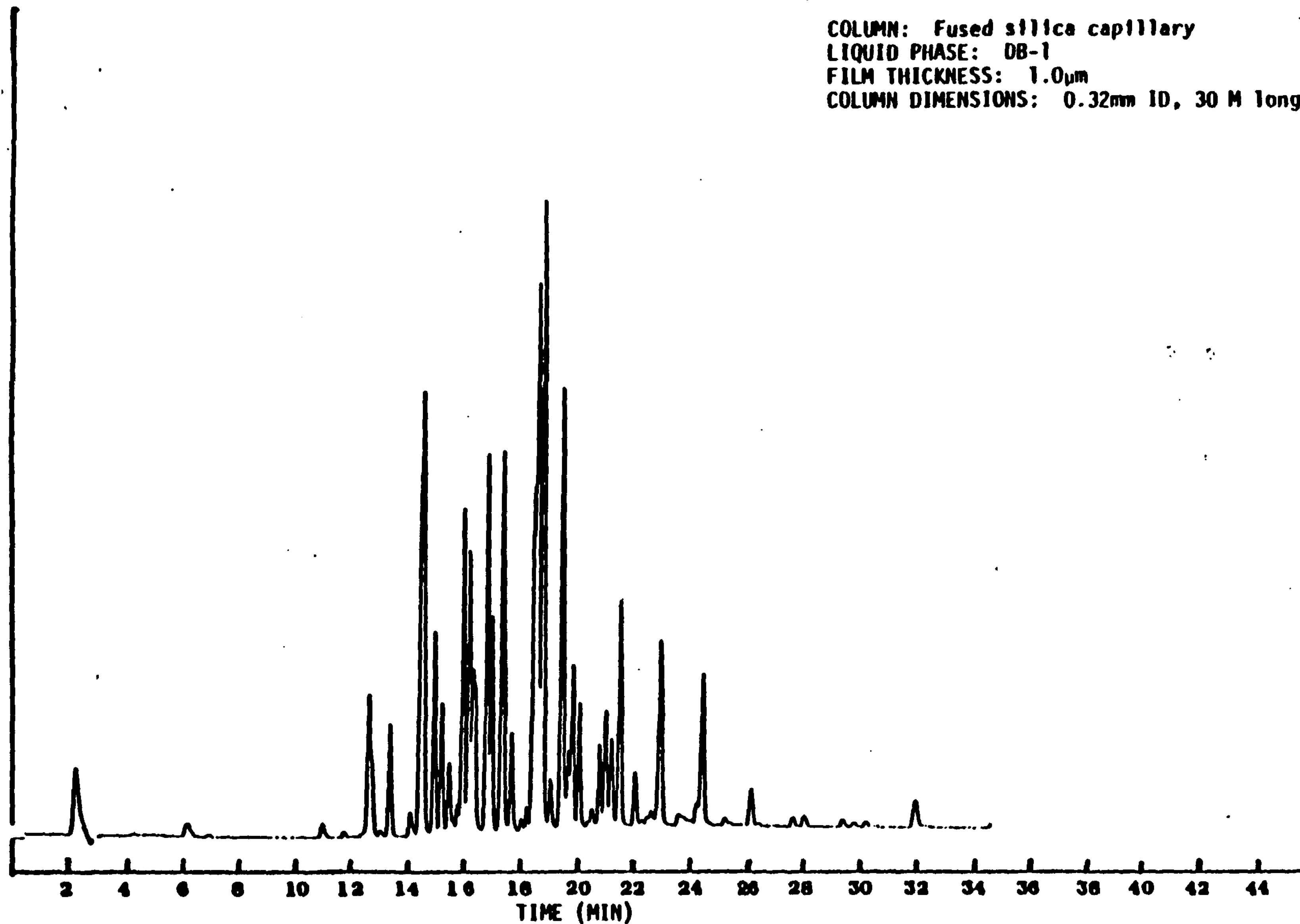

Figure 6. Hexane spiked at 57.1 ug/L with Aroclor 1248.

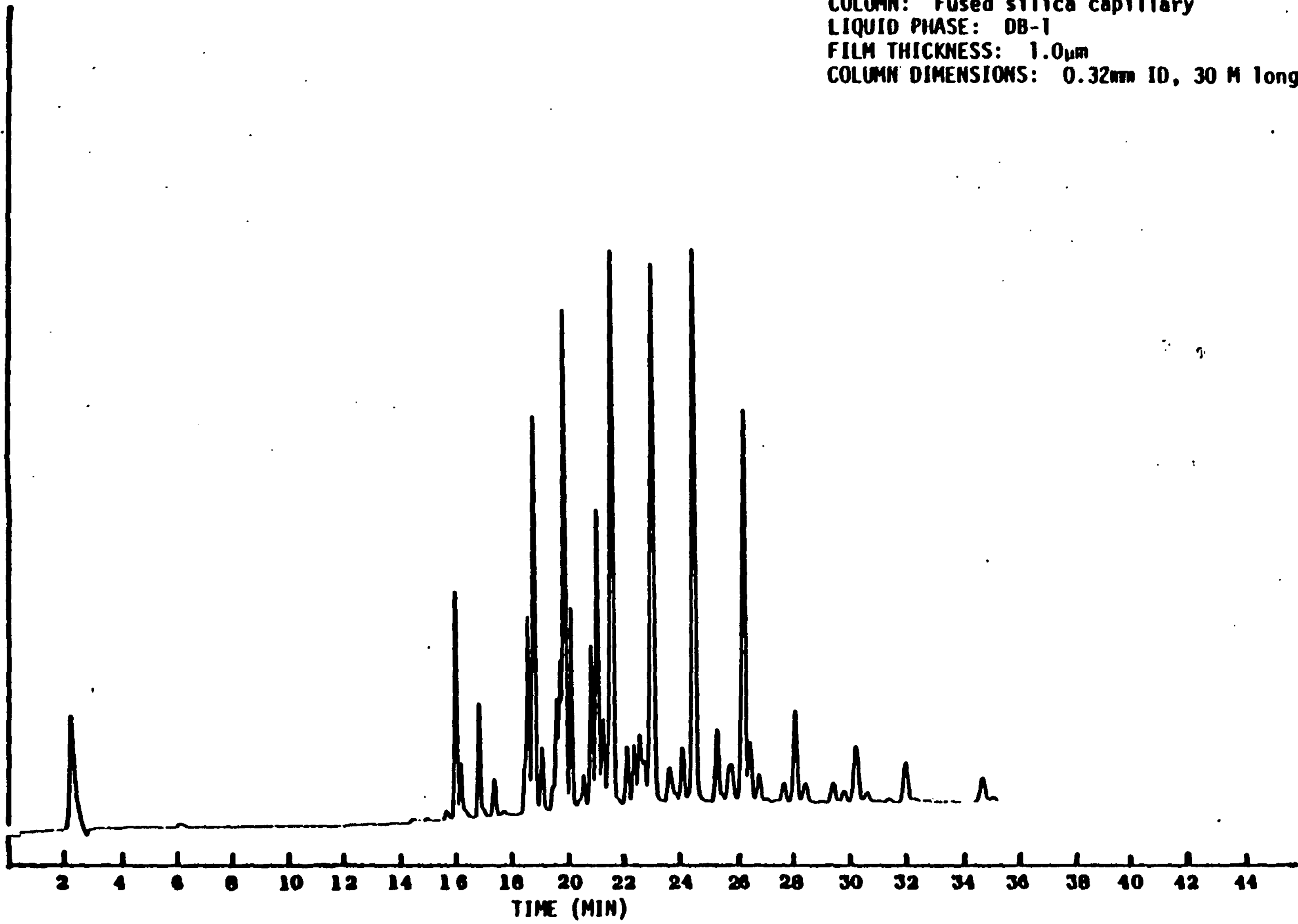

Figure 7. Hexane spiked at 42.9 ug/L with Aroclor 1254.

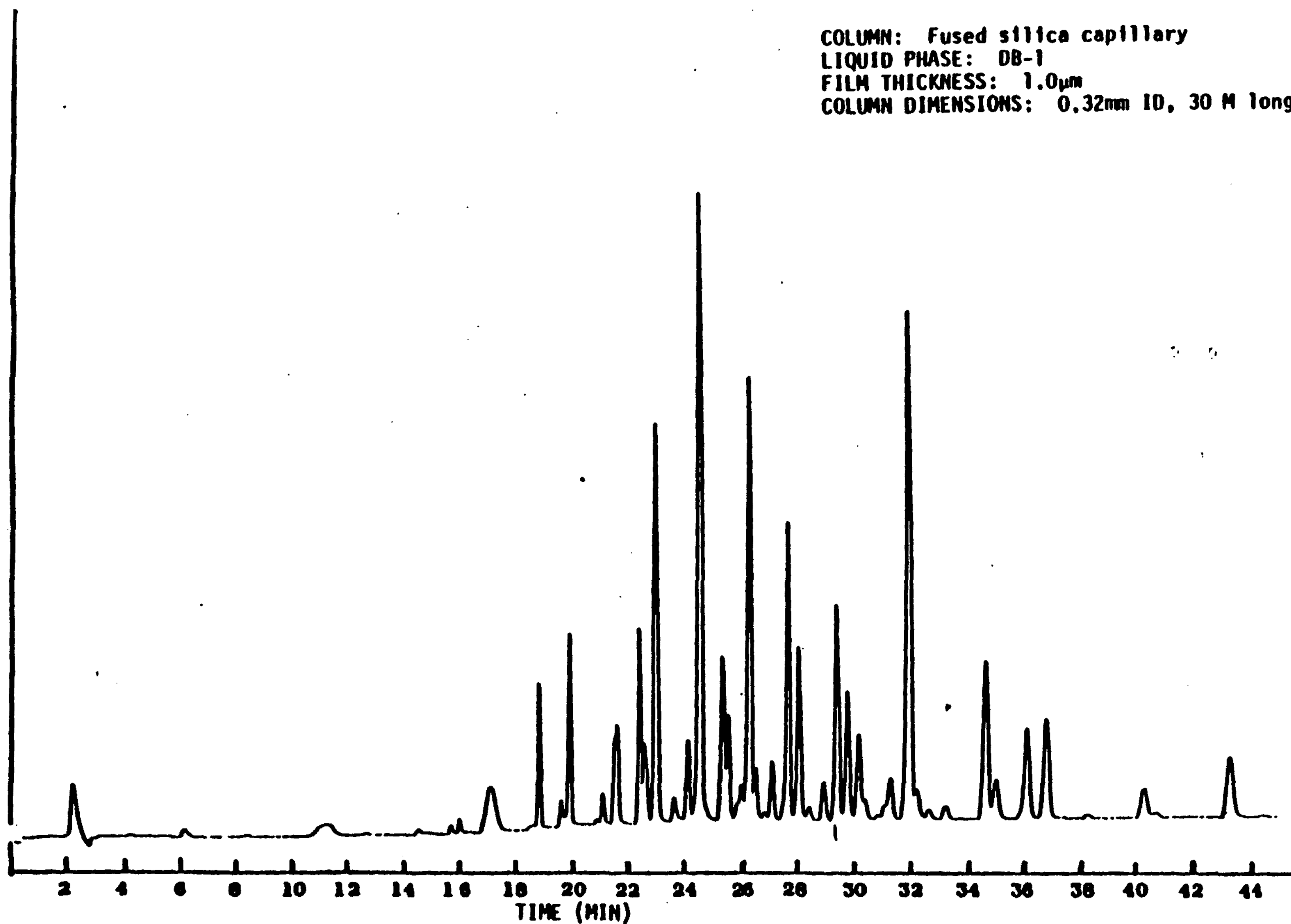

Figure 8. Hexane spiked at 34.3 ug/L with Aroclor 1260.

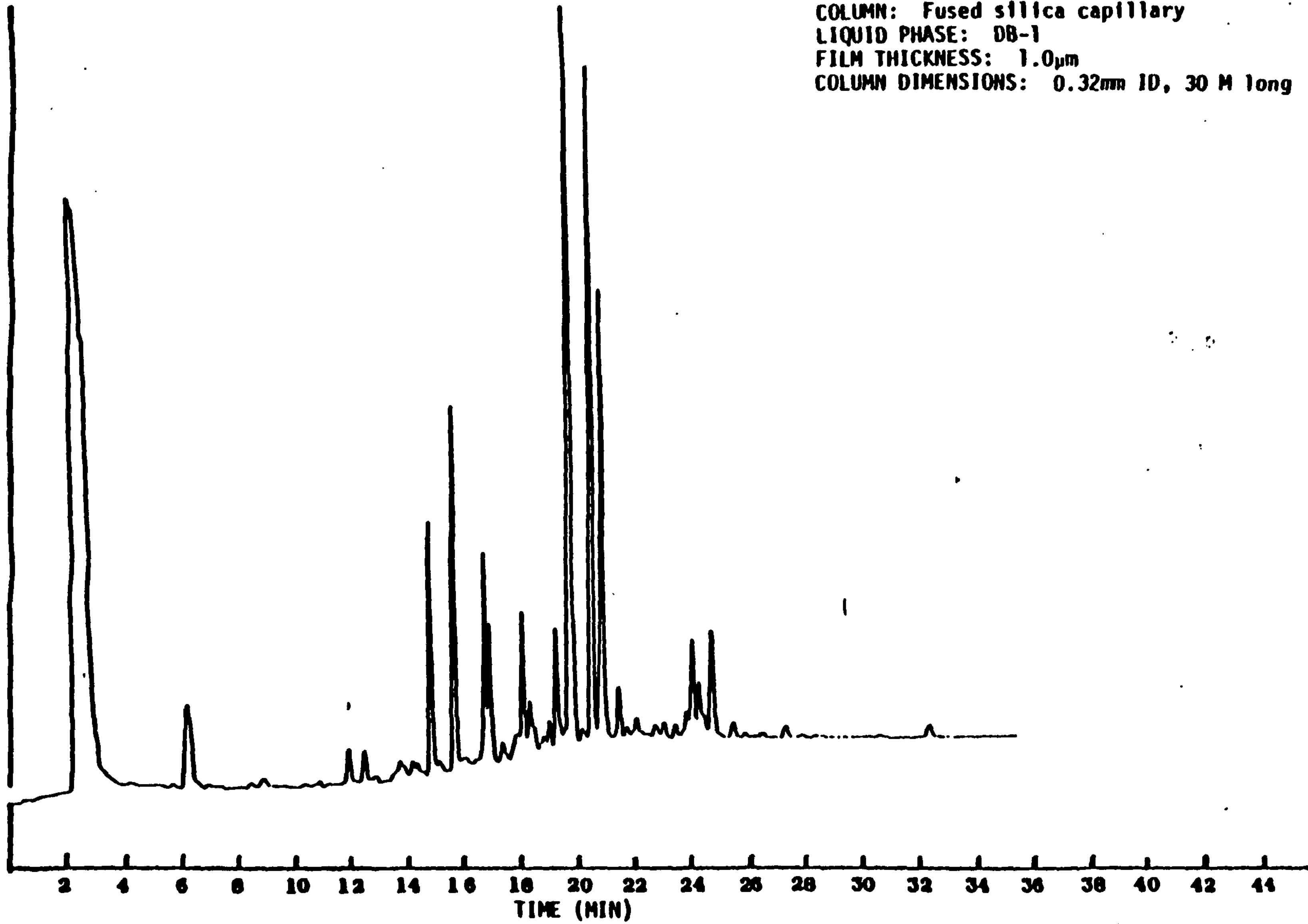

Figure 9. Hexane spiked at 28.6 ug/L with chlordane.