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ANALYSIS FOR POLYBROMINATED BIPHENYLS (PBBs) IN ENVIRONMENTAL SAMPLES

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ANALYSIS FOR POLYBROMINATED BIPHENYLS (PBBs) IN ENVIRONMENTAL SAMPLES

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DISCLAIMER

This report has been reviewed by the Office of Toxic Substances, U.S. Environmental Protection Agency, and approved for publication. Approval does not signify that the contents necessarily reflect the views and policies of the U.S. Environmental Protection Agency, nor does mention of trade names or commercial products constitute endorsement or recommendation for use.

ABSTRACT

Ninety-eight environmental samples consisting of fish, sediments, soils, and vegetation were analyzed by gas chromatographic/mass spectrometry (GC/MS) techniques for hexa-, hepta-, octa-, nona-, and decabromobiphenyls. The samples were collected by EPA regional office personnel. Hexabromobiphenyl was the most prevalent species detected. This species was detected in 84 percent of the samples over the range of 0.2 to 50,000 ppb. Heptabromobiphenyl was detected in 12 percent of the samples and octabromobiphenyl in 2 percent of the samples. Nonabromobiphenyl and decabromobiphenyl were not detected.

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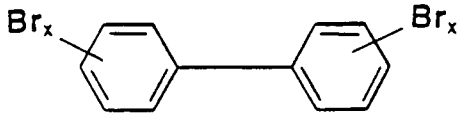
1.0 INTRODUCTION

Polybrominated biphenyls (PBBs) are a class of chemical compounds with a chemical structure as follows:



where X can be any number from 0 to 5. These compounds were first produced in the United States in commercial quantities in 1970. The compounds have been used as flame retardants for plastics, textiles, and other materials.

Polybrominated biphenyls (PBBs) are used extensively as flame retardants for polymers, textiles, and other materials.[1] The major component of the most common commercial mixture, Fire Master PB-6®[2], is hexabromobiphenyl.[3-7] Concern about the environmental impact and health effects of wide spread use of PBBs has been aroused by a recent major environmental catastrophe in Michigan.[4-8] Their chemical similarity to polychlorinated biphenyls (PCB) and polychlorinated naphthalenes (PCN) has raised the question of bioconcentration and toxicity to the point to warrant a test of the carcinogenicity of hexabromobiphenyl by the National Cancer Institute Bioassay Program.[9]

In response to the problem in Michigan, analytical methodologies for PBBs have been developed and applied to feeds[5], dairy products[4] and soils.[6,7] Although extensive analytical work has been carried out with respect to samples originating in Michigan, little attention has been directed to the possibility of pollution resulting from manufacture and use of PBBs in other locations throughout the United States.

In this document is reported the results of the analysis of environmental samples collected in the vicinity of several locations where PBBs have been used for the manufacture of various products. Samples were collected by personnel from the following organizations:

- Sets a/h New Jersey Division of Fish and Game
- Set b Ohio EPA, Southwest District
- Set c EPA Region III, Wheeling, West Virginia
- Set d EPA Region III, Nashville, Tennessee
- Set e EPA Region IX, Alameda, California
- Set f New York State Department of Environmental Conservation, Buffalo, New York
- Sets g/i EPA Region V, Central District Office
- Set j EPA Office of Toxic Substances

The samples were sent to Environmental Science and Engineering, Inc. for extraction, clean-up, and concentration. All samples were received in good condition except those taken in the vicinity of General Tire and Rubber, Corporation, Ionia, Michigan, which could not be analyzed. The sample extracts were shipped to Research Triangle Institute (RTI) for analysis.

2.0 METHODS

2.1 Sample Preparation

Soils. Soil samples were extracted with hexane in a soxhlet apparatus. The extract was concentrated to 1.0 ml on a Kuderna-Danish evaporator, cleaned up on a florisil chromatographic column, and packaged in hermetically sealed vials prior to GC/MS analysis.

Sediments and Sludges. Sediment and sludge samples were extracted with hexane in a soxhlet apparatus, and the extract cleaned up in the same manner as described above for soils. The moisture content was determined on a separate fraction of the homogenized sample.

Biological Tissue. Terrestrial vegetation was analyzed for PBB by rinsing a measured quantity with hexane in a beaker. The hexane was subsequently concentrated on a Kuderna-Danish evaporator concentrator to 1.0 ml and subjected directly to GC/MS analysis. The resulting analytical data was calculated as mass of PBB accumulated on a unit mass of vegetation.

Animal tissue was hard frozen shortly after collection. To a measured quantity of crushed, frozen tissue was added 5 g of clean, sharp sand and 5 g of anhydrous sodium sulfate (Na_2SO_4). Whole fish were used, since those analyzed were generally small in size. The tissue was then ground to obtain a uniform, dry granular mass. This material was extracted with hexane in a soxhlet extraction apparatus. The hexane extract was concentrated to 0.5 ml on a Kuderna-Danish apparatus and passed through a florisil chromatographic column using the procedures of Fehringer[4,5] and EPA[10]. The eluate was concentrated to 1.0 ml and placed in a hermetically sealed vial prior to GC/MS analysis. Moisture content of the tissue was determined on a separate fraction of the homogenate. Analytical results are reported on a dry weight basis.

2.2 Sample Analysis

The sample extracts were stored frozen until shipment to RTI for GC/MS analysis. The extracts were shipped on ice by air courier.

Upon receipt by RTI, samples were taken to dryness under a slow stream of dry nitrogen. The residue was then taken up in an appropriate volume of dry toluene and the appropriate standards (octachloronaphthalene and decachlorobiphenyl) added at a level of approximately 2 ng/ul. The samples were analyzed by gas chromatograph/mass spectrometer/computer utilizing either a Finnigan 3300 GC/MS equipped with a PDP-12 computer (chromatographic column, 45 cm x 0.2 cm i.d., glass, 2% OV-101 on Gas-Chrom Q; helium flow, 20 cc/min; initial temperature, 220°C programmed to 280°-300°C at 4°/min; separator temperature, 250°C; injector temperature, 280°C) or an LKB-2091-050 EI/CI (chromatographic column, 45 cm x 0.2 cm i.d., glass, 2% OV-101 on Gas-Chrom Q; helium flow, 18 cc/min; initial temperature, 220°C for one minute, then programmed to 290°C at 8°/min; separator temperature, 275°C; injector temperature, 280°C; ion source temperature, 250°C). The polybrominated biphenyls were analyzed by selected ion monitoring techniques (see Appendix C for ions monitored and retention times observed).

3.0 RESULTS

A total of 144 samples were received. Appendix A lists all the samples received and the origin of the shipment. Ninety-eight of these samples were selected by the EPA Project Officer for extraction and analysis. The results of these analyses are shown in Table 1.

Appendix B is a summary of the PBB concentration in each sample extract. The retention times and the ions monitored are listed in Appendix C. The ratios of ions used to confirm the presence of PBBs are described in Appendix D and full scan spectral confirmation of representative samples are shown in Appendix E. Appendix F are representative selected ion monitoring traces.

4.0 CONCLUSIONS

The frequence of detection of the various PBB species is summarized in Table 2. Hexabromobiphenyl was the most prevalent species detected. This species was detected in 84 percent of the samples over the range of 0.2 to 50,000 ppb. Heptabromobiphenyl was detected in 12 percent of the samples and octabromobiphenyl in 2 percent of the samples. Nonabromobiphenyl and decabromobiphenyl were not detected.

Table 1.
Estimated Levels of Polybrominated Biphenyls in Environmental Samples (Page 1 of 14)

Samples Collected By	Sample Code	Sample Type and Location	PBB Concentration (ng/g), parts per billion				
			$C_{12}H_4Br_6$	$C_{12}H_3Br_7$	$C_{12}H_2Br_8$	$C_{12}HBr_9$	$C_{12}Br_{10}$
a	5-2	FISH Hackensack R. at Sawmill Cr.	3.4	--	--	--	--
a	5-13	FISH Hackensack R. at Sawmill Cr.	0.7	--	--	--	--
a	5-17	FISH Hackensack River at Moonachie Cr.	0.6	--	--	--	--
a	7-2	FISH Newark Bay, Bayonne Area	3.1	1.6	1.8	--	--
a	7-4	FISH Newark Bay, Bayonne Area	<0.1	--	--	--	--
a	7-6	SEDIMENT Newark Bay, Bayonne Area	--	--	--	--	--
a	7-7	FISH Arthur Kill/Howland Hook Staten Is.	--	--	--	--	--
a	7-8	SEDIMENT Kill Van Kull, Staten Is.	--	--	--	--	--
a	7-9	FISH Newark Bay, Bayonne Area	0.4	--	--	--	--
a	7-10	FISH Newark Bay, Bayonne Area	0.3	--	--	--	--
a	7-14	FISH Newark Bay, Bayonne Area	0.7	--	--	--	--

Table 1.
Estimated Levels of Polybrominated Biphenyls in Environmental Samples (Continued, page 2 of 14)

Samples Collected By	Sample Code	Sample Type and Location	PBB Concentration (ng/g), parts per billion				
			$C_{12}H_4Br_6$	$C_{12}H_3Br_7$	$C_{12}H_2Br_8$	$C_{12}HBr_9$	$C_{12}Br_{10}$
a	9-1	FISH Raritan R. at South R.	1.5	0.5	--	--	--
a	9-3	FISH Raritan R. at South River	0.3	--	--	--	--
a	9-7	FISH Raritan R. at JCP&L Power Plant	0.4	--	--	--	--
a	9-10	FISH Raritan R. at JCP&L Power Plant	0.7	--	--	--	--
a	9-15	FISH Raritan R. at South River Light	<0.1	--	--	--	--
a	9-16	FISH Raritan R. at South River Light	0.3	--	--	--	--
a	9-18	FISH Raritan R. at South R. Light	2.3	--	--	--	--
a	9-20	FISH Raritan R. at South R. Light	0.4	--	--	--	--
a	9-22	FISH Raritan R. at South R. Canal	2.1	--	--	--	--
a	9-26	FISH Raritan R. at Lawrence Brook	0.2	--	--	--	--
a	9-28	FISH Raritan R. at Lawrence Brook	<0.1	--	--	--	--

Table 1.
Estimated Levels of Polybrominated Biphenyls in Environmental Samples (Continued, page 3 of 14)

Samples Collected By	Sample Code	Sample Type and Location	PBB Concentration (ng/g), parts per billion				
			$C_{12}H_4Br_6$	$C_{12}H_3Br_7$	$C_{12}H_2Br_8$	$C_{12}HBr_9$	$C_{12}Br_{10}$
a	9-30	FISH Raritan R. at Lawrence Brook	--	--	--	--	--
a	9-31	FISH South R. at South R. Marina	0.3	--	--	--	--
a	23-1	FISH Raritan Bay/Sandy Hook Bay	0.4	--	--	--	--
a	23-2	FISH Raritan Bay/Sandy Hook Bay	--	--	--	--	--

Table 1.
Estimated Levels of Polybrominated Biphenyls in Environmental Samples (Continued, page 4 of 14)

Samples Collected By	Sample Code	Sample Type and Location	PBB Concentration (ng/g), parts per billion				
			$C_{12}H_4Br_6$	$C_{12}H_3Br_7$	$C_{12}H_2Br_8$	$C_{12}HBr_9$	$C_{12}Br_{10}$
		SEDIMENT					
b	8503	Cincinnati Chem. Proc., North Ditch	5.9	--	--	--	--
		SEDIMENT					
b	8504	Cincinnati Chem. Proc., West Ditch	550	9.4	--	--	--
		SEDIMENT					
b	8528	Cincinnati Chem. Proc., East Ditch	270	9.2	--	--	--

Table 1.
Estimated Levels of Polybrominated Biphenyls in Environmental Samples (Continued, page 5 of 14)

Samples Collected By	Sample Code	Sample Type and Location	PBB Concentration (ng/g), parts per billion				
			$C_{12}H_4Br_6$	$C_{12}H_3Br_7$	$C_{12}H_2Br_8$	$C_{12}HBr_9$	$C_{12}Br_{10}$
c	7695	SOIL, #1 Borg-Warner, Washington Plant, WV	3,200	190	64	--	--
c	7696	SOIL #2 Borg-Warner, Washington Plant	2,800	430	--	--	--
c	7714	SEDIMENT, #3 Ohio R., Borg-Warner, Wash. Plant	5.6	--	--	--	--
c	7716	SEDIMENT, #5 Ohio R., Borg-Warner, Wash. Plant	10	--	--	--	--
c	7717	SOIL, #6 Borg-Warner, Washington Plant	<10	--	--	--	--
c	7718	SOIL, #7 Borg-Warner, Dupont and L. Washington Rd. Intersection	14	--	--	--	--
c	7719	SOIL, #8 Borg-Warner, Washington Plant	<10	--	--	--	--
c	7722	SOIL Corry Foam Products, PA, S.W.	14	--	--	--	--
c	7723	SOIL Corry Foam Products, PA, N.W.	16	--	--	--	--
c	7726	SOIL Corry Foam Products, PA, N.E.	12	--	--	--	--
c	7727	SOIL Corry Foam Products, PA, S.E.	480	--	--	--	--

Table 1.
Estimated Levels of Polybrominated Biphenyls in Environmental Samples (Continued, page 6 of 14)

Samples Collected By	Sample Code	Sample Type and Location	PBB Concentration (ng/g), parts per billion				
			$C_{12}H_4Br_6$	$C_{12}H_3Br_7$	$C_{12}H_2Br_8$	$C_{12}HBr_9$	$C_{12}Br_{10}$
d	7698	SOIL, #2 Corry Foam Prod., TN	6.4	--	--	--	--
d	7699	SOIL, #3 Corry Foam Prod., TN	3.5	--	--	--	--
d	7706	SOIL, #1A Corry Foam Prod., TN	26	--	--	--	--
d	7709	SOIL, #6 Corry Foam Prod., TN	26	--	--	--	--
d	7710	SOIL, #5 Corry Foam Prod., TN	4.2	--	--	--	--
d	7712	SOIL, #4 Corry Foam Prod., TN	7.2	--	--	--	--

Table 1.
Estimated Levels of Polybrominated Biphenyls in Environmental Samples (Continued, page 7 of 14)

Samples Collected By	Sample Code	Sample Type and Location	PBB Concentration (ng/g), parts per billion				
			$C_{12}H_4Br_6$	$C_{12}H_3Br_7$	$C_{12}H_2Br_8$	$C_{12}HBr_9$	$C_{12}Br_{10}$
e	7651	SOIL, #7 Borg-Warner, California Plant	640	--	--	--	--
e	7652	VEGETATION, #8 Borg-Warner, California Plant	--	--	--	--	--
e	7653	SOIL, #9 Borg-Warner, California Plant	710	47	--	--	--
e	7654	SOIL, #10 Borg-Warner, California Plant	7,300	440	--	--	--
e	7655	VEGETATION, #11 Borg-Warner, California Plant	<150	--	--	--	--
e	7656	SOIL, #12 Borg-Warner, California Plant	23	--	--	--	--
e	7657	SOIL, #13 Borg-Warner, California Plant	2,400	170	--	--	--
e	7658	VEGETATION, #14 Borg-Warner, California Plant	150	--	--	--	--
e	7659	SOIL, #15 Borg-Warner, California Plant	380	--	--	--	--
e	7660	VEGETATION, #16 Borg-Warner, California Plant	100	--	--	--	--
e	7661	SOIL, #17 Borg-Warner, California Plant	39	--	--	--	--

Table 1.
Estimated Levels of Polybrominated Biphenyls in Environmental Samples (Continued, page 8 of 14)

Samples Collected By	Sample Code	Sample Type and Location	PBB Concentration (ng/g), parts per billion				
			$C_{12}H_4Br_6$	$C_{12}H_3Br_7$	$C_{12}H_2Br_8$	$C_{12}HBr_9$	$C_{12}Br_{10}$
e	7662	SLUDGE, #1 Oxnard, STP, California	91	--	--	--	--
e	7663	SLUDGE, #2 Oxnard, STP, California	80	--	--	--	--
e	7728	SOIL, #1 Borg-Warner, California Plant	6,000	150	--	--	--
e	7729	SOIL, #2 Borg-Warner, California Plant	50,000	18,000	--	--	--
e	7730	SOIL, #3 Borg-Warner, California Plant	9,200	870	--	--	--
e	7731	VEGETATION, #4 Borg-Warner, California Plant	50	<40	--	--	--
e	7732	VEGETATION, #5 Borg-Warner, California Plant	93	--	--	--	--
e	7733	VEGETATION, #6 Borg-Warner, California Plant	64	--	--	--	--

Table 1.
Estimated Levels of Polybrominated Biphenyls in Environmental Samples (Continued, page 10 of 14)

Samples Collected By	Sample Code	Sample Type and Location	PBB Concentration (ng/g), parts per billion				
			$C_{12}H_4Br_6$	$C_{12}H_3Br_7$	$C_{12}H_2Br_8$	$C_{12}HBr_9$	$C_{12}Br_{10}$
g	4601	SOIL, #1 Burkart Randall, Cairo, IL 78-CM09S01	40	--	--	--	--
g	4602	SOIL, #2 Burkart Randall, 78-CM09S02	290	--	--	--	--
g	4603	SOIL, #3 Burkart Randall, 78-CM09S03	8	--	--	--	--
g	4604	SOIL, #4 Burkart Randall, 78-CM09S04	37	--	--	--	--
g	4605	SOIL, #5 Burkart Randall, 78-CM09S05	350	--	--	--	--
g	4606	SOIL, #6 Burkart Randall, 78-CM09S06	240	--	--	--	--
g	4607	SOIL, #7 Burkart Randall, 78-CM09S07	150	--	--	--	--
g	4608	SOIL, #8 Burkart Randall, 78-CM09S08	310	--	--	--	--
g	4609	SOIL, #9 Burkart Randall, 78-CM09S09	8	--	--	--	--
g	4610	SOIL, #10 Burkart Randall, 78-CM09S10	8	--	--	--	--

Table 1.
Estimated Levels of Polybrominated Biphenyls in Environmental Samples (Continued, page 11 of 14)

Samples Collected By	Sample Code	Sample Type and Location	PBB Concentration (ng/g), parts per billion				
			$C_{12}H_4Br_6$	$C_{12}H_3Br_7$	$C_{12}H_2Br_8$	$C_{12}HBr_9$	$C_{12}Br_{10}$
g	4611	SOIL, #12 Burkart Randall, 78-CM09S12	3	--	--	--	--
g	4612	SOIL, #13 Burkart Randall, 78-CM09S13	2	--	--	--	--
g	4613	SOIL, #1 Burkart Randall, 78-CM09S14	3	--	--	--	--
g	4614	SOIL, #15 Burkart Randall, 78-CM09S15	--	--	--	--	--
g	4615	SOIL, #16 Burkart Randall, 78-CM09S16	1	--	--	--	--

Table 1.
Estimated Levels of Polybrominated Biphenyls in Environmental Samples (Continued, page 12 of 14)

Samples Collected By	Sample Code	Sample Type and Location	PBB Concentration (ng/g), parts per billion				
			$C_{12}H_4Br_6$	$C_{12}H_3Br_7$	$C_{12}H_2Br_8$	$C_{12}HBr_9$	$C_{12}Br_{10}$
h	4616	SOIL, #1/7 Cincinnati Chemical Processing Co., Batavia, OH	1020	--	--	--	--
h	4617	SOIL, #2/7 Cincinnati Chemical Processing Co., Batavia, OH	84	--	--	--	--
h	4618	SOIL, #3/7 Cincinnati Chemical Processing Co., Batavia, OH	340	--	--	--	--
h	4619	SOIL, #4/7 Cincinnati Chemical Processing Co., Batavia, OH	390	--	--	--	--
h	4620	SOIL, #5/7 Cincinnati Chemical Processing Co., Batavia, OH	2	--	--	--	--
h	4621	SOIL, #6/7 Cincinnati Chemical Processing Co., Batavia, OH	17	--	--	--	--
h	4622	SOIL, #7/7 Cincinnati Chemical Processing Co., Batavia, OH	5	--	--	--	--

Table 1.
Estimated Levels of Polybrominated Biphenyls in Environmental Samples (Continued, page 13 of 14)

Samples Collected By	Sample Code	Sample Type and Location	PBB Concentration (ng/g), parts per billion				
			$C_{12}H_4Br_6$	$C_{12}H_3Br_7$	$C_{12}H_2Br_8$	$C_{12}HBr_9$	$C_{12}Br_{10}$
i	4623	SOIL, #1 Borg-Warner, Ottawa, IL 78-CM10S01	12	--	--	--	--
i	4624	SOIL, #2 Borg-Warner, Ottawa, IL 78-CM10S02	3	--	--	--	--
i	4625	SOIL, #7 Borg-Warner, Ottawa, IL 78-CM10S07	51	--	--	--	--
i	4626	SOIL, #8 Borg-Warner, Ottawa, IL 78-CM10S08	61	--	--	--	--
i	4627	SOIL, #9 Borg-Warner, Ottawa, IL 78-CM10S09	68	--	--	--	--
i	4628	SOIL, #12 Borg-Warner, Ottawa, IL 78-CM10S12	9	--	--	--	--
i	4629	SOIL, #13 Borg-Warner, Ottawa, IL 78-CM10S13	4	--	--	--	--
i	4630	SOIL, #14 Borg-Warner, Ottawa, IL 78-CM10S14	4	--	--	--	--

Table 1.
Estimated Levels of Polybrominated Biphenyls in Environmental Samples (Continued, page 14 of 14)

Samples Collected By	Sample Code	Sample Type and Location	PBB Concentration (ng/g), parts per billion				
			$C_{12}H_4Br_6$	$C_{12}H_3Br_7$	$C_{12}H_2Br_8$	$C_{12}HBr_9$	$C_{12}Br_{10}$
j	8669	CARPET AID Consulting Engineers	--	--	--	--	--

Table 2. Frequency and Range of Detection in Ninety-Eight Samples Analyzed

Species	Frequency	Range (ppb)	Material
Hexabromobiphenyl	84%	0.2-50,000	tissue, soil, sediment
Heptabromobiphenyl	12%	0.5-18,000	tissue, soil, sediment
Octabromobiphenyl	2%	1.8-64	tissue, soil
Nonabromobiphenyl	0%	--	--
Decabromobiphenyl	0%	--	--

5.0 REFERENCES

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APPENDIX A

SAMPLES RECEIVED

Table A-1. Sample Description

Set a

New Jersey Division of Fish and Game
Nacote Creek Research Station
Star Route
Absecon, New Jersey 08201

Sample No.	Sampling Location	Sample Description	Analyzed?
5-1	Hackensack River at Sawmill Creek	Blue Crab--Composite	Broken
5-2	Hackensack River at Sawmill Creek	Spot--Composite	Yes
5-3	Hackensack River at Rt. 1 & 9 Bridge	Sediment--1/3 pint	No
5-13	Hackensack River at Sawmill Creek	American Eel--Single	Yes
5-14	Hackensack River at Sawmill Creek	American Eel--Single	No
5-15	Hackensack River at Sawmill Creek	American Eel--Single	No
5-16	Hackensack River at Sawmill Creek	American Eel--Single	No
5-17	Hackensack River at Moonachie Creek	Br. Bullhead Catfish--Single	Yes
6-1	Passaic River at Rt. 1 & 9 Bridge	Sediment--1/3 Pint	No
7-2	Newark Bay, Bayonne Area	Menhaden--Composite	Yes
7-3	Newark Bay, Bayonne Area	Spot--Composite	No
7-4	Newark Bay, Bayonne Area	Blue Crab--Composite	Yes
7-5	Newark Bay, Bayonne Area	Killifish--Composite	No
7-6	Newark Bay, Bayonne Area	Sediment--1/3 Pint	Yes
7-7	Arthur Kill/ Howland Hook Staten Island	Sediment--1/3 Pint	Yes

Table A-1. Sample Description

Set a (continued)

Sample No.	Sampling Location	Sample Description	Analyzed?
7-8	Kill Van Kull, Bement Ave., Staten Island	Sediment--1/3 Pint	Yes
7-9	Newark Bay, Bayonne Area	Striped Bass--Single	Yes
7-10	Newark Bay, Bayonne Area	Striped Bass--Single	Yes
7-11	Newark Bay, Bayonne Area	Menhaden--Single	No
7-14	Newark Bay, Bayonne Area	Killifish--Composite	Yes
9-1	Raritan River at South River	Bluefish--Composite	Yes
9-3	Raritan River at South River	Blue Crab--Composite	Yes
9-5	Raritan River at South River	Spot--Composite	No
9-7	Raritan River at JCP&L Power Plant	Striped Bass--Single	Yes
9-9	Raritan River at JCP&L Power Plant	American Eel--Single	No
9-10	Raritan River at JCP&L Power Plant	Striped Bass--Single	Yes
9-11	Raritan River at JCP&L Power Plant	American Eel--Single	No
9-12	Raritan River at JCP&L Power Plant	White Perch--Single	No
9-13	Raritan River at JCP&L Power Plant	White Perch--Single	No
9-15	Raritan River at South River Light	Striped Bass--Single	Yes
9-16	Raritan River at South River Light	Striped Bass--Single	Yes
9-17	Raritan River at South River Light	Striped Bass--Single	No
9-18	Raritan River at South River Light	Striped Bass--Single	Yes
9-19	Raritan River at South River Light	Striped Bass--Single	No

Table A-1. Sample Description

Set a (continued)

Sample No.	Sampling Location	Sample Description	Analyzed?
9-20	Raritan River at South River Light	White Perch--Single	Yes
9-22	Raritan River at South River Canal	White Perch--Single	Yes
9-24	Raritan River at South River Light	American Eel--Single	No
9-26	Raritan River at Lawrence Brook	White Perch--Single	Yes
9-28	Raritan River at Lawrence Brook	Carp--Single	Yes
9-29	Raritan River at Lawrence Brook	Carp--Single	No
9-30	Raritan River at Lawrence Brook	Carp--Single	Yes
9-31	South River at South River Marina	White Catfish--Single	Yes
23-1	Raritan Bay/Sandy Hook Bay	Winter Flounder--Single	Yes
23-2	Raritan Bay/Sandy Hook Bay	Winter Flounder--Single	Yes
23-4	Raritan Bay/Sandy Hook Bay	Winter Flounder--Single	No
23-5	Raritan Bay/Sandy Hook Bay	Winter Flounder--Single	No

Table A-1. Sample Description

Set b

Southwest District
Ohio EPA

Sample No.	Sampling Location	Sample Description	Analyzed?
8503	Cincinnati Chemical Processing, North Ditch	Sediment	Yes
8504	Cincinnati Chemical Processing, West Ditch	Sediment	Yes
8528	Cincinnati Chemical Processing, East Ditch	Sediment	Yes

Table A-1. Sample Description

Set c

U.S. Environmental Protection Agency
Region III
303 Methodist Building
Wheeling, West Virginia 26003

Sample No.	Sampling Location	Sample Description	Analyzed?
7695	Borg-Warner, Washington Plant	Soil, Sample #1	Yes
7696	Borg-Warner, Washington Plant	Soil, Sample #2	Yes
7714	Borg-Warner, Ohio River below Borg-Warner Effluent	Sediment, Sample #3	Yes
7715	Borg-Warner, Washington Plant	Water, Sample #4	No
7716	Ohio River at Old Lock and Dike, just below Borg-Warner Washington Plant	Sediment, Sample #5	Yes
7717	Along Road to Old Lock and Dam, just downstream Borg-Warner Washington Plant	Soil, Sample #6	Yes
7718	DuPont and Lake Washington Roads Intersection	Soil, Sample #7	Yes
7719	DuPont Road at Entrance to Landfill Area of DuPont	Soil, Sample #8	Yes
7720	1/2 mile S.E. of Corry Foam Products, Corry, PA	Soil	No
7721	1/2 mile N.W. of Corry Foam Products, Corry, PA	Soil	No
7722	Corry Foam Products, Corry, PA, S.W.	Soil	Yes
7723	Corry Foam Products, Corry, PA, N.W.	Soil	Yes

Table A-1. Sample Description

Set c (continued)

Sample No.	Sampling Location	Sample Description	Analyzed?
7724	1/2 mile N.E. of Corry Foam Products, Corry, PA	Soil	No
7725	1/2 mile S.W. of Corry Foam Products, Corry, PA	Soil	No
7726	Corry Foam Products, Corry, PA, N.E.	Soil	Yes
7727	Corry Foam Products, Corry, PA, S.E.	Soil	Yes

Table A-1. Sample Description

Set d

U.S. Environmental Protection Agency
Pesticides Branch
Nashville, Tennessee

Sample No.	Sampling Location	Sample Description	Analyzed?
7697	Point #9	Soil	No
7698	Point #2	Soil	Yes
7699	Point #3	Soil	Yes
7700	Point #8, Sub 8A	Soil	No
7701	Point #1, Sub C., Corry Foam Products Milan, TN	Live Frog	No
7702	Point #8	2 Live Chub	No
7703	Point #1, Sub B	Soil	No
7706	Point #1, A	Soil	Yes
7707	Point #1, Sub	Soil	No
7708	Point #1, Sub E	Piece of Foam	No
7709	Point #6	Soil	Yes
7710	Point #5	Soil	Yes
7711	Point #8	Wood Soil	No
7712	Point #4	Soil	Yes
7713	Point #8, Sub 8	Soil	No

Table A-1. Sample Description

Set e

U.S. Environmental Protection Agency
Region IX
Alameda, California

Sample No.	Sampling Location	Sample Description	Analyzed?
7651	Borg-Warner, California Plant	Soil, Sample #7	Yes
7652	Borg-Warner, California Plant	Vegetation, Sample #8	Yes
7653	Borg-Warner, California Plant	Soil, Sample #9	Yes
7654	Borg-Warner, California Plant	Soil, Sample #10	No
7655	Borg-Warner, California Plant	Vegetation, Sample #11	Yes
7656	Borg-Warner, California Plant	Soil, Sample #12	Yes
7657	Borg-Warner, California Plant	Soil, Sample #13	Yes
7658	Borg-Warner, California Plant	Soil, Sample #14	Yes
7659	Borg-Warner, California Plant	Soil, Sample #15	Yes
7660	Borg-Warner, California Plant	Vegetation, Sample #16	Yes
7661	Borg-Warner, California Plant	Soil, Sample #17	Yes
7662	Oxnard STP	Sludge #1	Yes
7663	Oxnard STP	Sludge #2	Yes
7728	Borg-Warner, California Plant	Soil, Sample #1	Yes
7729	Borg-Warner, California Plant	Soil, Sample #2	Yes
7730	Borg-Warner, California Plant	Soil, Sample #3	Yes
7731	Borg-Warner, California Plant	Oleander Leaves, Veg., Sample #4	Yes
7732	Borg-Warner, California Plant	Oleander Leaves, Veg., Sample #5	Yes
7733	Borg-Warner, California Plant	Oleander Leaves, Veg., Sample #6	Yes

Table A-1. Sample Description

Set f

New York State Department of Environmental Conservation
Buffalo, New York

Sample No.	Sampling Location	Sample Description	Analyzed?
#1	Chemtrol Pollution Services	W. Faculatative Pond Liquid	No
#2	Chemtrol Pollution Services	E. Faculatative Pond Liquid	No
#3	Chemtrol Pollution Services	New Faculatative Pond Liquid	No
#4	Chemtrol Pollution Services	Landfill #5 Liquid N. Standpipe	No
#5	Chemtrol Pollution Services	Landfill #5 Liquid S. Standpipe	No
#6	Chemtrol Pollution Services	W. Faculatative Pond Sediment	No
#7	Chemtrol Pollution Services	E. Faculatative Pond Sediment	Yes
#8	Chemtrol Pollution Services	New Faculatative Pond Sediment	Yes

Table A-1. Sample Description

Set g

U.S. Environmental Protection Agency
Region V, Central District Office
Sampling and Analysis Division
E.N. Mortenson, Chemical Engineers

Sample No.	Sampling Location	Sample Description	Analyzed?
4601	1. SW Corner of Intersection 38th St. and Commercial, Cairo, IL	Soil 78-CM09S01	Yes
4602	2. Next to Bunge property fence at 36th St. and Commercial, Cairo, IL	Soil 78-CM09S02	Yes
4603	3. SE Corner of Intersection 34th and Commercial, Cairo, IL	Soil 78-CM09S03	Yes
4604	4. SW Corner of Intersection 34th and Poplar, Cairo, IL	Soil 78-CM09S04	Yes
4605	5. SW Corner of Intersection 34th and and Sycamore, Cairo, IL	Soil 78-CM09S05	Yes
4606	6. NW Corner of Intersection 36th and Sycamore, Cairo, IL	Soil 78-CM09S06	Yes
4607	7. SW Corner of Intersection 38th and Sycamore, Cairo, IL	Soil 78-CM09S07	Yes
4608	8 NW Corner of Intersection 38th and Poplar, Cairo, IL	Soil 78-CM09S08	Yes
4609	9. 500 feet NE of Burkart plant property, Cairo, IL	Soil 78-CM09S09	Yes

Table A-1. Sample Description

Set g (continued)

Sample No.	Sampling Location	Sample Description	Analyzed?
4610	10. 500 feet straight east of plant property, Cairo, IL	Soil 78-CM09S10	Yes
--	11. Sample not taken--location not accessible	--	No
4611	12. NW Corner of Intersection 34th and Poplar, Cairo, IL	Soil 78-SM09S12	Yes
4612	13. SW Corner of Intersection 33rd and Elm, Cairo, IL	Soil 78-SM09S13	Yes
4613	14. NW Corner of Intersection 36th and Elm, Cairo, IL	Soil 78-SM09S14	Yes
4614	15. NW Corner of Intersection 39th and Elm, Cairo, IL	Soil 78-SM09S15	Yes
4615	16. NW Corner of Intersection 40th and Poplar, Cairo, IL	Soil 78-SM09S16	Yes

Table A-1. Sample Description

Set h

Ohio EPA
Southwest District Office
7 East Fourth Street
Dayton, Ohio 45402
Ken M. Harsh

Sample No.	Sampling Location	Sample Description	Analyzed?
4616	NE of Cincinnati Chemical Processing Co., Batavia, OH	Soil, Sample #1/7	Yes
4617	SE of Cincinnati Chemical Processing Co., Batavia, OH	Soil, Sample #2/7	Yes
4618	SW of Cincinnati Chemical Processing Co., Batavia, OH	Soil, Sample #3/7	Yes
4619	NW of Cincinnati Chemical Processing Co., Batavia, OH	Soil, Sample #4/7	Yes
4620	West Bank Little Miami River near new SR-32 bridge	Soil, Sample #5/7	Yes
4621	Ditch along west bank Little Miami River near new SR-32 bridge	Soil, Sample #6/7	Yes
4622	West bank Little Miami River near old SR-32 bridge	Soil, Sample #7/7	Yes

Table A-1. Sample Description

Set i

U.S. Environmental Protection Agency
Region V, Central District Office
Sampling and Analysis Division
E.N. Mortenson, Chemical Engineer

Sample No.	Sampling Location	Sample Description	Analyzed?
4623	1. West end of north side of Bells' Island, south of B/W plant. 6-13-78, Ottawa, IL	78-CM10S01, Soil	Yes
4624	2. Soil with turf from Bells' Island, south of B/W plant and straight north of Lock-master control house, Ottawa, IL	78-CM10S02, Soil	Yes
--	3. Weitz's cornfield, adjacent east property of B/W plant, Ottawa, IL	78-CM10S03, Soil	Broken on Receipt
--	4. Weitz's cornfield, 500 feet east of B/W property, Ottawa, IL	78-CM10S04, Soil	Broken on Receipt
--	5. Weitz's cornfield, adjacent to NE corner of B/W property, Ottawa, IL	78-CM10S05, Soil	Broken on Receipt
--	6. Weitz's cornfield, 500 feet east of NE corner of B/W property, Ottawa, IL	78-CM10S06, Soil	Broken on Receipt
4625	7. Across Commercial Road at NE corner of B/W property, Ottawa, IL	78-CM10S07, Soil	Yes
4626	8. Approximately 700 feet north of NE corner of B/W property, Ottawa, IL	78-CM10S08, Soil	Yes

Set i

Sample No.	Sampling Location	Sample Description	Analyzed?
4627	9. Across Commercial Road at center of north side of B/W property, Ottawa, IL	78-CM10S09, Soil	Yes
--	10. Approximately 500 feet north of center of north side of B/W property, Ottawa, IL	78-CM10S10, Soil	Broken on Receipt
--	11. 50 feet west of NW corner of B/W property, in McLean's cornfield, Ottawa, IL (Note: duplicate of this sample retained at CDO, Region V)	78-CM10S11, Soil	Broken on Receipt
4628	12. 500 feet west of NW corner of B/W property in McLean's cornfield, Ottawa, IL	78-CM10S12, Soil	Yes
4629	13. 50 feet west of center of west property line of B/W plant, Ottawa, IL	78-CM10S13, Soil	Yes
4630	14. 500 feet west of center of west property line of B/W plant, Ottawa, IL	78-CM10S14, Soil	Yes

Set j

U.S. Environmental Protection Agency
Office of Toxic Substances
Washington, D.C. 20460

Sample No.	Sampling Location	Sample Description	Analyzed?
8669	AID Consulting Engineers	Carpet Sample	Yes

APPENDIX B

GC/MS ANALYSIS RESULTS FOR ENVIRONMENTAL SAMPLE EXTRACTS

Table B-1. ESTIMATED LEVELS OF POLYBROMINATED BIPHENYLS IN ENVIRONMENTAL SAMPLES[a,n]

Sample Code[b]	Type	$C_{12}H_4Br_6$[c]	$C_{12}H_3Br_7$	$C_{12}H_2Br_8$	$C_{12}HBr_9$	$C_{12}Br_{10}$
7-14	Killifish, Composite	2.9 ng/ml[d]	-[e]	-	-	-
5-17	Br. Bullhead Catfish	2.0 ng/ml	-	-	-	-
7-4	Blue Claw Crab, Composite	T[f]	-	-	-	-
7-10	Striped Bass	1.1 ng/ml[g]	-	-	-	-
5-13	American Eel	2.9 ng/ml[d]	-	-	-	-
9-30	Carp	-	-	-	-	-
7-6[m]	Sediment	-	-	-	-	-
7-8	Sediment	-	-	-	-	-
9-3	Blue Claw Crab, Composite	0.9 ng/ml[d]	-	-	-	-
8	Sediment	5.2 ng/ml[d]	-	-	-	-
9-7	Striped Bass	1.3 ng/ml[d]	-	-	-	-
9-16	Striped Bass	1.0 ng/ml	-	-	-	-
9-15	Striped Bass	T	-	-	-	-
7	Sediment	3.5 ng/ml	-	-	-	-
9-22	White Perch	9.0 ng/ml[d]	-	-	-	-
9-18	Striped Bass	8.1 ng/ml	-	-	-	-
7-9	Striped Bass	3.0 ng/ml	-	-	-	-
23-2	Winter Flounder	-	-	-	-	-
9-20	White Perch	1.3 ng/ml	-	-	-	-
9-1	Bluefish, Composite	5.6 ng/ml[d]	1.7 ng/ml[d]	-	-	-
7-2	Menhaden, Composite	5.1 ng/ml	2.6 ng/ml	2.9 ng/ml	-	-
23-1	Winter Flounder	1.5 ng/ml	-	-	-	-

Table B-1. (continued)

Sample Code[b]	Type	$C_{12}H_4Br_6$[c]	$C_{12}H_3Br_7$	$C_{12}H_2Br_8$	$C_{12}HBr_9$	$C_{12}Br_{10}$
7-7[m]	Sediment	-	-	-	-	-
9-31	White Catfish	0.6 ng/ml	-	-	-	-
9-28	Carp	T	-	-	-	-
9-26	White Perch	0.7 ng/ml	-	-	-	-
9-10	Striped Bass	2.4 ng/ml[d]	-	-	-	-
7657[h]	Soil	111,000 ng/ml[g]	7,600 ng/ml[g]	-	-	-
7727[h]	Soil	12,700 ng/ml[g]	-	-	-	-
7723	Soil	410 ng/ml[d]	-	-	-	-
7651[h]	Soil	31,000 ng/ml[i]	-	-	-	-
7659[h]	Soil	18,500 ng/ml[d]	-	-	-	-
8504[h]	Sediment	26,800 ng/ml[g]	460 ng/ml[g]	-	-	-
7728[h]	Soil	253,000 ng/ml[g]	6,400 ng/ml[g]	-	-	-
7695[h]	Soil	146,000 ng/ml[d]	8,700 ng/ml[d]	2,900 ng/ml[d]	-	-
7661[h]	Soil	1,700 ng/ml[d]	-	-	-	-
7716	Sediment	490 ng/ml[d]	-	-	-	-
7722	Soil	480 ng/ml[d]	-	-	-	-
7706	Soil	1,100 ng/ml[g]	-	-	-	-
7709	Soil	1,200 ng/ml[d]	-	-	-	-
7710	Soil	190 ng/ml[g]	-	-	-	-
7712	Soil	320 ng/ml[j]	-	-	-	-
7714	Sediment	280 ng/ml[g]	-	-	-	-
7726	Soil	450 ng/ml[g]	-	-	-	-

Table B-1. (continued)

Sample Code[b]	Type	$C_{12}H_4Br_6$[c]	$C_{12}H_3Br_7$	$C_{12}H_2Br_8$	$C_{12}HBr_9$	$C_{12}Br_{10}$
7732	Vegetation	880 ng/ml[k]	-	-	-	-
7698	Soil	290 ng/ml[g]	-	-	-	-
7699	Soil	160 ng/ml[i]	-	-	-	-
8503	Sediment	290 ng/ml[l]	-	-	-	-
7662[m]	Sludge	950 ng/ml[d]	-	-	-	-
5-2	Spot, Composite	7.1 ng/m	-	-	-	-
7730[h]	Soil	429,000 ng/ml[g]	40,300 ng/ml[g]	-	-	-
7654[h]	Vegetation	330,000 ng/ml[d]	20,000 ng/ml[d]	-	-	-
7663[m]	Sludge	590 ng/ml[d]	-	-	-	-
8528[m]	Sediment	11,500 ng/ml	400 ng/ml	-	-	-

[a]Samples as received from Environmental Science and Engineering, Inc., were taken to dryness under a slow stream of dry nitrogen. Samples were then concentrated or diluted to the appropriate volume with dry toluene and the appropriate standard (octachloronaphthalene or decachlorobiphenyl) was added at a level of 2 ng/µl. The samples were analyzed by gas chromatograph/mass spectrometer/computer using a Finnigan 3300 gc/ms equipped with a PDP-12 computer. The chromatographic column used was a 45 cm x 0.2 cm i.d. glass column packed with 2% OV-101 coated on Gas-Chrom Q with a helium flow rate of 20 cc/min, and an initial temperature of 220°C programmed to 280°-300°C at 4°/min. The temperature of the glass jet separator was 250°C, the injector 280°C. The polybrominated biphenyls were analyzed by selected ion monitoring techniques (see Appendix B for ions monitored and retention times observed).

[b]Sample code as received from Environmental Science and Engineering, Inc.

[c]Three positional isomers used as standards were not resolved under the chromatographic conditions used. Ratios between members of the isotope clusters were used for verification of compound identity (as well as retention time); full scan verification was obtained for some samples (see Appendices C and D).

[d]Average of two determinations.

Table B-1. (continued)

[e]Undetectable.

[f]Less than 0.5 ng/µl, but distinctly present.

[g]Average of three determinations.

[h]Presence of polybrominated biphenyls was confirmed by full scan (see Appendix D).

[i]Average of four determinations.

[j]Average of seven determinations, over a two-week interval.

[k]Average of six determinations.

[l]Average of five determinations.

[m]Decachlorobiphenyl (ion 502) was used as a standard instead of octachloronapthalene (ion 404) because of interferences in the 400-490 channels.

[n]See Appendix E for some representative Selected Ion Monitoring Traces.

Table B-2. ESTIMATED LEVELS OF POLYBROMINATED BIPHENYLS IN ENVIRONMENTAL SAMPLES[a]

Sample Code[b]	Type	$C_{12}H_4Br_6$[c]	$C_{12}H_3Br_7$	$C_{12}H_2Br_8$	$C_{12}HBr_9$	$C_{12}Br_{10}$
77-18	Soil	610 ng/mL[d]	-[e]	-	-	-
77-17	Soil	T[f]	-	-	-	-
77-19	Soil	T[f]	-	-	-	-
76-86	Soil	1,100 ng/mL[d]	-	-	-	-
76-60	Vegetation	900 ng/mL[d]	-	-	-	-
77-31	Oleander Leaves	650 ng/mL[d]	T	-	-	-
76-55	Vegetation	T[f]	-	-	-	-
76-52	Vegetation	-	-	-	-	-
76-58	Soil	1,300 ng/mL[d]	-	-	-	-
77-33	Oleander Leaves	500 ng/mL[g]	-	-	-	-
77-29[h]	Soil	2,291,000 ng/mL[g]	830,000 ng/mL	-	-	-
76-53[h,i]	Soil	35,000 ng/mL[d]	2,300 ng/mL	-	-	-
76-96[h,i]	Soil	132,000 ng/mL[d]	20,000 ng/mL[g]	-	-	-
86-69	Carpet	-	-	-	-	-

[a]Samples as received from Environmental Science and Engineering, Inc., were taken to dryness under a slow stream of dry nitrogen. Samples were then concentrated or diluted to the appropriate volume with dry toluene and the appropriate standard (octachloronaphthalene or decachlorobiphenyl) was added at a level of 2 ng/µl. The samples were analyzed by gas chromatograph/mass spectrometer/computer using a Finnigan 3300 gc/ms equipped with a PDP-12 computer. The chromatographic column used was a 45 cm x 0.2 cm i.d. glass column packed with 2% OV-101 coated on Gas-Chrom Q with a helium flow rate of 20 cc/min, and an initial temperature of 220°C programmed to 280°-300°C at 4°/min. The temperature of the glass jet separator was 250°C, the injector 280°C. The polybrominated biphenyls were analyzed by selected ion monitoring techniques (see Appendix B for ions monitored and retention times observed).

[b]Sample code as received from Environmental Science and Engineering, Inc.

Table B-2. (continued)

[c]Three positional isomers used as standards were not resolved under the chromatographic conditions used. Ratios between members of the isotope clusters were used for verification of compound identity (as well as retention time); full scan verification was obtained for some samples (see Appendices C and D).

[d]Average of two determinations.

[e]Undetectable.

[f]Less than 0.5 ng/μL, but distinctly present.

[g]Average of three determinations.

[h]Presence of polybrominated biphenyls was confirmed by full scan (see Appendix D).

[i]Decachlorobiphenyl (ion 502) was used as a standard instead of octachloronaphthalene (ion 404) because of interferences in the lower mass channels.

Table B-3. Estimated Levels of Polybrominated Biphenyls in Environmental Samples[a,h]

Sample Code[b]	$C_{12}H_4Br_6$[c]	$C_{12}H_3Br_7$	$C_{12}H_2Br_8$	$C_{12}HBr_9$	$C_{12}Br_{10}$
Solvent Blank #1[d]	--[f]	--	--	--	--
Soil Blank (11/8)[d]	--	--	--	--	--
Soil Blank (11/17)[3]	--	--	--	--	--
Soil Spike #1[d]	8,000 ng/ml	--	--	--	--
Soil Spike #2[d]	7,000 ng/ml	--	--	--	--
46-01[d]	1,700 ng/ml	--	--	--	--
46-02[e]	13,200 ng/ml	trace[g]	--	--	--
46-03[d]	400 ng/ml	--	--	--	--
46-04[d]	1,600 ng/ml	--	--	--	--
46-05[e]	11,100 ng/ml	trace	--	--	--
46-06[e]	8,300 ng/ml	trace	--	--	--
46-07[e]	6,300 ng/ml	trace	--	--	--
46-08[e]	11,300 ng/ml	trace	--	--	--
46-09[d]	300 ng/ml	trace	--	--	--
46-10[d]	330 ng/ml	trace	--	--	--
46-11[d]	100 ng/ml	--	--	--	--
46-12[d]	70 ng/ml	trace	--	--	--
46-13[d]	100 ng/ml	trace	--	--	--
46-14[d]	--	--	trace	--	--
46-15[d]	50 ng/ml	--	--	--	--
46-16[d]	43,100 ng/ml	trace	--	--	--
46-17[d]	3,700 ng/ml	trace	--	--	--
46-18[e]	14,900 ng/ml	trace	--	--	--
46-19[e]	16,500 ng/ml	trace	--	--	--
46-20[d]	60 ng/ml	--	--	--	--
46-21[d]	700 ng/ml	trace	--	--	--
46-22[d]	200 ng/ml	--	--	--	--
46-23[d]	570 ng/ml	--	--	--	--
46-24[d]	130 ng/ml	trace	--	--	--
46-25[d]	2,500 ng/ml	trace	--	--	--
46-26[d]	2,500 ng/ml	--	--	--	--
46-27[e]	3,400 ng/ml	trace	--	--	--

Table B-3. (continued)

Sample Code[b]	$C_{12}H_4Br_6$[c]	$C_{12}H_3Br_7$	$C_{12}H_2Br_8$	$C_{12}HBr_9$	$C_{12}Br_{10}$
46-28[d]	440 ng/ml	trace	--	--	--
46-29[d]	180 ng/ml	--	--	--	--
46-30[d]	170 ng/ml	--	--	--	--

[a]Samples as received from Environmental Science and Engineering, Inc. were taken to dryness under a slow stream of dry nitrogen. Samples were then taken up in an appropriate volume of dry toluene and the appropriate standards (octachloronaphthalene and decachlorobiphenyl) added at a level of approximately 2 ng/μl. The samples were analyzed by gas chromatograph/mass spectrometer/computer utilizing either a Finnigan 3300 GC/MS equipped with a PDP-12 computer (chromatographic column, 45 cm x 0.2 cm i.d., glass, 2% OV-101 on Gas-Chrom Q; helium flow, 20 cc/min; initial temperature, 220°C programmed to 280°-300°C at 4°/min; separator temperature, 250°C; injector temperature, 280°C) or an LKB-2091-050 EI/CI (chromatographic column, 45 cm x 0.2 cm i.d., glass, 2% OV-101 on Gas-Chrom Q; helium flow, 18 cc/min; initial temperature, 220°C for one minute, then programmed to 290°C at 8°/min; separator temperature, 275°C; injector temperature, 280°C; ion source temperature, 250°C). The polybrominated biphenyls were analyzed by selected ion monitoring techniques (see Appendix B for ions monitored and retention times observed).

[b]Sample code as received from Environmental Science and Engineering, Inc.

[c]Positional isomers used as standards were not resolved under the chromatographic conditions used. Ratios between members of the isotope clusters were used for verification of compound identity (as well as retention time); full scan verification was obtained for some samples (see Appendices C and D).

[d]Average of two determinations, one instrument.

[e]Average of three determinations, both instruments.

[f]Undetectable.

[g]Present as a distinct peak but not reproducibly quantifiable.

[h]See Appendix E for some representative Selected Ion Monitoring Traces.

APPENDIX C

RETENTION TIMES AND IONS MONITORED FOR ANALYSIS OF POLYBROMINATED BIPHENYLS

APPENDIX C

Table C -1. RETENTION TIMES AND IONS MONITORED FOR ANALYSIS OF POLYBROMINATED BIPHENYLS*

Compound	Retention Time	Ion(s) Monitored
Octachloronaphthalene (Standard)	∿ 1.3 min	404
Decachlorobiphenyl[a] (Standard)	∿ 1.5 min	502
Hexabromobiphenyls,[b]		
2,2',4,4',6,6'-	∿ 1.7 min	626, 628, 630, 632
2,4,5,2',4',5'-	∿ 1.7 min	626, 628, 630, 632
3,3',4,4',5,5'-	∿ 1.7 min	626, 628, 630, 632
Heptabromobiphenyl[c]	∿ 2.5 min	706
Octabromobiphenyl, 2,2',3,3',5,5',6,6'-	∿ 7.8 min	784
Nonabromobiphenyl[c]	∿ 8.2 min	862
Decabromobiphenyl	∿10.2 min	942

[a]Decachlorobiphenyl was used as a standard for those samples in which the ion characteristic of octachloronaphthalene (404) could not be used for quantitation.

[b]Under the chromatographic conditions used, the hexabromobiphenyl isomers used as standards were not resolved.

[c]Heptabromobiphenyl was present as an impurity in the octabromobiphenyl standard; the positional isomer was not identified. Nonabromobiphenyl was also observed.

*Applies to samples reported in Tables B -1 and B -2.

Table C-2. Retention Times and Ions Monitored for Analysis of Polybrominated Biphenyls*

Compound	Retention Time	Ion(s) Monitored
Octachloronaphthalene (Standard)	∿2.5 min	404
Decachlorobiphenyl[a] (Standard)	∿2.8 min	502
Hexabromobiphenyls[b]		
2,2',4,4',6,6'-	∿3.0 min	626, 628, 630
3,3',4,4',5,5'-	∿3.0 min	626, 628, 630
Heptabromobiphenyl[c]	∿5.4 min	706, 708
Octabromobiphenyl[d]	∿7.2 min	784, 786
Nonabromobiphenyl[e]	∿9.5 min	864, 866
Decabromobiphenyl[f]	∿10.8 min	942, 944

[a]Decachlorobiphenyl was used as an auxiliary standard to provide a reference in those cases in which the ion characteristic of octachloronaphthalene (404) could not be used for quantitation because of interferences in that channel.

[b]Under the chromatographic conditions used, the hexabromobiphenyl isomers used as standards were not resolved.

[c]Heptabromobiphenyl (isomer unidentified) was present as an impurity in the standards labeled "octabromobiphenyl" and "decabromobiphenyl."

[d]Octabromobiphenyl (any isomer in a relatively pure form) can no longer be purchased for use as a standard.

[e]Nonabromobiphenyl (isomer unidentified) was present as an impurity in the standards labeled "octabromobiphenyl" and "decabromobiphenyl."

[f]Decabromobiphenyl in a pure form can no longer be purchased for use as a standard.

*Applies to samples reported in Table B-3.

APPENDIX D

RATIOS OF IONS USED TO CONFIRM THE PRESENCE OF POLYBROMINATED BIPHENYLS

APPENDIX D

Table D-1. RATIOS OF IONS USED TO CONFIRM THE PRESENCE OF POLYBROMINATED BIPHENYLS*

Hexabromobiphenyl Theoretical			Observed	
626:628	0.766	7-10	(Striped Bass)	
628:630	1.363		626:628	0.715
630:632	2.553		628:630	1.346
			630:632	2.102
		9-1	(Bluefish, Composite)	
			626:628	0.754
			628:630	1.373
			630:632	NR[a]
		23-1	(Winter Flounder)	
			626:628	0.762
			628:630	1.367
			630:632	NR[a]
		7659	(Soil)	
			626:628	0.725
			628:630	1.345
			630:632	2.345
		7728	(Soil)	
			626:628	0.772
			628:630	1.370
			630:632	2.655
		7651	(Soil)	
			626:628	0.727
			628:630	1.390
			630:632	2.640
		7716	(Sediment)	
			626:628	0.727
			628:630	1.481
			630:632	2.192
		7732	(Vegetation)	
			626:628	0.716
			628:630	1.509
			630:632	2.404
		8503	(Sediment)	
			626:628	0.714
			628:630	1.334
			630:632	2.053

Table D-1. (continued)

Hexabromobiphenyl Theoretical		Observed	
	7654	(Vegetation)	
		626:628	0.746
		628:630	1.373
		630:632	2.404
	7662	(Sludge)	
		626:628	0.886
		628:630	1.318
		630:632	1.987

[a]Not Recorded

*Applies to samples reported in Table B-1.

APPENDIX D

Table D-2. RATIOS OF IONS USED TO CONFIRM THE PRESENCE OF POLYBROMINATED BIPHENYLS*

Hexabromobiphenyl Theoretical		Observed		
626:628	0.766	76-53	Soil	
628:630	1.363		626:628	0.750
630:632	2.553		628:630	1.364
			630:632	2.712
		76-58	Soil	
			626:628	0.761
			628:630	1.323
			630:632	2.239
		77-29	Soil	
			626:628	0.700
			628:630	1.382
			630:632	NR[a]
		77-33	Oleander Leaves	
			626:628	0.716
			628:630	1.377
			630:632	NR[a]

[a]Not recorded.

*Applies to samples reported in Table B-2.

Table D-3. Ratios of Ions Used to Confirm the Presence of Hexabromobiphenyl[a*]

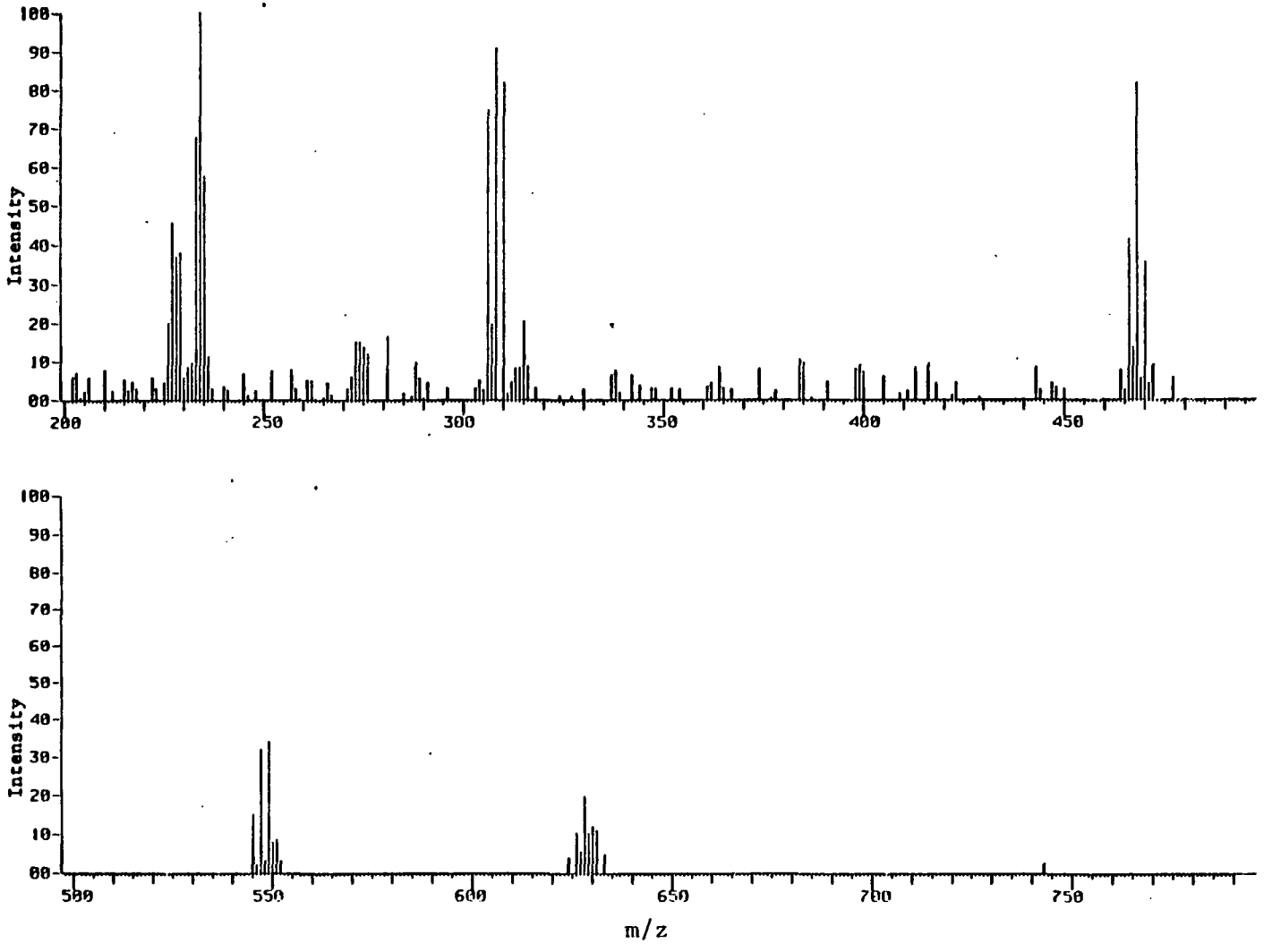
Theoretical			Observed	
626:628	0.766	46-02	626:628	0.732
628:630	1.363		628:630	1.345
630:632	2.553		630:632	2.628
		46-05	626:628	0.732
			628:630	1.340
			630:632	2.430
		46-06	626:628	0.753
			628:630	1.367
			630:632	2.566
		46-07	626:628	0.755
			628:630	1.407
			630:632	2.378
		46-08	626:628	0.750
			628:630	1.308
			630:632	2.433
		46-18	626:628	0.729
			628:630	1.322
			630:632	2.451
		46-19	626:628	0.798
			628:630	1.246
			630:632	2.311
		46-27	626:628	0.701
			628:630	1.380
			630:632	2.623

[a]Selected Ion Monitoring to confirm ion ratios was performed on the Finnigan 3300 under conditions delineated in footnote a of Table B-3.

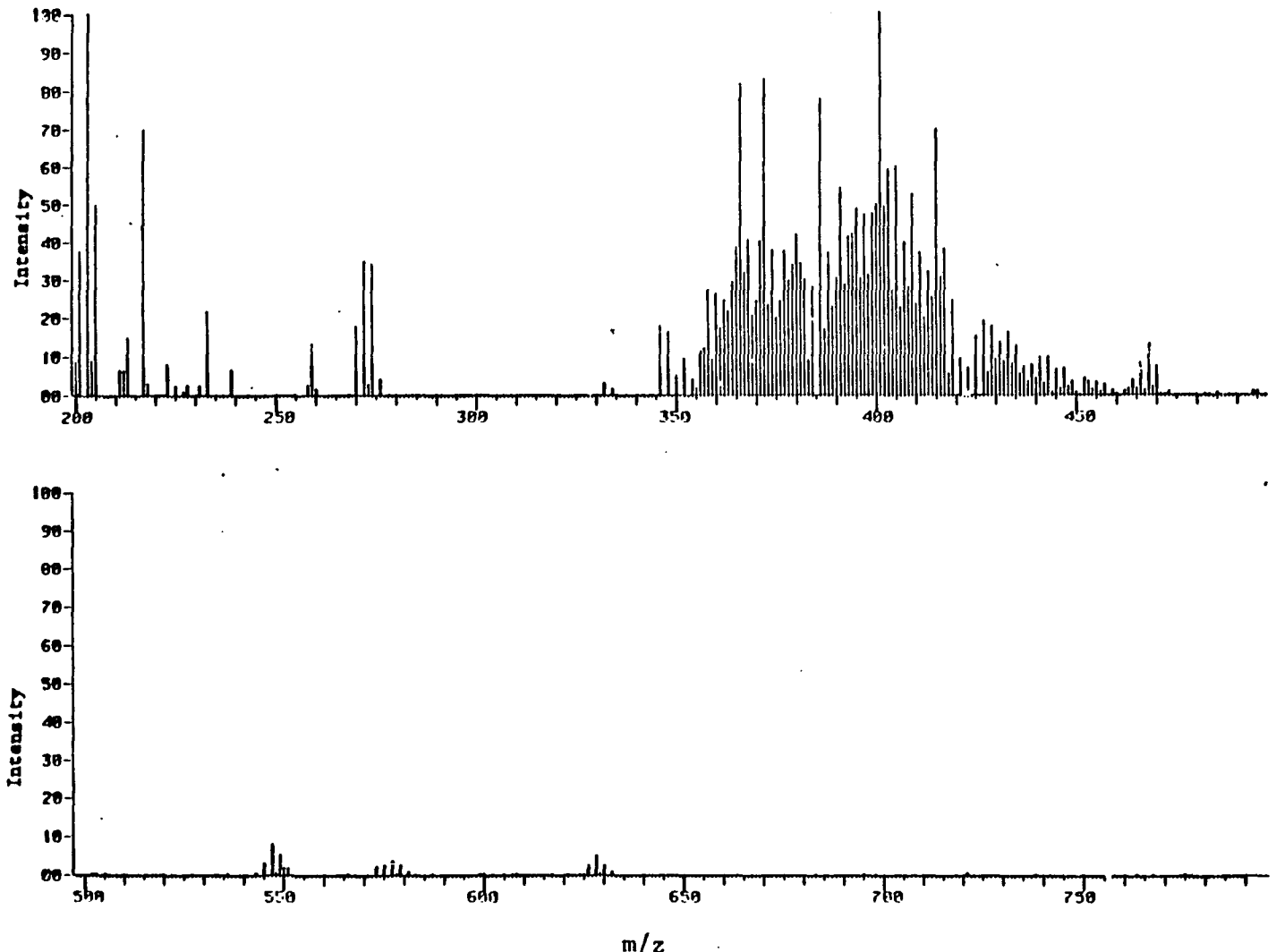
*Applies to samples reported in Table B-3.

APPENDIX E

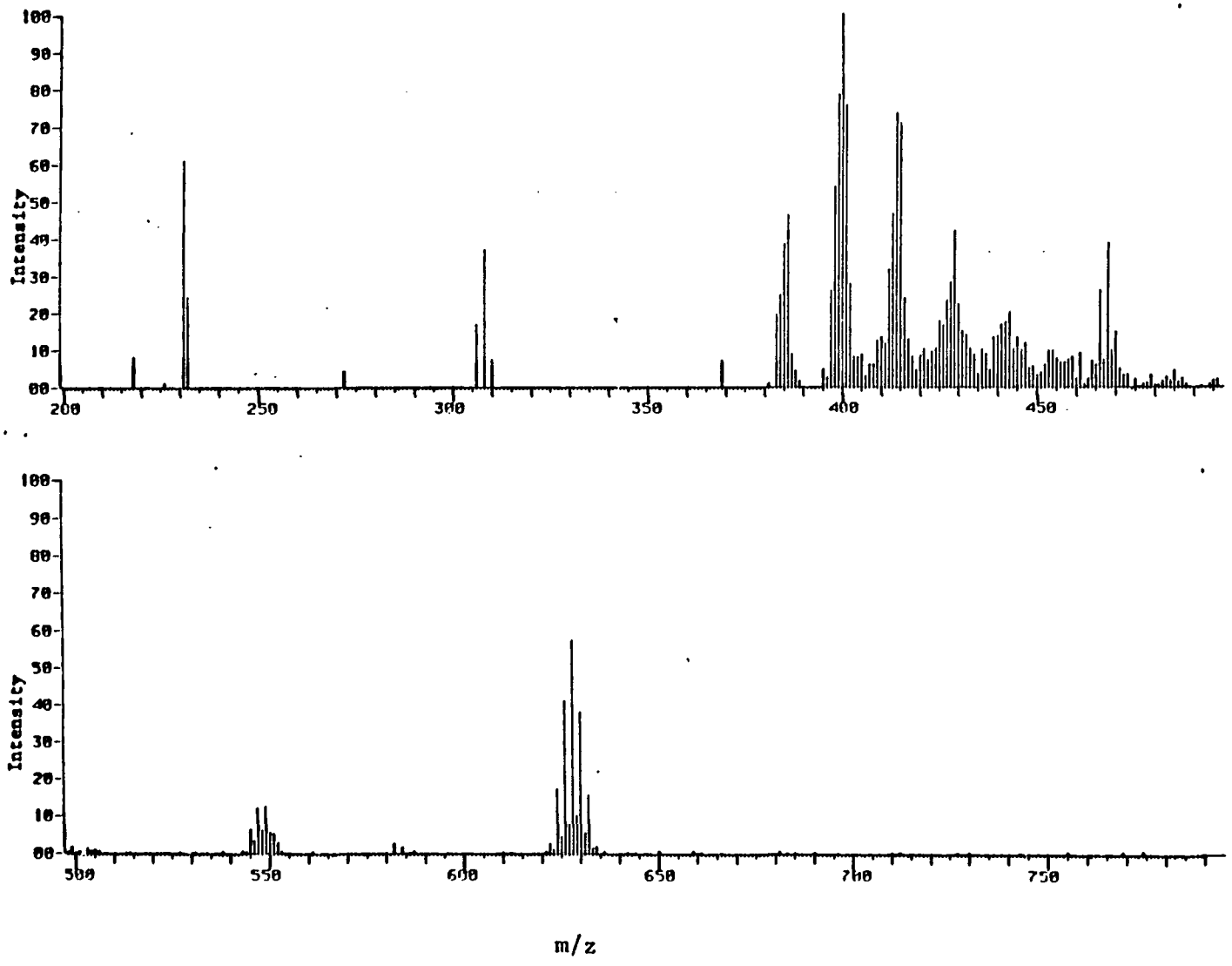
FULL SCAN SPECTRAL CONFIRMATION OF THE PRESENCE OF POLYBROMINATED BIPHENYLS FOR REPRESENTATIVE SAMPLES



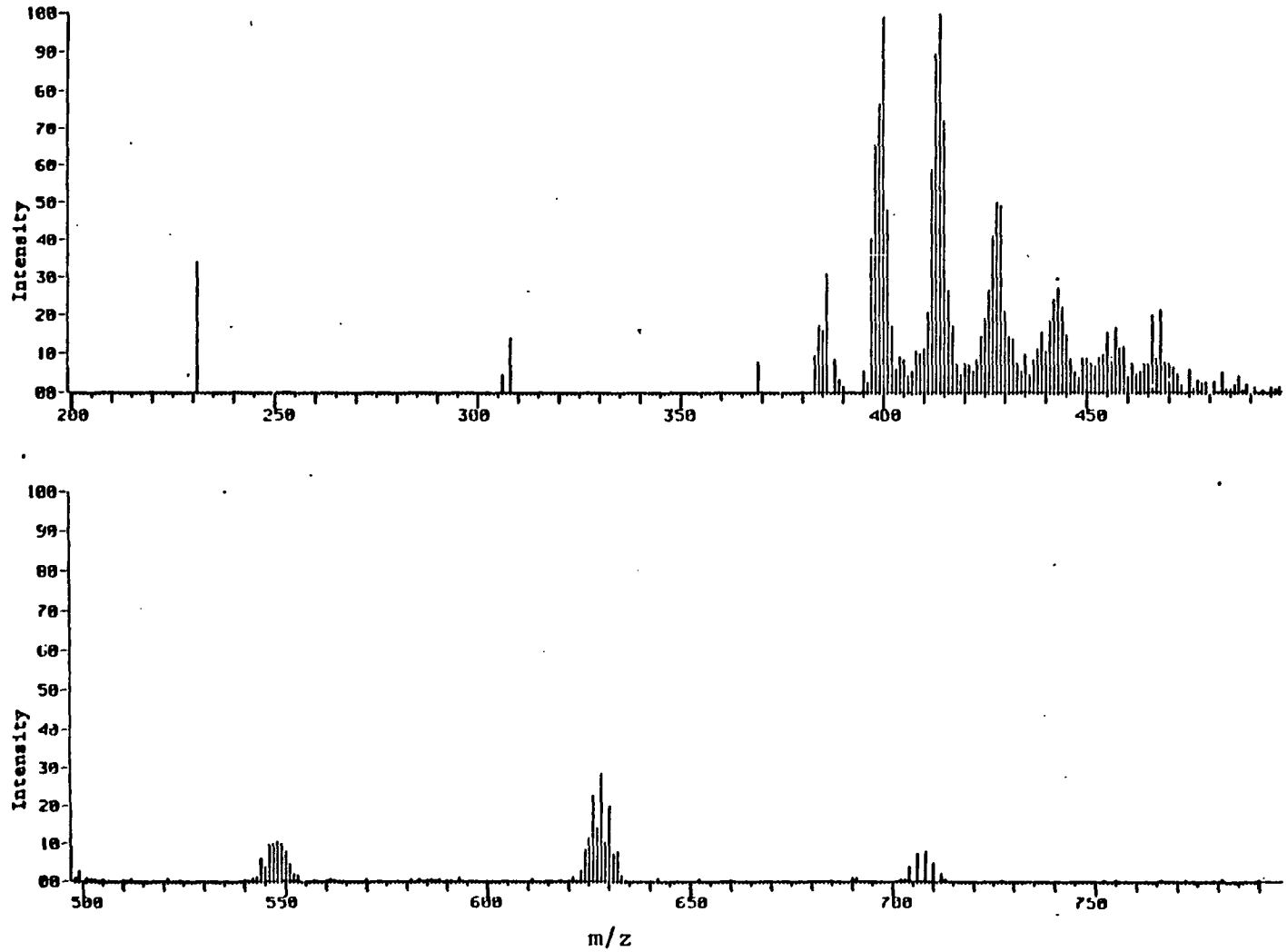
7728 (Soil), 1.66 minutes, hexabromobiphenyl



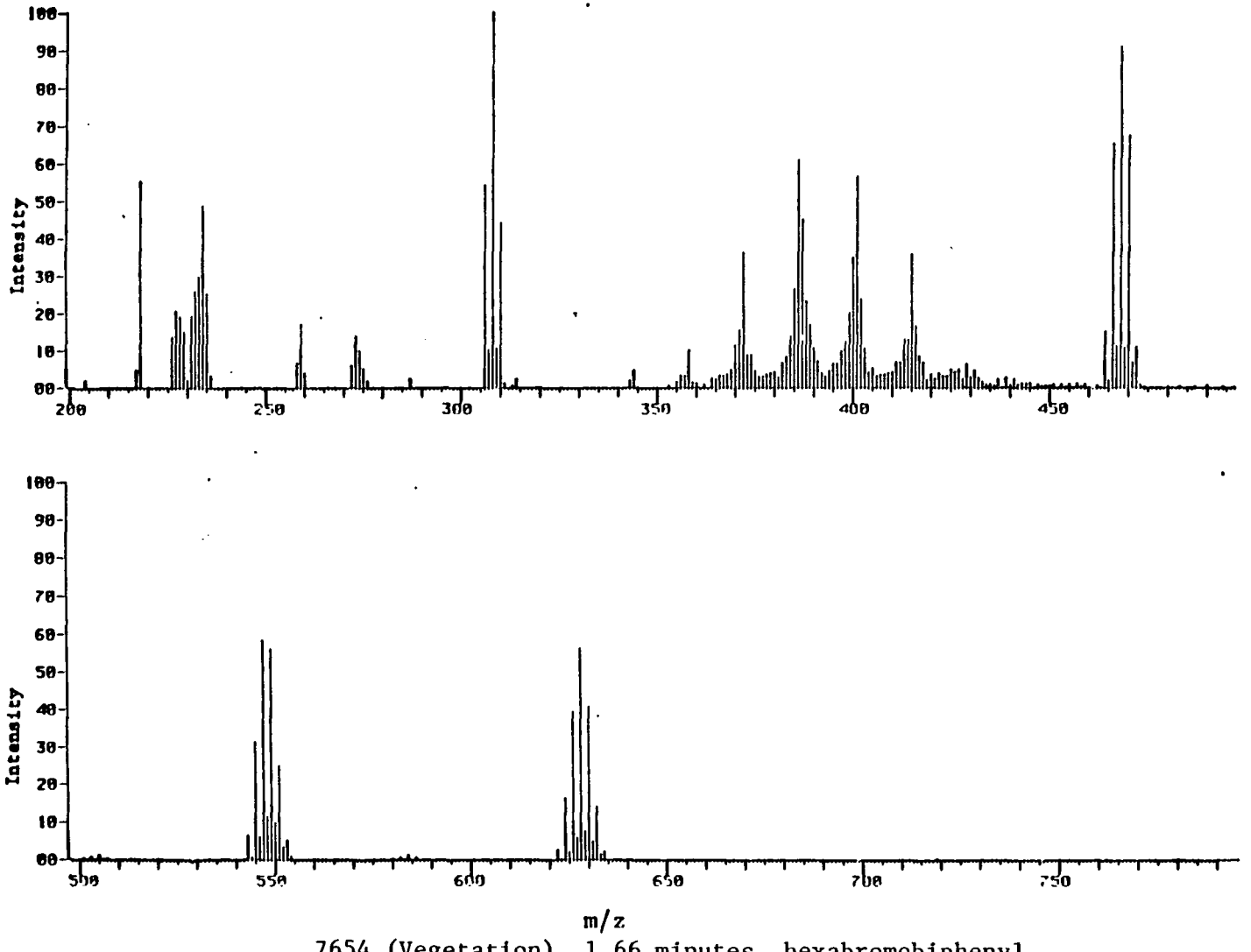
8528 (Sediment), 1.58 minutes, hexabromobiphenyl



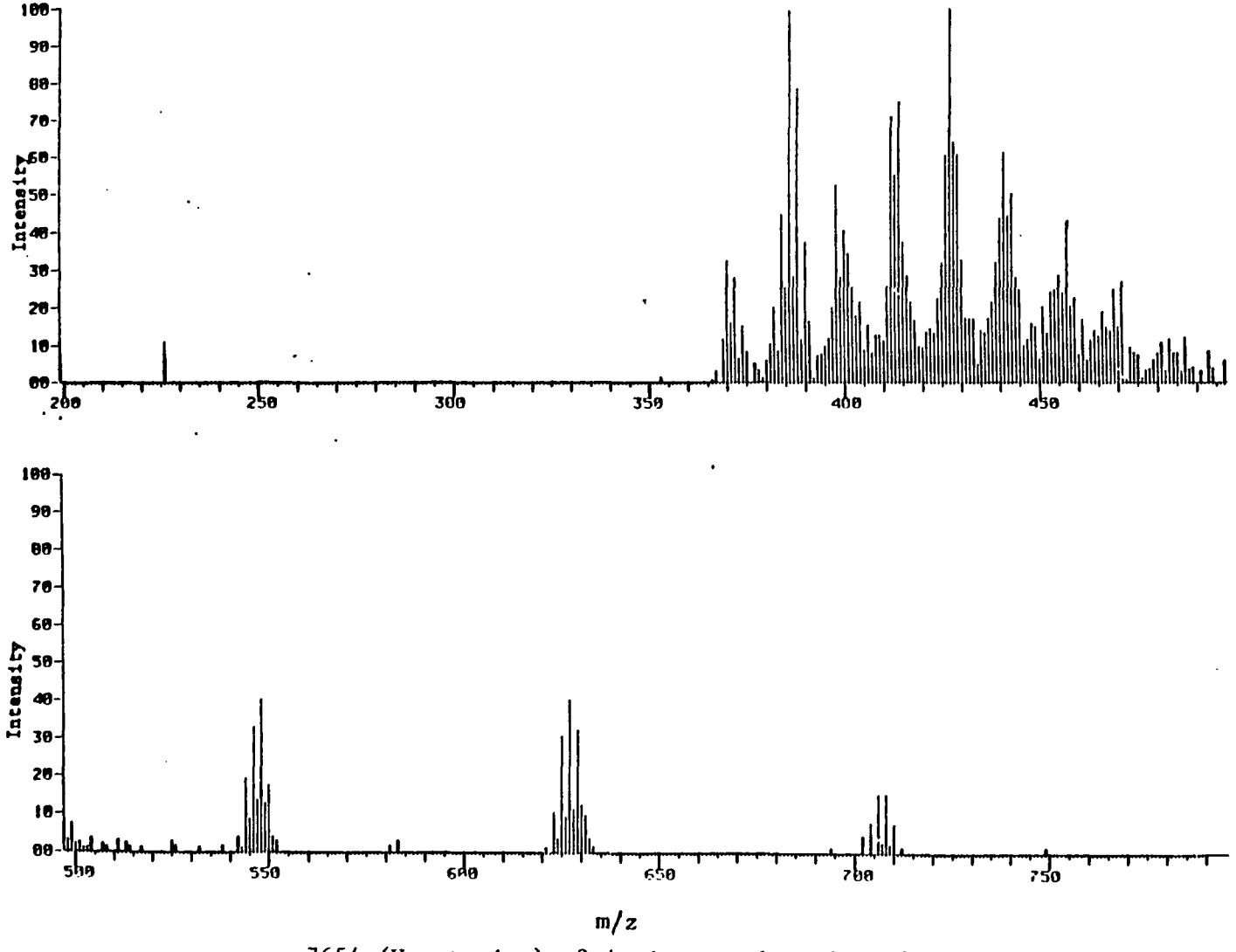
7730 (Soil), 1.66 minutes, hexabromobiphenyl



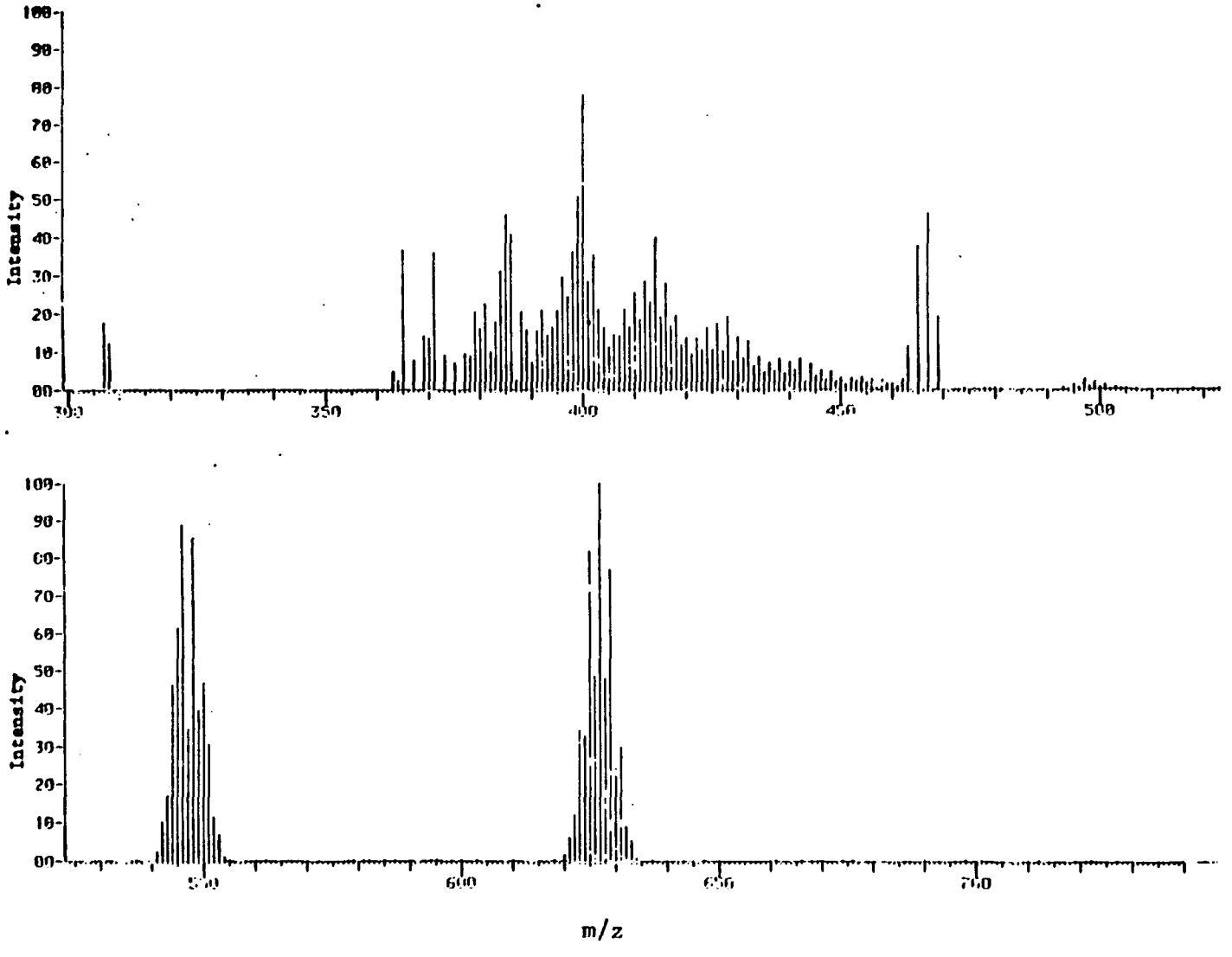
7730 (Soil), 2.38 minutes, heptabromobiphenyl



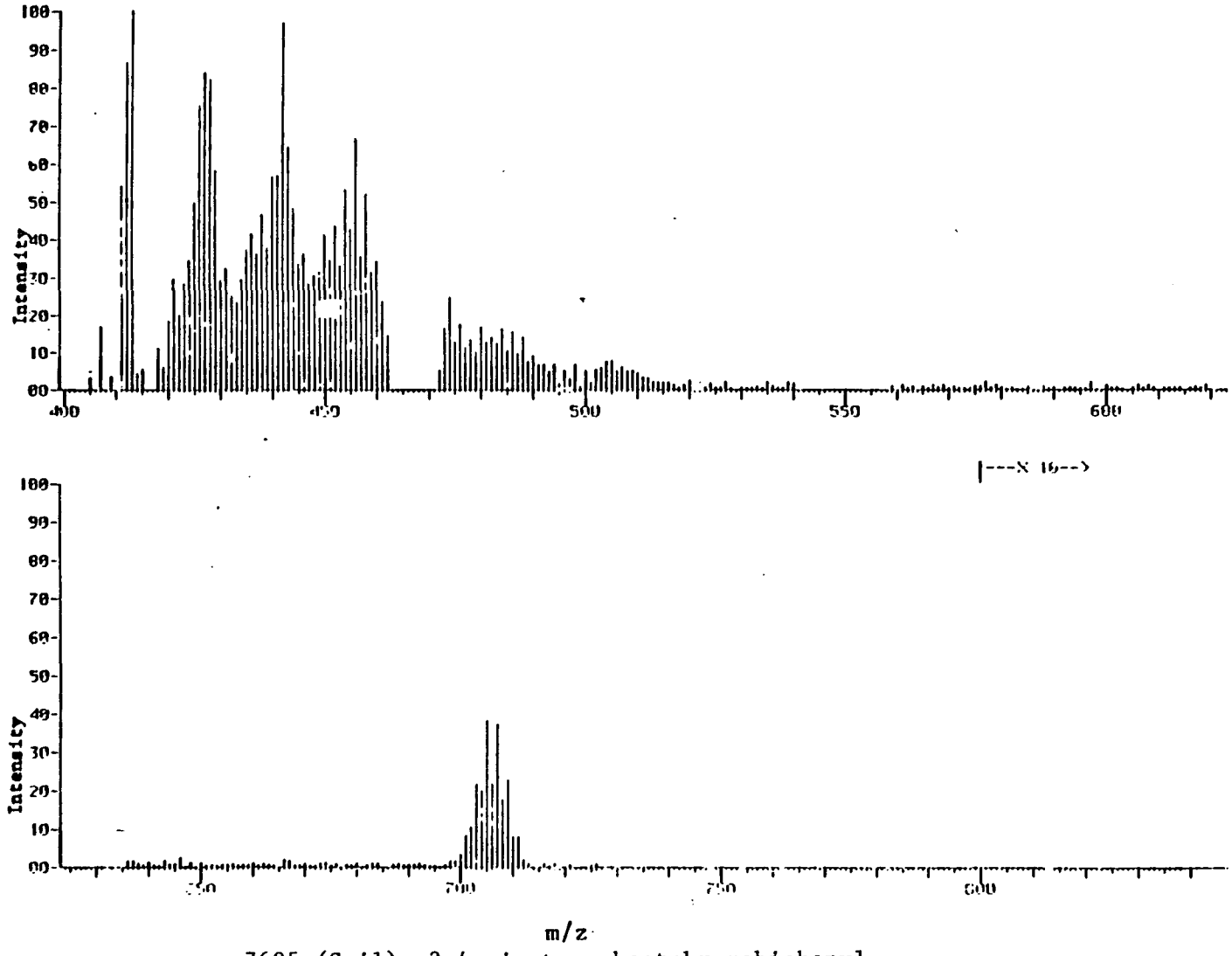
7654 (Vegetation), 1.66 minutes, hexabromobiphenyl



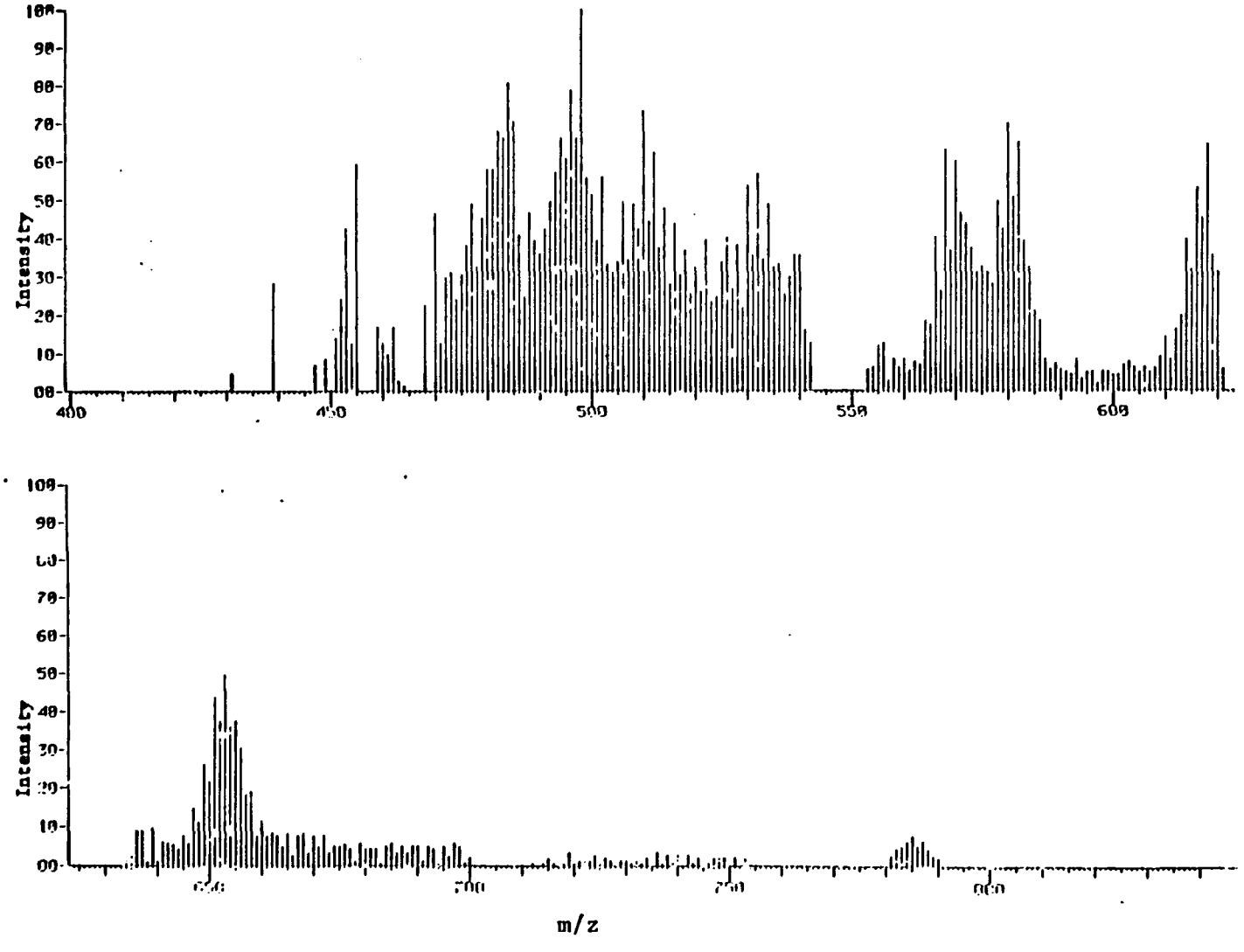
7654 (Vegetation), 2.4 minutes, heptabromobiphenyl



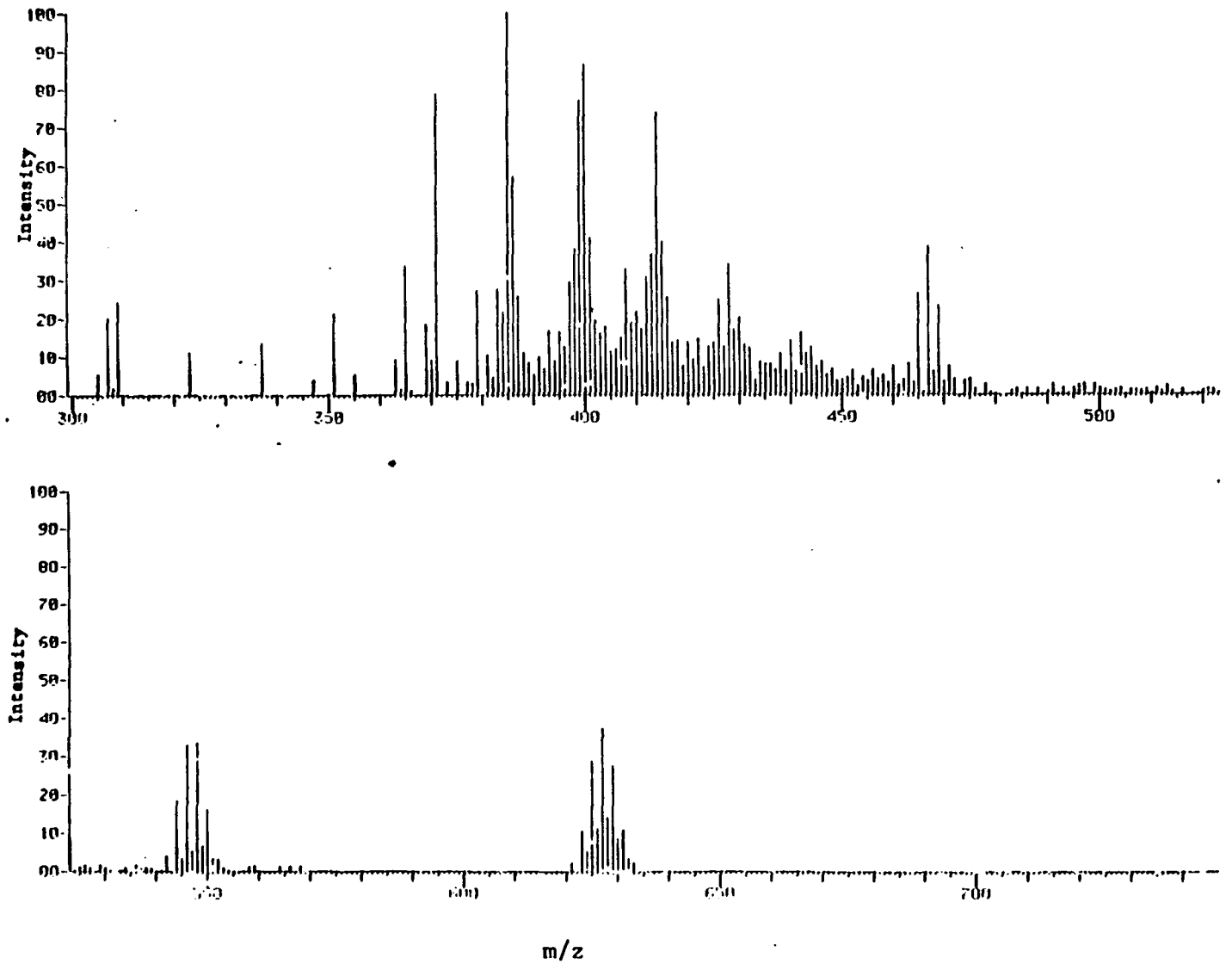
7695 (Soil), 1.66 minutes, hexabromobiphenyl



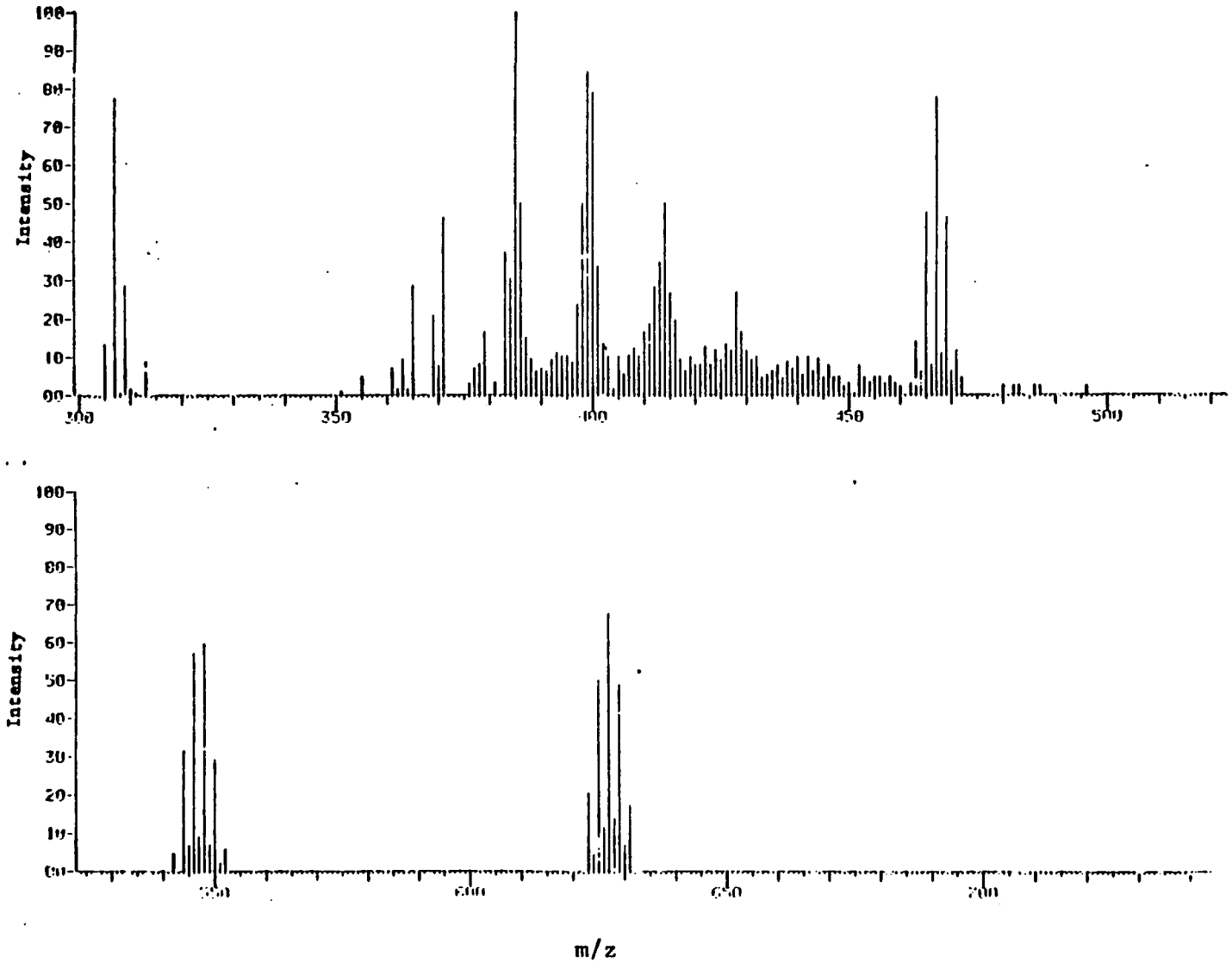
7695 (Soil), 2.4 minutes, heptabromobiphenyl



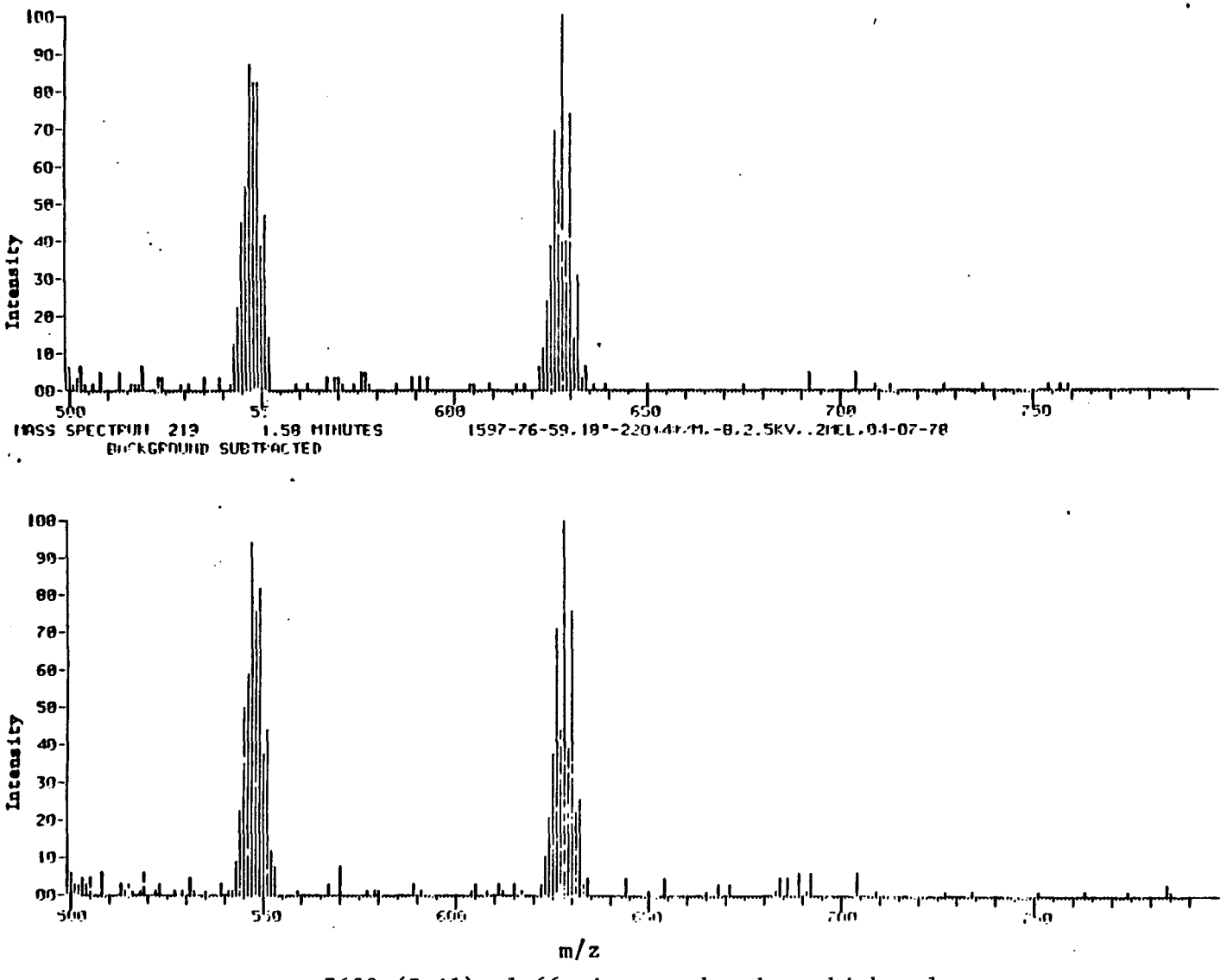
7695 (Soil), 7.7 minutes, octabromobiphenyl



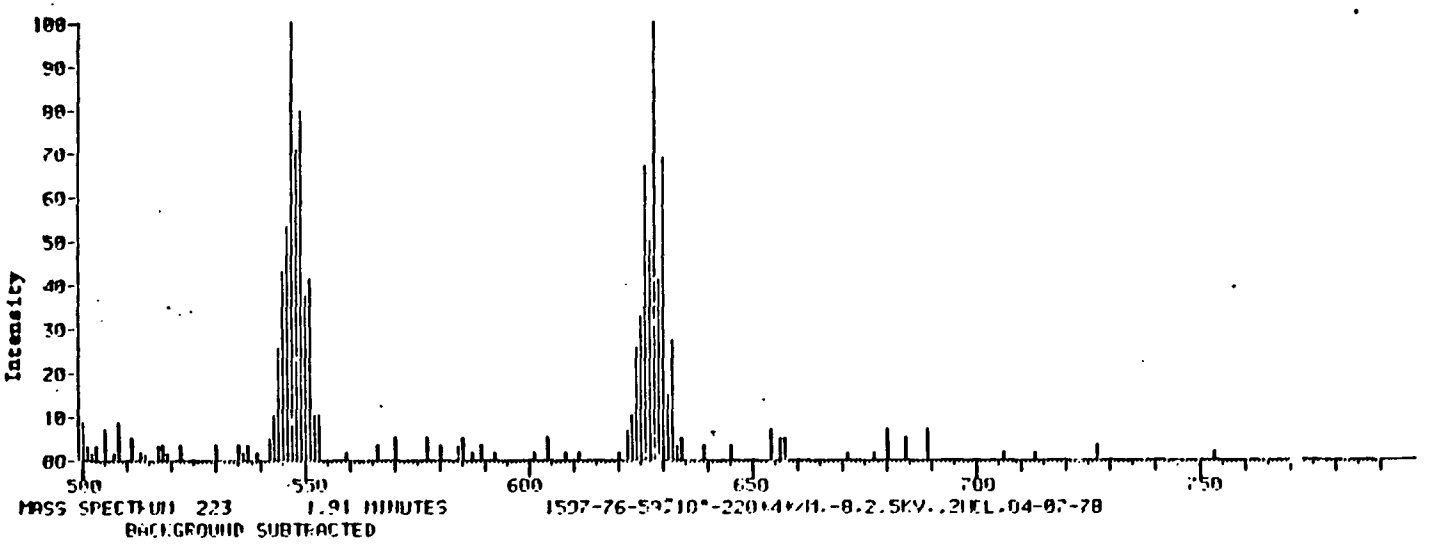
7727 (Soil), 1.66 minutes, hexabromobiphenyl

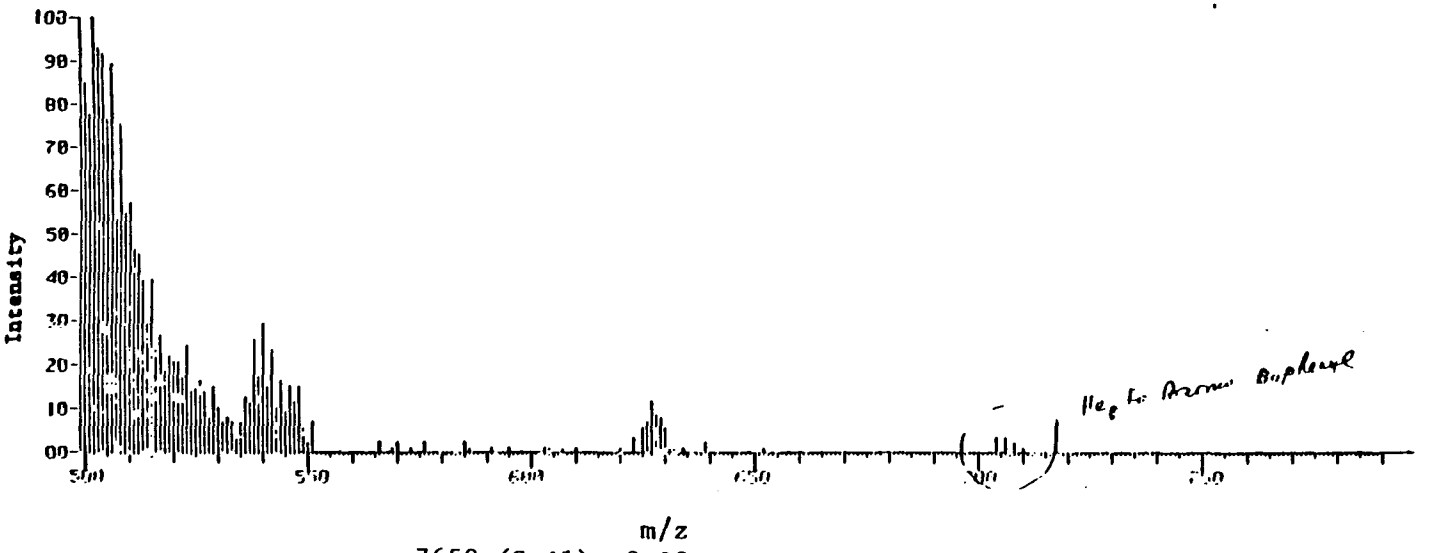


7657 (Soil), 1.66 minutes, hexabromobiphenyl

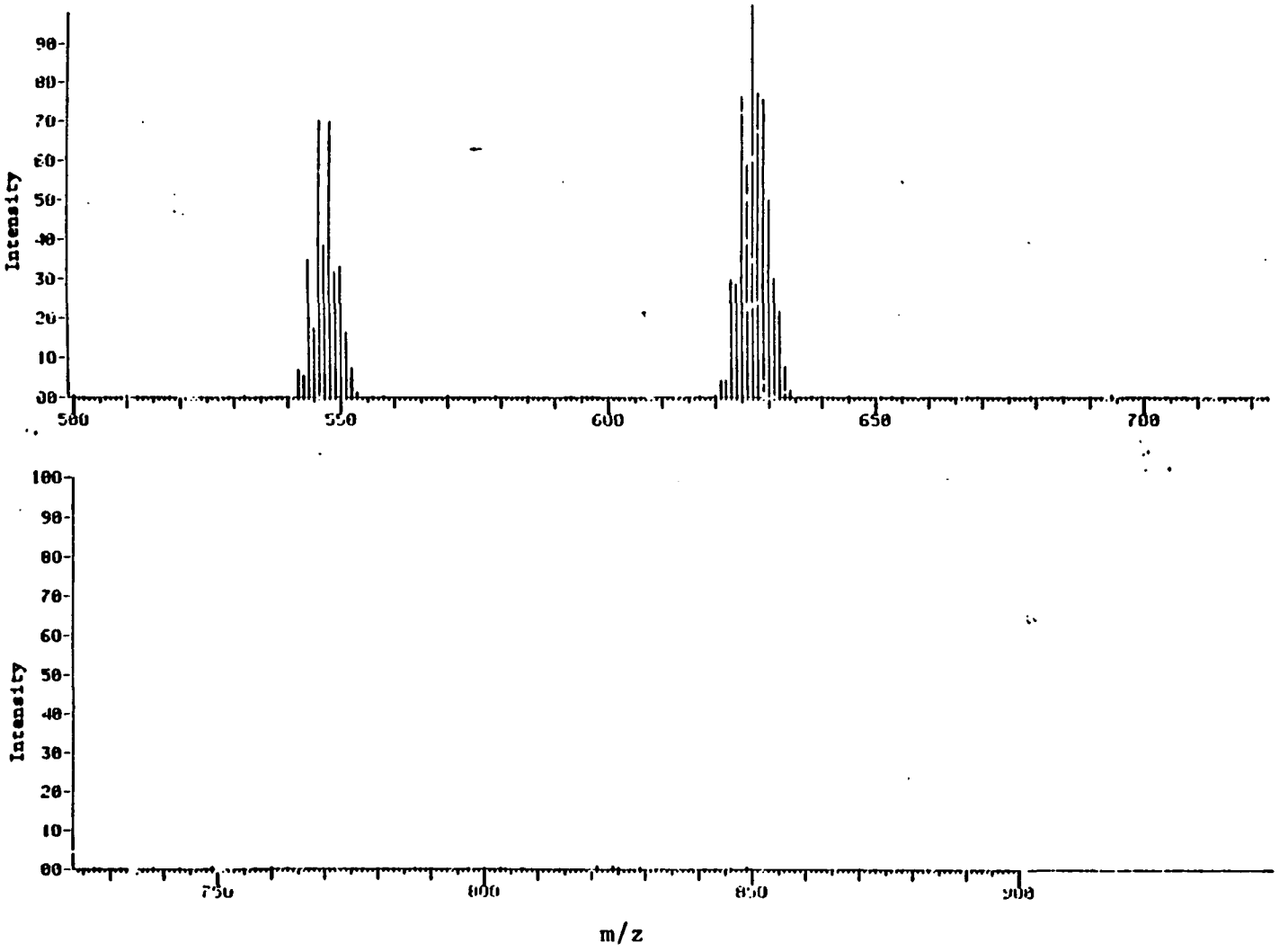


7659 (Soil), 1.66 minutes, hexabromobiphenyl

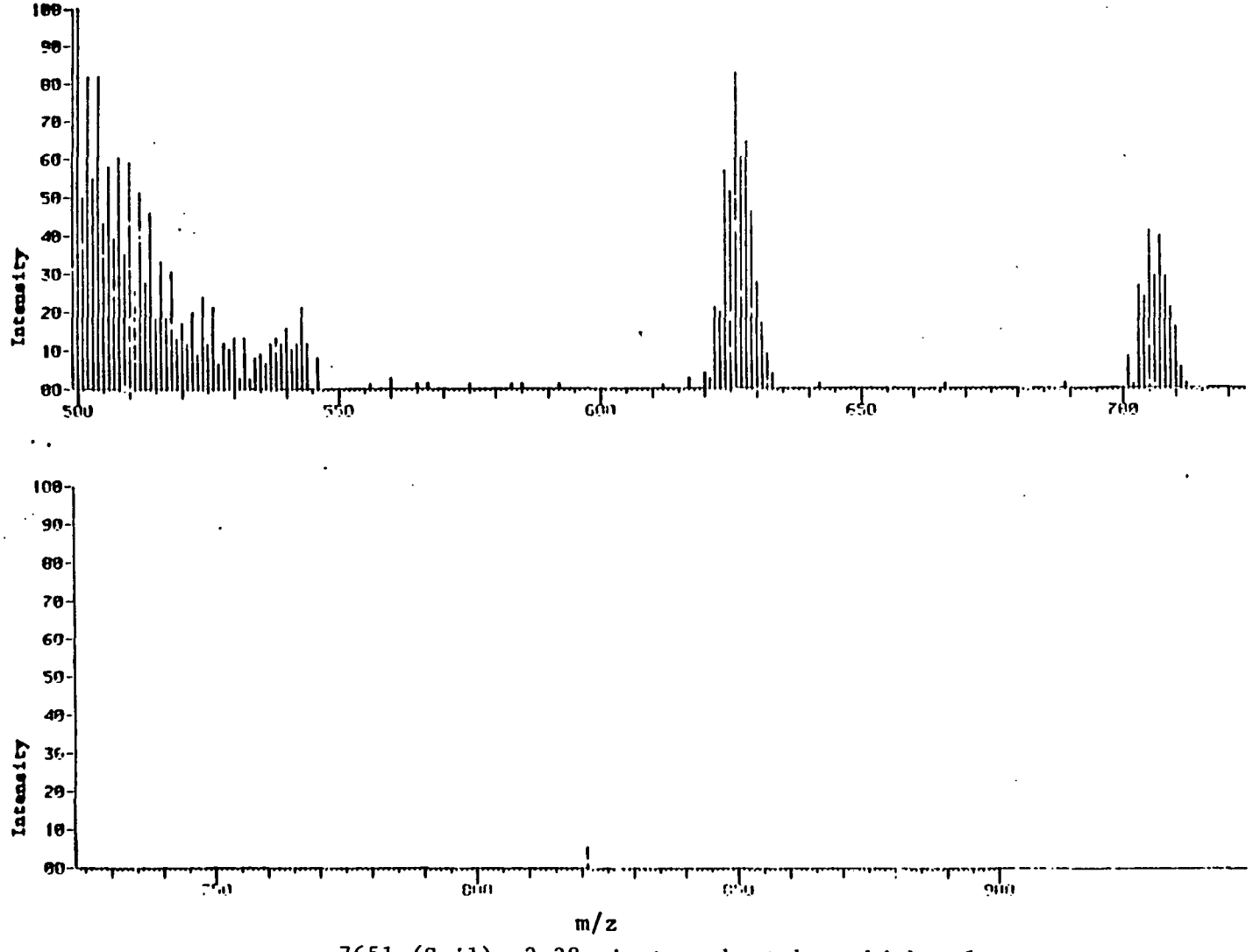




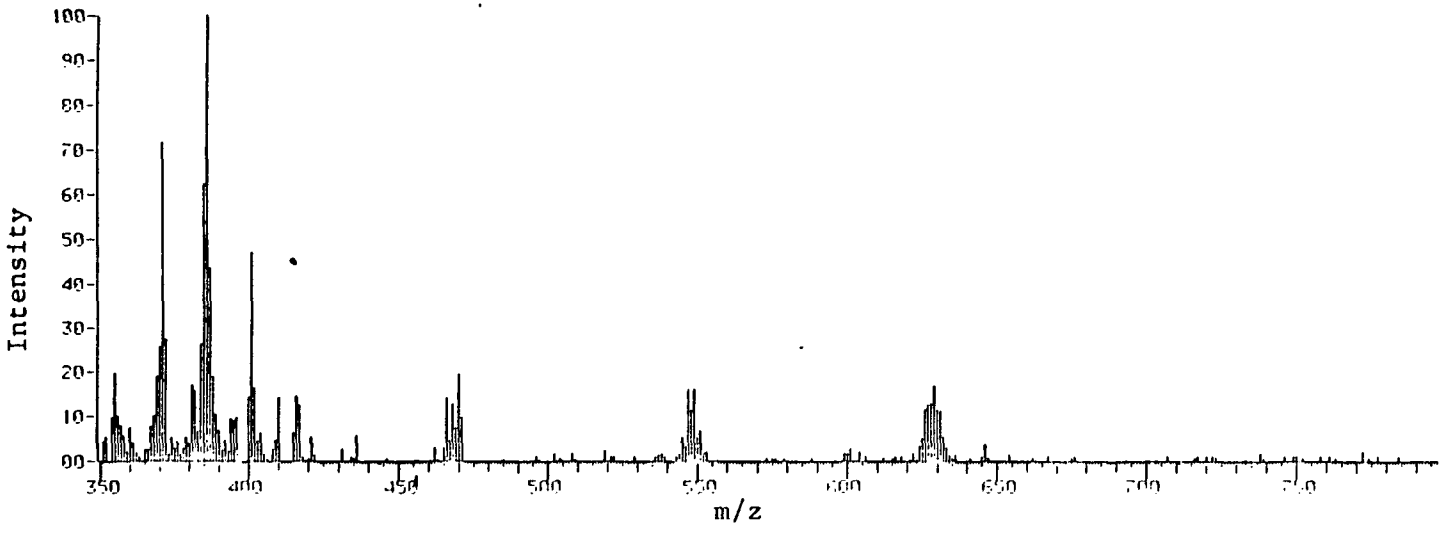
7659 (Soil), 2.38 minutes, heptabromobiphenyl

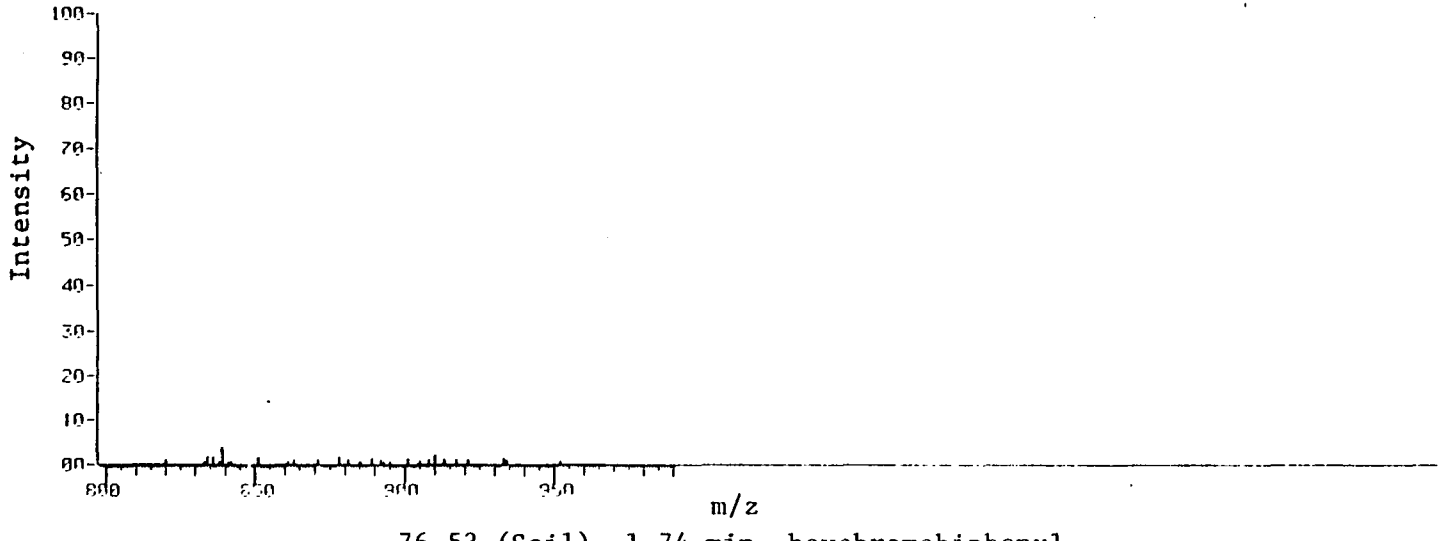


7651 (Soil), 1.66 minutes, hexabromobiphenyl

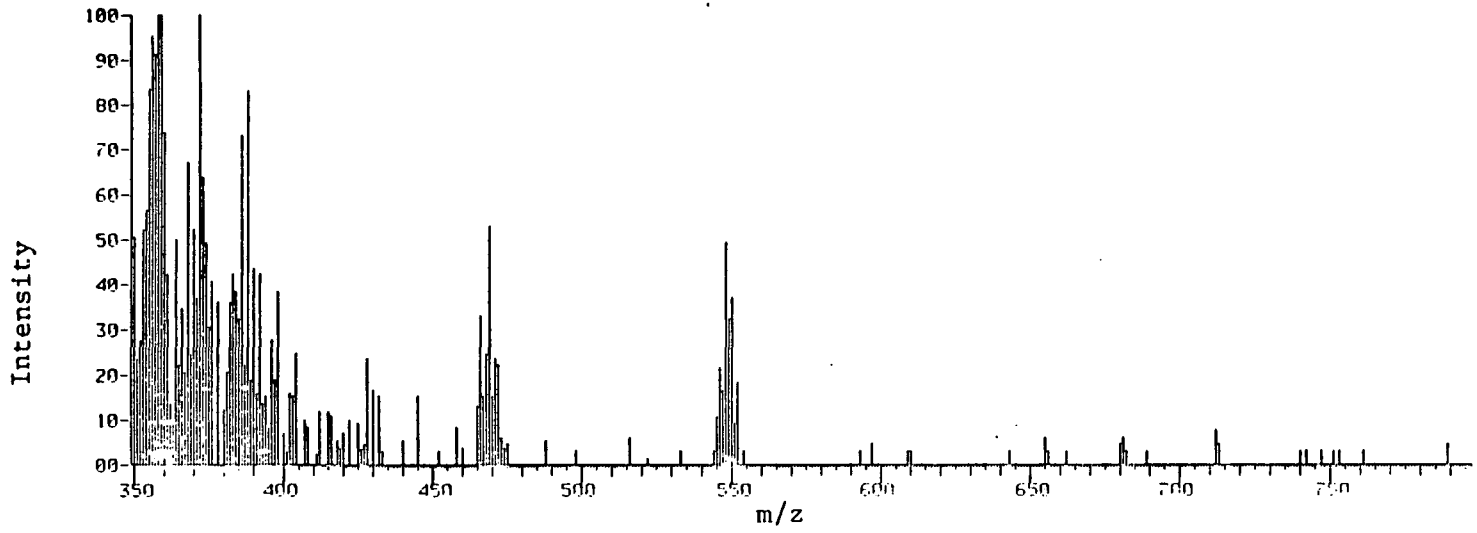


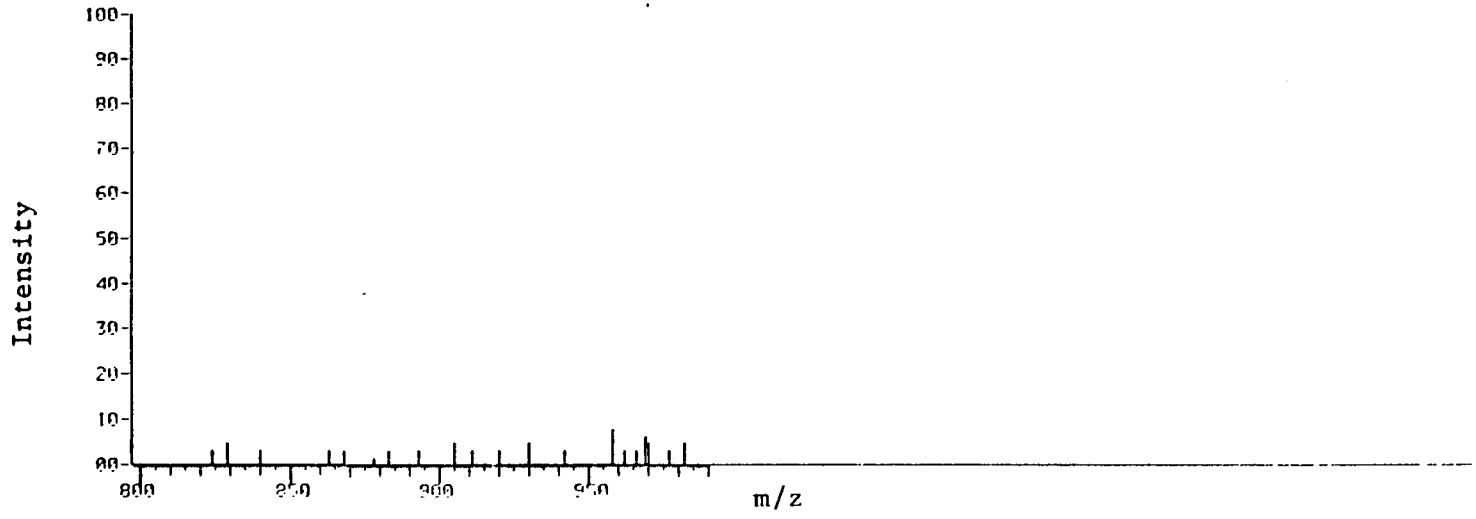
7651 (Soil), 2.38 minutes, heptabromobiphenyl



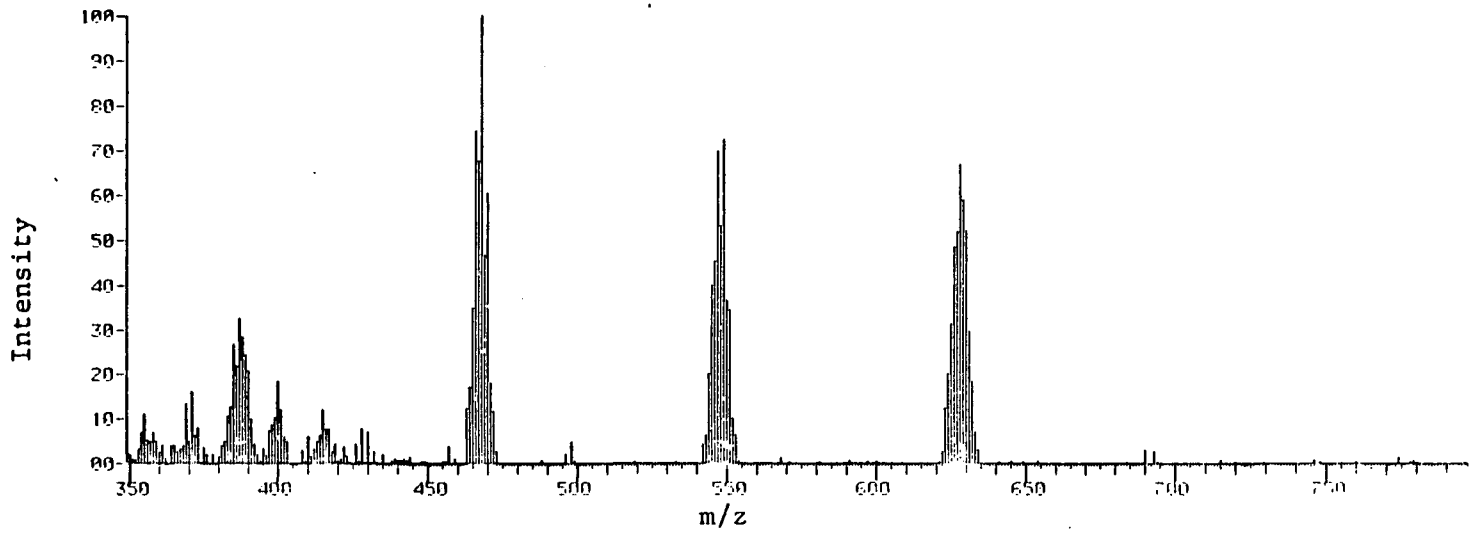


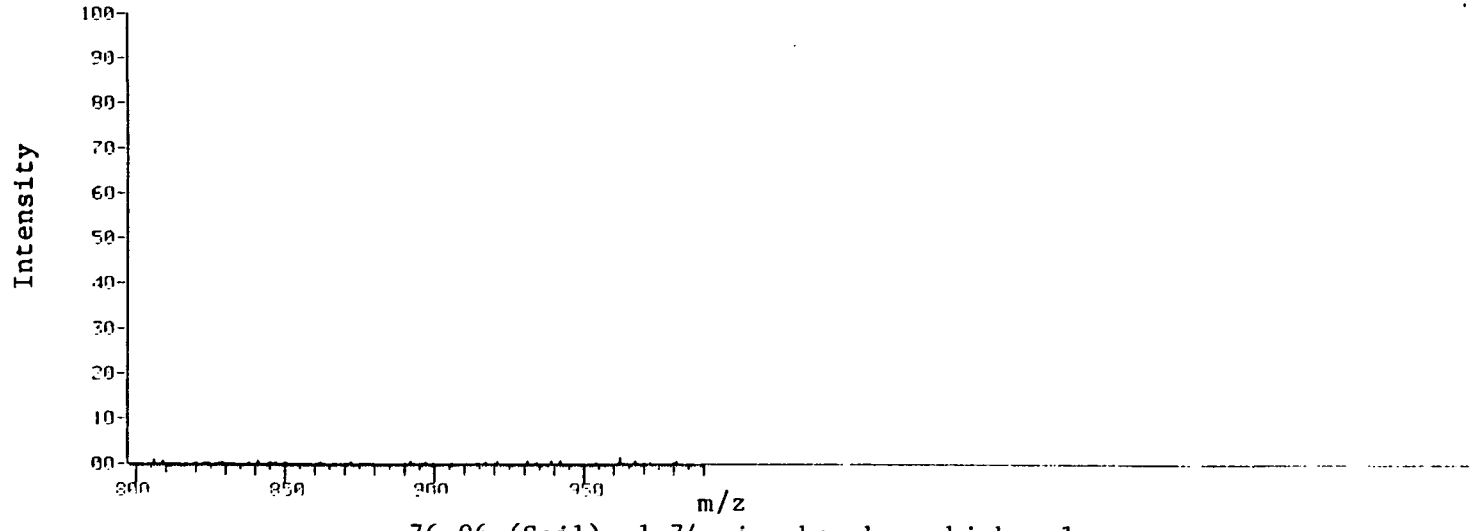
76-53 (Soil), 1.74 min, hexabromobiphenyl



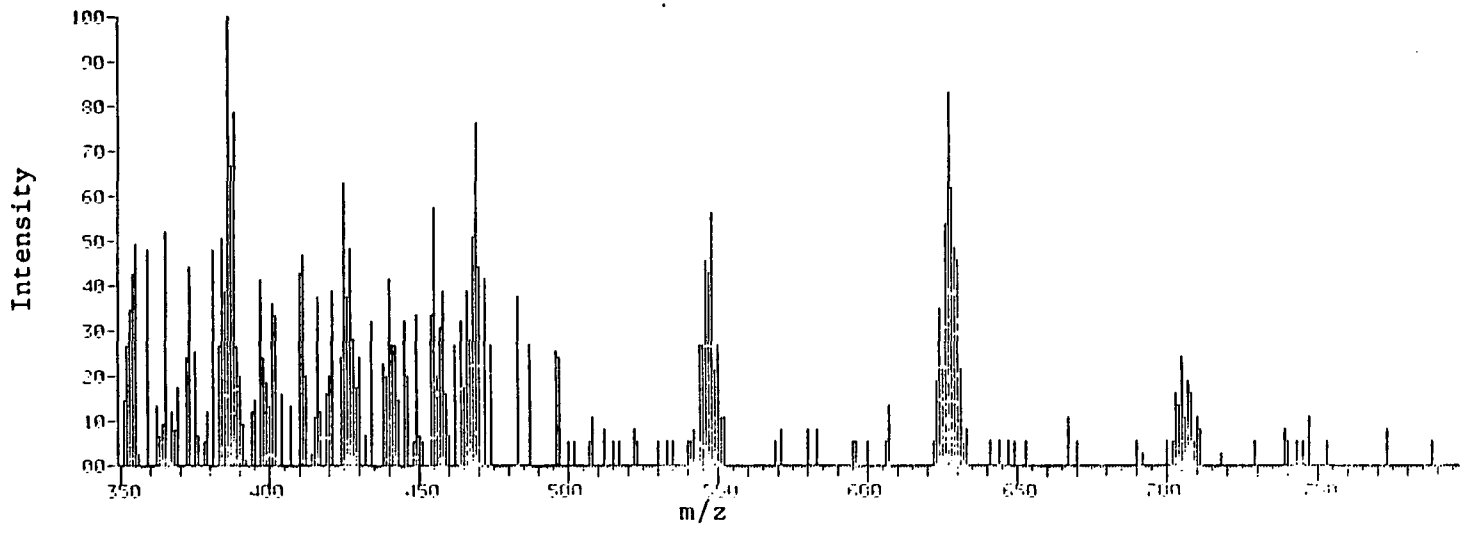


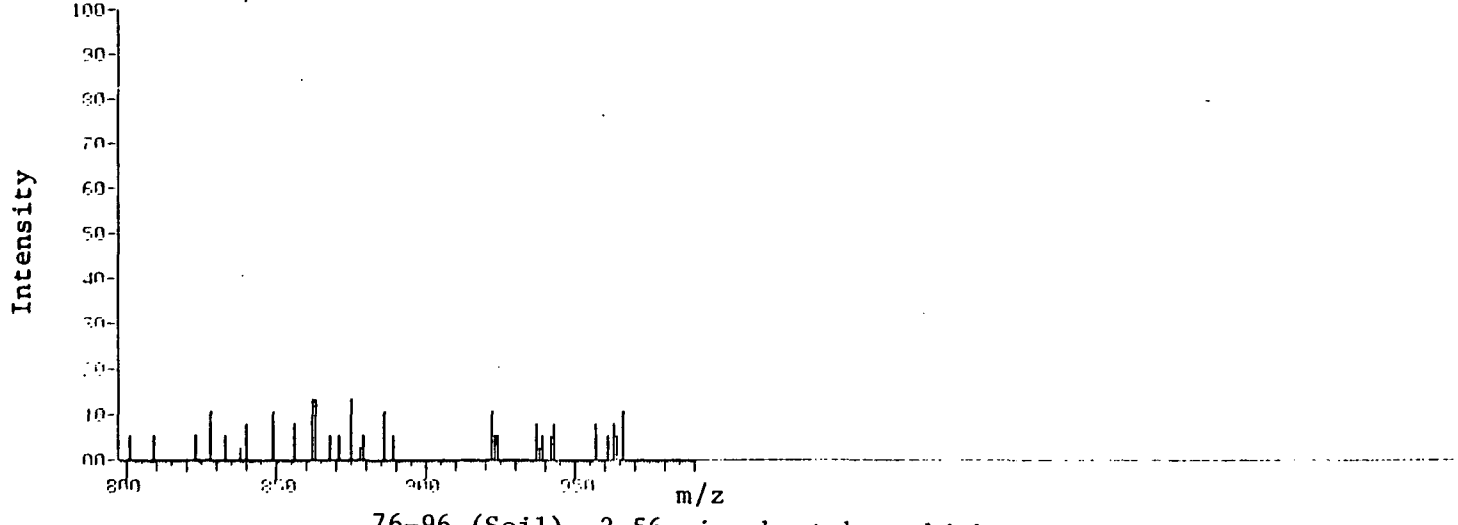
76-96 (Soil), 0.74 min, pentabromobiphenyl



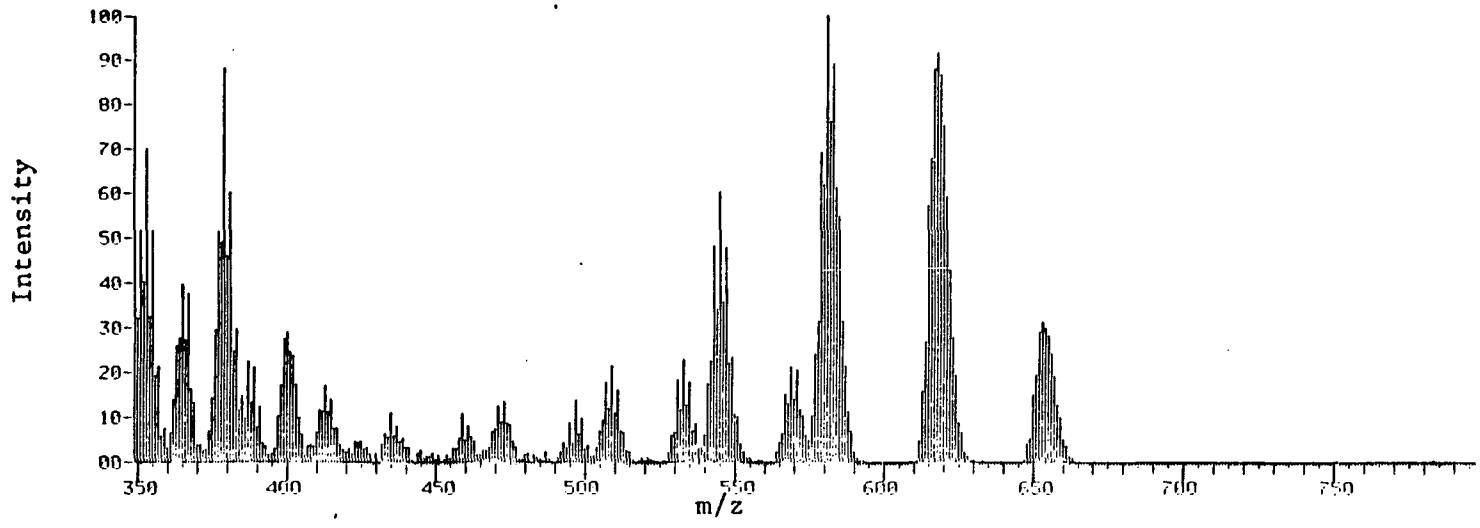


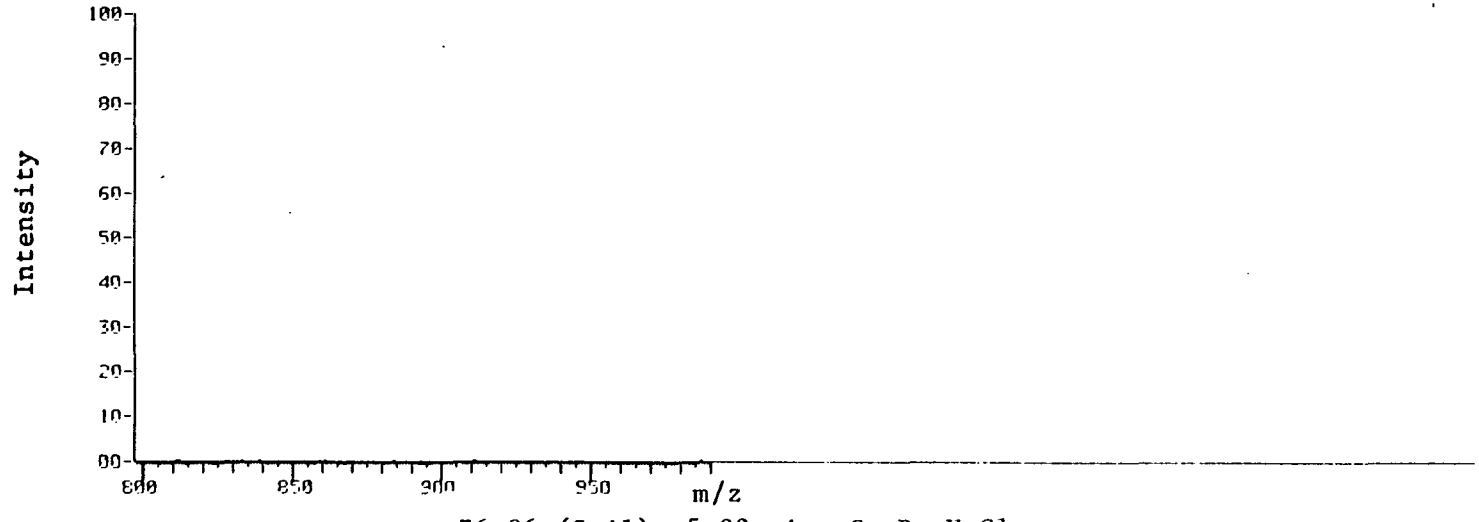
76-96 (Soil), 1.74 min, hexabromobiphenyl



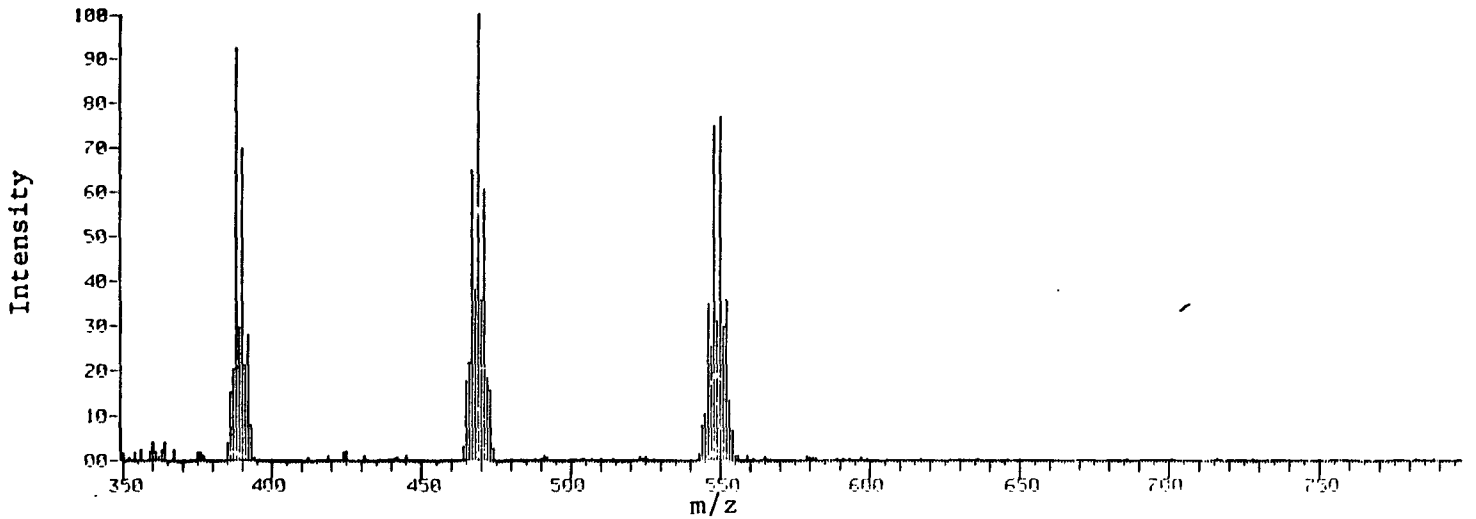


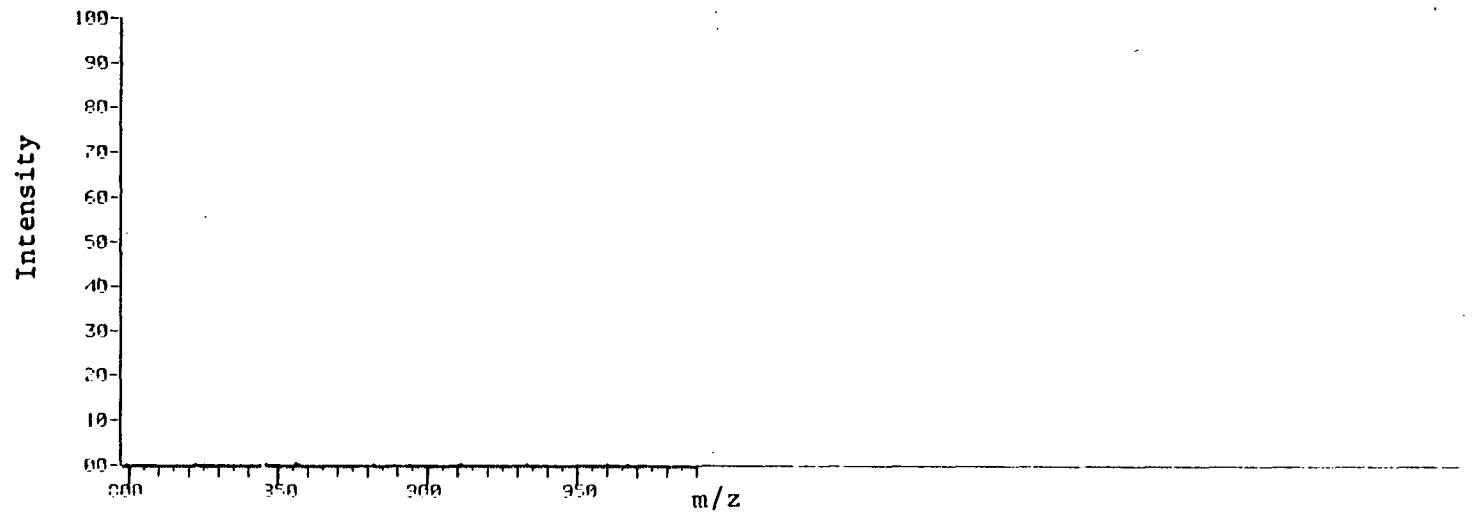
76-96 (Soil), 2.56 min, heptabromobiphenyl



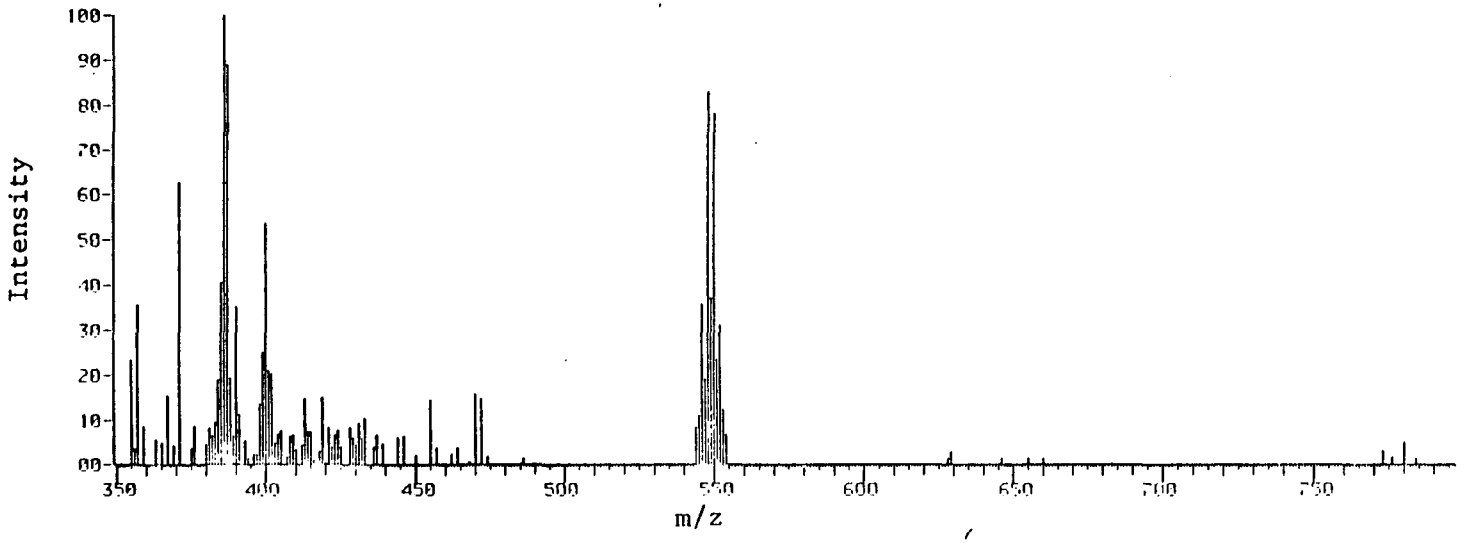


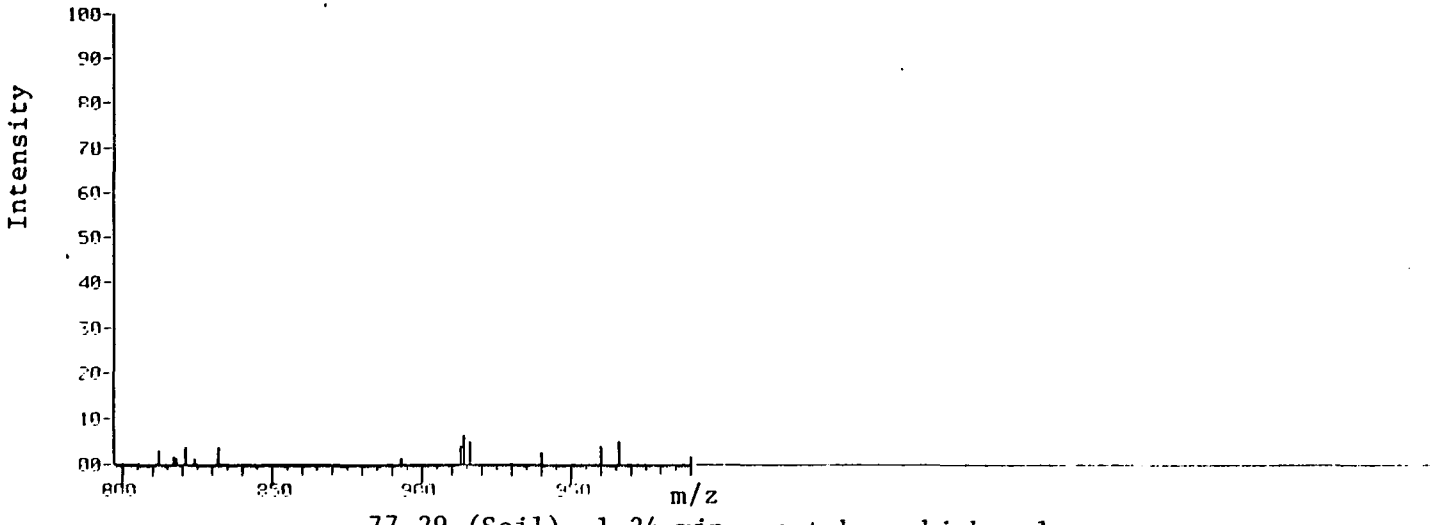
76-96 (Soil), 5.33 min, $C_{12}Br_6H_3Cl$



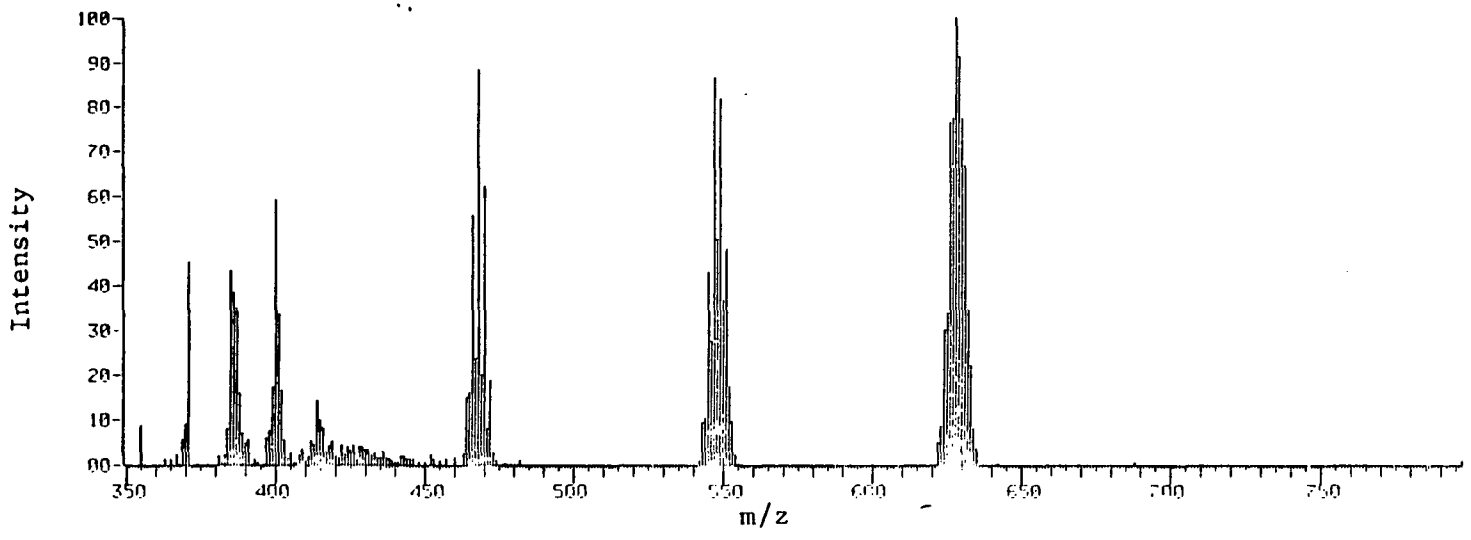


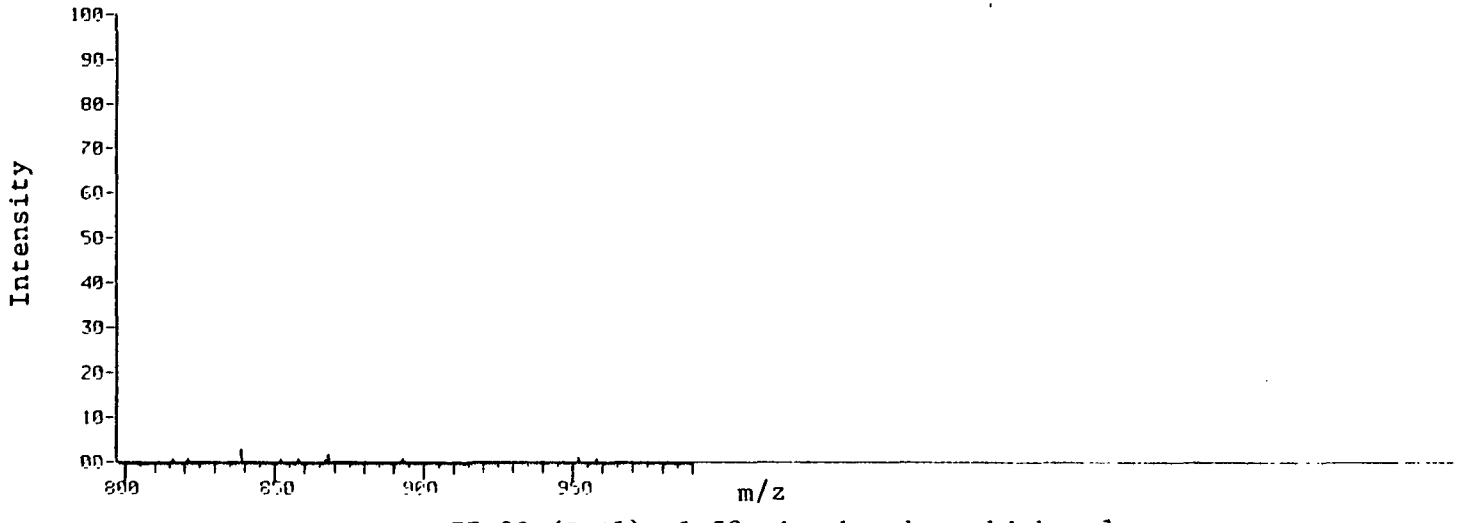
77-29 (Soil), 0.83 min, pentabromobiphenyl



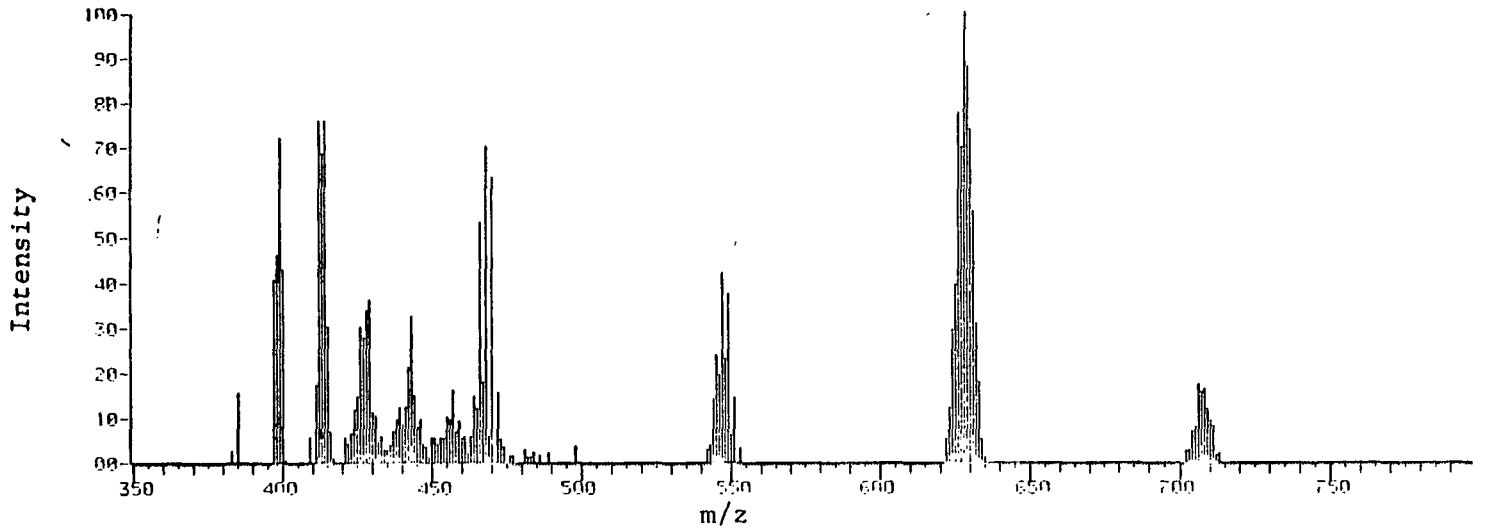


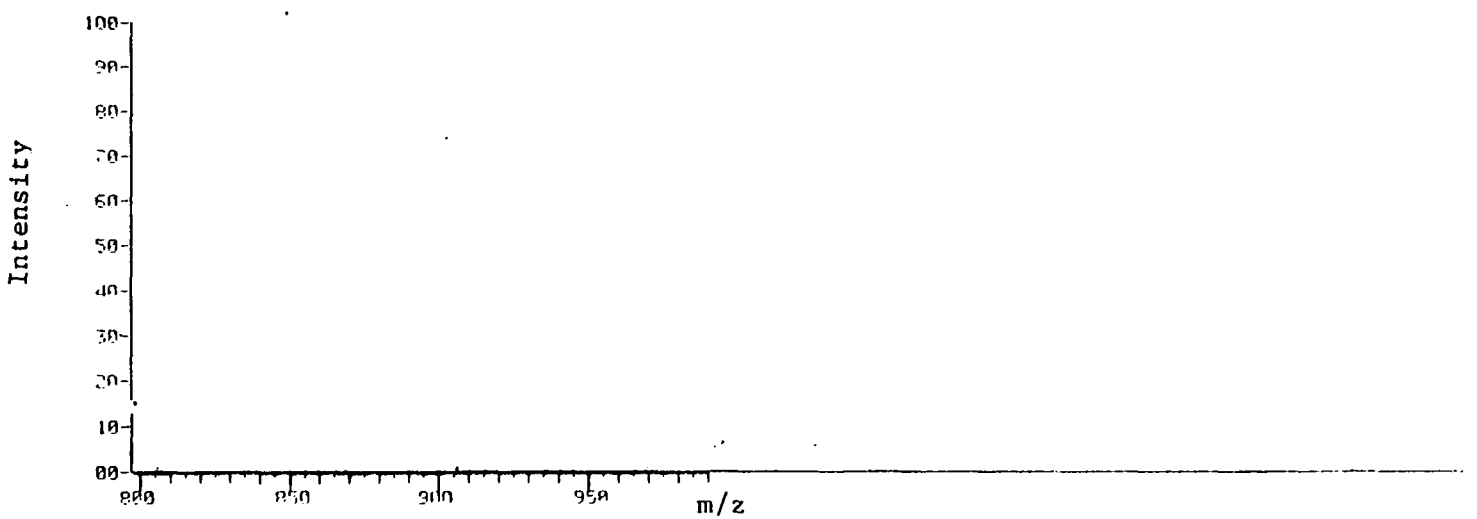
77-29 (Soil), 1.24 min, pentabromobiphenyl



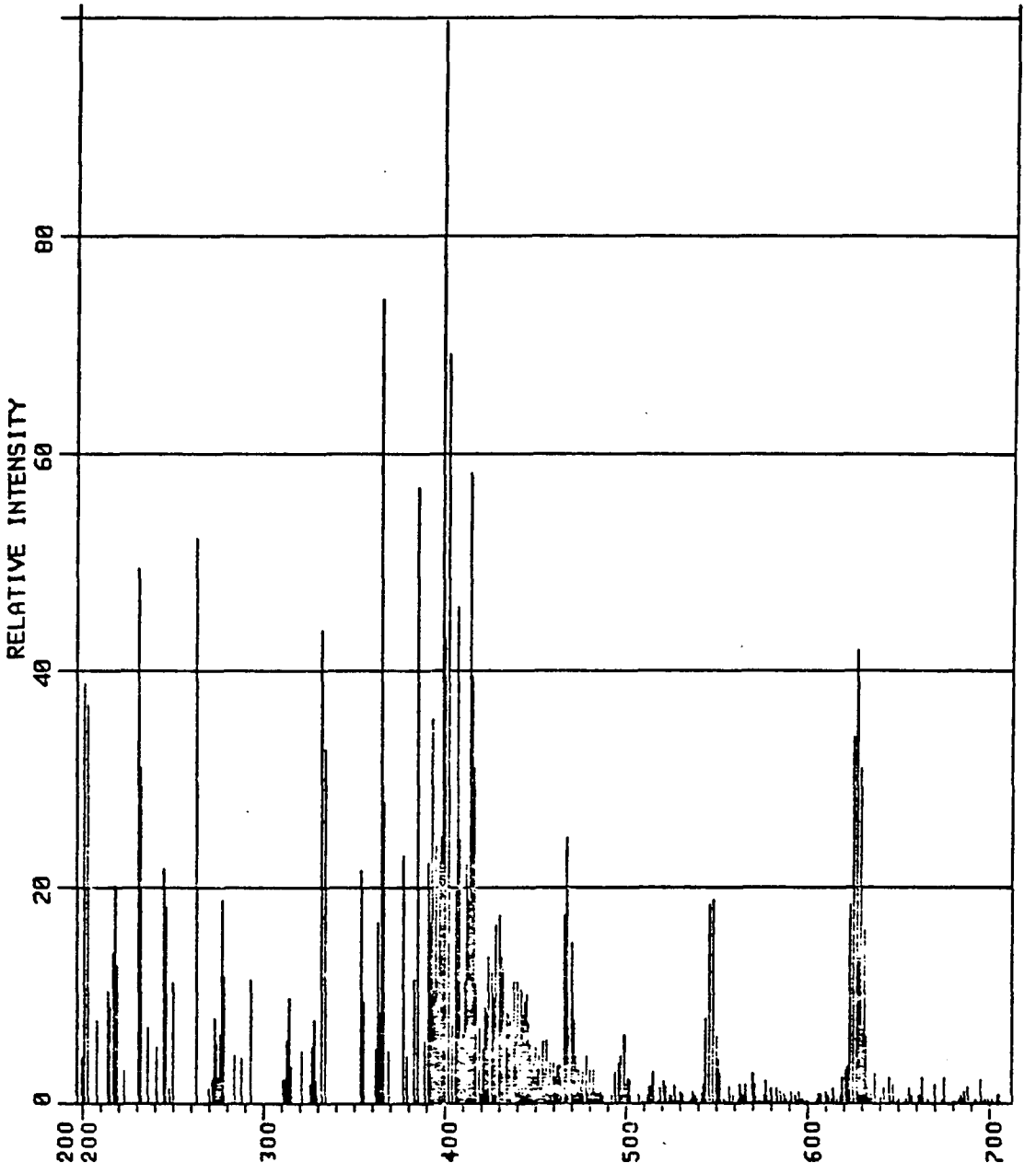


77-29 (Soil), 1.58 min, hexabromobiphenyl

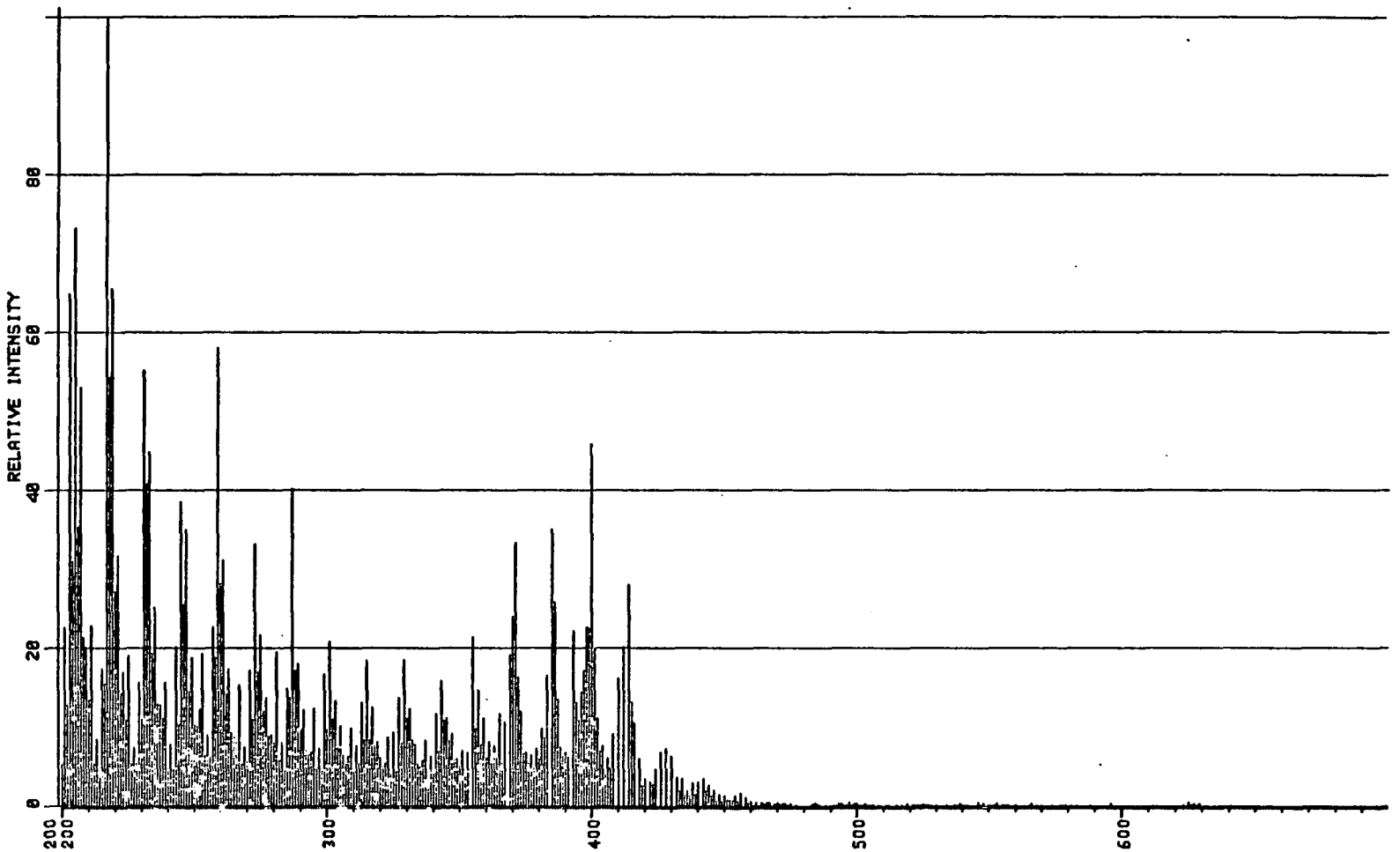




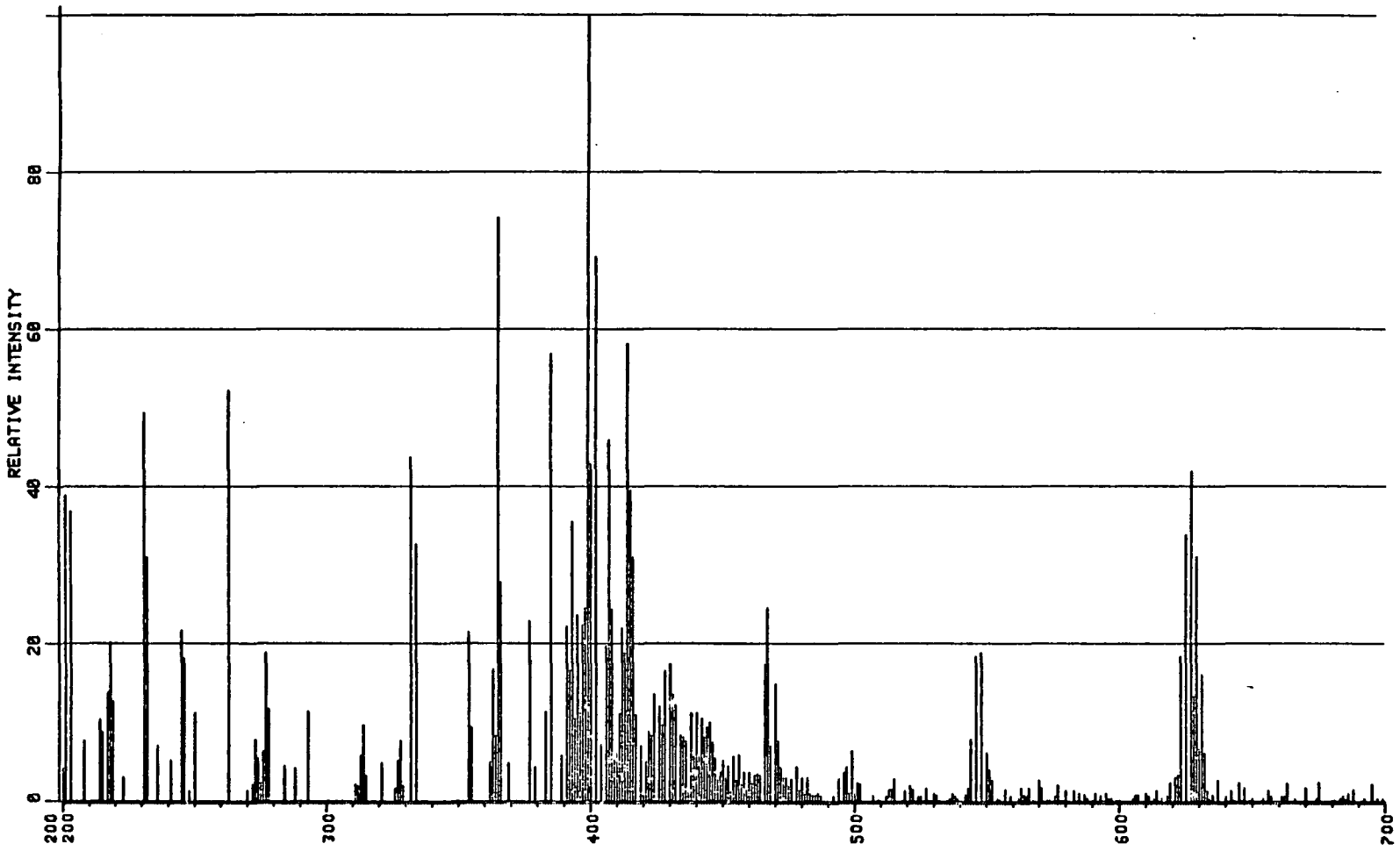
77-29 (Soil), 2.58 min, heptabromobiphenyl



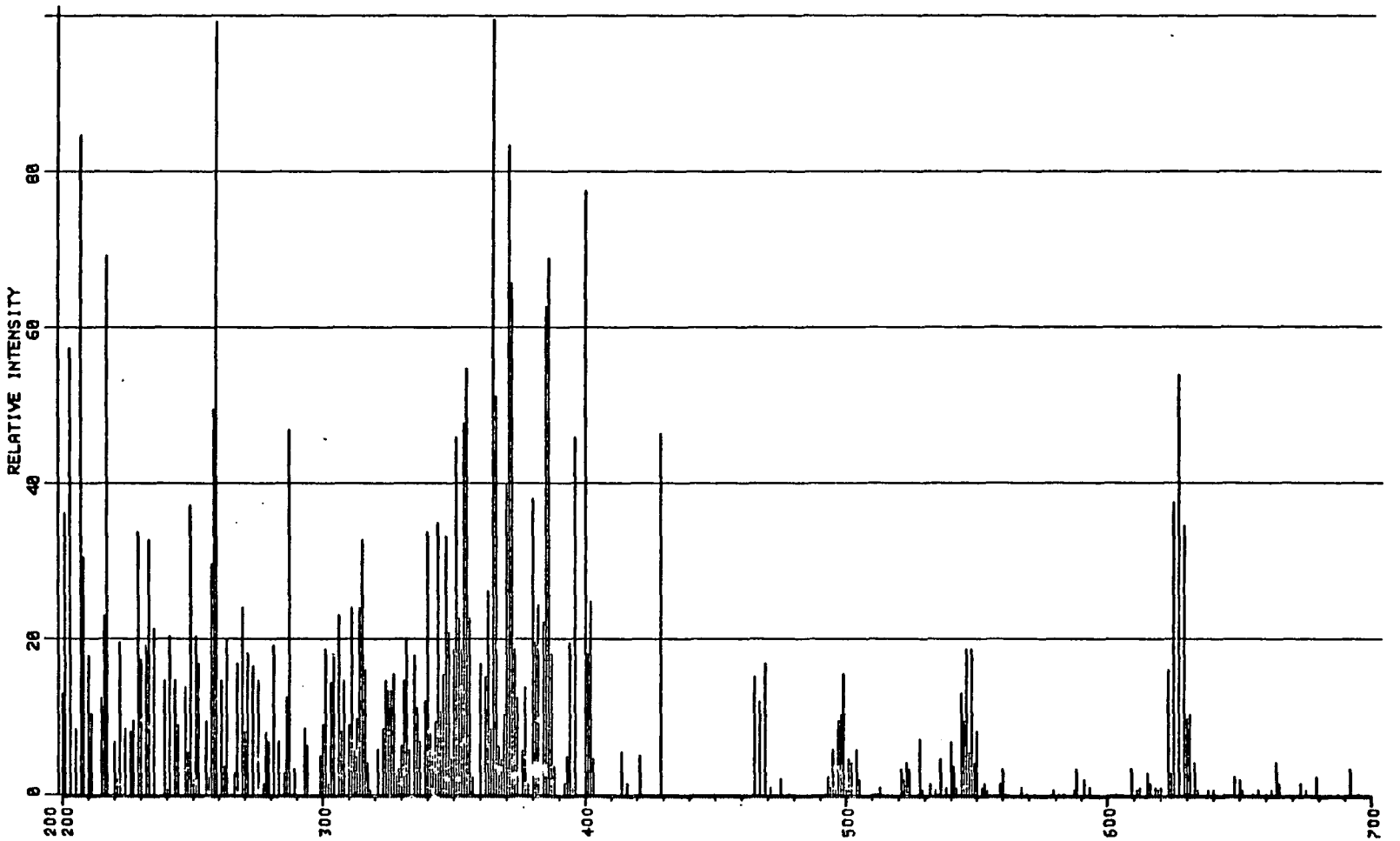
Sample 46-25, concentrated 20:1. Hexabromobiphenyl (isotope cluster centered at m/z 628) co-elutes to some extent with the octachloronaphthalene used as a standard.



Sample 46-13, concentrated 50:1. Hexabromobiphenyl (isotope cluster barely visible centered at m/z 628) can just be seen at this level.



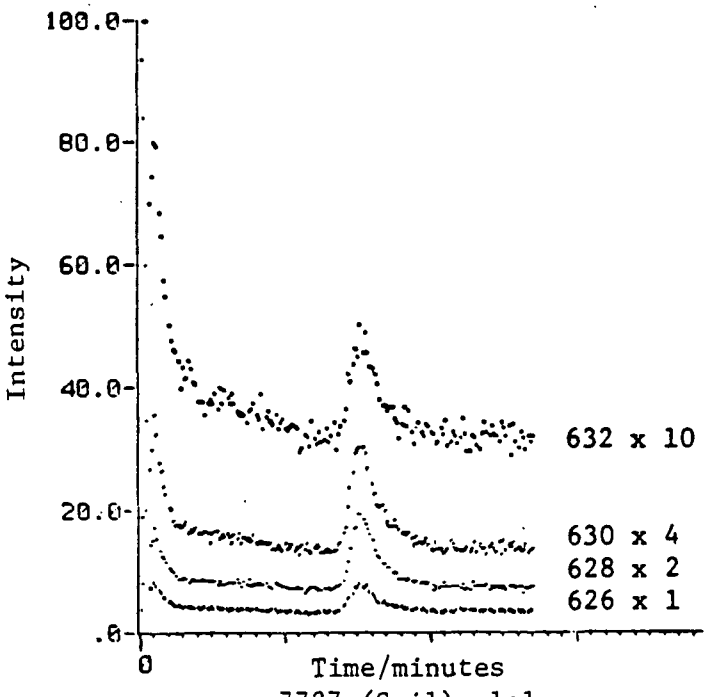
Sample 46-25, concentrated 20:1. The hexabromobiphenyl isotope cluster is visible.



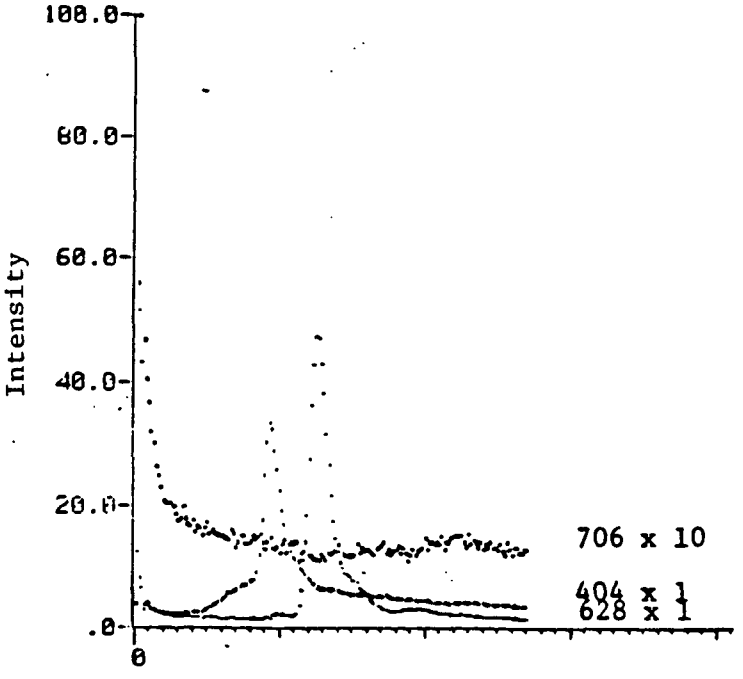
Sample 46-16, unconcentrated. The hexabromobiphenyl isotope cluster is evident.

APPENDIX F

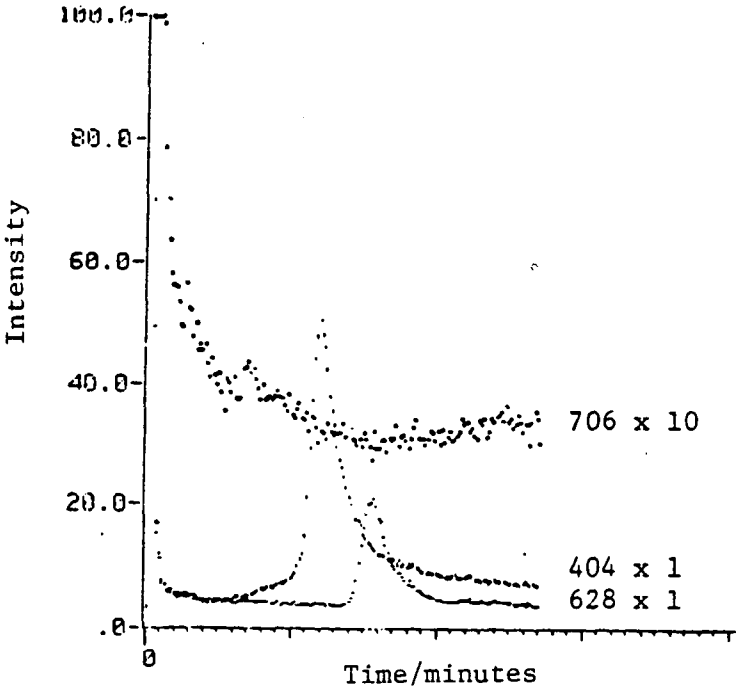
REPRESENTATIVE SELECTED ION MONITORING TRACES



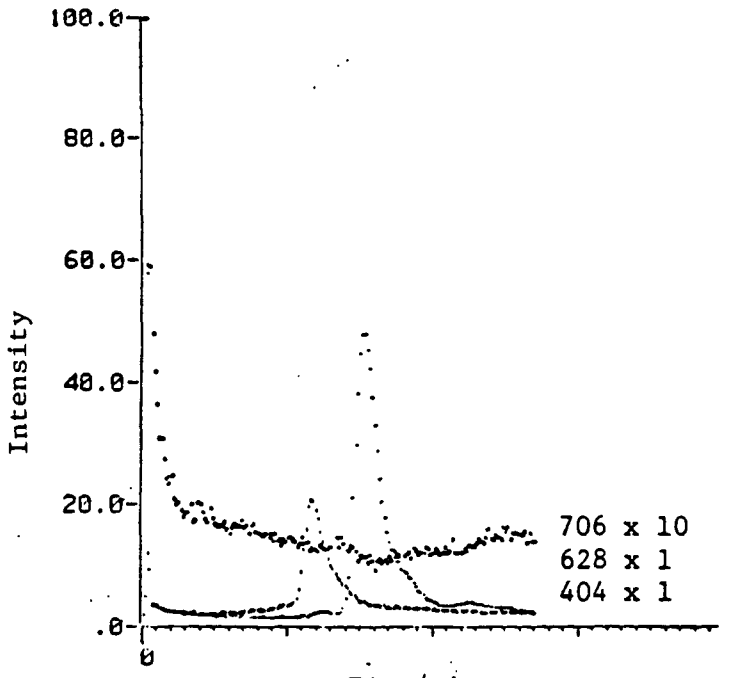
7727 (Soil), 1:1



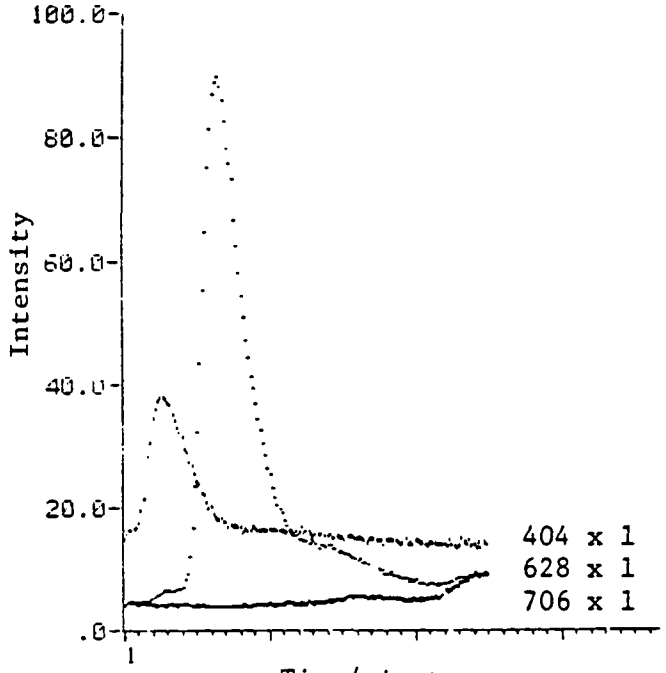
7657 (Soil), 1:1



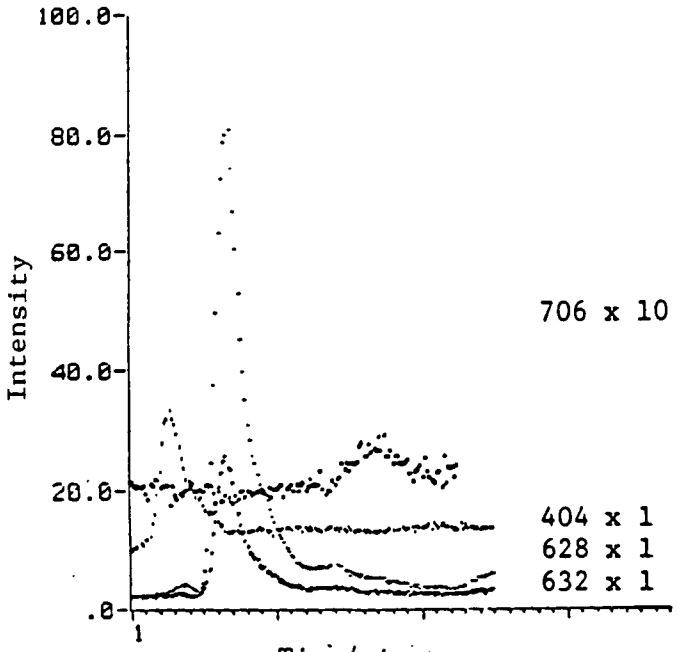
7651 (Soil), 1:1



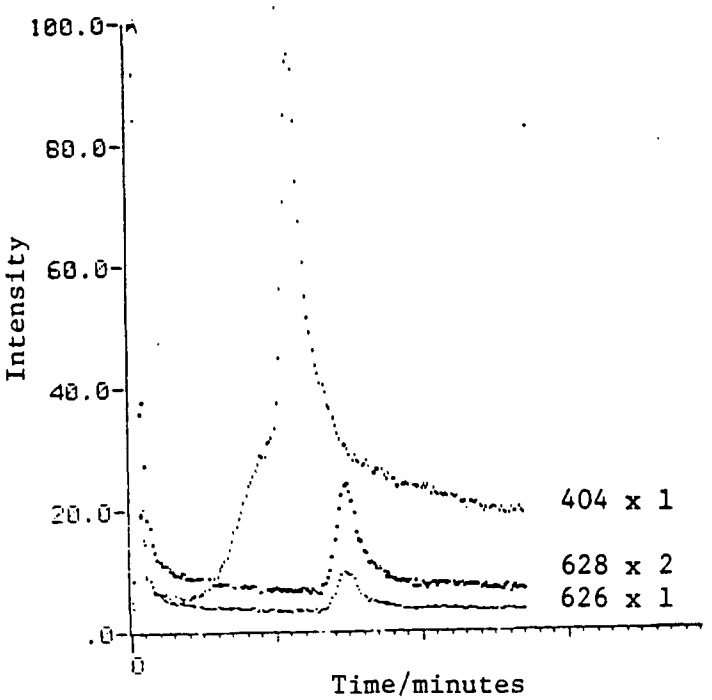
7695 (Soil), 1:1



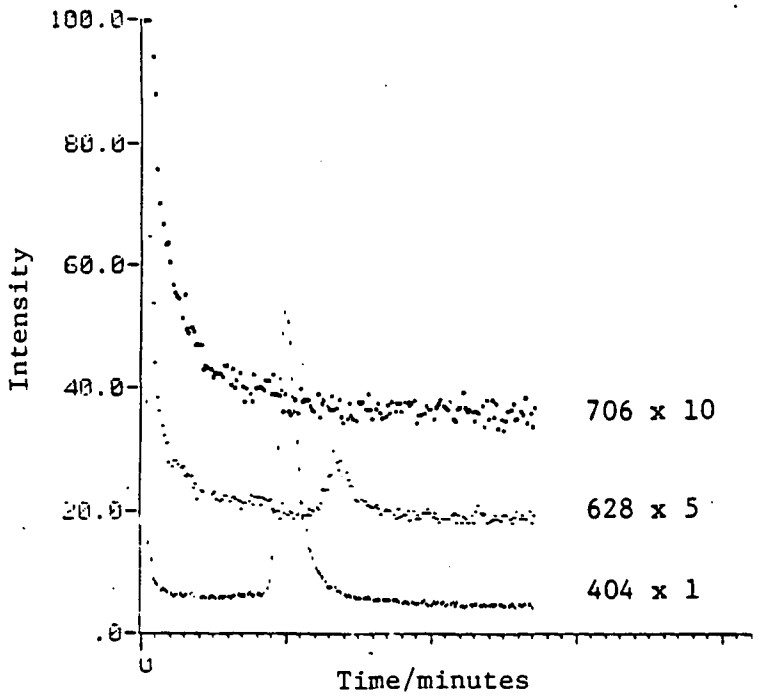
7695 (Soil), 1:1



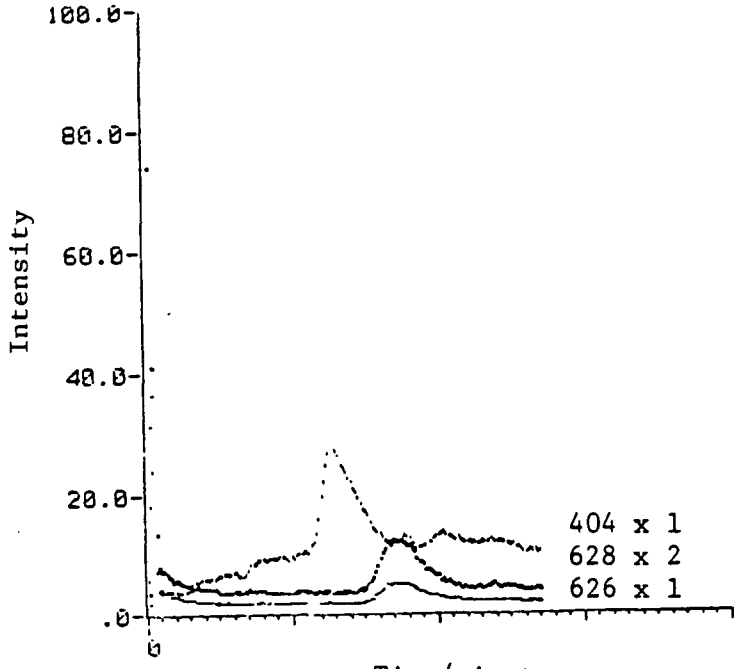
7728 (Soil), Diluted
1:100



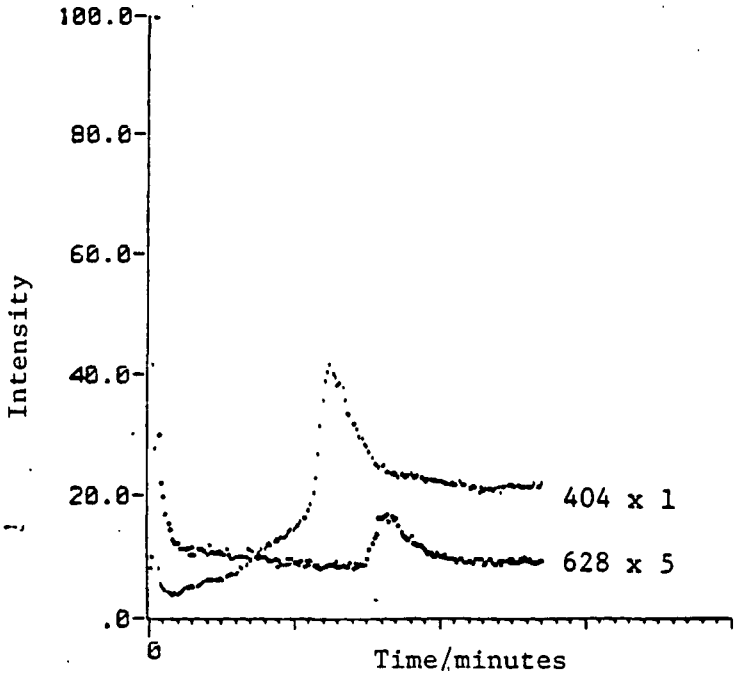
7661 (Soil), 1:1



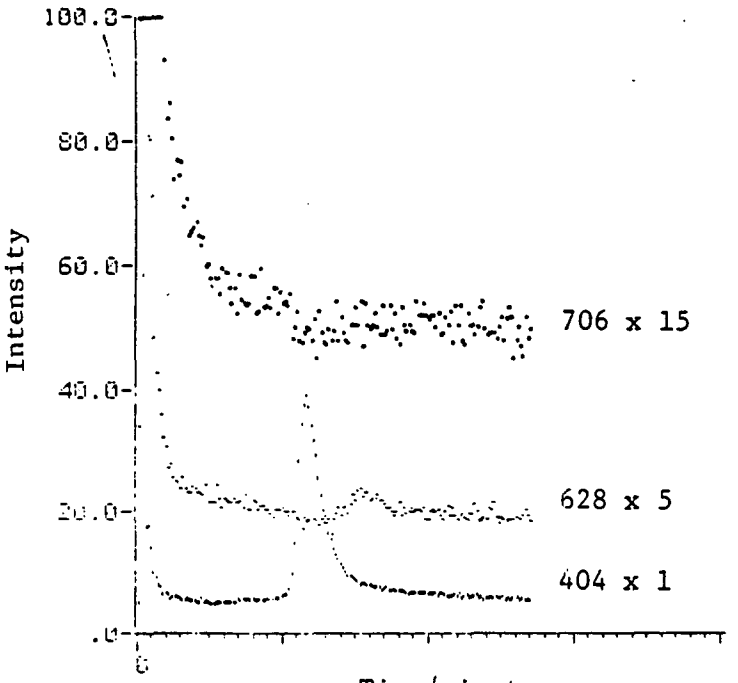
7712 (Soil), 1:1



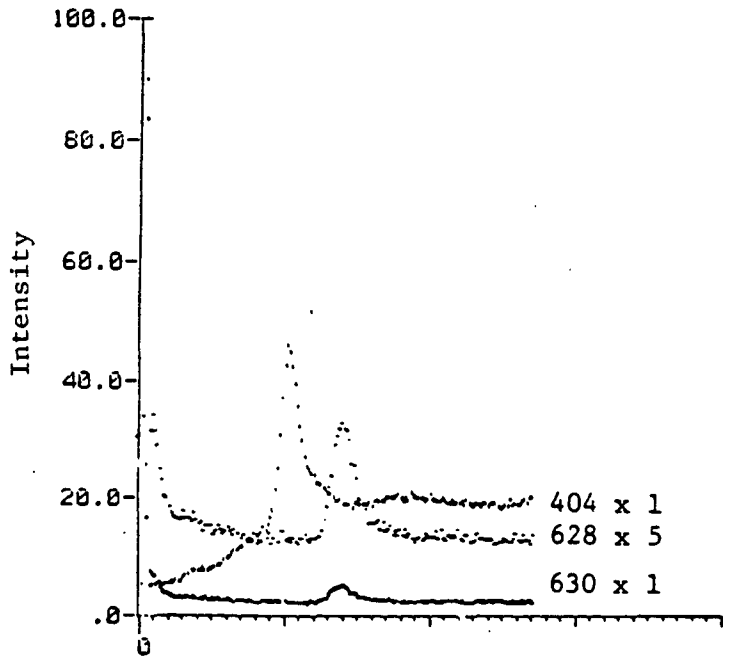
7732 (Vegetation), Concentrated
5:1



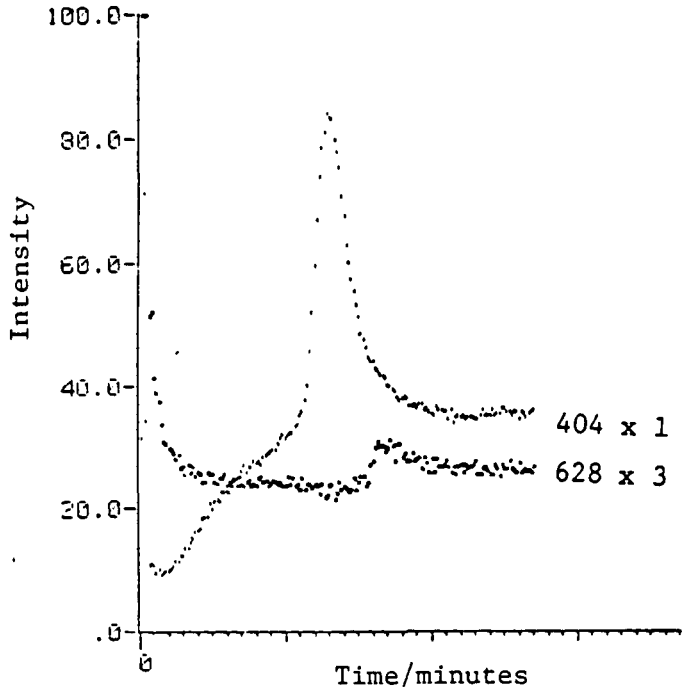
7699 (Soil), Concentrated
5:1



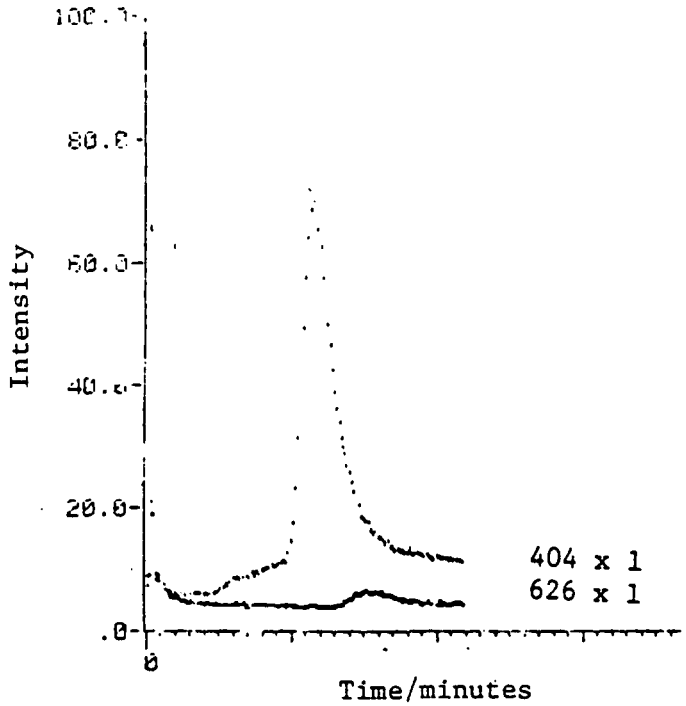
7698 (Soil), 1:1



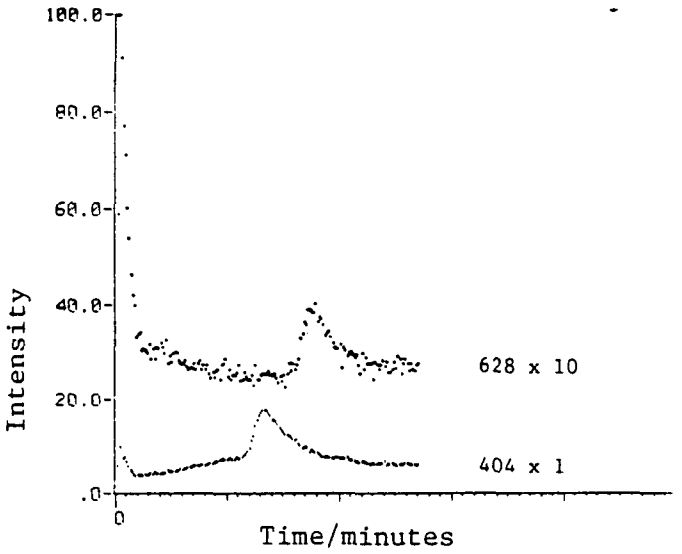
7698 (Soil), Concentrated
5:1



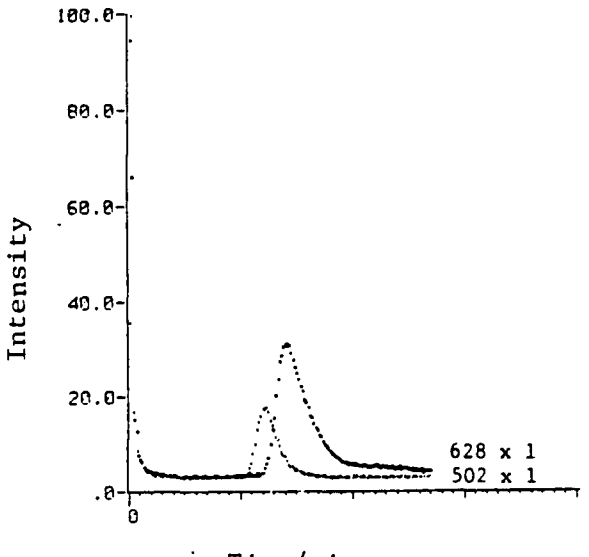
7698 (Soil), 1:1



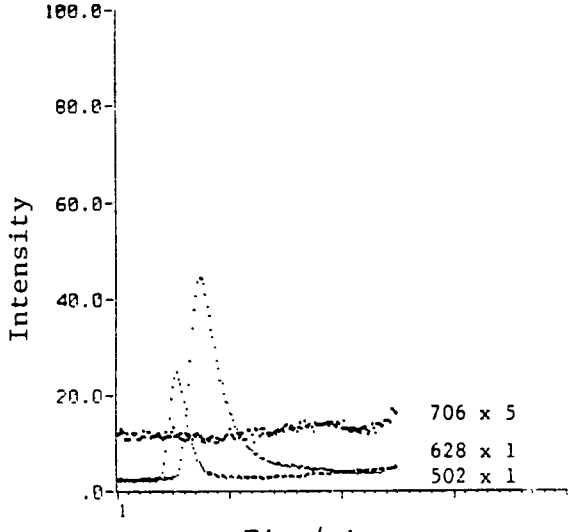
7699 (Soil), 1:1



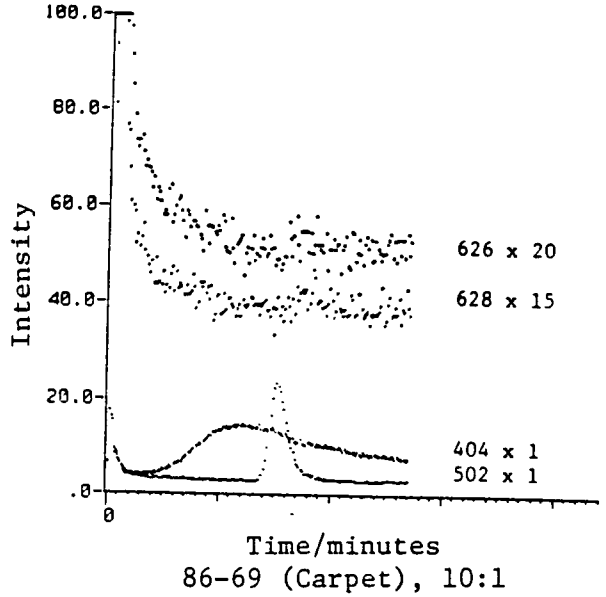
77-18 (Soil), 1:1



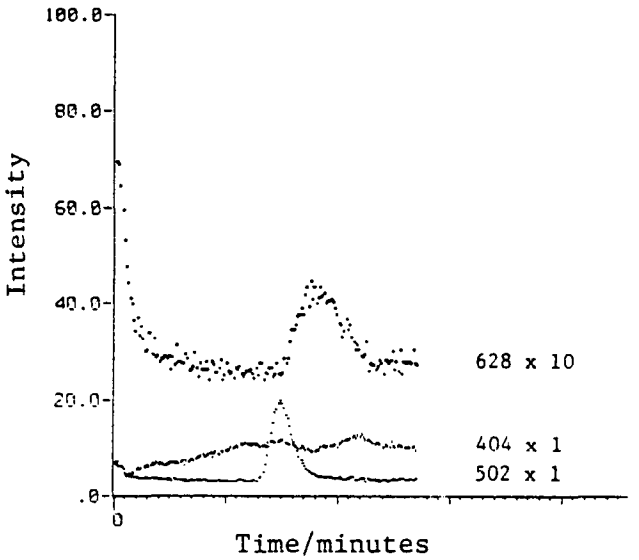
77-96 (Soil), 1:5



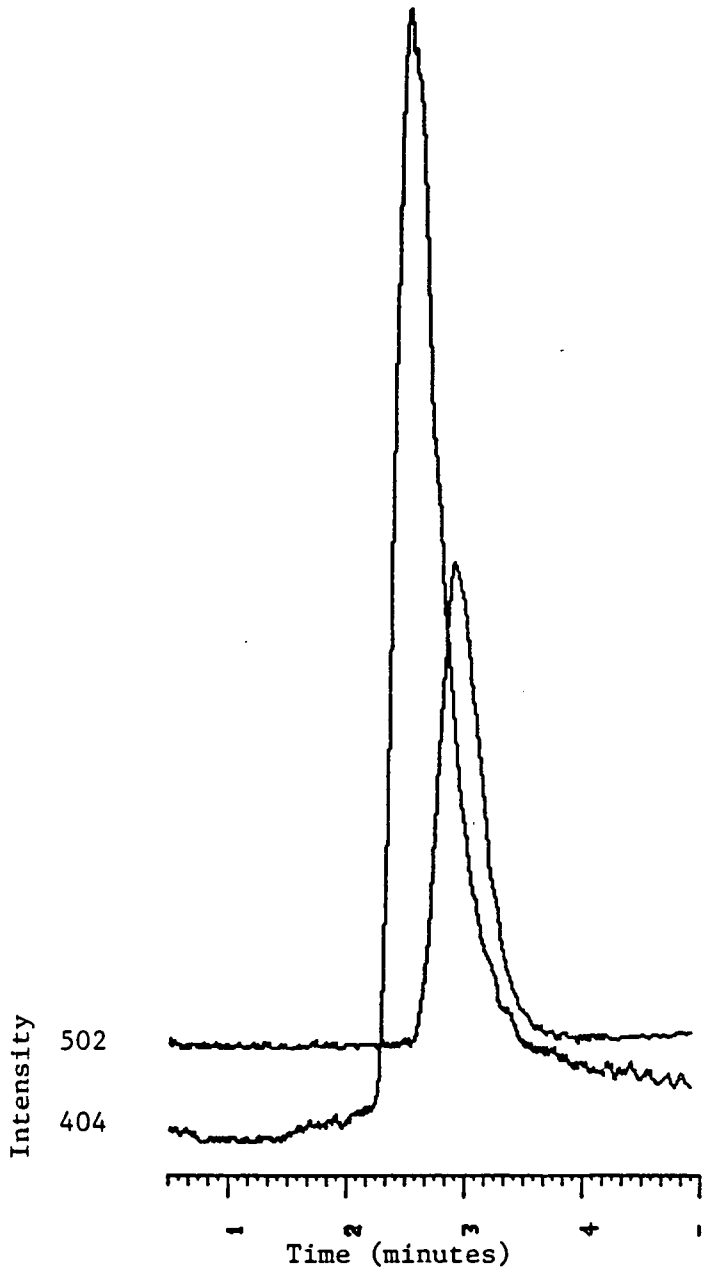
76-53 (Soil), 1:1



86-69 (Carpet), 10:1

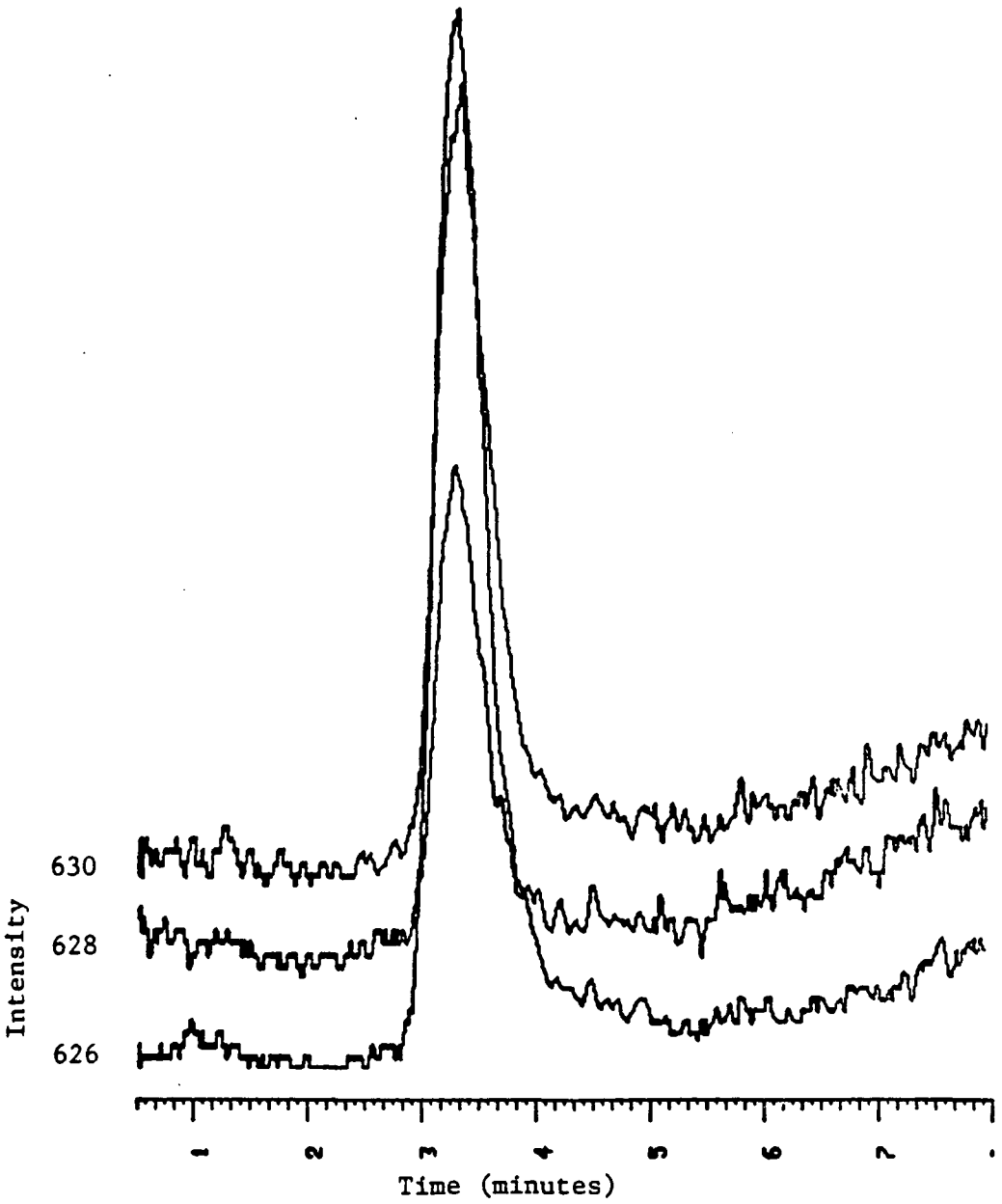


77-33 (Oleander Leaves), 33:1

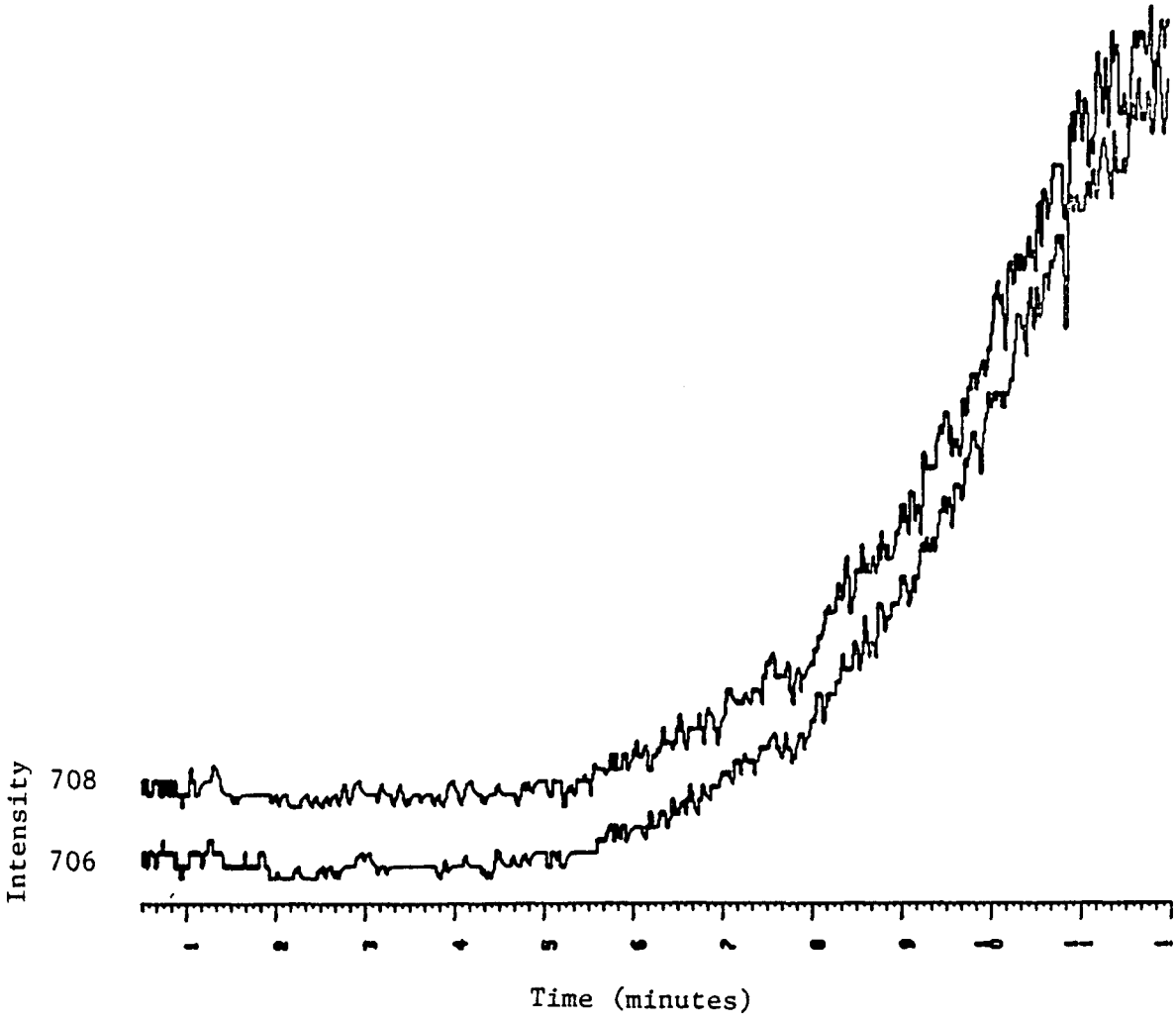


Sample: Soil Spike #2, Unconcentrated

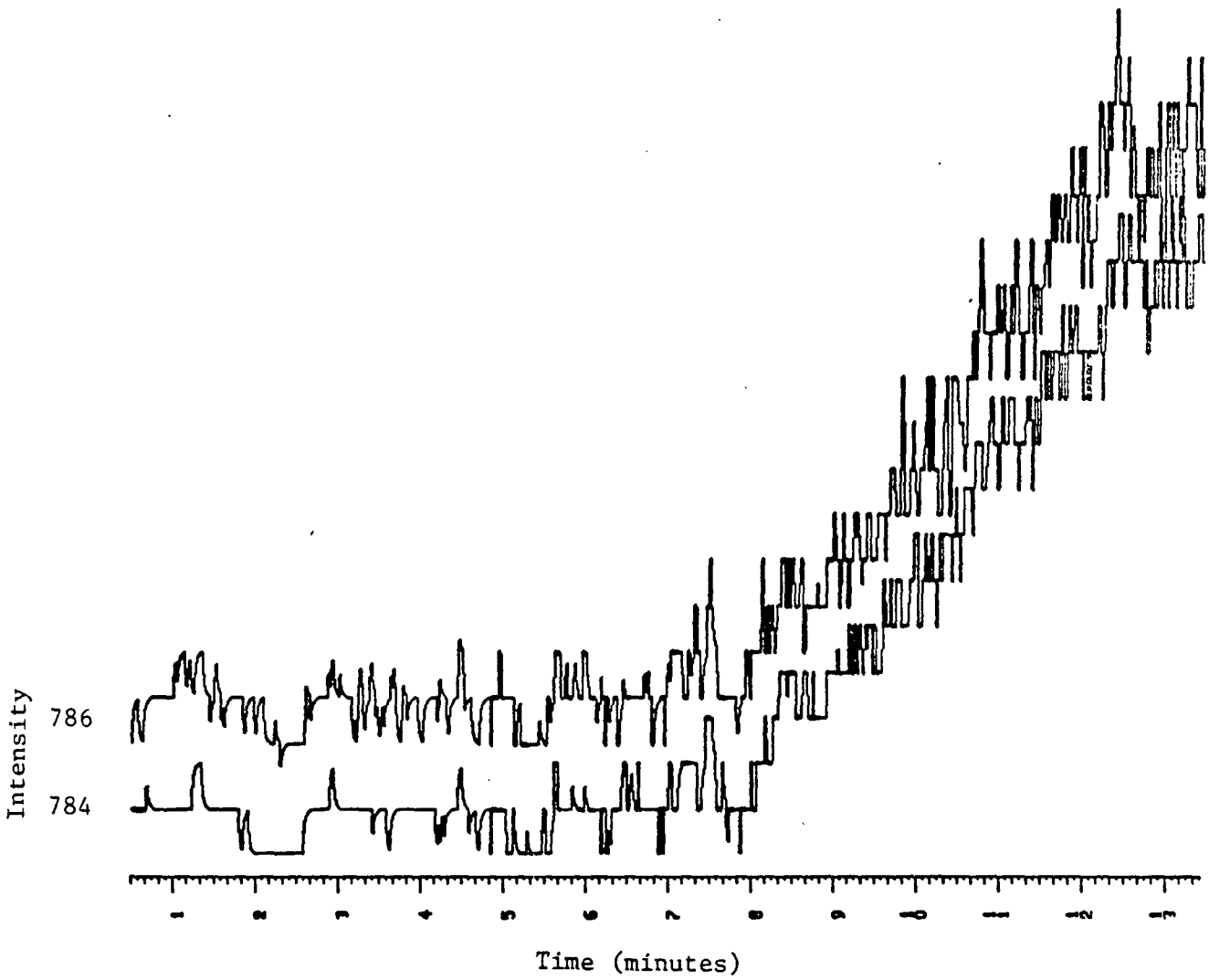
Standards: octachloronaphthalene (m/z 404) and decachlorobiphenyl (m/z 502).



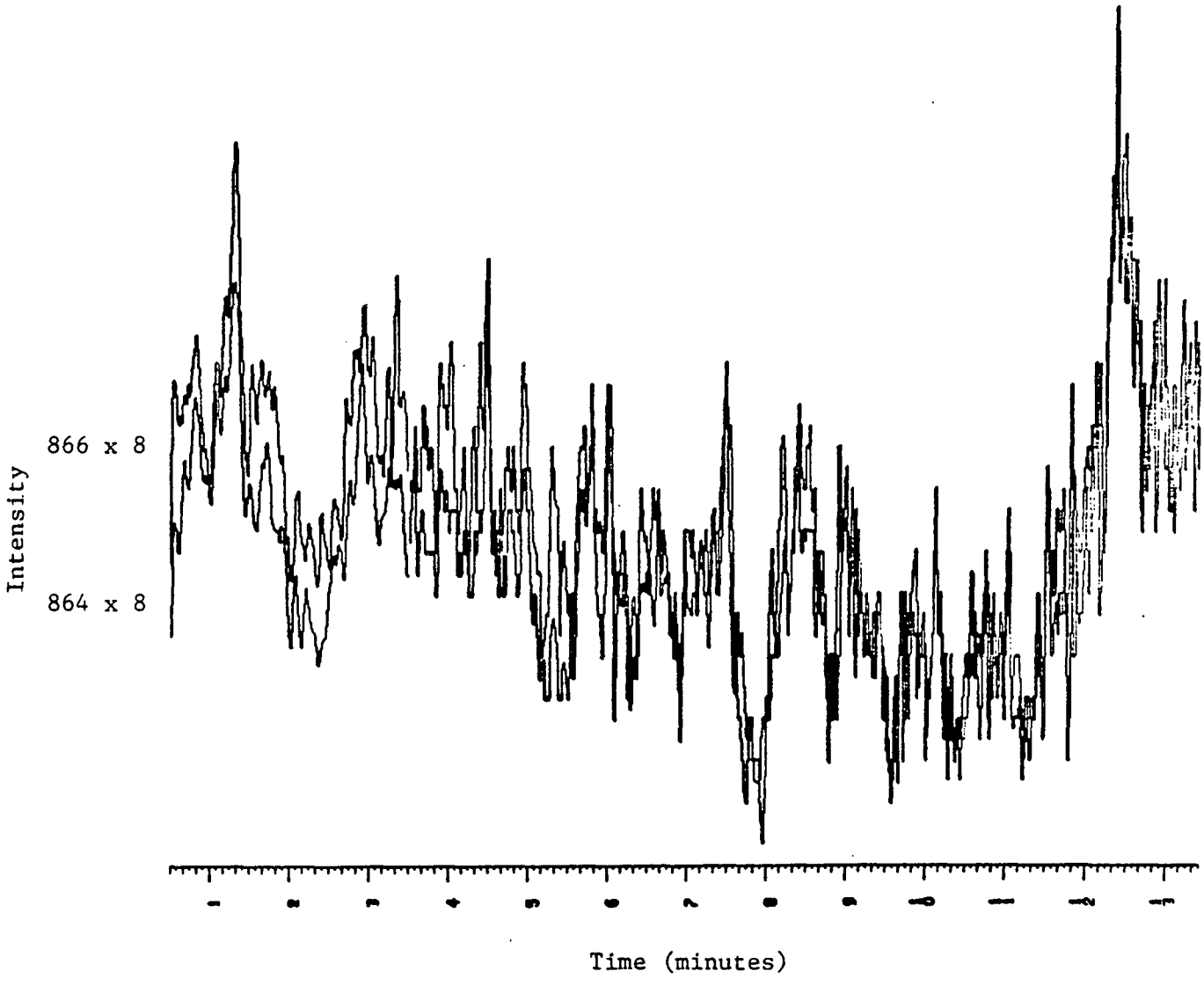
Ions monitored for hexabromobiphenyl, observed in Soil Spike #2.



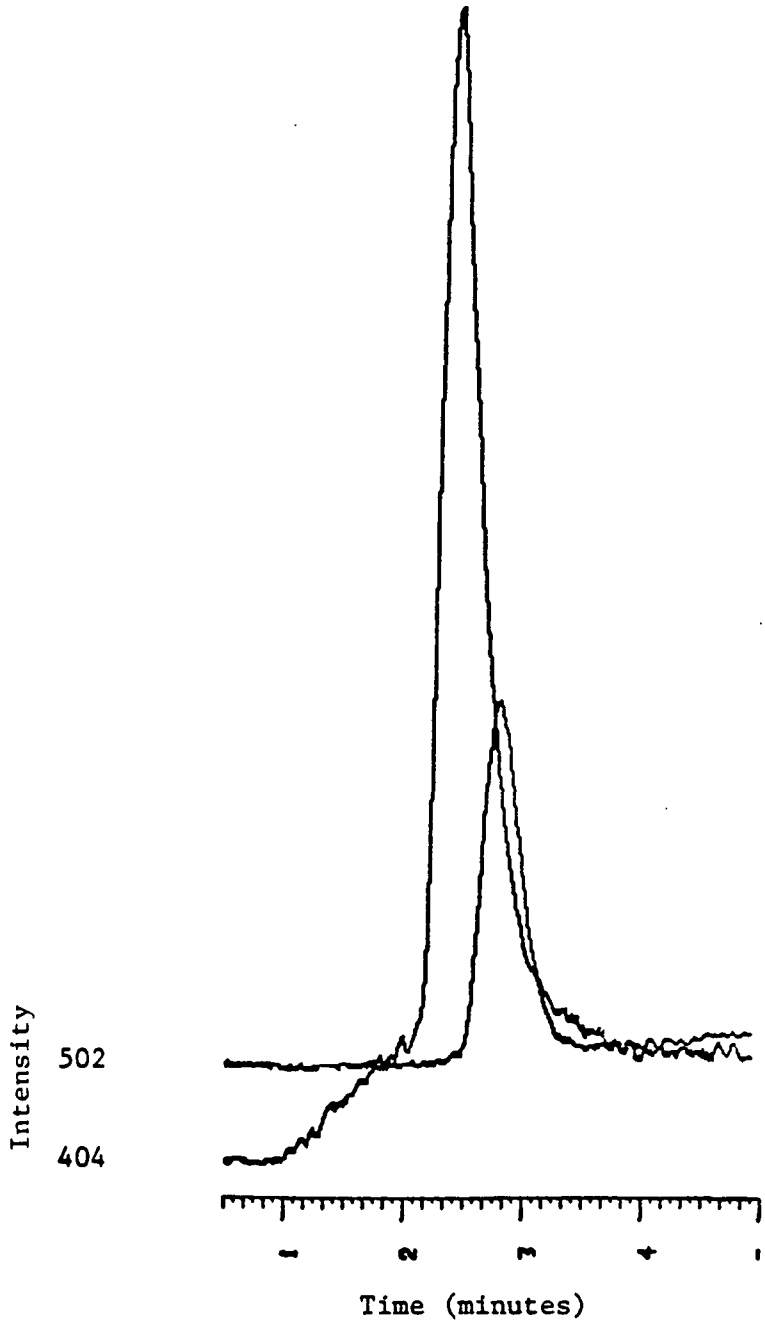
Ions monitored for heptabromobiphenyl. Not observed in Soil Spike #2.



Ions monitored for octabromobiphenyl. Not observed in Soil Spike #2.

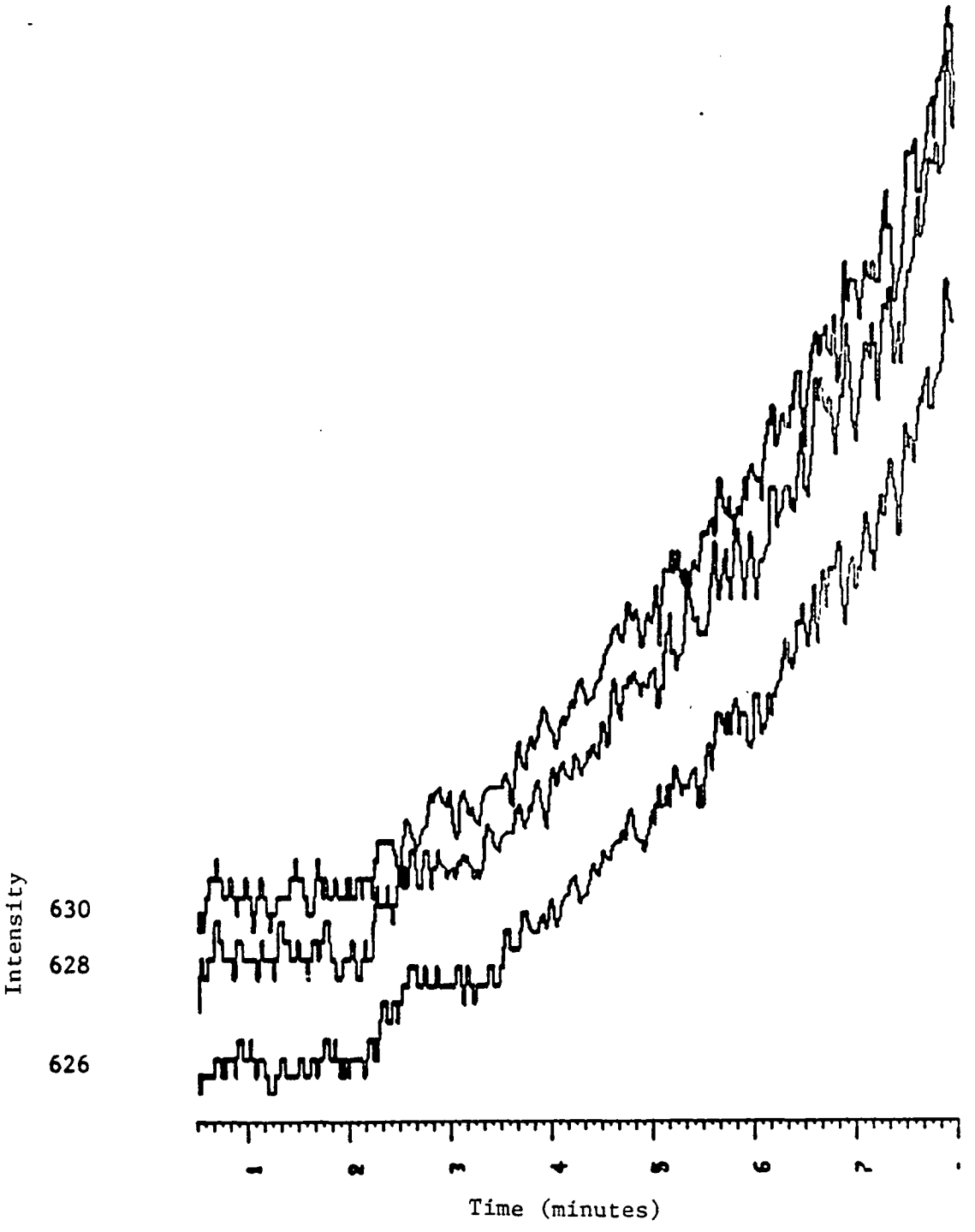


Ions monitored for nonabromobiphenyl. Not observed in Soil Spike #2, even amplified by a factor of 8 (note enhancement of noise).

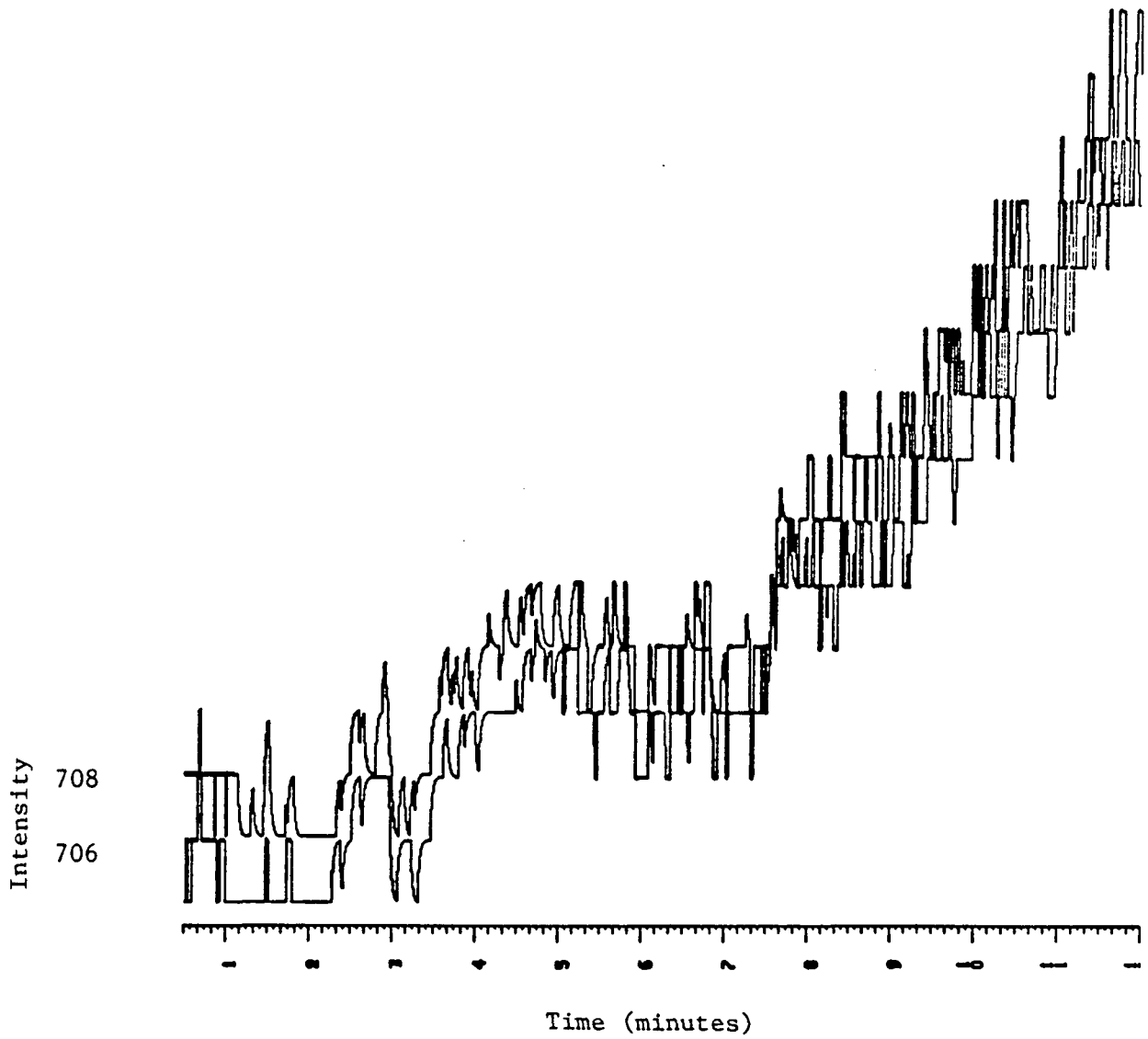


Sample: Soil Blank #1, Unconcentrated

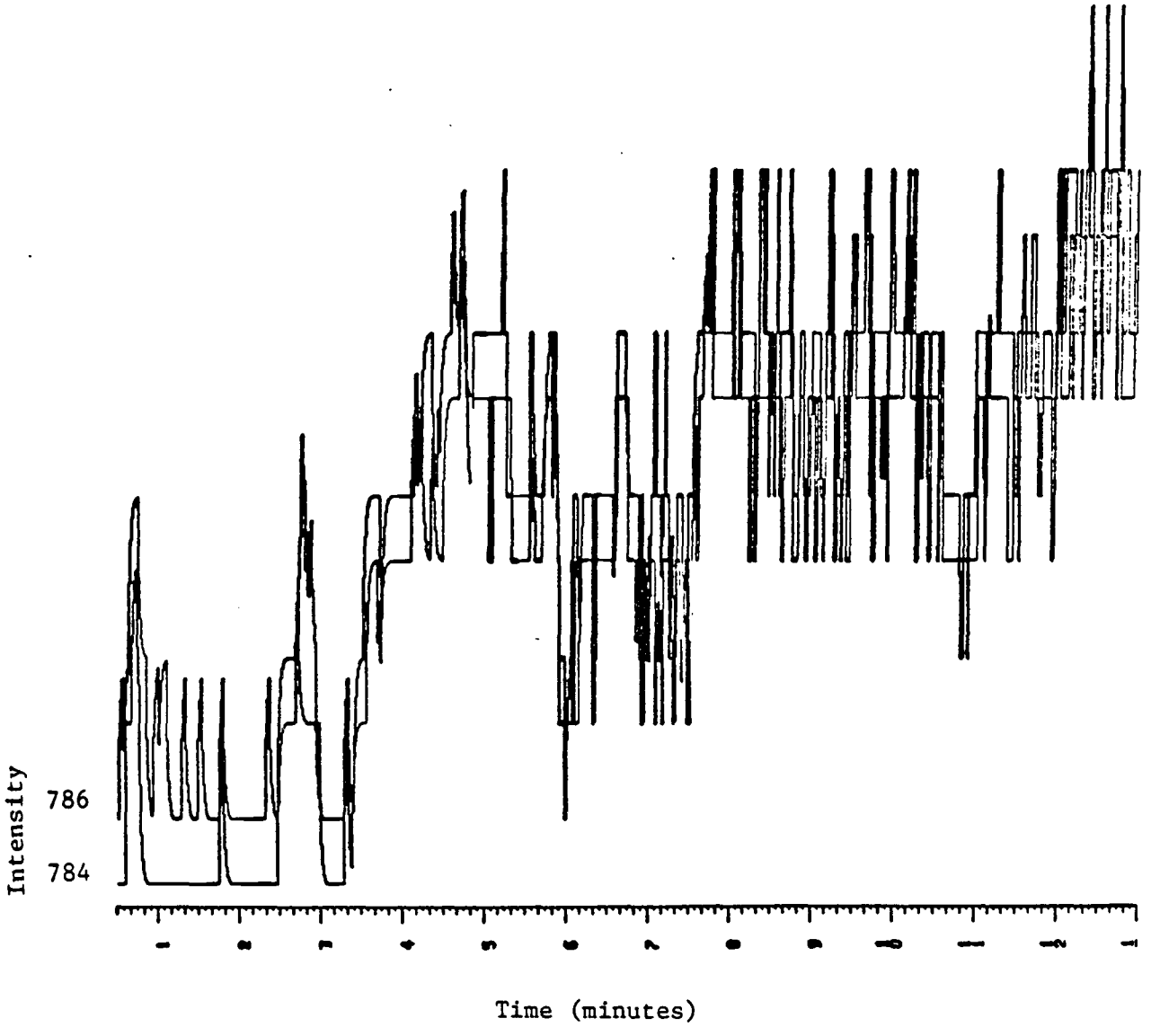
Standards: octachloronaphthalene (m/z 404) and decachlorobiphenyl (m/z 502).



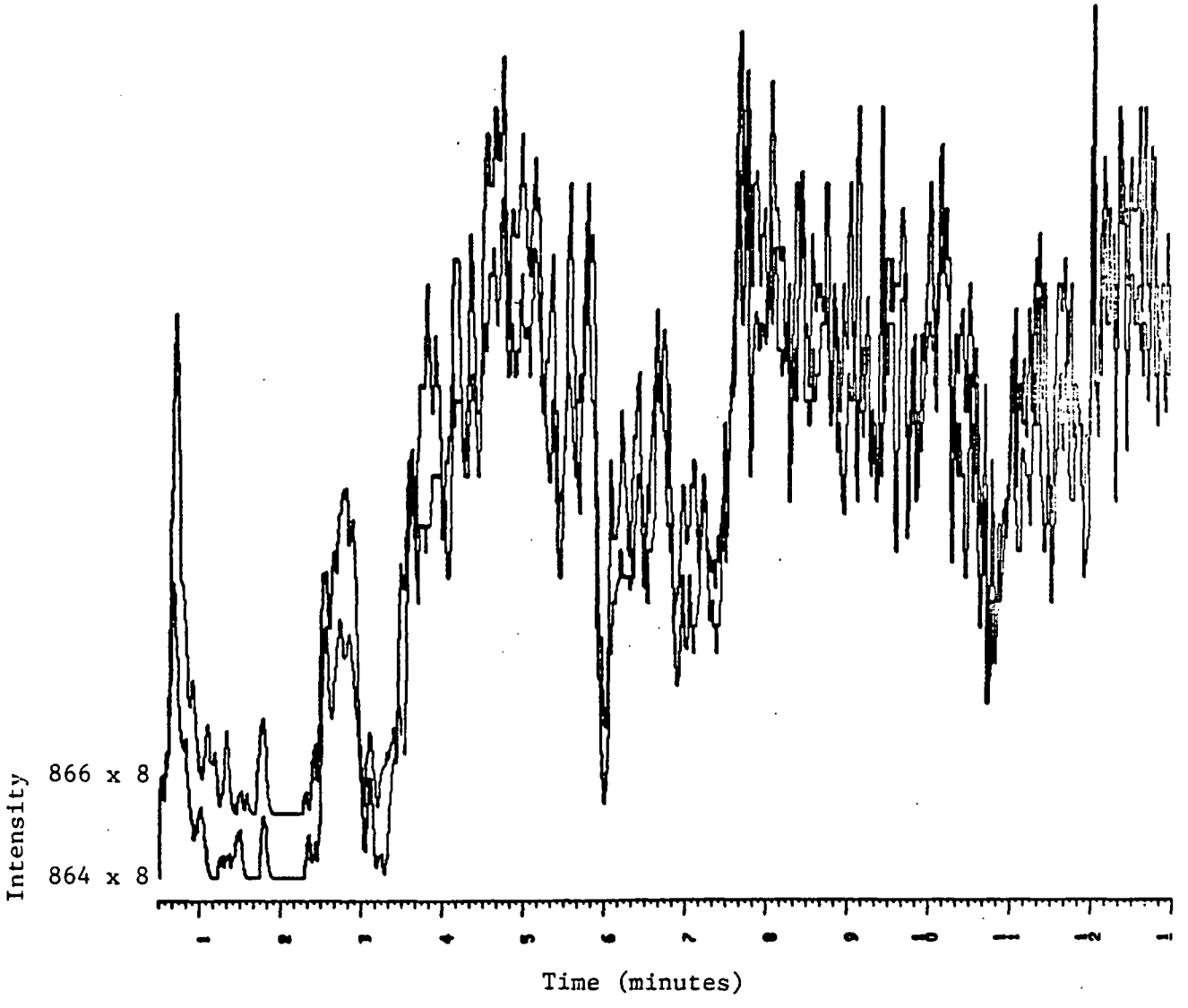
Ions monitored for hexabromobiphenyl.
Not observed in Soil Blank #1.



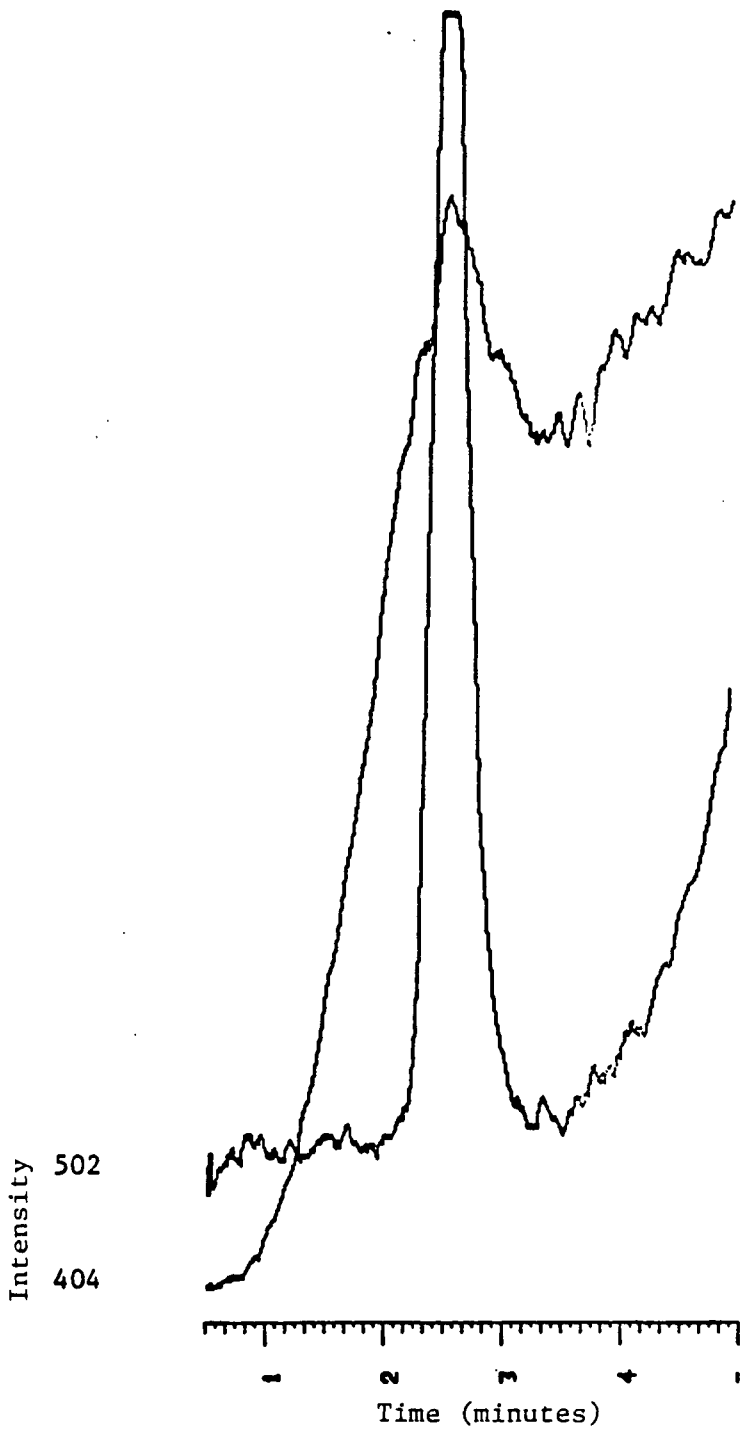
Ions monitored for heptabromobiphenyl. Not observed in Soil Blank #1.



Ions monitored for octabromobiphenyl. Not observed in Soil Blank #1.

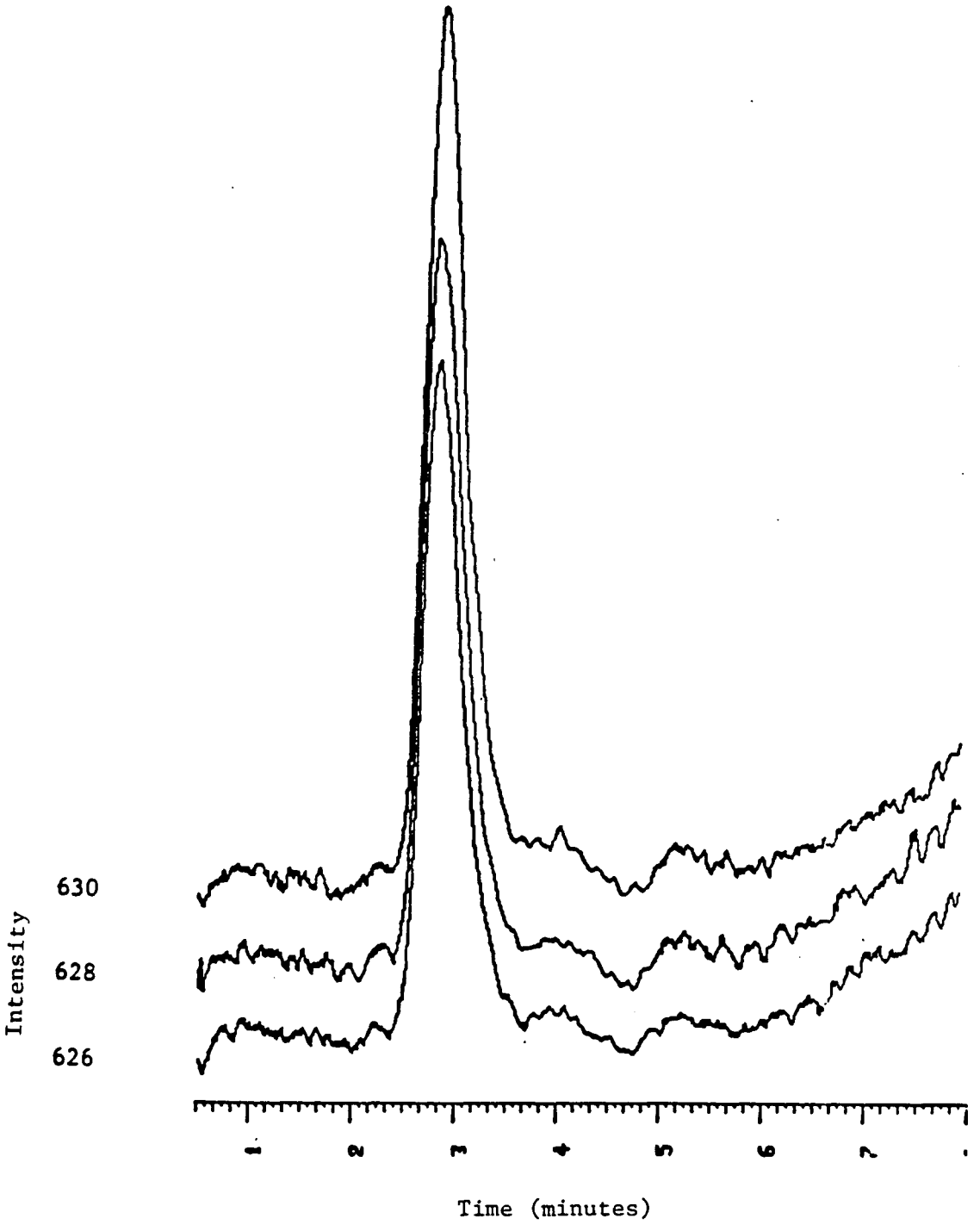


Ions monitored for nonabromobiphenyl. Not observed in Soil Blank #1.

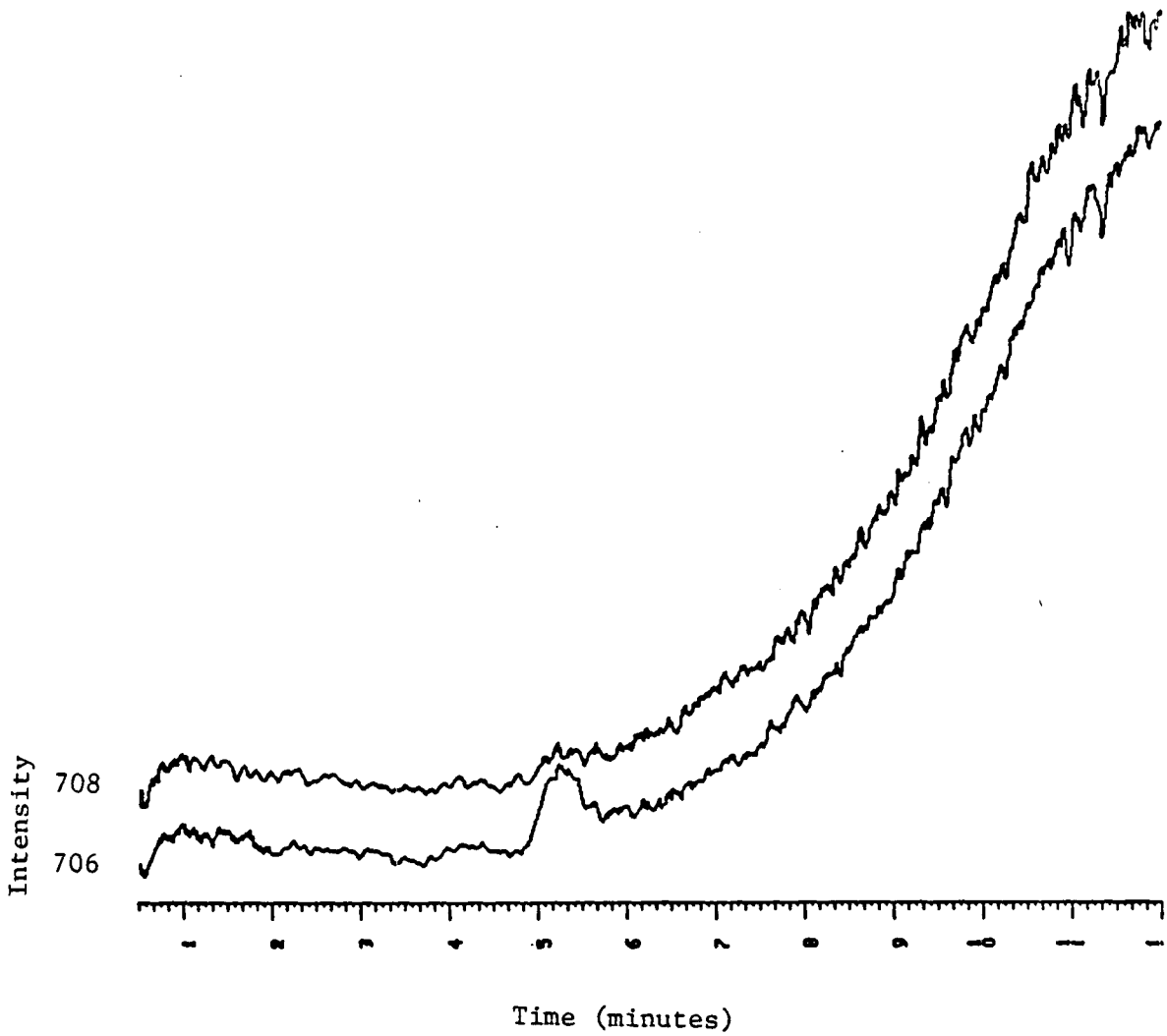


Sample: 46-02, Unconcentrated

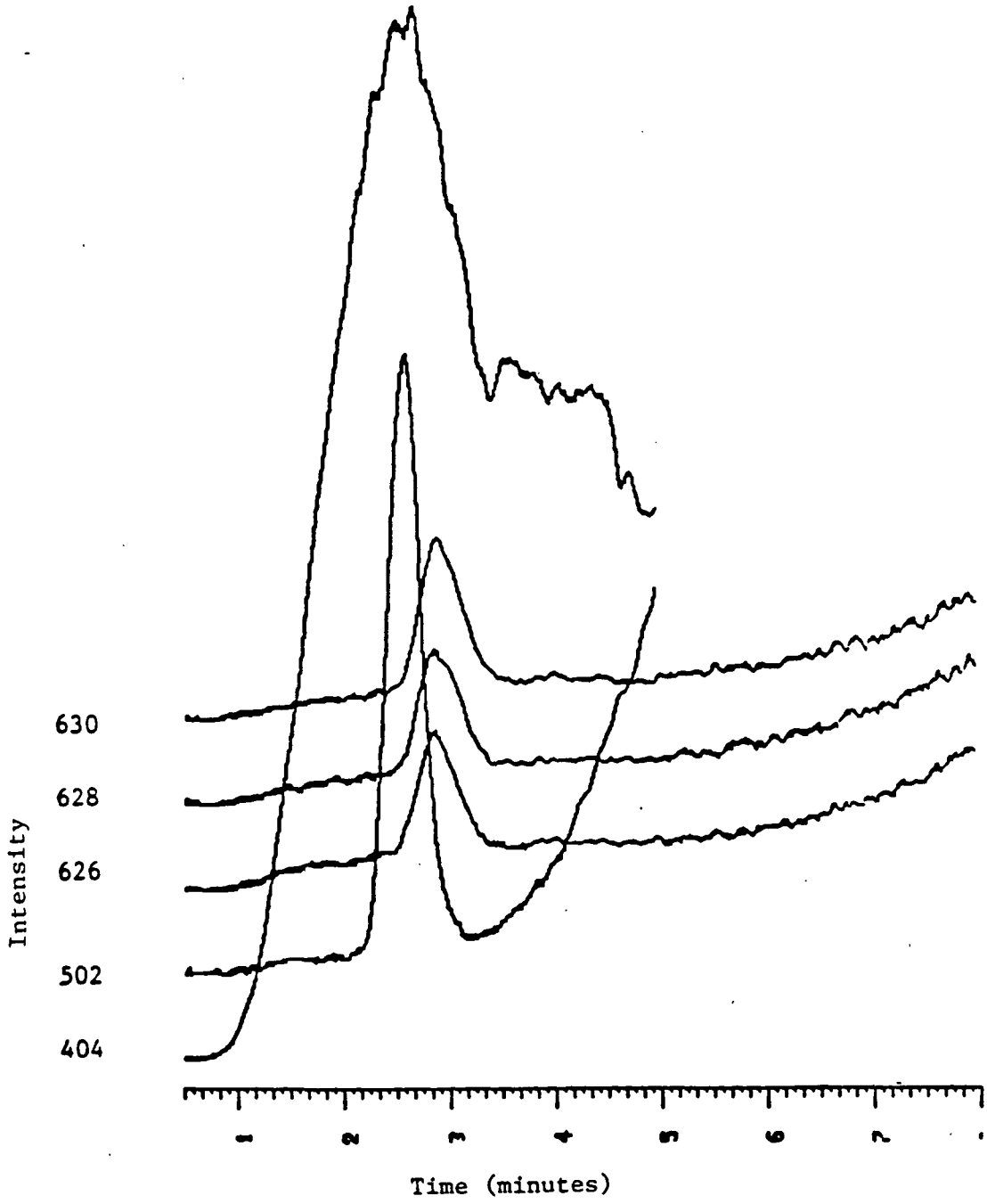
Standards: octachloronaphthalene (m/z 404) and decachlorobiphenyl (m/z 502). Note that while there is interference in the 404 channel, the 502 channel is clear.



Ions monitored for hexabromobiphenyl, present in Sample 46-02.



Ions monitored for heptabromobiphenyl, present in trace quantities in Sample 46-02.

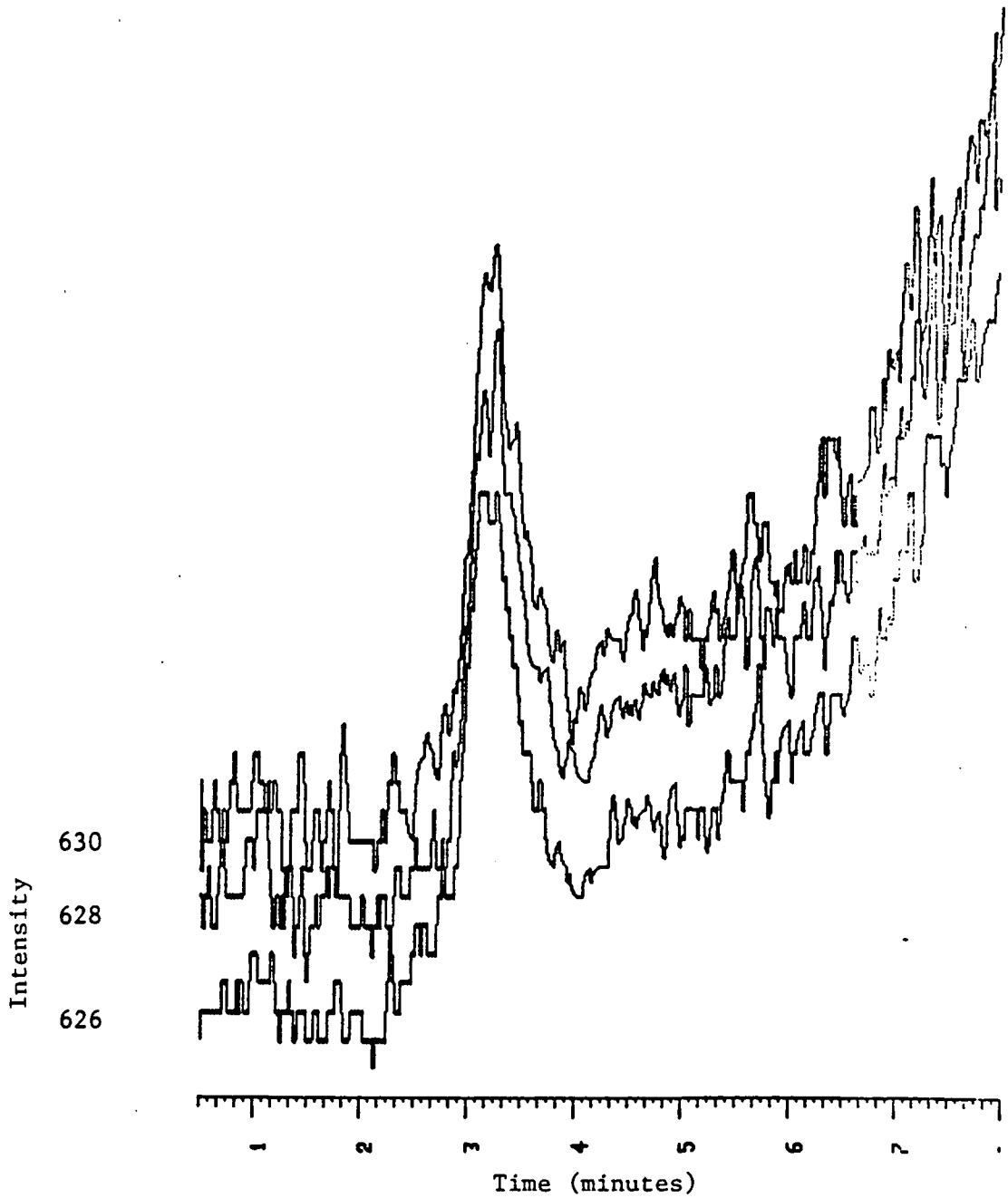


Sample: 46-12, Concentrated 50:1

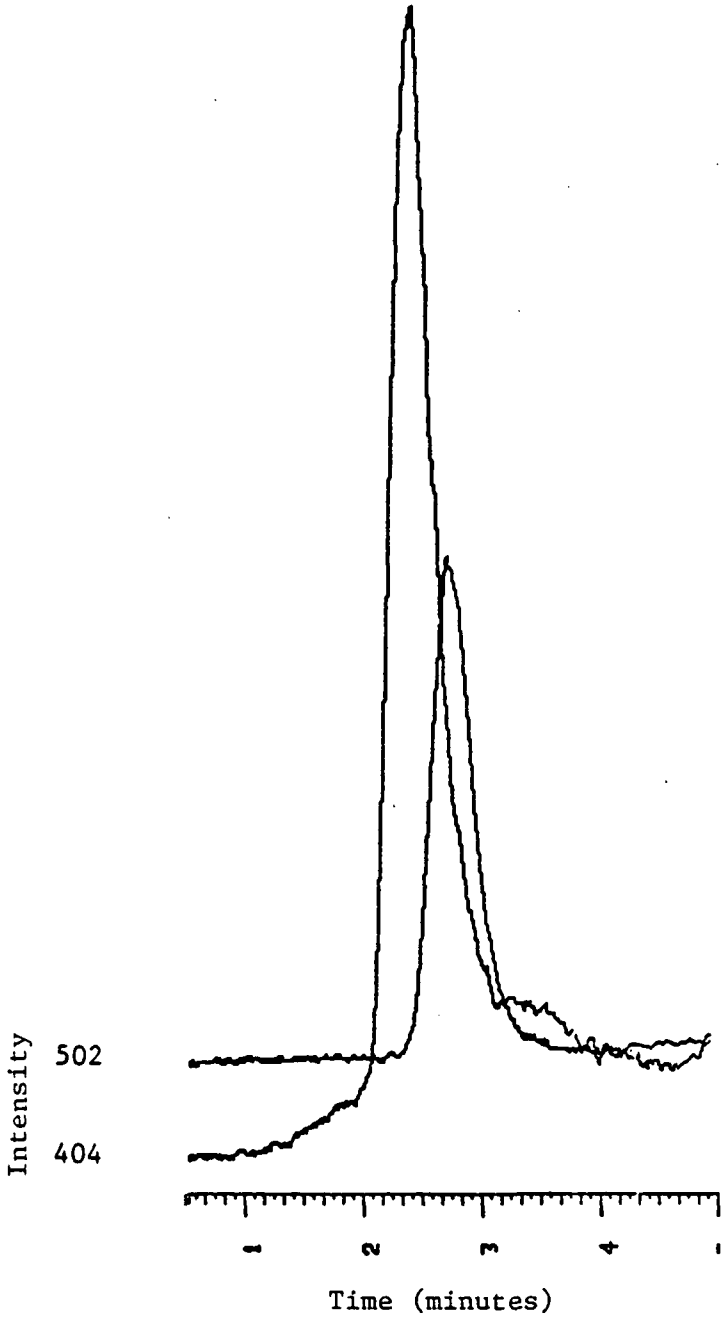
Standards: octachloronaphthalene (m/z 404, note interference) and decachlorobiphenyl (m/z 502, clear channel). Ions monitored for hexabromobiphenyl, present in this sample.

Sample: 46-25, Unconcentrated

Standards: octachloronapthalene (m/z 404)
and decachlorobiphenyl (m/z 502).

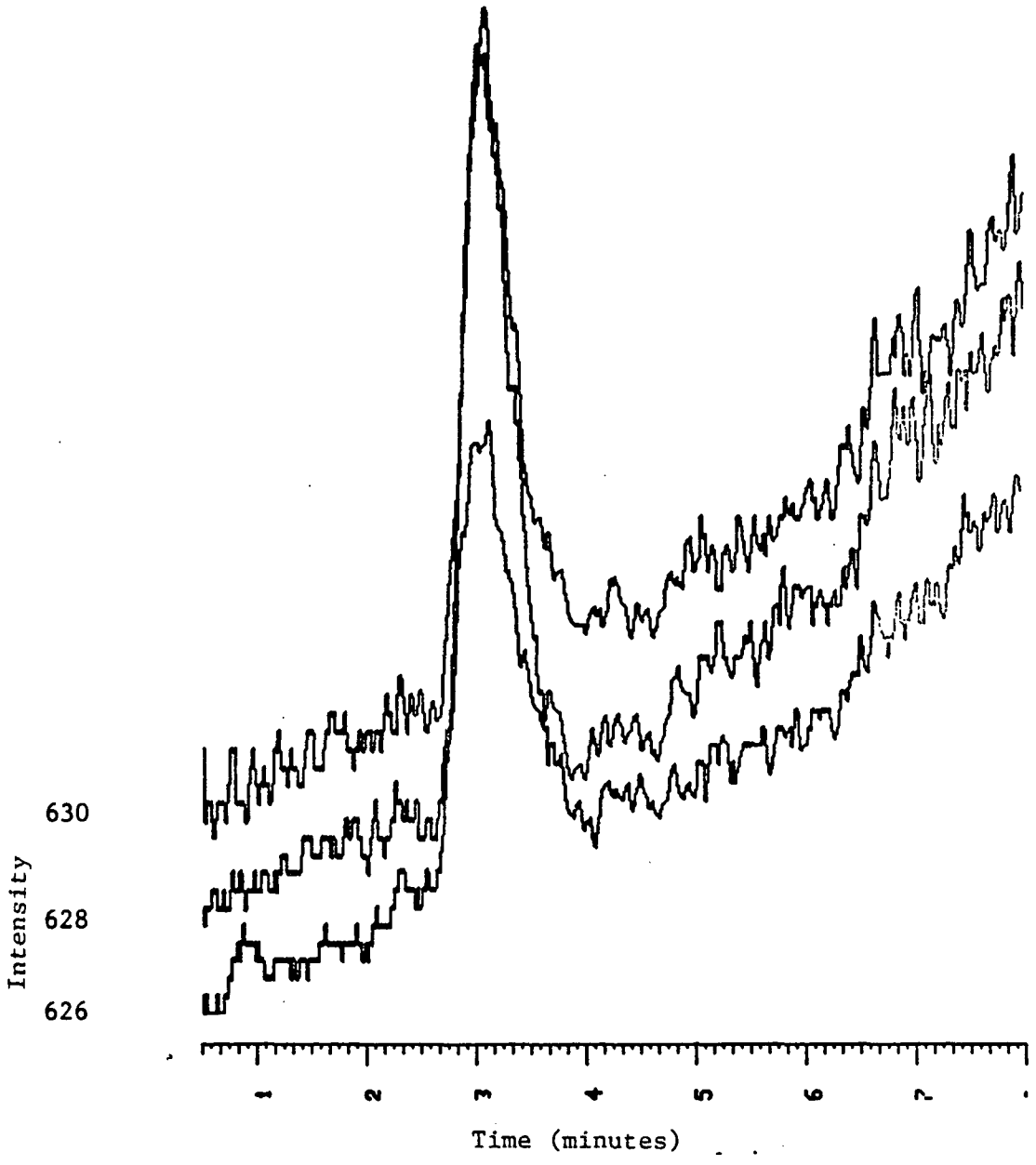


Ions monitored for hexabromobiphenyl, present in Sample 46-25.

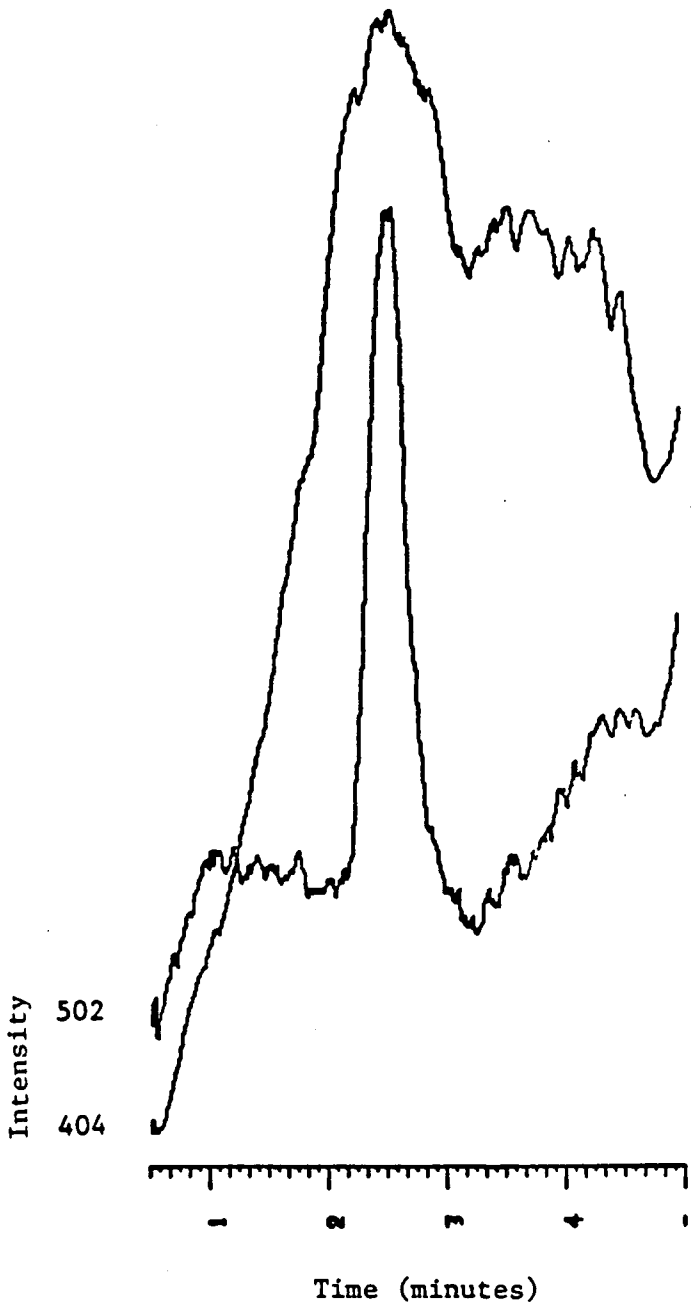


Sample: 46-26, Unconcentrated

Standards: octachloronaphthalene (m/z 404) and decachlorobiphenyl (m/z 502).

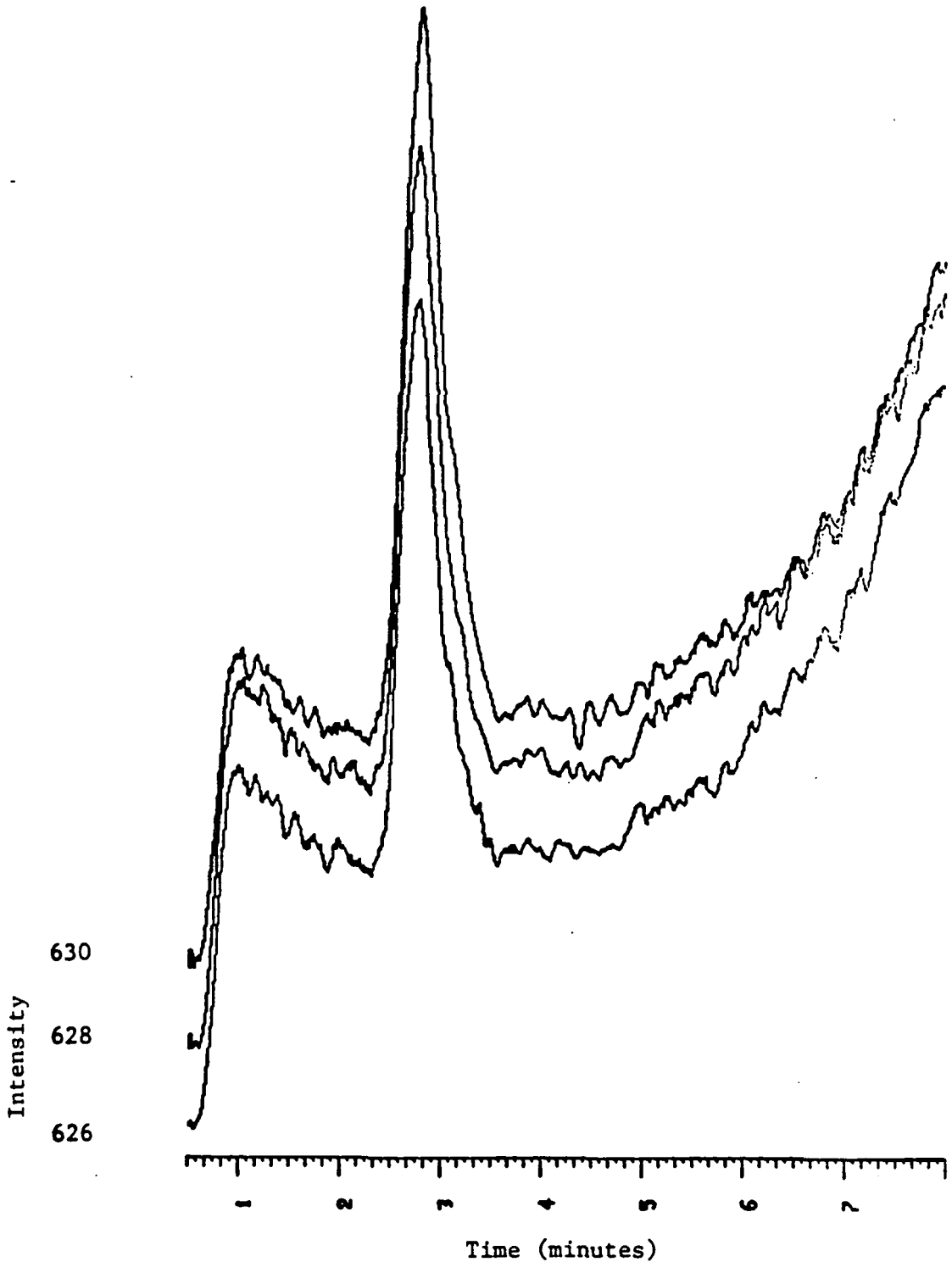


Ions monitored for hexabromobiphenyl, present in Sample 46-26.



Sample: 46-29, Concentrated 50:1

Standards: octachloronaphthalene (m/z 404, note interference) and decachlorobiphenyl (m/z 502, clear channel).



Ions monitored for hexabromobiphenyl, present in Sample 46-29.

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO. EPA-560/13-79-001	2.	3. RECIPIENT'S ACCESSION NO.
4. TITLE AND SUBTITLE Analysis for Polybrominated Biphenyls (PBBs) in Environmental Samples		5. REPORT DATE March, 1979
		6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) Charles L. Stratton, John J. Mousa and Joan T. Bursey		8. PERFORMING ORGANIZATION REPORT NO. Research Request #3, Final Report
9. PERFORMING ORGANIZATION NAME AND ADDRESS Environmental Science and Engineering, Inc. P. O. Box 13454 Gainesville, Florida 32604		10. PROGRAM ELEMENT NO. Task IIIB
		11. CONTRACT/GRANT NO. 68-01-3248
12. SPONSORING AGENCY NAME AND ADDRESS U.S. Environmental Protection Agency Office of Toxic Substances 401 M. Street, SW Washington, DC 20460		13. TYPE OF REPORT AND PERIOD COVERED Final 4/78-4/79
		14. SPONSORING AGENCY CODE
15. SUPPLEMENTARY NOTES		

16. ABSTRACT

Ninety-eight environmental samples consisting of fish, sediment, soils, and vegetation, were analyzed by gas chromatographic/mass spectrometry (GC/MS) techniques for hexa-, hepta-, octa-, nona-, and decabromobiphenyls. The samples were collected by EPA regional office personnel. Hexabromobiphenyl was the most prevalent species detected. This species was detected in 84 percent of the samples over the range of 0.2 to 50,000 ppb. Heptabromobiphenyl was detected in 12 percent of the samples and octabromobiphenyl in 2 percent of the samples. Nonabromobiphenyl and decabromobiphenyl were not detected.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Polybrominated biphenyls PBBs GC/MS Brominated organics		

18. DISTRIBUTION STATEMENT Release unlimited	19. SECURITY CLASS (This Report) unclassified	21. NO. OF PAGES
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