

FILE ON EDOC'S ☒ YES ☐ NO

SITE NAME _____

SITE # _____

COUNTY _____ TOWN _____

FOILABLE ☒ YES ☐ NO

SC/PSA _____ RI/FS _____

RD _____ RA _____

SM _____ OTHER _____

NAME DESCRIPTION:

Report HW 130 004-1984-10-01. OOC GW & Siols

Investigation Report



OCCIDENTAL CHEMICAL CORPORATION

**Special Environmental Programs
Niagara Falls, New York**

**Report of Groundwater & Soils Investigation
at
The Former Ruco Division Plantsite
Hicksville, New York**

SECTION III

Analytical

October 1984

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REFERENCES

- 1). Proposed Hicksville Plant Groundwater Study, D. R. Thielen and R. G. Badger, 4/03/83.
- 2). Parsons, F., Lage, G, Rice, R., Astvaskis, M., and Nassau, R., "Behaviour and Fate of Hazardous Organic Chemicals in Contaminated Groundwater", Report to Florida Department of Environmental Regulation, December 1982.
- 3). Wood, P. R., Parsons, F., Lang, R. F., Payan, I. L., Espinet-Tracey, S. S., and Harwin, H. J., "Pilot Plant Project for Removing Organic Substances from Drinking Water", EPA Report EPA600/2-84-009, January, 1984.
- 4). Harris, D., and Davids, H. W., "Interim Report #2, Vinyl Chloride Contamination of Groundwater, North Bay Shore, New York", Suffolk County Department of Health Services, November, 1983.

CONCLUSIONS

Soil - Well Sites

With the exception of sites E and F, soil from the well sites did not show the presence of organic or inorganic compounds at significant levels.

At site E, tetrachloroethylene was found at a level of 240 ppm in a sample near the surface with much smaller amounts in deeper samples. Very low concentrations of Aroclor 1248 (less than 1 ppm) were also detected in some of these soils. At site F tetrachloroethylene only was found in much smaller concentrations (less than 2 ppm) in samples at 36 and 46 feet below grade level. Sites B and C each had one sample (the uppermost one) with less than 0.4 ppm of tetrachloroethylene. Sites C and D had very low concentrations of Aroclor 1248 (less than 1 ppm) in three of the seven samples analyzed.

Groundwater

Water samples from twelve locations were analyzed for a variety of parameters. None of the organic compounds analyzed for were found in six of these locations. Of the remaining six locations, two had only one compound, three had two compounds and one had three compounds. Only four values were over 50 ppb and none were over 200 ppb. Vinyl chloride was found at concentrations of 7, 140 and 50 ppb at sites E-1, F-1 and F-2 respectively. TOC and COD values at sites C-1, D-1, E-1, E-2, F-1 and F-2 indicate the possible presence of additional organic or oxidizable compounds at these sites. There is no consistent pattern of groundwater contamination by organics found in the soil samples.

Soil - Therminol Spill Area

In the limited area of the therminol spill region, Aroclor 1248 was found near the surface at the four sites samples. There was a rapid decrease of concentration with increasing depth. A value of less than 50 ppm was reached at a depth between 2.5 and 8 ft.

1.0 SAMPLING

1.1 Introduction

All soil and water sampling was done by personnel from the firm of Leggette, Brashears and Graham. The general principles were outlined in the Proposed Hicksville Plant Groundwater Study document⁽¹⁾ which is included in this report as Appendix B-1. Additional details are given in this report in Section II - Hydrogeology. All samples were split with the NYS DEC representative who was present for all sampling operations.

1.2 Soil - Well Sites

The general procedure for sampling soil from the well sites was to use a split spoon in advance of the casing. Samples were taken at 5 foot intervals from the surface down to the top of the saturated zone. At each site, three of these samples were selected for analysis, while the others were archived at the analytical laboratory.

The initial work at Site E encountered an oily material at approximately 48 feet below grade. Samples of soil and water with this material were taken for limited worker health related analysis at the Occidental Chemical Corporation's laboratory at Grand Island, NY. This material was not observed in a subsequent resampling from a spot about 5 feet east of the original site. Additional detail is supplied in Section II (Hydrogeology) of this report. The site locations are shown in Figure 1.1. Note that samples F-36 and F-61 were from the actual location of the F wells at the edge of the sump.

Figure 1.1
Monitor Well Locations

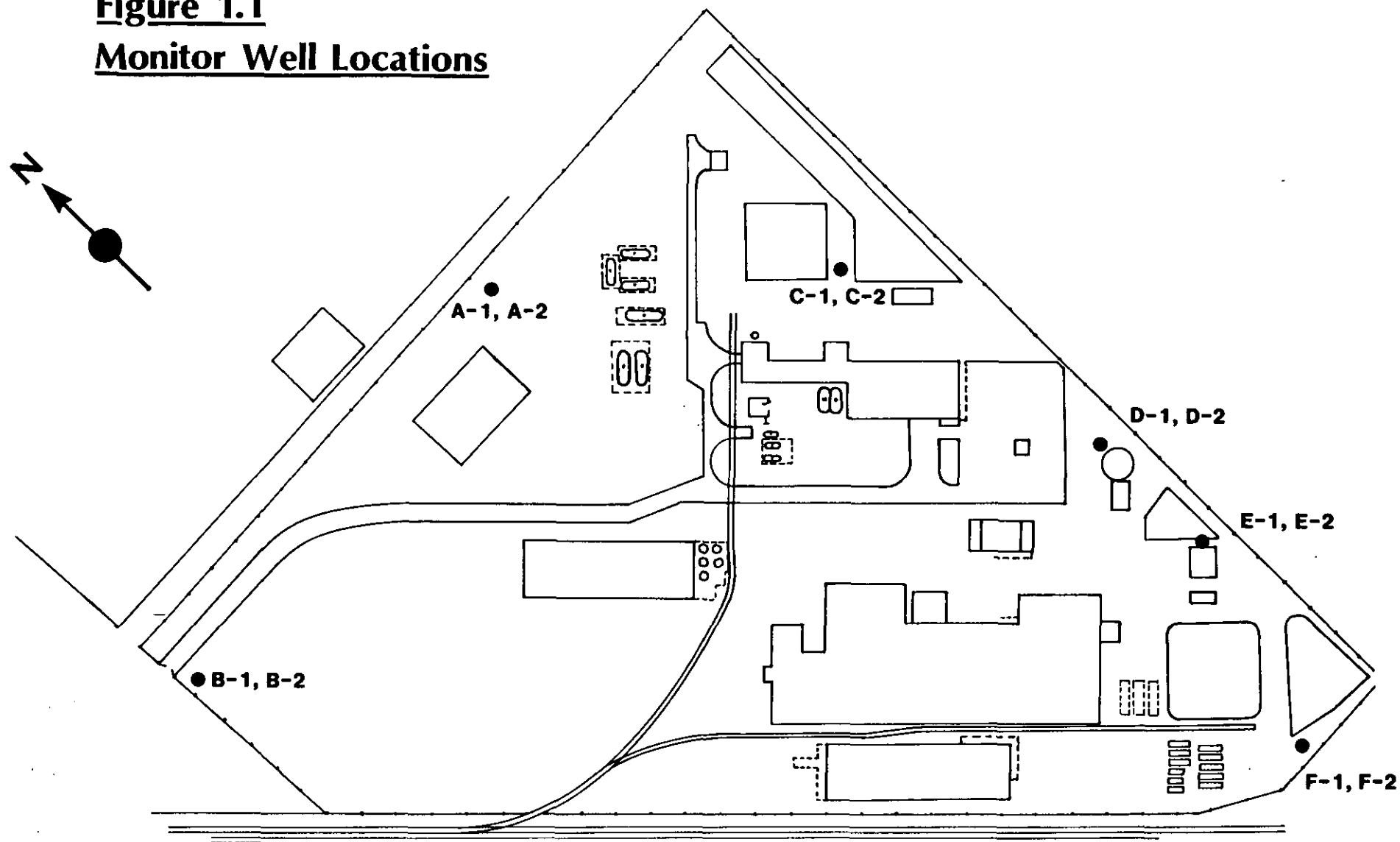
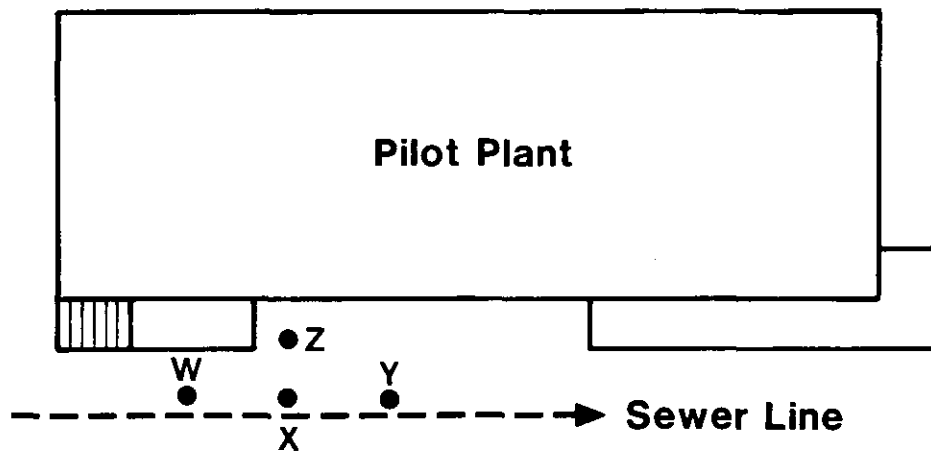


Figure 1.2
Locations of Pilot Borings

1 inch = 20 feet



Plant #1

1.3 Groundwater

The procedure for water sampling was to use a small submersible pump for purging the well casing and taking the samples except for volatiles. A manual bailer was used to obtain volatiles samples. Details on the procedures used and observations made are provided in Section II (Hydrogeology) of this report. The sampling site locations are shown in Figure 1.1.

1.4 Soil - Therminol Spill Area

The therminol spill area is presently paved with asphalt. The soil samples were taken starting just below the paving. Continuous split spoon samples were taken except for the uppermost sample which was taken manually. The sites for sampling in this area are shown in Figure 1.2.

2.0 ANALYTICAL RESULTS

2.1 Introduction

The analysis results reported here were obtained by the Environmental Testing and Certification Corp. (ETC), Edison, NJ. The analytical methods were those specified in the original proposal (Appendix B-1). These methods are given in Appendix B-2. A complete set of the ETC reports is available in Appendix B-4 (bound separately).

Comments on the quality assurance of the analytical work are found in Section 4.0. As noted in 4.3.1, the EPA method used to determine the phthalates proved to be inadequate. The magnitude of this problem and

TABLE 2.2

HICKSVILLE SOIL FROM WELL SITES AT VARIOUS DEPTHS (b)

Parameter	MDL (ug/kg)	A-5	A-25	A-50	B-5	B-25	B-50	C-6 (a)	C-31 (a)	C-56 (a)	D-5	D-25	D-50	D-55	E-7 (a)	E-6 (a)	E-25 (a)	E-51 (a)	E-55	E-60	F-17	F-36	F-46	F-36 (e)	F-61 (d,e)
1,1-Dichloroethylene	100	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Tetrachloroethylene	100	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Toluene	100	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,2-Tran Dichloroethylene	100	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Trichloroethylene	100	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Vinyl Chloride	100	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Styrene	100	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Bis(2-Ethylhexyl)phthalate	*	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Butylbenzylphthalate	*	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Diethylphthalate	*	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Dimethylphthalate	*	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Di-n-Butylphthalate	*	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Di-n-Octylphthalate	*	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Mono	*	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Arochlor 1061 (mg/kg)	0.1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1221 "	0.1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1232 "	0.1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1242 "	0.1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1248 "	0.1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1254 "	0.1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1260 "	0.1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Barium (mg/L) ***	1.0	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Cadmium "	0.05	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Copper "	0.03	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Lead (ug/L)	5	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Mercury "	0.3	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Zinc (mg/L)	0.05	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Nitrate as N (mg/L)	0.04	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Phenolics (Total) "	0.05	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Sulfate as SO ₄ "	9	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
ETC No.		C5429	C5433	C5468	C7342	C7336	C5444	C3597	C5439	C5454	C5449	C5453	C5413	C5419	C5234	C5247	C5240	C5394	C5371	C5372	C5445	C5448	C5380	C5424	C5428
								C5374 (a)	C5375 (a)	C5376 (a)					C5384 (a)	C5385 (a)	C5399 (a)	C5369 (a)							

* - No Detection limit established.

** - Detection Limit except where otherwise noted.

*** - All parameters below are the analysis of liquid from EP Toxicity Test Procedures, Resource Conservation and Recovery Act.

- Parameters not determined.

(a) - Separate sample for Arochlor analysis. *Ref. 100?*

(b) - The numeral in the column heading is the approximate sample depth (feet) below grade.

(c) - ND means not determined at or above the concentration of xx.

(d) - Sample taken from the driller's trough.

(e) - Samples taken from second boring at rim of sump.

TABLE 2.3
HICKSVILLE WATER SAMPLE ANALYSIS^(c)

	MDL (ug/L)**	H DMSHURCB City Water	ENDCAS21NB Casing Blank	XFLDNJKEEA1 Blank (b)	A1	A2	B1	B2	C1	C2	D1	D2	E1	E2	F1	F2
1,1-Dichloroethylene	10	ND 100	ND 100	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10
Tetrachloroethylene	10	ND 100	ND 100	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	50	160	ND 10	ND 10	ND 10	ND 10	ND 10
Toluene	10	ND 100	ND 100	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10
1,2-Transdichloroethylene	10	ND 100	ND 100	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	24	ND 10	30	ND 10	130	200
Trichloroethylene	10	ND 100	ND 100	ND 10	ND 10	25	ND 10	ND 10	ND 10	ND 10	16	ND 10	ND 10	ND 10	ND 10	ND 10
Vinyl Chloride	5	ND 100	ND 100	ND 5	ND 5	ND 5	ND 5	ND 5	ND 5	ND 5	ND 5	ND 5	7	ND 5	140	50
Styrene	10	ND 100	ND 100	ND 5	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10
Bis(2-Ethylhexyl)phthalate	10	ND 10	ND 13	ND 10	ND 10	ND 10	*	*	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10
Butylbenzylphthalate	*	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Diethylphthalate	*	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Dimethylphthalate	*	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Di-n-butylphthalate	*	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Di-n-octylphthalate	10	ND 10	ND 13	ND 10	ND 10	ND 10	*	*	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10
Mnca	10	ND 25	*	ND 10	ND 10	ND 10	*	*	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 25
Aroclors 1242	10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10
1254	10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10
1260	10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10
1248	10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10
1232	10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10
1221	10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10
1016	10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10	ND 10
Cadmium	50	ND 6	ND 6	ND 50	ND 50	ND 50	ND 50	ND 50	ND 50	ND 50	ND 50	ND 50	ND 50	ND 50	ND 50	ND 50
Copper	200	ND 20	20	ND 200	ND 200	ND 200	ND 200	ND 200	ND 200	ND 200	ND 200	ND 200	ND 200	ND 200	ND 200	ND 200
Lead	6	0.0	9.0	ND 6	ND 6	ND 6	ND 6	ND 6	ND 6	ND 6	ND 6	ND 6	ND 6	ND 6	ND 6	ND 6
Mercury	0.3	ND 0.1	ND 0.1	ND 0.3	ND 0.3	ND 0.3	ND 0.3	ND 0.3	ND 0.3	ND 0.3	ND 0.3	ND 0.3	ND 0.3	ND 0.3	ND 0.3	ND 0.3
Zinc	50	ND 10	ND 10	60	64	ND 50	ND 50	ND 50	ND 50	ND 50	ND 50	ND 50	84	ND 50	ND 50	ND 50
Barium	1000	ND 6	ND 6	ND 1000	ND 1000	ND 1000	ND 1000	ND 1000	ND 1000	ND 1000	ND 1000	ND 1000	ND 1000	ND 1000	ND 1000	ND 1000
Sulfate as SO ₄ (mg/L)	1	ND 2	ND 2	ND 2	13	15	20	34	4	36	19	27	ND 2	ND 2	4	5
ODD (mg/L)	2	-	-	ND 2	3	4	3	4	13	3	9	ND 5	25	15	46	66
Nitrate as N (mg/L)	0.1	-	-	ND 0.1	13	1.7	1.1	2.2	1.1	1.2	ND 0.1	0.53	ND 0.1	ND 0.1	ND 0.1	0.17
Specific Cond. (umhos/cm) ^(a)	100	-	-	6400	300	120	220	240	110	170	240	200	180	280	290	400
pH	-	-	-	4.7	6.8	7.0	7.9	7.1	7.5	7.5	6.1	6.7	6.7	6.8	6.4	6.2
Phenolics (Total) (mg/L)	0.05	-	-	ND 0.05	ND 0.05	ND 0.05	ND 0.05	ND 0.05	ND 0.05	ND 0.05	ND 0.05	ND 0.05	ND 0.05	ND 0.05	ND 0.05	ND 0.05
TOC (mg/L)	1	-	-	1,1,ND,ND	1.2	1.5	1.6	1.4	4.2	1.8	2.4	1.3	8.2	0.7	22	14
ETC No.		D3262	D3263	D9766	D8923	D3907	D3915	D3913	D3911	D3917	D3918	D3916	D8547	D3912	D9921	D9922

* - No detection limit established

** - Detection Limit (DL) except where otherwise stated.

- - Not analyzed

(a) - 186G Field Notes except for 6400 value.

(b) - Field Blank - Blank water through the pump & tubing and from the boiler.

(c) - ND means not detected at or above the concentration of xx.

(d) - Average of 4 determinations.

TABLE 2.4

HICKSVILLE SOIL FROM THERMINOL SPILL AREA^(a,b)

Site	Depth (ft.)	ETC No.	1061	1221	1232	1242	1248	1254	1260
W	1	D5513	ND ₅₀₀	ND ₅₀₀	ND ₅₀₀	ND ₅₀₀	20,000	ND ₅₀₀	ND ₅₀₀
	1 - 2.5	D5514	ND ₅₀₀	ND ₅₀₀	ND ₅₀₀	ND ₅₀₀	2,200	ND ₅₀₀	ND ₅₀₀
	2.5 - 4	D5515	ND ₅₀	ND ₅₀	ND ₅₀	ND ₅₀	ND ₅₀	ND ₅₀	ND ₅₀
	4 - 5.5	D5516	ND _{0.5}	ND _{0.5}	ND _{0.5}	ND _{0.5}	3.3	ND _{0.5}	ND _{0.5}
	5.5 - 7	D5517	ND _{1.0}	ND _{1.0}	ND _{1.0}	ND _{1.0}	13	ND _{1.0}	ND _{1.0}
	7 - 8.5	D5493	ND _{0.5}	ND _{0.5}	ND _{0.5}	ND _{0.5}	7.0	ND _{0.5}	ND _{0.5}
	8.5 - 10	D5494	ND _{2.5}	ND _{2.5}	ND _{2.5}	ND _{2.5}	21	ND _{2.5}	ND _{2.5}
X	0.5 - 1.0	D5475	ND ₅₀₀	ND ₅₀₀	ND ₅₀₀	ND ₅₀₀	23,000	ND ₅₀₀	ND ₅₀₀
	1.0 - 2.5	D5476	ND ₅₀	ND ₅₀	ND ₅₀	ND ₅₀	1,300	ND ₅₀	ND ₅₀
	2.5 - 4.0	D5477	ND _{1.0}	ND _{1.0}	ND _{1.0}	ND _{1.0}	21	ND _{1.0}	ND _{1.0}
	4.0 - 5.5	D5478	ND _{2.5}	ND _{2.5}	ND _{2.5}	ND _{2.5}	54	ND _{2.5}	ND _{2.5}
	5.5 - 7.0	D5479	ND _{1.0}	ND _{1.0}	ND _{1.0}	ND _{1.0}	8.6	ND _{1.0}	ND _{1.0}
	7.0 - 8.5	D5499	ND ₅	ND ₅	ND ₅	ND ₅	18	ND ₅	ND ₅
	8.5 - 10.0	D5501	ND _{0.5}	ND _{0.5}	ND _{0.5}	ND _{0.5}	10	ND _{0.5}	ND _{0.5}
Y	1 - 2.5	D5481	ND ₅₀₀	ND ₅₀₀	ND ₅₀₀	ND ₅₀₀	11,000	ND ₅₀₀	ND ₅₀₀
	2.5 - 4.0	D5482	ND ₅₀	ND ₅₀	ND ₅₀	ND ₅₀	500	ND ₅₀	ND ₅₀
	4.0 - 5.5	D5483	ND _{1.0}	ND _{1.0}	ND _{1.0}	ND _{1.0}	30	ND _{1.0}	ND _{1.0}
	5.5 - 7.0	D5484	ND _{0.5}	ND _{0.5}	ND _{0.5}	ND _{0.5}	11	ND _{0.5}	ND _{0.5}
	7.0 - 8.5	D5498	ND _{1.0}	ND _{1.0}	ND _{1.0}	ND _{1.0}	7.2	ND _{1.0}	ND _{1.0}
	8.5 - 10.0	D5499	ND _{1.0}	ND _{1.0}	ND _{1.0}	ND _{1.0}	7.0	ND _{1.0}	ND _{1.0}
Z	0.5 - 2.0	C5434	ND ₅₀₀	ND ₅₀₀	ND ₅₀₀	ND ₅₀₀	22,000	ND ₅₀₀	ND ₅₀₀
	2.0 - 3.5	C5435	ND ₅₀₀	ND ₅₀₀	ND ₅₀₀	ND ₅₀₀	7,300	ND ₅₀₀	ND ₅₀₀
	3.5 - 5.0	C5436	ND ₅₀	ND ₅₀	ND ₅₀	ND ₅₀	1,900	ND ₅₀	ND ₅₀
	5.0 - 6.5	C5437	ND _{2.5}	ND _{2.5}	ND _{2.5}	ND _{2.5}	87	ND _{2.5}	ND _{2.5}
	6.5 - 8.5	C5438	ND _{2.5}	ND _{2.5}	ND _{2.5}	ND _{2.5}	28	ND _{2.5}	ND _{2.5}
	8.5 - 10.0	D5480	ND _{1.0}	ND _{1.0}	ND _{1.0}	ND _{1.0}	35	ND _{1.0}	ND _{1.0}

(a) Concentration in mg/Kg dry weight basis.

(b) ND_{xx} means not detected at or above the concentration of xx.

its' cause were not known until after the April review of the groundwater results. At this point, it was not possible to correct the situation. Thus, it must be recognized that there are no valid results for phthalates in either the soil or groundwater samples.

2.2 Soil - Well Sites

At least three soil samples from each well site were analyzed. Soil from near the surface and at approximately 25 and 50 ft. depths were generally chosen as providing a vertical section of the sites. The list of parameters and the results are given in Table 2.2. Due to difficulties originating at the laboratory, ETC, sites C and E had to be sampled a second time to obtain soil for the Aroclor analyses. Thus, the results for the Aroclors at these sites were obtained using different samples from those used for the other parameters. Regarding the special soil and water samples taken at the 48 ft. depth at Site E (see 1.2), the water, the oily phase and some sediment were examined qualitatively with GC/MS to determine the major components. The results are given in a report located in Appendix B-3. 3 ✓

2.3 Groundwater

Water samples collected from each well site at two depths were analyzed. The parameters and results are given in Table 2.3.

2.4 Soil - Therminol Spill Area

All of the soil samples from the therminol spill area were analyzed for the seven Aroclors listed by the USEPA as priority pollutants. A total of 26 samples from the four sites were analyzed. The results are listed in Table 2.4 along with the laboratory sample numbers for cross referencing.

3.0 Discussion of Results

3.1 Soil - Well Sites

Except for sites E and F the results on the soil samples indicate a very low level presence of two organic compounds, tetrachloroethylene (TECE) and Aroclor 1248 in isolated samples. Two phthalates were identified at site E, but no conclusions about the presence of phthalates can be reached relative to other sites. Other parameters were not detected at levels of note.

The TECE was detected in seven soil samples from four sites. Sites A and D had none at all. At sites B and C the top samples only had very low concentrations, less than 0.4 ppm. At site F the samples taken 20 and 30 feet below the sump bottom had 1.7 and 0.12 ppm respectively, while the samples near the sump bottom (F-17) and the samples taken from the rim of the sump had nothing. At site E samples taken at depths of 2, 6 and 25 feet had concentrations of 244, approximately 1 and 0.16 ppm respectively. These data indicate that the source of the TECE at Site E is near the surface and that the TECE from this source will not be found in significant quantitation below the 25 foot depth.

Traces of Aroclor 1248 (less than 1 ppm) were found in seven samples from three sites. Four of these were at site E with three of them being in samples where TECE also was found. Two more were in samples from site C and one at Site D. The levels are so low (two are near the

detection limit) that no significance can be given to the small differences in concentrations.

The qualitative analysis of the special samples taken from the 48' level of site E showed the presence of Aroclor 1248, bis(2-ethylhexyl)-phthalate and di-n-butylphthalate. These compounds were found in the mixtures of the oily phase with water and/or sediment, but not in the water phase. The fact that this oily phase was not observed in the resampling for Aroclors analysis indicates that this area contains a boundary zone for a localized source or a plume of unknown origin.

3.2 Groundwater

Low concentrations of four chlorinated organic compounds were found in water from sites A-2, C-2, D-1, E-1, F-1 and F-2. The trichloroethylene (TCE) value for A-2 (25 ppb) and the TECE for C-2 (50 ppb) are low and seem to originate from upgradient (off-site) source. The TECE data for the groundwater do not correlate with the TECE found in the soils. The pattern of 1,2-transdichloroethylene and vinyl chloride seen in E-1, F-1 and F-2 is consistent with the hypothesis that TCE or TECE can biodegrade to yield these compounds. (2,3,4)

The relatively high values (compared to other sites) of TOC and COD for sites C-1, D-1, E-1, E-2, F-1 and F-2 indicate the presence of some organic compounds and/or oxidizable substances. The presence of extra peaks in the volatiles and base neutral chromatograms for samples from sites E-1, F-1 and F-2 tend to reinforce the idea that other unidentified materials are present at these sites.

Values for groundwater parameters, other than those mentioned above, are not considered to be notable. The values for zinc reported in A-1 and E-1 are doubtful since they are not much above the detection limit and a similar value was reported for one of the blank samples. The value for specific conductance for the blank water is very likely to be an error of decimal location.

3.3 Soil - Therminol Spill Area

Aroclor 1248 was the only Aroclor found in these soil samples. Concentrations were highest at the surface and decreased rapidly with increasing depths. For sites W, X, and Y, a relatively constant value of less than 50 mg/Kg was reached at depths between 2.5 and 4 ft. At site Z this constant value was reached at between 6.5 and 8.5 ft. These constant values may result from small amounts of soil from the top being moved to lower depths by the drilling-sampling procedures.

4.0 ANALYTICAL QUALITY ASSURANCE

4.1 Summary

Reviews of the Hicksville Analytical Program were made February 24th and April 12, 1984 at ETC Laboratories, N.J. In general, all analyses have been performed according to the requirements of the study as described in the document "Proposed Hicksville Plant Groundwater Study", D. R. Thielen and R. G. Badger, March 3, 1983.

Documentation is complete for all phases of the quality assurance program including chain of custody, analytical methodology, calibration and quality control (spikes and duplicates).

Quality control data indicates that no major problems existed in the analytical program, except for the analysis of phthalates where the EPA procedure proved inadequate. The performance of the laboratory was acceptable.

4.2 Introduction

The analytical requirements of the Hicksville Groundwater Study are contained in the document "Proposed Hicksville Plant Groundwater Study", D. R. Thielen and R. G. Badger, March 3, 1983.

The analytical services required by the study were provided by Environmental Testing and Certification Corporation (ETC), 284 Raintan Center Parkway, Edison, NJ.

All groundwater and soil samples submitted were analyzed according to

the study requirements for all parameters.

4.3 Specific Points

4.3.1 Phthalates

USEPA Method 625 was selected for the analysis of phthalates, either directly in groundwater or from water generated by the EP TOX leach test. This GC/MS method is a general method to analyze a number of classes of compounds including phthalates. Detection limits for some of phthalates were not reported by the laboratory because acceptable spike recoveries ($>50\%$) were not obtained. The reason given was that the method calls for a pH of greater than 11, at this pH, phthalates hydrolyze and can not be recovered. This is an inadequacy of the method rather than of the laboratory. Neither the laboratory nor Occidental were aware of this problem prior to beginning the study.

*what method?
EP-504*

*what about
our lab's that
perform this
method*

4.3.2 Holding Times

In some instances the 7 day holding period prior to extraction of samples for Method 625 was not met. They were extracted within 22 days of receipt. The laboratory feels, based on private communications from researchers at Rutgers University, that this would not affect the validity of the analyses. We agree with the laboratory that the longer holding time should not affect the analysis.

which ones?

*Policy + legal
Basis?
then what about
ours - as to
non routine*

4.3.3 Quality Control

Excellent documentation of internal quality control

procedures was received. This included chain of custody, method summary and GC/MS performance data for every sample. Calibration curves were shown to be linear. Analyses were repeated when a blank sample was shown to be contaminated. The method detection limits were calculated based on the lowest standard run.

4.3.4 Precision and Accuracy

Precision and Accuracy data for all compounds over the course of the study have been compiled: Table I, Soil/Leachate Data; Table II, Water Data. The first two columns of the tables shown accuracy and precision for a spike into a reagent water blank. The accuracy is the average recovery observed for each compound/parameter. The precision is the % relative standard deviation of all the recoveries performed. The third and fourth columns show accuracy and precision for a matrix (actual sample) spike. Spiking level and method detection limit are shown in columns five and six.

The tables show that accuracy and precision were similar for the blank and matrix spikes. The matrix spike was slightly less accurate and precise, as would be expected. In general, the precision and accuracy data were acceptable for all parameters. The only exception was vinyl chloride in soil where

the precision is 42%. The loss of very volatile compounds is to be expected during handling of soil samples. Recoveries of Butylbenzyl, Diethyl, Dimethyl and Di-n-butyl phthalates were very poor, this was discussed earlier. Matrix spike recoveries for Moca were low, but are considered acceptable.

TABLE I

Soil/Leachate

Compound	Spike Accuracy	Blank Precision	Spike Accuracy	Sample Precision	Spiking Level	MDL
<u>PCB</u>						
A1248	-	-	93	39	0.2	0.1 mg/kg
A1248	-	-	117	14	15-17	0.1 mg/kg
<u>Phthalates</u>						
Bis 2 ethylhexyl	107	13	70	18	300	100 ug/kg
Butyl Benzyl	13	5	13	5	300	100 ug/kg
Diethyl	3	1	5	4	300	100 ug/kg
Dimethyl	1	1	1	1	300	100 ug/kg
Di-n-butyl	23	8	25	13	300	100 ug/kg
Di-n-octyl	97	16	30	8	300	100 ug/kg
Moca	97	16	30	8	500	250 ug/kg
<u>Volatiles</u>						
1,1-Dichloroethylene	95	12	98	13	36	10 ug/kg
Tetrachloroethylene	103	8.5	95	12	36	10 ug/kg
Toluene	98	5.6	99	6.2	36	10 ug/kg
1,2-Trans-dichloroethylene	95	11	92	4.7	36	10 ug/kg
Trichloroethylene	101	5.3	97	15	36	10 ug/kg
Vinyl Chloride	94	18	67	42	36	5 ug/kg
Styrene	105	8.5	107	15	36	10 ug/kg
<u>Metals, Conventional</u>						
Barium	101	2.4	100	4.9	4.0	1.0 mg/l
Cadmium	99	2.9	95	9.2	0.400	0.050 mg/l
Copper	101	5.4	92	8.3	0.400	0.200 mg/l
Lead	101	3.5	100	4.0	1.6	0.01 mg/l
Zinc	102	2.7	93	6.2	1.6	0.050 mg/l
Mercury	99	3.6	96	10	0.002	0.0003 mg/l
Nitrate	95	8.4	94	9.2	0.5-10	0.10 mg/l
Sulfate	99	2.2	103	3.9	25	9 mg/l
Phenols, Total	103	6.7	98	3.3	0.100	0.050 mg/l
COD	102	8.4	95	11	500	2 mg/l
TOC	-	-	-	-	-	-
TOC	-	-	-	-	-	-
TOC	-	-	-	-	-	-

TABLE II

Water

<u>Compound</u>	<u>Spike Accuracy</u>	<u>Blank Precision</u>	<u>Spike Accuracy</u>	<u>Sample Precision</u>	<u>Spiking Level</u>	<u>MDL</u>
<u>PCB</u>						
A1248	73	7.6	72	13	20	10 ug/l
<u>Phthalates</u>						
Bis 2 ethylhexyl	83	13	69	5	30	10 ug/l
Butyl Benzyl	15	5	17	3	30	10 ug/l
Diethyl	3	2	4	3	30	10 ug/l
Dimethyl	2	2	2	2	30	10 ug/l
Di-n-butyl	20	5	23	1	10	10 ug/l
Di-n-octyl	71	14	57	9	30	10 ug/l
Moca	63	15	47	7	60	25 ug/l
<u>Volatiles</u>						
1,1-Dichloroethylene	122	13	109	7.3	14	10 ug/l
Tetrachloroethylene	97	15	110	7.4	14	10 ug/l
Toluene	103	12	112	16	14	10 ug/l
1,2-Trans-dichloroethylene	120	4.9	120	3.7	14	10 ug/l
Trichloroethylene	99	16	100	13	14	10 ug/l
Vinyl Chloride	92	18	97	13	14	5 ug/l
Styrene	100	4	105	5.6	14	10 ug/l
<u>Metals, Conventional</u>						
Barium	102	31	107	14	2.0	1.0 mg/l
Cadmium	112	1.1	111	1.6	0.120	0.050 mg/l
Copper	107	6.4	108	2.3	0.400	0.200 mg/l
Lead	118	1.6	102	2.1	0.02	0.10 mg/l
Zinc	103	3.7	112	2.2	0.120	0.050 mg/l
Mercury	103	0.1	96	0.1	0.0005	0.0003 mg/l
Nitrate	95	8.4	94	9.2	0.5-10	0.10 mg/l
Sulfate	99	2.2	103	3.9	25	2 mg/l
Phenols, Total	103	6.7	98	3.3	0.100	0.050 mg/l
COD	102	8.4	95	11	500	2 mg/l
TOC	-	-	125	0.1	2	1 mg/l
TOC	-	-	95	4.7	44	1 mg/l
TOC	-	-	108	0.4	19	1 mg/l

APPENDIX B-1

PROPOSED HICKSVILLE PLANT GROUNDWATER STUDY

PROPOSED HICKSVILLE PLANT GROUNDWATER STUDY

A study of the groundwater and certain soils at the Occidental Chemical Corporation's former Hicksville plant (Hicksville, Long Island, New York) is being planned. The work will be divided into two tasks, sampling and analytical. The requirements for both of these tasks are outlined in this document to aid in the estimation of the cost of the program.

I. SAMPLING

Sampling will be performed by a two-person team with experience in environmental sampling. The senior member of the team will be responsible for complete documentation of sampling which will be kept in a field notebook with bound pages, appropriately dated and signed. The sampling team will be responsible for supplying proper sample containers, the filtration of water samples, for the preservation of all samples and performing any tests required in the field. The team also will maintain chain of custody records for all samples until they are shipped to the analytical laboratory.

Twelve (12) well sites will be sampled for water and samples of soil will be taken during the construction of six (6) of these wells. Six (6) of these wells will be screened at the 50-70 ft. depth and six (6) will be screened at the 80-100 ft. depth. Additional soil samples will be taken at 4 to 7 other sites during the same time that the wells are being constructed.

Groundwater Details

Wells will be sampled after pumping at least four volumes of the well casing, or until the well has been completely evacuated, whichever comes first. Placement of the pump inlet tubing should be such as to assure that the water in the casing will be exchanged with fresh water from the aquifer. Pumping and sampling will be performed using a peristaltic, centrifugal or gas lift pump which contain materials of construction shown not to compromise or contaminate the sample in any way. Samples for volatile organics will be taken by bailing after the well has been purged. With the exception of the Group A compounds, all water will be pressure filtered using a 0.5u pore size "Teflon" membrane filter and placed into an appropriate sample container. Group A compounds will be taken and analyzed as unfiltered samples (after any solids have separated by settling or mild centrifugation). The sample must be properly preserved as noted in Table I and stored at 4° C until analysis.

Detailed preservation techniques are noted in reference (1). Conductivity and pH measurements will be made on unfiltered samples in the field.

Soil Details

Soil samples at well sites will be taken during well construction at approximately five (5) foot intervals in the unsaturated zone. A 2"x24" split spoon driven in advance of the auger will be the mode of sampling. The top six (6) inches of the split spoon sample will be discarded in all cases and the remainder will be placed in a suitable size glass jar with a "Teflon" lined screw cap. All soil samples will be cooled to 4°C for transportation to the laboratory. It is expected that separate samples (using special precautions to avoid loss of volatiles) will be taken for volatiles analysis.

Soil samples in the Therminol handling area will be taken by continuous split spoon sampling to a depth of approximately 6 feet. The initial sampling will be at the center of the handling area and 10 feet from the center in three radial directions. If contamination is found in the initial samples, additional sampling will be required to define the area of contamination.

Sampling Cleanup

Cross contamination between sites for either water or soil sampling must be avoided. This can be done either by dedicated pumping equipment for water or by rigorous clean up between sites (for water) or samples (for soil). Details on the procedures to protect sample integrity should be provided.

II. METHODOLOGY

Table 2 contains the groupings of those compounds which must be determined in the samples. The required detection limits are also included.

Groundwater

Group A. EPA Method 624 is required using GC/MS for quantitation. Styrene has been included as per the attached memo (Simon, N., September 29, 1982).

Group B. EPA Method 625 is required using GC/MS for quantitation. MOCA has been included as per the attached memo (Simon, N., September 29, 1982).

Group C. EPA Method 608 is required using GC/EC for quantitation.

Group D. The required EPA Methods are listed in Table II.

Soil

Group A. The required method is a modification of a Midwest Research Report (5). The specific modifications of this method are found in the attached report (Simon and Johnson, August 16, 1982). Quantitation will be by GC/MS.

Group B. The soil will be prepared by obtaining an aqueous extract of the soil using the EPA's EP Toxicity digestion procedure 2. The aqueous extract will be analyzed using EPA Method 625 and GC/MS for quantitation. The limits of detection stated in Table II are based on the limits for the aqueous extract using Method 625 and related back to the original soil sample.

Group C. The required method is that described in Reference (3). In cases of interferences from organochlorine pesticides, an additional clean-up procedure, as outlined in Section 9C of the same manual, will be considered. Quantitation will be by GC/EC. NOTE: Due to the nature of the program, special priority should be given these samples to obtain the most rapid turnaround possible. Please state what this will be.

Group D. The required EPA procedures listed in Table II will be carried out on an aqueous extract of the soil obtained by using the EPA's EP Toxicity digestion procedure (2). The parameters of pH, conductivity COD and TOC will not be required for soils.

The USEPA Methods defined above may be modified in your proposal if valid technical reasons exist. In all cases, your proposed methodology must attain the expected detection limits and be fully documented. Full verification of any non-EPA methods must be made.

III. QUALITY ASSURANCE

As a general rule, EPA practices outlined in Reference (4) will be followed. In particular, the following QC procedures will be required for every batch of samples or at a minimum of every ten samples:

- (1). Replicate sample analysis as randomly selected by the contractor with approval of the project liaison.
- (2). Recovery of all analyzed compounds at two to three times the detection limit using laboratory distilled water.
- (3). Recovery of spikes made to a sample selected by the contractor with approval of the technical liaison. Spiking will be done for all analyzed compounds at a level which approximately doubles the concentration found in the sample. In samples where compounds of interest are not detected, spiking must be at levels not exceeding two to three times the detection limit.
- (4). Reagent and method blanks.

All standards used for quantitation must be traceable to a verified standard; that is, a compound whose purity has been determined by at least two different analytical procedures. A linearity of detector response for each compound must be demonstrated by generation of a linearity curve containing five concentrations of that compound. All sample calculations must be made from responses which fall within this linear range. During the course of the analysis, standards must be interspersed at frequent intervals to check the calibration. The preparation of all standards including purity verification, dilutions, linearities, etc. must be recorded in the bound notebook.

Samples and extracts must be retained and properly stored until time of disposal. After acceptance of the final report by Occidental, the contractor must request and receive permission prior to disposing of samples.

Records containing all relevant data must be easily accessible and kept for a specified period of time as determined by Occidental's technical liaison. These records must include all logbooks, workbooks, worksheets, graphs, charts and/or any records of pertinent nature relating to this study.

All chromatography scans must remain connected in the sequence in which they were generated, i.e., no scans shall be cut, torn or otherwise removed from the body of the chromatographic data attached to it.

The final report must include sample identification information, methods used, analysts, and all samples and quality control data. The calculated data must include units of concentration and limits of detection given with the proper significant figures. In cases where compounds are not detected at or above the stated detection limit, the reporting protocol will be ND_x where x is the required detection limit. An assessment of analytical precision and accuracy must also be stated.

The contractor will designate a project manager who has direct responsibility for the technical aspects of the study. The project manager will be available for detailed technical reviews during the course of the program.

III. QUOTATION AND TECHNICAL PROPOSAL

One technical proposal should cover the complete sample program outlined above. It should contain the following:

- (1). Documented methodology for each analysis.
- (2). Detailed procedures for and the cost of sampling. Also, the precise number, size and type of samples required from each sampling point to allow the contractor to do all the analyses which may be necessary i.e. spikes, duplicates, etc.
- (3). Timing for completion of analyses after receipt of samples. To include issuing of preliminary (verbal) and final (draft) reports.
- (4). A separate cost estimate broken down by analysis and sample including necessary development work.
- (5). An estimate of timing starting from receipt of samples to when a report including documentation, QA/QC and results can be expected.

One quotation should be submitted separately and cover the complete program. Included in the quotation should be the cost broken down by analysis and sample.

The technical proposal and quotation should be sent to our attorney, who will also refer any questions to the appropriate technical personnel.

John Hanna, Esq.
WHITEMAN, OSTERMAN AND HANNA
99 Washington Avenue
Albany, New York 12210
PHONE: 518/449-7600

DATE: _____

PREPARED BY:

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Central Sciences

Richard G. Badger
Sr. Research Chemist
Central Sciences

/jb
03/02/83

REFERENCES

- (1). "Handbook for Sampling and Sample Preparation of Water and Wastewater", EPA-600/4-82-029, Sept. 1982.
- (2). "RCRA Test Methods for Evaluating Solid Waste - Physical/Chemical Methods", SW-846, May 1980.
- (3). "Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples", EPA-600/8-30-038, June 1980, Section 11A.
- (4). "Handbook for Analytical Quality Control in Water and Wastewater Laboratories", EPA-600/4-79-019, March 1979.
- (5). MRI Special Report No. 1, "Development of Analytical Test Procedures for the Measurement of Organic Priority Pollutants in Sludges and Sediments", June 26, 1979, Midwest REsearch Institute Project No. 4583-A.

PRIVILEGED & CONFIDENTIAL
ATTORNEY-CLIENT COMMUNICATION PREPARED AT THE REQUEST
OF LEGAL COUNSEL IN CONTEMPLATION OF LITIGATION

TABLE 1
 PRESERVATION METHODS - WATER

PARAMETER	PRESERVATION METHOD
VOLATILE ORGANICS	4° C
BASE/NEUTRAL ORGANICS	4° C
PCB'S	4° C
NITRATES	2ML H ₂ SO ₄ PER LITER AND 4° C
SULFATES	4° C
CADMIUM •	ADD 1:1 REDISTILLED HNO ₃ TO PH OF <2
MERCURY •	
BARIUM •	
COPPER •	
LEAD •	
ZINC •	
COD	ADD SULFURIC ACID TO PH OF <2 AND 4° C
TOC	ADD H ₂ SO ₄ OR HCL TO PH OF <2 AND 4° C
PHENOLICS	ADD H ₂ PO ₄ TO PH OF <4, ADD 1G/L OF CUSO ₄ , AND 4° C

ATTORNEY-CLIENT COMMUNICATION PREPARED AT THE REQUEST
OF LEGAL COUNSEL IN CONTEMPLATION OF LITIGATION

GROUP A

VOLATILES FRACTION	WATER		SOIL	
	DETECTION LIMIT (UG/L)		DETECTION LIMIT (NG/G)	
TETRACHLOROETHYLENE	10		100	
TRICHLOROETHYLENE	10		100	
DICHLOROETHYLENE	10		100	
TOLUENE	10		100	
VINYL CHLORIDE	5		100	
STYRENE	10		100	

GROUP B

BASE NEUTRAL FRACTION	WATER		SOIL	
	DETECTION LIMIT (UG/L)		DETECTION LIMIT (NG/G)	
BIS(2-ETHYLHEXYL)PHTHALATE	10		100	
BUTYL BENZYL PHTHALATE	10		100	
DIETHYL PHTHALATE	10		100	
DIMETHYL PHTHALATE	10		100	
DI-N-BUTYL PHTHALATE	10		100	
DI-N-OCTYL PHTHALATE	10		100	
MOCA (3,3'-DICHLORO-4,4'- DIAMINODIPHENYLMETHANE)	25		250	

GROUP C

AROCHLOR FRACTION	WATER		SOIL	
	DETECTION LIMIT (UG/L)		DETECTION LIMIT (NG/G)	
AROCHLOR-1016	10		100	
AROCHLOR-1221	10		100	
AROCHLOR-1232	10		100	
AROCHLOR-1242	10		100	
AROCHLOR-1248	10		100	
AROCHLOR-1254	10		100	
AROCHLOR-1260	10		100	

GROUP D

OTHER PARAMETERS	WATER AND SOIL OPTIMUM RANGE	USEPA METHOD #
NITRATES	0.1 TO 2.0MG NO ₃ -N/LITER	352.1
SULFATES	3 TO 400 MG SO ₄ /LITER	375
CADMIUM*	0.05 TO 2 MG/LITER	213.1
MERCURY*	0.2 UG/LITER	245.1
BARIUM*	1 TO 20 MG/LITER	208.1
COPPER*	0.2 TO 5 MG/LITER	220.1
LEAD*	5 TO 100 UG/LITER	239.2
ZINC*	0.05 TO 1 MG/LITER	289.1
CONDUCTIVITY	--	120.1
PH	--	150.1
COD	20 TO 900 MG/LITER	410.4
TSS	01 MG/LITER	415.1
PHENOLICS	05 UG/LITER	420

* - THE DETECTION LIMIT IS BASED ON THE ANALYSIS OF AN AQUEOUS EXTRACT AND RELATED BACK TO THE ORIGINAL WEIGHT OF THE SOIL.

** - THE DETECTION LIMIT IS BASED ON THE ANALYSIS OF THE WATER OBTAINED FROM THE AQUEOUS EXTRACTION OF THE SOIL (EP TOX).



Occidental Chemical Corporation

Research Center

MEMO

To A. F. Weston Date September 29, 1982

From N. Simon

Subject GC/MS Analysis of Styrene, Moca, Phthalates and Five Volatile Organics

COPIES: D. Johnson, P. Skotnicki, R. Badger, TIC

I. SUMMARY

The EPA Priority Pollutant Method for base neutral organics was extended to include styrene and 3',3'-dichloro 4,4'-diamino diphenyl methane (MOCA). Standard curves were generated and extraction efficiencies calculated. Detection limits were set at 10 µg/L for styrene and 25 µg/L moca. The volatiles analyses could also be used to analyze for styrene and appears to be the preferred method.

A. Extractables

1). Instrumental Parameters

Gas Chromatographic Conditions (Finnigan 96100)

Column	- 15 m DB5-NB fused silica capillary (J&W)
Carrier	- Helium 15.0 psi
Injector Temperature	- 275°C
Injection	- Grob, 60/1 split after 60 seconds
Detector Temperature	- 275°C
GC/MS Interface	- 265°C-275°C
Column Program	1) - 20°C to 250°C at 10°C/minute after a 1 minute hold at 20°C, hold at 250°C for 20 minutes. 2) - Without styrene - 50°C to 250°C.

Mass Spectrometer Conditions (Finnigan 4000)

Instrument	- Finnigan 4000 GC/MS interfaced with an Incos Data Acquisition System
Source Parameters	- 85°C, Electron Impact Source with 70eV ionizing electrons
EM Volts	- 1380 volts
Scan Parameters	- Total scan sequence - .5 second consisting of acquisition during .45 second up scan, .05 second hold at bottom. Mass range scanned 350-45.

2). Sample Preparation

for the base neutral extraction, one liter of sample was adjusted to pH 11 with 6N NaOH; extracted three times with methylene chloride according to EPA protocol; dried through a sodium sulfate column; and concentrated to 5 ml using a Kuderna-Danish evaporator and nitrogen.

An internal standard, deuterated phenanthrene was added 15 minutes prior to the analysis.

3). Standard Preparation

A stock solution containing the six phthalates was purchased from Supelco. Styrene and MOCA standards were prepared in-house. The standards were prepared to give 1,5,10 and 20 times the detection limit. The detection limit for MOCA was set at 25 µg/L to give a relatively equivalent response when compared to styrene and the phthalates at 10 µg/L.

4). Extraction Efficiencies

Since the method has routinely been used for phthalates it was only necessary to verify its efficiency for styrene and MOCA. Three blank water samples were spiked at 10X the detection limit, extracted and analyzed by the method noted above.

Sample	% Recovery			
	Styrene		MOCA	
	Day 1	Day 2	Day 1	Day 2
20832	51	55	72	88
20833	74	60	79	85
20834	88	74	69	83

The ions used to identify and quantitate were m/e 266, 268, and 131 for MOCA, and m/e 104, 102, 51 for styrene.

(B). VOLATILES

Extending Method 624 to include styrene.

(see Page 3 for Volatiles)

(B). VOLATILES

1) Instrumental Parameters

Purge and Trap Conditions
(Tekmar Liquid Sample Concentrator-Model LSC-2)

Plumbing	- Hard plumbed from trap effluent to the GC flow controller via a 1/8 inch O.D. copper line
Trap Column	- 12" x 1/4" stainless steel tubing packed with Tenax 60/80 mesh. Baked after each run at 250° for 20+ min.
Purge	- 12 minutes at 30 cc/minute
Desorb	- 4 minutes at 195°
Sample Size	- 5 ml transferred by Blenco gas/liquid syringe

Gas Chromatographic Conditions (Finnigan 9610)

Column	- 8 foot by 1/4 inch (2mm I.D.) glass packed with 0.1% SP-1000 on Carbowack C
Carrier	- Helium at 30 cc/minute
Injector	- 180°
GC/MS Interface	- 250°
Column Program	- 50° for purge, desorb and three minutes after desorb; 8°/min. to 180°; held for 30 min. at 180°

Mass Spectrometer Conditions

Instrument	- Finnigan 4000 GC/MS interfaced with an Incos Data Acquisition System
Source Parameters	- 260°, Electron Impact Source with 70 eV ionizing electrons
Manifold Temperature	- 90°
Electron Multiplier	- 1080 volts
Scan Parameters	- Total scan sequence of 2 seconds consisting of data acquisition during 1.95 sec. up scan, 0.05 sec. hold at bottom. Mass range scanned 45-270.

2. Standards

The standards used were supplied by Supelco and are described as "Standards for EPA Consent Decree Protocol". They are further referenced to (I.F.B. No. WA77-B133, Appendix B, Sampling and Analysis for Priority Pollutants, US EPA). A solution of styrene at the same concentration as the above standards, was prepared in the lab.

Bromochloromethane, 2-Bromo-1-chloropropene and 1,4-dichlorobutane were used as internal standards.

The stock solutions, as received from Supelco, were stored in a freezer. Dilutions were stored in the refrigerator in 15 ml hypovials until one hour before analysis. Standards were prepared to give concentration levels of 10 µg/L (50 ng injected) and 100 µg/L (500 ng injected). An additional standard at 25 µg/L (125 ng injected) was analyzed to verify linearity. Internal standards were prepared at 20 µg/L; 5 µl (100 ng injected) was used to spike each standard and sample.

Standards were stored in the refrigerator until one hour before analysis.

Standards were poured into a 5 ml syringe; the volume adjusted; the needle removed and 5 µl internal standard added immediately before injection into the Tekmar.

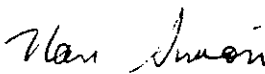
Standards could be prepared by weighing pure materials into methanol instead of using the commercial mix since only five of the priority pollutants are required: perchloroethylene, trichloroethylene, trans-1,2-dichloroethylene, toluene, and vinyl chloride. It should also be noted that the required detection limit for VCM is 5 µg/L while the detection limit for the other volatile components is 10 µg/L.

3. Results and Discussion

The EPA Priority Pollutant base neutral method can be extended to include styrene and MOCA. The chromatogram following (Figure 1) demonstrates the relative retention times of styrene and MOCA compared to the phthalates.

It seems preferable to analyze styrene with the volatiles rather than the extractables for a number of reasons: The gas chromatographic oven will not need sub-ambient conditions to separate styrene from the solvent (see Figure 2); loss of styrene will not be a problem; a narrower range of internal standards will be acceptable, styrene carryover will be limited in the volatiles analysis, etc.

The RIC's from the analyses (Figures 1,2,4) and the mass spectrum of MOCA (Figure 3) follow.



Nan Simon
Central Sciences

RIC

08/31/82 16:00:00

SAMPLE: PHTHALATE STYRENE MOCA STDs

RANGE: G 1,2900 LABEL: N 0, 4.0 QUAN: A 0, 1.0 BASE: U 20, 3

DATA: ETC TEST1 #379

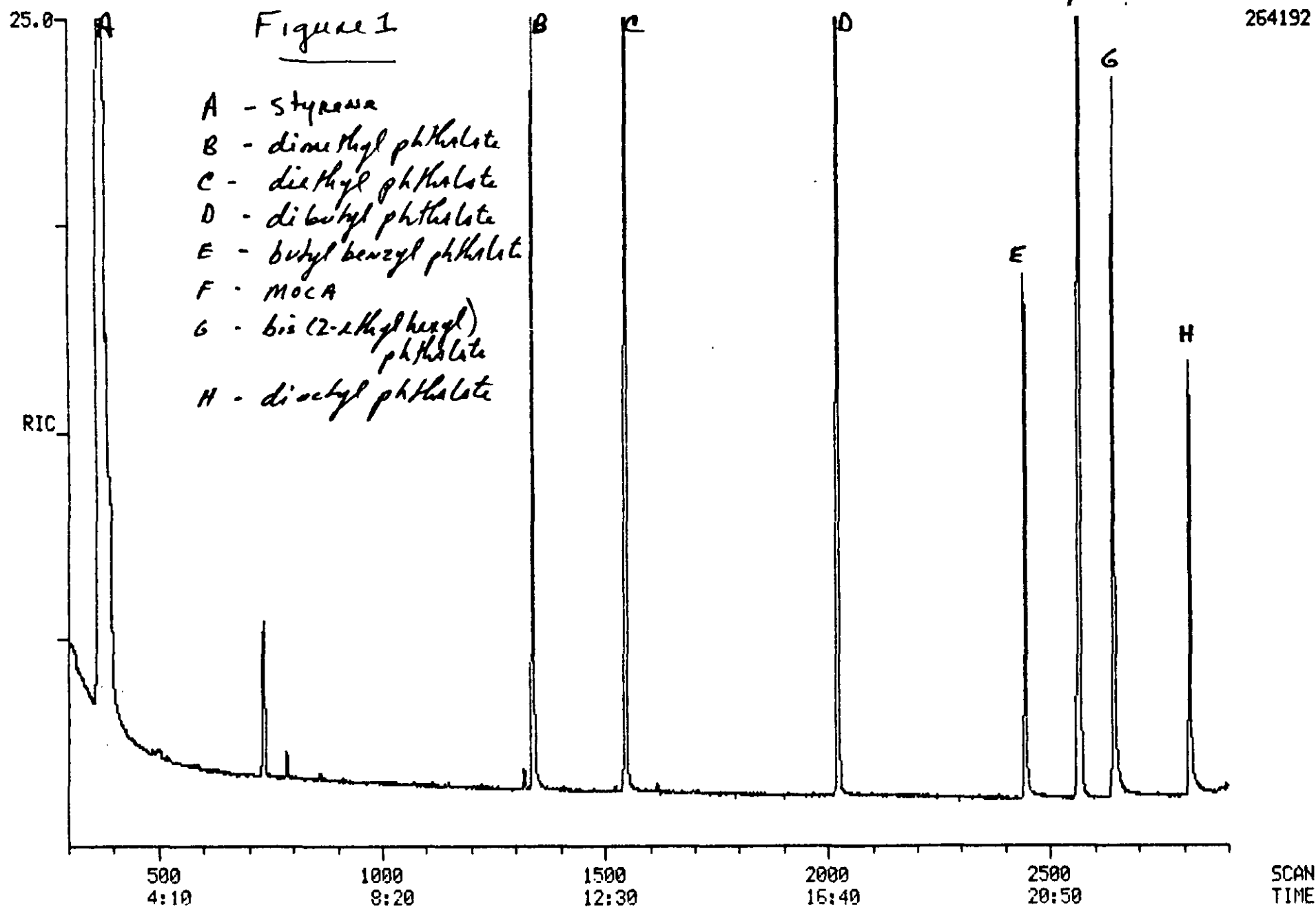
CALI: N50831A #1

SCANS 300 TO 2900

264192

Figure 1

- A - styrene
- B - dimethyl phthalate
- C - diethyl phthalate
- D - dibutyl phthalate
- E - butyl benzyl phthalate
- F - MOCA
- G - bis (2-ethylhexyl)
phthalate
- H - dioctyl phthalate



RIC

09/02/82 15:01:00

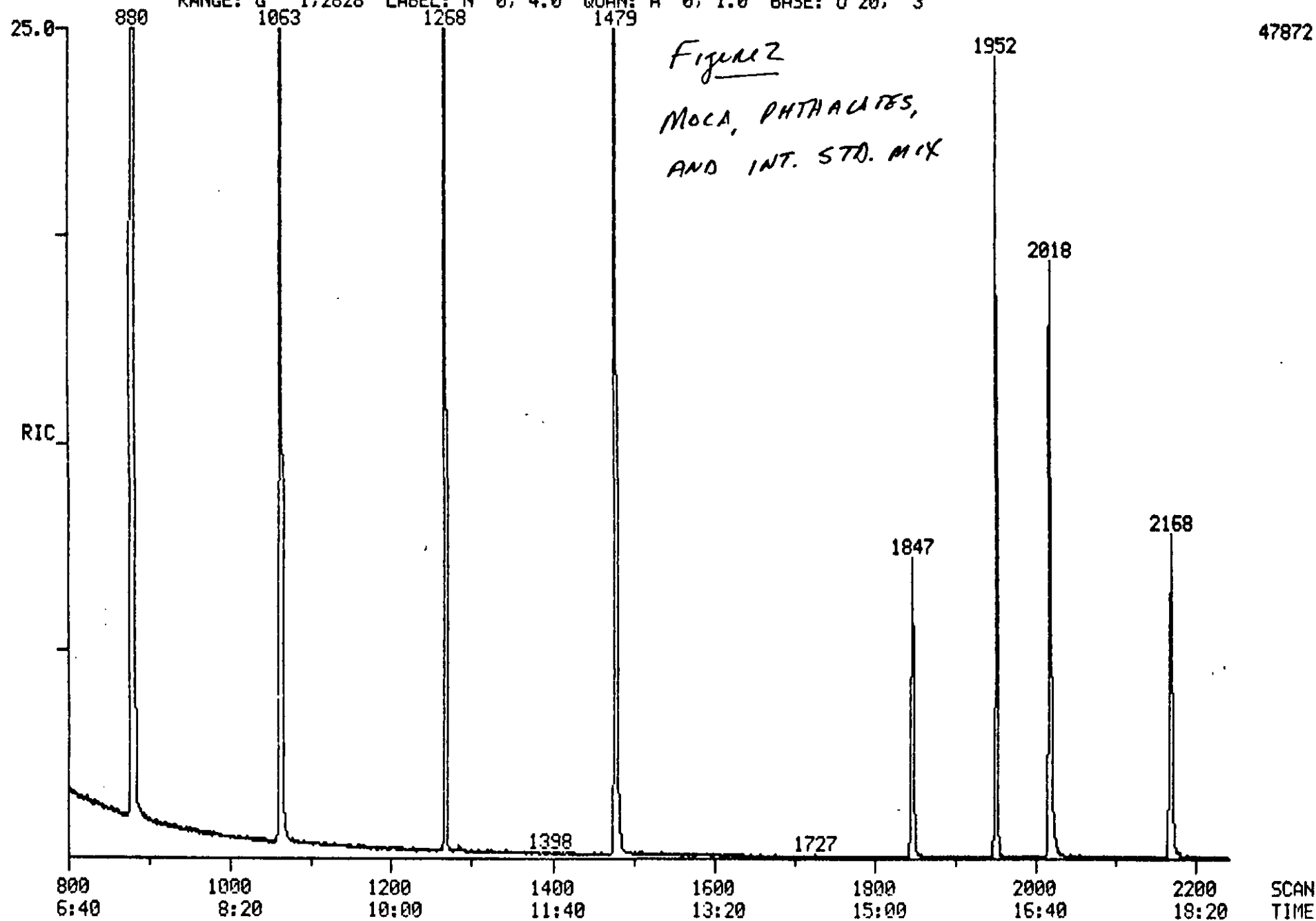
SAMPLE: PHTH AND MOCA

RANGE: G 1.2628 LABEL: N 0, 4.0 QUAN: A 0, 1.0 BASE: U 20, 3

DATA: PMTEST #503

CALI: NS0902 #1

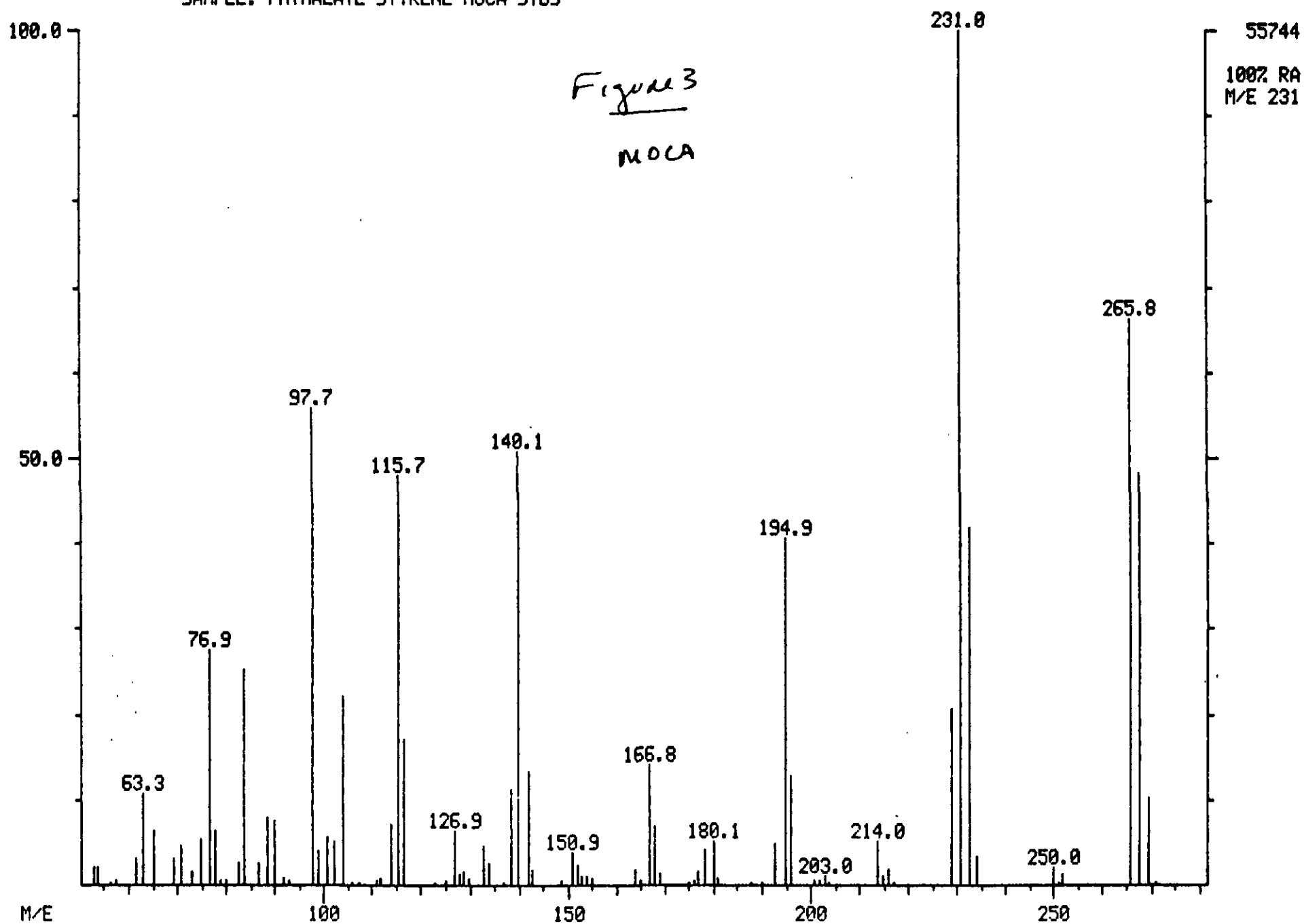
SCANS 800 TO 2240



MASS SPECTRUM
08/31/82 15:22:00 + 21:27
SAMPLE: PHTHALATE STYRENE MOCA STD5

DATA: ETCTEST #2574
CALI: NS0831A #1

BASE M/E: 51
RIC: 516096.



RIC

09/03/82 10:50:00

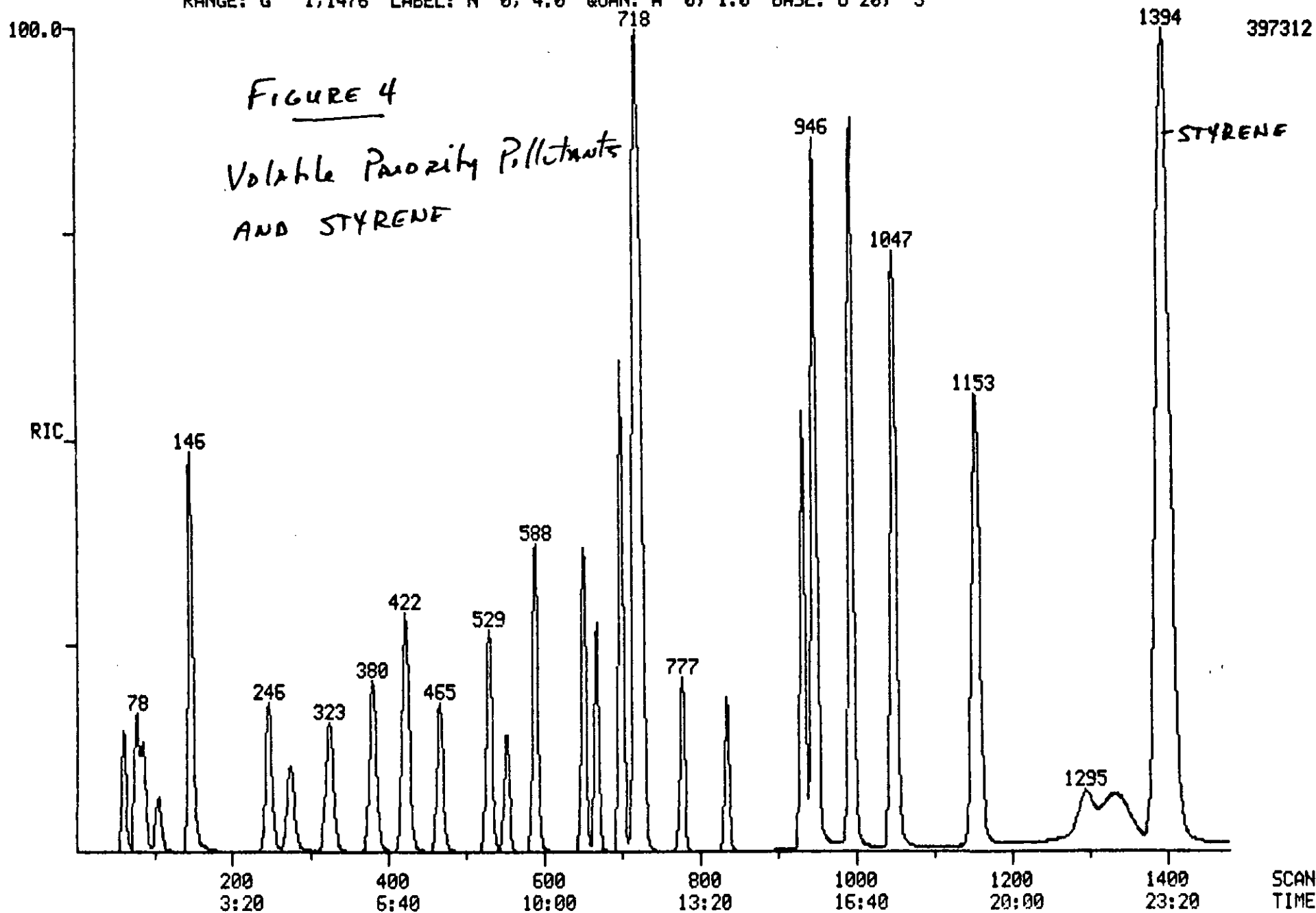
SAMPLE: VOLATILES WITH STYRENE

RANGE: G 1,1476 LABEL: N 0, 4.0 QUAN: A 0, 1.0 BASE: U 20, 3

DATA: UPPSTD #1332

CALI: 0903DJ #3

SCANS 1 TO 1479



August 16, 1982

To: R. Hall

From: N. Simon, D. Johnson

Distribution: P. Skotnicki, A. Weston

Reference: GC/MS Analysis of Soil Samples for Volatile Priority Pollutants

I. Summary

This report summarizes the GC/MS sample preparation and analyses of six soil samples taken at the Arecibo facility on 8/ /82. The methodology used was as developed for the EPA. It is considered semi-quantitative because of variances in the sampling, sample handling and the sample matrix.

Sample 00003 (STP Plant across from PRC/sewer bottoms in sewer dumping spot #5) was the only sample where priority pollutant volatile organics were detected at greater than 10 ug/L. The compounds found were benzene, toluene and chlorobenzene. Vinyl chloride, 1,1-dichloroethylene, trans-1,2-dichloroethylene and trichloroethylene were not detected in any of the samples. Toluene was only detected in 00003. Non-volatile priority pollutants found were xylenes in sample 00003 and dichlorobenzene in 00092.

II. Experimental

The EPA priority pollutant method is described in Special Report No. 1 "Development of Analytical Test Procedures for the Measurement of Organic Priority Pollutants in Sludges and Sediment", published June 26, 1979 under contract No. 68-03-2695, MRI Project No. 4583-A. The only significant deviation from the published method was the use of a larger sample to give a lower detection limit.

A. Instrumental Parameters

Purge and Trap Conditions (Tekmar Liquid Sample Concentrator-Model LSC-2)

Plumbing	-	Hard plumbed from trap effluent to the GC flow controller via a 1/8 inch O.D. copper line.
Trap Column	-	12" X 1/4" stainless steel tubing packed with Tenax 60/80 mesh. Baked after each run at 250° for 20+ min.
Purge	-	12 minutes at 30 cc/minute
Desorb	-	4 minutes at 195°C
Sample Size	-	0.5g in 5ml distilled water

Gas Chromatographic Conditions (Finnigan 9610)

Column - 8 foot by 1/4 inch (2 mm I.D.) glass packed
with 60/80 Carbopack C/0.2% CW 1500

Carrier - Helium at 25 cc/minute

Injector - 180°C

GC/MS Interface - 250°

Column Program - 50° for purge, desorb and three minutes after
desorb; 8°/min. to 180°; held for 30 min. at
180°

Mass Spectrometer Conditions

Instrument - Finnigan 4000 GC/MS interfaced with an Incos
Data Acquisition System

Source Parameters- 260°, Electron Impact Source with 70 eV
ionizing electrons

Manifold Temperature- 90°

Electron Multiplier- 1330

Scan Parameters - Total scan sequence of 1 second consisting of
data acquisition during 0.95 sec. up scan,
0.05 sec. hold at bottom. Mass range scanned
45-180

B. Sample Preparation

The sample for each site was received in a wide mouth glass quart bottle with a teflon cover. (There was considerable head space in each bottle). One half ml. (~ 0.5g) was transferred, using a tipless disposable pipet, to a Tekmar tube. Five mls of distilled water and 5 ml of an internal standard solution were added. The tube was immediately attached to the Tekmar and purged.

Since the samples did not appear to be homogenous and since there was one to three inches of headspace, the 0.5ml aliquot was taken from the bottom half of the bottle and each sample was analyzed in duplicate.

The samples were refrigerated until one hour before analysis.

C. Standards

The standards used were supplied by Supelco and are described as "Standards for EPA Consent Decree Protocol". They are further referenced to (I.F.B. No. WA77-B133, Appendix B, Sampling and Analysis for Priority Pollutants, US EPA).

Bromochloromethane, 2-Bromo-1-chloropropene and 1,4-dichlorobutane were used as internal standards.

The stock solutions, as received from Supelco, were stored in a freezer. Dilutions were stored in the refrigerator in 15 ml hypovials until one hour before analysis. Standards were prepared to give concentration levels of 10 µg/L (5 ng injected) and 100 µg/L (50 ng injected). An additional standard at 50 µg/L (25 ng injected) was analyzed to verify linearity. Internal standards were prepared at 20 µg/L; 5 µl (100 ng injected) was used to spike each standard and sample.

III. Quality Assurance

All six samples were analyzed in duplicate. A blank was prepared using 1/2 ml ^{gravel (a mg)} of soil and 5 mls of distilled water. The blank was analyzed each day to verify the absence of sample handling contamination. Three spiked samples were prepared at 10 or 20 µg/L, two from the lab blank and one an actual sample.

Linearity was verified with a three point curve (10, 50 and 100 µg/L) and a three component internal standard was added to each sample and standard.

The significant amount of headspace and the non uniformity of each sample limits the quantitative conclusions that normally could be assumed with the rigorous quality assurance protocol. Sample 00003 was the most obvious example; a mixture of soil and black sludge that was impossible to accurately reproduce in the transfer.

IV. Results and Conclusions

The results are listed in Table 1. % recoveries from the three spikes are listed in Table 2. Chromatograms of each sample follow the tables.

Nan Simon
Nan Simon

jmw/

attachments

TABLE 1
RESULTS SUMMARY

C.S. Log #	20811	20812	20813*	20814**	20815	20816
Sample I.D.	00061	00002	00003	00092	00090	00062
Chloromethane	ND	ND	ND	ND	ND	ND
Bromomethane	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀
Vinylchloride	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀
Chloroethane	ND ₅₀	ND ₅₀	ND ₅₀	ND ₅₀	ND ₅₀	ND ₅₀
Methylene Chloride	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀
Trichlorofluoromethane	ND	ND	ND	ND	ND	ND
1,1-Dichloroethylene	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀
1,1-Dichloroethane	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀
Trans-1,2-Dichloroethylene	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀
Chloroform	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀
1,2-Dichloroethane	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀
1,1,1-Trichloroethane	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀
Carbon Tetrachloride	ND ₅₀	ND ₅₀	ND ₅₀	ND ₅₀	ND ₅₀	ND ₅₀
Bromodichloromethane	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀
1,2-Dichloropropane	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀
Trans-1,3-Dichloropropene	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀
Trichloroethylene	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀
Dibromochloromethane	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀
Cis-1,3-Dichloropropene	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀
Benzene	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀
Bromoform	ND ₅₀	ND ₅₀	ND ₅₀	ND ₅₀	ND ₅₀	ND ₅₀
1,1,2,2-Tetrachloroethene	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀
1,1,2,2-Tetrachloroethane	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀
Toluene	ND ₁₀	ND ₁₀	19 11	ND ₁₀	ND ₁₀	ND ₁₀
Chlorobenzene	ND ₁₀	ND ₁₀	134 66	ND ₁₀	ND ₁₀	ND ₁₀
Ethylbenzene	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀	ND ₁₀

* Xylenes also detected

** A significant amount of dichlorobenzene was detected

TABLE II

% RECOVERY FROM SPIKED SOIL

	D.L. ug/L	Blank Soil @ 10 ug/L	Blank Soil @ 20 ug/L	20811-00061 Soil @ 20 ug/L
Chloromethane	No std.	ND	ND	ND
Bromomethane	10	136%	103%	110%
Vinylchloride	10	103	105	117
Chloroethane	50	ND	ND	123
Methylene Chloride	10	143	161	550*
Trichlorofluoromethane	No std.	ND	ND	ND
1,1-Dichloroethylene	10	108	105	103
1,1-Dichloroethane	10	102	92	114
Trans-1,2-Dichloroethylene	10	100	94	111
Chloroform	10	106	97	100
1,2-Dichloroethane	10	140	100	110
1,1,1-Trichloroethane	10	109	102	121
Carbon Tetrachloride	50	ND	ND	ND
Bromodichloromethane	10	105	101	115
1,2-Dichloropropane	10	147	103	84
Trans-1,3-Dichloropropene	10	90	78	148
Trichloroethylene	10	84	76	95
Dibromochloromethane	10	82	98	101
Cis-1,3-Dichloropropene	10	143	100	110
Benzene	10	96	88	105
Bromoform	50	ND	ND	ND
1,1,2,2-Tetrachloroethene	10	158	155	144
1,1,2,2-Tetrachloroethane	10	83	74	67
Toluene	10	125	138	580*
Chlorobenzene	10	94	85	105
Ethylbenzene	10	108	98	124

* It can reasonably be assumed that the large recovery is contribution from the sample #20811 - identified as 00061. However, neither compound was found in the unspiked sample.

RIC
08/12/82 11:26:00
SAMPLE: SOIL SAMPLE #4 TANK TRUCK DISPOSAL PIT
RANGE: G 1,1376 LABEL: N 0, 4.0 QUAN: A 0, 1.0 BASE: U 20, 3

DATA: 20811 #1030
CALI: 0812DJ #2

SCANS 1 TO 1300

110848

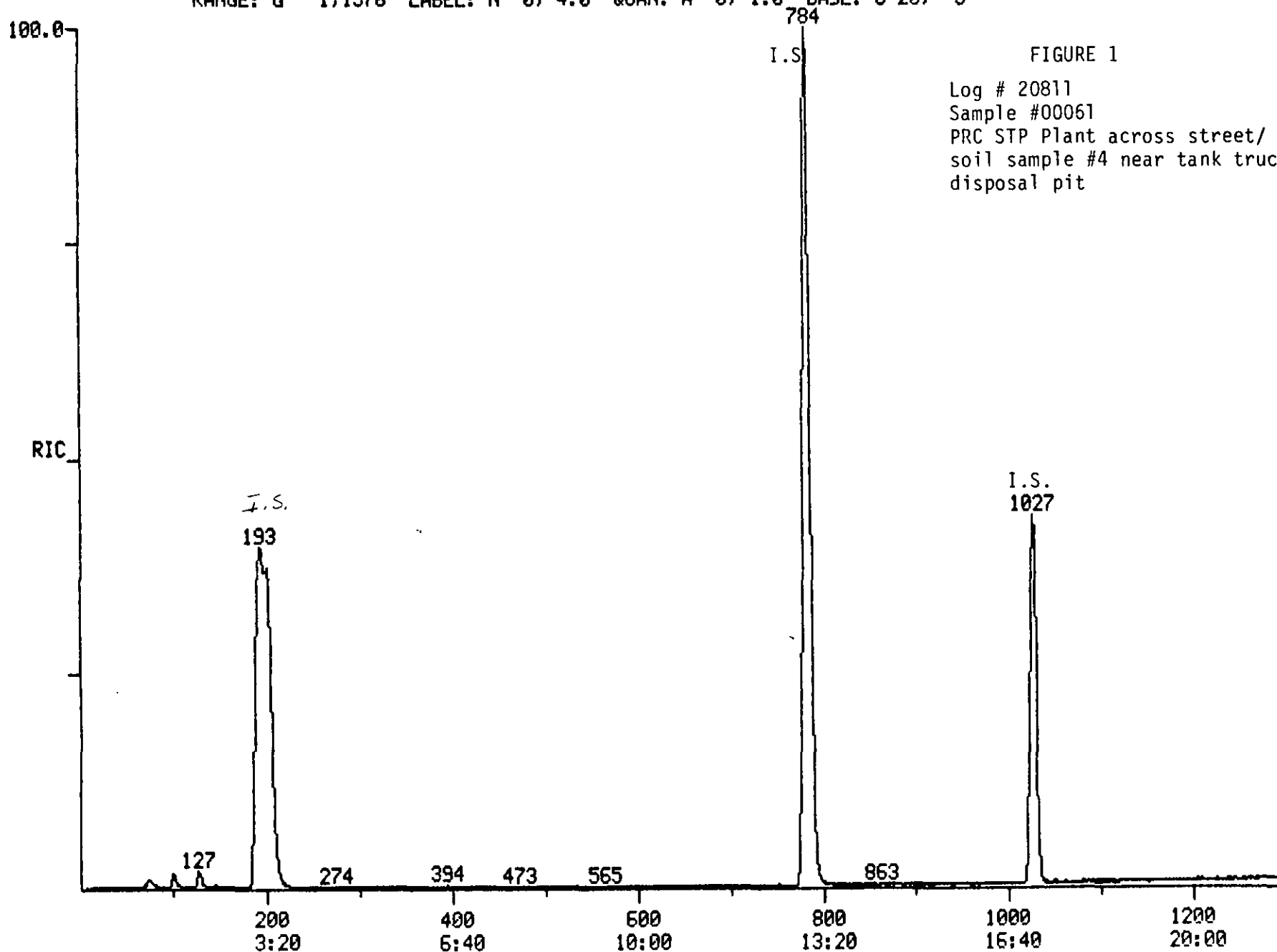


FIGURE 1

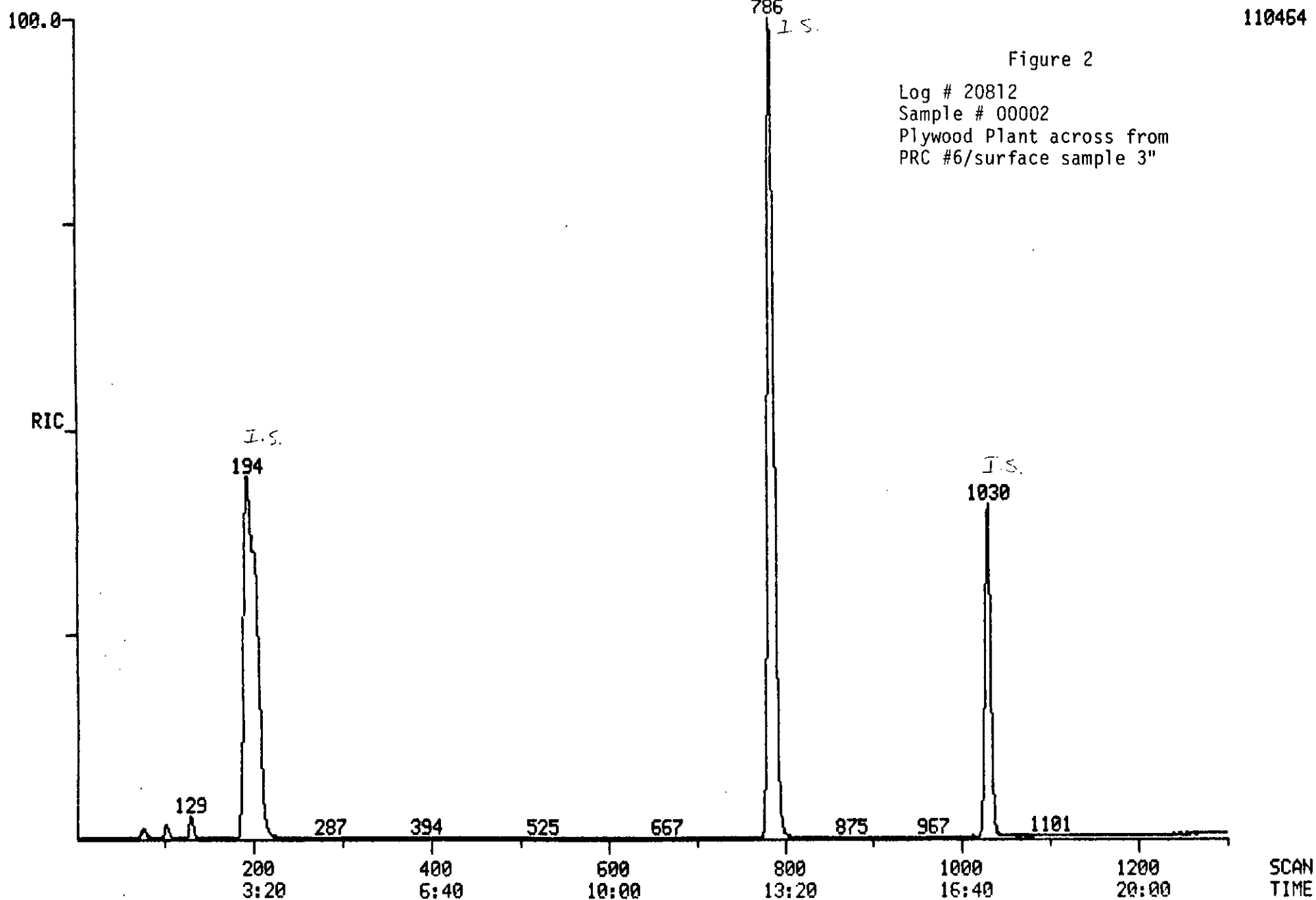
Log # 20811
Sample #00061
PRC STP Plant across street/
soil sample #4 near tank truck
disposal pit

RIC
08/12/82 13:15:00
SAMPLE: 00002 PLYWOOD PLANT ACROSS FROM PRC#6/ SURFACE SAMPLE 3"
RANGE: G 1.1337 LABEL: N 0, 4.0 QUAN: A 0, 1.0 BASE: U 20, 3

DATA: 20812 #1
CALI: 0812DJ #2

SCANS 1 TO 1300

110464

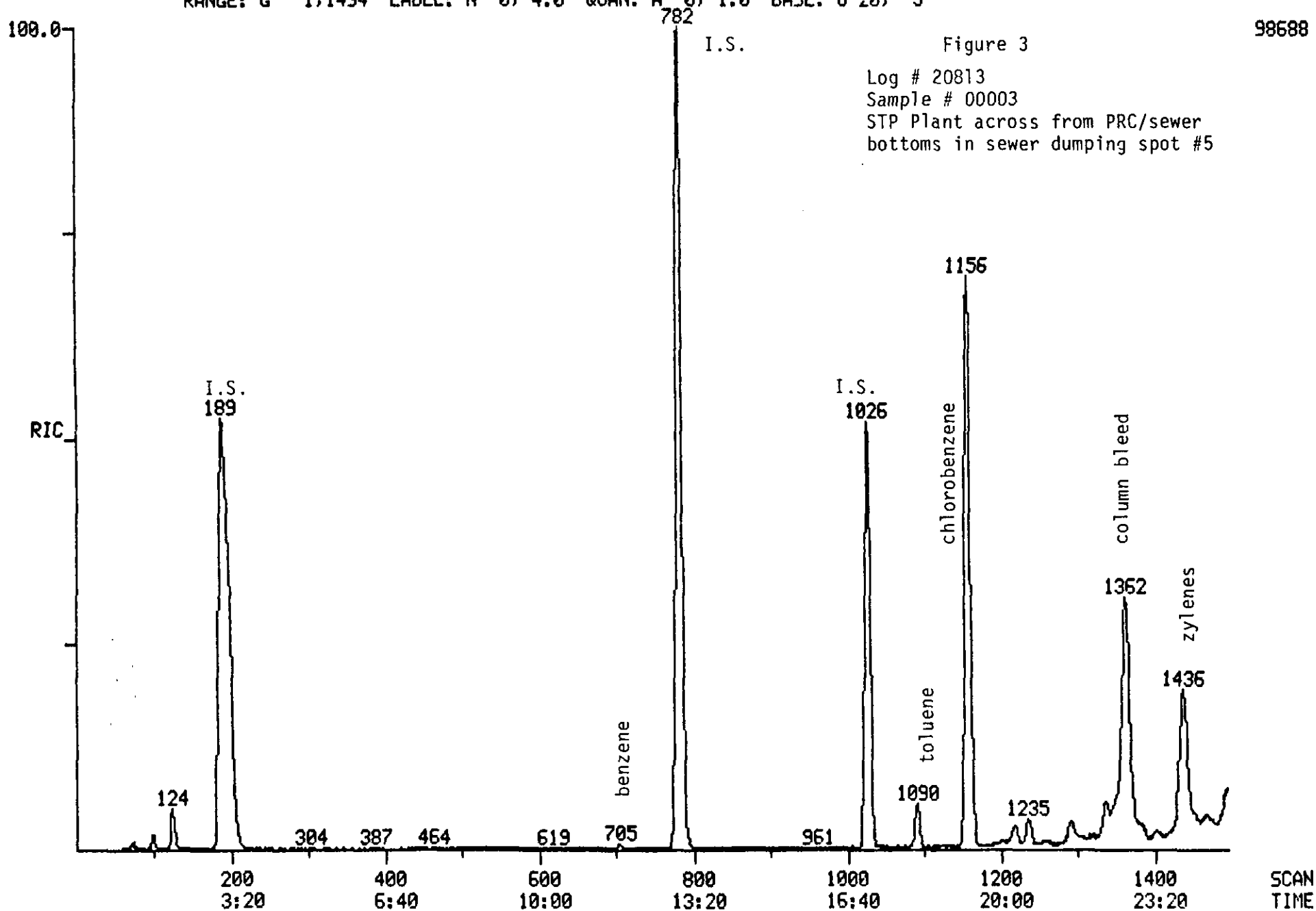


RIC
08/12/82 13:49:00
SAMPLE: 00003 STP PLANT ACROSS PRC SEWER BOTTOMS IN SEWER DUMP SPOT #5
RANGE: G 1,1494 LABEL: N 0, 4.0 QUAN: A 0, 1.0 BASE: U 20, 3

DATA: 20813 #1
CALI: 0812DJ #2

SCANS 1 TO 1494

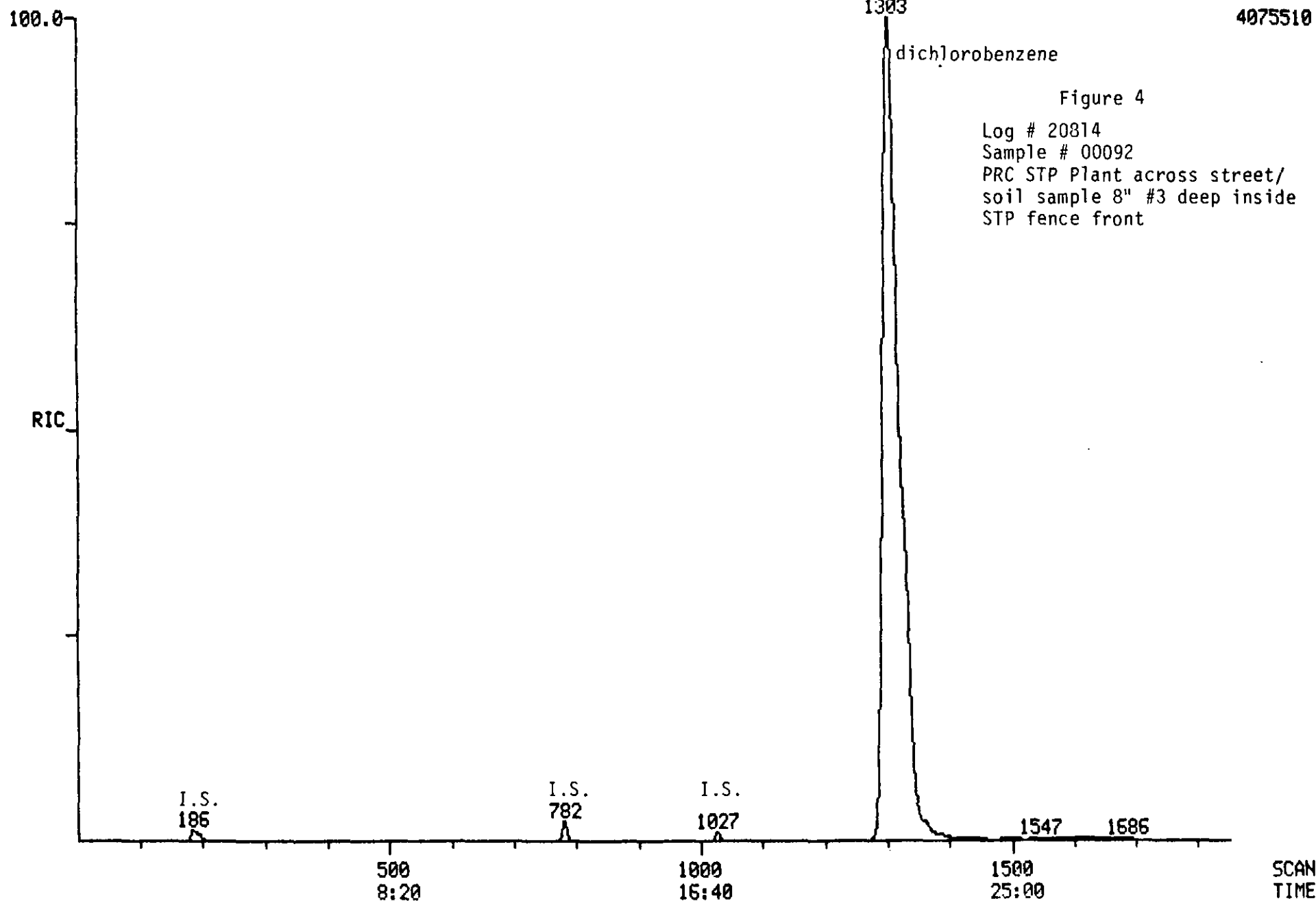
98688



RIC
08/12/82 14:24:00
SAMPLE: 00092 PRC STP PLANT ACROSS ST.SOIL SAMPLE#3 8"DEEP INSIDE STP FR
RANGE: G 1,1851 LABEL: N 0, 4.0 QUAN: A 0, 1.0 BASE: U 20, 3

DATA: 20814 #1
CALI: 08120J #2

SCANS 1 TO 1851



RIC
08/12/82 14:57:00
SAMPLE: 00090 PRC PLANT SOIL SAM. #2FRONT OIL STOR. TANK
RANGE: G 1.1347 LABEL: N 0, 4.0 QUAN: A 0, 1.0 BASE: U 20, 3

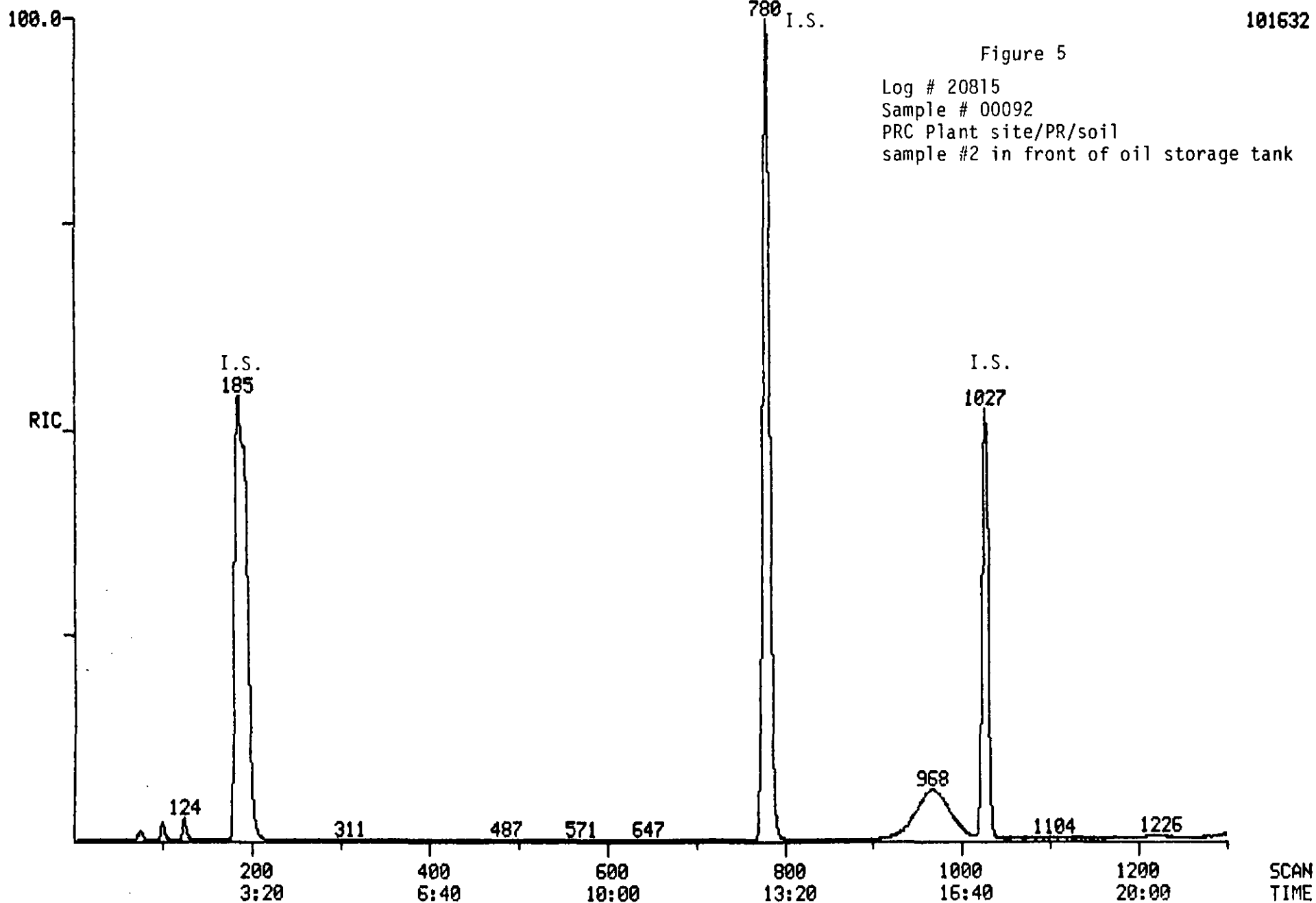
DATA: 20815 #1
CALI: 0812DJ #2

SCANS 1 TO 1300

101632

Figure 5

Log # 20815
Sample # 00092
PRC Plant site/PR/soil
sample #2 in front of oil storage tank



RIC
08/12/82 15:30:00
SAMPLE: 00062 PRC PLANT SITE SOIL SAM. #1 FRONT OF PLANT
RANGE: G 1,1369 LABEL: N 0, 4.0 QUAN: A 0, 1.0 BASE: U 20, 3

DATA: 20816 #1
CALI: 0812DJ #2

SCANS 1 TO 1300

99200

782
I.S.

Figure 6

Log # 20816
Sample # 00062
PRC Plant site Arecibo PR/
soil sample #1 front of plant

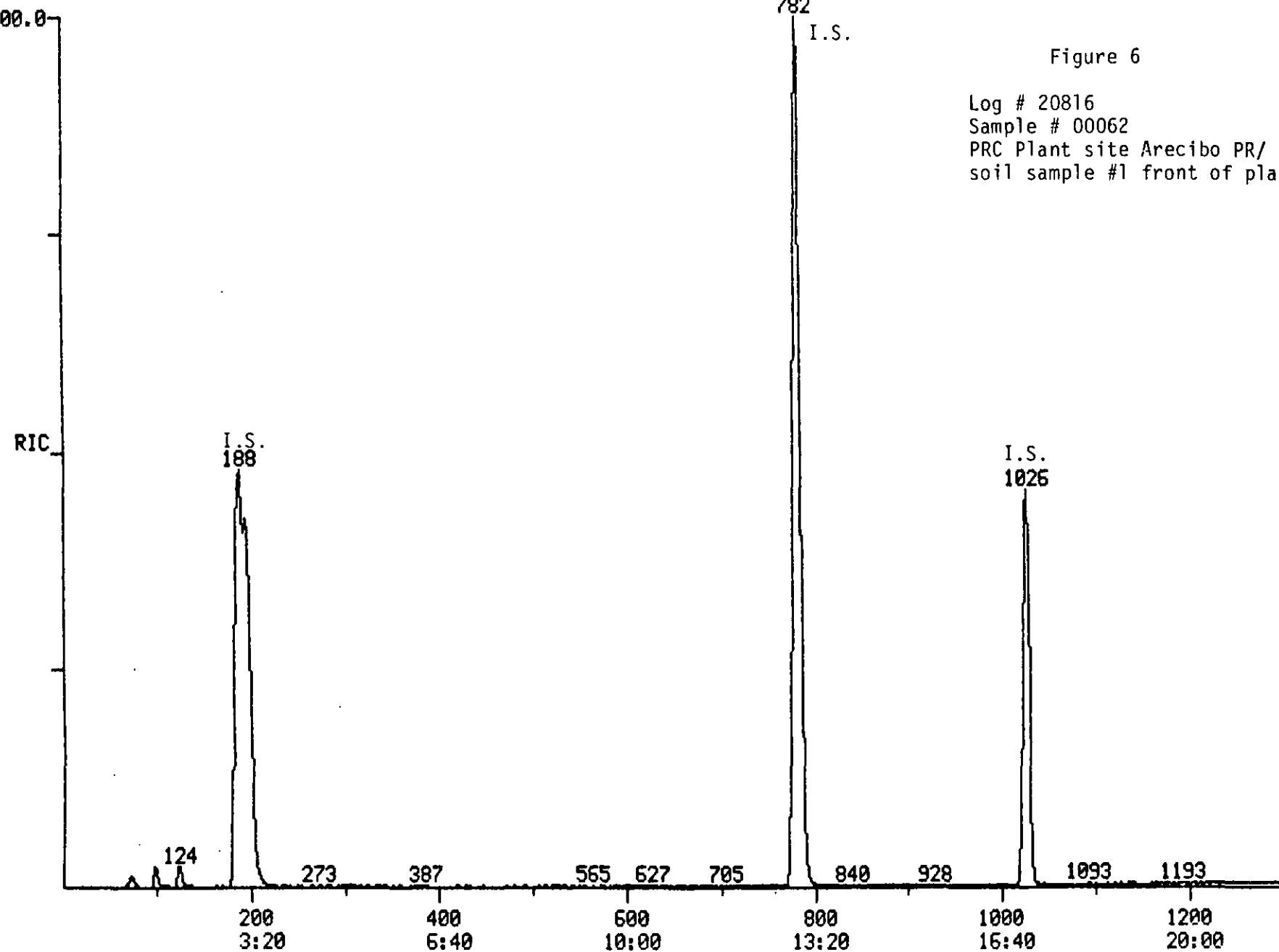


TABLE 2: METHOD PERFORMANCE DATA

Surrogate Recovery - GC/MS Data (QR20)

Chain of Custody Data Required for ETC Data Management Summary Reports

D8923

ETC Sample No.

Company

Facility

Sample Point

Date

Time

Elapsed
Hours

Compound	Amount Added ug	% Recovery	Control Limits *	
			Lower	Upper
VOLATILE FRACTION				
Bromochloromethane	.200	103	79	127
Benzene, d ₆	.150	116	63	122
Fluorobenzene	.150	123	74	122
1,4-Dichlorobutane	.200	76	75	117
Pentafluorobenzene	.150	133	58	124
Ethylbenzene, d ₁₀	.150	121	78	114
ACID FRACTION				
2-Fluorophenol	-	-	20	86
Pentafluorophenol	-	-	37	127
BASE/NEUTRAL FRACTION				
2-Fluorobiphenyl	80	78	62	122
1-Fluoronaphthalene	80	102	64	104
Nitrobenzene, d ₅	80	103	58	105
* Three Standard Deviations About the Mean.				

February 17, 1984

TABLE 2: METHOD PERFORMANCE DATA

GC/MS Tuning Data – Bromofluorobenzene (BFB) for Volatiles Analysis (QR21)

Chain of Custody Data Required for ETC Data Management Summary Reports

D8923

ETC Sample No.	Company	Facility	Sample Point	Date	Time	Elapsed Hours
----------------	---------	----------	--------------	------	------	---------------

m/z	Ion Abundance Criteria	Abundance (% Base Peak)
50	15-40% of the base peak	28
75	30-60% of the base peak	50
95	Base Peak, 100% relative abundance	100
96	5-9% of the base peak	8
173	Less than 1% of the base peak	<1
174	Greater than 50% of the base peak	67
175	5-9% of mass 174	5
176	Greater than 50% of the base peak	62
177	5-9% of mass 176	4

Date: 840204
Run No: >A0173
Spectrum No: 174
Analyst: R. Albert

February 27, 1984

TABLE 2: METHOD PERFORMANCE DATA

GC/MS Tuning Data - Decafluorotriphenylphosphine (DFTPP) for Base/Neutrals Analysis (QR23)

Chain of Custody Data Required for ETC Data Management Summary Reports

D8923

ETC Sample No.

Company

Facility

Sample Point

Date

Time

Elapsed
Hours

m/z	Ion Abundance Criteria	Abundance (% Base Peak)
51	30-60% of mass 198	60
68	Less than 2% of mass 69	<2
70	Less than 2% of mass 69	<2
127	40-60% of mass 198	44
197	Less than 1% of mass 198	<1
198	Base peak, 100% relative abundance	100
199	5-9% of mass 198	7
275	10-30% of mass 198	21
365	Greater than 1% of mass 198	2
441	Less than mass 443	10
442	Greater than 40% of mass 198	67
443	17-23% of mass 442	13

Date: 840223
Run No: G1058
Spectrum No: 192
Analyst: K. Weiner

Methodology for GC Analysis of Polychlorinated Biphenyls

The methods employed in the analysis of your water sample for polychlorinated biphenyls are established EPA methods taken from the "Manual of Analytical Methods for the Analysis of Pesticide Residues in Human and Environmental Samples," June, 1980.

The water method can be summarized as follows: A measured volume of sample, approximately 500 ml, to which sodium sulfate has been added is extracted with methylene chloride. The methylene chloride extract is dried and concentrated to approximately 1 ml. The concentrated extract is transferred to a silica gel column and eluted with hexane. The eluate is concentrated to a final volume of 1 ml and injected into a gas chromatograph equipped with a ⁶³Ni electron capture detector.

The GC operating parameters were as follows:

COLUMN

6' x 4 mm glass 1.5% SF-2250 & 1.95% SP-2401
Supelcoport 100/120 mesh

CARRIER FLOW

60 ml/min. Argon/Methane

COLUMN OVEN

220° C

INJECTOR TEMPERATURE

225° C

DETECTOR TEMPERATURE

325° C

METHODOLOGY

The methods employed in the analysis of your samples are both established EPA methods for priority pollutants in water and modified EPA procedure for priority pollutants in sediments and sludges. Gas chromatography combined with electron impact mass spectrometry (GC/MS) was used for this analysis.

1982 → For the analysis of the volatile organic compounds, EPA Method 624 (Federal Register, December 3, 1979; page 69532) was used. A September 1982 modification of EPA Method 624 which allows for the analysis of styrene was included. The method can be summarized as follows: Helium is bubbled through a 5-ml water sample contained in a specially designed purging chamber at ambient temperature. The purgeable volatile organic compounds are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and back flushed with helium to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer.

For the analysis of volatile organic compounds in sediments and sludges, methods taken from special report No. 1, "Development of Analytical Test Procedures for the Measurement of Organic Priority Pollutants in Sludges and Sediments" June 1979 were used. That method can be summarized as follows: 0.5 ml (0.5 grams) of sediment/sludge was transferred to a Tekmar purging chamber using a tipless disposable pipet. Five mls of reagent water and five mls of an internal standard water solution were added to the purging chamber. The mixture was purged and trapped following the same procedure used in Method 624 for water samples.

For the analysis of the target Base/Neutral priority pollutants, EPA Method 625 (Federal Register, December 3, 1979; page 69540) was used. The procedure includes a September 1982 modification to include the analysis of 3',3'-dichloro 4,4'-diamino dephenyl methane (MOCA). The method can be summarized as follows: A measured volume of sample, approximately 1-liter, was serially extracted with methylene chloride at a pH greater than 11 using a separatory funnel or a continuous extractor. The methylene chloride extract was dried and concentrated to a volume of 1 ml. The concentrate was injected into GC/MS systems set specifically for the separation and measurement of the priority pollutants.

For the analysis of target base neutrals and MOCA in sediment and soils, EPA Method 625 (previously referenced) was applied to an aqueous extract of the sample obtained by using the EP Toxicity extraction procedure found in "RCRA Test Methods For Evaluating Solid Wastes- Physical/Chemical Methods", SW846, May 1980. The EP Toxicity extraction procedure can be summarized as follows: 100g of sediment or sludge are stabilized at pH 5 using 0.5 Normal acetic acid solution. The mixture is diluted to a final volume of 2 liters with distilled water. The entire sample is tumble shaken for 24 hours followed by positive pressure filtration at 75 PSI. The filtrate is extracted and analyzed using EPA Method 625.

For the analysis of PCB's in water and sediment, methods taken from "Manual of Analytical methods for the analysis of Pesticides in Human and Environmental Samples." EPA 600-8-30-038 were used. The water method can be summarized as follows: A measured volume of water sample, approximately 500 ml, to which sodium sulfate has been added, is extracted with methylene chloride. The methylene chloride extract is dried and concentrated to approximately 1 ml. The concentrated extract is transferred to a silica gel column and eluted with hexane. The eluate is concentrated to a final volume of 1 ml and injected into a gas chromatograph equipped with a ⁶³Ni electron capture detector. 4

The soil method can be summarized as follows: A weighed amount of air dried sample, approximately 2 grams, is soxhlet extracted for 5 hours with 1:1 acetone/hexane solution. The extract is dried and concentrated to approximately 3 ml. The concentrated extract is transferred to a silica gel column and eluted with hexane. The eluate is concentrated to a final volume of 1 ml and injected into a gas chromatograph equipped with a ⁶³Ni electron capture detector.

Qualitative Identification of the target priority pollutants was performed initially using the relative retention times, the relative abundance of three characteristic ions, and their ratios. The entire mass spectrum was reviewed before an identification was recorded. Quantitative analysis was performed using an internal standard with a single characteristic ion.

GC/MS

was the
the only

my file
P. H. H. G.
G. H. H. G.

Quality Assurance/Quality Control Procedures (QA/QC)

ETC bases its quality assurance protocols on the following government guidelines:

- "Handbook for Analytical Quality Control in Water and Wastewater Laboratories", EPA-600/4-79-019, March 1979; ~ E2
- National Enforcement Investigation Center Policies, and Procedures manual; EPA-330/9/79/001-R, October 1979; - 3
- the recommended guidelines for EPA Methods 624 and 625. (Federal Register, December 3, 1979, pp. 69532-69559); and
- "Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples," EPA 600/8-80-036, June 1980.
- "Determination of 2,3,7,8-TCDD in Soil and Sediment" EPA, Region VII, Kansas City, September 1983.

However, we have modified our protocols to provide a higher level of QA/QC than the guidelines require. For example, we analyze a higher than required number of quality control samples and we pay especially careful attention to the certification of the "reference standard" compounds we use in analysis. Below are listed the key QA/QC elements for the methods we used.

Analysis of Volatile Organic Compounds (EPA Method 624)

- Long as the results*
- Each batch of 12 samples consists of 9 customer samples (at maximum), one blank sample, one spiked blank, one spiked sample and one replicate sample. This amounts to a 30% quality control factor.
 - Three surrogate compounds are added to each sample in the batch of 13.
 - At least one blind quality control sample is introduced to the laboratory for analysis for each hundred samples analyzed.
 - Each GC/MS is checked and retuned, if necessary, every 8 hours to ensure that its performance on bromofluorobenzene (BFB) meets the EPA criteria.
 - A calibration curve for quantitation is prepared using a mixture of Volatile Organic Priority Pollutant "standards" at a minimum of 3 different concentrations and using a mixture of 3 internal standards at a constant concentration.
 - The calibration curve is verified with a mixture of priority pollutant standards every 8 hours.
 - Results are compared to the acceptance criteria given in Method 624; any that do not meet the criteria are re-analyzed.

Analysis of Organic Compounds Extracted in Acid or Base/Neutral Solutions (EPA Method 625)

- Each batch of 20 samples consists of 16 customer samples (at maximum), one blank sample, one spiked blank, one sample spiked with the priority pollutant standard mixture and a duplicate customer sample. This amounts to a 20% quality control factor.
- Five surrogate compounds are added to each sample in the batch of 20.
- At least one blind quality control sample is introduced to the laboratory for analysis for each hundred samples analyzed.

- Each GC/MS is checked and retuned, if necessary, every eight hours to ensure that its performance on decafluorotriphenylphosphine (DFTPP) meets the EPA criteria.
- A calibration curve for quantitation is prepared using a mixture of standards composed of either the Organic Acid or Base/Neutral Extractable Compounds at a minimum of 3 concentrations and using 2,2'-difluorobiphenyl as an internal standard.
- The calibration curve is verified with a mixture of priority pollutant standards every eight hours.
- Results are compared to the acceptance criteria given in Method 625; any that do not meet the criteria are re-analyzed.

Analysis of Metals

All Samples

- New standards are prepared for each batch of samples.
- Normal calibration is performed using a blank sample and four standards that have been through the sample preparation procedure. A regression analysis is used to construct the calibration curve.
- For each sample analysis that requires the use of the "method of additions" technique, a three point calibration is performed using U.S. EPA "Methods for Chemical Analysis of Water and Wastes, 1979". Results are obtained using linear regression analysis. Any results with a coefficient of correlation below 0.990 are considered erroneous, necessitating raw data editing or sample re-analysis.
- In constructing the normal calibration curves the lowest concentration levels we use are values greater than or equal to 5 times the Instrumental Detection Limit (IDL).
- All calibration standards are analyzed in duplicate, at a minimum.
- Independent reference standards are used to check the accuracy of calibration standards.
- A check standard is analyzed every ten samples to validate the normal calibration curve.

Homogeneous Samples (except for Mercury analysis)

Samples are analyzed in batches of 30 or less. For batches in which the sample matrices are homogeneous, the QC program is a minimum of 42% and consists of analyzing:

- 3 Replicates;
- 2 Replicate spikes;
- 2 Replicate independent reference standards;
- 8 Calibration standards (processed using the sample preparation method);
- 2 Blanks (processed using the sample preparation method);
- 4 Calibration standards (without sample preparation); and
- 1 Blank (without sample preparation).

Heterogeneous Samples (except for Mercury analysis)

Samples are analyzed in batches of 30 or less. For batches in which the sample matrices are heterogeneous, the QC program is a minimum of 65% and consists of analyzing:

- each of the 30 customer samples in duplicate;
- 4 Replicates;
- 4 Replicate spikes;
- 2 Replicate independent reference standards;
- 8 Calibration standards (processed using the sample preparation method);
- 2 Blanks (processed using the sample preparation method);
- 4 Calibration standards (without sample preparation); and
- 1 Blank (without sample preparation).

Analysis of Mercury:

To analyze samples for mercury we group them by matrix in batches of 20 or less. Our QC program is a minimum of 66% and consists of analyzing:

- each of the 20 customer samples in duplicate;
- 3 Replicates;
- 2 Replicate spikes;
- 2 Replicate independent reference standards;
- 10 Calibration standards (processed using the sample preparation method); and
- 2 Blanks.

Analysis of Pesticides, Herbicides and PCB's (EPA Method 608)

*{ not the method used - does it
not have a quality/QA control*

Pesticide, herbicide and PCB samples are grouped in batches of 16 customer samples or less according to the type of analysis to be performed. The QC program for each of these three types of analyses is a minimum of 20% and consists of analyzing:

- 1 blank sample;
- 1 spiked blank sample;
- 1 replicate sample;
- 1 replicate spiked sample; and
- 1 blank QC sample for at least each 100 samples analyzed.

The GC instruments are tuned daily to meet performance criteria in Method 608. Because Method 608 lacks data acceptance criteria, ETC has developed its own upper and lower quality control limits. When a test result falls outside the control limits, the test is re-run.

What is the control limit

Analysis of Phenols

Phenols are analyzed using a Technicon AutoAnalyzer II GT.

- Absorbance of full scale standards must be within +/- 25% of nominal absorbance.
- Duplicate calibration standards at four different concentrations are run with each batch of customer samples.
- At least one intersample standard is run for each 20 customer samples.
- Gain and carryover standards are analyzed at the end of each run.

Chain-of-Custody

The chain-of-custody procedure is part of our quality assurance protocol. We believe our chain-of-custody record fully complies with the legal requirements of federal, state and local government agencies and of the courts of law. The record covers:

- labeling of sample bottles, packing the Sample Shuttle and transferring the Shuttle under seal to the custody of a shipper;
- outgoing shipping manifests;
- the chain-of-custody form completed by the person(s) breaking the Shuttle seal, taking the sample, resealing the Shuttle and transferring custody to a shipper;
- incoming shipping manifests;
- breaking the Shuttle's reseal;
- storing each labeled sample bottle in a secured area;
- disposition of each sample to an analyst or technician; and
- the use of the sample in each bottle in a testing procedure appropriate to the intended purpose of the sample.

The record shows for each link in this process:

- the person with custody; and
- the time and date each person accepted or relinquished custody.

Appendix A1

Gas Chromatographic Spectral Data

for

Quantitated Compounds

- 1) A reconstructed gas chromatogram for each sample analysed by a GC instrument.
- 2) A reconstructed gas chromatogram for the appropriate standard compounds analyzed with the same GC under the same operating conditions.

APPENDIX B-2

TYPICAL ETC REPORT

October 4, 1984

TECHNICAL REPORT

for

**WHITEMAN, OSTERMAN & HANNA
99 WASHINGTON AVE.
ALBANY, NY 12210**

PRIVILEGED & CONFIDENTIAL
PREPARED AT THE REQUEST
OF LEGAL COUNSEL

Chain of Custody Data Required for ETC Data Management Summary Reports

D8923 WHITEMAN, OSTERMAN, & HANNA WOHHICKGWM W137A1001A1 840130 1530

ETC Sample No.	Company	Facility	Sample Point	Date	Time	Elapsed Hours
D8923	WHITEMAN, OSTERMAN, & HANNA	WOHHICKGWM	W137A1001A1	840130	1530	

ENVIRONMENTAL TESTING and CERTIFICATION CORPORATION

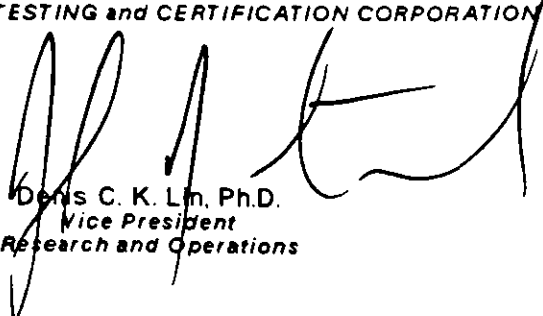

Denis C. K. Lin, Ph.D.
Vice President
Research and Operations

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Table 1: Results and Quality Assurance Data

Table 2: Method Performance Data

Methodology

QA Protocol

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INTRODUCTION

This report contains the analytical results on your water samples, submitted on February 2, 1984. It is designed to satisfy the needs of your people at various levels in your organization.

The results we obtained on your sample are presented in a tabular format immediately after this introduction. Included with the sample results, the quality assurance data on your specific sample are tabulated to verify the validity of the results obtained. The quality assurance data include those obtained on the blank, the spiked blank, the replicate and the spiked sample (commonly known as matrix spike). Also presented in the quality assurance data report is the verification of the proper functioning of the instruments used. The gas chromatograms and/or mass spectra generated in the analysis of your sample are included in the Appendix of this report. The chain of custody record for your sample is included at the end of this report.

The established methods we used in the analysis of your sample are described in the Methodology section after the Results. In the analysis we followed a rigidly controlled Quality Assurance Protocol. This Protocol is described after the Methodology section.

We hope our report format is useful in assisting you to obtain pertinent information on your sample.

RESULTS

The results obtained on your sample and the accompanying quality assurance data are listed in Table 1.

The data on the recovery of the surrogates in your sample and the certification of the GC/MS systems used in the analysis of your sample are listed in Table 2.

The sample extract was qualitatively analyzed by GC/ECD for the presence of Aroclors. If present, the Aroclors were quantitated.

The sample chromatograms were compared qualitatively to chromatograms of all 7 Aroclors - 1016, 1221, 1232, 1242, 1248, 1254, and 1260 for matching peaks. Quantitation was based on a three point calibration curve for Aroclor 1248. The data are tabulated in Table 1; this quality assurance data obtained on the Method Blank, Replicate and Matrix Spike analyses. The methodology and quality assurance protocol follow Table 1. Sample and standard chromatograms are included in the appendix of this report.

The Chain-of-Custody Record on your sample is also included at the end of this Report.

MAY 8, 1984

TABLE 1: QUANTITATIVE RESULTS and QUALITY ASSURANCE DATA									
--	--	--	--	--	--	--	--	--	--

Volatile Compounds - GC/MS Analysis Data (QR01)

Chain of Custody Data Required for ETC Data Management Summary Reports

D8923 WHITEMAN, DSTERMAN & HANNA WOHHICKGLM W137A1001A1 840130 1530

Elapsed
Hours

[illegible]

ETCENVIRONMENTAL
TESTING and CERTIFICATION

JUN 26, 1984

TABLE 1: QUANTITATIVE RESULTS and QUALITY ASSURANCE DATA
BASE/NEUTRAL COMPOUNDS - GC/MS ANALYSIS DATA (QR03)

Chain of Custody Data Required for ETC Data Management Summary Reports

D8923 WHITEMAN, OSTERMAN & HANNA WOHMICKGMM W137A1001A1 840130 1530

ETC Sample No. Company Facility Sample Point Date Time Elapsed Hours

NPDES Number	Compound	Results		QC Replicate		QC Blank and Spiked Blank			QC Matrix Spike		
		Sample Concen. ug/l	MDL ug/l	First ug/l	Second ug/l	Blank Data ug/l	Concen. Added ug/l	% Recov	Unspiked Sample ug/l	Concen. Added ug/l	% Recov
	bis(2-Ethylhexyl)phthalate	ND	10	ND	ND	ND	30	108	ND	30	55
	Butyl benzyl phthalate	-		ND	ND	ND	30	18	ND	30	20
	Diethyl phthalate	-		ND	ND	ND	30	3	ND	30	3
	Dimethyl phthalate	-		ND	ND	ND	30	0	ND	30	0
	Di-n-butyl phthalate	-		ND	ND	ND	30	28	ND	30	51
	Di-n-octyl phthalate	ND	10	ND	ND	ND	30	102	ND	30	58
	Moca	ND	10	ND	ND	ND	60	94	ND	60	58

A EPA published Method Detection Limit.

B Not Determinable.

C ETC estimated method detection limit.

March 30, 1984

TABLE 1: QUANTITATIVE RESULTS

Conventional Analysis Data (QR12)

Chain of Custody Data Required for ETC Data Management Summary Reports

D8923

WHITEMAN, OSTERMAN & HANNA

WOHHICKGWM W137A1001A1 840130 1530

ETC Sample No.

Company

Facility

Sample Point

Date _____

Line

Elapsed
Hours

Parameter	Results		
	Sample Measure	MDL	Units of Measure
Chemical Oxygen Demand (COD)	3	2	mg/l
pH	5.1		std
Specific Conductance	310	100	umhos/cm
Sulfate as SO ₄	13	2	mg/l
Total Organic Carbon	1.2	1	mg/l

Chain of Custody Data Required for ETC Data Management Summary Reports

D8923

WHITEMAN, OSTERMAN & HANNA

WOHHICKGUM

W137A1001A1 840130 1530

Elapsed

ETC Sample No.

Company

Facility

Sample Point

Däte

7 June

Hours

[illegible]

ETCENVIRONMENTAL
TESTING and CERTIFICATION

MAR 12, 1984

TABLE 1: QUANTITATIVE RESULTS and QUALITY ASSURANCE DATA**Aroclors - GC Analysis Data (QR14)**

Chain of Custody Data Required for ETC Data Management Summary Reports

D8923 WHITEMAN, OSTERMAN & HANNA WOHHICKGMM W137A1001A1 840130 1530

ETC Sample No.

Company

Facility

Sample Point

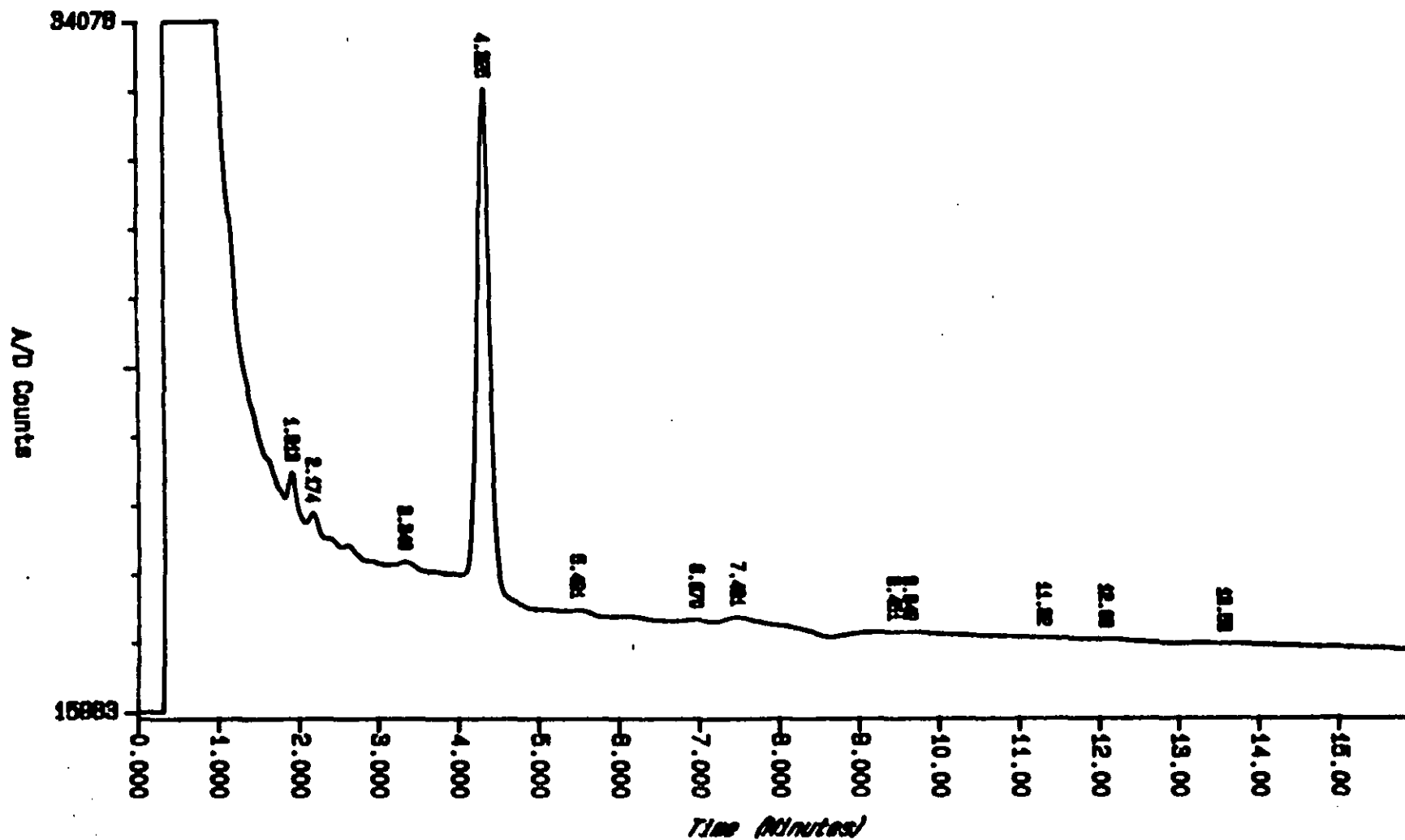
Date

Time

Elapsed
Hours

NPDES Number	Compound	Results		QC Replicate		QC Blank and Spiked Blank			QC Matrix Spike		
		Sample Concen. ug/l	MDL ug/l	First ug/l	Second ug/l	Blank Data ug/l	Concen. Added ug/l	% Recov	Unspiked Sample ug/l	Concen. Added ug/l	% Recov
	Aroclor 1242	ND	10	ND	ND	ND	0	-	-	-	-
	Aroclor 1254	ND	10	ND	ND	ND	0	-	-	-	-
	Aroclor 1260	ND	10	ND	ND	ND	0	-	-	-	-
	Aroclor 1248	ND	10	ND	ND	ND	20	97	ND	20	104
	Aroclor 1232	ND	10	ND	ND	ND	0	-	-	-	-
	Aroclor 1221	ND	10	ND	ND	ND	0	-	-	-	-
	Aroclor 1016	ND	10	ND	ND	ND	0	-	-	-	-

* MDL calculated for each sample matrix.



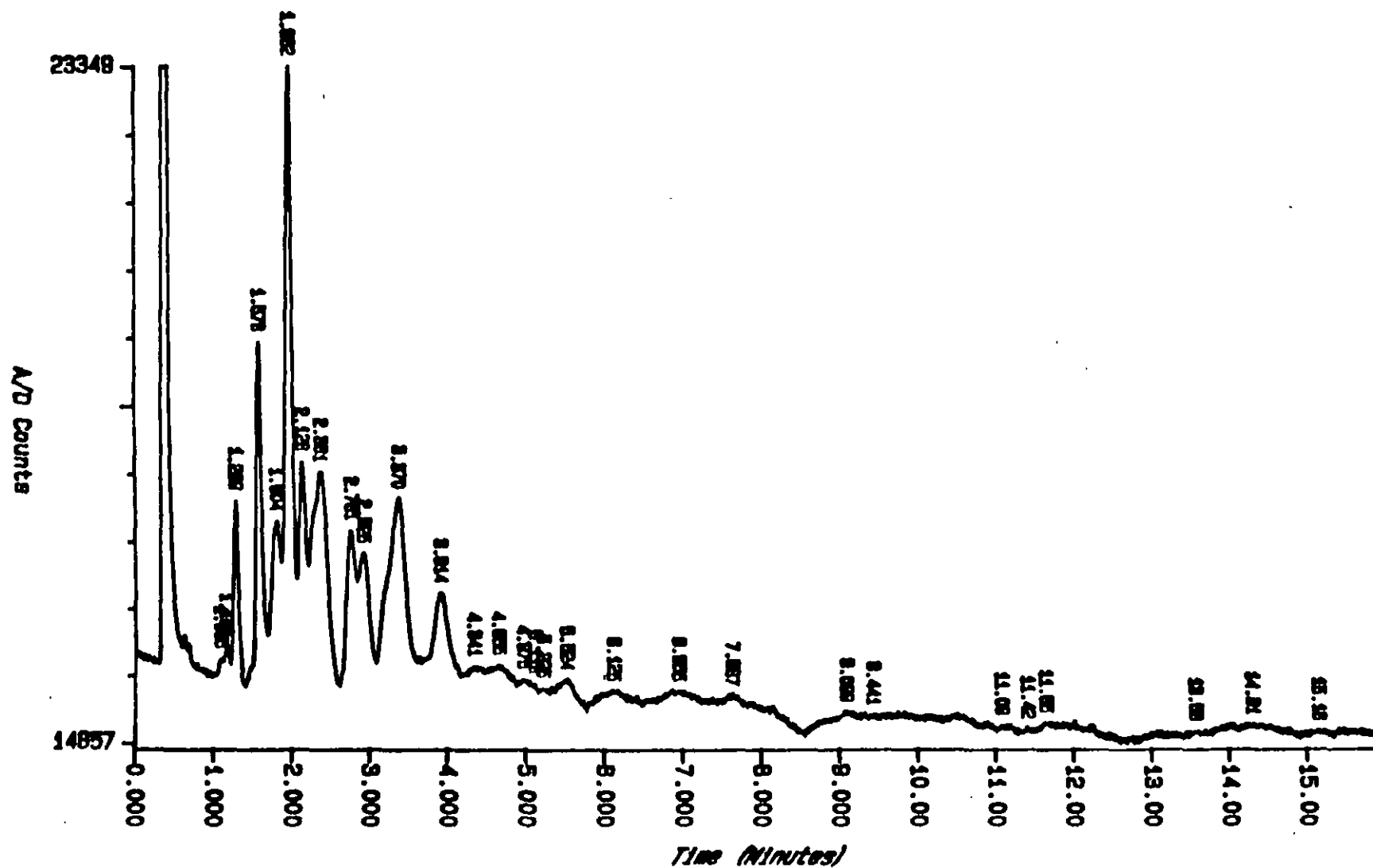
Sample: D8923

Injected at 18:45:28 ON FEB 22, 1984

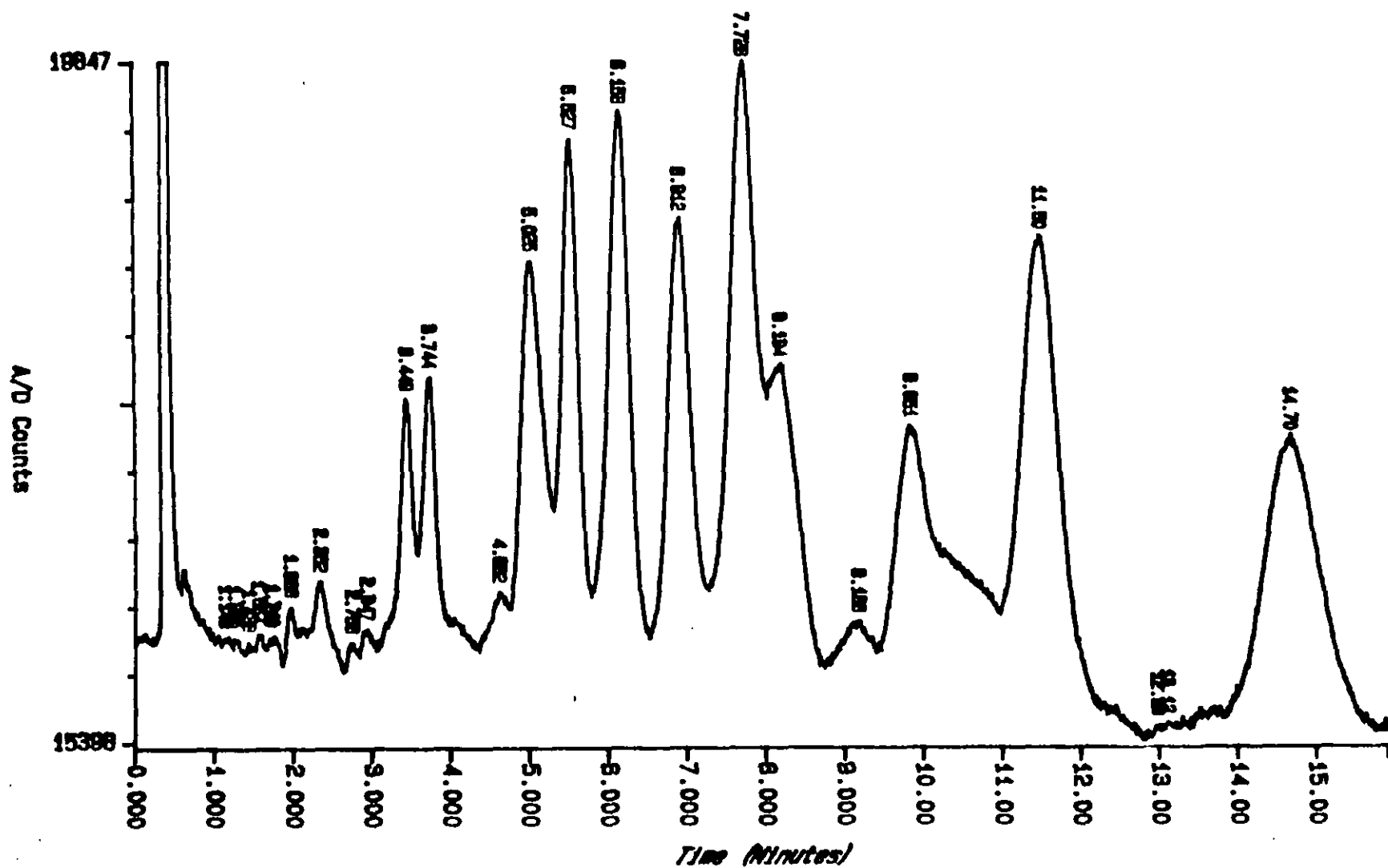
Raw File: D8923B

Proc File: D8923A

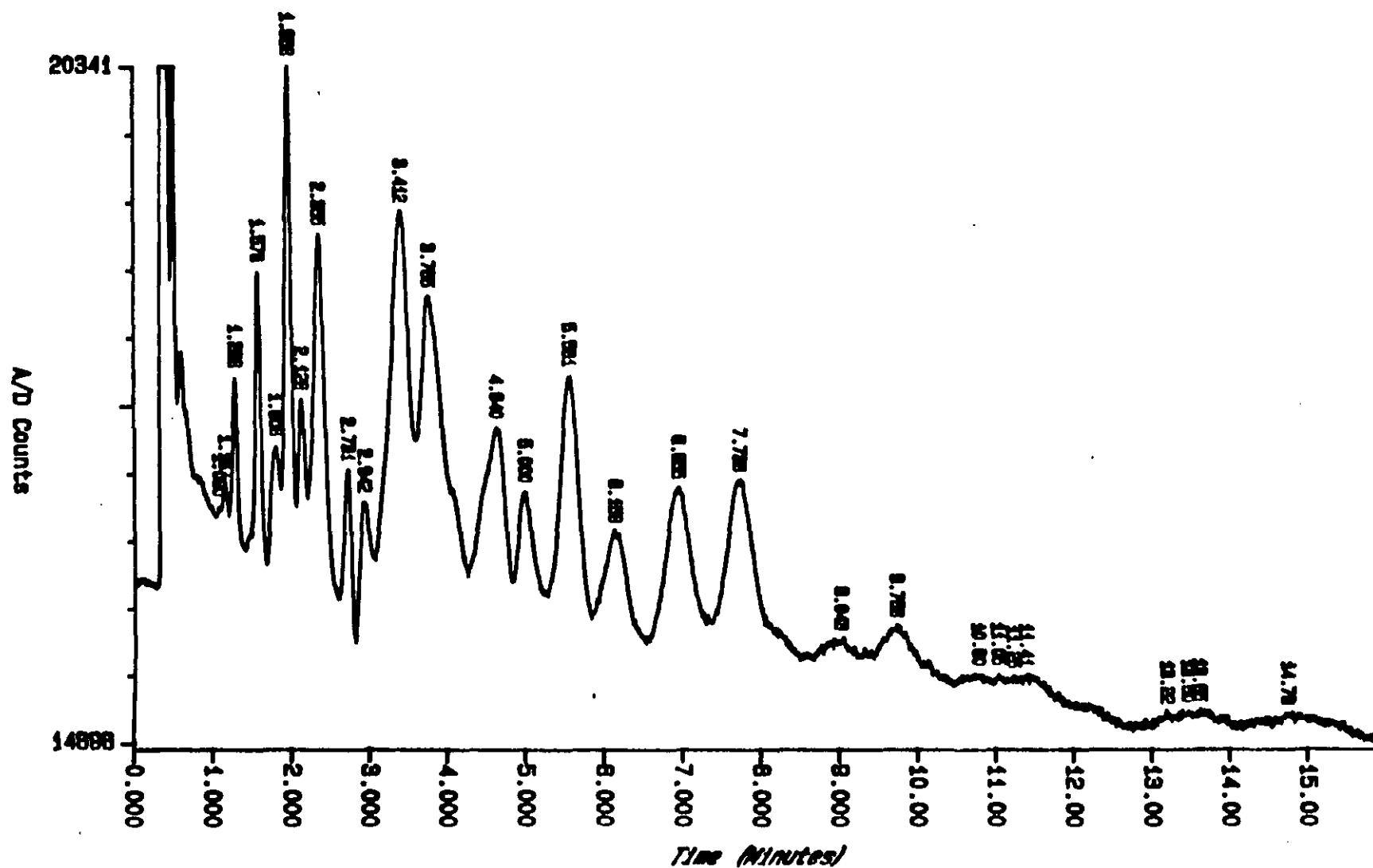
Method: PCBB5



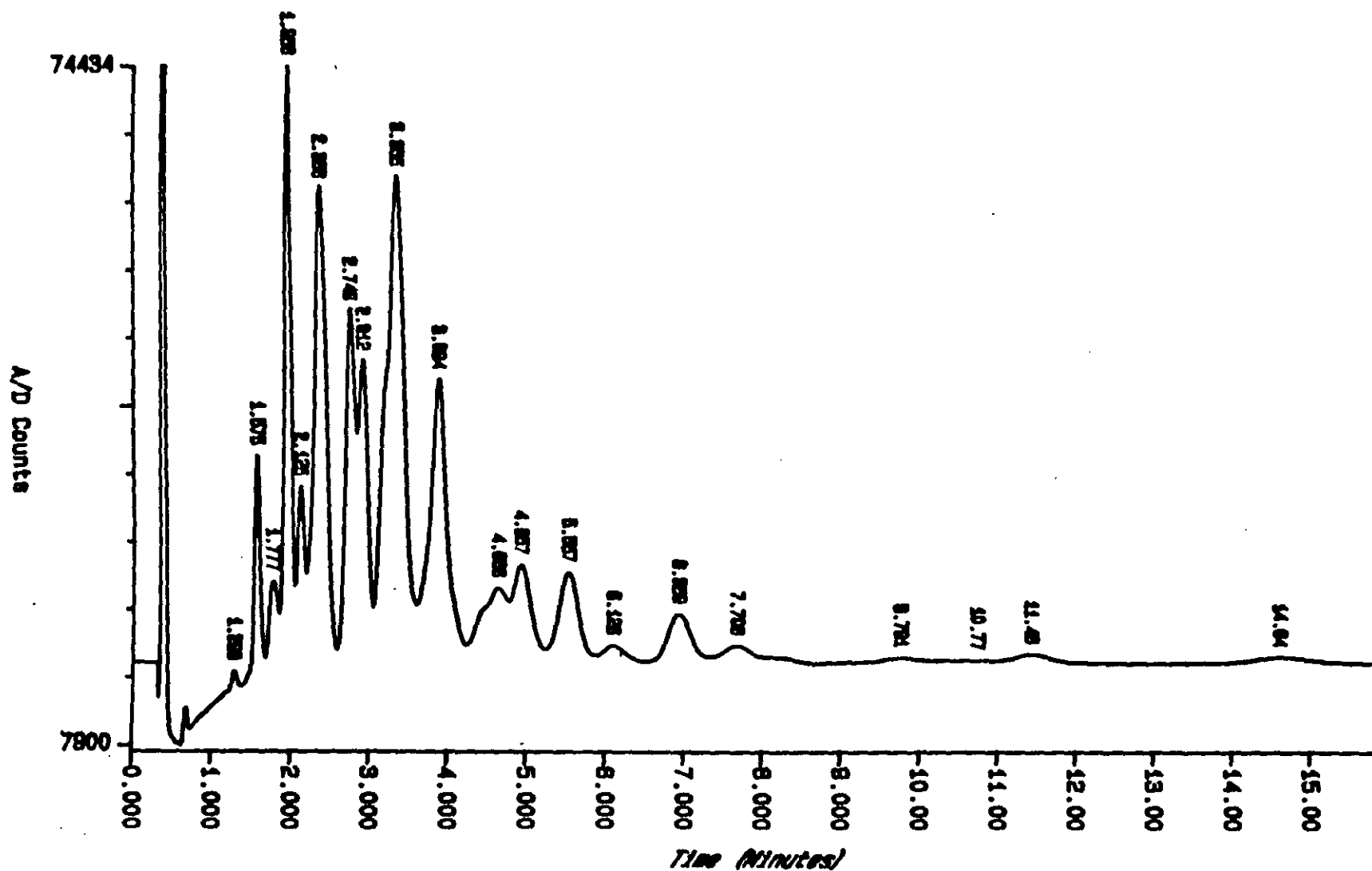
Sample: A1242 STD0.5 Injected at 17:57:10 ON MAR 8, 1984
Raw File: R8032 Proc File: P8032 Method: PCB85



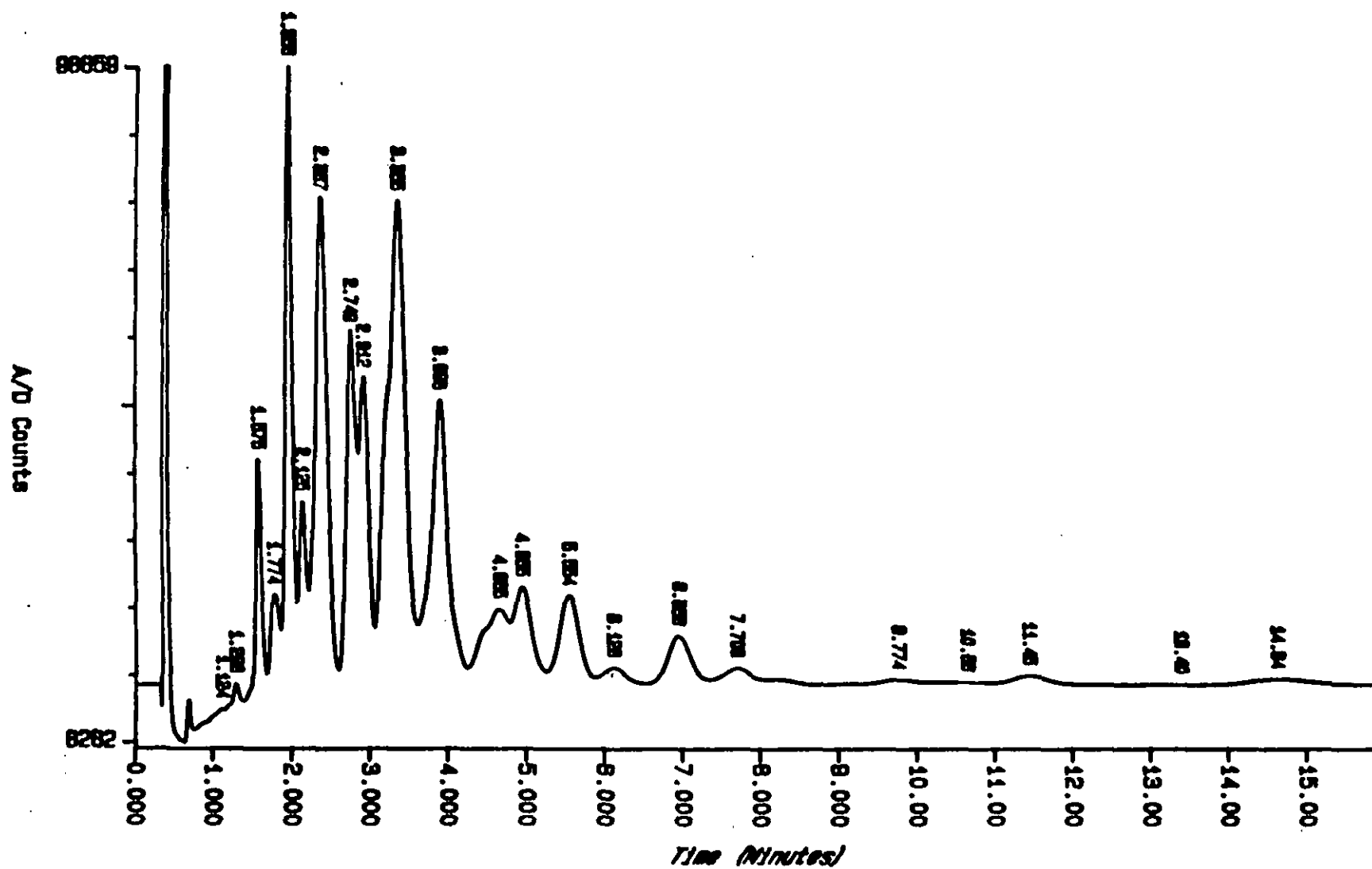
Sample: A1280 STD0.5 Injected at 17:21:28 ON MAR 8, 1984
Raw File: R8030 Proc File: P8030 Method: PCBB5



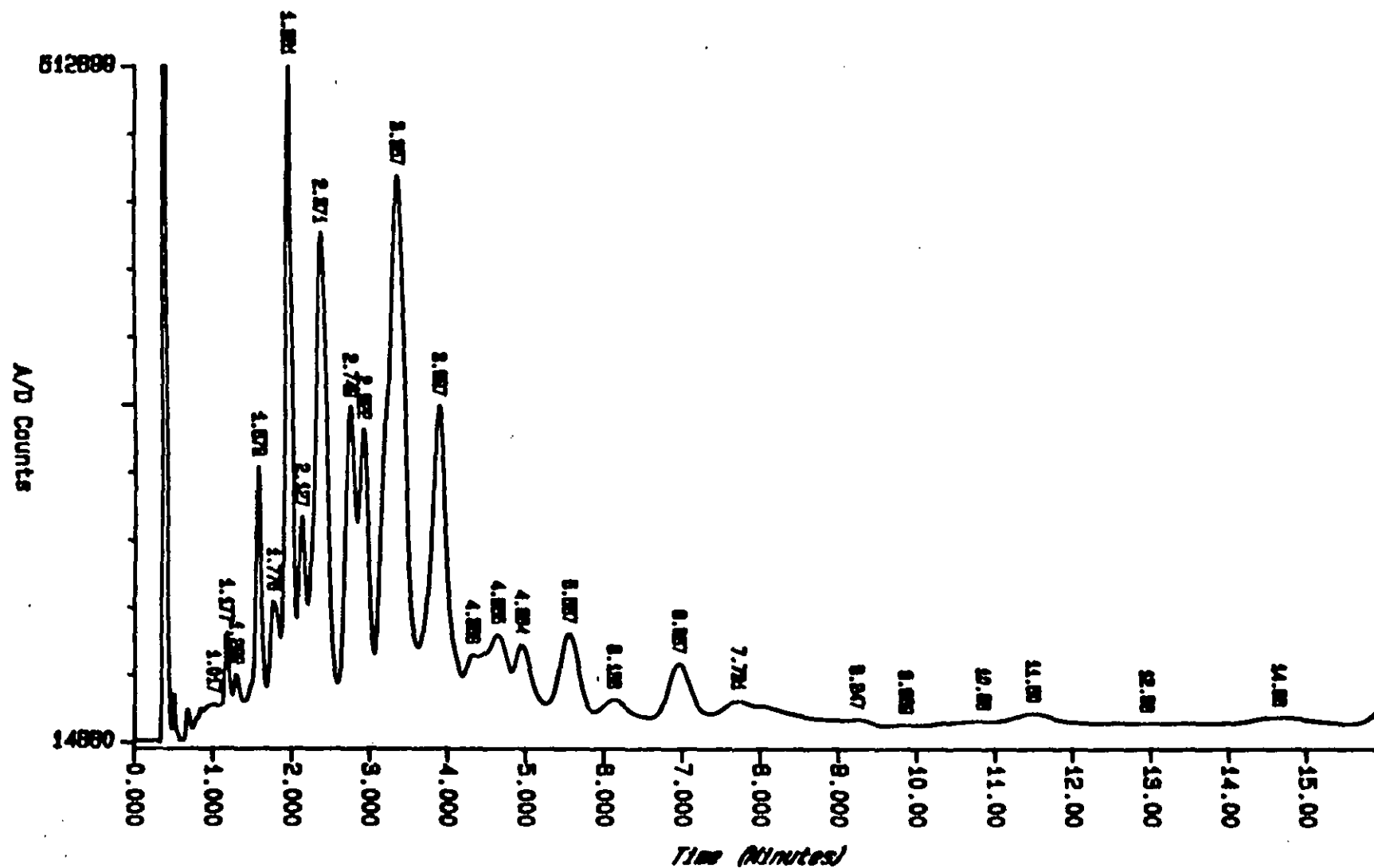
Sample: A1254 STD0.5 Injected at 17:39:17 ON MAR 8, 1984
Raw File: R8031 Proc File: P8031 Method: PCB85



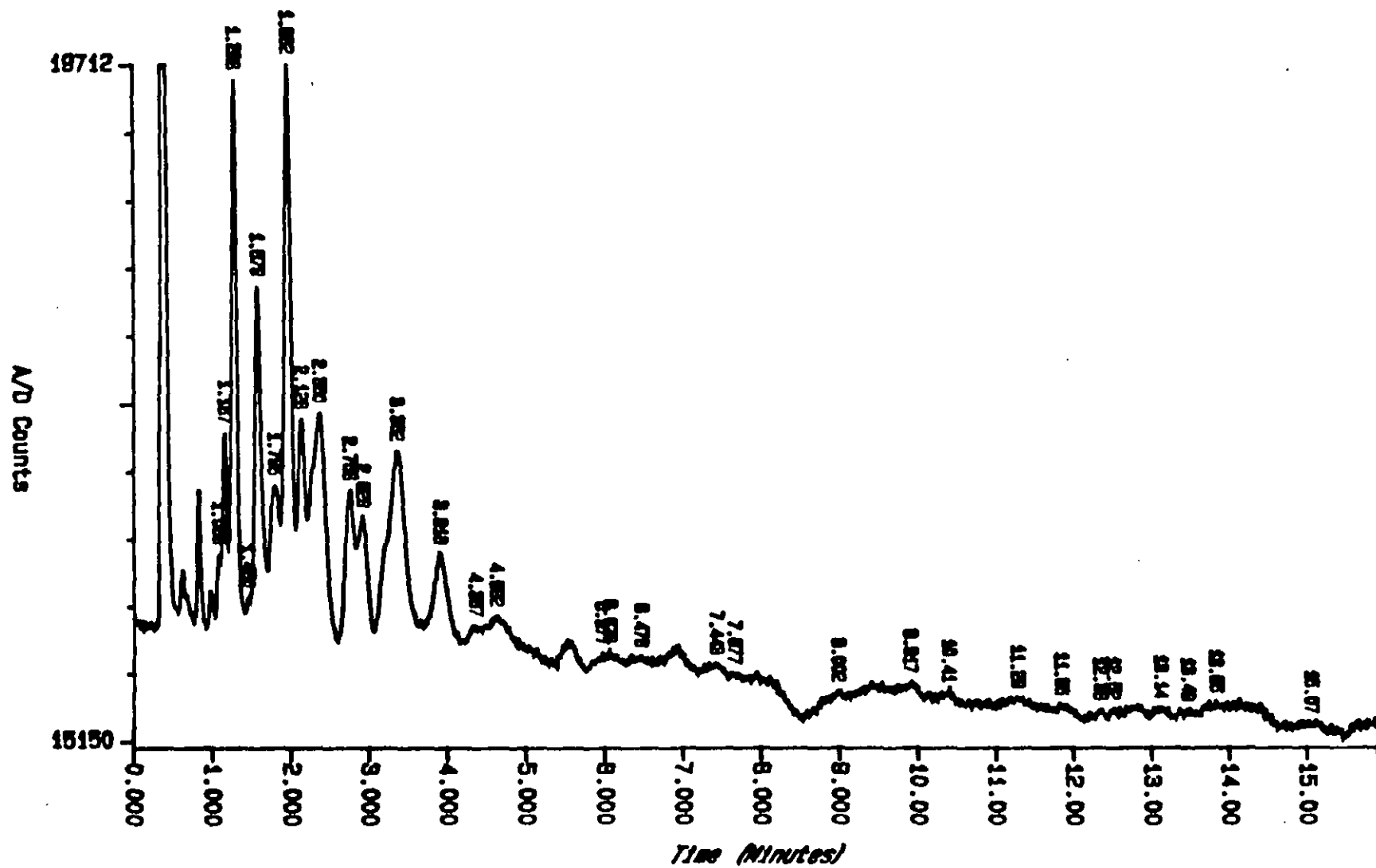
Sample: A1248 STD0.5 Injected at 15:05:51 ON FEB 22, 1984
Raw File: R7728 Proc File: P7728 Method: PCB85



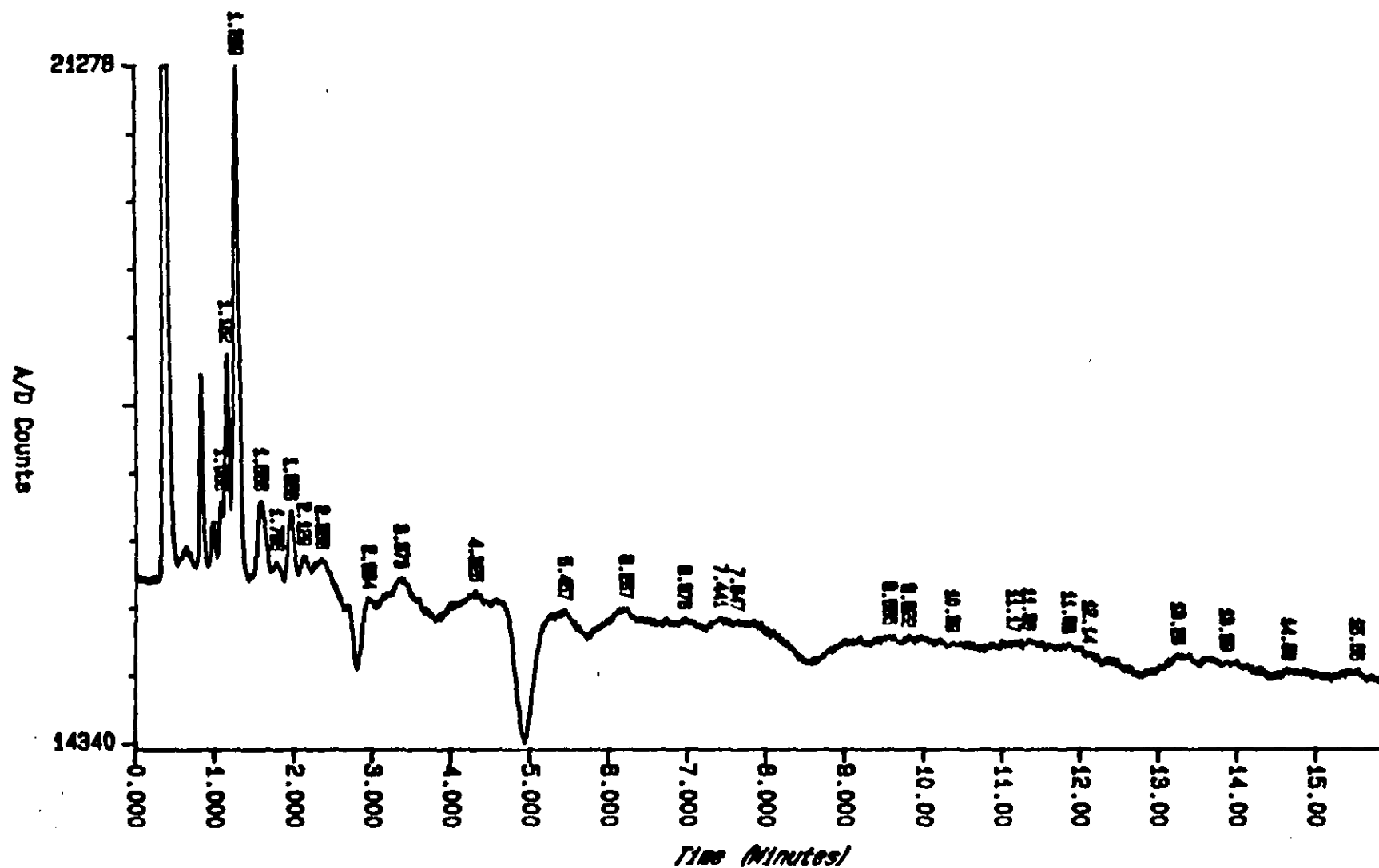
Sample: A1248 STD1.0 Injected at 15:23:25 ON FEB 22, 1984
Raw File: R7729 Proc File: P7729 Method: PCB85



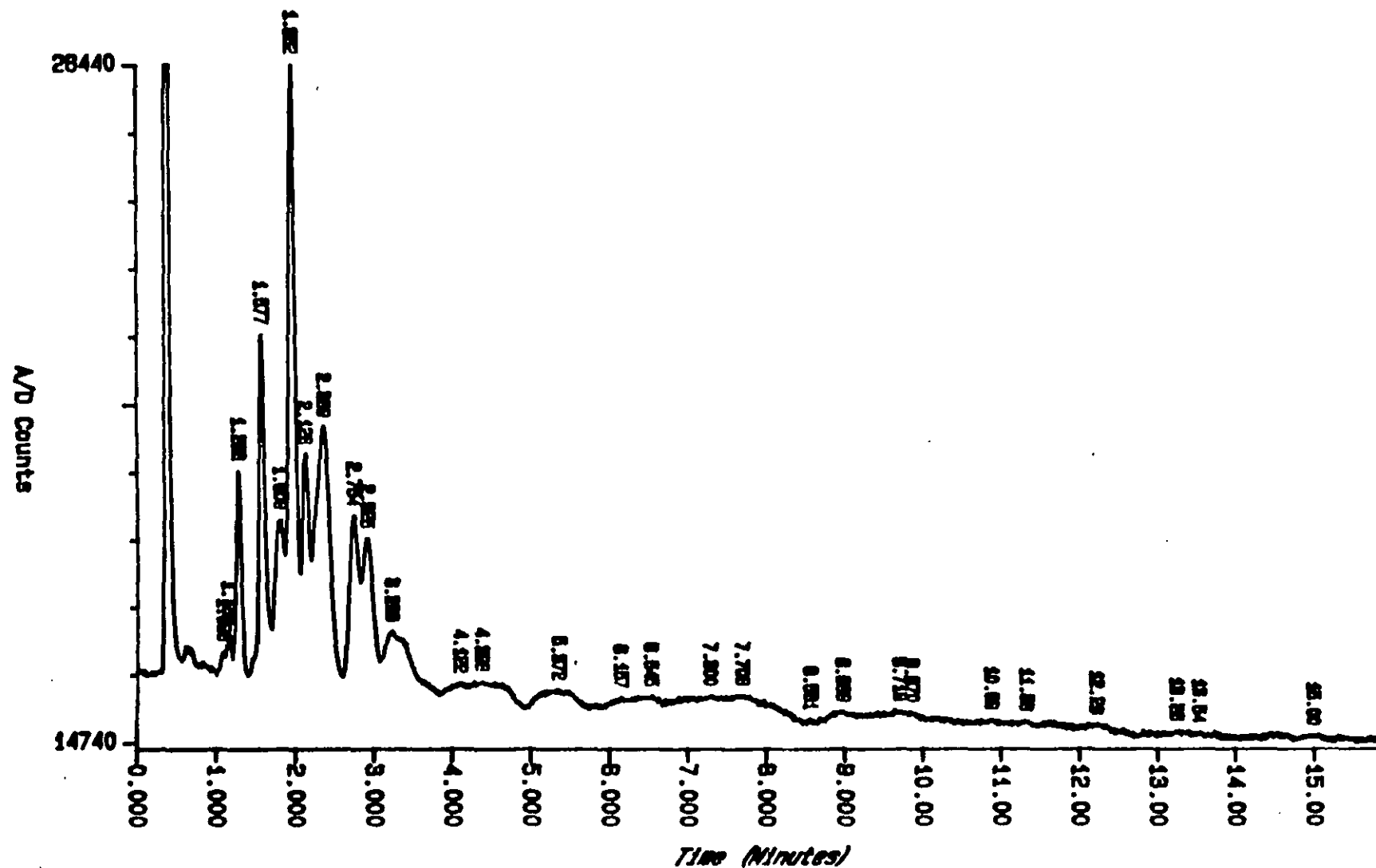
Sample: A1248 STD5.0 Injected at 15:47:18 ON FEB 22, 1984



Sample: A1232 STD0.5 Injected at 18:15:04 ON MAR 8, 1984
Raw File: R8033 Proc File: P8033 Method: PCBB5



Sample: A1221 STD0.5 Injected at 18:32:59 ON MAR 8, 1984
Raw File: R8034 Proc File: P8034 Method: PC885



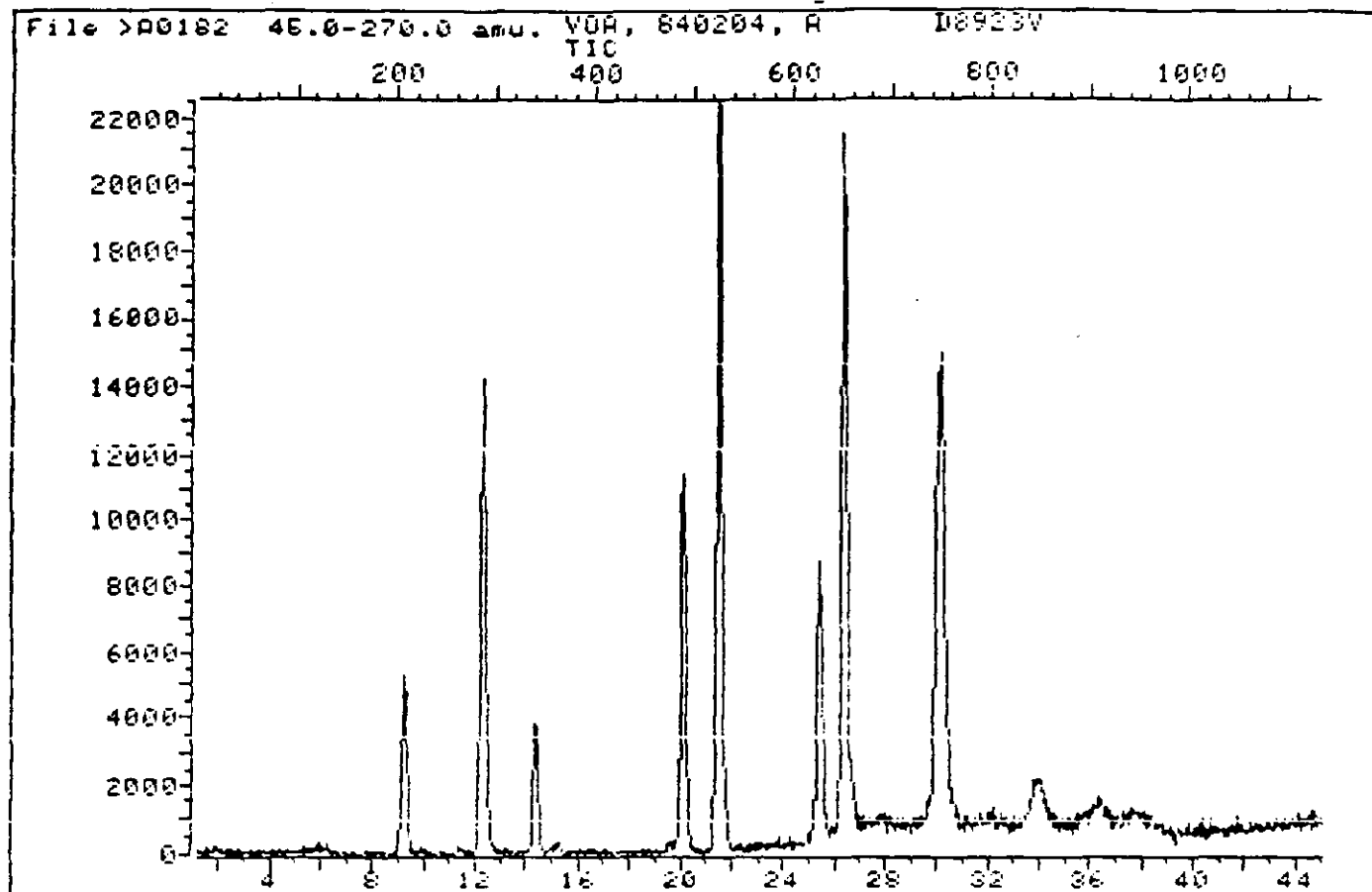
Sample: A1018 STD0.5 Injected at 18:50:59 ON MAR 8, 1984
Raw File: R8035 Proc File: P8035 Method: PCBB5

Appendix A
Mass Spectral Data
for
Quantitated Compounds

- 1) A total ion chromatogram for each sample analysed by a GC/MS instrument.
- 2) A mass spectrum and a reference spectrum for each priority pollutant compound detected in the sample.

**THE
FOLLOWING
DOCUMENTS
WERE OF
POOR QUALITY**

**IMAGE
DATA**



Data File: >A0182::U1

Name: VOA, 840204, A

Misc: D8923V

Id File: VOA

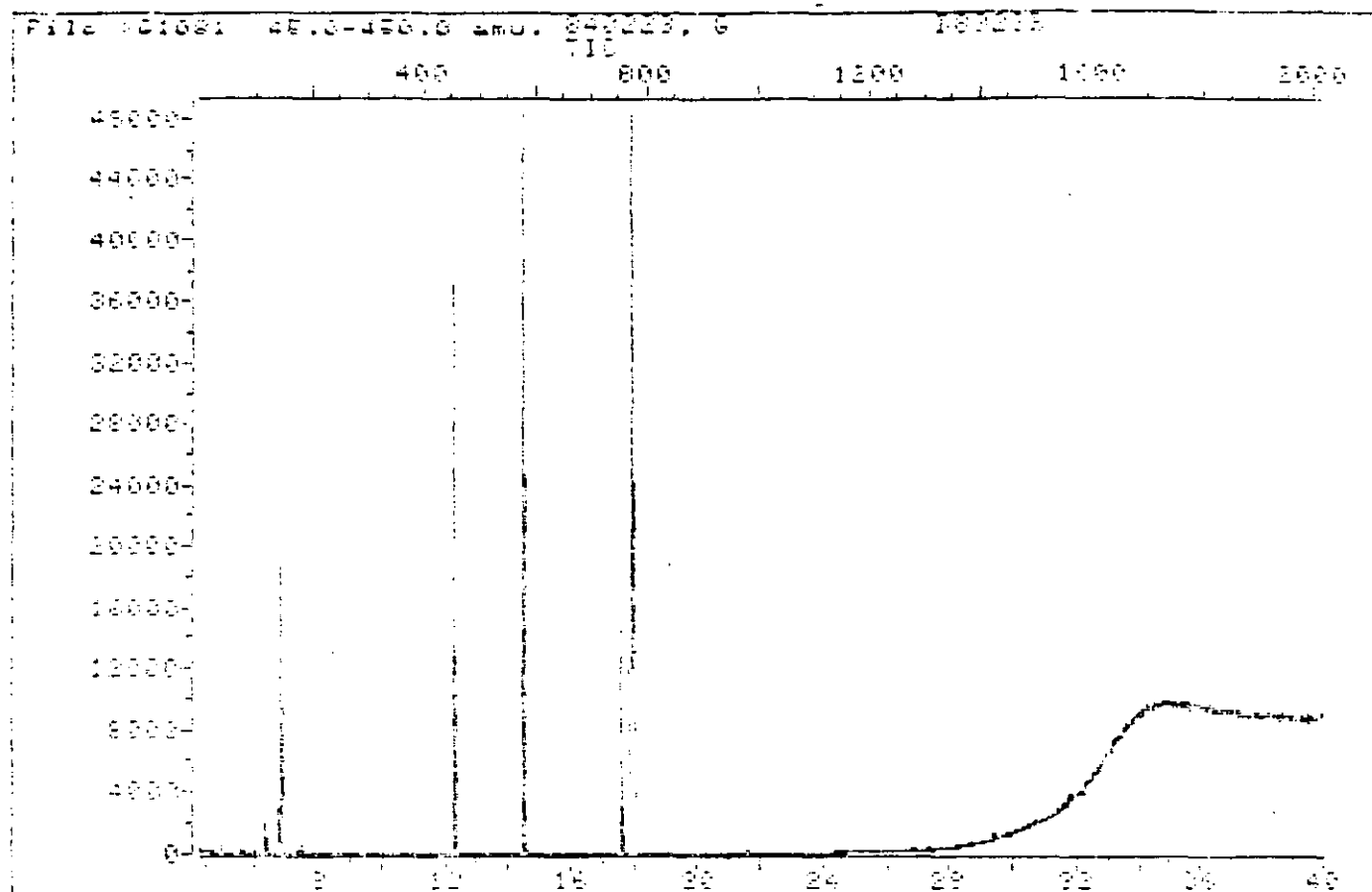
Title: DFILE, PURGEABLE PRIORITY POLLUTANTS, A

Last Calibration: 840205 00:57

Operator ID: RA0157

Quant Time: 840205 02:44

Total Ion Chromatogram



Date: 11/18/80
 File: 801081
 Name: 849227

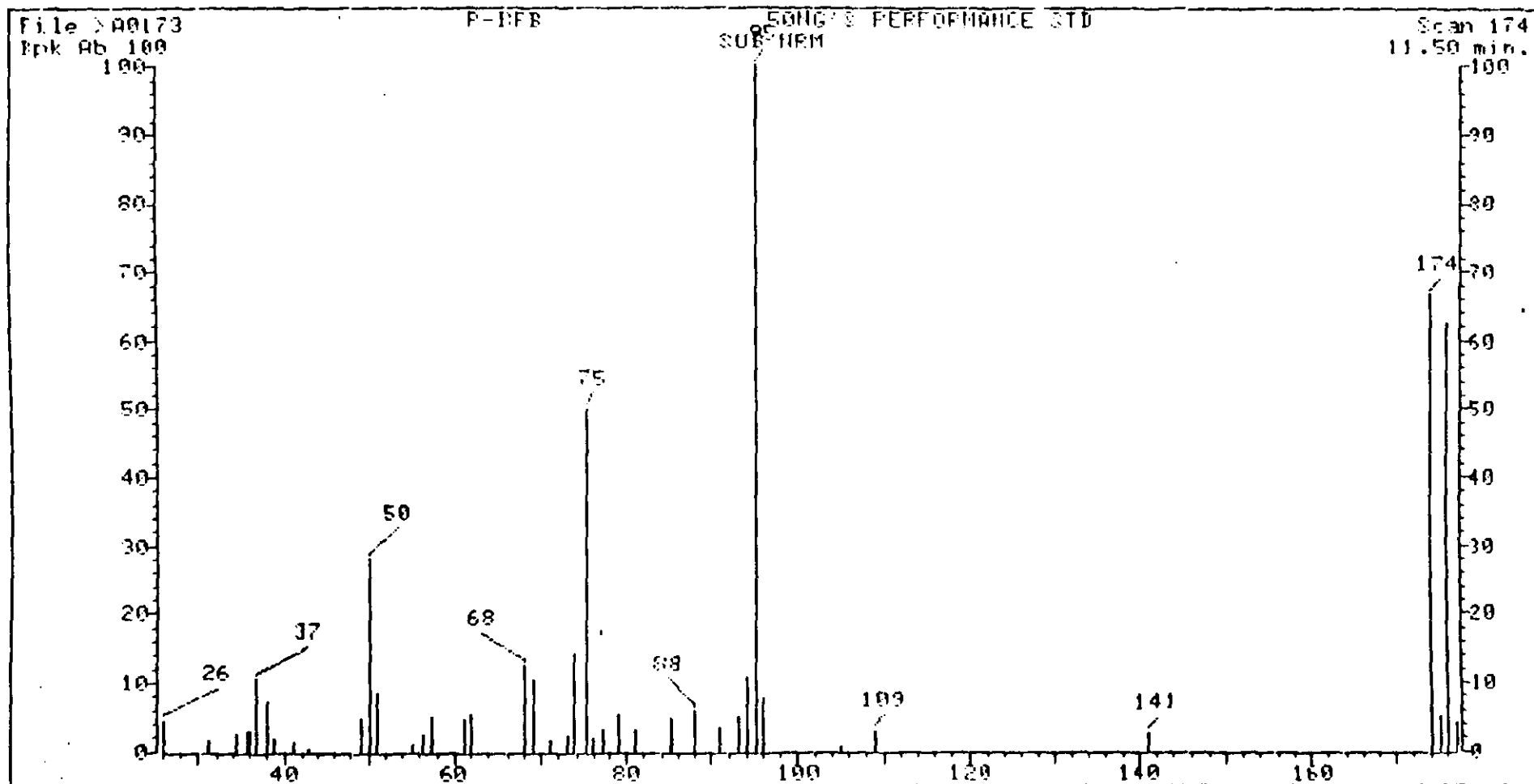
In File: 801081
 In File: 801081 45.3-458.8 LmU. 849227, G
 Last Calibration: 07/02/80 09:18

Version: II: 11/01/80
 Quant Time: 849227 16.07

Appendix B

Mass Spectral Data
for
Calibration Compounds

- 1) If the sample analysis included the determination of purgeable organic compounds then a mass spectrum for 4-bromofluorobenzene (BFB) is included. This data was used in the instrument calibration protocol on the day of analysis.
- 2) If the sample analysis included the determination of non-purgeable organic compounds then a mass spectrum for decafluorotriphenylphosphine (DFTPP) is included. This data was used in the instrument calibration protocol on the day of analysis.



>A0173
174

P-BFB
SUB NRM

SONG'S PERFORMANCE STD

File: >A0173 Scan #: 174 Retn. time: 11.50

m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.	m/z	Int.
26.00	4.40	43.05	.26	68.05	12.52	78.95	5.35	96.05	7.86
31.00	1.30	49.05	4.75	69.05	10.45	81.05	3.02	105.00	.60
34.55	2.42	49.95	27.98	71.05	1.38	85.15	4.75	109.00	2.76
35.75	2.76	51.05	8.29	73.05	1.99	88.05	5.87	141.10	2.42
35.95	2.76	55.05	.78	73.95	14.16	91.05	3.28	173.95	66.58
36.85	10.62	56.05	2.33	75.05	49.91	93.05	4.84	174.95	5.01
37.95	7.08	57.05	4.92	75.95	1.73	94.05	10.54	175.95	62.35
38.85	1.90	61.05	4.75	77.05	2.94	95.05	100.00	176.95	4.06
40.95	1.21	61.95	5.18						

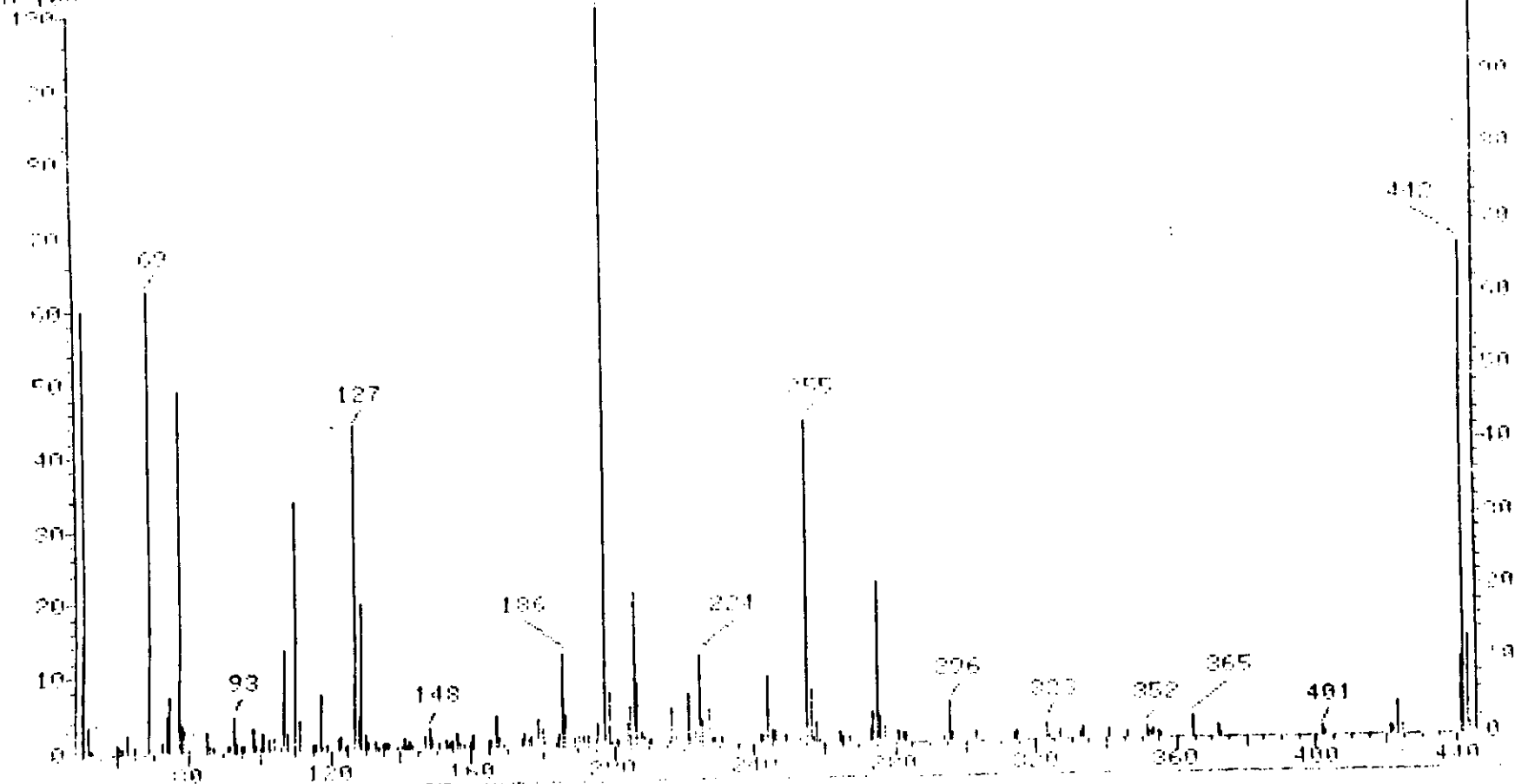
AL.,,3 Move cursor; then press carriage return :

File: 541058
Exp: 05 196

300.23 100
1000.00 100

1000.00 100

1000.00 100
11.00 100



001058 840223, 1 5000, D. TRF
 192 SUB ADI. NRK

terminal set to type-check mode.

END, 001058

File: 001058 Scan #: 192 Page: 11.00

Page: 00517

6/2	Int	6/2	Int	6/2	Int	6/2	Int	6/2
49.95	15.75	110.00	33.44	157.90	.29	209.95	.42	293.90
50.95	59.87	111.00	3.81	159.80	.40	210.45	.63	295.80
51.95	3.41	115.00	.36	160.80	1.48	211.15	.61	296.70
52.95	.23	115.80	.56	165.00	.92	215.95	.31	302.70
53.25	.36	116.90	7.01	165.80	.73	216.75	4.62	302.90
60.05	.77	117.70	.31	166.90	3.95	217.85	.67	313.80
60.85	.44	119.00	.38	167.90	1.17	220.95	6.46	314.60
61.85	.38	122.00	1.07	169.00	.25	221.95	.44	314.80
63.05	2.20	122.90	1.44	174.00	.82	222.05	1.51	322.90
64.95	.54	124.10	.15	175.00	1.51	224.05	11.71	323.60
68.95	62.58	125.00	.42	176.00	.73	225.15	2.61	324.00
73.95	1.92	127.00	43.69	176.80	1.21	226.75	4.11	326.11
75.95	4.48	121.10	3.01	177.90	.79	227.81	.61	331.85
76.95	7.21	120.80	15.67	178.70	3.43	228.90	1.41	333.75
77.05	48.75	129.90	1.11	179.90	2.15	231.85	.31	340.75
77.95	3.37	131.90	.75	186.10	.57	241.95	.17	340.65
78.05	2.61	132.70	.31	183.95	.00	242.75	.17	343.11
81.15	1.99	132.90	.81	181.85	1.51	242.85	3.07	351.95
81.15	2.55	123.90	.29	181.95	15.21	243.85	1.81	352.85
85.15	2.85	121.90	.61	185.75	3.89	245.65	1.61	352.95
85.95	.40	126.10	.51	186.95	.77	249.91	.75	353.25
86.85	.65	131.70	.52	191.75	.98	250.95	.11	353.65
96.85	.40	133.90	.10	197.75	1.80	251.05	43.31	354.51
91.35	.46	141.11	.17	192.15	1.07	255.07	6.77	355.11
92.95	4.63	141.10	1.11	194.85	.44	256.07	.13	364.65
93.85	.75	141.90	.11	195.95	2.88	257.71	11.11	367.15
94.05	.42	141.40	.81	197.85	100.00	258.70	.29	372.51
95.15	.57	142.71	.32	198.85	7.07	264.90	1.11	411.11
95.85	.61	147.80	1.11	199.85	.31	264.91	.41	411.61
98.05	2.78	147.91	2.47	201.15	.42	266.93	.52	402.61
99.05	.46	148.90	.04	201.35	.41	271.70	1.59	421.91
99.95	.11	151.75	.64	202.85	.34	273.80	3.82	421.91
100.95	1.93	152.70	.91	203.85	2.63	274.80	21.44	423.00
102.75	1.22	153.10	.13	204.05	5.03	275.00	2.91	423.25
103.85	1.46	151.90	.56	205.85	20.28	276.90	1.63	441.10
105.05	.96	155.00	.71	205.85	8.18	280.80	1.12	442.00
106.05	.11	155.90	1.72	207.85	1.49	281.80	.80	443.00
106.85	13.20	156.90	.36	208.65	.73	282.80	.69	444.10
108.05	2.05							

AL, Z Move cursor; then press carriage return :

Appendix D

Subcontractor's Data

- 1) A copy of the originating subcontractor's report is included for all data not generated within ETC's laboratory.



RECEIVED FEB 8 1984

1101 State Road, Building B
Princeton, New Jersey 08540
609-924-5151

LABORATORY ANALYSIS REPORT

Client: ETC Corporation
Address: 284 Raritan Center Parkway
Edison, New Jersey 08837
Attention: Mr. R. Smith

Test Number: 184055
Date Received: January 31, 1984
Date Sampled: Unknown
Job Number: 03001-22F

SAMPLE NUMBER	SAMPLE DESIGNATION DESCRIPTION
1	D3907
2	D8923
DL	Detection Limit

[illegible]

All results in mg/l (ppm) except where noted.

Page 3 of 4

Laboratory ID No. 11198

February 3, 1984
Date


Michael Wright
Laboratory Supervisor

Appendix E

Chain-of Custody Forms

- 1) A field Chain-of-Custody form (CC1) is included for all samples shipped by ETC shuttle.
- 2) An in-house sample Chain-of Custody form is included for the period the sample was in ETC's possession.
- 3) A subcontractor's Chain-of-Custody form is included for any analytical work not performed within ETC's laboratory.
- 4) Any additional Chain-of-Custody material provided by a client or by a client's sampling agent is also included.

CHAIN OF CUSTODY FORM (CC1)

ETC Sample #

78923

Start Date

1/24/84

Sealed By

Langis

SHIP TO:

Company: Hooker - Ruco

Facility/Addr: New South Rd

Attn: Robert LaMonica

Address: Hicksville, NY 11802

Phone: () -

SAMPLE IDENTIFICATION

Facility/Addr Code

WIOHICKIS

Source Codes

Soil ☐ (M)

Sliver/Stream ☐ (M)

Surface Impoundment ☐ (M)

Lake/Ocean ☐ (M)

Sed ☐ (S)

Bottom Sediment ☐ (S)

Pre-treatment Facility ☐ (P)

Treatment Facility ☐ (P)

Outfall ☐ (O)

Generation Point ☐ (G)

Leachate Collection Sys. ☐ (C)

Other ☐ (O)

W

1137A11001A1

013084

115310

11

Source Code (from above)

Your Sample Point ID (left justify)

Start Date (mo/day/yr)

Start Time (2400 hr. clock)

Elapsed Hour (compute)

Example:

W

1101121613P

015115811

091310

113

SAMPLE CONTENTS

Sample Bottle		Condition	Sample Bottle		Condition
D8923	E1 ✓	SAMPLE NOT FILTERED ANALYZE AS PER FULL WOH RFP All samples taken thru pump except VOUU (leaked)	D8923	V3 ✓	Per WOH methodology
D8923	E2 ✓		D8923	V4 ✓	
D8923	E3 ✓		D8923	A1 ✓	
D8923	E4 ✓		D8923	B1 ✓	
D8923	V1 ✓				
D8923	V2 ✓				
Rec'd one - 40 ml trip blank					

CHAIN OF CUSTODY CHRONICLE

1. Shuttle Opened By: (print) Robert LaMonica Date: 01/30/84 Time: 14:46
Signature: Robert LaMonica Seal #: 0018677 Intact: YES
I have received these materials in good condition from the above person.

2. Name: _____ Signature: _____
Date: _____ Time: _____ Remarks: _____
I have received these materials in good condition from the above person.

2. Name: _____ Signature: _____
Date: _____ Time: _____ Remarks: _____
I have received these materials in good condition from the above person.

4. Name: _____ Signature: _____
Date: _____ Time: _____ Remarks: _____
I have received these materials in good condition from the above person.

2. Shuttle Sealed By: (print) Robert LaMonica Date: 01/30/84 Time: 15:53
Signature: Robert LaMonica Seal #: 9018678
ETC USE ONLY Opened By: G. Morrison Date: 1/31/84 Time: 9:50am
Seal #: 18678 Condition: Seal intact

189
12

D8923

ETC Sample No.

SAMPLE POINT INFORMATION FORM (CC2)

FIELD MEASUREMENT DATA

Select up to 3 parameters to be recorded by entering the appropriate code letter in the first space for each of the 3 data entry fields provided. Enter the actual measurement data in the units specified for the three parameters you have selected.

PARAMETERS	ACTUAL (unit entry)	SAMPLE
Flow (GPM) _____	_____	C 2.5 _____
Volume (Gal) _____	_____	F 5.0 _____
Depth to Water (ft) _____	_____	G 1.1 0.3 _____
Discharge Rate (GPM) _____	_____	
Depth to Bottom (ft) _____	_____	
Flow Time (1/100-sec) _____	_____	
Depth to Screen (ft) _____	_____	

FIELD TEST DATA

DO (mg/L) _____	Sample Temp. (°C) _____		
pH _____	_____	_____	_____
Specific Conductance (microhm/cm) _____	_____	_____	_____

Single Measurement 2nd of Quadruplicate 3rd of Quadruplicate 4th of Quadruplicate

THE FOLLOWING DATA IS FOR YOUR RECORDS ONLY

SAMPLING METHOD (choose one)

AIR-LIFT PUMP ()	POTABLE PUMP ()	WELL ()
AUGER ()	PETROLEUM ()	WELL ()
BAILER ()	PUMP PUMP ()	WELL ()
BOTTLE ()	ROCK/SHOULDER ()	OTHER _____
COLUMBA ()	SOCKET PUMP ()	
DIFFER ()	SUBSIDENCE PUMP ()	
EDMETER ()	SUCKER LIFT PUMP ()	
ROCK ()	SURFACE ()	

SAMPLE TYPE (choose one)

RAW ()	COMPOSITE ()	OTHER ()
_____	_____	_____
_____	_____	_____

(describe) (describe)

WEATHER

SAMPLE DESCRIPTION (e.g., water, soil)

_____	_____
_____	_____
_____	_____

(describe) (describe)

Form Prepared By: _____ Employer: _____

Request for Analysis

Name of Subcontractor: Chymun

ETC Sample Number(s) D3907
D8923

Send bill to: Mr. John Birri
Send report to: Mr. R. F. Smith

ETC Corporation
284 Raritan Center Pkw.
Edison, NJ 08837
(201) 225-5600

Date Data Required: 2/11/84
If deadline cannot be met, contact R. F. Smith immediately.

Please perform the analyses requested below:

☐ Color	☐ Coliform, Total
☐ Conductance, Specific	☐ Coliform, Fecal
☐ Odor	☐ Biological Oxygen Demand
☐ pH	☐ (5 day, 20 degree C)
☐ Turbidity	☒ Chemical Oxygen Demand (COD)
☐ Total Solids	☐ Oil & Grease (Gravimetric)
☐ Total Suspended Solids	☐ Petroleum Hydrocarbons
☐ Total Dissolved Solids	☐ (Infrared)
☐ Total Volatile Solids	☐ Organic Carbon, Total (TOC)
☐ Gross Alpha and Gross Beta*	☐ Phenols, Total (as Phenolics)
☐ Radium 226 if Gross Alpha	☐ Methylene Blue Active
exceeds 5 pCi/l	☐ Substances (MBAS) (Foaming
☐ Radium 228 if Radium 226	☐ Agents, Surfactants)
exceeds 3 pCi/l	

* If Gross Alpha exceeds 5 pCi/l, R. F. Smith must be notified immediately.

☐ Acidity	☐ Nitrate-Nitrite
☐ Alkalinity	☐ Nitrite
☐ Bromide	☐ Oxygen, Dissolved
☐ Chloride	☐ Phosphorous, Ortho Phosphate
☐ Chlorine, Total Residual	☐ Silica, Dissolved
☐ Cyanide, Total	☒ Sulfate (as SO ₄)
☐ Ammonia (as N)	☐ Sulfide (as S)
☐ Total Kjeldahl Nitrogen (TKN)	☐ Sulfite (as SO ₃)
☐ Nitrate	☐ Fluoride

OTHERS

Sample(s) Relinquished by: Greg Morris

Date 1/31/84 Time 5:00 P.M.

Sample(s) Received by: L. V. R.

Date 1/31/84 Time 5:10 P.M.

LABORATORY CHAIN-OF-CUSTODY CHRONICLE

ETC Sample Number(s) D8923

Sample Preparation For:	Analyst	Date
Base/Neutral/PCB's & Pesticides	<u>A. Migliaccio</u>	<u>2/21/84</u>
Acids	_____	_____
Metals	_____	_____
Others <u>PP/BN</u>	<u>A. Migliaccio</u>	<u>2/21/84</u>
Others <u>PLB (GC)</u>	<u>David B. Long</u>	<u>2/21/84</u>
Others	_____	_____
Others	_____	_____

Sample Analysis For:	Analyst	Date
Base/Neutral/PCB's & Pesticides	<u>Keith Sher</u>	<u>2/23/84</u>
Acids	_____	_____
VOA/Purgeables	<u>R Albert</u>	<u>840204</u>
Metals	_____	_____
Others <u>TOC</u>	<u>C. Cullinan</u>	<u>2/14/84</u>
Others <u>TOC report</u>	<u>Lee Figueroa</u>	<u>2/21/84</u>
Others <u>Lead/Dangler PP/PCB/GC</u>	_____	<u>2/23/84</u>
Others	_____	_____
Others	_____	_____
Others	_____	_____

Verified By Ken Baker

LABORATORY CHAIN-OF-CUSTODY CHRONICLE

ETC Sample Number(s) D3907, D8923

Sample Preparation For:

Analyst

Date

Base/Neutral/PCB's & Pesticides

Acids

Metals

Others

Others

Others

Others

Xpnd

1/31/84

Sample Analysis For:

Analyst

Date

Base/Neutral/PCB's & Pesticides

Acids

VOA/Purgeables

Metals

Others

Others

Others

Others

Others

Others

Xpnd

2/25/84

Verified By

Ken Baker

APPENDIX B-3

Site E - Special Samples



Occidental Chemical Corporation

Research Center

MEMO

To R. Badger Date July 15, 1983
From N. Simon
Subject GC/MS ANALYSIS OF C3588 FOR AROCLORS & PHTHALATES
COPIES: R. Schuttler, M. Kargatis, D. Thielen, A. Weston, TIC

I. SUMMARY

Fractions of the sample contained Aroclor 1248, bis(2-ethylhexyl)phthalate, di-N-butylphthalate and other unidentified phthalates. Very approximate estimates of the concentrations show Aroclor 1248 present at greater than 100 ug/g and the two identified phthalates at 1-3 mg/g.

II. EXPERIMENTAL

A). Sample Preparation

Aliquots of the sample were weighed into a 40 ml hypo-vial and diluted approximately 50:1 with methylene chloride. Each dilution or extract was shaken vigorously by hand for five minutes and then sonificated for ten minutes. The hypo-vials were inverted and 1 ul of each extract analyzed by GC/MS.

B). Instrumental Parameters

Gas Chromatographic Conditions (Finnigan 9610)

Column	- 15 m DB5-NB fused silica capillary (J&W)
Injection	- Grob, 60/1 split after 48 secs.
Carrier	- Helium 14.5 psi
Injector Temp.	- 280°C
Detector Temp.	- 280°C
GC/MS Interface	- 280°C
Column Program	- 10° to 280° at 12°/min. after a 1 min. hold at 10°, hold at 280° for 20 min.



Occidental Chemical Corporation

Research Center

R. Badger
GC/MS ANALYSIS OF C3588 FOR AROCLORS AND PHTHALATES
July 15, 1983

Page 2

Mass Spectrometer Conditions (Finnigan 4000)

- | | |
|-------------------|--|
| Instrument | - Finnigan 4000 GC/MS interfaced with an Incos Data Acquisition System |
| Source Parameters | - 85 ⁰ , Electron Impact Source with 70 eV ionizing electrons, ionizer temp. 270 ⁰ C. |
| EM Volts | - 1080 |
| Scan Parameters | - MID for Aroclors on six ions 2 each representing Cl ₃ , Cl ₄ , Cl ₅ for phthalates data acquisition in .45 sec. with .5 sec. hold. Scan 140-350 |

(MID descriptor shown in Figure 1)

(C). Standard Preparation

Bis(2-ethylhexyl)phthalate, di-n-butylphthalate and Aroclor 1254 were prepared by weighing pure standards in methylene chloride. Aroclors 1242, 1248, 1232, were obtained in solution from Supelco. Dilutions were made in methylene chloride.

III. RESULTS AND DISCUSSION

Aliquots of the sample representing soil and water, oil (or other organics) and water, and water alone were extracted with methylene chloride. An analysis of the extracts showed that the PCBs present are from Aroclor 1248. Figures 2, 3 and 4 compare a soil/water extract to Aroclors 1242, 1248 and 1254. An estimate of the concentration of Aroclor 1248 was made based on two concentration levels of a standard. The calculation was based on the sum of the trichlorobiphenyl isomers. It should be noted that the extracts analyzed represented mixtures of soil or oil and water. The water alone did not contain a detectable concentration of Aroclor 1248 (ND₅₀ ug/g). The water present could have diluted the Aroclor in the soil or oil.

<u>Sample</u>	<u>Conc. of Aroclor 1248/ug/g)</u>
1 soil/water <u>A</u>	400
2 soil/water <u>B</u>	150
3 oil/water <u>A</u>	250
4 oil/water <u>B</u>	220
5 water	ND ₅₀



Occidental Chemical Corporation

Research Center

R. Badger

GC/MS ANALYSIS OF C3588 FOR AROCLORS AND PHTHALATES.

July 15, 1983

Page 3

Samples 1, 3 and 5 were also analyzed for phthalates. The two phthalates identified were quantitated based on two concentration levels of a standard. It should be noted again that the concentrations found were very approximate.

<u>Sample</u>	<u>di-N-butylphthalate ug/g</u>	<u>bis(2-ethylhexyl)phthalate</u>
1 soil/water <u>A</u>	1000	280
3 oil/water <u>A</u>	2500	2200
5 water	less than 100	ND ₇₅

Other phthaltes were detected. They were not identified or quantitated. Figures 5, 6 and 7 show reconstructed ion chromatograms of samples 1, 3 and 5 on the lower trace and an ion chromatogram of m/e 149 - the most common fragment to phthalates.

Nan Simon

Nan Simon

Associate Chemist
Central Sciences

/jb
Attachments

Figure 1
MID Descriptor for
identification of
Aroclor

MID DESC: PC
INST: FINN CALI: 0714DJ

MASS DEFECT AT 100 AMU 30 MMU

MASTER RATE 1024

TOTAL ACQU TIME 0.628 SECS

TOTAL SCAN TIME 0.700 SECS

CENT SAMP INT 0.200 MS

MASS RANGE 1 TO 1024 AMU

6	255.576	326.597	1.000	0.700	1	80	0	1	0	POS
INT	BEGIN	END	TIME	(SECS)	MPW	MFW	MA	TH	BL	IGN
#	MASS	MASS	REQUEST	ACTUAL						

1	255.576	256.576	0.100	0.105	2	150	50	1	0	POS
2	257.577	258.577	0.100	0.105	2	150	50	1	0	POS
3	289.586	290.587	0.100	0.105	2	150	50	1	0	POS
4	291.587	292.587	0.100	0.103	2	150	50	1	0	POS
5	323.596	324.597	0.100	0.105	2	150	50	1	0	POS
6	325.597	326.597	0.100	0.105	2	150	50	1	0	POS

MID RIC
07/05/83 14:57:00
SAMPLE: 5804-07-1
RANGE: G 1.2287

DATA: PCBHICKS #1,29PPM1242
CALI: N5706 #1

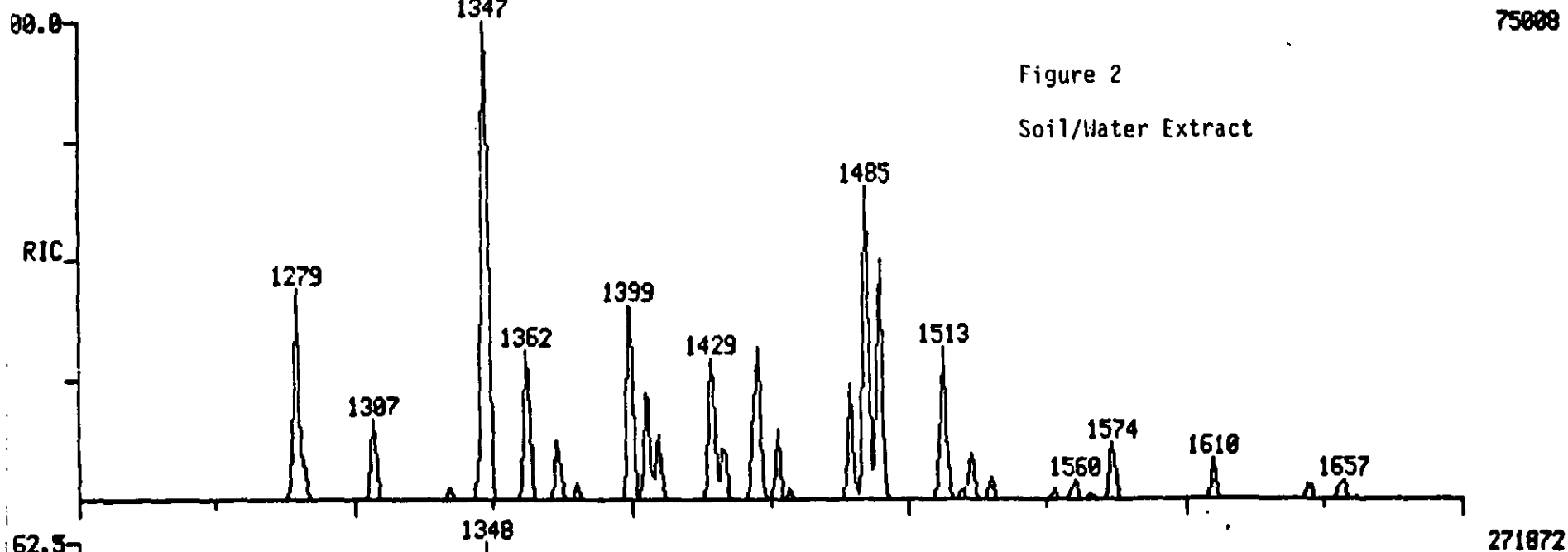
SCANS 1200 TO 1700

LABEL: N 0, 4.0 BASE: U 20, 3

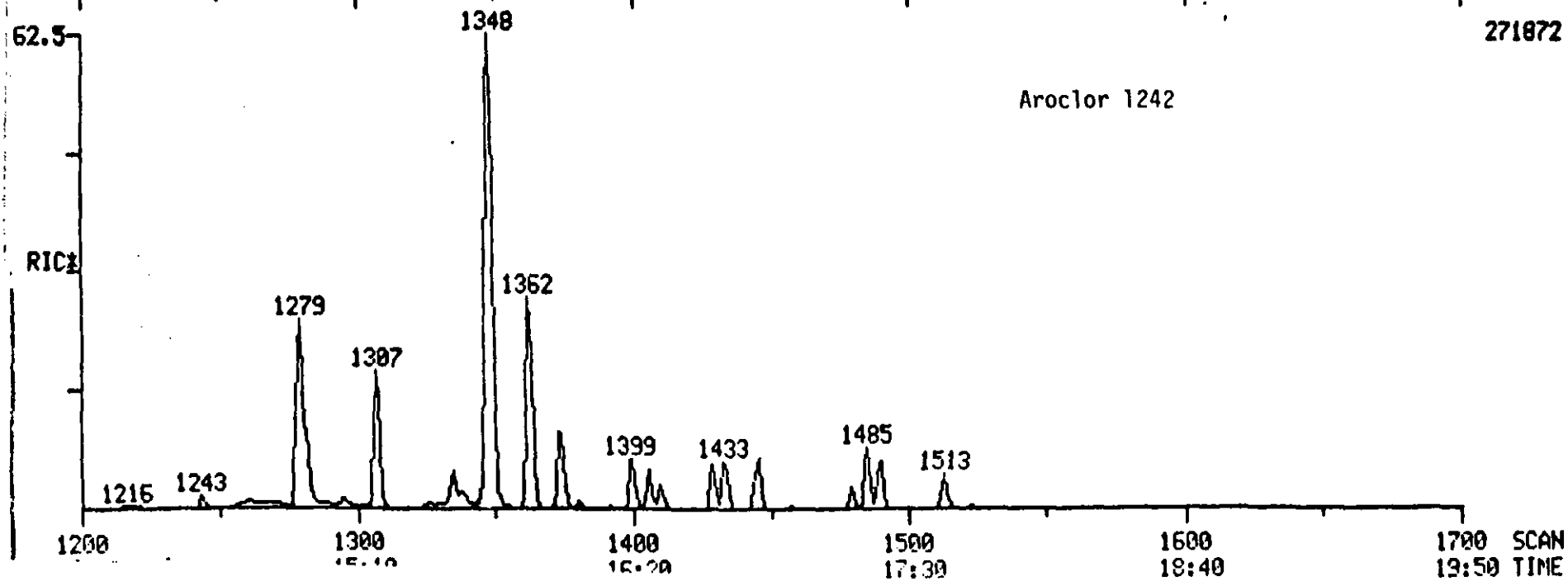
75008

Figure 2

Soil/Water Extract



Aroclor 1242



MID RIC
07/06/93 14:57:00
SAMPLE: 5804-07-1
RANGE: G 1.2287

DATA: PCBHICKS #1,20PPM1248
CALI: NS706 #1

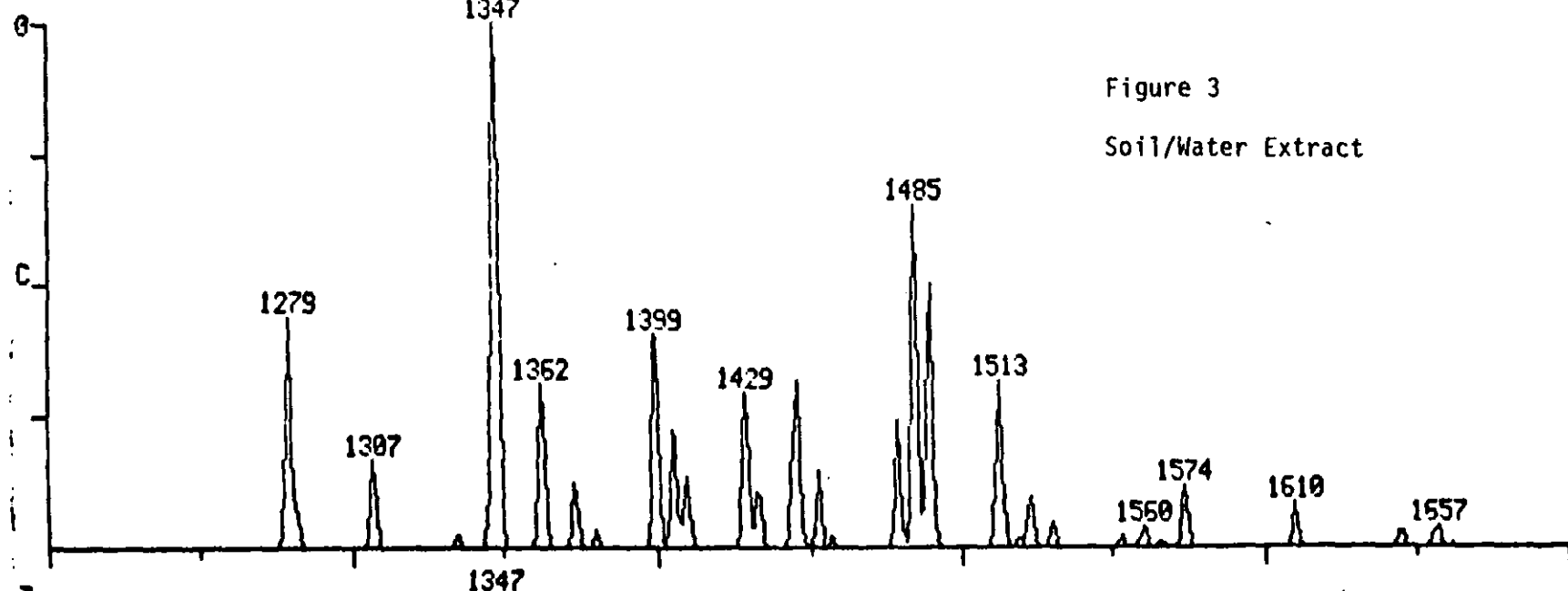
SCANS 1200 TO 1700

LABEL: N 0, 4.0 EASE: U 20, 3

75008

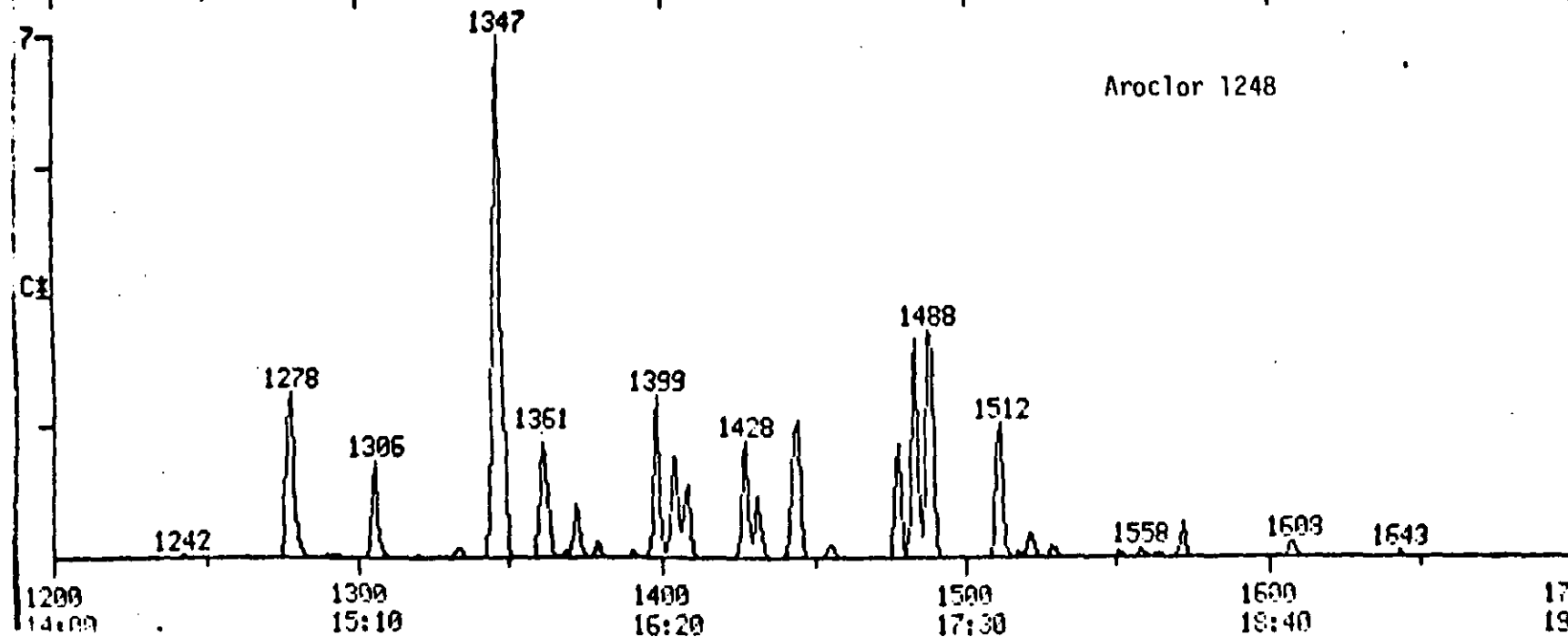
Figure 3

Soil/Water Extract



281088

Aroclor 1248



1200 1300 1400 1500 1600 1700 SCAN
14:00 15:10 16:20 17:30 18:40 19:50 TIME

MID RIC
07/06/83 14:57:00
SAMPLE: 5804-07-1
RANGE: G 1.2552

DATA: PCBHICKS #1,1254
CALI: N5706 #1

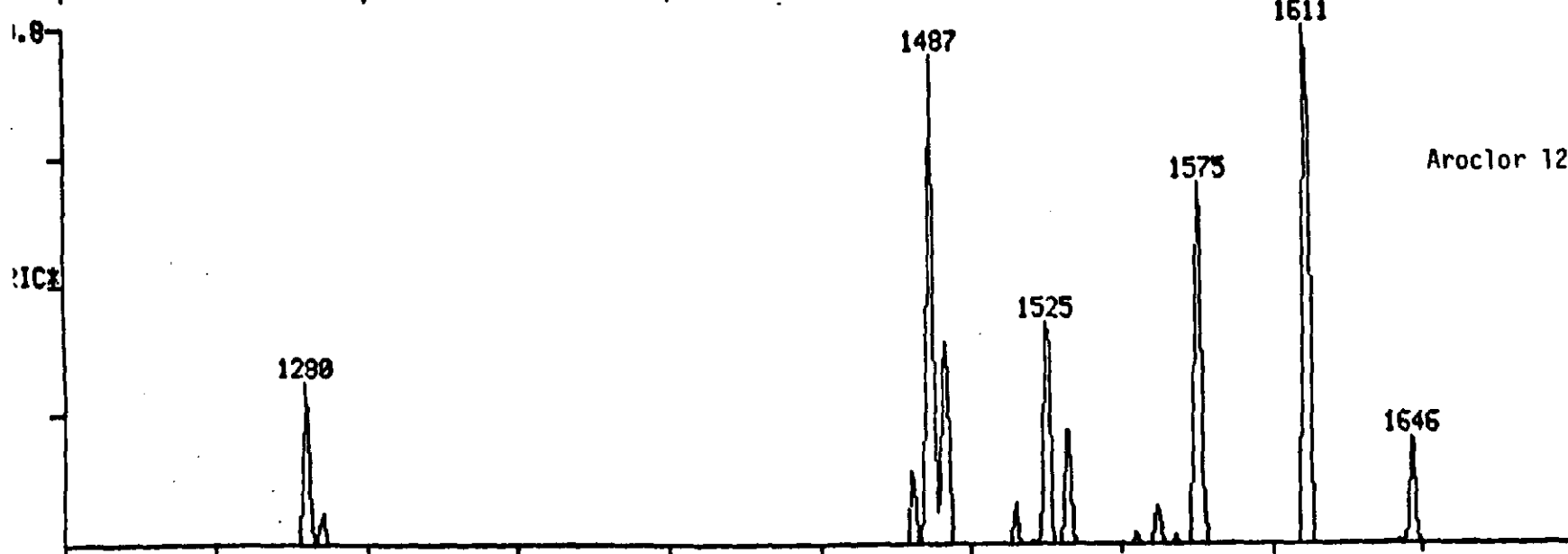
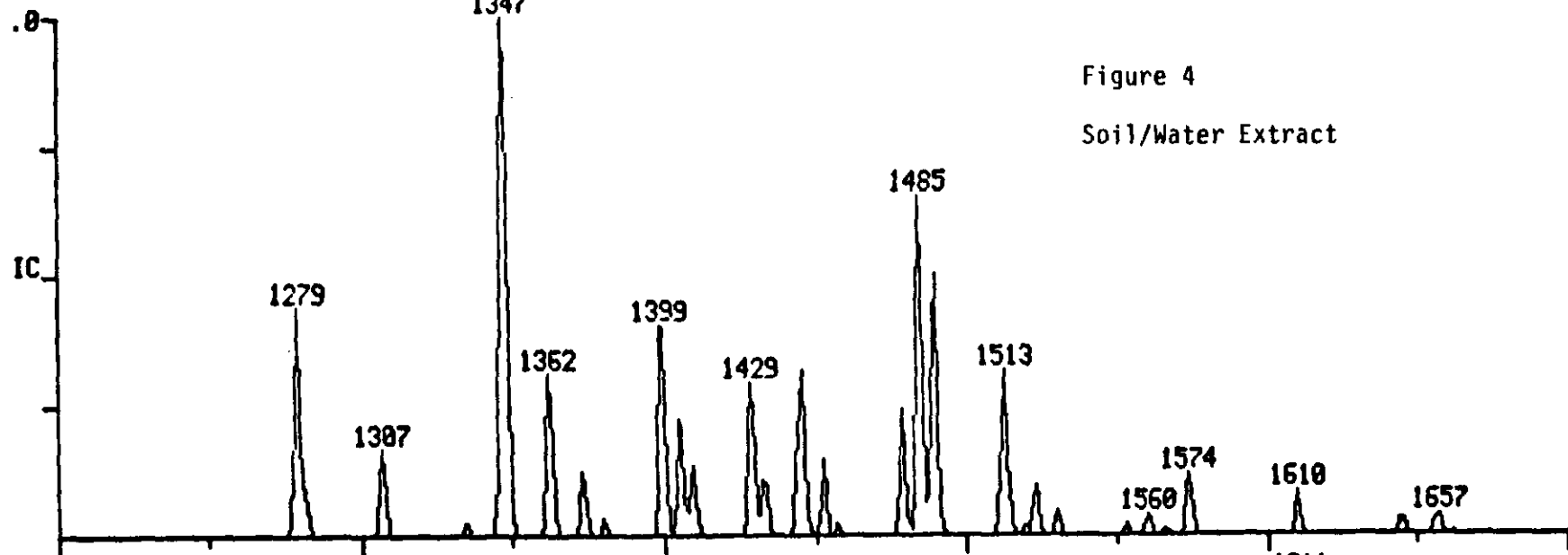
SCANS 1200 TO 1700

LABEL: N 0, 4.0 BASE: U 20, 3
1347

75008

Figure 4

Soil/Water Extract



7376

Aroclor 1254

1200 14:00 1300 15:10 1400 16:20 1500 17:30 1600 18:40 1700 19:50 SCAN TIME

RIC + MASS CHROMATOGRAM

07/12/83 10:05:00

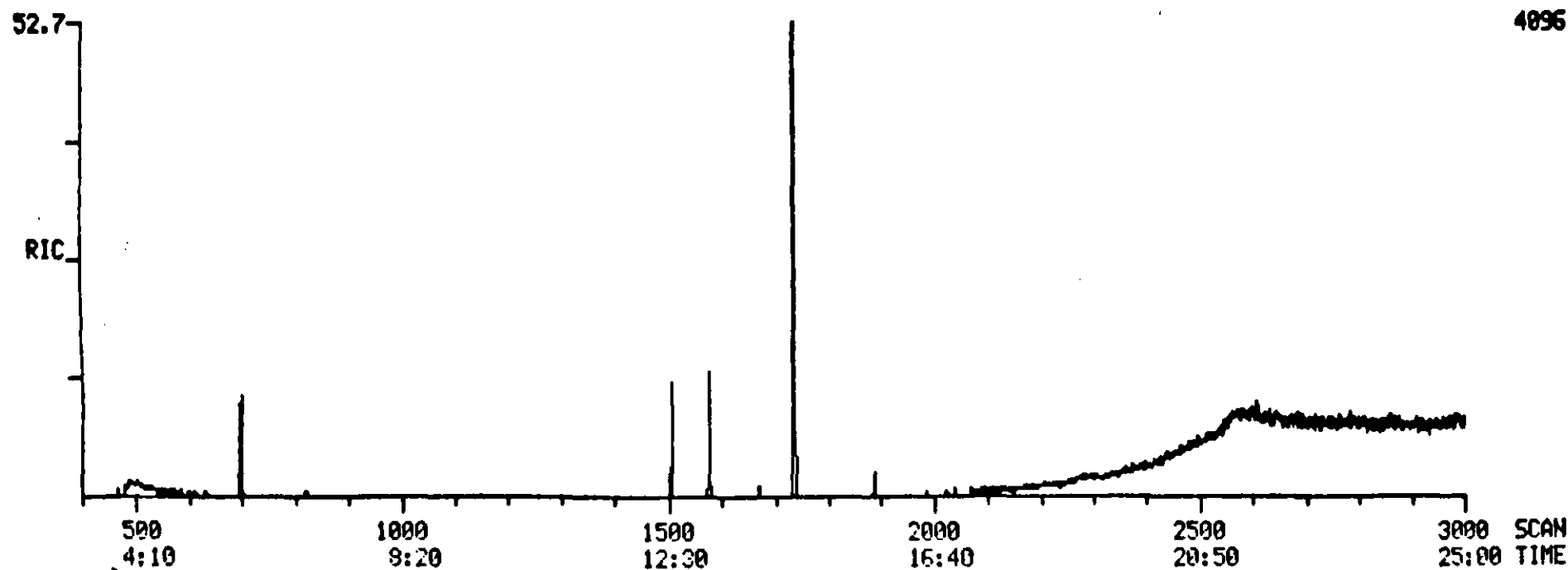
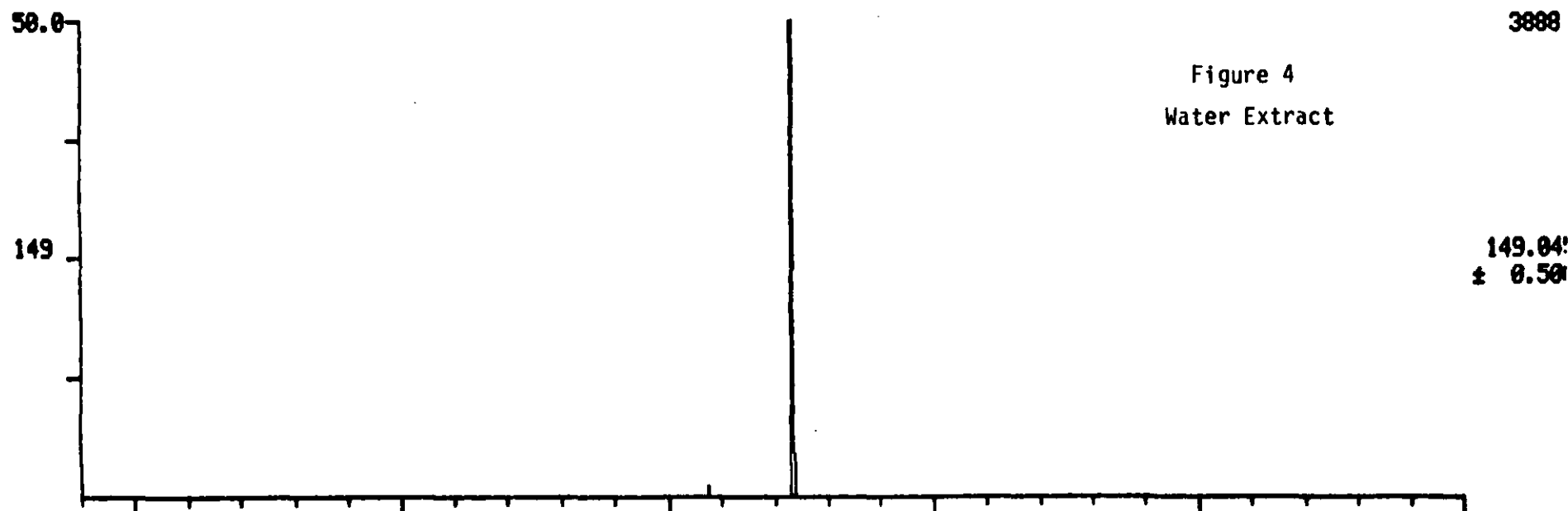
SAMPLE: EXTRACT OF WATER

RANGE: G 1.3137 LABEL: N 0, 4.0 QUAN: A 0, 1.0 BASE: U 20, 3

DATA: WATER #1

CALI: NS0712 #1

SCANS 400 TO 3000



RIC + MASS CHROMATOGRAM

07/12/83 10:42:00

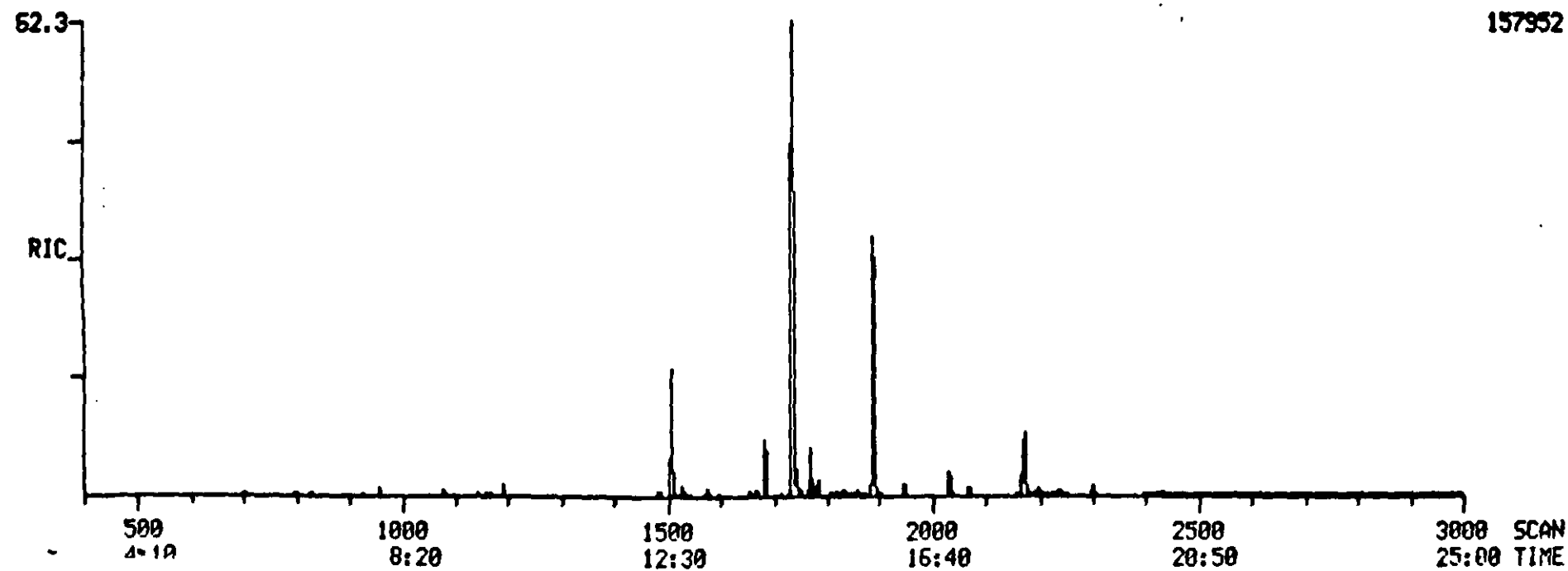
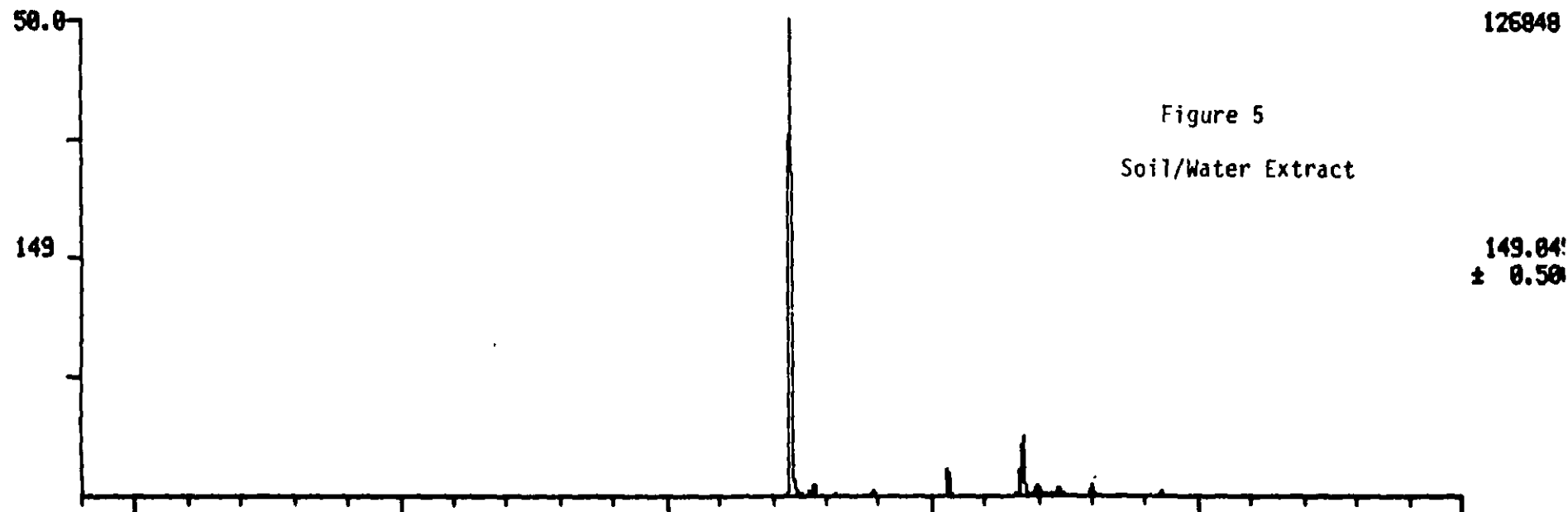
SAMPLE: EXTRACT OF SLUDGE

RANGE: G 1.3521 LABEL: N 0, 4.0 QUAN: A 0, 1.0 BASE: U 20, 3

DATA: SLUDGE #1

CALI: NS0712 #1

SCANS 400 TO 3000



RIC + MASS CHROMATOGRAM

07/12/83 11:19:00

SAMPLE: EXTRACT OF OIL

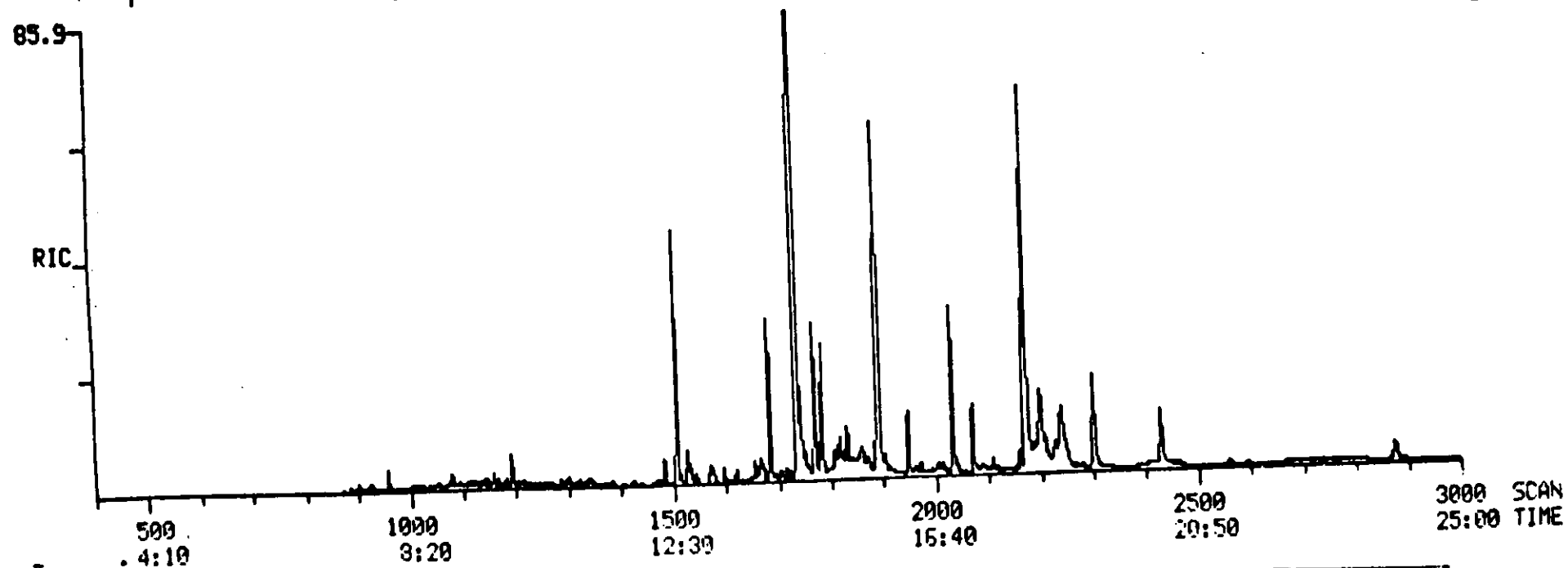
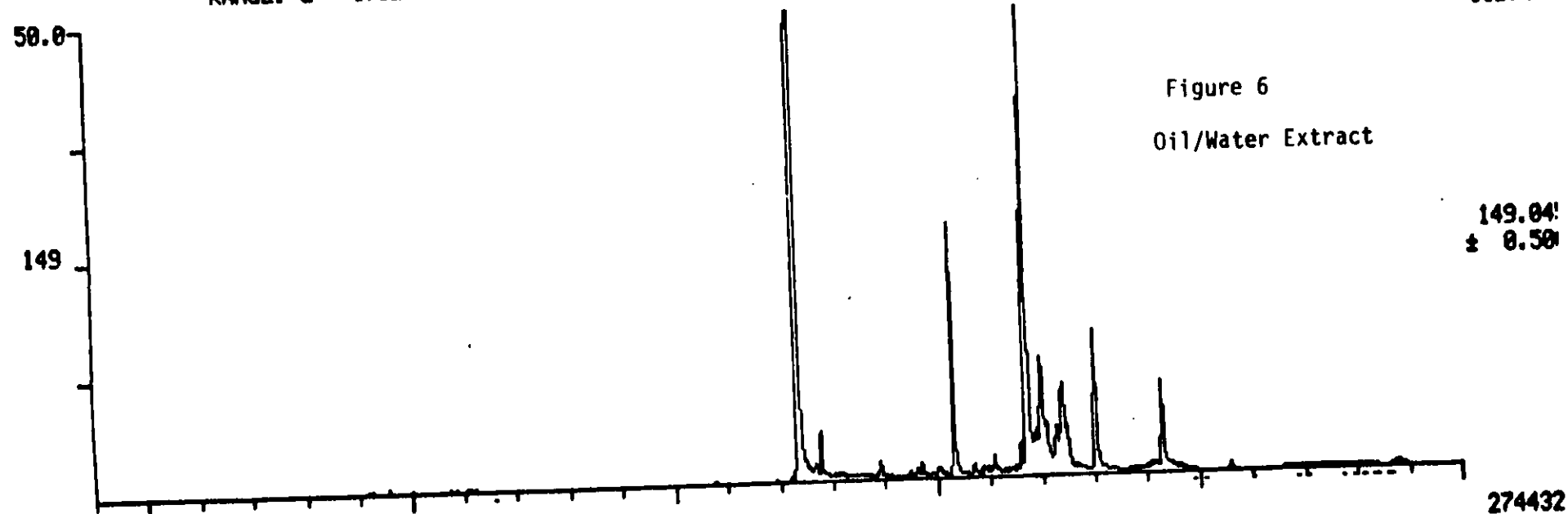
RANGE: G 1.3230 LABEL: N 0. 4.0 QUAN: A 0. 1.0 BASE: U 20. 3

DATA: OIL #1

CALI: N50712 #1

SCANS 400 TO 3000

159744



APPENDIX B-4

COMPLETE ETC REPORTS

(Bound Separately)