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ARGONNE NATIONAL LABORATORY
9700 South Cass Avenue
Argonne, Illinois 60439

THE DETERMINATION OF PCBs IN ROCKY FLATS TYPE IV WASTE SLUDGE BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTION PART II

by

Kathleen J. Parish
Daniel V. Applegate
Pamela D. Postlethwait
Amrit S. Boparai
Gerald T. Reedy

Analytical Chemistry Laboratory
Chemical Technology Division

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TABLE OF CONTENTS

Page

ABSTRACT 1

INTRODUCTION 2

MATERIALS AND METHODS 3

1. Preparation of the Spiked Simulated Sludges 3

1.1 ANL-E Simulated Sludges 3

1.2 ANL-W Simulated Sludges 3

2. Extraction and Analysis 4

2.1 Gas Chromatographic Conditions 7

2.2 Gas Chromatographic Maintenance 7

2.3 PCB Identification 8

2.3.1 Identification of Weathered PCBs 8

2.3.2 Determination of Retention Time Windows 9

2.4 PCB Quantitation 9

RESULTS AND DISCUSSION 10

REFERENCES 18

APPENDIX A 19

LIST OF TABLES

No.	Title	Page
1.	Formula for Simulated Sludges	4
2.	Gas Chromatographic Conditions Used to Analyze ANL-E Sludge	5
3.	Gas Chromatographic Conditions Used to Analyze ANL-W Sludge	6
4.	Percent Recoveries of ANL-E Simulated Sludge Spiked with Aroclor 1016 at 50 ppm	11
5.	Percent Recoveries of ANL-E Simulated Sludge Spiked with Aroclor 1221 at 50 ppm	11
6.	Percent Recoveries of ANL-E Simulated Sludge Spiked with Aroclor 1232 at 50 ppm	12
7.	Percent Recoveries of ANL-E Simulated Sludge Spiked with Aroclor 1242 at 50 ppm	12
8.	Percent Recoveries of ANL-E Simulated Sludge Spiked with Aroclor 1248 at 50 ppm	13
9.	Percent Recoveries of ANL-W Simulated Sludge Spiked with Aroclor 1016 at 50 ppm	14
10.	Percent Recoveries of ANL-W Simulated Sludge Spiked with Aroclor 1221 at 50 ppm	14
11.	Percent Recoveries of ANL-W Simulated Sludge Spiked with Aroclor 1232 at 50 ppm	15
12.	Percent Recoveries of ANL-W Simulated Sludge Spiked with Aroclor 1242 at 50 ppm	15
13.	Percent Recoveries of ANL-W Simulated Sludge Spiked with Aroclor 1248 at 50 ppm	16
14.	Percent Recoveries of ANL-W Simulated Sludge Spiked with Aroclor 1254 50 ppm	16
15.	Percent Recoveries of ANL-W Simulated Sludge Spiked with Aroclor 1260 50 ppm	17
16.	Summary of Results: Average Recoveries of the Aroclors in ANL-E and ANL-W Sludge	17

LIST OF FIGURES

No.	Title	Page
1.	Aroclor 1016 Standard at 0.50 ppm Concentration	20
2.	Aroclor 1221 Standard at 0.50 ppm Concentration	21
3.	Aroclor 1232 Standard at 0.50 ppm Concentration	22
4.	Aroclor 1242 Standard at 0.50 ppm Concentration	23
5.	Aroclor 1248 Standard at 0.50 ppm Concentration	24
6.	Extract of ANL-E Simulated Sludge Spiked with 50 ppm of Aroclor 1016	25
7.	Extract of ANL-E Simulated Sludge Spiked with 50 ppm of Aroclor 1221	26
8.	Extract of ANL-E Simulated Sludge Spiked with 50 ppm of Aroclor 1232	27
9.	Extract of ANL-E Simulated Sludge Spiked with 50 ppm of Aroclor 1242	28
10.	Extract of ANL-E Simulated Sludge Spiked with 50 ppm of Aroclor 1248	29
11.	Extract of ANL-E Simulated Sludge with No Aroclors Added	30
12.	Aroclor 1016 Standard at 0.50 ppm Concentration	31
13.	Aroclor 1221 Standard at 0.50 ppm Concentration	32
14.	Aroclor 1232 Standard at 0.50 ppm Concentration	33
15.	Aroclor 1242 Standard at 0.50 ppm Concentration	34
16.	Aroclor 1248 Standard at 0.50 ppm Concentration	35
17.	Aroclor 1254 Standard at 0.50 ppm Concentration	36
18.	Aroclor 1260 Standard at 0.50 ppm Concentration	37
19.	Extract of ANL-W Simulated Sludge Spiked with 50 ppm of Aroclor 1016	38
20.	Extract of ANL-W Simulated Sludge Spiked with 50 ppm of Aroclor 1221	39

LIST OF FIGURES
(Continued)

No.	Title	Page
21.	Extract of ANL-W Simulated Sludge Spiked with 50 ppm of Aroclor 1232	40
22.	Extract of ANL-W Simulated Sludge Spiked with 50 ppm of Aroclor 1242	41
23.	Extract of ANL-W Simulated Sludge Spiked with 50 ppm of Aroclor 1248	42
24.	Extract of ANL-W Simulated Sludge Spiked with 50 ppm of Aroclor 1254	43
25.	Extract of ANL-W Simulated Sludge Spiked with 50 ppm of Aroclor 1260	44
26.	Extract of ANL-W Simulated Sludge with No Aroclors Added	45

THE DETERMINATION OF PCBs IN ROCKY FLATS TYPE IV WASTE SLUDGE BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTION PART II

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ABSTRACT

Before disposal, radioactive sludge (Type IV) from Rocky Flats Plant (RFP) must be evaluated for polychlorinated biphenyl (PCB) content. The Type IV sludge consists of organic solvents, degreasers, cutting oils, and transuranic (TRU) waste mixed with calcium silicate (MicroCel E®) and Oil Dri® to form a grease or paste-like material. For laboratory testing, a nonradioactive simulated Type IV RFP sludge was prepared at Argonne National Laboratory-East (ANL-E). This sludge has a composition similar to that expected from field samples. In an earlier effort, a simplified method was developed for extraction, cleanup of extract, and determination of PCBs in samples of simulated sludge spiked with Aroclors 1254 and 1260. The simplified method has now been used to determine the presence and quantities of other Aroclors in the simulated sludge, namely, Aroclors 1016, 1221, 1232, 1242, and 1248. The accuracy and precision of the data for these Aroclors were found to be similar to the data for sludges spiked with Aroclors 1254 and 1260. Since actual sludges may vary in composition, the method was also verified by analyzing another source of Type IV simulated sludge, prepared by Argonne National Laboratory-West (ANL-W).

INTRODUCTION

The data contained in this report supplements the data in the earlier report, *Determination of PCBs in Rocky Flats Type IV Waste Sludge by Gas Chromatography/Electron Capture Detection* (ANL/ACL-93/1).[1] The Type IV sludge consists of organic solvents, degreasers, cutting oils, and transuranic (TRU) waste mixed with calcium silicate (MicroCel E®) and Oil Dri® to form a grease or paste-like material. The original report contains data on the accuracy and precision of the ANL-developed method for simulated Type IV sludges spiked with Aroclors 1254 and 1260 prepared at ANL-E. This method was compared to an Environmental Protection Agency (EPA) method (SW-846) for extraction and analysis of PCBs. The accuracy and precision of the data for the sludges spiked with Aroclors 1254 and 1260 were found to be similar for the two methods. This report addresses questions about the applicability of the ANL method to other Aroclors, namely, Aroclors 1016, 1221, 1232, 1242, and 1248. We found that the data obtained for Aroclors 1016 through 1248 were similar to those obtained for Aroclors 1254 and 1260. Therefore, the ANL method is applicable to sludges containing a variety of Aroclors.

Since actual sludges may vary in composition, the method was also tested with a simulated sludge prepared by ANL-W. The ANL-W sludge varied slightly in composition from the ANL-E sludge. Data on accuracy and precision were similar for both the ANL-W and ANL-E simulated sludges. Therefore, the variation in sludge composition did not affect the performance of the method.

MATERIALS AND METHODS

1. Preparation of the Spiked Simulated Sludges

1.1 ANL-E Simulated Sludges

The simulated RFP Type IV sludge was prepared in the proportions as listed in Table 1. Three replicate sludge samples were spiked separately at 50 parts per million with each Aroclor (1016, 1221, 1232, 1242, and 1248) for a total of 15 spiked sludge samples. As described in Appendix A of the original report, the RFP sludge consists of an organic liquid portion (organic solvents, degreasers, and cutting oils) mixed with calcium silicate and Oil Dri® to solidify the waste. When preparing simulated sludges spiked with Aroclors, the Aroclor is added to the organic liquid portion first, and the solidifying agents are added later. Addition of the Aroclors to the unsolidified organic liquid most closely simulates the generation of actual PCB-contaminated waste.

1.2 ANL-W Simulated Sludges

A solidified simulated sludge was received from ANL-W. Unlike the sludge prepared at ANL-E, the Aroclors were not able to be added to the organic liquid portion because the sludge was solidified upon receipt. Therefore, the Aroclors were spiked into the solidified sludges. The recipe of the simulated sludge prepared by ANL-W is given in Table 1. Three replicate sludge samples were spiked separately at 50 parts per million with each Aroclor (1016, 1221, 1232, 1242, 1248, 1254, and 1260), which yielded 21 spiked sludge samples.

Table 1. Formula for Simulated Sludges

Individual Sludge Component	ANL-E Sludge Amount	ANL-W Sludge Amount, lb (kg)
Calcium Silicate: (Microcel E®)	150 g	88 (40)
Oil Dri®	23 g	--
Texaco Regal Oil	29 mL	260 (118)
Carbon tetrachloride	20 mL	61 (28)
Hydraulic Oil: Texaco Rando Oil HD150	49 mL	--
Spindle Oil: Mobile Velocite 6	49 mL	--
Gearbox Oil: Swepco SAE 90	49 mL	--
Freon 113	49 mL	34 (15)
Mineral Spirits (Varsol equivalent)	49 mL	--
Paint Thinner	--	28 (13)
Trichloroethane	49 mL	29 (13)
Water	35 mL	--

2. Extraction and Analysis

Two-gram portions of the spiked sludges were prepared for analysis by the procedure described in Appendix A. The sludge extracts were analyzed by the same procedures described in Section 7.0 of the original report,[1] with the exception of a few chromatographic differences. The gas chromatographic conditions of the analysis are outlined in Table 2 for the ANL-E samples and Table 3 for ANL-W samples. The gas chromatograms obtained for those conditions are given in Figs 1-11 (Table 2) and Figs. 12-26 (Table 3).

Table 2. Gas Chromatographic Conditions Used to Analyze ANL-E Sludge

Chromatographic Conditions	Original Report Data (Aroclor 1254 & 1260)	Additional Data (Aroclor 1016-1248)
Column type	DB-5 (J&W Scientific)	Rtx-1701 (Restek Corp.)
Column length	30 m	30 m
Column ID	0.32 mm	0.53 mm
Column film thickness	0.25 μm	1.0 μm
GC Conditions:		
Carrier gas/flow	Nitrogen, ~2 mL/min	Helium, ~6 mL/min
Makeup gas/flow	Nitrogen, ~60 mL/min	Nitrogen, ~60 mL/min
Injector temperature	270°C	225°C
Detector temperature	280°C	280°C
Initial oven temperature	100°C	150°C
Initial time	1 min	0.5 min
Oven temperature program	100-200°C at 25°C/min; then 200-240°C at 2°C/min, hold 6 min, finally 240-260°C at 5°C/min, hold 2 min	150-275°C at 4°C/min, hold 15 min
Total run time	37 min	47 min
Injection volume	2 μL splitless injection	1 μL splitless injection
PCB quantitation	Sum 5 major Aroclor peaks	Sum 3 to 5 major Aroclor peaks

Table 3. Gas Chromatographic Conditions Used to Analyze ANL-W Sludge

Chromatographic Conditions	Actual Conditions	Recommended Conditions for Improved Chromatography
Column type	Rtx-1701 (Restek Corp.)	Rtx-1701 (Restek Corp.)
Column length	30 m	30 m
Column ID	0.53 mm	0.53 mm
Column film thickness	1.0 μm	1.0 μm
GC Conditions:		
Carrier gas/flow	Helium, 10 mL/min (Electronic Pressure Control)	Helium, ~10 mL/min (Electronic Pressure Control)
Makeup gas/flow	Nitrogen, ~60 mL/min	Nitrogen, ~60 mL/min
Injector temperature	Initial temp. 150°C with oven tracking	Initial temp. 50°C with oven tracking
Detector temperature	280°C	280°C
Initial oven temperature	150°C	50°C
Initial time	1.5 min	0.5 min
Oven temperature program	150-270°C at 4°C/min, hold 5.50 min	50-150°C at 20°C/min, 150-270°C at 5°C/min, hold 5.50 min
Total run time	37 min	35 min
Injection volume	1 μL on-column	1 μL on-column
PCB quantitation	Sum 3 of 5 major peaks	Sum 3 to 5 major peaks

2.1 Gas Chromatographic Conditions

The columns and conditions in Tables 2 and 3 are based on recommendations found in the U.S. EPA Contract Laboratory Program Statement of Work for Organic Analysis.[2] Other columns and conditions may be used if adequate method performance data are demonstrated.

2.2 Gas Chromatographic Maintenance

If chromatographic problems are encountered or corrective measures are required, the following system maintenance should performed. Clean and deactivate the glass injection port liner or replace it with a clean deactivated liner. Inspect the injection end of the column for discoloration or plugs of foreign material. Depending on the column, remove the first few inches, up to one foot. Remove and solvent backflush the column according to the manufacturer's instructions.

Figures 12 through 26 show a relatively broad peak for a compound that elutes at approximately 23 min. It is suspected that this peak is due to contamination in the gas lines, which is caused by back-flashing of sample injections resulting from the high injector temperature (150°C). For on-column injections using electronic pressure control, the initial injector and oven temperatures should be relatively low (50°C instead of 150°C). For this reason, the revised chromatographic conditions as listed in Table 3 are recommended.

To remove contamination due to back flashing of sample injections, the oven and injection port temperatures are raised to 270°C. Then, the gas lines going into the injector are heated with a VariTemp® heat gun (Master Appliance Corp., Racine, WI). The gas lines are heated for 5 to 7 min approximately two to three inches upstream from the injection port. This should achieve the necessary decontamination.

The metal injector body may also be cleaned and deactivated. For that purpose, purchase a deactivation solution and follow the manufacturer's instructions.

The electron capture (EC) detector signal may rise with use, causing chromatographic problems such as an elevated noisy baseline. In this case, the detector may be baked out. Before baking is done, the exit end of the column must be disconnected from the detector, and the detector must be plugged. The detector may be baked at 380°C overnight. If the bakeout procedure does not adequately lower the detector signal, a new EC detector should be installed.

2.3 PCB Identification

Qualitative identification of PCBs is based on retention time and the relative peak intensities as compared to known standards on a single analytical column. Except in cases where the PCBs in the sample are severely weathered, the chromatographic fingerprint of a particular Aroclor can be readily identified by an experienced analytical chemist; a second dissimilar column may be used for confirmation.

2.3.1 Identification of Weathered PCBs

For samples where the Aroclor patterns or relative peak intensities do not readily appear to match any of the Aroclor standard patterns, it must be considered that the sample contains weathered PCBs. In examining such samples, the analytical chemist should keep in mind that weathering of PCBs often results in loss of the lower molecular weight PCBs. The EPA SW-846 Method 8081,[3] "Organochlorine Pesticides and PCBs as Aroclors by Gas Chromatography/Capillary Column Technique," identifies several diagnostic peaks of the different Aroclors. It is recommended that any samples producing chromatograms with these peaks be closely examined, as they may contain PCBs. If chromatographic conditions other than those listed in Method 8081 are used, an experienced analytical chemist should be able to identify diagnostic Aroclor peaks for a given set of chromatographic conditions. The diagnostic peaks chosen should be fairly immune to the effects of PCB degradation and weathering.

2.3.2 Determination of Retention Time Windows

In determining the presence of Aroclors in the sample, retention time should be used. The retention time "windows" for PCBs may be determined as described in Section 7.5 of SW-846 Method 8000A "Gas Chromatography."[3] According to this method, make three injections of the Aroclor standards over a full 72-hour period. Since Aroclors are multicomponent products, choose one major peak for each Aroclor standard injected. Calculate the standard deviation of the retention times for the three injections. The retention time window will be equal to plus or minus three times the standard deviation.

Once the presence of Aroclors has been established by use of retention time, identification of the Aroclors should be based primarily on pattern recognition.

2.4 PCB Quantitation

The recoveries of the Aroclors in the simulated sludges spiked separately with the Aroclors at 50 parts per million were determined by summing the areas of three to five major chromatographic peaks contributed by the Aroclor of interest and comparing the summed area to the sum of the same three to five peaks in the matching Aroclor standard. Chromatograms of the standards and spiked sludges prepared at ANL-E are shown in Figs. 1 through 10 for Aroclors 1016, 1221, 1232, 1242, and 1248. The unspiked sample chromatogram for ANL-E sludge appears in Fig. 11. Chromatograms of the standards and spiked sludges prepared at ANL-W are shown in Figs. 12 through 25 for Aroclors 1016, 1221, 1232, 1242, 1248, 1254, and 1260. The unspiked sample chromatogram for ANL-W sludge appears in Fig. 26. The peaks used for quantitation are indicated on the chromatograms by an asterisk.

RESULTS AND DISCUSSION

Tables 4 through 8 list the percent recoveries and percent relative standard deviation (%RSD) of the ANL-developed method for the Aroclors 1016, 1221, 1232, 1242, and 1248 in the ANL-E sludge samples. For comparison, results are given for two surrogate compounds, tetrachloro-meta-xylene and decachloro-biphenyl. The accuracy and precision of the data for these Aroclors are similar to the data obtained in the original report for Aroclors 1254 and 1260.[1] These results indicate that the developed method is capable of determining the presence and quantities of Aroclors 1016, 1221, 1232, 1242, and 1248 in Type IV sludge.

Tables 9 through 15 list the percent recoveries and %RSD of the method for the two surrogate compounds and Aroclors 1016, 1221, 1232, 1242, 1248, 1254, and 1260 in the ANL-W sludge samples. The accuracy and precision of the data for these Aroclors in the ANL-W simulated sludge are similar to the data obtained for the ANL-E sludge. These results demonstrate that the method is applicable to sludges of various compositions. We plan to further test the method using Type IV sludge samples from the field; however, up to this point, field sludge samples have not been available.

Table 16 is a summary and comparison of the percent recoveries of all seven Aroclors in the ANL-E and ANL-W simulated sludge samples.

Table 4.
Percent Recoveries of ANL-E
Simulated Sludge Spiked with Aroclor 1016 at 50 ppm

Replicate	Surrogate 1[a]	Surrogate 2[b]	Aroclor
1	93	80	85
2	79	70	74
3	90	79	81
Avg.	87	76	80
%RSD	8.5%	7.2%	7.0%

Table 5.
Percent Recoveries of ANL-E
Simulated Sludge Spiked with Aroclor 1221 at 50 ppm

Replicate	Surrogate 1[a]	Surrogate 2[b]	Aroclor
1	69	70	66
2	69	71	71
3	64	68	63
Avg.	67	70	67
%RSD	4.3%	2.2%	6.0%

[a]Surrogate 1 is tetrachloro-meta-xylene.
[b]Surrogate 2 is decachlorobiphenyl.

Table 6.
Percent Recoveries of ANL-E
Simulated Sludge Spiked with Aroclor 1232 at 50 ppm

Replicate	Surrogate 1[a]	Surrogate 2[b]	Aroclor
1	69	78	79
2	63	72	76
3	64	74	68
Avg.	65	75	74
%RSD	4.9%	4.1%	7.7%

Table 7.
Percent Recoveries of ANL-E
Simulated Sludge Spiked with Aroclor 1242 at 50 ppm

Replicate	Surrogate 1[a]	Surrogate 2[b]	Aroclor
1	72	79	73
2	71	77	69
3	78	87	78
Avg.	74	81	73
%RSD	5.1%	6.5%	6.2%

[a]Surrogate 1 is tetrachloro-meta-xylene.
[b]Surrogate 2 is decachlorobiphenyl.

Table 8.
Percent Recoveries of ANL-E
Simulated Sludge Spiked with Aroclor 1248 at 50 ppm

Replicate	Surrogate 1[a]	Surrogate 2[b]	Aroclor
1	70	76	80
2	69	73	76
3	57	62	65
Avg.	65	70	74
%RSD	11.1%	10.5%	10.5%

[a]Surrogate 1 is tetrachloro-meta-xylene.
[b]Surrogate 2 is decachlorobiphenyl·

Table 9.
Percent Recoveries of ANL-W
Simulated Sludge Spiked with Aroclor 1016 at 50 ppm

Replicate	Surrogate 1[a]	Surrogate 2[b]	Aroclor
1	89	92	68
2	87	90	69
3	102	101	77
Avg.	93	94	71
%RSD	8.8%	6.2%	6.9%

Table 10.
Percent Recoveries of ANL-W
Simulated Sludge Spiked with Aroclor 1221 at 50 ppm

Replicate	Surrogate 1[a]	Surrogate 2[b]	Aroclor
1	90	85	80
2	90	85	77
3	87	83	77
Avg.	89	84	78
%RSD	1.9%	1.4%	2.2%

[a]Surrogate 1 is tetrachloro-meta-xylene.
[b]Surrogate 2 is decachlorobiphenyl.

Table 11.
Percent Recoveries of ANL-W
Simulated Sludge Spiked with Aroclor 1232 at 50 ppm

Replicate	Surrogate 1[a]	Surrogate 2[b]	Aroclor
1	98	97	73
2	109	103	76
3	100	100	75
Avg.	102	100	75
%RSD	5.7%	3.0%	2.0%

Table 12.
Percent Recoveries of ANL-W
Simulated Sludge Spiked with Aroclor 1242 at 50 ppm

Replicate	Surrogate 1[a]	Surrogate 2[b]	Aroclor
1	83	83	70
2	78	75	67
3	87	79	68
Avg.	83	79	68
%RSD	5.4%	5.1%	2.2%

[a]Surrogate 1 is tetrachloro-meta-xylene.
[b]Surrogate 2 is decachlorobiphenyl.

Table 13.
Percent Recoveries of ANL-W
Simulated Sludge Spiked with Aroclor 1248 at 50 ppm

Replicate	Surrogate 1[a]	Surrogate 2[b]	Aroclor
1	65	60	73
2	64	59	69
3	68	63	74
Avg.	66	61	72
%RSD	3.2%	3.4%	3.7%

Table 14.
Percent Recoveries of ANL-W
Simulated Sludge Spiked with Aroclor 1254 at 50 ppm

Replicate	Surrogate 1[a]	Surrogate 2[b]	Aroclor
1	84	80	69
2	65	62	69
3	98	90	74
Avg.	82	77	71
%RSD	20.2%	18.4%	4.1%

[a]Surrogate 1 is tetrachloro-meta-xylene.
[b]Surrogate 2 is decachlorobiphenyl.

Table 15.
Percent Recoveries of ANL-W
Simulated Sludge Spiked with Aroclor 1260 at 50 ppm

Replicate	Surrogate 1[a]	Surrogate 2[b]	Aroclor
1	87	81	65
2	85	78	60
3	82	78	62
Avg.	85	79	62
%RSD	3.0%	2.2%	4.1%

Table 16.
Summary of Results: Average Recoveries of the Aroclors in
ANL-E and ANL-W Simulated Sludge

Aroclor	Percent Recoveries	
	ANL-E	ANL-W
1016	80	71
1221	67	78
1232	74	75
1242	73	68
1248	74	72
1254	75	71
1260	73	62

[a]Surrogate 1 is tetrachloro-meta-xylene.
[b]Surrogate 2 is decachlorobiphenyl.

REFERENCES

1. K. J. Parish, D. V. Applegate, A. S. Boparai and G. T. Reedy, Determination of PCBs in Rocky Flats Type IV Waste Sludge by Gas Chromatography/Electron Capture Detection, Argonne National Laboratory Technical Report ANL/ACL-93/1 (December 1993).

2. U.S. Environmental Protection Agency, Statement of Work for Organic Analysis, Contract Laboratory Program, OLM02.0 Including Revision OLM02.1, U.S. EPA, Washington, DC.

3. U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Ed., Proposed Update II, U.S. EPA, Washington, DC (November 1992).

APPENDIX A

Two-gram portions of the sludge samples were prepared for analysis using the same procedure described in Section 7.0 and 7.1 of the original report, with two exceptions. In the original report, the step of adding sodium sulfate to the sludge was performed, however it was omitted in error from the report. After the data in the original report were collected, it was discovered that pre-wetting the Florisil cartridge with hexane during the cleanup step was unnecessary and may decrease the capacity of the cartridge to remove interferences from oil extracts, therefore the step of pre-wetting the cartridge was omitted. The corrected extraction procedure should read as follows:

Extraction Procedure

Weigh approximately 2.0 g (measured to 0.01 g) of a sludge sample into a 20 mL extraction vial in a radiochemical hood or a glovebox. Also add 2 g of sodium sulfate to the vial and mix using a vortex mixer. Add 50 μL of surrogate stock standard solution to each sample and blank so the final extract concentration of each surrogate is 0.05 μg/mL. Then add 10 mL of hexane to each sample and vortex mix for at least 30 seconds.

Cleanup

After vortexing, allow the sample to settle (one minute or more). Remove a 50 μL portion of the supernatant and add to a 1 mL glass or polypropylene syringe connected to a Florisil cartridge. Add approximately 0.4 mL of hexane to the syringe. Apply pressure or vacuum to move the solution through the cartridge without introducing air into it. Then add approximately 0.5 mL of hexane to the syringe as a rinse for a final volume of 1.0 mL. Collect the extract and rinses in an autosampler vial calibrated to 1.0 mL.

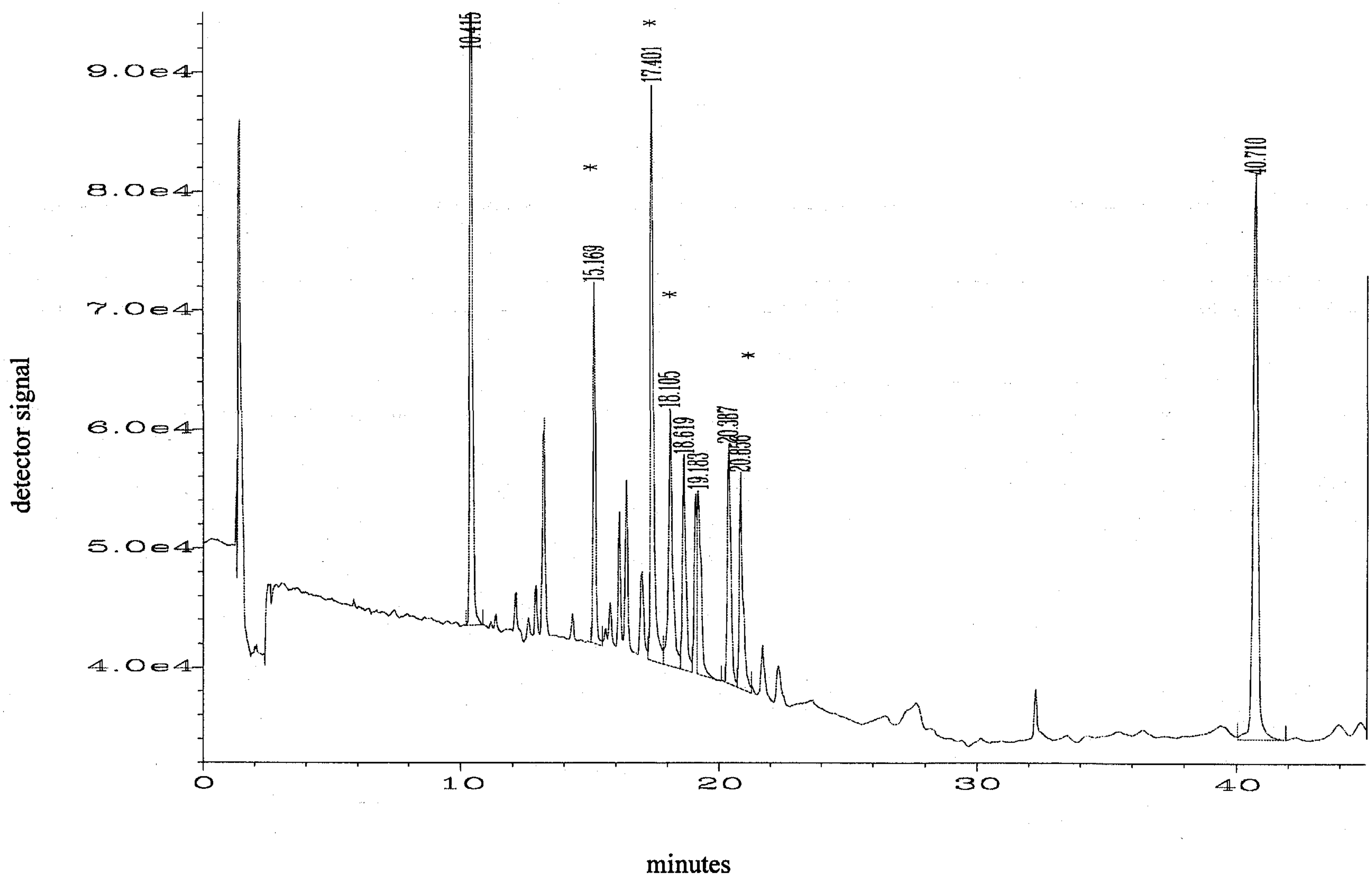

Figure 1. Aroclor 1016 Standard at 0.50 ppm Concentration

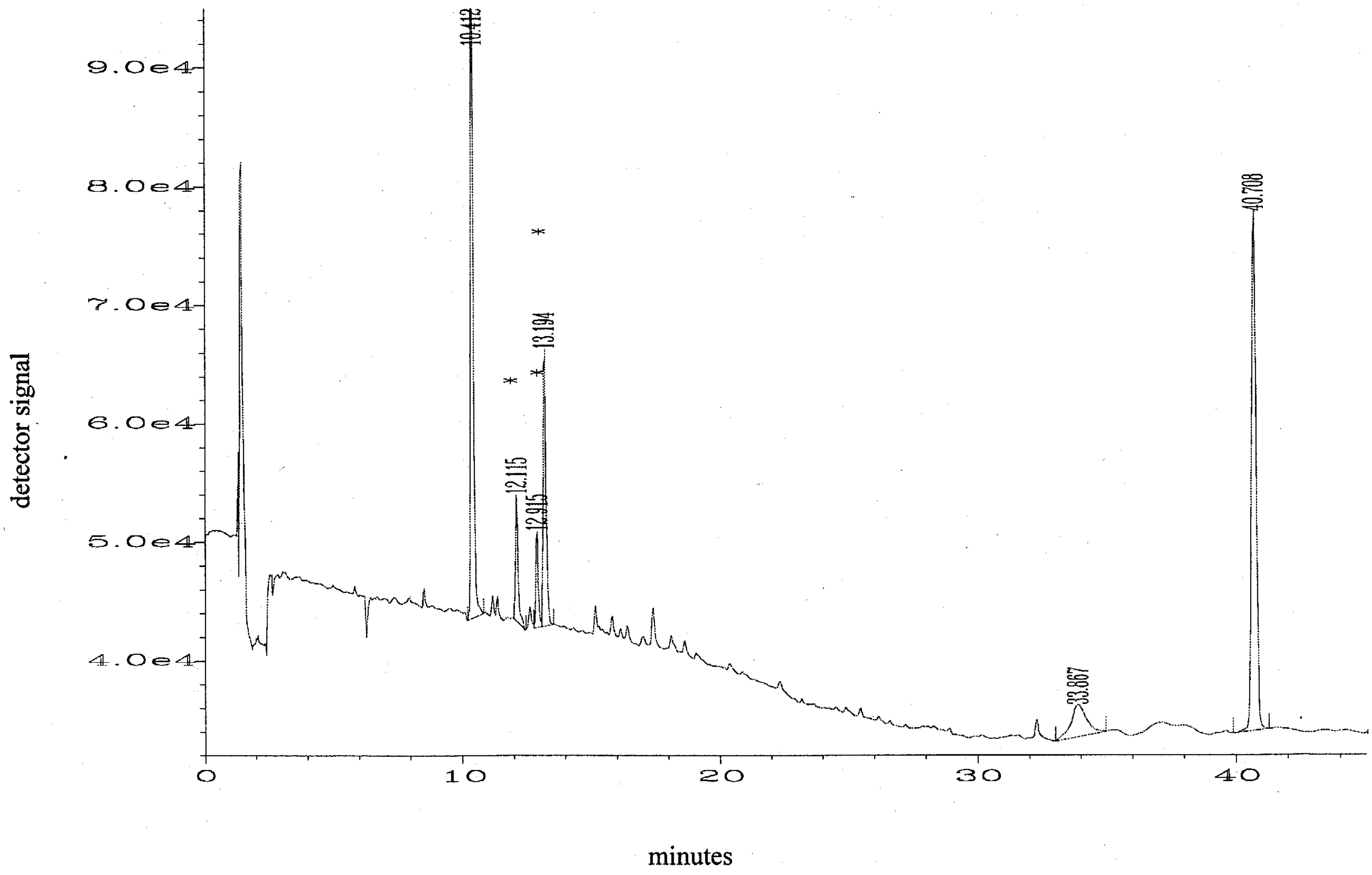

Figure 2. Aroclor 1221 Standard at 0.50 ppm Concentration

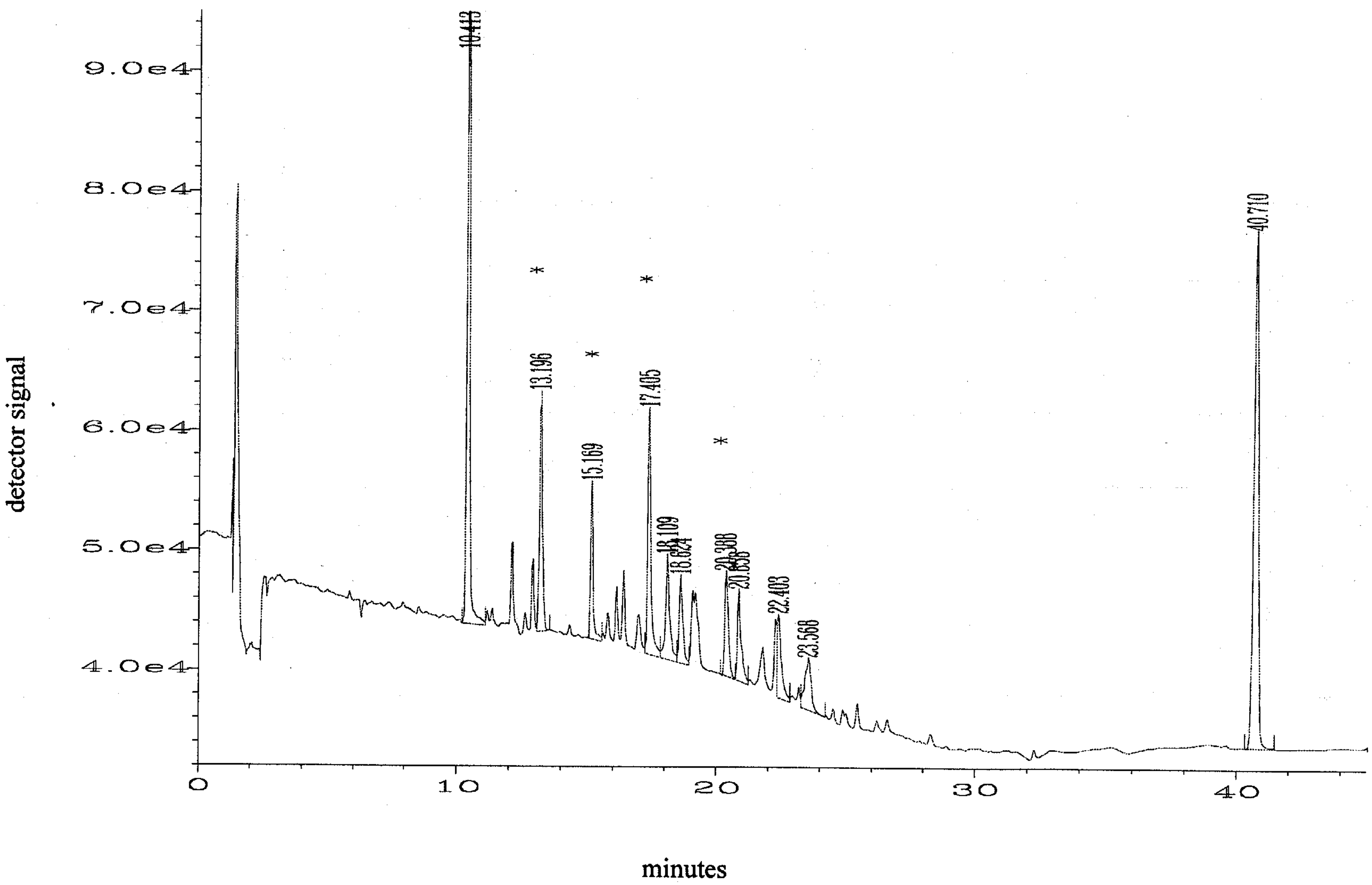

Figure 3. Aroclor 1232 Standard at 0.50 ppm Concentration

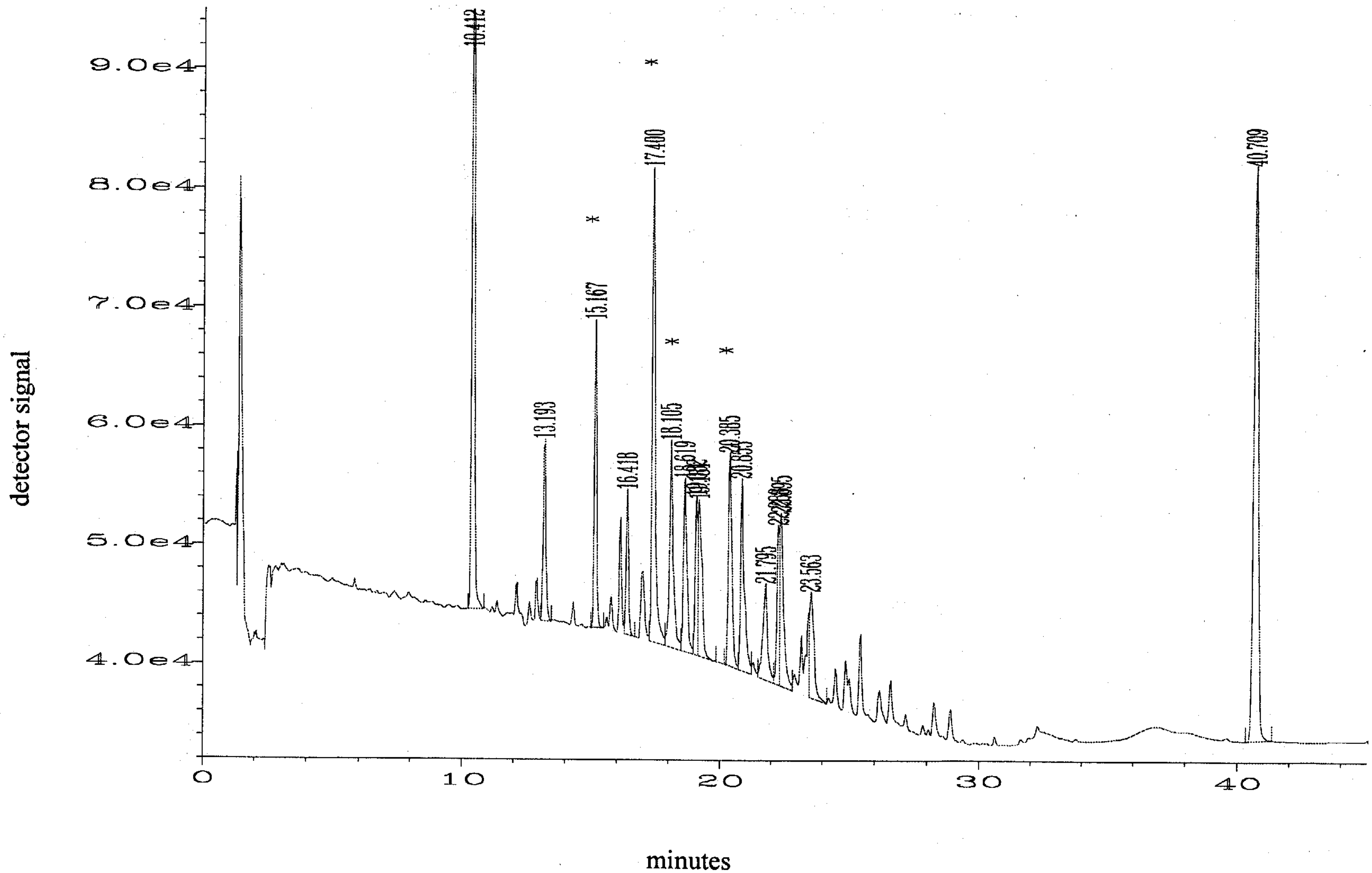

Figure 4. Aroclor 1242 Standard at 0.50 ppm Concentration

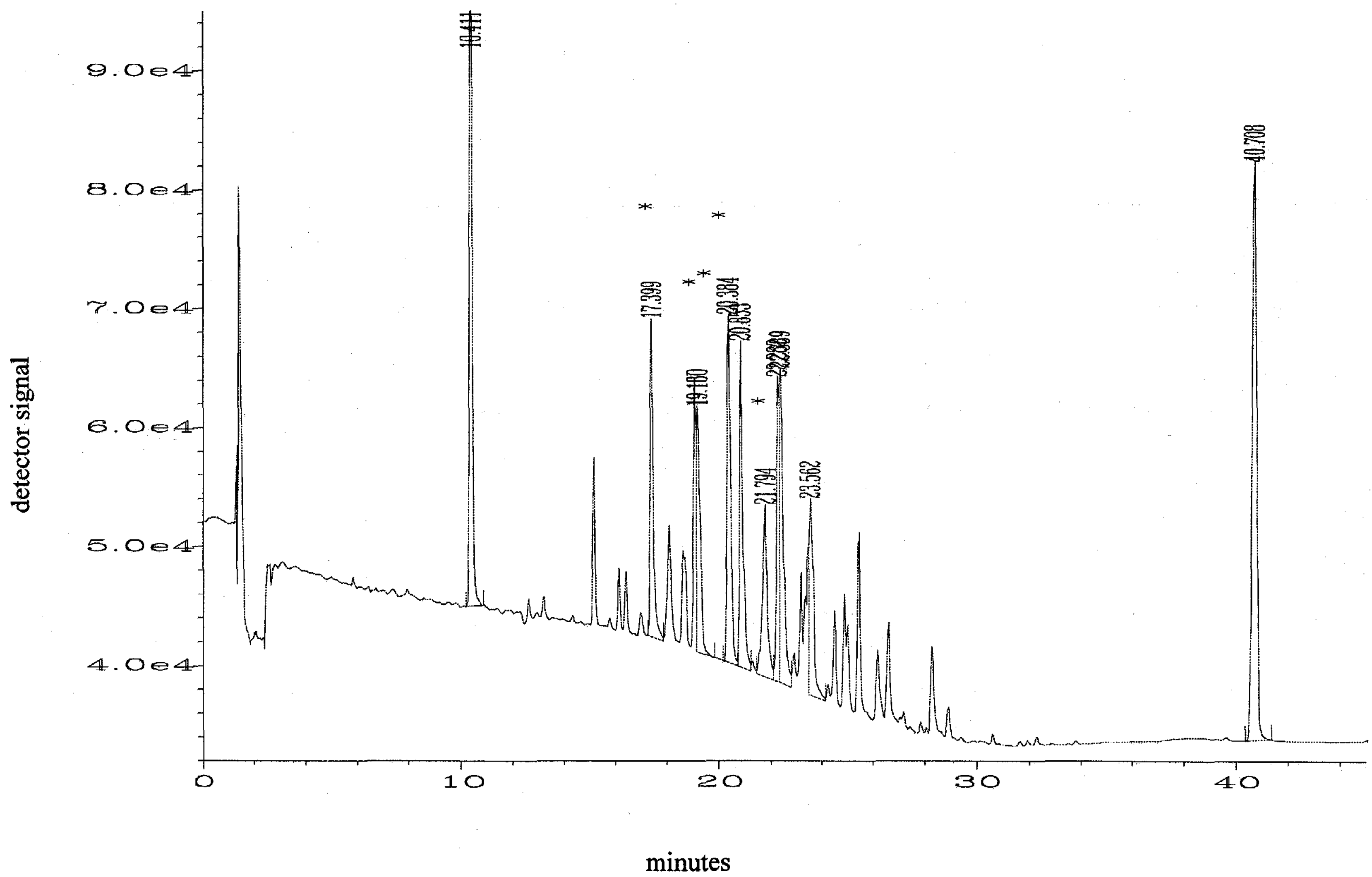

Figure 5. Aroclor 1248 Standard at 0.50 ppm Concentration

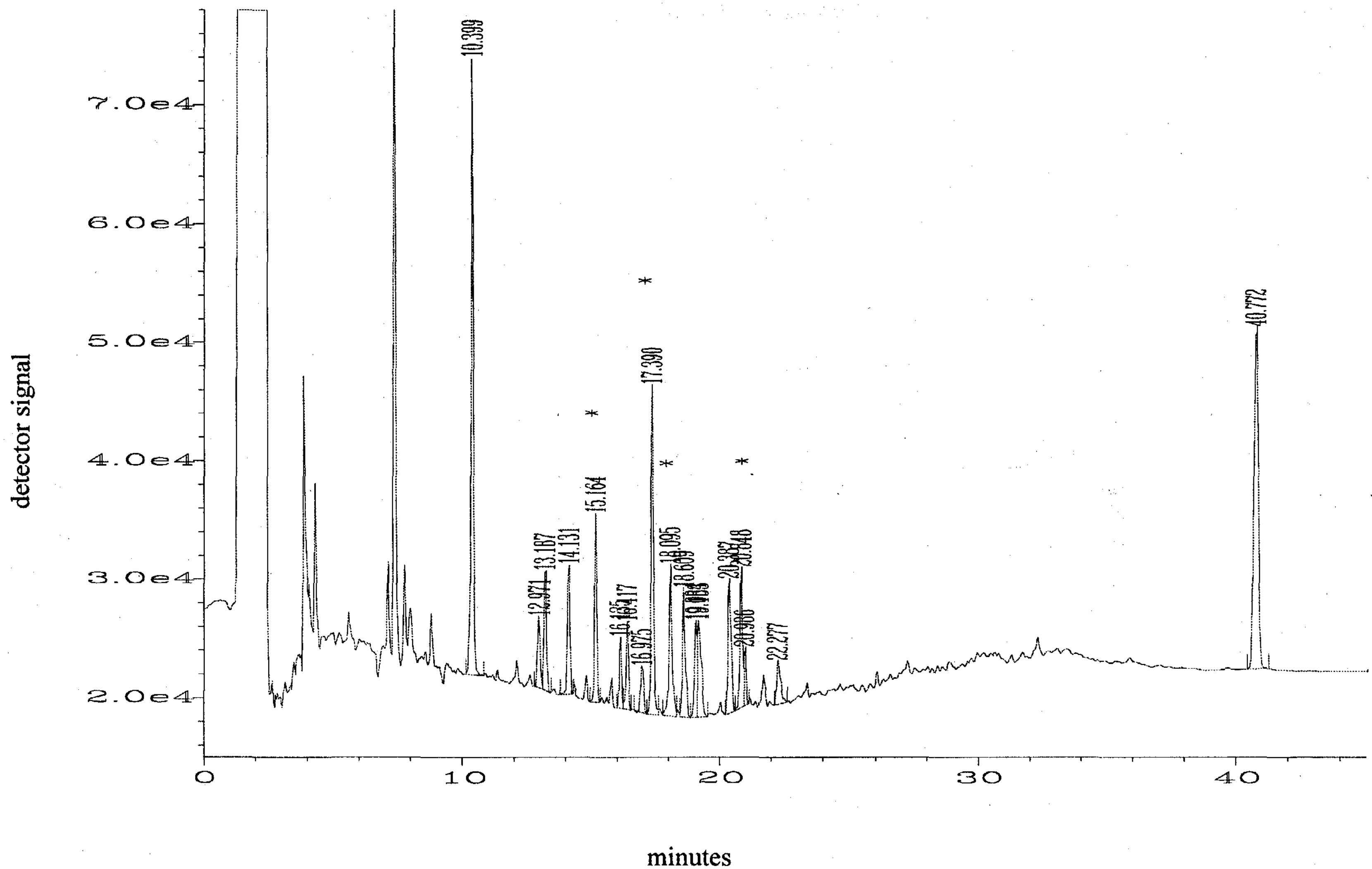

Figure 6. Extract of ANL-E Simulated Sludge Spiked with 50 ppm of Aroclor 1016

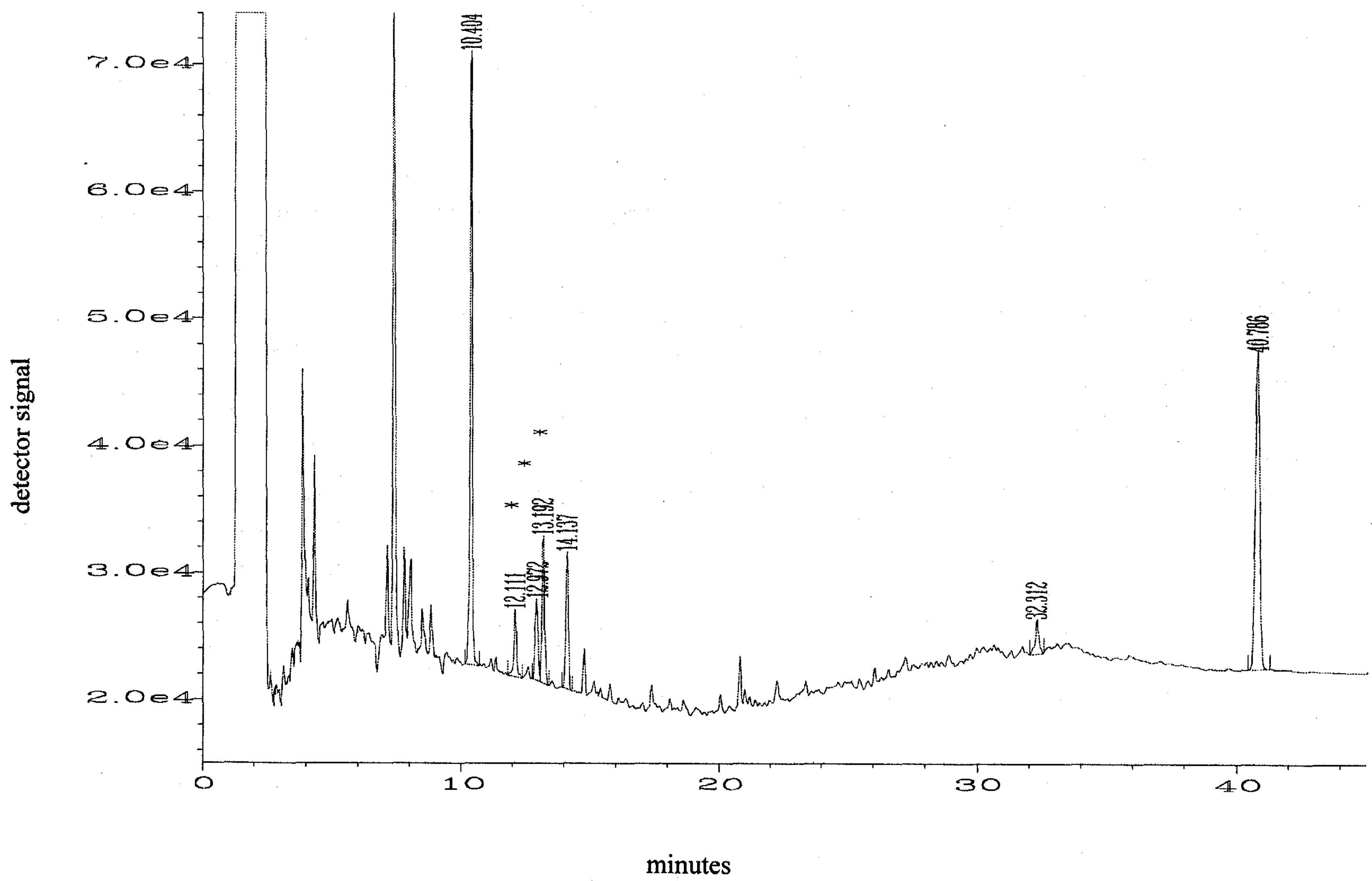

Figure 7. Extract of ANL-E Simulated Sludge Spiked with 50 ppm of Aroclor 1221

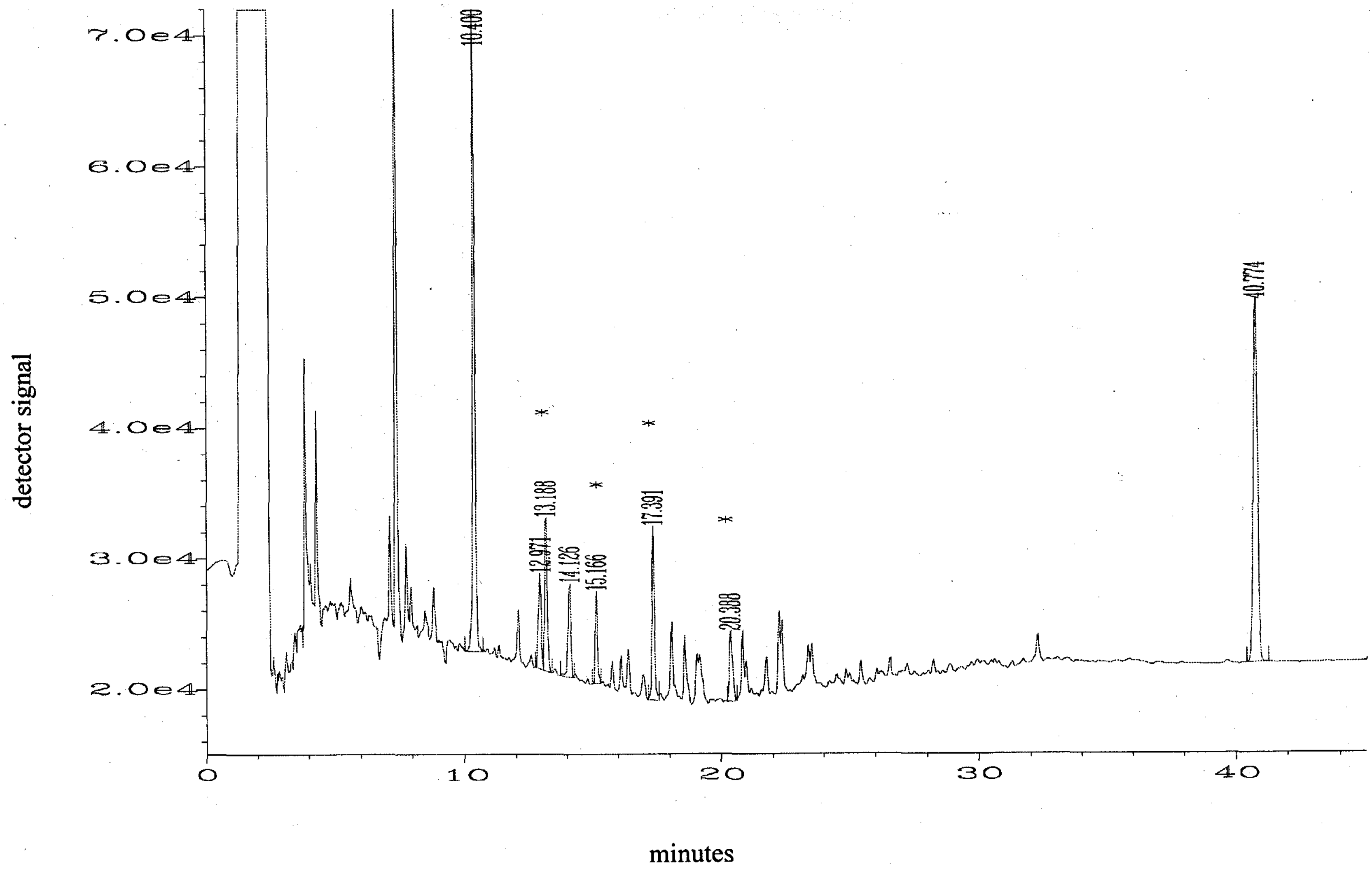

Figure 8. Extract of ANL-E Simulated Sludge Spiked with 50 ppm of Aroclor 1232

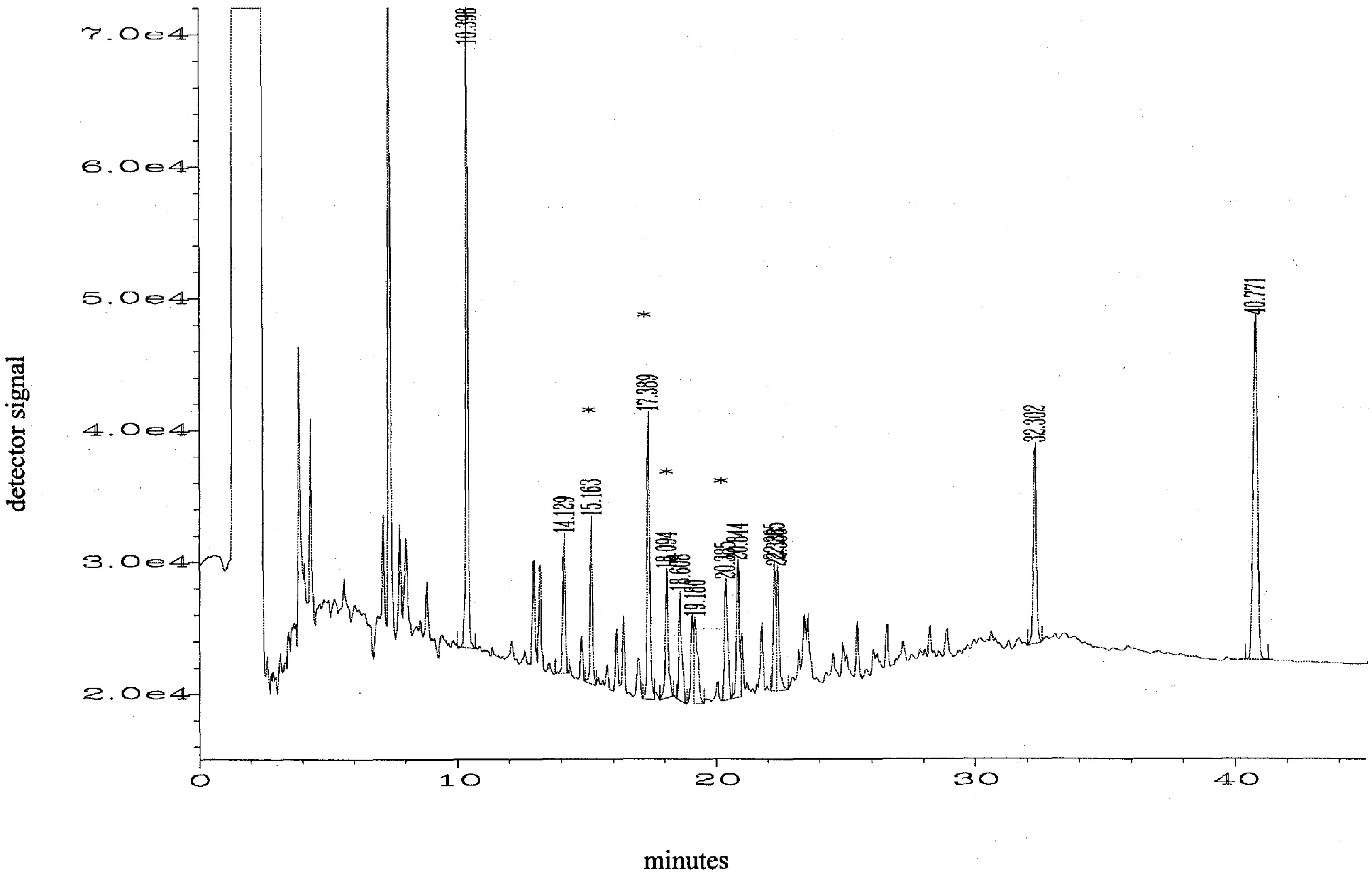

Figure 9. Extract of ANL-E Simulated Sludge Spiked with 50 ppm of Aroclor 1242

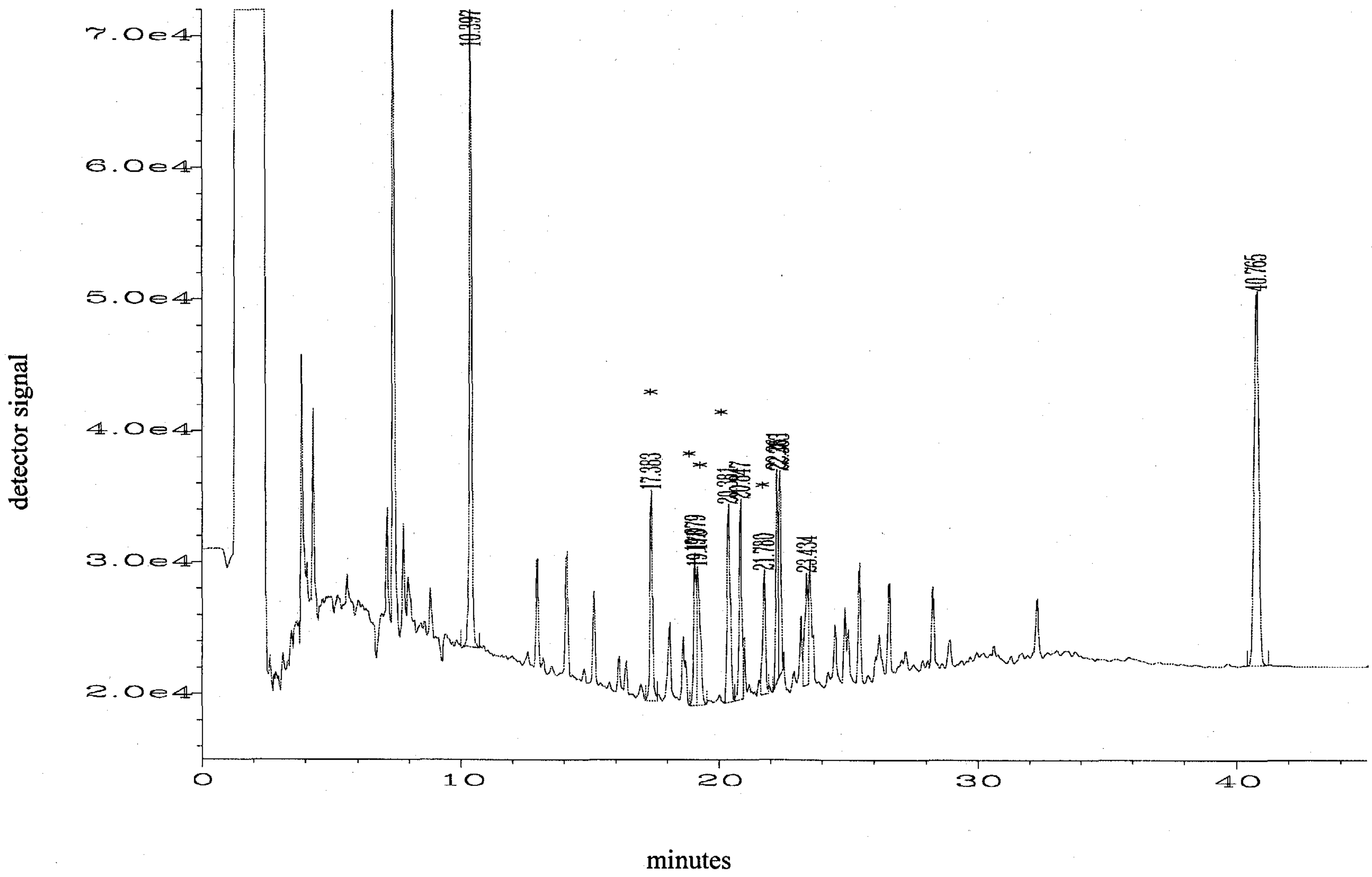

Figure 10. Extract of ANL-E Simulated Sludge Spiked with 50 ppm of Aroclor 1248

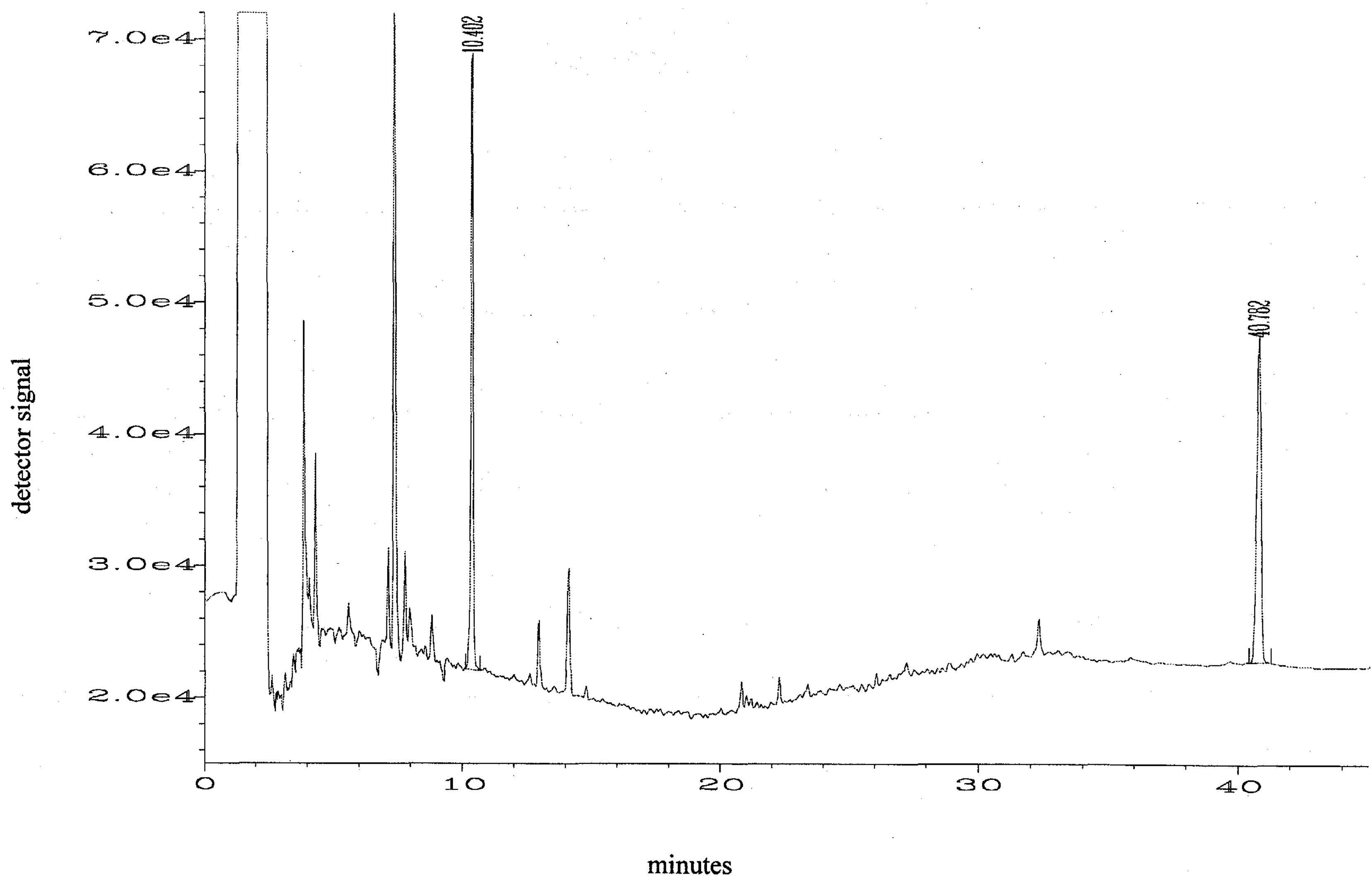

Figure 11. Extract of ANL-E Simulated Sludge with No Aroclors Added

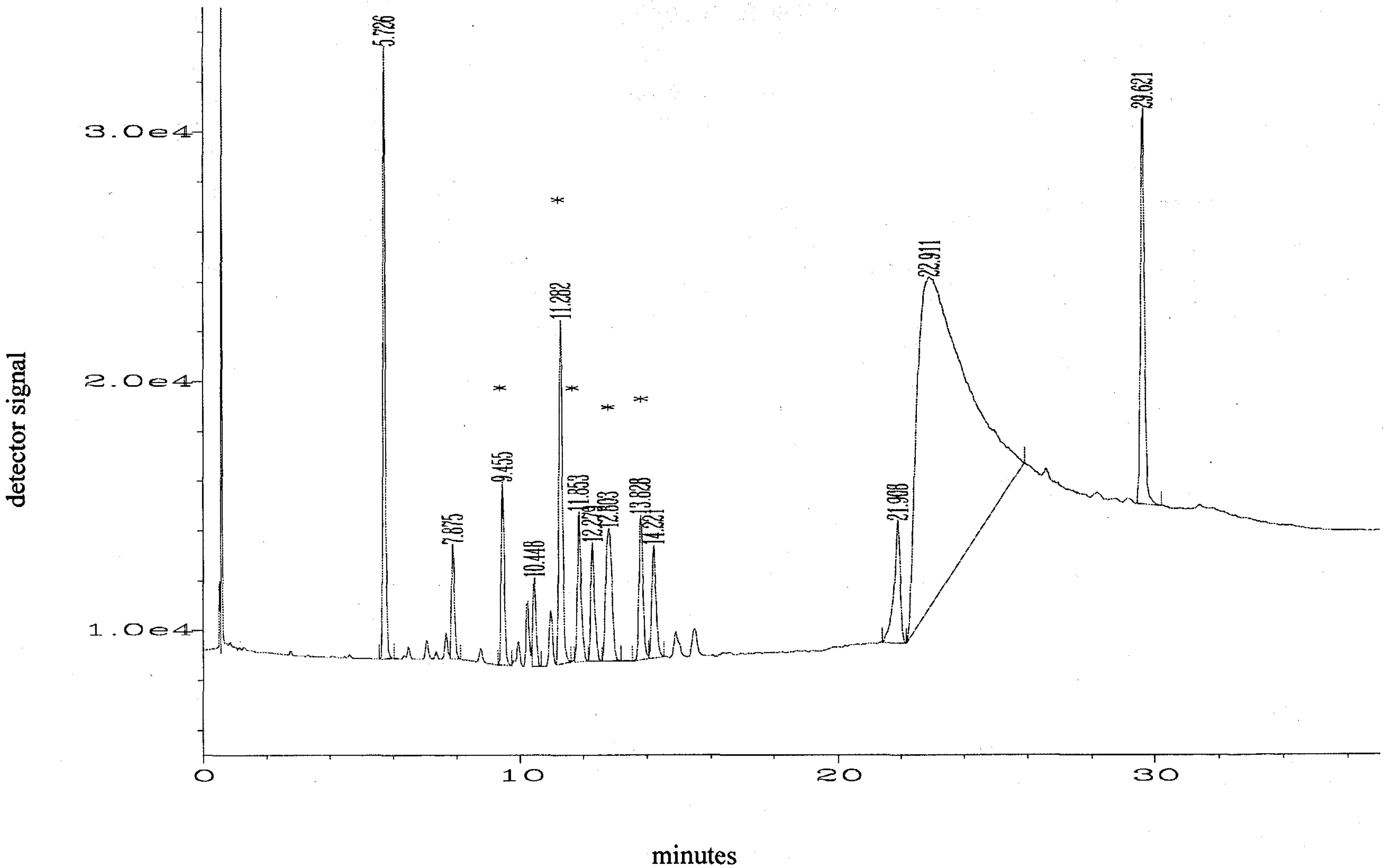

Figure 12. Aroclor 1016 Standard at 0.50 ppm Concentration

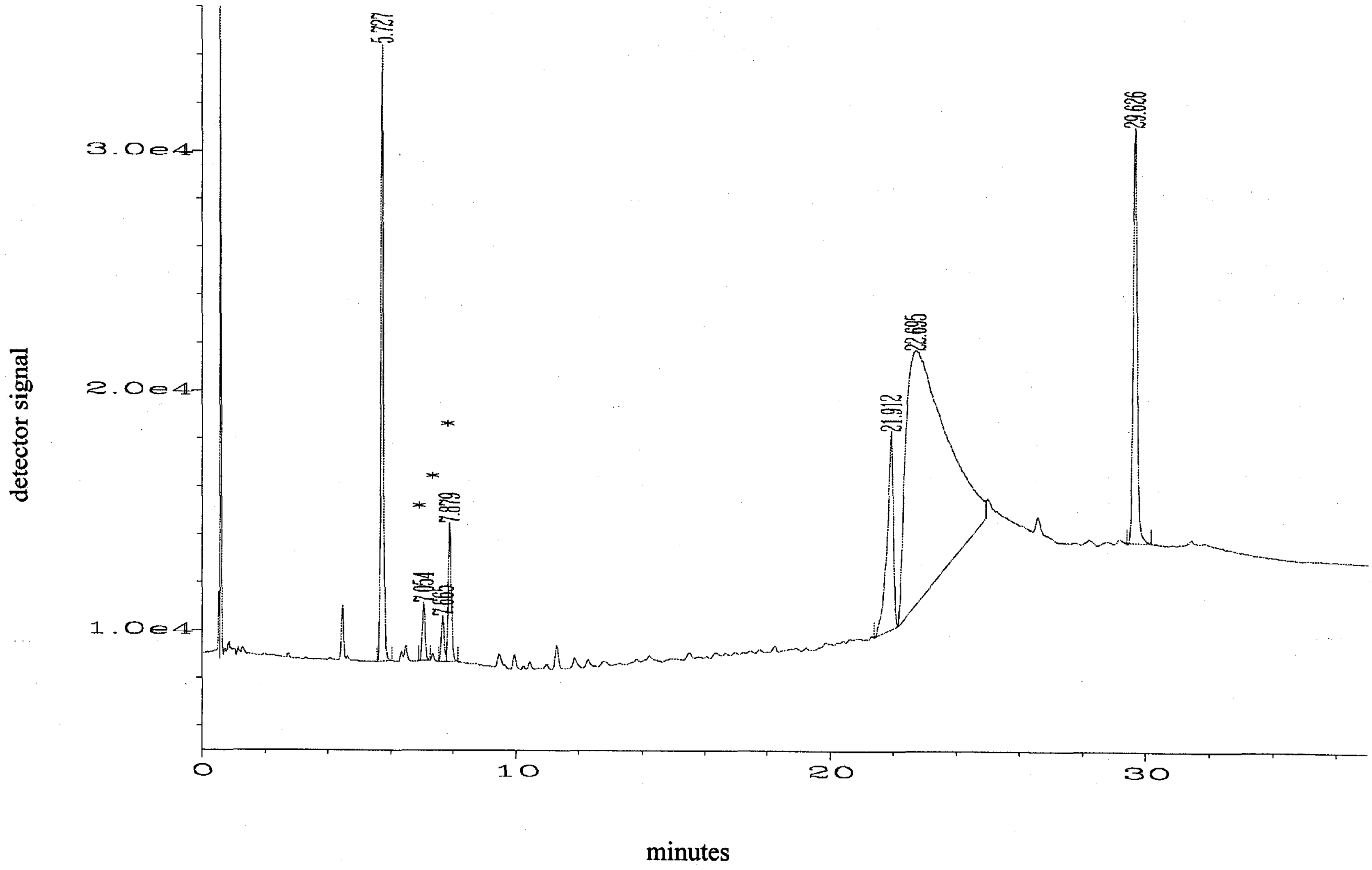

Figure 13. Aroclor 1221 Standard at 0.50 ppm Concentration

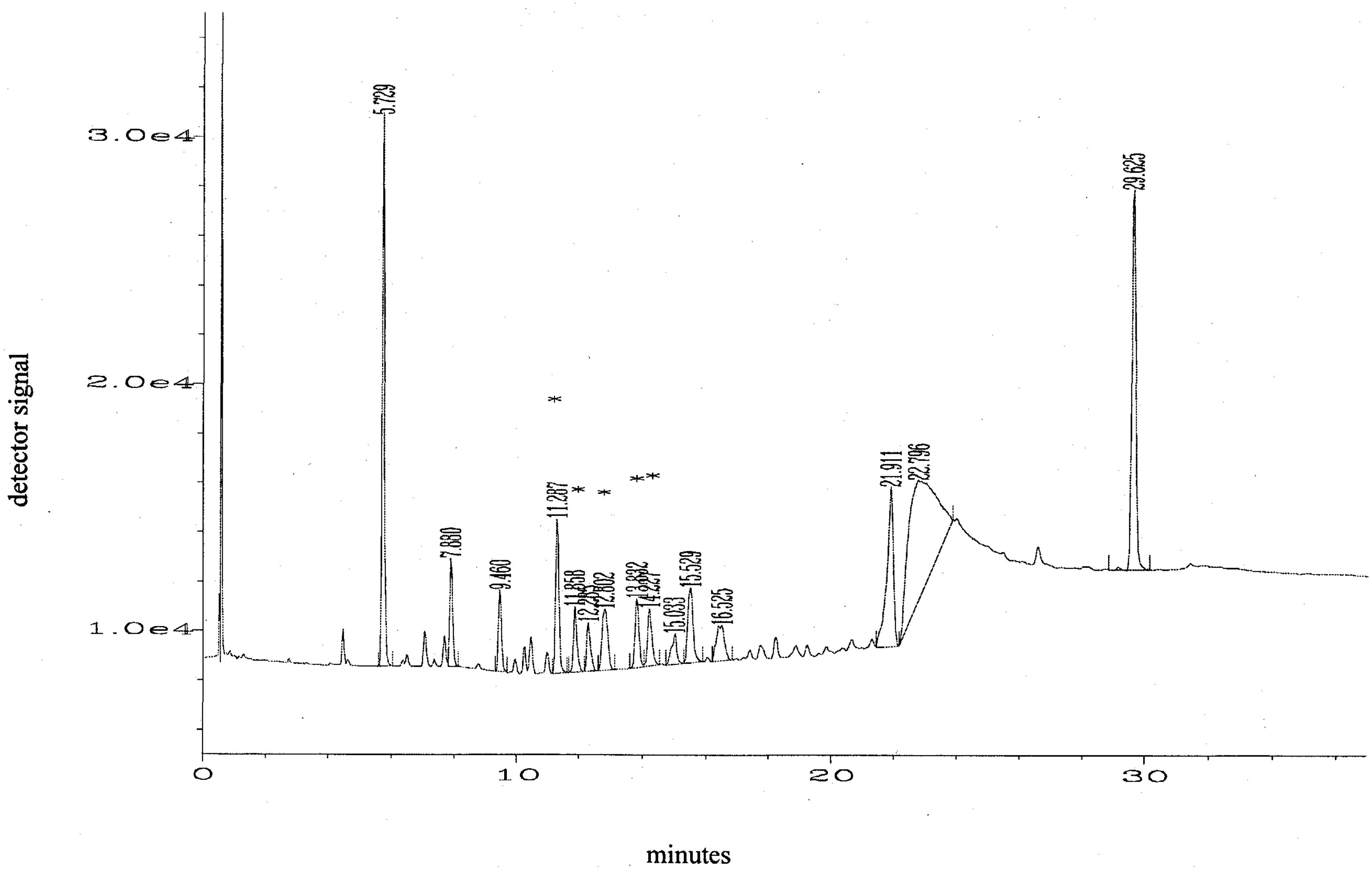

Figure 14. Aroclor 1232 Standard at 0.50 ppm Concentration

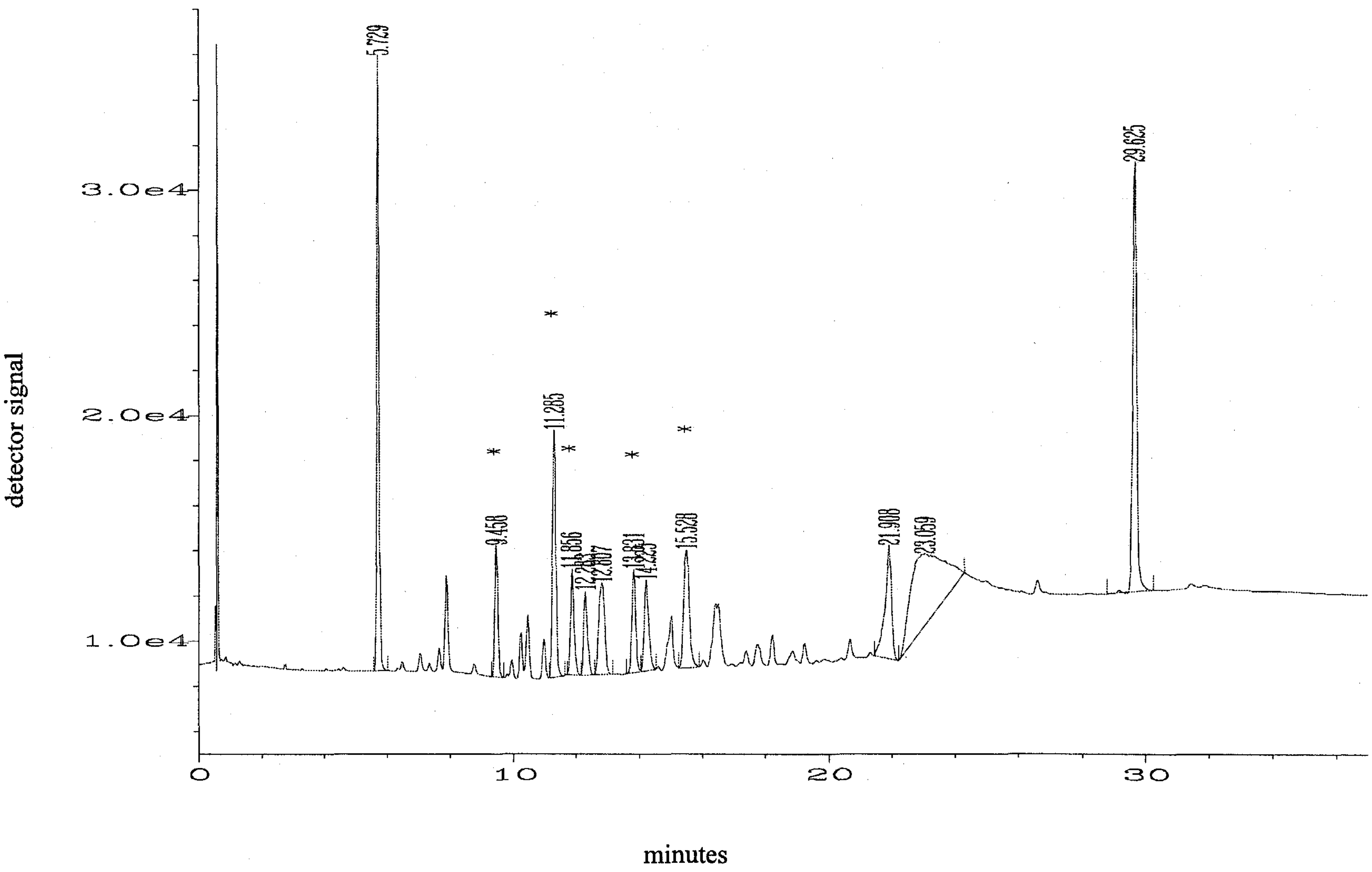

Figure 15. Aroclor 1242 Standard at 0.50 ppm Concentration

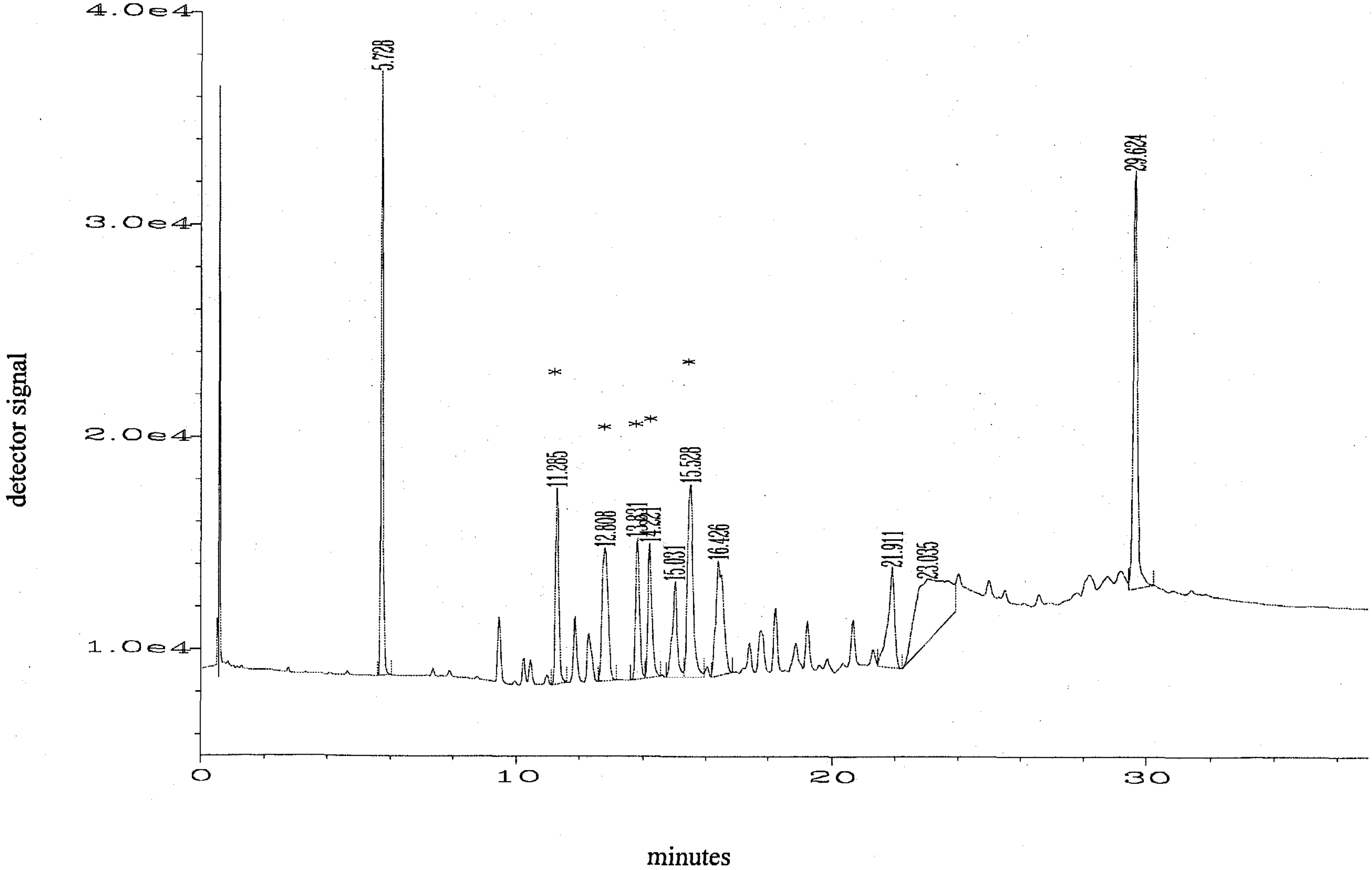

Figure 16. Aroclor 1248 Standard at 0.50 ppm Concentration

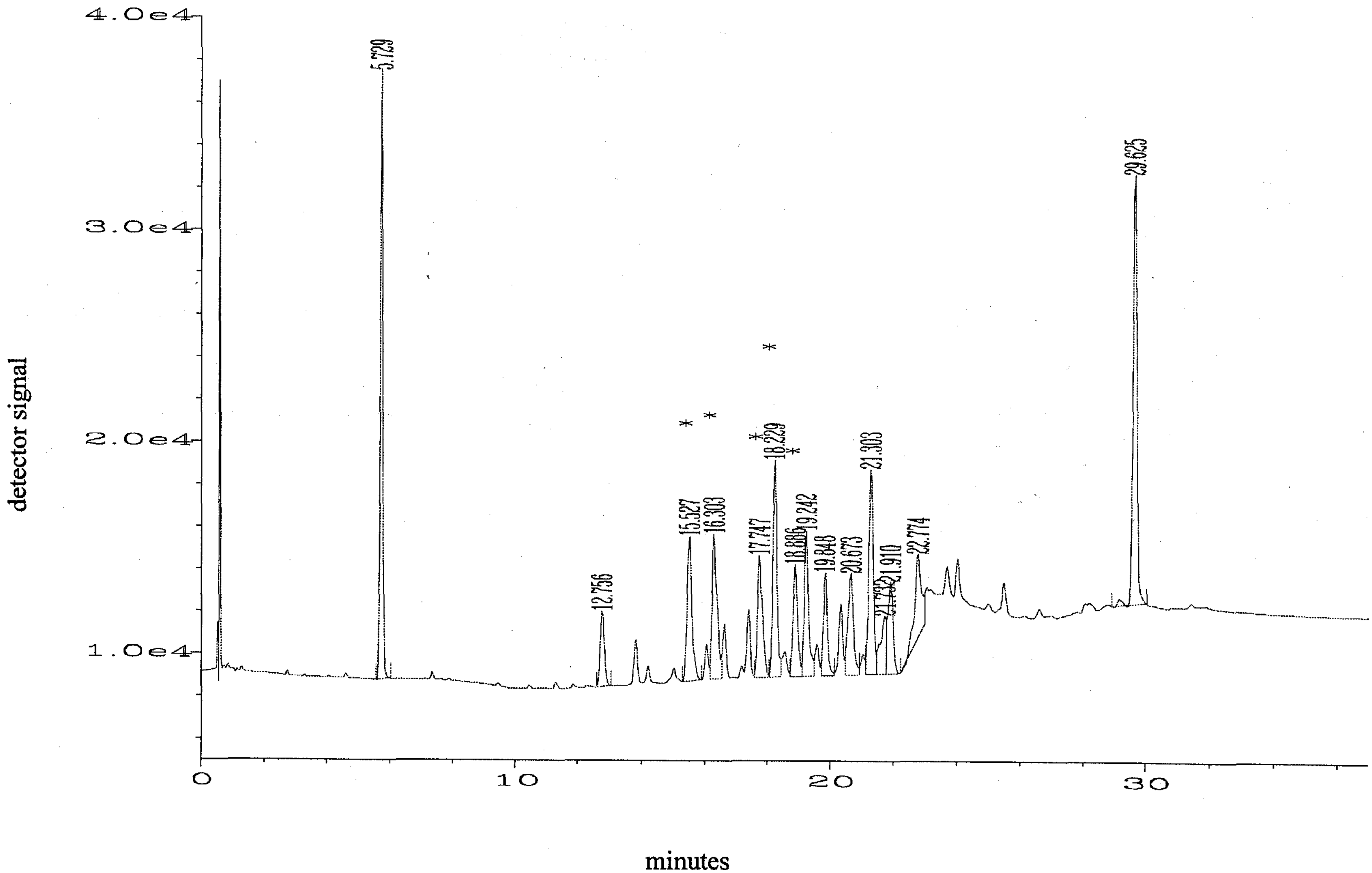

Figure 17. Aroclor 1254 Standard at 0.50 ppm Concentration

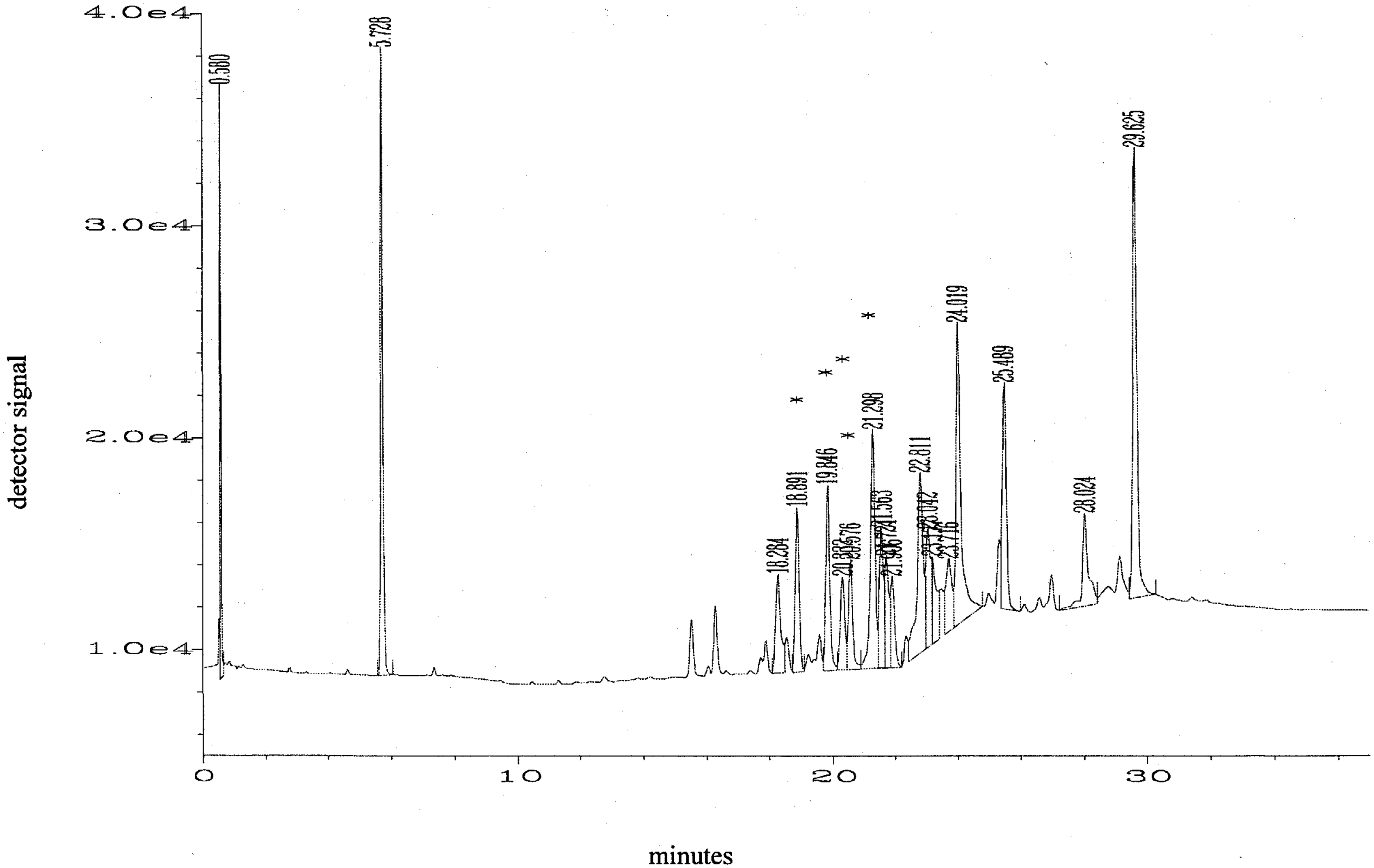

Figure 18. Aroclor 1260 Standard at 0.50 ppm Concentration

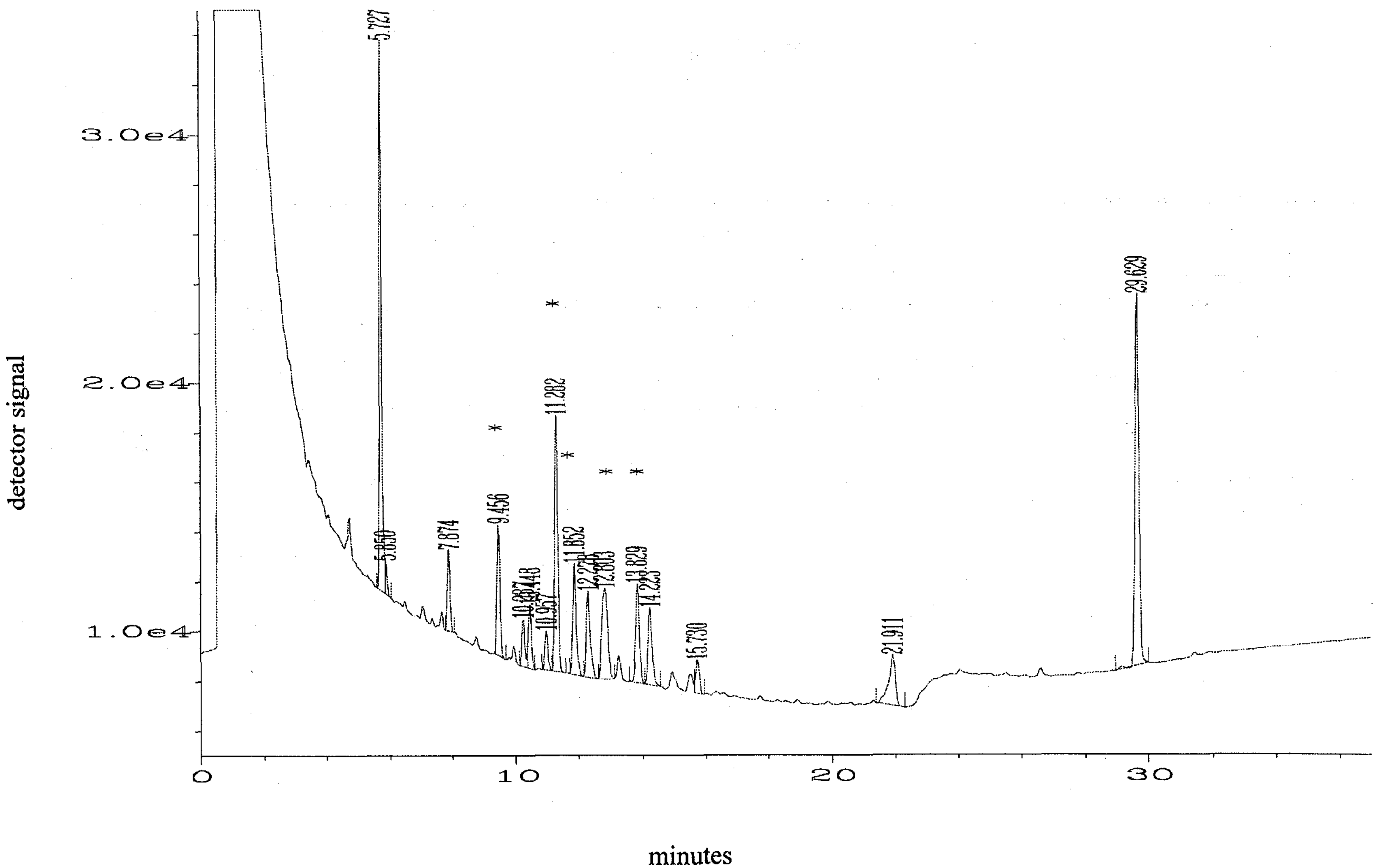

Figure 19. Extract of ANL-W Simulated Sludge Spiked with 50 ppm of Aroclor 1016

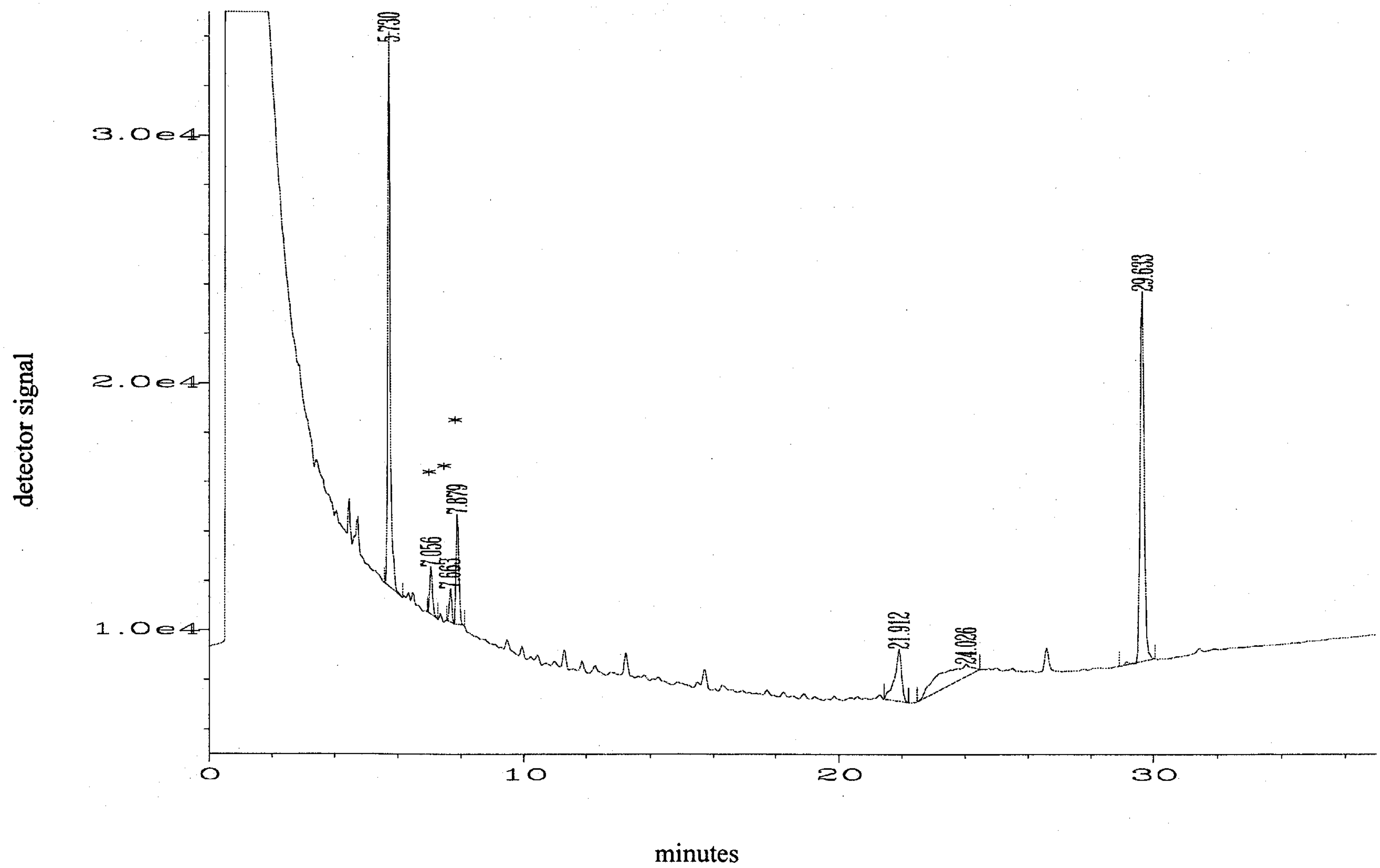

Figure 20. Extract of ANL-W Simulated Sludge Spiked with 50 ppm of Aroclor 1221

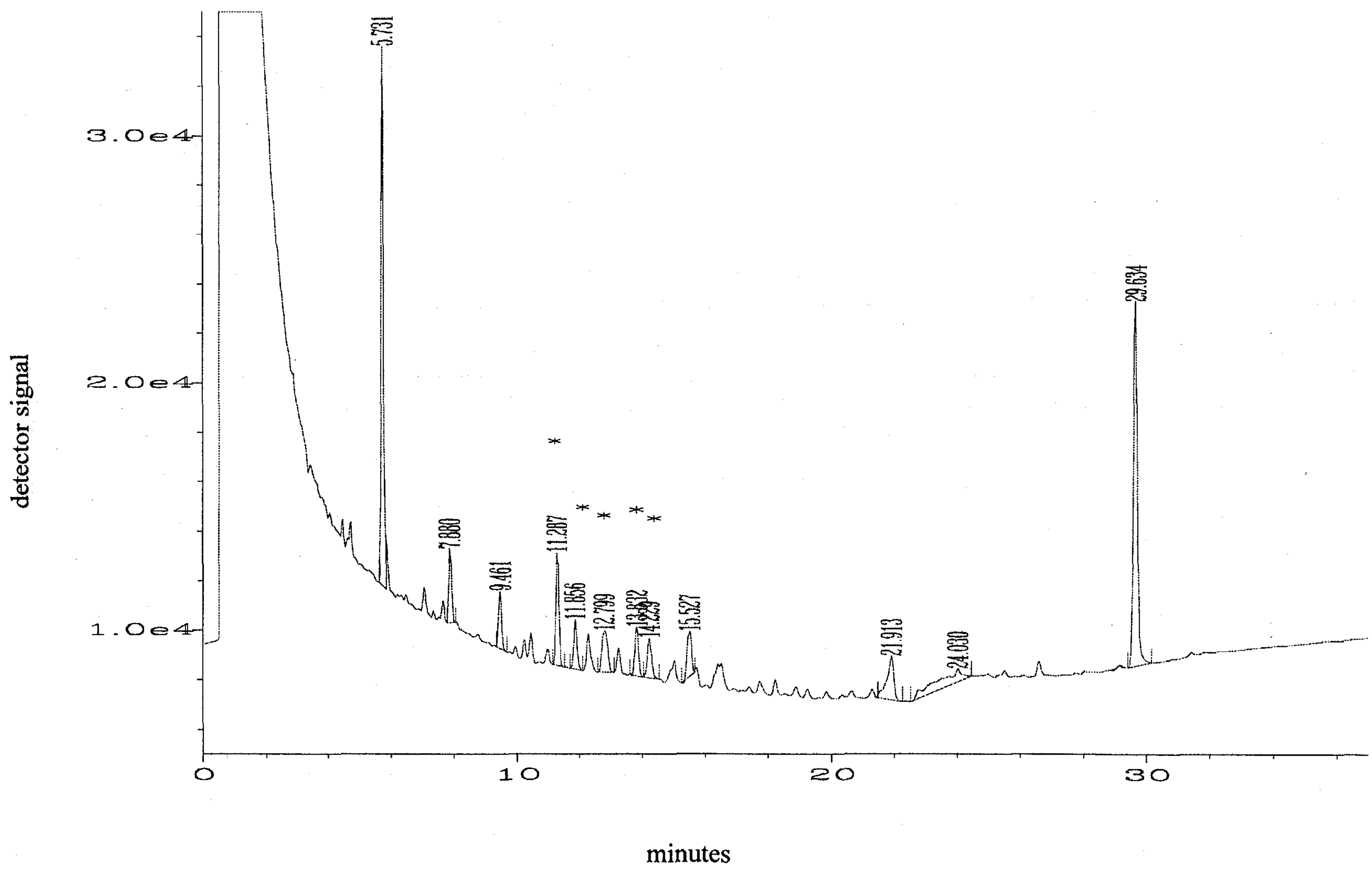

Figure 21. Extract of ANL-W Simulated Sludge Spiked with 50 ppm of Aroclor 1232

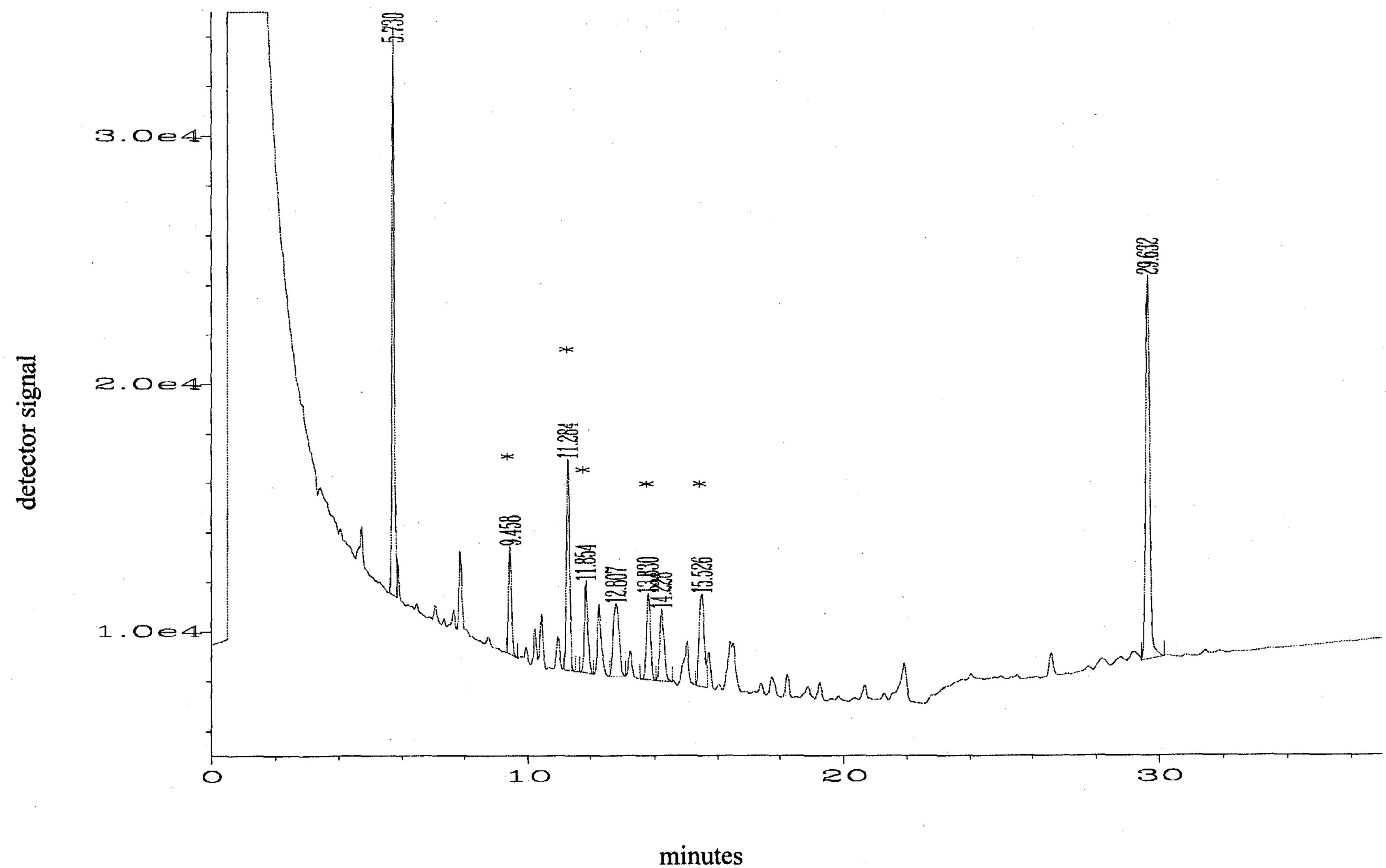

Figure 22. Extract of ANL-W Simulated Sludge Spiked with 50 ppm of Aroclor 1242

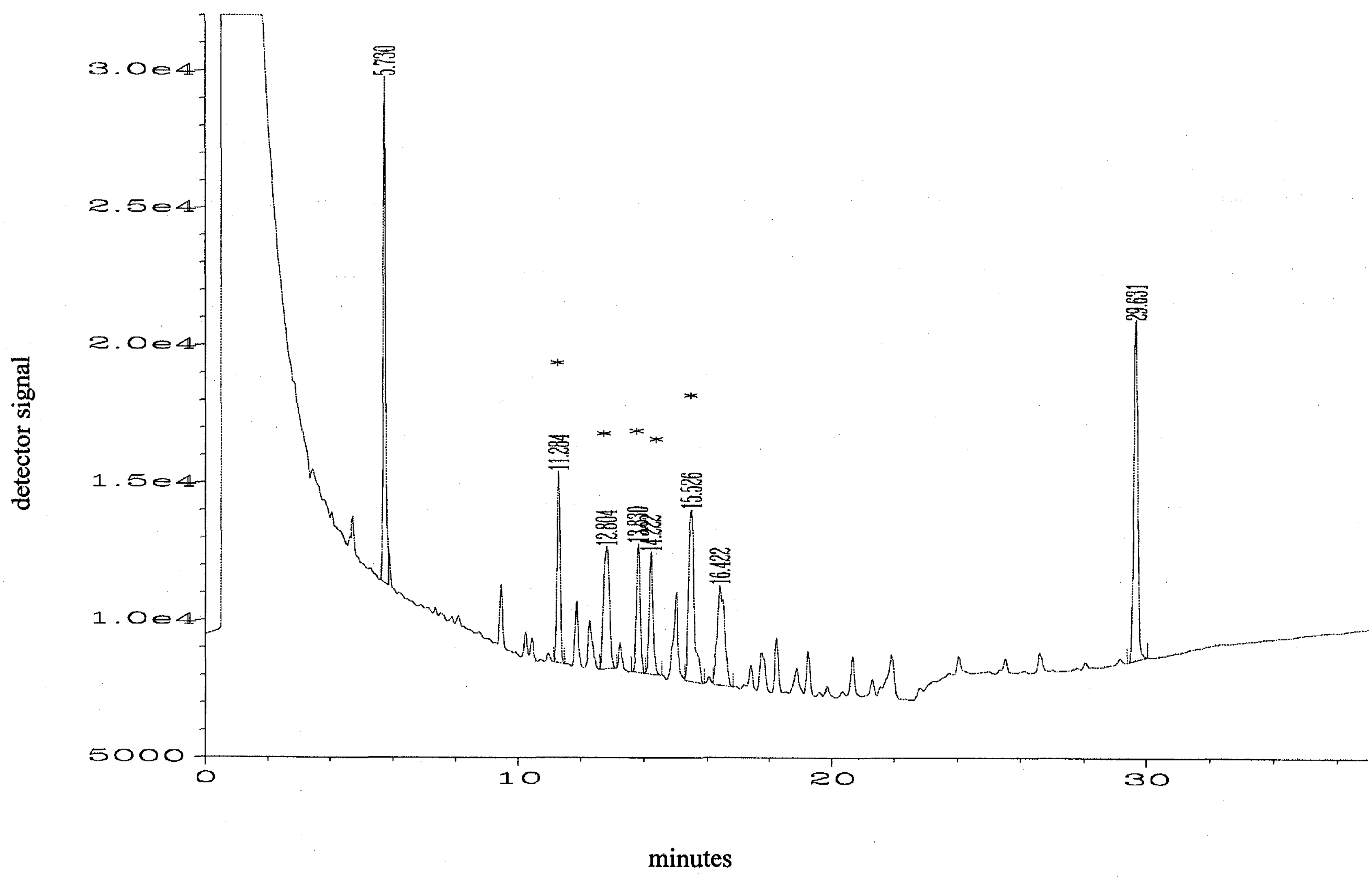

Figure 23. Extract of ANL-W Simulated Sludge Spiked with 50 ppm of Aroclor 1248

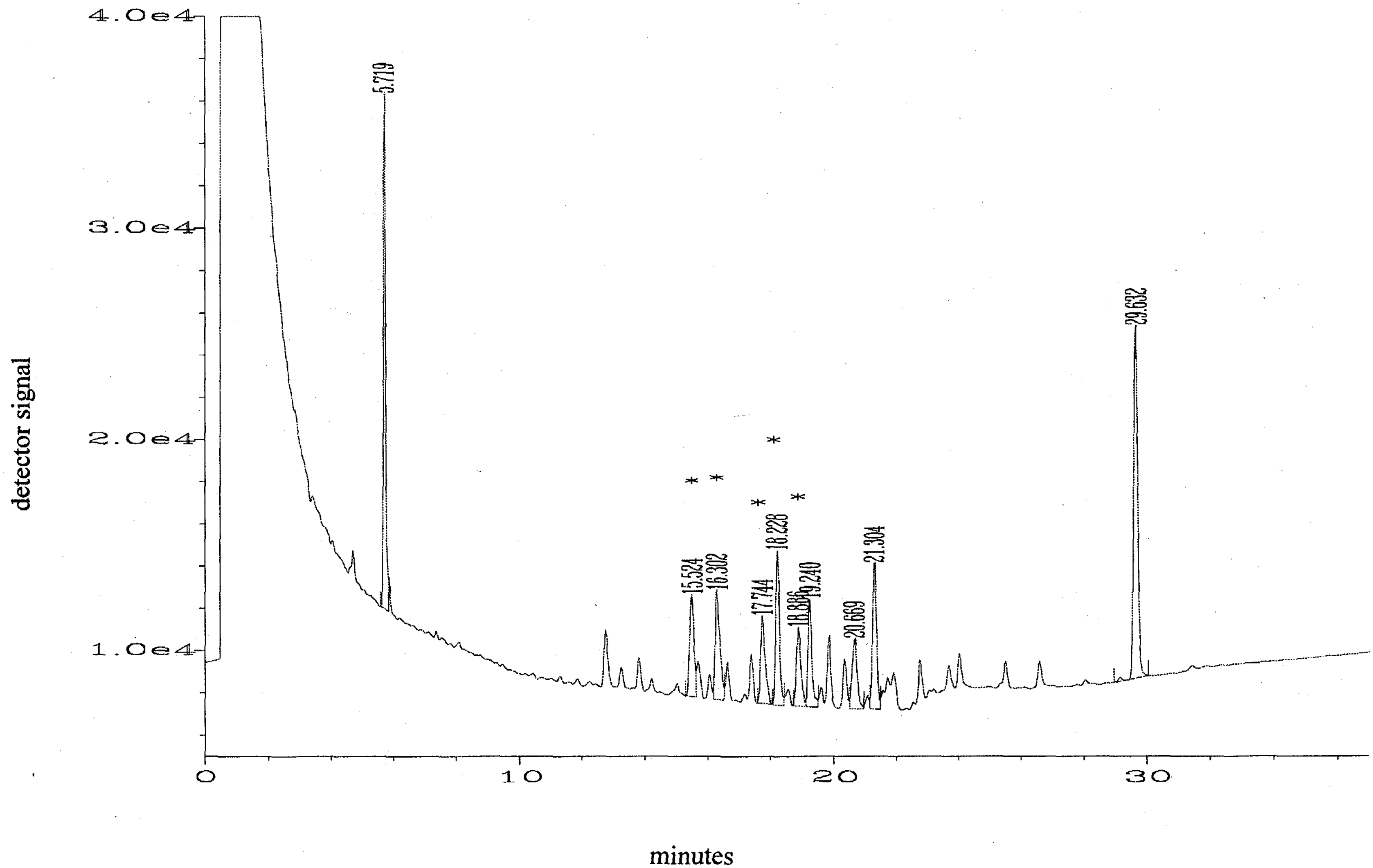

Figure 24. Extract of ANL-W Simulated Sludge Spiked with 50 ppm of Aroclor 1254

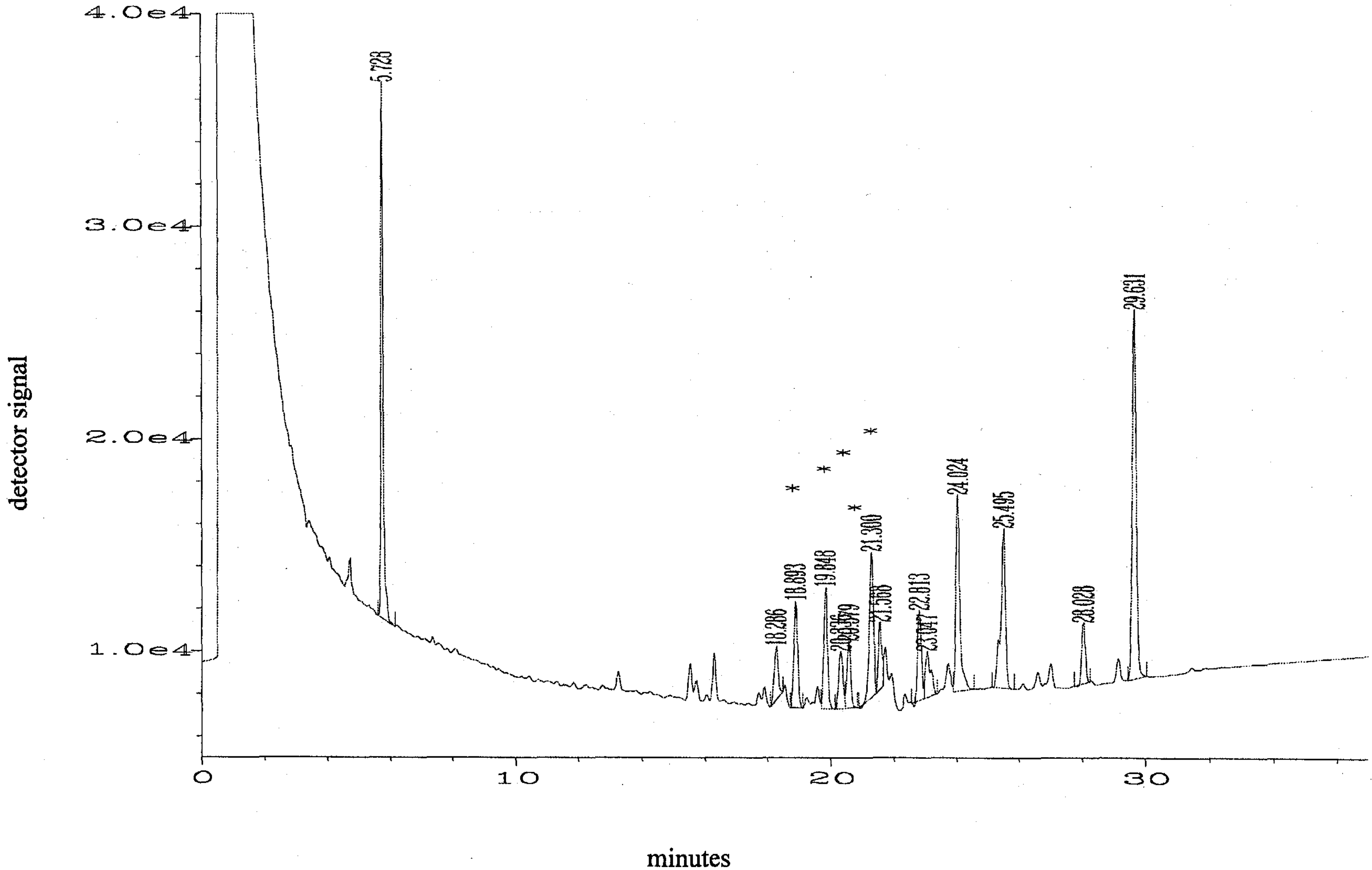

Figure 25. Extract of ANL-W Simulated Sludge Spiked with 50 ppm of Aroclor 1260

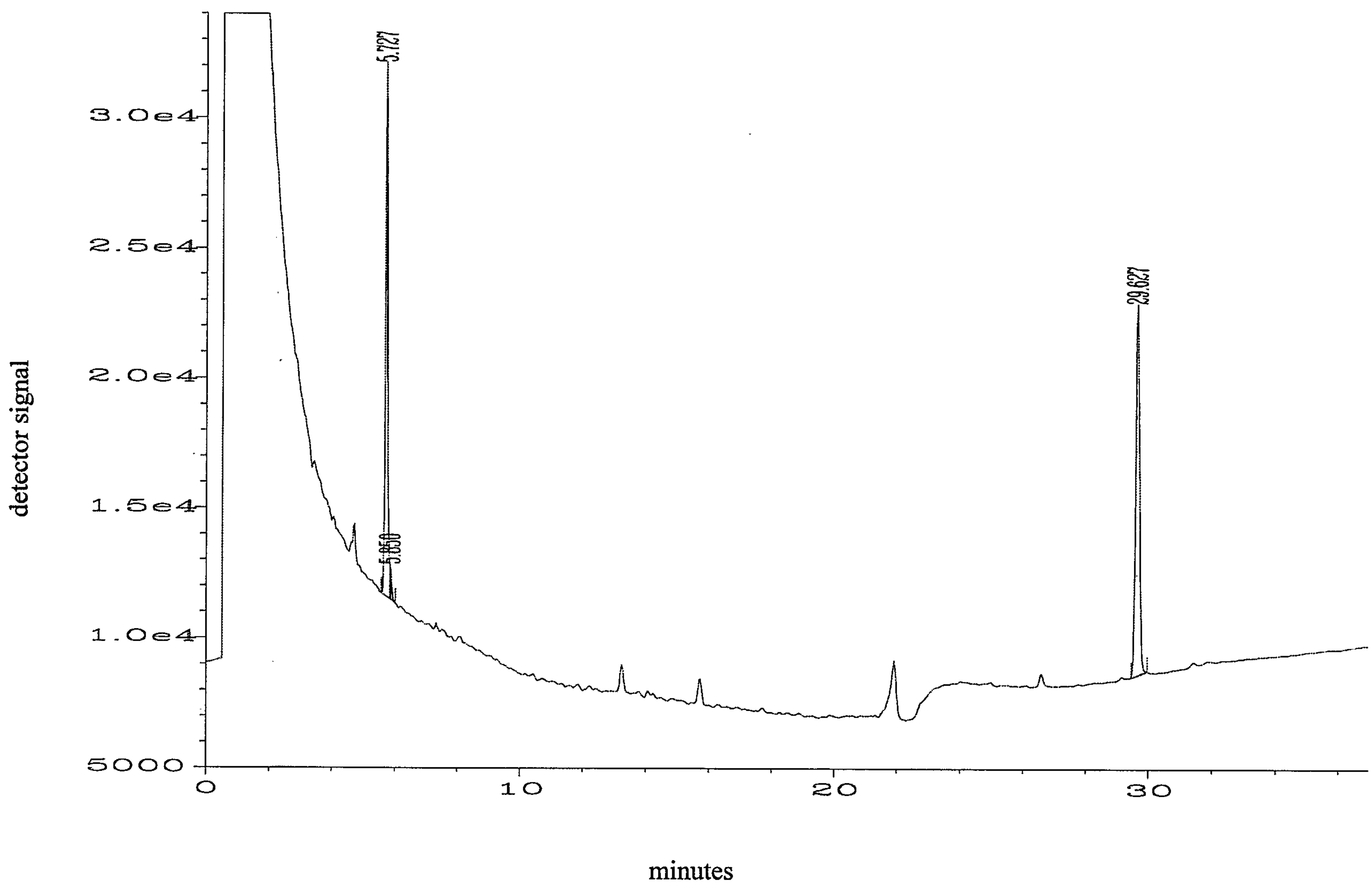

Figure 26. Extract of ANL-W Simulated Sludge with No Aroclors Added

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