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TITLE: ANALYSIS OF WATER AND SEDIMENT SAMPLES FROM
THE SAUGET, ILLINOIS WASTE TREATMENT PLANT

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ABSTRACT: Water and sediment samples from the Sauget,
Illinois Waste Treatment Plant were analyzed
for the U.S. Environmental Protection Agency's
for the priority and non-priority pollutants.
The results of this analyses are presented in
this report.

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1. INTRODUCTION

Water and sludge samples collected at the Sauget, Illinois Waste Treatment plant were submitted to the Dayton Laboratory for priority and non-priority screening analyses. All analyses were conducted in accordance with the U.S. Environmental Protection Agency's (EPA) approved procedures (1, 2, 3, 4, 5).

2. SAMPLING

Sampling was performed at the Sauget, Illinois by Waste Treatment Plant. Samples were received at the Dayton Laboratory and submitted to the various analysts.

3. SAMPLE EXTRACTION AND SCREENING FOR ORGANIC POLLUTANTS

One influent, one effluent and one sediment sample from the Sauget, Illinois treatment plant were received for priority pollutant and wide scan organic analyses. A method blank, consisting of a one liter Burdick and Jackson distilled in glass organic free water, a duplicate of the effluent and a duplicate of the sediment sample were also analyzed for quality control purposes. Each water sample was spiked with 100 µg/L each of phenol- d_5 , Cl_2 -benzene- d_4 , pyrene- d_{10} and chrysene- d_{12} before liquid liquid extraction according to the EPA Method 625 analysis protocol. The sediment sample and duplicate were spiked with the same surrogate spiking compounds, but at levels corresponding to 187 µg/g for the sediment and 145 µg/g for the sediment duplicate.

All sample extracts were screened using capillary GC/FID analysis techniques. Figure 1 shows the chromatogram from the GC/FID analysis of the surrogate compound spiking standard added to each sample before extraction and Figures 2-11 show chromatograms from the analysis of the base/neutral and acid extracts from the water samples and methylene chloride extracts of the sediment and sediment duplicate. The method blank extracts were analyzed with no dilution of the extract, while the remaining water and sediment extracts required dilutions of 1:10 due to the very high levels of organic compounds present.

The laboratory data system was used to generate a summary report of extracts analyzed in Figures 4-11. Tables 1-8 show these results. They contain a retention time, an area, and an estimated concentration for each peak observed in the capillary GC/FID chromatograms (Figures 2-11). Note that very high levels of phenol- d_5 and Cl_2 -benzene- d_4 are reported in the water extracts, due to other co-eluting compounds. The identity of the major

components shown in Tables 1-8 will be discussed in later sections on the analysis of priority pollutants and other organic compounds in these samples.

The total amounts of chromatographable compounds are summed at the end of each table. These totals are based upon the assumption that the FID response of the compounds are equal to the anthracene-d₁₀ internal standard. Table 9 summarizes these values found from the GC/FID analysis. This table shows that there is very little difference between the influent and effluent water samples and that very good agreement is observed for the water and sediment duplicate analyses. Table 9 also shows the total amount of compounds identified from GC/MS analyses, to be discussed in a later section.

The following sections give detailed mass spectrometric results concerning the levels of priority pollutants and other organic compounds in these samples. Due to the requirement of sample dilution before analysis, only approximately 40 major components are identified in the water and sediment extracts. Also note that it is not certain what extraction efficiencies these compounds exhibit, especially in water samples containing high concentration levels of organic compounds. Therefore the levels which are reported in the following sections present lower level concentrations of compounds which are present in these samples. Other more direct analyses, such as direct aqueous sample injection, were beyond the scope of the present study.

3.1 Volatile Priority Pollutant Analyses

One organic free water sample (used as a method blank) and three water samples labelled 9:15 a.m., 10:23 p.m. and 11:15 a.m. were analyzed for the volatile priority pollutants listed in Table 10 using EPA Method 624 (1, 2). Table 12 gives the results of these analyses for those priority pollutants detected.

3.2 Extractable Organic Priority Pollutants

A method blank, an influent sample, an effluent sample, a duplicate effluent sample, a sediment sample and a duplicate sediment sample were analyzed for base/neutral, pesticide and acid extractable priority pollutants listed in Tables 10 and 11 using EPA Method 625 (1, 2) analysis protocol. Tables 13 and 14 summarize the results for those priority pollutants detected. Before extraction began, the water and sediment samples were each spiked with phenol-d₈, Cl₂-benzene-d₄, pyrene-d₁₀ and chrysene-d₁₂ surrogate spiking compounds. Tables 15 and 16 summarize recoveries of these compounds in the various extracts analyzed.

3.3 Wide Scan Organic Analyses

Large amounts of non-priority pollutant organic compounds were observed in the volatile and extractable organic priority pollutant analyses, given above. Table 17 gives the tentative identification of compounds and estimated amounts of detected compounds in the volatiles analysis and Tables 18 through 27 give these data for extractable compounds. Appendix A gives detailed information on the methods used for identification of non-priority pollutant extractable compounds.

3.4 Discussion of Priority Pollutant and Wide Scan Analyses

Table 9 summarizes the total concentrations of extractable organic compounds measured in water and sediment extracts from the Sauget, Illinois Water Treatment Plant. The total amounts reported in this table are calculated by three different analytical methods. The totals measured under "GC/FID" assumes all chromatographable compounds have FID responses equal to anthracene- d_{10} (used as an internal standard). The totals measured under "GC/MS-Total" assumes all chromatographable compounds have mass spectrometric responses equal to anthracene- d_{10} . The totals measured using "GC/MS-PP" are totals of all priority pollutants measured using EPA Method 625. This latter method uses authentic standards for each of the detected priority pollutants and thus are more accurate (for the priority pollutants) than either of the previous totals listed in the table. However this last total does not contain the amounts of non-priority pollutants which are present in the various sample extracts. All three base/neutral extractable water analyses show that the major organic components are not priority pollutants and thus the GC/FID and GC/MS-Total amounts are from 4 to 10 times larger than the GC/MS-PP totals. All three acid extractable water analyses show that the major organic components are the acid priority pollutants and therefore the GC/MS-PP totals for these extracts are approximately the same as the totals measured using the other two methods of analysis. The fact that the latter acid extractable analysis actually produces total amounts approximately twice that measured from GC/FID and GC/MS-Total is due to the more accurate method of quantitation for the acid priority pollutants.

4. ANALYSES FOR CHLORINATED DIBENZO-P-DIOXINS AND DIBENZOFURANS

Water samples were prepared by extracting one liter of water with 40 mL of petroleum ether by shaking for 15 minutes. The ether was removed and further cleaned up by extracting with two 50 mL washes of 1N KOH. This ether extract was dried over sodium sulfate, concentrated to 2 mL and then chromatographed on a column of silica gel, silica gel plus H_2SO_4 , and silica gel plus KOH. The column was eluted with 45 mL of hexane which was again concentrated to

2 mLs. The concentrate was then chromatographed on a 2.5 gm Woelm B alumina column. This column was first eluted with 10 mL of 2% CH_2Cl_2 /Hexane and this fraction discarded. A second elution was carried out with 15 mL of 50% CH_2Cl_2 /Hexane; 300 μL of dodecane was added as a keeper. The sample was concentrated to 300 μL and submitted for GC/MS analysis.

The sludge samples were prepared in a similar manner. One gram of sludge was applied to the top of a four gram silica gel column. The sample was eluted with 30 mL of CH_2Cl_2 and the collected fraction was concentrated to 2 mL and applied to the multi stage silica column as described for the water samples. The work up then proceeded in a manner comparable to the water samples.

Replicates, low and high level spiked and blanks were processed with all samples. The GC/MS analysis was carried out on a Hewlett-Packard 5985B GC/MS. A 30 meter SE-54 capillary column was used for the separation of the PCDDs and PCDFs. The mass spectrometer was operated in the selected ion monitoring mode following ions characteristic of mono through octachloro dibenzo-p-dioxins and dibenzofurans.

The results of the analyses are given in Tables 28 through 31. A pair of hexachlorodibenzo-p-dioxins (HCDDs) were found in the influent water samples (see table 28). The reproducibility of the analysis of these HCDDs was not good and no reasonable explanation can be given especially in view of the reasonable quantitative agreement of the heptachloro- and octachlorodibenzo-p-dioxin results. In certain instances interferences in the influent samples significantly raised the detection limits and this is noted as a ND (LXX) meaning none detected with a detection limit of XX.

The sludge samples were very difficult to analyze and therefore the method detection limits are quite high for both PCDDs and PCDFs. The low level spikes for several of the lower chlorinated PCDDs were not detected above the background interferences and have not been reported.

5. ANALYSIS OF OIL SAMPLE FOR PCBs

One oil sample was analyzed for low $\mu\text{g/mL}$ levels of PCBs. Molecular ions of the major chlorinated biphenyl components of all Aroclor formulations were monitored using capillary GC/MS-SIM analysis techniques. Figure 12 shows the analysis of a 59 $\mu\text{g/mL}$ Aroclor 1254 standard and Figure 13 shows the analysis of the oil sample. Both standard and oil were diluted 100 times before analysis. The response at 24.5 minutes is very likely due to Cl_4 -biphenyl isomers. This level of Cl_4 -biphenyl corresponds to 19 $\mu\text{g/mL}$, using Aroclor 1254 for calibration of the mass spectrometer response. However, the signal/noise is too low to verify what Aroclor formulation is present in this sample.

6. STATIC DAPHNIA MAGNA BIOASSAY

A static Daphnia magna bioassay was performed on a grab effluent sample obtained from the Sauget Waste Treatment Facility on March 2, 1962.

Procedures utilized for the Daphnia bioassay were based upon procedures outlined in "Methods for Measuring the Acute Toxicity of Effluents to Aquatic Organisms" (EPA-600/4-78-012). Six effluent concentrations (100%, 56%, 32%, 18%, 10%, 5.6%) and a control were set-up in duplicate. Ten first instar Daphnia magna (>24 hrs old) were placed in each replicate concentration. Well water, which was used for dilution purposes, was adjusted to simulate the quality of the Mississippi River near Sauget, IL. (i.e., total hardness = 230; pH = 8.10).

The results of the Sauget bioassay were as follows:

Effluent concentration (%)	Percent mortality	
	24 Hours	48 Hours
100	100	100
56	100	100
32	50	90
18	0	65
10	0	30
5.6	0	35
Control	0	0

Statistical analysis of the Sauget test data by the Binomial, Moving Average, and Probit Test Methods resulted in 48-hour EC_{50} values (95% confidence limits) of 14% (0, 32), 14% (9, 18), and 11% (8, 14), respectively.

Test concentration physical characteristics during the March 17-19, 1962 bioassay are presented below:

Physical parameter	Exposure period, hrs			Effluent concentration, %
	0	24	48	
Dissolved oxygen, mg/L	8.3	3.7	3.5	100
	8.1	5.6	4.8	56
	-	-	-	32
	-	-	-	18
	-	-	-	10
	7.8	6.7	7.3	5.6
	7.6	7.4	7.7	Control

(continued)

Physical parameter	Exposure period, hrs			Effluent concentration, %
	0	24	48	
pH ↓	8.3	7.8	7.6	100
	8.1	8.1	7.8	56
	-	-	-	32
	-	-	-	18
	-	-	-	10
	8.1	8.2	8.1	5.6
	8.0	8.0	7.9	Control
Conductivity, μ mhos ↓	7.200	7.150	7.200	100
	4.600	4.650	4.600	56
	-	-	-	32
	-	-	-	18
	-	-	-	10
	1.100	1.150	1.050	5.6
	600	600	700	Control
Temperature, °C ↓	22	22	22	100
	22	22	22	56
	-	-	-	32
	-	-	-	18
	-	-	-	10
	22	22	22	5.6
	22	22	22	Control
Alkalinity, mg/L as CaCO ₃ ↓	530			100
	312			56
	-			32
	-			18
	-			10
	224			5.6
	196			Control
Hardness, mg/L as CaCO ₃ ↓	>500			100
	>500			56
	-			32
	-			18
	-			10
	274			5.6
	236			Control

7. EP TOXICITY TESTING OF SLUDGE

The sludge sample was extracted per the U.S. EPA RCRA procedure as shown in Figure 14. The resulting leachate was then analyzed in accordance with approved EPA analytical procedures (3, 4 and 5). Results of the metals analyses are shown in Table 32 with the applicable quality control data. Table 33 shows the herbicides and pesticides concentration found in the leachate. While not a

part of the RCRA requirements, the results for requested analysis of the leachate for cyanide and phenol concentrations are shown in Table 34 with the applicable quality control data.

8. ADDITIONAL ANALYSES OF SLUDGE SAMPLE

Additional analyses requested for the sludge sample include pH, total cyanide and its total metal content. To obtain the pH a 10 g sample of the sludge was mixed with 20 mL of distilled water and slurried. The pH of this slurry was measured to be 11.07. The total cyanide content of the sludge was determined to be <1.3 ug/g (dry weight) using an EPA approved method [4]. The total metal content was determined by atomic absorption (AA) spectroscopy and an Inductivity Coupled Plasma (ICP) excitation technique. The methods used are described in Reference 4. Results of these analyses are shown in Table 35.

9. ANALYSES FOR PRIORITY AND NON-PRIORITY METAL POLLUTANTS IN WATER

In addition to the organics the EPA-designated priority pollutants include 13 metals which are to be analyzed for under the protocol [1]. Four metals (arsenic, mercury, selenium and thallium) are analyzed by AA spectroscopy. All others are determined by ICP excitation technique. Results are shown in Table 36 with the applicable quality control data shown in Table 37. Table 38 lists the least observable quantities (LOQ) for metals by ICP analyses.

10. ANALYSES FOR TOTAL CYANIDE AND TOTAL PHENOL IN WATER SAMPLES

Total cyanide in the water samples was determined according to the conventional wet chemistry technique described in Reference 3. Independent standards were included with the water samples for quality assurance.

In addition to specific phenolic compounds and phenol measured by GC/MS, total phenol in the water samples were also measured by typical wet chemistry techniques [3,4]. Independent phenol standards were also analyzed for quality assurance.

The results for these analyses were shown in Table 39 with the applicable quality control data.

11. NON-PRIORITY POLLUTANT COMPOUNDS

A number of pollutants as designated by the NPDES Consolidated Permits [3] as Group A and B pollutants were analyzed for. The compound was analyzed for in accordance with approved EPA methods [4]. The results for these analyses are shown in Table 40.

12. REFERENCES

1. Draft Final Report: Sampling and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants. U.S. Environmental Protection Agency, Cincinnati, Ohio, April 1977. 145 pp.
2. EPA Guidelines Establishing Test Procedures for the Analysis of Pollutants, Proposed Regulation, Federal Register, Monday, December 3, 1979.
3. U.S. Federal Register, Book 3, Vol. 45, No. 98, Monday, May 1, 1980.
4. Standard Methods for Examination of Water and Wastewater, Fourteenth Edition. American Public Health Association, Washington, D.C., 1976. 1193 pp.
5. Manual of Methods for Chemical Analysis of Water and Wastes. EPA-625/6-76-003a (PB 259 973). U.S. Environmental Protection Agency, Cincinnati, Ohio. 319 pp.

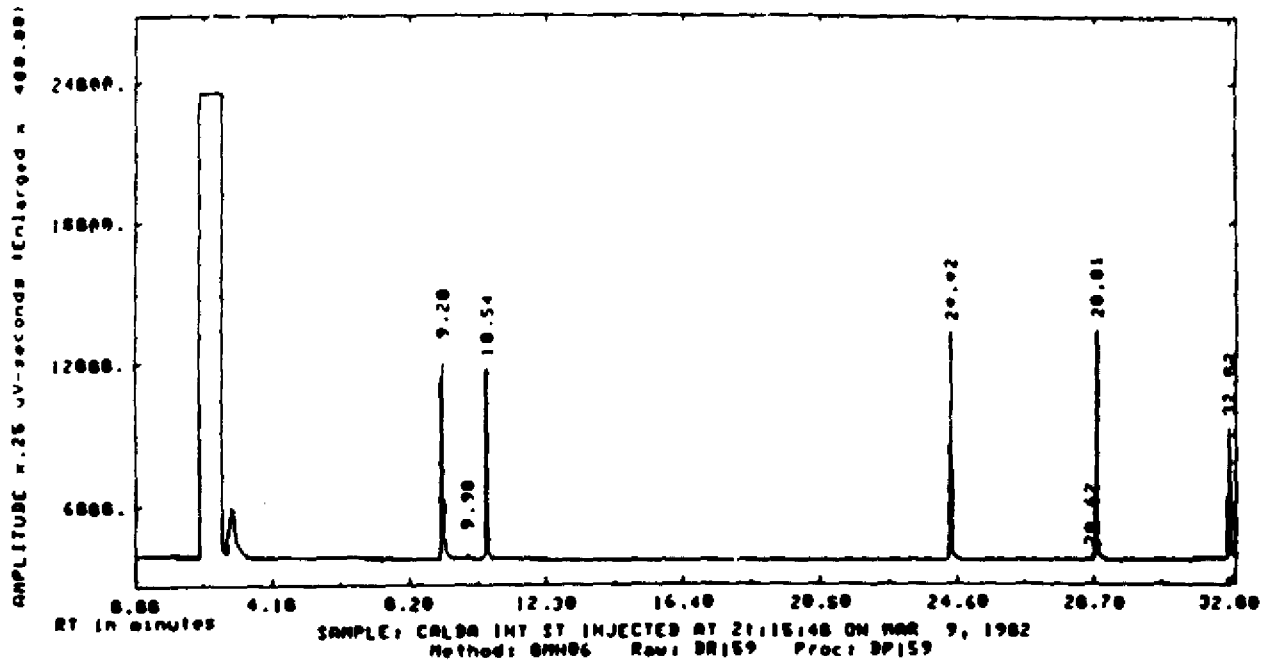


Figure 1. Capillary GC/FID chromatogram of surrogate compound spiking standard added to each sample before extraction.

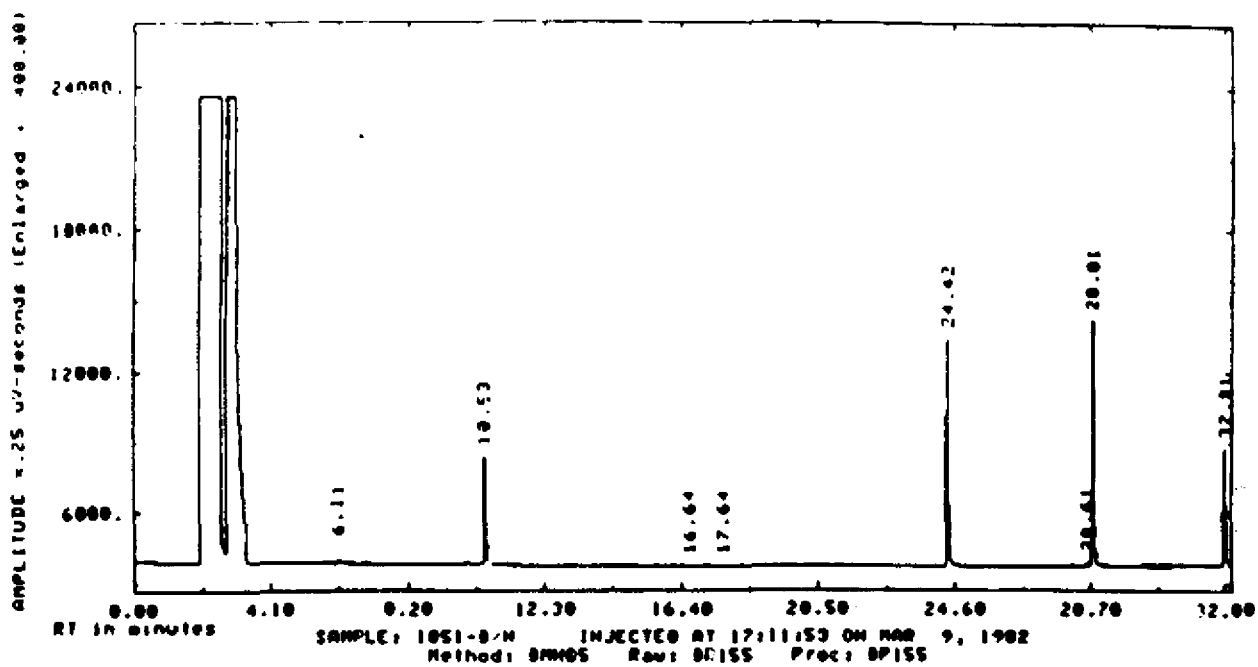


Figure 2. Capillary GC/FID chromatogram of the base/neutral extract of the method blank (Burdick and Jackson Water, distilled in glass).

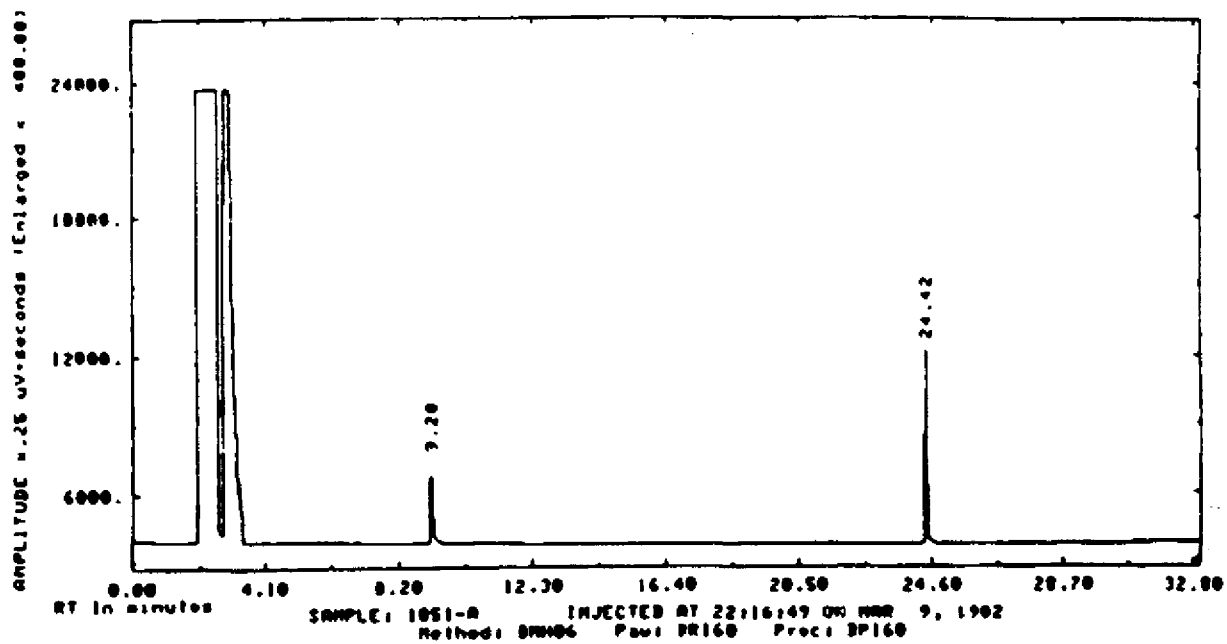


Figure 3. Capillary GC/FID chromatogram of the acid extract of the method blank (Burdick and Jackson Water, distilled in glass).

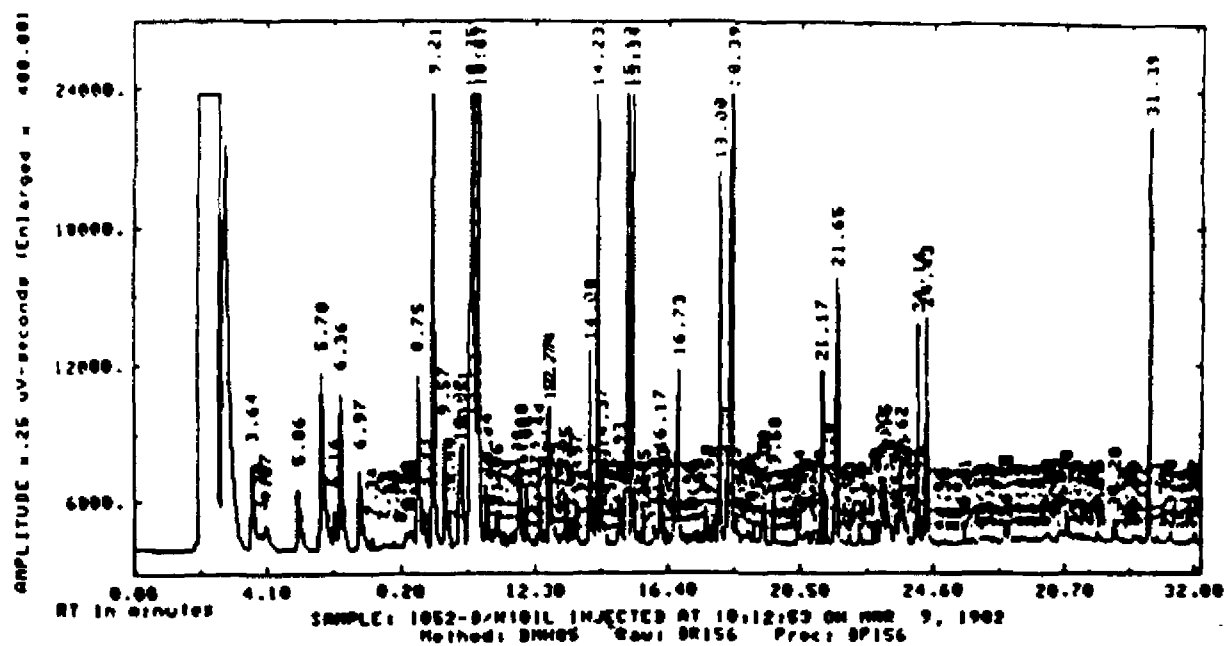


Figure 4. Capillary GC/FID chromatogram of the base/neutral extract of influent sample from Sauget, IL Treatment Plant, diluted ten times before analysis.

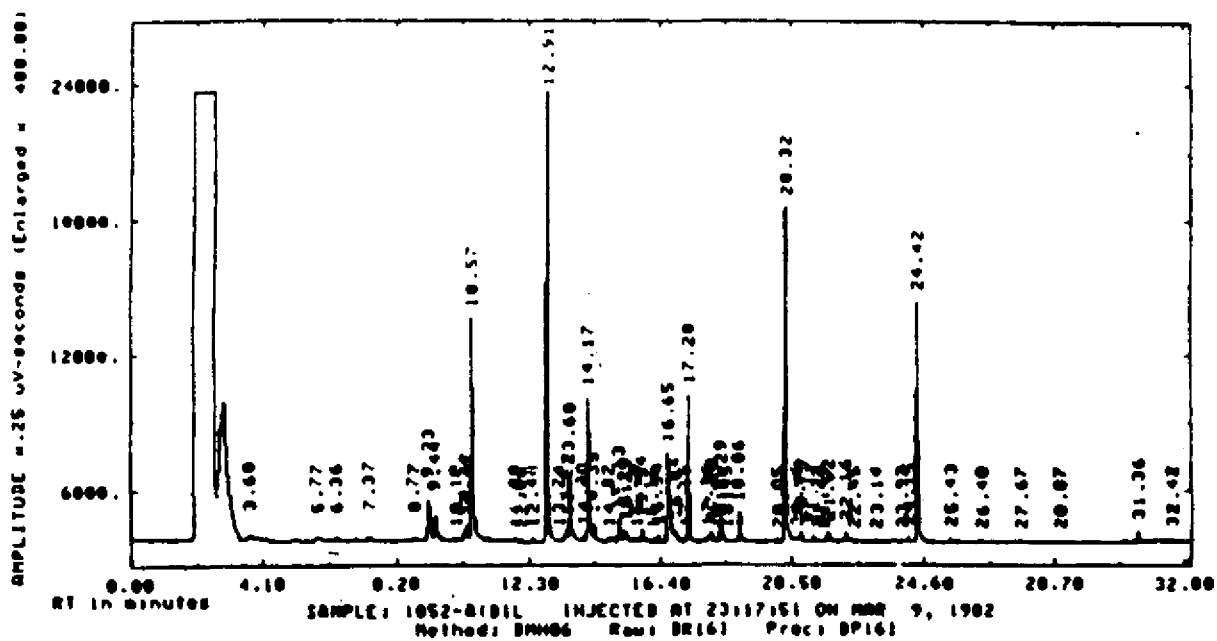


Figure 5. Capillary GC/FID chromatogram of the acid extract of the influent sample from Sauget, IL Treatment Plant, diluted ten times before analysis.

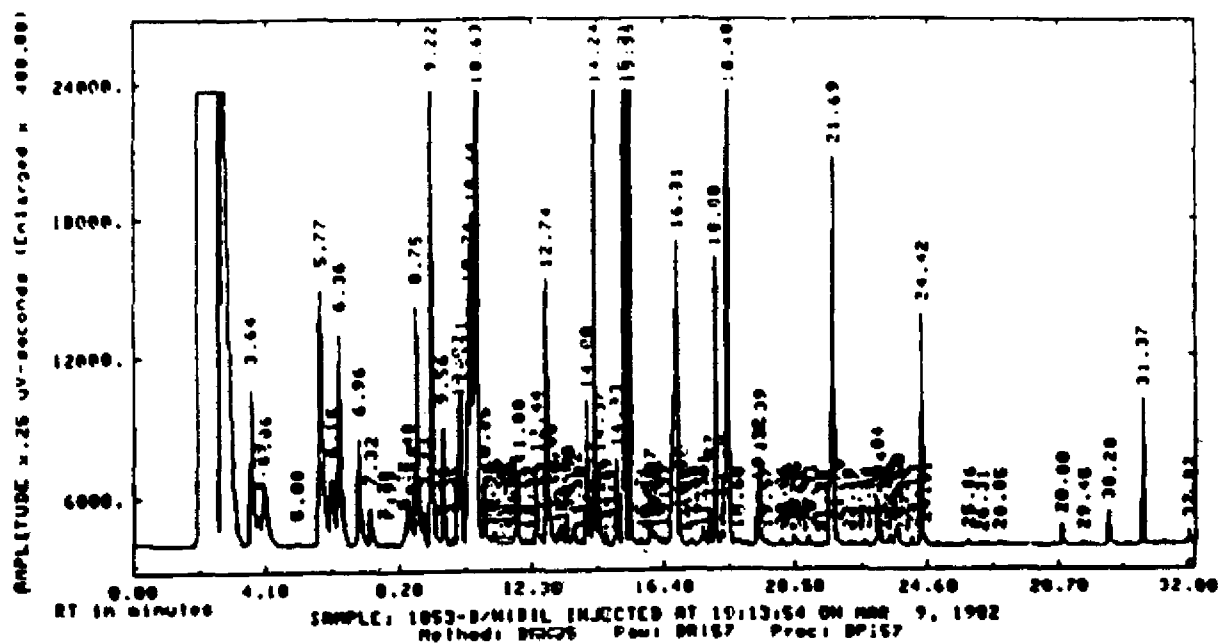


Figure 6. Capillary GC/FID chromatogram of the base/neutral extract of the effluent sample from Sauget, IL Treatment Plant, diluted ten times before analysis.

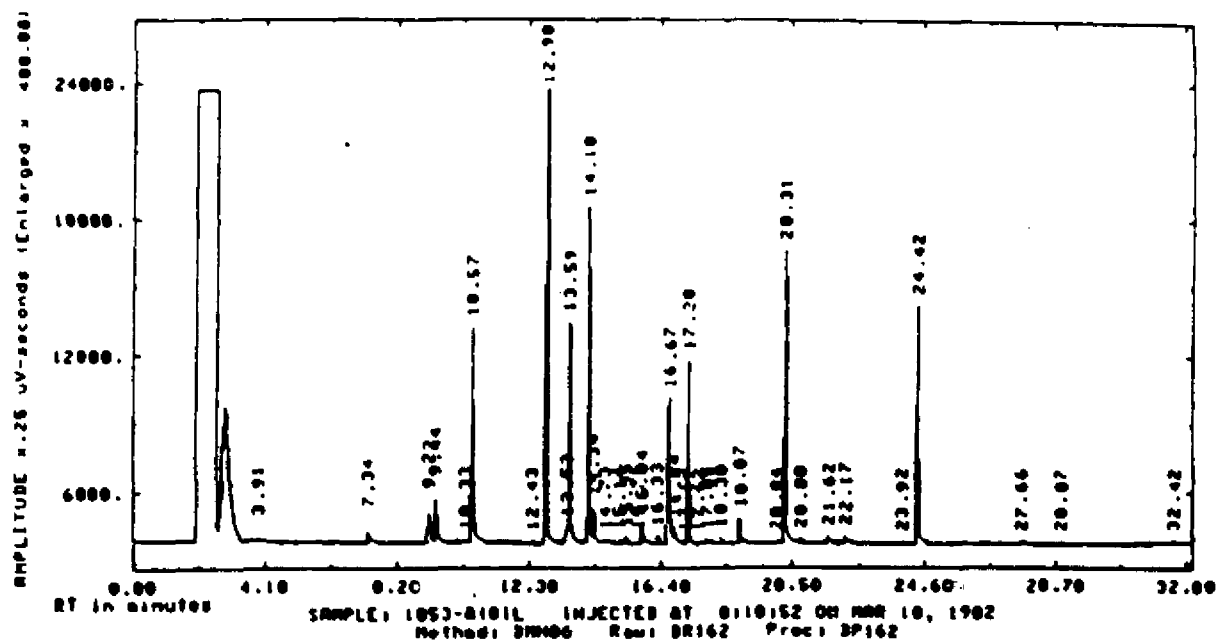


Figure 7. Capillary GC/FID chromatogram of the acid extract of the effluent sample from Sauget, IL Treatment Plant, diluted ten times before analysis.

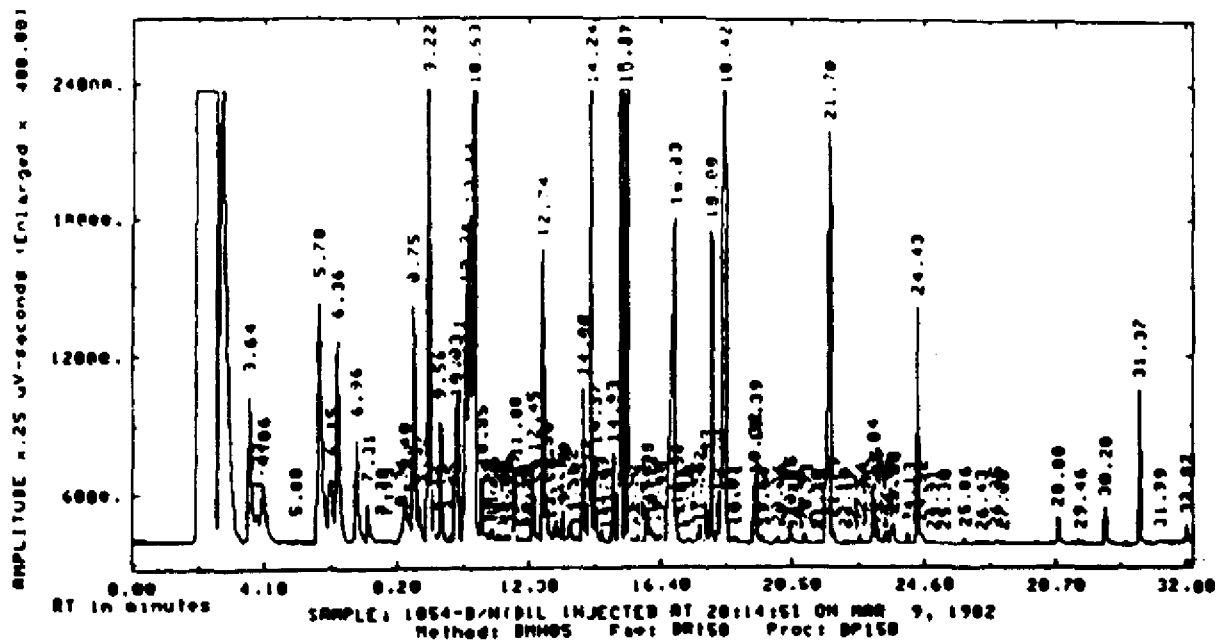


Figure 8. Capillary GC/FID chromatogram of the base/neutral extract of a duplicate effluent sample from Sauget, IL Treatment Plant, diluted ten times before analysis.

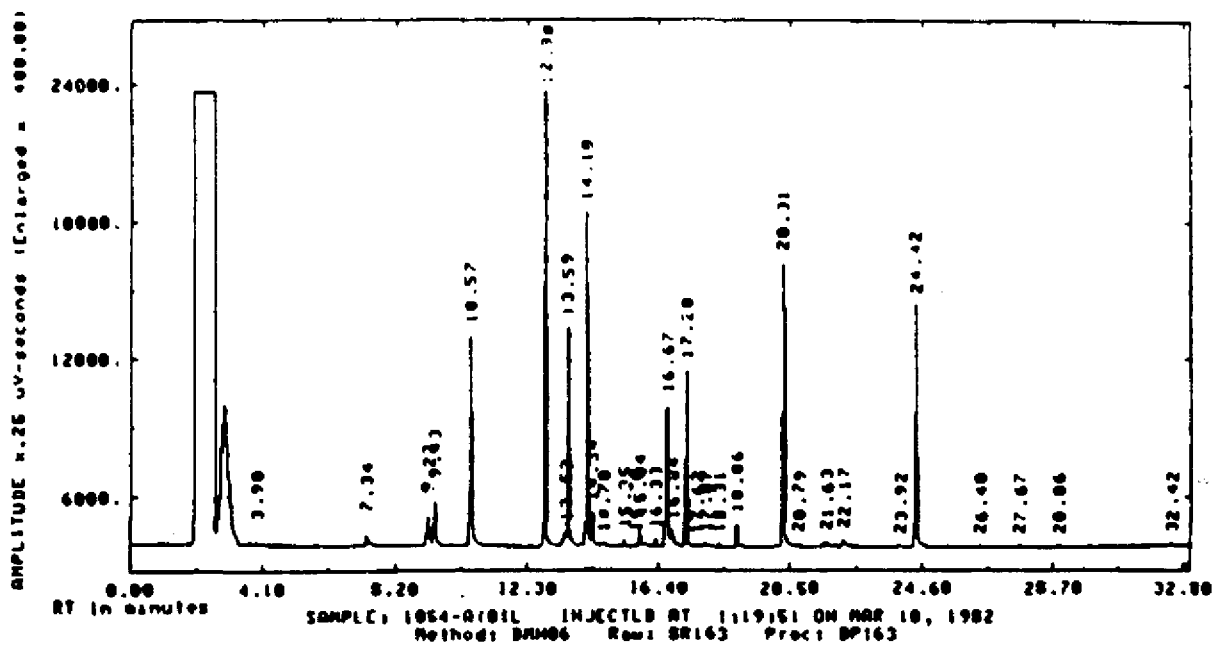


Figure 9. Capillary GC/FID chromatogram of the acid extract of a duplicate effluent sample from Sauget, IL Treatment Plant, diluted ten times before analysis.

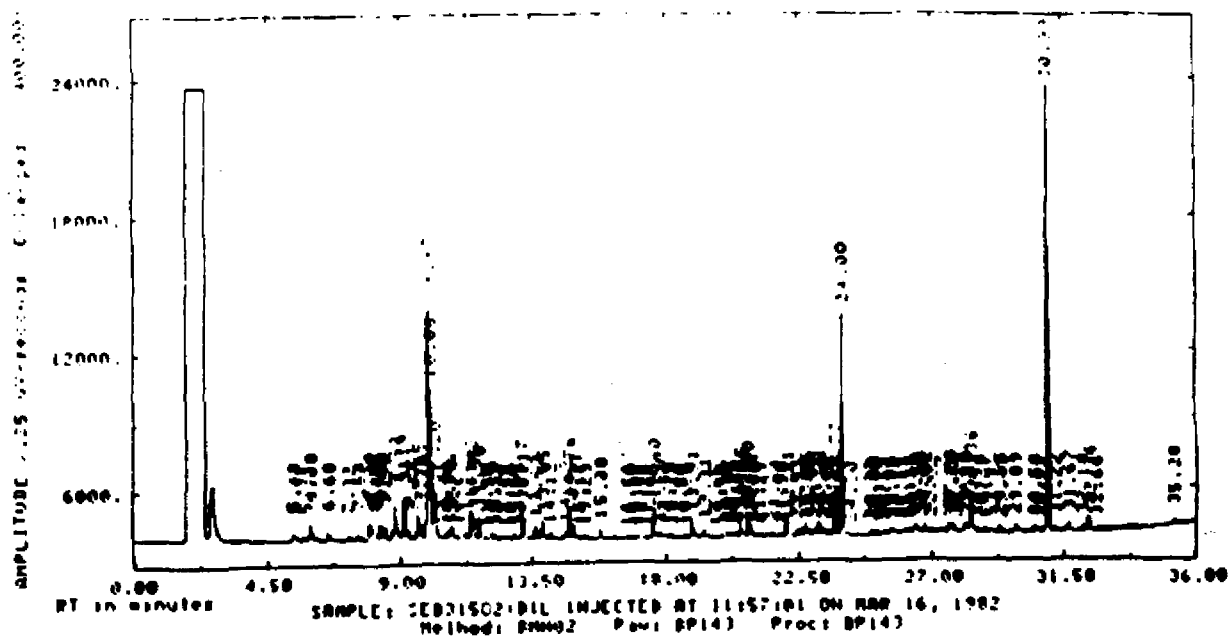


Figure 10. Capillary GC/FID chromatogram of the extract of a 1.07 g sediment sample from Sauget, IL Treatment Plant, diluted ten times before analysis.

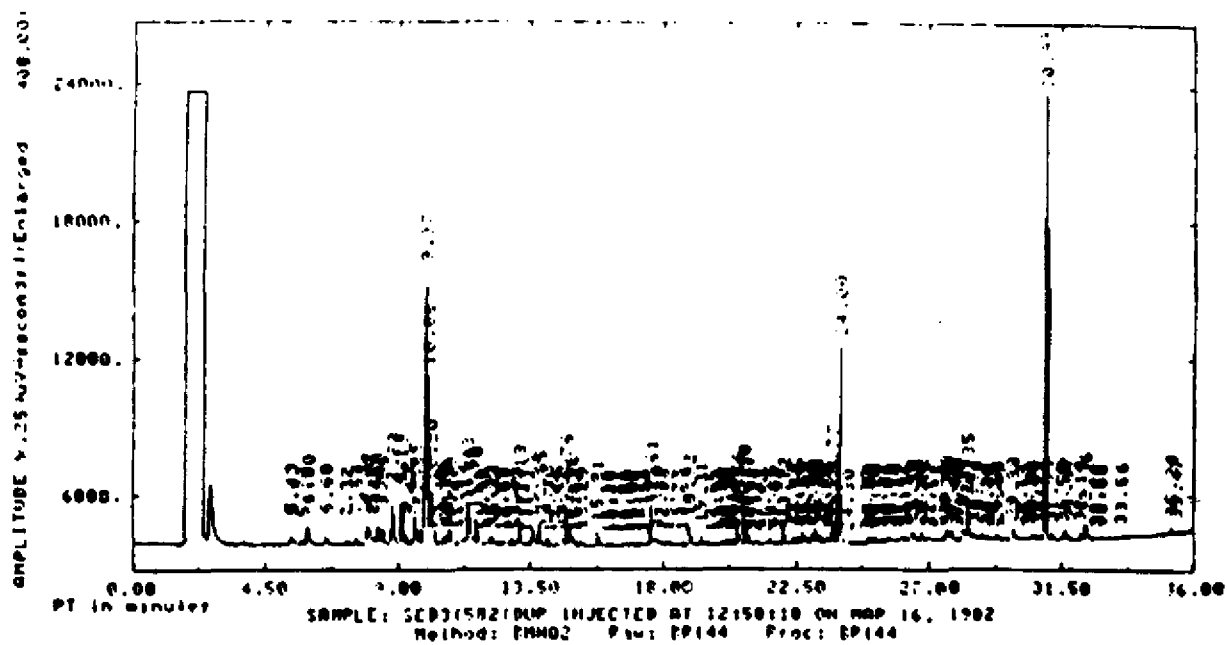


Figure 11. Capillary GC/FID chromatogram of the extract of a 1.38 g duplicate sediment sample from Sauget, IL Treatment Plant, diluted ten times before analysis.

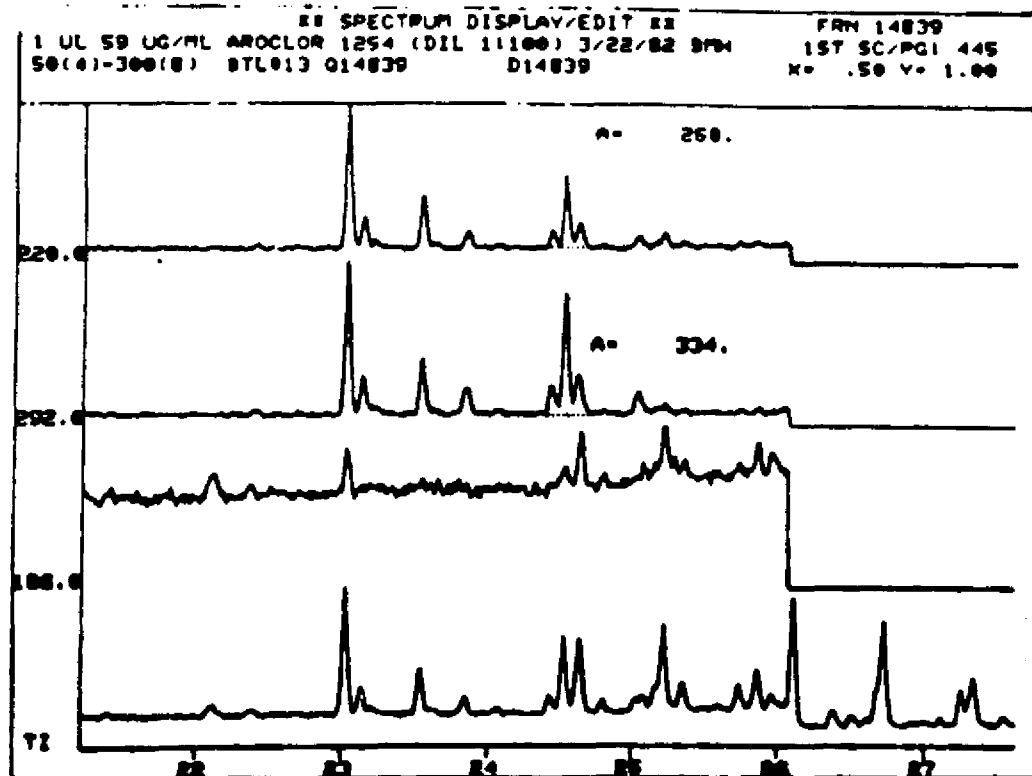


Figure 12. Analysis of 59 $\mu\text{g/mL}$ Aroclor 1254 standard, using $(\text{GC})^2/\text{MS-SIM}$ analysis techniques.

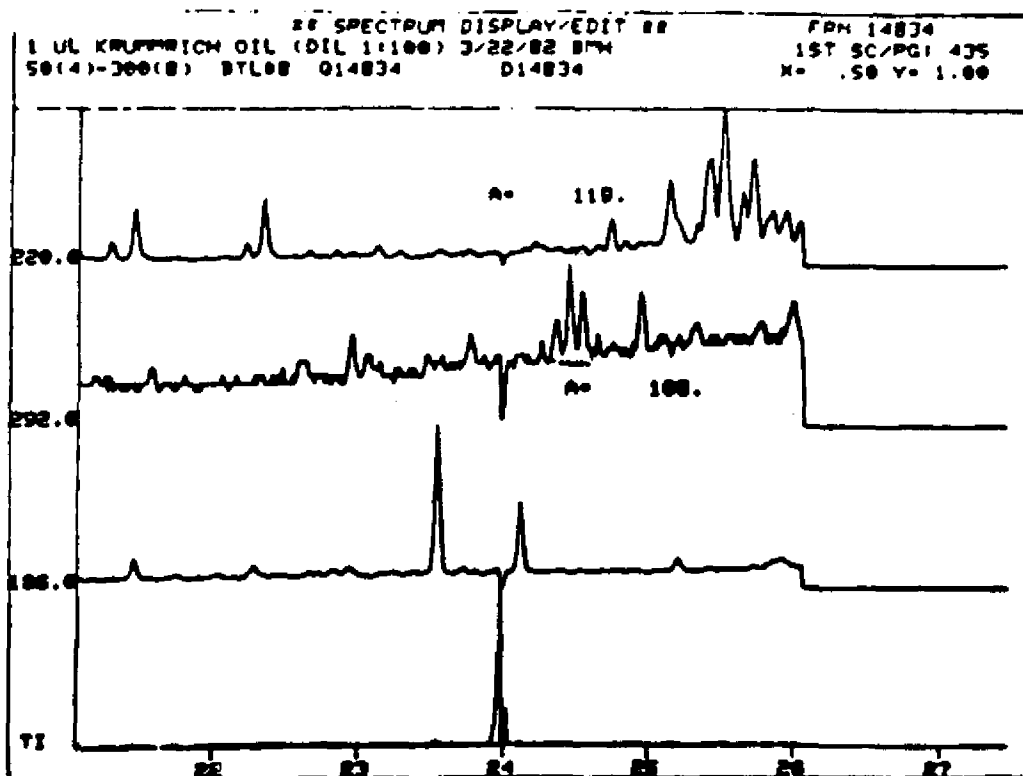


Figure 13. Analysis of an oil sample, using (GC)²/MS-SIM analysis techniques.

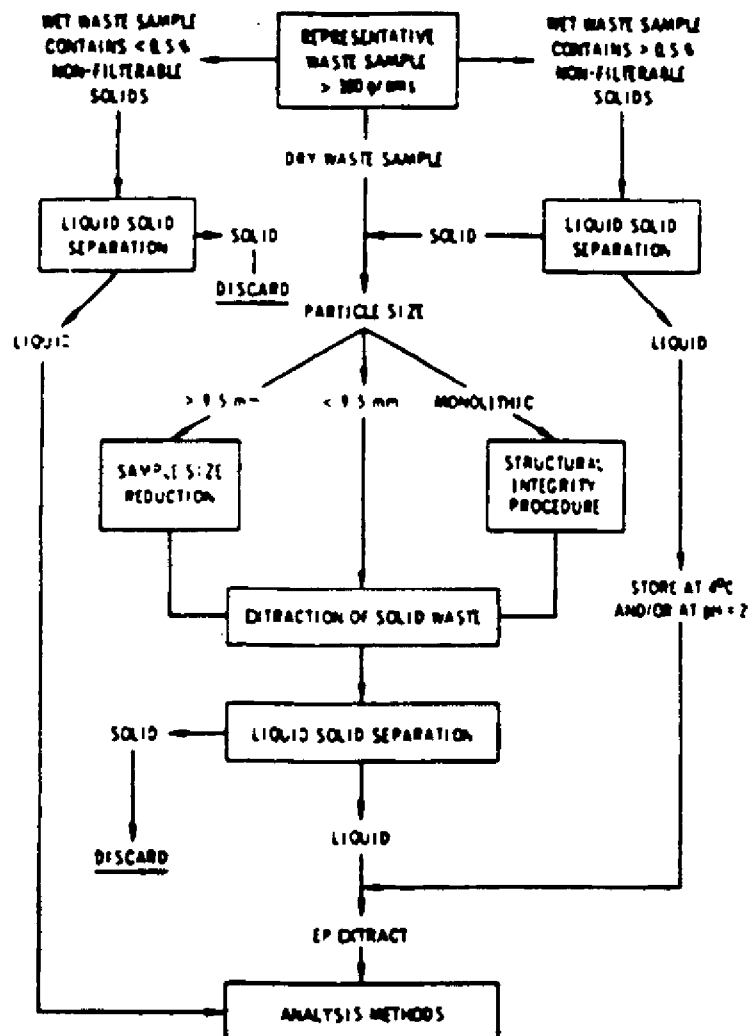


Figure 14. Extraction procedure flowchart.

TABLE 1. AMOUNT OF CHROMATOGRAPHABLE COMPOUNDS DETECTED IN THE BASE/NEUTRAL EXTRACT OF THE INFLUENT SAMPLE, DILUTED TEN TIMES BEFORE ANALYSIS

RT	AREA	WG/L	NAME
3.64	49354	BV 684.169	
3.84	9440	WV 137.072	
4.07	22463	VB 312.313	
5.04	34777	VB 514.780	
5.76	131334	BV 1825.87	
6.17	20322	WV 224.231	
6.38	98460	WV 1363.26	
6.57	46741	BV 647.406	
7.57	3573	VB 51.905	
7.65	1057	VB 14.415	
8.67	2842	BV 37.505	
8.18	673	VB 9.356	
8.19	7133	BV 95.171	
8.48	4061	WV 55.615	
8.54	9427	WV 131.053	
8.75	73064	WV 1035.56	
8.81	16543	WV 227.504	
9.21	301748	WV 4047.99	PHENOL-D5
9.77	34462	BV 506.079	
9.8	12740	WV 177.253	
9.87	1215	VB 16.857	
10.62	22144	BV 446.887	
10.81	57227	WV 517.656	
10.85	171714	WV 2387.54	
10.84	210552	WV 2726.93	
10.81	20767	WV 319.585	CL2-MENZENE-D4
10.81	219466	WV 3051.13	
10.84	20186	WV 280.352	
10.91	6164	WV 83.469	
11.04	5542	WV 74.550	
11.16	14611	WV 205.132	
11.36	8790	WV 80.495	
11.62	859	BV 9.720	
11.60	15155	WV 210.172	
11.88	15524	WV 245.651	
11.93	1502	WV 20.835	
12.04	13433	WV 187.445	
12.16	2318	WV 32.221	
12.27	1061	WV 15.050	
12.54	1546	WV 21.450	
12.44	21354	WV 297.293	
12.56	4948	WV 66.790	
12.67	1879	WV 26.125	
12.74	25160	WV 349.792	

TABLE 1 (continued)

RT	AREA	W/L	NAME
12.77	34176	W	473.163
12.84	12099	W	148.210
12.94	1917	W	26.657
13.04	3405	W	47.399
13.12	1874	W	26.054
13.17	4310	W	60.888
13.25	10453	W	214.923
13.38	8070	W	112.183
13.47	2741	W	33.111
13.52	10747	W	141.170
13.74	1124	W	13.993
13.82	1131	W	16.413
14.01	22107	W	742.001
14.12	6147	W	114.937
14.2	12273	W	1700.14
14.37	23331	W	321.713
14.44	2723	W	32.824
14.4	2174	W	74.425
14.5	622	W	12.044
14.67	272	W	5.134
14.74	241	W	3.442
14.87	2114	W	40.306
14.97	1211	W	111.421
15.0	2134	W	32.322
15.07	1051	W	14.200
15.17	11011	W	424.11
15.24	12473	W	1176.45
15.47	4190	W	60.233
15.61	1710	W	24.044
15.67	2101	W	70.721
15.77	422	W	4.211
15.92	2521	W	74.811
16.11	3111	W	42.042
16.17	18112	W	251.241
16.2	6234	W	87.243
16.37	3637	W	50.357
16.52	2411	W	34.219
16.59	2227	W	31.936
16.73	104647	W	1399.25
16.82	4440	W	63.471
17.01	2717	W	32.205
17.10	1874	W	26.084
17.16	2299	W	31.901
17.24	3010	W	41.842
17.34	2760	W	38.247
17.47	2115	W	29.409
17.52	10313	W	143.372
17.71	263	W	3.623
17.80	6341	W	113.940
18.00	104451	W	1452.13
18.20	14022	W	222.744
18.39	300249	W	3004.34
18.44	7541	W	104.835
18.55	2753	W	38.674
18.61	2590	W	34.008
18.71	944	W	13.378

TABLE 1 (continued)

RT	AREA	Wt%	NAME
18.85	1844 BV	26.186	
18.92	700 WV	9.731	
19.00	4247 WV	67.575	
19.19	1111 WV	15.342	
19.31	11130 WV	154.754	
19.32	12152 WV	169.026	
19.54	605 VB	8.447	
19.65	14440 BV	201.444	
19.82	1074 WV	15.072	
19.91	224 WV	4.260	
19.93	276 VB	3.870	
20.09	872 BV	12.117	
20.19	452 VB	6.278	
20.27	671 BV	9.171	
20.31	950 WV	12.627	
20.44	7252 VV	103.247	
20.51	1111 WV	21.071	
20.69	4375 WV	47.775	
20.77	2415 WV	40.377	
20.79	1554 WV	22.050	
20.81	1911 WV	25.244	
20.81	4025 WV	62.514	
21.09	4510 BV	62.497	
21.17	25411 WV	428.641	
21.21	10111 WV	145.718	
21.34	2481 WV	34.491	
21.57	11244 WV	1271.101	
21.73	1245 WV	17.348	
21.71	471 BV	4.014	
21.80	1464 WV	21.120	
21.83	413 VB	6.021	
21.95	4474 BV	61.205	
22.47	1575 WV	21.595	
22.51	171 VB	11.474	
22.85	4541 BV	61.230	
22.97	8157 WV	68.124	
23.17	12117 WV	251.115	
23.15	15279 WV	267.474	
23.25	10511 WV	150.842	
23.32	5423 WV	75.365	
23.42	10143 WV	141.090	
23.52	2421 WV	31.641	
23.61	28111 WV	373.744	
23.71	5707 WV	154.521	
23.76	7554 WV	105.820	
23.84	4571 WV	63.547	
23.98	305 VB	4.302	
24.07	4542 BV	63.423	
24.16	49078 WV	681.318	
24.24	5491 WV	76.334	
24.32	2193 WV	30.490	
24.42	71974 WV		BANTHRACENE-D10
24.69	1241 VB	17.257	
24.90	462 BV	6.421	
25.03	1160 BV	16.407	
25.23	543 BV	7.548	
25.42	204 BV	2.833	
25.52	1128 WV	15.915	
25.61	2923 VB	40.635	
25.86	911 BV	12.667	
25.99	292 VB	4.064	

TABLE 1 (continued)

RT	AREA	ug/L	NAME
26.15	1855 BV	23.006	
26.24	964 VB	15.405	
26.35	543 BV	7.829	
26.37	542 WV	7.953	
26.56	1916 BV	26.640	
26.71	301 VB	5.396	
26.75	559 BV	7.776	
26.85	3755 WV	51.749	
26.91	2972 VB	54.701	
27.10	1554 BV	21.659	
27.24	1376 VB	19.132	
27.27	908 BV	15.457	
27.41	951 VB	15.785	
27.67	504 BV	6.254	
27.84	513 VB	3.722	
27.86	205 BV	3.545	
28.01	265 BV	4.013	
28.12	265 BV	4.494	
28.17	1554 BV	27.715	
28.25	577 BV	8.016	
28.41	1024 WV	14.141	
28.57	4575 WV	60.877	
28.64	3472 WV	56.985	
28.72	9455 WV	122.767	PYRENE-D10
28.74	1475 BV	20.557	
29.01	221 WV	5.973	
29.05	211 VB	5.611	
29.12	1417 BV	19.715	
29.27	150 BV	5.649	
29.31	215 BV	8.555	
29.47	417 BV	5.767	
29.48	754 BV	11.067	
29.75	220 BV	2.461	
29.85	1220 BV	17.752	
30.25	9257 BV	121.081	
30.74	211 BV	7.656	
30.75	441 BV	6.455	
30.80	654 BV	9.091	
30.84	205 BV	5.741	
31.01	754 BV	10.205	
31.12	104 BV	14.565	
31.25	127004 BV	1661.95	
31.65	1453 BV	20.265	
31.85	215 BV	2.959	
31.89	516 BV	7.206	
32.01	2035 BV	28.345	
32.05	205 BV	2.822	
32.44	344 BV	4.806	
32.54	984 BV	13.704	
32.69	1747 WV	24.573	
32.82	4644 WV	107.285	CHRYSENE-D12
33.06	967 BV	7.868	
33.26	1442 BV	22.633	
33.34	552 BV	7.390	
33.34	442 BV	6.149	
34.00	444 BV	6.757	
34.22	254 BV	3.538	
34.32	251 BV	3.494	
34.48	647 BV	9.002	
34.60	644 BV	9.263	
35.24	1710 BV	23.888	

TABLE 1 (continued)

27.72	708 BP	9.847
31.92	870 BP	11.297
34.42	822 BP	9.151
37.31	1992 BP	27.497
38.77	342 BP	4.765

TOTAL AREA = 3903967 TOTAL ug/L = 84295.414

PROCESSED DATA FILE: BP154 RAW DATA FILE: BR154

NOTE: The above report is generated from the capillary GC/MS analysis of a methylene chloride extract of a basic water sample (see Table 1). The retention time units are minutes; the area units are detector counts and the component concentrations (ug/L) are calculated using authentic standards. If the peak is named, it is assumed the unknown compound response is equal to the internal standard, anthracene-d10. Other deuterated compounds identified are successful when the compounds which are added to the water sample during extraction leave. Each unknown compound was added to produce a water concentration of 100 ug/L.

The above analysis includes all chromatographable compounds with retention times between 20 min and 34.00 min which corresponds to C1 through C14 aromatic hydrocarbons and which can be extracted using the EPA Method 625 Extraction Protocol.

The identification of non-chromatographic pollutant components should be a part of the above analysis at levels greater than 5 ug/L can be carried out. Further information of the mass spectral data obtained from the capillary GC/MS analysis for priority pollutants is discussed in a later section.

TABLE 2. AMOUNT OF CHROMATOGRAPHABLE COMPOUNDS DETECTED IN THE ACID
EXTRACTS OF THE INFLUENT SAMPLE, DILUTED TEN TIMES

RT	AREA	UG/L	NAME
1.11	207 BB	2.776	
1.77	1121 BB	12.447	
2.31	736 BB	9.891	
2.77	546 BB	7.349	
3.77	830 BB	11.136	
7.22	17075 BV	185.422	PHENOL-DS
7.84	10795 BV	146.408	
10.11	1710 BV	20.343	
10.15	2712 BV	42.105	
10.41	4111 BV	57.513	
10.77	7170 BV	896.345	CL2-BENZENE-DA
11.31	425 BV	5.795	
12.01	338 BV	4.543	
12.44	526 BV	7.975	
12.51	32786 BV	4432.748	
12.54	104 BV	1.757	
13.01	6753 BV	85.428	
13.11	21032 BV	286.593	
14.01	908 BV	12.705	
14.17	32774 BV	520.924	
14.17	4415 BV	62.195	
14.51	425 BV	5.715	
15.11	6132 BV	82.321	
15.21	2855 BV	38.131	
15.25	2724 BV	31.219	
15.41	634 BV	9.344	
15.54	5790 BV	50.919	
16.11	546 BV	4.912	
16.34	1341 BV	16.261	
16.51	71417 BV	529.252	
16.64	5454 BV	75.651	
17.18	654 BV	8.850	
17.26	36420 BV	491.922	
17.54	841 BV	11.292	
17.54	2152 BV	28.915	
18.20	274 BV	3.676	
18.21	8479 BV	116.063	
18.45	219 BV	2.949	
18.51	7504 BV	100.840	
20.05	772 BV	10.372	
20.52	120942 BV	1626.83	
20.61	525 BV	7.047	
20.71	2249 BV	30.149	
21.17	1049 BV	14.096	

TABLE 2 (CONTINUED)

RT	AREA	U/L	NAME
21.24	341 VB	0.034	
21.47	425 BB	0.715	
21.61	2601 BB	21.044	
22.15	2601 BB	21.727	
22.60	372 BB	0.726	
22.80	303 BB	0.060	
22.87	303 BB	0.067	
24.17	1044 BB	10.717	
24.41	7444 BB		ANTHRACENE-D10
25.12	71 BB	0.711	
25.40	240 BB	0.217	
25.87	450 BB	0.171	
26.07	114 BB	1.141	
26.26	2754 BB	26.701	
26.41	210 BB	0.141	
26.71	222 BB	0.066	
TOTAL AREA =		24747	TOTAL U/L = 10214.775
PROCESSED DATA FILE: B141		RAW DATA FILE: B141	

NOTE: The above report is generated from the Capillary GC/FID analysis of a methylene chloride extract of an acidic water sample. The retention time units are minutes. The area units are three digit counts and the component concentrations (U/L) are calculated by using authentic standards. If the peak is near, or is, assigned the unknown compound response is equal to the internal standard, anthracene-d10. Other deuterated compounds identified are sufficiently similar compounds which are added to the water sample during extraction process. Each spiking compound was added in creating a water concentration of 100 ug/L.

The above analysis includes all chromatographable compounds with retention indices between 7,000 and 34,000 (which corresponds to C7 through C24 normal hydrocarbons) and which can be extracted using the EPA Method 625 Extraction Protocol.

The search for unidentified pollutant components shown to be present in the above analysis at levels greater than 5 ug/L can be obtained by further interpretation of the mass spectral data obtained from the capillary GC/MS analysis for priority pollutants discussed in a later section.

TABLE 3. AMOUNT OF CHROMATOGRAPHABLE COMPOUNDS IN DETECTED IN THE BASE/NEUTRAL EXTRACT OF THE EFFLUENT SAMPLE, DILUTED TEN TIMES BEFORE ANALYSIS

RT	AREA	UG/L	NAME
3.64	9134	8V 1403.01	
3.87	18434	WV 269.476	
4.04	54441	VB 837.653	
5.08	777	BB 11.913	
5.77	171544	8V 2634.63	
6.11	4247	VB 63.247	
6.17	571	8V 8.776	
6.21	110217	WV 1693.53	
6.54	20413	WV 924.464	
7.21	10000	VB 270.164	
7.74	400	BB 9.810	
7.89	334	BB 3.810	
8.37	5547	8V 87.116	
8.41	2750	WV 291.918	
8.61	10174	WV 151.445	
8.77	10174	WV 1514.11	
8.81	14007	WV 212.754	
9.21	327181	WV 4920.79	PHENOL-D5
9.44	500	VB 7.763	
9.54	41456	8V 634.277	
9.87	2000	WV 34.201	
10.01	40261	WV 449.713	
10.11	54151	WV 872.191	
10.34	62408	WV 1061.55	
10.44	13473	WV 1069.47	
10.51	12471	WV 321.662	CL2-BENZENE-D4
10.61	35471	WV 5451.27	
10.87	11521	WV 517.640	
10.91	1730	WV 42.677	
11.07	1752	WV 26.918	
11.16	5547	WV 82.503	
11.36	2914	WV 44.937	
11.45	220	VB 3.477	
11.59	1405	8V 21.583	
11.64	910	WV 13.983	
11.79	2500	WV 79.900	
11.86	20121	WV 309.186	
12.17	708	BB 10.875	
12.27	604	WV 9.280	
12.34	1071	WV 16.440	
12.44	21866	WV 324.303	
12.58	2933	WV 45.047	
12.74	95790	WV 1555.37	
12.84	17345	WV 272.983	

TABLE 3 (continued)

RT	AREA	Wt%	NAME
13.00	3336	WV	31.262
13.17	6851	WV	105.379
13.25	6784	WV	76.144
13.35	8271	WV	127.393
13.42	9700	WV	149.182
13.74	1292	WV	19.854
13.85	1581	VB	24.249
14.00	3844	BV	599.216
14.12	7802	WV	119.881
14.24	22704	WV	3499.04
14.37	2638	WV	402.462
14.51	2849	VB	44.241
14.70	404	BB	6.264
14.95	27135	BV	416.952
15.21	433320	WV	6652.87
15.34	217521	VB	3229.54
15.71	2018	BV	31.004
15.81	375	BB	5.752
15.97	8204	WV	124.641
16.11	9292	WV	81.315
16.22	1914	WV	29.464
16.51	2411	VB	37.042
16.61	201574	BV	3047.70
16.83	6471	WV	124.315
17.00	292	VB	4.621
17.23	1414	BB	42.549
17.55	274	VB	4.221
17.61	3559	BV	51.175
17.82	10219	WV	127.022
18.00	71221	WV	1154.51
18.24	6923	WV	127.325
18.40	403492	WV	6241.62
18.63	2554	VB	39.277
19.22	14561	BV	230.201
19.35	21672	WV	343.321
19.57	1341	VB	17.824
20.14	3020	BV	46.401
20.24	274	BB	4.214
20.45	4617	BV	70.444
20.64	547	VB	8.405
20.91	2501	BB	37.820
21.28	544	BB	5.847
21.35	318	BB	4.890
21.49	181673	BV	2910.52
21.78	451	VB	6.261
22.19	624	BB	12.440
22.37	927	BB	14.713
22.64	1527	BB	29.742
22.90	210	BB	3.227
23.04	13551	BV	202.250
23.14	7674	WV	89.944
23.25	1048	WV	16.172
23.32	444	VB	6.847
23.44	3370	BV	51.731
23.62	3331	WV	51.181
23.86	4453	WV	99.183
23.88	322	VB	4.944
24.13	1829	BB	28.099

TABLE 3 (continued)

RT	AREA	u/L	NAME
24.41	87179 BA		ANTHRACENE-DIO
24.51	3505 VB	33.836	
25.64	1671 BB	25.344	
26.31	291 BA	6.472	
26.82	953 BA	16.670	
26.84	7017 BA	104.910	PYRENE-DIO
26.87	687 BA	9.966	
26.91	1171 BA	121.152	
26.97	3405 BA	600.101	
26.98	6441 BA	115.526	CHRYSENE-DIO
TOTAL AREA =		419411	TOTAL u/L = 62766.703
REQUESTED DATA FILE: BR157		RAW DATA FILE: BR157	

NOTE: The above report is generated from the capillary GC/MS analysis of a methylene chloride extract of a basic water sample (see Table 1). The retention time units are minutes, the area units are integrator counts and the component concentrations (u/L) are calculated by using authentic standards. If the peak is needed, it is assumed the unknown compound response is equal to the internal standard anthracene-dio. Other deuterated compounds identified are surrogate organic compounds which are added to the water sample before extraction begins. Each surrogate compound was added to produce a water concentration of 100 u/L.

The above analysis includes all chromatographable compounds with mass indices between 7,000 and 24,000 (which corresponds to C7 through C24 normal hydrocarbons) and which can be extracted using the EPA Method 825 Extraction Protocol.

The identity of non-petroleum pollutant components shown to be present in the above analysis at levels greater than 5 u/L can be obtained by further interpretation of the mass spectral data obtained from the capillary GC/MS analysis for priority pollutants discussed in a later section.

TABLE 14. AMOUNTS OF CHROMATOGRAPHABLE COMPOUNDS DETECTED IN THE ACID EXTRACT OF THE EFFLUENT SAMPLE, DILUTED TEN TIMES BEFORE ANALYSIS

RT	AREA	ug/L	NAME
5.11	215 BF	2.921	
7.54	3424 BF	21.545	
9.22	18032 BF	171.544	PHENOL-D5
9.44	17407 BF	132.473	
10.51	555 BF	4.464	
10.57	44751 BF	446.024	CL2-BENZENE-D4
11.4	210 BF	2.601	
12.1	27145 BF	24.151	
12.51	47 BF	2.151	
12.9	40401 BF	100.471	
14.15	6404 BF	112.141	
14.59	10007 BF	141.406	
14.71	111 BF	14.565	
17.14	10 BF	1.001	
17.17	221 BF	27.211	
17.71	40 BF	3.241	
18.59	2471 BF	74.741	
18.71	141 BF	21.551	
18.87	5470 BF	701.201	
18.94	410 BF	51.651	
17.17	10 BF	4.411	
17.21	47015 BF	405.554	
17.41	50 BF	6.441	
17.59	1141 BF	15.211	
18.21	174 BF	10.251	
18.57	744 BF	95.255	
20.04	10 BF	1.272	
20.21	97570 BF	1299.06	
20.41	257 BF	11.657	
21.41	1720 BF	36.321	
21.17	2174 BF	51.505	
22.54	141 BF	4.576	
24.41	74377 BF	54.377	ANTHRACENE-D10
27.44	50 BF	7.915	
28.17	25 BF	3.221	
32.41	50 BF	5.101	
TOTAL AREA =		82672	TOTAL ug/L = 10077.671
PROCESSED DATA FILE: BF162			RAW DATA FILE: BR162

NOTE: The above report is generated from the capillary GC/MS analysis of a methylene chloride extract of an acidic water sample (see C 21). The retention time units are minutes, the area units are detector counts and the component concentrations (ug/L) are calculated by using methylene chloride as the peak response is by assuming the unknown compound response is equal to the internal standard, anthracene-d10. Other deuterated compounds identified are surrogate spiking compounds which are added to the water sample before extraction began. Each spiking compound was added to produce a water concentration of 100 ug/L.

The above analysis includes all chromatographable compounds with Kovats indices between 7,000 and 34,000 (which corresponds to C7 through C34 normal hydrocarbons) and which can be extracted using the EPA Method 625 Extraction Protocol.

The identity of non-priority pollutant components shown to be present in the above analysis at levels greater than 5 ug/L can be obtained by further interpretation of the mass spectral data obtained from the capillary GC/MS analysis for priority pollutants, discussed in a later section.

TABLE 5. AMOUNT OF CHROMATOGRAPHABLE COMPOUNDS DETECTED IN THE
BASE/NEUTRAL EXTRACT OF THE REFLUENT DUPLICATE SAMPLE,
DILUTED TEN TIMES BEFORE ANALYSIS

RT	AREA	ug/L	NAME
3.44	82117 BV	1294.55	
3.64	16161 VV	246.361	
4.04	51354 VV	755.042	
5.07	211 BF	3.282	
5.71	174055 BV	2650.42	
6.15	53334 VV	783.515	
6.37	127145 VV	1938.15	
6.51	71457 VV	111.337	
7.01	16723 VV	249.754	
7.17	211 BF	3.151	
7.17	511 BF	4.645	
8.14	1258 BF	27.267	
8.47	17521 BV	227.417	
8.61	4763 VV	99.445	
9.71	100546 VV	1478.49	
9.91	12367 VV	189.029	
9.91	325445 VV	4994.41	PHENOL-DS
9.84	1519 VV	22.604	
9.91	44103 VV	649.372	
9.95	2175 VV	34.835	
10.01	41717 VV	627.544	
10.11	55457 VV	818.219	
10.24	84321 VV	1294.30	
10.44	145121 VV	2150.61	
10.71	19051 VV	309.626	CL2-BENZENE-DA
10.71	377118 VV	5556.42	
10.87	21167 VV	321.451	
10.91	1150 VV	16.698	
11.07	1112 VV	28.045	
11.14	5445 VV	80.057	
11.56	3101 VV	49.250	
11.56	100 BF	3.023	
11.59	227 BF	3.354	
11.65	221 BF	3.414	
11.75	4544 VV	73.222	
11.81	21175 VV	311.678	
12.17	900 BV	13.215	
12.20	293 VV	4.301	
12.27	543 VV	8.245	
12.34	1165 VV	17.108	
12.45	23012 VV	336.354	
12.51	3614 VV	44.272	
12.74	106214 VV	1219.73	
12.84	17197 VV	252.954	

TABLE 5 (continued)

RT	AREA	W/L	NAME
13.00	3237	WV	67.848
13.17	6141	WV	91.983
13.27	8754	WV	84.023
13.37	7547	WV	110.870
13.47	5997	BV	84.094
13.57	918	WV	13.461
13.67	1405	VB	20.609
14.04	41905	BV	412.604
14.12	8745	WV	122.499
14.24	139155	WV	3214.79
14.37	15141	WV	423.293
14.55	2501	WV	51.901
14.70	840	VB	11.775
14.81	15501	BV	414.761
15.21	471529	WV	6927.05
15.37	214479	WV	3471.06
15.50	541	VB	7.469
15.71	2241	BB	31.925
15.81	47	WV	4.325
15.92	9525	WV	137.872
16.13	15	WV	89.653
16.27	2745	WV	35.032
16.40	2771	VB	40.710
16.6	111417	BV	3414.62
16.7	8479	WV	124.561
17.0	421	VB	6.167
17.1	1151	BB	40.541
17.27	1007	BV	10.301
17.42	6274	WV	91.673
17.67	11745	WV	173.225
17.69	61435	WV	1194.57
18.20	6761	WV	145.474
18.41	459181	WV	6747.12
18.61	250	VB	4.267
18.71	1602	BB	23.557
18.81	17029	BV	254.727
18.91	14441	WV	323.665
19.37	1707	WV	25.071
19.64	727	VB	10.445
20.04	1372	BB	20.164
20.14	402	BB	5.993
20.45	5307	BV	77.965
20.65	743	VB	11.211
20.91	2327	BB	34.179
21.21	464	BB	5.929
21.31	393	BB	5.775
21.70	217290	BV	3192.12
21.79	5215	VB	76.616
22.19	843	BV	12.378
22.37	574	BB	6.194
22.64	2514	BB	37.113
22.90	514	BB	3.147
23.04	13429	BV	224.594
23.15	6409	WV	100.634
23.25	1419	WV	20.647
23.35	884	WV	12.984
23.45	4443	WV	63.889
23.61	3721	WV	84.671

TABLE 5 (continued)

RT	AREA	ug/L	NAME
23.45	6659 VB	97.620	
24.10	7047 BB	30.072	
24.45	61073 BV		ANTHRACENE-D10
24.55	4206 VB	61.790	
24.91	296 BB	4.340	
25.30	227 BB	3.317	
25.74	1847 BB	27.787	
26.47	402 BB	5.906	
26.87	1054 BB	32.420	
27.06	207 BB	3.077	
28.80	6415 BB	126.961	PYRENE-D10
29.44	971 BB	14.287	
30.01	1101 BB	144.457	
31.37	4105 BB	60.1602	
31.93	170 BB	2.457	
32.62	5276 BB	137.451	CHRYSENE-D12
TOTAL AREA =		4489726	TOTAL ug/L = 64778.951
REQUESTED DATA FILE: M154		RAW DATA FILE: M152	

NOTE: The above report is generated from the capillary GC/MS analysis of a methylene chloride extract of a basic water sample (m = 11). The retention time units are minutes. The area units are integrator counts and the component concentrations (ug/L) are calculated using authentic standards. If the peak is needed, it is assumed the unknown compound response is equal to the internal standard, anthracene-D10. Other deuterated compounds identified are carbazole-D10 and benzo[a]pyrene-D10 which are added to the water sample before extraction begins. Each polycyclic compound was added to produce a water concentration of 100 ug/L.

The above analysis includes all chromatographable compounds with Kovats indices between 7.000 and 34.000 (which corresponds to C7 through C14 normal n-alkanes) and which can be extracted using the EPA Method 600 Extraction Protocol.

The identity of non-priority pollutant compounds shown to be present in the above analysis at levels greater than 2 ug/L can be obtained by further interpretation of the mass spectral data obtained from the capillary GC/MS analysis for priority pollutants discussed in a later section.

TABLE 6. AMOUNT OF CHROMATOGRAPHABLE COMPOUNDS DETECTED IN THE ACID EXTRACT OF THE EFFLUENT DUPLICATE SAMPLE, DILUTED TEN TIMES BEFORE ANALYSIS

RT	AREA	ug/L	NAME
3.90	342 BP	4.829	
7.54	3712 BP	50.750	
9.22	15467 BV	146.060	PHENOL-D5
9.4	17121 VP	231.126	
10.57	63055 BP	841.292	CLJ-BENZENE-D4
12.5	241407 BP	3507.25	
13.22	6189 BV	83.041	
13.57	71513 VP	796.523	
13.72	22247 BP	1312.24	
14.12	107705 VP	143.657	
14.24	177 BP	15.061	
15.27	1576 BP	25.176	
15.5	604 BP	8.962	
15.64	5611 BP	75.005	
16.77	1670 BP	21.431	
16.77	52555 BV	704.912	
16.84	7805 VP	104.742	
17.22	42655 BP	572.340	
17.22	550 BP	7.112	
17.27	645 BP	12.657	
17.37	847 BP	11.622	
18.2	7164 BP	94.445	
19.23	75155 BP	1251.25	
19.5	814 BP	10.916	
19.8	1102 BP	24.210	
21.37	1593 BP	34.799	
22.2	170 BP	4.926	
24.42	74217 BP		ANTHRACENE-D10
24.4	320 BP	4.294	
27.67	461 BP	4.203	
28.86	275 BP	3.682	
32.42	301 BP	4.034	
TOTAL AREA =		623134	TOTAL ug/L = 9958.256
PROCESSED DATA FILE: BK163			RAW DATA FILE: BK163

NOTE: The above report is generated from the capillary GC/FID analysis of a methylene chloride extract of an acidic water sample (pH < 2). The retention time units are minutes, the area units are integrator counts and the component concentrations (ug/L) are calculated by using authentic standards if the peak is named, or by assuming the unknown compound response is equal to the internal standard, anthracene-D10. Other deuterated compounds identified are surrogate organic compounds which are added to the water sample before extraction began. Each organic compound was added to produce a water concentration of 500 ug/L.

The above analysis includes all chromatographable compounds with Kovats indices between 7,000 and 26,000 (which corresponds to C7 through C34 normal hydrocarbons) and which can be extracted using the EPA Method 625 Extraction Protocol.

The identity of non-priority pollutant components shown in be present in the above analysis at levels greater than 5 ug/L can be obtained by further interpretation of the mass spectral data obtained from the capillary GC/MS analysis for priority pollutants, discussed in a later section.

TABLE 7. AMOUNT OF CHROMATOGRAPHABLE COMPOUNDS DETECTED IN THE METHYLENE CHLORIDE EXTRACT OF THE SEDIMENT SAMPLE, DILUTED TEN TIMES BEFORE ANALYSIS

RT	AREA	Wt%	NAME
5.42	1110 BV	30.020	
5.67	926 VB	13.173	
5.72	604 BB	11.460	
5.77	2773 BB	99.270	
5.82	2424 BB	35.050	
5.87	1007 BB	19.320	
5.92	1121 BV	31.150	
5.97	5422 BV	84.847	
6.02	4227 WV	60.704	
6.07	1024 VB	21.110	
6.12	1121 VB	10.000	
6.17	5422 BV	64.640	
6.22	4227 WV	69.510	
6.27	14427 BV	110.420	PHENOL-05
6.32	5422 BV	124.210	
6.37	2121 WV	30.310	
6.42	4227 WV	7.607	
6.47	1142 BV	120.040	
6.52	5422 WV	46.780	
6.57	4227 WV	818.220	
6.62	4227 WV	592.070	
6.67	2121 WV	212.900	CL2-BENZENE-14
6.72	5422 WV	5.260	
6.77	2422 BV	34.510	
6.82	1121 VB	24.640	
6.87	4227 WV	52.760	
6.92	1121 VB	11.640	
6.97	2422 BV	3.700	
7.02	5422 WV	112.410	
7.07	4227 WV	87.900	
7.12	4227 WV	65.850	
7.17	2121 BV	7.560	
7.22	2121 BV	3.214	
7.27	1522 WV	22.667	
7.32	1121 VB	17.006	
7.37	2773 BB	3.661	
7.42	2121 BV	3.090	
7.47	2422 BV	3.694	
7.52	233 BB	4.737	
7.57	525 VB	8.315	
7.62	2121 BV	30.710	
7.67	4227 WV	7.076	
7.72	2121 BV	7.306	
7.77	744 WV	111.020	

TABLE 7 (continued)

BT	AREA	W/F	WPC
13.62	3709 BB	88.680	
13.85	3026 BV	71.630	
14.12	974 BV	13.880	
14.41	2443 WV	34.844	
14.76	6130 WV	122.770	
14.91	4648 WV	64.113	
15.11	640 VB	6.344	
15.26	1076 BB	16.306	
15.80	2464 BB	25.344	
16.44	315 BV	4.484	
16.73	270 VB	3.280	
16.88	425 BB	4.020	
16.97	514 BB	7.244	
17.0	270 VB	5.020	
17.20	415 BB	5.680	
17.31	200 VB	2.844	
17.41	757 BB	10.407	
17.60	8844 BV	122.970	
17.60	1711 WV	28.670	
17.66	417 BB	6.929	
18.15	270 VB	3.394	
18.21	270 BB	5.077	
18.26	215 BB	5.104	
18.41	270 VB	13.648	
18.51	2171 BB	75.500	
18.51	1750 VB	24.171	
18.77	240 BB	4.720	
18.81	210 BB	4.410	
18.88	410 BB	5.071	
20.07	1159 BV	10.200	
20.15	264 VB	5.440	
20.25	250 BB	5.059	
20.35	410 BB	9.709	
20.37	270 VB	3.956	
20.45	923 WV	13.135	
20.55	5041 WV	71.764	
20.70	632 VB	8.992	
20.80	4740 BV	65.154	
20.91	2271 WV	32.800	
20.95	222 WV	3.163	
21.16	306 BB	4.358	
21.21	470 BB	5.315	
21.44	1420 BB	20.200	
21.45	471 BB	4.400	
21.98	496 BB	7.024	
22.11	3490 BB	49.453	
22.42	1000 BV	15.074	
22.51	1447 WV	23.431	
22.64	1911 WV	27.037	
22.74	2946 WV	61.907	
22.86	1907 WV	27.122	
22.95	1010 WV	14.345	
23.07	2658 BV	37.813	
23.21	5136 WV	73.088	
23.23	1934 WV	27.512	
23.40	976 WV	14.188	
23.45	1329 WV	18.899	
23.57	1825 WV	24.575	
23.6	864 WV	12.193	

TABLE 7 (continued)

23.37	2.811 W	129.001	
23.84	23.9 W	32.988	
24.00	43699 W		ANTHRACENE-D10
24.30	436 W	4.515	
24.42	223 W	3.172	
25.05	1541 W	22.205	
25.14	1021 W	14.517	
25.21	1226 W	17.476	
25.37	348 W	4.950	
25.47	732 W	10.774	
25.61	395 W	5.424	
25.71	249 W	3.541	
25.81	240 W	3.789	
25.96	394 W	5.599	
26.15	872 W	13.294	
26.21	417 W	4.517	
26.31	292 W	4.204	
26.46	1449 W	23.451	
26.54	2220 W	30.504	
26.65	1340 W	19.341	
26.81	2344 W	33.345	
27.00	650 W	11.855	
27.11	311 W	3.259	
27.21	2972 W	42.394	
27.31	151 W	2.776	
27.71	2100 W	29.849	
27.81	440 W	6.351	
28.12	243 W	34.541	
28.21	174 W	2.712	
28.31	1017 W	114.406	PYRENE-D10
28.54	792 W	11.272	
28.71	334 W	4.756	
28.81	1127 W	14.257	
28.91	1257 W	17.494	
29.14	417 W	4.214	
29.21	1701 W	31.176	
29.31	1196 W	17.045	
29.54	744 W	10.411	
29.61	799 W	11.344	
29.71	435 W	5.940	
29.81	10444 W	270.74	
29.91	1075 W	15.250	
30.07	1498 W	24.997	
30.11	971 W	12.809	
30.21	435 W	89.627	CHRYSENE-D12
30.31	261 W	4.044	
30.41	1421 W	20.214	
TOTAL AREA =		447993	TOTAL ug/g = 8540.514
RAW DATA FILE: MP143			RAW DATA FILE: BR143

NOTE: The above report is generated from the capillary GC/FID analysis of a methylene chloride extract of a sediment sample. The retention time units are minutes, the area units are integrator counts and the component concentrations (ug/g) are calculated by using authentic standards. If the peak is named, we assume the unknown compound response is equal to the internal standard, anthracene-D10. Other deuterated compounds identified are surrogate and line compounds which are added to the sample before extraction began. Each surrogate compound was added to produce a sediment concentration of 200 ug/g.

The above analysis includes all chromatographic compounds with retention indices between 7.000 and 36.000 (which corresponds to...)

TABLE 7 (continued)

to C7 through C34 normal hydrocarbons) and which can be extracted using methylene chloride as an extraction solvent.

The identity of non-priority pollutant components shown to be present in the above analysis at levels greater than 5 ug/g can be obtained by further interpretation of the mass spectral data obtained from the capillary GC/MS available for priority pollutants, discussed in a later section.

TABLE 8 AMOUNT OF CHROMATOGRAPHABLE COMPOUNDS DETECTED IN THE METHYLENE CHLORIDE EXTRACT OF THE SEDIMENT DUPLICATE SAMPLE, DILUTED TEN TIMES BEFORE ANALYSIS

RT	AREA	CONC	NAME
5.43	2151	BV	27.474
5.67	1142	VB	14.676
5.80	1109	BP	14.113
6.00	7470	BV	95.154
6.42	3751	BV	44.874
7.31	1453	BP	18.541
7.44	2544	BP	30.190
7.71	6772	BV	84.084
8.10	2141	VV	26.746
8.17	2112	VV	26.889
8.26	2215	VV	26.341
8.40	7596	VV	97.090
8.54	5927	VB	75.707
8.88	16725	BV	199.917
9.19	10470	BV	121.730
9.31	2212	VV	26.691
9.67	680	VB	8.752
9.85	10771	BV	132.732
9.77	2515	VV	44.894
9.87	20014	VV	694.457
10.06	46050	VV	592.095
10.20	24672	VV	310.917
10.44	211	VB	4.221
10.62	2455	BV	34.236
10.71	2102	VV	26.270
10.74	9014	VV	59.505
10.87	1761	VV	15.615
11.01	1177	VB	14.098
11.07	141	BP	4.151
11.40	6923	BV	113.944
11.52	7207	VV	93.092
11.70	5841	VB	69.754
11.80	274	BP	7.409
11.97	254	BP	3.174
12.11	1840	BV	23.427
12.21	1671	VV	18.810
12.49	205	BP	2.596
12.51	243	BP	3.170
12.54	374	BP	4.774
12.91	2685	BV	34.294
13.01	743	VV	10.122
13.10	751	VV	9.593
13.18	7664	VV	101.713
13.41	210	VB	2.771
13.45	4957	BV	63.305
13.67	5933	VB	75.028

PHENOL-D5

CL7-BENZENE-D4

TABLE 8 (continued)

RT	AREA	wt%	NAME
14.12	1202 BV	12.383	
14.21	205 VB	2.622	
14.44	274 BB	3.302	
14.62	2879 BV	36.771	
14.74	10467 WV	133.688	
14.82	4854 WV	61.992	
15.07	279 VB	3.822	
15.12	555 BB	7.091	
15.24	1247 BB	15.954	
15.81	3232 BB	41.153	
16.14	252 BV	3.254	
16.20	219 VB	2.803	
16.31	317 BB	4.180	
16.61	519 BF	5.574	
16.74	585 BV	5.674	
16.80	269 VB	3.440	
16.87	517 BF	6.859	
16.97	776 BB	9.829	
17.10	419 BB	5.354	
17.21	554 VB	6.351	
17.31	351 BB	4.496	
17.40	501 BV	11.131	
17.61	10611 WV	133.501	
17.80	2001 WV	25.664	
17.97	541 BB	6.941	
18.21	318 BF	4.066	
18.72	1473 BB	13.767	
18.82	5752 BV	73.740	
19.11	1831 BB	24.030	
19.61	419 BB	5.430	
19.82	519 VB	3.941	
19.91	475 BB	6.067	
20.07	1949 BV	17.121	
20.18	511 BV	6.771	
20.20	455 VB	5.855	
20.24	753 BV	10.189	
20.38	265 VB	3.495	
20.41	1023 BV	13.066	
20.51	5345 WV	68.519	
20.71	753 WV	9.359	
20.80	5126 WV	65.448	
20.91	2519 WV	31.055	
20.99	300 VB	4.221	
21.14	591 BV	7.551	
21.21	859 VB	10.970	
21.51	202 BB	2.584	
21.65	1340 BB	19.474	
21.91	443 BB	5.635	
21.99	516 BB	6.595	
22.12	3426 BB	44.558	
22.42	1113 BV	14.217	
22.52	1784 VB	22.780	
22.65	2024 BV	25.852	
22.77	3584 WV	39.448	
22.87	1967 WV	24.867	
22.91	1084 WV	13.848	
23.08	2791 BV	35.648	
23.21	5367 WV	68.355	
23.33	2028 WV	26.031	

TABLE 8 (continued)

RT	AREA	wt%	NAME
22.40	982 W	12.544	
23.40	1378 VB	17.352	
23.51	1833 BV	23.413	
23.69	872 W	11.139	
23.77	9423 W	123.034	
23.81	2544 W	32.498	
24.00	5472 VB		ANTHRACENE-D10
24.30	561 BB	7.148	
24.37	260 BV	3.325	
25.01	1810 W	20.360	
25.15	1058 W	14.025	
25.25	1326 VB	16.951	
25.31	522 BB	6.710	
25.47	751 BB	9.444	
25.61	371 BB	4.824	
25.71	261 BV	3.437	
25.84	501 W	6.394	
25.87	337 VB	4.302	
25.91	464 BB	5.925	
26.11	1448 W	18.494	
26.21	719 VB	9.107	
26.31	551 BB	4.229	
26.41	1224 BV	22.327	
26.54	174 W	25.111	
26.68	1365 W	17.479	
26.81	2544 BV	32.748	
26.91	71 BB	6.505	
27.11	214 BB	2.742	
27.11	75 VB	9.578	
27.24	751 BB	9.219	
27.31	3221 BV	41.148	
27.51	144 W	24.343	
27.57	1547 W	27.171	
27.67	671 VB	8.657	
27.87	516 BB	6.591	
27.97	2644 BV	33.765	
28.07	2041 W	26.064	
28.25	10717 W	107.968	PYRENE-D10
28.54	1141 W	14.354	
28.71	710 BV	3.954	
28.87	1370 BV	17.753	
29.00	484 W	6.213	
29.03	133 VB	3.052	
29.11	285 BB	3.643	
29.24	1404 BV	29.501	
29.31	67 VB	1.154	
29.61	4125 BB	53.174	
29.74	251 BB	3.307	
29.77	284 BB	3.626	
29.80	545 BB	7.016	
30.61	422 BV	5.384	
30.73	803 VB	10.757	
30.99	214874 BV	2776.02	
31.21	1623 VB	20.735	
31.25	424 BB	7.997	
31.67	1974 BV	25.216	
32.19	1562 BV	19.981	
32.36	6124 VB	108.098	CHRYSENE-D12
32.40	260 BB	3.332	
32.77	248 BB	3.389	
32.81	314 BB	4.027	

TABLE 8 (continued)

32.36	392 BB	5.010
32.38	2030 BB	25.933
32.47	293 BB	5.010

TOTAL AREA = 760671 TOTAL WGT = 8780.371

PROCESSED DATA FILE: BP144 RAW DATA FILE: BR144

NOTE: The above report is generated from the capillary GC/MS analysis of a methylene chloride extract of a sediment sample. The retention time units are minutes. The area units are arbitrary units and the component concentrations listed are calculated by using authentic standards. If the peak is named, or by estimating the unknown compound response is equal to the internal standard, entire are listed. Other identified compounds identified are surrogate spike compounds which are added to the sample before extraction step. Each spike compound was added to produce a sediment concentration of 200 ug/g.

The above analysis includes all chromatographable compounds with Kovats indices between 7,000 and 34,000 which corresponds to C7 through C24 normal alkanes and which can be extracted using methylene chloride as an extraction solvent.

The list of nonhydrocarbon pollutant compounds shown to be present in the above analysis at levels greater than 5 ug/g can be obtained. Further interpretation of the mass spectral data obtained from the capillary GC/MS analysis for organic pollutants is given in a later section.

TABLE 9. TOTAL CONCENTRATIONS OF EXTRACTABLE COMPOUNDS PRESENT IN WATER AND SEDIMENT EXTRACTS. ANALYZED USING THREE DIFFERENT METHODS

Extract identification	Total concentrations ($\mu\text{g/L}$ for water sampler and $\mu\text{g/g}$ for sediment samples)		
	GC/FID	GC/MS-Total	GC/MS-PP
Influent base/neutral extract	54,000	20,000	5,200
Influent acid extract	10,000	5,500	12,000
Effluent base/neutral extract	63,000	34,000	3,700
Effluent acid extract	10,000	6,800	11,200
Effluent (duplicate) base/neutral extract	65,000	36,000	3,900
Effluent (duplicate) acid extract	10,000	6,000	10,600
Sediment extract	8,500	9,700	7,810
Sediment (duplicate) extract	8,800	8,900	7,360

TABLE 10. PRIORITY POLLUTANT SCREENING LEVELS GC/MS

Compound	Screening level, $\mu\text{g/L}$	Compound	Screening level, $\mu\text{g/L}$
Acids:		Base/Neutrals:	
2-Chlorophenol	25	1,3-Dichlorobenzene	10
2-Nitrophenol	25	1,4-Dichlorobenzene	10
Phenol	25	Hexachloroethane	10
2,4-Dimethylphenol	25	1,2-Dichlorobenzene	10
2,4-Dichlorophenol	25	Bis(2-chloroisopropyl) ether	10
2,4,6-Trichlorophenol	25	Hexachlorobutadiene	10
4,6-Dinitro-o-cresol	250	1,2,4-Trichlorobenzene	10
2,4-Dinitrophenol	250	Naphthalene	10
p-Chloro-m-cresol	25	Bis(2-chloroethyl) ether	10
Pentachlorophenol	25	Hexachlorocyclopentadiene	10
4-Nitrophenol	25	Nitrobenzene	10
Volatiles:		Bis(2-chloroethoxy) methane	10
Chloromethane	10	2-Chloronaphthalene	10
Dichlorodifluoromethane	10	Acenaphthylene	10
Bromomethane	10	Acenaphthene	10
Vinyl chloride	10	Isophorone	10
Chloroethane	10	Fluorene	10
Methylene chloride	10	2,6-Dinitrotoluene	10
Trichlorofluoromethane	10	1,2-Diphenylhydrazine	10
1,1-Dichloroethylene	10	2,4-Dinitrotoluene	10
1,1-Dichloroethane	10	N-nitrosodiphenylamine	10
Trans-1,2-dichloroethylene	10	Hexachlorobenzene	10
Chloroform	10	4-Bromophenyl phenyl ether	10
1,2-Dichloroethane	10	Phenanthrene/anthracene	10
1,1,1-Trichloroethane	10	Dimethyl phthalate	10
Carbon tetrachloride	10	Diethyl phthalate	10
Bromodichloromethane	10	Fluoranthene	10
Bis(chloromethyl) ether	10	Pyrene	10
1,2-Dichloropropane	10	Di-n-butyl phthalate	10
Trans-1,3-dichloropropene	10	Benzidine	10
Trichloroethylene	10	Butyl benzyl phthalate	10
Dibromochloromethane	10	Chrysene	10
Cis-1,3-dichloropropene	10	Bis(2-ethylhexyl) phthalate	10
1,1,2-Trichloroethane	10	Benz(a)anthracene	10
Benzene	10	Benzo(b)fluoranthene	10
2-Chloroethyl vinyl ether	10	Benzo(k)fluoranthene	10
Bromoform	10	Benzo(a)pyrene	10
Tetrachloroethylene	10	Indeno(1,2,3-cd)pyrene	25
1,1,2,2-Tetrachloroethane	10	Dibenzo(a,h)anthracene	25
Toluene	10	Benzo(g,h,i)perylene	25
Chlorobenzene	10	N-Nitrosodimethylamine	10
Direct injectables:		N-nitrosodi-n-propylamine	10
Acrolein	200	4-Chlorophenyl phenyl ether	10
Acrylonitrile	100	3,3'-Dichlorobenzidine	10
		Di-n-octyl phthalate	10

TABLE 11. SCREENING LEVELS FOR PESTICIDES/PCB's
EXTRACTABLE PRIORITY POLLUTANTS

Compound	Screens (ug/liter)	
	CC/MS	CC/EC
Aldrin	10	1
α-BHC	10	1
β-BHC	10	1
γ-BHC (lindane)	10	1
δ-BHC	10	1
Chlordane	10	1
4,4'-DDT	10	1
4,4'-DDE	10	1
4,4'-DDD	10	1
Dieldrin	10	1
Endosulfan I	10	1
Endosulfan II	10	1
Endosulfan sulfate	10	1
Endrin	10	1
Aroclor 1248	10	1
Aroclor 1260	10	1
Aroclor 1016	10	1
Toxaphene	10	1
Methoxychlor	10	1

TABLE 12. ANALYTICAL RESULTS FOR VOLATILE ORGANIC PRIORITY
POLLUTANTS IN WATER SAMPLES. µg/L (ppb)

Analyte	Sample ID			
	Blank	9:15 a.m.	10:23 a.m.	11:15 a.m.
Methylene chloride	ND ^a	20	80	150
1,2-trans-dichloroethylene	ND	<10	<10	<10
1,1-Dichloroethane	<10 ^b	<10	<10	<10
Chloroform	ND	690	460	430
Dichlorobromomethane	ND	30	20	20
1,1,1-Trichloroethane	ND	10	20	20
Carbon tetrachloride	ND	<10	ND	ND
Trichloroethylene	ND	<10	<10	<10
Benzene	<10	5,350	6,020	5,900
Chlorodibromomethane	ND	<10	<10	<10
Tetrachloroethylene	<10	<10	<10	<10
Toluene	<10	530	710	1,000
Ethylbenzene	<10	430	480	380
Chlorobenzene	<10	2,540	2,660	2,560

^aND - compound not detected.

^b<10 - compound observed, but was below screening level.

TABLE 13. ANALYTICAL RESULTS FOR EXTRACTABLE ORGANIC PRIORITY POLLUTANTS IN WATER SAMPLES, µg/L (ppb)

Analyte	Sample 1b			
	Blank	Influent	Effluent	Effluent duplicate
<u>Base/neutral</u>				
1,3-Dichlorobenzene	ND ^a	60	70	70
1,4-Dichlorobenzene	ND	720	970	1,000
1,2-Dichlorobenzene	ND	700	1,290	1,350
Napthalene	ND	530	350	380
Diethyl phthalate	<10 ^b	ND	<10	<10
Di-n-butyl phthalate	<10	10	<10	<10
Butylbenzyl phthalate	ND	2,990	1,040	1,070
Bis(2-ethylhexyl) phthalate	<10	80	<10	<10
Di-n-octyl phthalate	ND	70	ND	ND
Phenanthrene	<10	ND	<10	<10
Anthracene	<10	ND	<10	<10
Fluoranthene	<10	ND	ND	ND
Pyrene	<10	ND	ND	ND
<u>Acid</u>				
2-Chlorophenol	ND	160	290	260
2-Nitrophenol	ND	2,790	4,760	4,470
Phenol	<25	175	130	110
2,4-Dichlorophenol	ND	390	1,200	1,090
2,4,6-Trichlorophenol	ND	960	1,250	1,110
Pentachlorophenol	ND	30	<25	<25
4-Nitrophenol	ND	4,380	3,580	3,540

^aND - compound not detected.

^b"<" - compound observed, but was below screening level.

TABLE 14. ANALYTICAL RESULTS FOR EXTRACTABLE ORGANIC PRIORITY POLLUTANTS IN SEDIMENT SAMPLES, $\mu\text{g/g}$ (ppm)

Analyte	Sample ID	
	Sludge	Sludge duplicate
1,3-Dichlorobenzene	20	10
1,4-Dichlorobenzene	240	180
1,2-Dichlorobenzene	330	240
Napthalene	70	60
Di-n-butyl phthalate	30	20
Butylbenzyl phthalate	7.100	6.820
Di-n-octyl phthalate	20	30

TABLE 15. QUALITY CONTROL DATA FOR ORGANIC PRIORITY POLLUTANT ANALYSIS OF WATER SAMPLES

Spike ^a	Sample ID, percent recovery			
	Blank	Influent	Effluent	Effluent duplicate
Dichlorobenzene-d ₄	66	56	88	80
Phenol-d ₅	39	28	30	26
Pyrene-d ₁₀	117	29	20	21
Chrysene-d ₁₂	64	31	73	82

^aSpike - 100 $\mu\text{g/L}$.

TABLE 16. QUALITY CONTROL DATA FOR ORGANIC PRIORITY POLLUTANT ANALYSIS OF SEDIMENT SAMPLE

Spike ^a	Sample ID, percent recovery	
	Sediment	Sediment duplicate
Dichlorobenzene-d ₄	53	50
Phenol-d ₅	53	39
Pyrene-d ₁₀	53	64
Chrysene-d ₁₂	49	63

^aSpike level

Sediment - 187 $\mu\text{g/g}$

Sediment duplicate - 145 $\mu\text{g/g}$.

TABLE 17. NON-PRIORITY POLLUTANT ORGANIC COMPOUNDS DETECTED IN THREE VOLATILE SAMPLES FROM SAUGET TREATMENT PLANT

RT (min)	Tentative identification ^b	Concentration ^a (µg/L)		
		Sample identification		
		9:15 a.m.	10:23 p.m.	11:15 a.m.
9.9	Unknown (44, 43, 42, 41) ^c	1	1	ND ^d
13.4	Unknown (56, 43, 41, 39)	ND	4	ND
15.5	Acetone	17	13	24
17.1	Unknown (45, 43, 59)	29	70	35
20.6	Methyl ethyl ketone	1	4	8
22.0	Unknown (43, 42, 41)	8	29	10
28.9	Methyl isobutyl ketone	86	170	98
33.6	Unknown (43, 58, 57, 59)	17	18	47
36.8	Unknown (81, 43, 154, 108)	22	44	21
38.0	Benzaldehyde	55	81	33
39.5	Xylene-isomer	890	850	670

^a Estimated concentration based on the area of the major ion of the identified compound to the major ion area of benzene-d₆, internal standard.

^b Tentative identification, not confirmed with authentic standards.

^c Major masses of unknown compounds are listed in parentheses according to decreasing intensities.

^d ND - detection limit is 1 µg/L.

TABLE 18. IDENTIFICATION OF MAJOR EXTRACTABLE ORGANIC COMPOUNDS IN THE BASE/NETURAL EXTRACT OF THE METHOD BLANK^a

Peak No	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, µg/L	Amount spiked, µg/L	Percent recovery
<u>Internal standard^b</u>						
2	21.28	Anthracene-d ₁₀	109007	100	-	-
<u>Recovery standard^{c,d}</u>						
1	7.83	1,2-Dichlorobenzene-d ₄	279584	66	100	66.0
3	25.42	Pyrene-d ₁₀	425301	117	100	117.0
4	29.27	Chrysene-d ₁₂	197093	64	100	64.0
<u>Organics detected</u>						
None detected						
TOTAL CONCENTRATION						

^aIncludes all substances that were found at or above a 5% threshold on the BATCH peak identification program in the Hewlett-Packard system, corresponding to a detection limit of 2 µg/L.

^bAnthracene-d₁₀ internal standard added immediately prior to analysis; 50 µg/L injected equivalent to 100 µg/L in original sample.

^cDeuterated recovery standard (spike), added to original sample before extraction.

^dQuantitated relative to area of major ion for authentic standard.

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TABLE 19. IDENTIFICATION OF MAJOR EXTRACTABLE ORGANIC COMPOUNDS IN THE ACID EXTRACT OF THE METHOD BLANK^a

Peak No	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, µg/L	Amount spiked, µg/L	Percent recovery
<u>Internal standard^b</u>						
2	21.28	Anthracene-d ₁₀	370013	100	-	-
<u>Recovery standard^{c,d}</u>						
1	6.70	Phenol-d ₅	39178	39	100	39.5
<u>Organics detected</u>						
None detected						
TOTAL CONCENTRATION						

^aIncludes all substances that were found at or above a 5% threshold on the BATCH peak identification program in the Hewlett-Packard system, corresponding to a detection of limit of 2 µg/L.

^bAnthracene-d₁₀ internal standard added immediately prior to analysis; 50 ng/L injected equivalent to 100 µg/L in original sample.

^cDeuterated recovery standard (spike), added to original sample before extraction.

^dQuantitated relative to area of major ion for authentic standard.

TABLE 20. IDENTIFICATION OF MAJOR EXTRACTABLE ORGANIC COMPOUNDS IN THE BASE/NEUTRAL EXTRACT OF THE INFLUENT SAMPLE, DILUTED 1:10, NOT CORRECTED FOR BACKGROUND^{a,b}

Peak No.	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, $\mu\text{g/L}$	Amount spiked, $\mu\text{g/L}$	Percent recovery
Internal standard ^c						
29	21.27	Anthracene- d_{10}	353040	1000	-	-
Recovery standard ^{d,e}						
		1,2-Dichlorobenzene- d_4		54	100	56.0
		Pyrene- d_{10}		29	100	29.0
		Chrysene- d_{12}		31	100	31.0
Organics detected ^{f,g,h}						
1	3.22	Chlorobenzene	181167	5.1×10^2		
2	3.73	Xylene (isomer)	149084	4.2×10^2		
3	4.32	Xylene (isomer)	71867	2.0×10^2		
4	6.08	Benzaldehyde and C_7 -benzene (isomer)	171284	4.9×10^2		
5	6.58	Aniline	411008	1.2×10^3		
6	6.90	C_7 -benzene (isomer)	113840	3.2×10^2		
7	7.33	Dichlorobenzene (isomer)	138653	3.9×10^2		

(continued)

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TABLE 20 (continued)

Peak No.	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, $\mu\text{g/L}$	Amount spiked, $\mu\text{g/L}$	Percent recovery
Organics detected (continued)						
8	7.47	Unknown (111,125,154, 71)	45052	1.3×10^2		
9	7.58	C ₃ -benzene (isomer)	17707	50		
10	7.70	C ₄ -benzene (isomer)	588111	1.7×10^1		
11	7.87	Dichlorobenzene (isomer)	411639	1.2×10^1		
12	8.02	Benzyl alcohol	262696	7.4×10^2		
13	9.13	Unknown (119,121,117,93,91)	36927	1.0×10^2		
14	10.10	Chloroaniline (isomer)	145425	4.1×10^2		
15	11.23	Naphthalene and/or azulene and/or isomer	114324	3.2×10^2		
16	11.57	Unknown (91,148,163,92)	255942	7.2×10^2		
17	11.68	Chloroaniline (isomer)	42047	1.2×10^2		
18	12.20	Chloronitrobenzene (isomer)	41779	1.2×10^2		
19	12.47	Chloronitrobenzene (isomer)	995000	2.8×10^3		
20a	12.60	Chloronitrobenzene (isomer)	477200	1.4×10^3		
20b	14.1	N-phenyl formamide	165858	4.7×10^2		
21	15.18	Dichloronitrobenzene (isomer)	434779	1.2×10^3		
22	15.38	Dichloronitrobenzene (isomer)	101830	2.9×10^2		
23	15.55	Nitroaniline (isomer)	774189	2.2×10^3		
24	15.57	Nitroaniline (isomer)	12295	35		
25	15.60	Nitroaniline (isomer)	21936	62		
26	18.43	C ₁₀ -alkene or cycloalkane	66006	1.9×10^2		

(continued)

TABLE 20 (Continued)

Peak No.	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, $\mu\text{g/L}$	Amount spiked, $\mu\text{g/L}$	Percent recovery
<u>Organics detected (continued)</u>						
27	18.73	Nitroaniline	172253	4.9×10^2		
28	20.28	C ₈ -phenol	68944	2.0×10^2		
30	24.52	Sulfur (S ₈)	104131	2.9×10^2		
31	28.15	Butyl benzyl phthalate	577042	1.6×10^3		
TOTAL CONCENTRATION ¹				20.0×10^3		

^aFor background, see method blank analysis, Table 18.

^bIncludes all substances that were found at or above a 5% threshold on the BATCH peak identification program in the Hewlett-Packard system, corresponding to a detection of limit of 100 $\mu\text{g/L}$.

^cAnthracene-d₁₀ internal standard added immediately prior to analysis; 50 mg/L injected equivalent to 1000 $\mu\text{g/L}$ in original sample.

^dDeuterated recovery standard (spike), added to original sample before extraction.

^eQuantitated relative to area of major ion for authentic standard.

^fEstimated concentration based on area of total ion relative to that of anthracene-d₁₀ internal standard.

^gTentative identification, not confirmed with authentic standards.

^hMajor masses of unknown compounds are listed in parentheses according to decreasing intensities. Possible molecular ions are underlined.

ⁱSummation of concentrations of detected compounds, excluding standards.

TABLE 21. IDENTIFICATION OF MAJOR EXTRACTABLE ORGANIC COMPOUNDS IN THE ACID EXTRACT OF THE INFLUENT SAMPLE, DILUTED 1:10, NOT CORRECTED FOR BACKGROUND^{a, b}

Peak No.	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, µg/L	Amount spiked, µg/L	Percent recovery
Internal standard ^c						
10	21.28	Anthracene-d ₁₀	431025	1000	-	-
Recovery standard ^{d, e}						
		Phenol-d ₅		28	100	28.0
Organics detected ^{f, g, h}						
1	8.00	Benzenemethanol	154148	3.6×10^2		
2-3	10.25	Nitrophenol isomer	1129666	2.6×10^3		
4	10.92	Dichlorophenol isomer	100080	2.3×10^2		
5	11.65	Mixture of 25% dichlorophenol isomer and 75% chlorophenol isomer	133692	3.1×10^2		
6	14.02	Cl-containing unknown (121, 93.66, 173)	128939	3.0×10^2		
7	14.50	Trichlorophenol isomer	357472	8.3×10^2		

(continued)

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TABLE 21 (continued)

Peak No.	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, $\mu\text{g/L}$	Amount spiked, $\mu\text{g/L}$	Percent recovery
Organics detected (continued)						
8	16.02	Cl ₂ -containing unknown (<u>207</u> 209.97.133)	46427	1.1×10^2		
9	17.63	Nitrophenol isomer	317844	7.4×10^2		
TOTAL CONCENTRATION ¹				5.5×10^1		

^aFor background, see method blank analysis, Table 19.

^bIncludes all substances that were found at or above a 5% threshold on the BATCH peak identification program in the Hewlett-Packard system, corresponding to a detection of limit of 80 $\mu\text{g/L}$.

^cAnthracene-d₁₀ internal standard added immediately prior to analysis; 50 $\mu\text{g/L}$ injected equivalent to 1000 $\mu\text{g/L}$ in original sample.

^dDeuterated recovery standard (spike), added to original sample before extraction.

^eQuantitated relative to area of major ion for authentic standard.

^fEstimated concentration based on area of total ion relative to that of anthracene-d₁₀ internal standard.

^gTentative identification, not confirmed with authentic standards.

^hMajor masses of unknown compounds are listed in parentheses according to decreasing intensities. Possible molecular ions are underlined.

ⁱSummation of concentrations of detected compounds, excluding standards.

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TABLE 22. IDENTIFICATION OF MAJOR EXTRACTABLE ORGANIC COMPOUNDS IN THE
BASE/NEUTRAL EXTRACT OF THE EFFLUENT SAMPLE, DILUTED 1:10,
NOT CORRECTED FOR BACKGROUND^{a,b}

Peak No.	Retention Time, min	Compound (major masses)	Peak area, total counts	Amount detected, µg/L	Amount spiked, µg/L	Percent recovery
<u>Internal standard^c</u>						
29	21.28	Anthracene-d ₁₀	375998	1000	-	-
<u>Recovery standard^{d,e}</u>						
		1,2-Dichlorobenzene-d ₄		88	100	88.0
		Pyrene-d ₁₀		20	100	20.0
		Chrysene-d ₁₂		73	100	73.0
<u>Organics detected^{f,g,h}</u>						
1	3.25	Chlorobenzene	423801	1.1×10^3		
2	3.77	Xylene (isomer)	230111	6.1×10^2		
3	4.37	Xylene (isomer)	125069	3.3×10^2		
4	6.12	Mixture of 95% benzaldehyde and 5% C ₃ -benzene (isomer)	296079	7.9×10^2		
5	6.63	Aniline	577450	1.5×10^3		
6	6.92	C ₃ -benzene (isomer)	170110	4.5×10^2		

(continued)

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TABLE 22 (continued)

Peak No.	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, $\mu\text{g/L}$	Amount spiked, $\mu\text{g/L}$	Percent recovery
Organics detected (continued)						
7	7.33	Dichlorobenzene (isomer)	387403	1.0×10^3		
8	7.50	Unknown (111,125,154.71)	144063	3.8×10^2		
9	7.70	C ₆ -benzene (isomer)	493985	1.3×10^3		
10	7.88	Dichlorobenzene (isomer)	847373	2.3×10^3		
11	8.08	Benzyl alcohol	719824	1.9×10^3		
12	10.12	50% Propyl benzene and 50% chloroaniline (isomer)	357095	9.5×10^2		
13	11.23	Naphthalene and/or azulene and/or isomer	121498	3.2×10^2		
14	11.60	Unknown (91,148,163.92)	825794	2.2×10^3		
15	11.68	Mixture of 67% chloroaniline (isomer) and 33% chlorophenol (isomer)	142507	3.8×10^2		
16	12.20	Chloronitrobenzene	124660	3.3×10^2		
17						
18	12.48	Chloronitrobenzene (isomer)	2210187	5.9×10^3		
19	12.65	Chloronitrobenzene (isomer)	1107879	2.9×10^3		
20	14.12	Mixture of 80% N-phenyl formamide and 20% unknown (151,153)	577294	1.5×10^3		
21	15.18	Dichloronitrobenzene (isomer)	562688	1.5×10^3		
22	15.37	Dichloronitrobenzene (isomer)	68923	1.8×10^2		
23-						
25	15.55	Nitroaniline (isomer)	1340059	3.6×10^3		

(continued)

TABLE 22 (continued)

Peak No	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, $\mu\text{g/L}$	Amount spiked, $\mu\text{g/L}$	Percent recovery
<u>Organics detected (continued)</u>						
26	16.45	Chloronitroaniline (isomer)	66353	1.8×10^2		
27	16.57	Ethoxynitrobenzene (isomer)	91565	2.4×10^2		
28	18.73	Nitroaniline (isomer)	487626	1.3×10^3		
30	28.13	Butyl benzyl phthalate	323572	8.6×10^2		
TOTAL CONCENTRATION ¹				34.0×10^3		

^aFor background, see method blank analysis, Table 18.

^bIncludes all substances that were found at or above a 5% threshold on the BATCH peak identification program in the Hewlett-Packard system, corresponding to a detection limit of 200 $\mu\text{g/L}$.

^cAnthracene- d_{10} internal standard added immediately prior to analysis, 50 ng/L injected equivalent to 1000 $\mu\text{g/L}$ in original sample.

^dDeuterated recovery standard (spike), added to original sample before extraction.

^eQuantitated relative to area of major ion for authentic standard.

^fEstimated concentration based on area of total ion relative to that of anthracene- d_{10} , internal standard.

^gTentative identification, not confirmed with authentic standards.

^hMajor masses of unknown compounds are listed in parentheses according to decreasing intensities. Possible molecular ions are underlined.

¹Summation of concentrations of detected compounds excluding standards.

TABLE 23. IDENTIFICATION OF MAJOR EXTRACTABLE ORGANIC COMPOUNDS IN THE ACID EXTRACT OF THE EFFLUENT SAMPLE, DILUTED 1:10, NOT CORRECTED FOR BACKGROUND^{a,b}

Peak No	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, µg/L	Amount spiked, µg/L	Percent recovery
Internal standard ^c						
10	21.28	Anthracene-d ₁₀	415039	1000	-	-
Recovery standard ^{d,e}						
		Phenol-d ₅		10	100	10.0
Organics detected ^{f,g,h}						
1	6.77	Chlorophenol isomer	81181	2.0×10^2		
2	8.00	Benzenemethanol	174088	4.2×10^2		
3	10.25	Nitrophenol isomer	920624	2.2×10^3		
4	10.92	O-chlorophenol isomer	370139	8.9×10^2		
5	11.65	Mixture of 25% dichlorophenolisomer and 75% chlorophenol isomer	353570	8.5×10^2		
6	14.03	Cl-containing unknown (121, 93, 66, 173)	192132	4.6×10^2		
7	14.52	Trichlorophenol isomer	470451	1.1×10^3		

(continued)

TABLE 23 (continued)

Peak No	Retention time min	Compound (major masses)	Peak area total counts	Amount detected $\mu\text{g/L}$	Amount spiked $\mu\text{g/L}$	Percent recovery
Organics detected (continued)						
8	16.02	Cl ₂ -containing unknown (207, 97, 209, 133)	42090	1.9×10^2		
9	17.62	Nitrophenol isomer	235463	5.7×10^2		
TOTAL CONCENTRATION ⁱ				6.8×10^3		

^aFor background, see method blank analysis, Table 19.

^bIncludes all substances that were found at or above a 5% threshold on the BATCH peak identification program in the Hewlett-Packard system, corresponding to a detection limit of 70 $\mu\text{g/L}$.

^cAnthracene-d₁₀ internal standard added immediately prior to analysis, 50 ng/L injected equivalent to 1000 $\mu\text{g/L}$ in original sample.

^dDeuterated recovery standard (spike), added to original sample before extraction.

^eQuantitated relative to area of major ion for authentic standard.

^fEstimated concentration based on area of total ion relative to that of anthracene-d₁₀ internal standard.

^gTentative identification, not confirmed with authentic standards.

^hMajor masses of unknown compounds are listed in parentheses according to decreasing intensities. Possible molecular ions are underlined.

ⁱSummation of concentrations of detected compounds, excluding standards.

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TABLE 24. IDENTIFICATION OF MAJOR EXTRACTABLE ORGANIC COMPOUNDS IN THE
BASE/NEUTRAL EXTRACT OF THE EFFLUENT DUPLICATE SAMPLE,
DILUTED 1:10. NOT CORRECTED FOR BACKGROUND^{a, b}

Peak No	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, µg/L	Amount spiked, µg/L	Percent recovery
Internal standard ^c						
28	21.28	Anthracene-d ₁₀	361258	1000	-	-
Recovery standard ^{d, e}						
		1,2-Dichlorobenzene-d ₄		80	100	80.0
		Pyrene-d ₁₀		21	100	21.0
		Chrysene-d ₁₂		82	100	82.0
Organics detected ^{f, g, h}						
1	3.23	Chlorobenzene	344954	9.5×10^2		
2	3.78	Xylene (isomer)	226741	6.3×10^2		
3	4.33	Xylene (isomer)	153266	4.2×10^2		
4	6.10	Mixture of 95% benzaldehyde and 5% C ₃ -benzene (isomer)	235243	6.5×10^2		
5	6.63	Aniline	603662	1.7×10^3		

(continued)

TABLE 24 (continued)

Peak No	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, $\mu\text{g/L}$	Amount spiked, $\mu\text{g/L}$	Percent recovery
Organics detected (continued)						
6	6.92	C ₃ -benzene (isomer)	211513	5.9×10^2		
7	7.35	Dichlorobenzene (isomer)	341836	9.5×10^2		
8	7.72	C ₄ -benzene (isomer)	501962	1.4×10^3		
9	7.88	Oichlorobenzene (isomer)	824287	2.3×10^3		
10	8.10	Benzyl alcohol	748664	2.1×10^3		
11	10.12	Mixture of 70% chloroaniline (isomer) and 30% unknown (91.120, 133.134)	355915	9.9×10^2		
12	11.23	Naphthalene and/or azulene and/or isomer	126080	3.5×10^2		
13	11.58	Unknown (91.148, 163.92)	815700	2.3×10^3		
14	11.70	Mixture of 70% chloroaniline (isomer) and 30% chlorophenol (isomer)	134788	3.7×10^2		
15	12.22	Chloronitrobenzene (isomer)	131556	3.6×10^2		
16	12.53	Chloronitrobenzene (isomer)	2317029	6.4×10^3		
17 &						
18a	12.65	Chloronitrobenzene (isomer)	1169099	3.2×10^3		
18b	14.1	N-phenyl formamide	591344	1.6×10^3		
19	15.20	Dichloronitrobenzene (isomer)	598057	1.7×10^3		
20	15.37	Dichloronitrobenzene (isomer)	72987	2.0×10^2		
21-						
24	15.55	Nitroaniline	1392402	3.9×10^3		

(continued)

TABLE 24 (continued)

Peak No	Retention time, min	Compound (major masses)	Peak area total counts	Amount detected, $\mu\text{g/L}$	Amount spiked, $\mu\text{g/L}$	Percent recovery
Organics detected (continued)						
25	16.45	Chloronitroaniline	67294	1.9×10^2		
26	16.57	Ethoxynitrobenzene	90437	2.5×10^2		
27	18.80	Nitroaniline and 5% chloronitroaniline	480195	1.3×10^3		
29	28.13	Butyl benzyl phthalate	324456	9.0×10^2		
TOTAL CONCENTRATION ¹				35.7×10^3		

^aFor background, see method blank analysis, Table 18.

^bIncludes all substances that were found at or above a 5% threshold on the BATCH peak identification program in the Hewlett-Packard system, corresponding to a detection of limit of 200 $\mu\text{g/L}$.

^cAnthracene- d_{10} internal standard added immediately prior to analysis; 50 mg/L injected equivalent to 1000 $\mu\text{g/L}$ in original sample.

^dDeuterated recovery standard (spike), added to original sample before extraction.

^eQuantitated relative to area of major ion for authentic standard.

^fEstimated concentration based on area of total ion relative to that of anthracene- d_{10} , internal standard.

^gTentative identification, not confirmed with authentic standards.

^hMajor masses of unknown compounds are listed in parentheses according to decreasing intensities. Possible molecular ions are underlined.

¹Summation of concentrations of detected compounds, excluding standards.

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TABLE 25. IDENTIFICATION OF MAJOR EXTRACTABLE ORGANIC COMPOUNDS IN THE ACID EXTRACT OF THE EFFLUENT DUPLICATE SAMPLE, DILUTED 1:10, NOT CORRECTED FOR BACKGROUND^{a, b}

Peak No.	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, $\mu\text{g/L}$	Amount spiked, $\mu\text{g/L}$	Percent recovery
Internal standard ^c						
10	21.28	Anthracene- d_{10}	425158	1000	-	-
Recovery standard ^{d, e}						
		Phenol- d_6		26	100	26.0
		1,2-Dichlorobenzene- d_4				
		Pyrene- d_{10}				
		Chrysene- d_{12}				
Organics detected ^{f, g, h}						
1	6.77	Chlorophenol isomer	60426	1.4×10^2		
2	7.98	Benzenemethanol	130238	3.1×10^2		
3	10.23	Nitrophenol isomer	861552	2.0×10^3		
4	10.92	Dichlorophenol isomer	331313	7.8×10^2		
5	11.65	Mixture of 25% dichlorophenol isomer and 75% chlorophenol isomer	324950	7.6×10^2		

(continued)

TABLE 25 (continued)

Peak No	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, $\mu\text{g/L}$	Amount spiked, $\mu\text{g/L}$	Percent recovery
Organics detected (continued)						
6	14 05	Cl-containing unknown (121, 73, 66, <u>171</u>)	193047	4.5×10^2		
7	14 52	Trichlorophenol isomer	416595	8.8×10^2		
8	16 02	Cl ₂ -containing unknown (<u>207</u> , 97, 133, 209)	34361	81		
9	17 62	Nitrophenol isomer	241521	5.7×10^2		
TOTAL CONCENTRATION ¹				6.0×10^3		

^aFor background, see method blank analysis, Table 19.

^bIncludes all substances that were found at or above a 5% threshold on the BATCH peak identification program in the Hewlett-Packard system, corresponding to a detection limit of 70 $\mu\text{g/L}$.

^cAnthracene- d_{10} internal standard added immediately prior to analysis; 50 mg/L injected equivalent to 1000 $\mu\text{g/L}$ in original sample.

^dDeuterated recovery standard (spike), added to original sample before extraction.

^eQuantitated relative to area of major ion for authentic standard.

^fEstimated concentration based on area of total ion relative to that of anthracene- d_{10} internal standard.

^gTentative identification, not confirmed with authentic standards.

^hMajor masses of unknown compounds are listed in parentheses according to decreasing intensities. Possible molecular ions are underlined.

ⁱSummation of concentrations of detected compounds, excluding standards.

TABLE 26. IDENTIFICATION OF MAJOR EXTRACTABLE COMPOUNDS IN THE METHYLENE CHLORIDE EXTRACT OF THE SEDIMENT SAMPLE, DILUTED 1:10, NOT CORRECTED FOR BACKGROUND

Peak No	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, µg/g	Amount spiked, µg/g	Percent recovery
<u>Internal standard^b</u>						
24	21.15	Anthracene-d ₁₀	158680	935	-	-
<u>Recovery standard^{c,d}</u>						
4	6.58	Phenol-d ₅	9676	99	187	53.0
8b	7.7	1,2-Dichlorobenzene-d ₄	35670	99	187	53.0
30	25.25	Pyrene-d ₁₀	10800	99	187	53.0
40	29.10	Chrysene-d ₁₂	11207	92	187	49.0
<u>Organics detected^{e,f,g}</u>						
1	3.62	1,4-Dimethylbenzene (isomer)	13562	80		
2	5.53	Bicyclo[2.2.1]heptane, 2,2-dimethyl-3-methylene - or Bicyclo[3.1.1]hept-2-ene, 2,6,6-trimethyl-cyclohexene, 5-methyl-3-(1-methylethenyl)-, <u>trans</u> -(-)	11247	66		

(continued)

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TABLE 26 (continued)

Peak No.	Retention Time, min	Compound (major masses)	Peak area, total counts	Amount detected, $\mu\text{g/g}$	Amount spiked, $\mu\text{g/g}$	Percent recovery
Organics detected (continued)						
3	5.95	C ₃ -benzene (isomer) or 1,3-cyclopentadiene, 5-(1-methylpropylidene)	13927	82		
5	6.73	C ₃ -benzene (isomer)	24832	1.5×10^2		
6	7.20	Dichlorobenzene (isomer)	31070	1.8×10^2		
7	7.57	C ₄ -benzene (isomer)	249691	1.5×10^3		
8a	7.63	80% C ₄ -benzene (isomer) and 20% unknown (93, 107, 121, 136)	71340	4.2×10^2		
8b	7.7	30% Cl ₂ -benzene (isomer) and 70% Cl ₂ -benzene-d ₄ spiking standard	35670	2.1×10^2		
9	9.00	50% C ₄ -benzene (isomer) and 50% unknown (109, 93, 107, 136)	12172	71		
10	10.62	Unknown (152, 87, 88, 154)	9322	55		
11	11.10	Bicyclo[4.4.1]undeca-1,3,5,7,9-pentaen-11-one and/or naphthalene	9181	54		
12	12.27	Chloronitro-benzene (isomer)	25310	1.5×10^2		
13	12.42	Chloronitro-benzene (isomer)	8718	51		
14	15.03	Dichloronitrobenzene (isomer)	21225	1.3×10^2		
15	16.47	Unknown (97, 69, 83, 70, 119)	3677	22		
16	18.07	Unknown (83, 97, 69, 111, 70)	6360	37		
17	18.30	Unknown (97, 83, 111, 69)	7763	46		
18	19.57	Unknown (97, 83, 111, 69)	4591	27		

(continued)

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TABLE 76 (continued)

Peak No.	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, $\mu\text{g/g}$	Amount spiked, $\mu\text{g/g}$	Percent recovery
Organics detected (continued)						
19	20.02	Phenol, 4-(1,1,1,3-tetramethylbutyl)	6362	37		
20	20.13	Nonyl phenol (isomer)	8281	49		
21	20.27	Nonyl phenol (isomer)	9903	58		
22a	20.4	Unknown (135, 107, 121, 134)	2760	16		
22b	20.50	Unknown (91, 210, 121, 85, 107)	6279	49		
22c	20.5	Unknown (149, 107, 91, 121)	2760	16		
23	20.67	Unknown (135, 107, 136, 91)	5189	31		
25	23.57	Unknown (91, 119, 134, 167)	3857	23		
26	23.80	Unknown (134, 97, 83, 91)	4411	26		
27	24.08	Unknown alkyl benzene (105, 106, 134)	10945	65		
28	24.35	Sulfur, molecular (S_8)	8845	52		
29	24.90	Unknown (150, 97, 83, 164)	4877	29		
31	25.37	Unknown (91, 105, 150)	4764	28		
32	26.50	Unknown alkyl benzene (105, 106, 91)	6037	36		
33	26.88	Benzenamine, 4-nitro-N-phenyl-	6617	39		
34	27.63	Unknown (91, 115, 149, 119, 133)	3613	21		
35	27.88	Unknown (91, 149, 147)	3534	21		
36-						
37	28.03	Unknown phthalate (149, 91, 206, 150)	968426	5.7×10^3		

(continued)

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TABLE 26 (continued)

Retention				Amount	Amount	
Peak	time		Peak area	detected	spiked	Percent
No	min	Compound (major masses)	total counts	µg/g	µg/g	recovery
<u>Organics detected (continued)</u>						
38	28.32	Unknown (91,119,105,133)	2849	17		
39	28.73	Unknown alkyl benzene (105,106,91)	9943	59		
41	32.08	Unknown phthalate (149,107,91,207)	5796	34		
TOTAL CONCENTRATION ^h				9.7 × 10 ³		

^aIncludes all substances that were found at or above a 0.5% threshold on the BATCH peak identification program in the Hewlett-Packard system, corresponding to a detection limit of 10 $\mu\text{g/g}$.

^bAnthracene- d_{10} internal standard added immediately prior to analysis: 50 mg/L injected equivalent to 935 $\mu\text{g/g}$ in original sample.

^cDeuterated recovery standard (spike), added to original sample before extraction.

^dQuantitated relative to area of major ion for authentic standard.

^eEstimated concentration based on area of total ion relative to that of anthracene- d_{10} internal standard.

^fTentative identification, not confirmed with authentic standards.

^gMajor masses of unknown compounds are listed in parentheses according to decreasing intensities. Possible molecular ions are underlined.

^hSummation of concentrations of detected compounds, excluding standards.

TABLE 27. IDENTIFICATION OF MAJOR EXTRACTABLE COMPOUNDS IN THE METHYLENE CHLORIDE EXTRACT OF THE SEDIMENT DUPLICATE SAMPLE, DILUTED 1:10, NOT CORRECTED FOR BACKGROUND

Peak No.	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, µg/g	Amount spiked, µg/g	Percent recovery
<u>Internal standard^b</u>						
28	21.13	Anthracene-d ₁₀	171907	725	-	-
<u>Recovery standard^{c,d}</u>						
4	6.58	Phenol-d ₅	8575	73	145	50.0
8	7.57	1,2-Dichlorobenzene-d ₄	88738	57	145	39.0
35	25.25	Pyrene-d ₁₀	29681	93	145	64.0
48a	29.10	Chrysene-d ₁₂	16423	91	145	63.0
<u>Organics detected^{e,f,g}</u>						
1	3.62	(See previous table for compound identifications)	10472	44		
2	5.55		8609	36		
3	5.95		10659	45		
5	6.75		28990	1.2×10^2		
6	7.18		42636	1.8×10^2		
7	7.53		266215	1.1×10^3		
9	8.38		7364	31		
10a	9.00		19898	84		
10b	9.1		19898	84		
11	11.63		13516	57		
12	11.08		8593	36		

(continued)

TABLE 27 (continued)

Peak No.	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, $\mu\text{g/g}$	Amount spiked, $\mu\text{g/g}$	Percent recovery
Organics detected (continued)						
13	12.25	(See previous table for compound identifications)	28546	1.2×10^2		
14	12.40		9775	41		
15	15.02		32086	1.4×10^2		
16	16.45		6439	27		
17	18.07		8968	38		
18	18.30		9267	39		
19	18.42		4118	17		
20	19.57		7727	33		
21	19.88		2701	16		
22	20.02		8040	34		
23	20.12		18718	79		
24	20.27		11119	47		
25	20.50		5277	22		
26	20.67		6192	26		
27	21.02		1593	7		
29	22.97		6090	26		
30	23.57		6759	29		
31	23.80		2297	10		
32	24.05		14737	62		
33	24.33		5187	22		
34	24.88		15369	65		
36	25.38		9231	39		
37	25.55		5036	21		
38	25.73		3311	14		
39	26.50		9585	40		
40	26.88		1200	5		
41	27.65		10268	43		

(continued)

TABLE 27 (continued)

Peak No.	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, $\mu\text{g/g}$	Amount spiked, $\mu\text{g/g}$	Percent recovery
<u>Organics detected (continued)</u>						
42	27.90	(See previous table for compound identifications)	2018	9		
43 & 44	28.05-28.08	↓	1406794	5.9×10^3		
45	28.32		4897	25		
46	28.53		4338	18		
47	28.73		14553	61		
48b	29.2		2053	9		
48c	29.2		2053	9		
49	32.07		9124	38		
TOTAL CONCENTRATION ^h				8.9×10^3		

^a Includes all substances that were found at or above a 0.5% threshold on the BATCH peak identification program in the Hewlett-Packard system, corresponding to a detection limit of 10 $\mu\text{g/g}$.

^b Anthracene- d_{10} internal standard added immediately prior to analysis; 50 ng/L injected equivalent to 725 $\mu\text{g/g}$ in original sample.

^c Deuterated recovery standard (spike), added to original sample before extraction.

^d Quantitated relative to area of major ion for authentic standard.

^e Estimated concentration based on area of total ion relative to that of anthracene- d_{10} internal standard.

^f Tentative identification, not confirmed with authentic standards.

^g Major masses of unknown compounds are listed in parentheses according to decreasing intensities. Possible molecular ions are underlined.

^h Summation of concentrations of detected compounds, excluding standards.

TABLE 28. ANALYSIS OF CHLORINATED DIBENZO-P-DIOXINS IN WATER SAMPLES

Sample	ng Dibenzo(furan)/liter (ppt)							
	Isomers							
	Cl ₁	Cl ₂	Cl ₃	Cl ₄	Cl ₅	Cl ₆	Cl ₇	Cl ₈
Effluent	ND ^a	ND	ND	ND	ND	ND	ND	ND
Influent	<14	ND (<17) ^b	ND (<7)	ND (<6)	6	61	17	100
Influent (rep)	<14	ND (<16)	ND (<8)	ND (<5)	12	140	20	110
Blank	ND	ND	ND	ND	ND	ND	ND	ND
Method detection limit	6	8	3	2	5	4	5	6
Spike level	13	12	14	10	12	12	13	10
Effluent (spiked)	9	11	14	12	12	9	12	14
Recovery percent	69	92	100	120	100	75	92	140
Spike level	130	120	140	99	120	120	130	100
Influent (spiked)	97	110	120	99	130	220	120	170
Recovery percent	75	92	86	100	101	100	78	65

^aND - none detected.^bND (<xx) - none detected (detection limit).

TABLE 29. ANALYSIS OF CHLORINATED DIPENZO-P-DIOXINS IN WATER SAMPLES

Sample	ng Dibenzofuran/liter (ppt)							
	Isomers							
	Cl ₁	Cl ₂	Cl ₃	Cl ₄	Cl ₅	Cl ₆	Cl ₇	Cl ₈
Effluent	ND ^a	ND	ND	ND	ND	ND	ND	ND
Influent	ND (<11) ^b	ND (<90)	ND	ND (<4)	ND	ND	4	11
Influent (rep)	ND (<14)	ND (<110)	ND	ND (<4)	ND	ND	6	11
Blank	ND	ND	ND	ND	ND	ND	ND	ND
Method detection limit	2	3	4	3	7	4	3	6
Spike level	4	6	7	4	10	18	4	11
Effluent (spiked)	4	4	8	4	11	14	5	11
Recovery percent	100	67	114	100	110	78	125	100
Spike level	36	64	69	45	104	176	38	110
Influent (spiked)	39	70	70	31	81	131	33	110
Recovery percent	108	109	101	69	78	74	74	90

^aND - none detected.^bND (<xx) - none detected (detection limit).

TABLE 30. ANALYSIS OF CHLORINATED DIBENZO-P-DIOXINS
IN SLUDGE SAMPLES

Sample	ng Dibenzo-p-dioxin/gm sludge (ppb)							
	Isomers							
	Cl ₁	Cl ₂	Cl ₃	Cl ₄	Cl ₅	Cl ₆	Cl ₇	Cl ₈
Sludge	ND ^a	ND	ND	ND	ND	11	65	620
Sludge (rep)	ND	ND	ND	ND	ND	17	81	830
Blank	ND	ND	ND	ND	ND	ND	ND	ND
Method detection limit	20	20	15	15	10	10	10	10
Spike level	13	12	14	10	12	12	13	10
Sludge (spiked)	ND	ND	ND	ND	18	34	93	700
Recovery percent	-	-	-	-	150	167	154	-
Spike level	130	120	140	100	120	120	130	100
Sludge (spiked)	69	120	87	120	150	153	220	860
Recovery percent	53	100	62	120	125	116	113	55

^aND - none detected.

TABLE 31. ANALYSIS OF CHLORINATED DIBENZOFURANS IN SLUDGE SAMPLES

Sample	ng Dibenzofuran/gm sludge (ppb)							
	Isomers							
	Cl ₁	Cl ₂	Cl ₃	Cl ₄	Cl ₅	Cl ₆	Cl ₇	Cl ₈
Sludge	ND ^a	ND	ND	ND	ND	ND	33	39
Sludge (rep)	ND	ND	ND	ND	ND	ND	25	73
Blank	ND	ND	ND	ND	ND	ND	ND	ND
Method detection limit	10	20	25	10	30	45	20	20
Spike level	110	200	220	140	310	520	110	370
Sludge (spiked)	27	110	130	110	290	580	130	410
Recovery percent	25	55	59	85	94	112	92	89
Spike level	1.100	2.000	2.200	1.400	3.100	5.200	1.110	3.700
Sludge (spiked)	380	2.000	2.700	1.300	3.100	5.300	940	3.400
Recovery percent	35	100	123	93	100	102	83	90

^aND - none detected.

TABLE 32. RCRA METALS IN SLUDGE LEACHATE^a

Analyte	µg/L (ppb)
Arsenic	<3
Barium	1.75×10^3
Cadmium	1.88×10^3
Chromium	348
Lead	514
Mercury	2
Selenium	8.2
Silver	96

Quality Control Data

Reference standard	Observed value, µg/L	True value, µg/L	Percent recovery
Arsene TM-7	35.7	35	102
Barium ERA 2767	429	460	93
Cadmium TM-NR	313.5	300	105
Chromium ERA-1120	165.3	160	103
Lead ERA-1120	278	265	105
Mercury TM-Cl	16.5	20	93
Selenium SRMA	10.2	10	102
Silver SPEX-A	111	100	111

Spike^b

Analyte	Concentration added, µg/L	Percent recovery
Arsenic	50	102

^aAll analyses by AA.^b50 µg/L of standard solution is added to leachate and amount recovered is determined.

TABLE 33. HERBICIDE AND PESTICIDE CONCENTRATION IN LEACHATE

Analyte	Concentration, µg/L (ppb)	Lower detection limit, µg/L
Lindane	ND ^a	1,000
Endrin	ND	400
Methoxychlor	ND	50
Toxaphene	ND	1 x 10 ⁴
2,4-D ^b	ND	10
2,4,5-TP Silvex ^b	ND	10

Quality Control Data

A known amount of 2,4-D and 2,4,5-TP Silvex were spiked into the sludge sample. The percent recovery was:

Analyte	Percent recovery
2,4-D	120
2,4,5-TP Silvex	105

^aND - not detected above lower detection limit.

^bDuplicate analyses performed.

TABLE 34 CYANIDE AND PHENOL ANALYSIS OF LEACHATE

Analyte	Concentration, µg/L
Cyanide	10
Phenol	1,297

Quality Control Data			
Analyte	Observed value, µg/L	True value, µg/L	Percent recovery
Cyanide	60	80	75
Phenol	118	125	94

TABLE 35. TOTAL METALS ANALYSIS OF SLUDGE SAMPLE

Analyte	$\mu\text{g/g}$ (ppm)
Arsenic ^a	16.9
Mercury ^a	<0.2
Selenium ^a	5.7
Aluminum	2.8×10^3
Antimony	<73.0
Barium	96.8
Beryllium	<23.0
Boron	78.8
Cadmium	45.0
Calcium	4.5×10^4
Chromium	156.0
Cobalt	15.0
Copper	995.0
Iron	1.1×10^4
Lead	361.0
Magnesium	2.5×10^3
Manganese	230.0
Molybdenum	84.0
Nickel	820.0
Potassium	$<2.4 \times 10^3$
Silver	23.7
Sodium	1.2×10^4
Tin	<519.0
Titanium	105.0
Vanadium	<14.8
Zinc	3.0×10^3

^aAnalyses by AA, all
others by ICP.

TABLE 36. ANALYTICAL RESULTS FOR PRIORITY AND NON-PRIORITY METAL POLLUTANTS IN WATER SAMPLES

	Sample Id. $\mu\text{g/L}$ (ppb)	
	Influent	Effluent ^a
<u>Priority metals</u>		
Arsenic ^b	31.7	4.3
Mercury ^b	<0.8	<0.8
Selenium ^b	14.3	9.95
Thallium ^b	<63	<63
Antimony	<830	<830
Beryllium	7	12
Cadmium	71	<18
Chromium	227	91
Copper	1.7×10^3	<35
Lead	777	<460
Nickel	851	303
Silver	<250	<250
Zinc	3.9×10^3	133
<u>Non-priority metals</u>		
Aluminum	890	<450
Barium	187	77
Boron	388	674
Calcium	2.6×10^5	<210
Cobalt	<70	<70
Iron	6.8×10^3	253
Magnesium	3.6×10^4	<70
Manganese	457	186
Molybdenum	562	849
Potassium	1.6×10^5	1.1×10^5
Sodium	6.4×10^5	$<1.5 \times 10^4$
Tin	$<2.2 \times 10^3$	$<2.2 \times 10^3$
Titanium	54	<43
Vanadium	<150	<150

^aAverage of duplicate analyses.

^bAnalyses by AA; all others by ICP.

TABLE 37. QUALITY CONTROL DATA FOR METALS
ANALYSES OF WATER SAMPLES

Analyte	Reference standard	Observed value, µg/L	True value, µg/L	Percent recovery
Arsenic ^a	TM-7	31.7	35.0	106
Mercury ^a	TM-C1	18.5	20.0	93
Selenium ^a	SRMA	12.2	11.0	111
Thallium ^a	SPEX-A	91.3	100	91
Aluminum	ICP	410	515	80
Antimony	ICP	1,010	1,000	101
Barium	ICP	831	920	90
Beryllium	ICP	173	165	105
Cadmium	ICP	51	50	103
Calcium	ICP	1,180	1,000	118
Chromium	ICP	153	160	96
Cobalt	ICP	123	115	107
Copper	ICP	602	615	98
Iron	ICP	1,100	1,000	110
Lead	ICP	254	265	96
Manganese	ICP	1,090	1,000	109
Nickel	ICP	1,160	1,000	116
Titanium	ICP	1,100	1,000	110
Vanadium	ICP	114	110	104
Zinc	ICP	157	165	95

^aAnalyses by AA; all others by ICP.

TABLE 38. LOQ^a VALUES FOR METALS BY ICP ANALYSES

Analyte	µg/L
Aluminum	450
Antimony	825
Barium	23
Beryllium	6
Boron	230
Cadmium	18
Calcium	210
Chromium	33
Cobalt	70
Copper	35
Iron	56
Lead	460
Magnesium	70
Manganese	3
Molybdenum	65
Nickel	60
Potassium	4.3×10^4
Silver	250
Sodium	1.4×10^4
Tin	2.2×10^3
Titanium	43
Vanadium	150
Zinc	26

^aLeast observable
quality.

TABLE 39. ANALYTICAL RESULTS FOR TOTAL CYANIDE
AND TOTAL PHENOL IN WATER SAMPLES

Analyte	Sample ID, $\mu\text{g/L}$ (ppb)	
	Influent	Effluent
Cyanide	NR ^a	10
Phenol	1384	1898

Quality Control Data			
Reference standard	Observed value,	True value,	Percent recovery
	$\mu\text{g/L}$	$\mu\text{g/L}$	
Cyanide	110	160	69
Phenol	222	250	89

^aNR - analysis not requested.

TABLE 40. ANALYTICAL RESULTS FOR NON-PRIORITY
POLLUTANTS IN WATER SAMPLES

Analyte	Sample ID	
	Influent	Effluent
Biological Oxygen Demand (BOD)	NR ^a	117 mg/L
Total Suspended Solids (TSS)	NR	19 mg/L
pH	NR	8.54
Total Organic Carbon (TOC)	NR	184 mg/L
Fluoride	NR	1.56 mg/L
Oil and grease	NR	16 mg/L

Quality Control Data				
Analyte	Reference standard	Observed	True	Percent recovery
		value,	value,	
		mg/L	mg/L	
TSS	Hardness 3253	34	34	100
Oil and grease	PCS	24.9	26	96
TOC	PCS	48.6	48	101
Fluoride	ERA 3253	3.3	3.4	97

^aNR - analysis not requested.

APPENDIX A

GC/MS ANALYSES OF EXTRACTABLE ORGANICS

A-1

MONS 220704

GC/MS ANALYSES OF EXTRACTABLE ORGANICS

Introduction

All GC/MS analyses were performed using the protocol developed for the EPA-sponsored Chesapeake Bay analysis program. These analyses were performed by automated capillary column chromatography using a Hewlett-Packard 5985-A GC/MS with a 5934 data system, which used Revision C Software and included a 7920 multi-drive disc unit.

Method

The relevant GC/MS parameters used for the present analyses are summarized below:

- 30 m fused silica SE-54 WCOT column
- Column head pressure, 7-9 psi
- MS operating pressure, 1×10^{-5} to 4×10^{-6} torr
- Septem purge on at 0.5 min
- Initial temperature, 50°C
- Initial temperature held for 4 minutes
- Heating rate, 8°C/min
- Final temperature, 300°C
- Total analysis time, 40 min
- Injector, splitless mode, temperature, 250°C
- Transfer line temperature, 250°C (fused silica column through this zone, directly to the MS source)
- Electron energy, 70eV
- Source temperature, 200°C
- A/D rate, 1 measurement/0.125 amu;
scan rate, approximately 0.6 s/scan
- Mass spectrometer scan delay, 3.0 min

The routine procedure used for GC/MS analysis was to transfer 150 μ L of the sample extract into 1.5 mL sample vials along with 150 μ L of a 100 μ g/mL anthracene- d_{10} in methylene chloride internal standard. The vial was capped and placed in the autosampler, which injected approximately 1 μ L into the GC, operating in the splitless mode.

Interpretation of Mass Spectra

The following protocol was followed in interpreting the mass spectral data obtained from analysis of water and sediment extracts:

- 1) Spectra of compounds detected as peaks in the Hewlett-Packard BATCH program with a 0.5% or 5% threshold (depending upon the complexity of the sample) were automatically compared with standard spectra in the computerized EPA/NIH mass spectral data base using the Probability Based Search (PBS) software supplied by Hewlett-Packard. A goodness of fit parameter was generated (1.0 being a perfect match) for the ten matches closest to 1.0.
- 2) The results from these searches were reviewed by an experienced mass spectroscopist using the Hewlett-Packard supplied spectral comparison software, SPDIF, to compare the sample spectra with the standard spectra identified by the SEARCH program.
- 3) In cases where the spectral agreement was judged to be adequate, compound identities were assigned. When compound isomers produced similar spectra, no specific isomer could be identified based solely upon mass spectral data.
- 4) In cases where the agreement was not adequate, identifications were made on the basis of manual interpretation (when obvious), or compounds were listed as unknown with their major masses given in parentheses. If a high even mass was present after which followed isotope peaks in the proper ratio, the underlined to indicate a possible molecular ion.

The approach outlined above was adopted to minimize the cost and time required for the analyses of large amounts of capillary chromatographic mass spectra by maximizing the use of computerized data interpretation techniques.

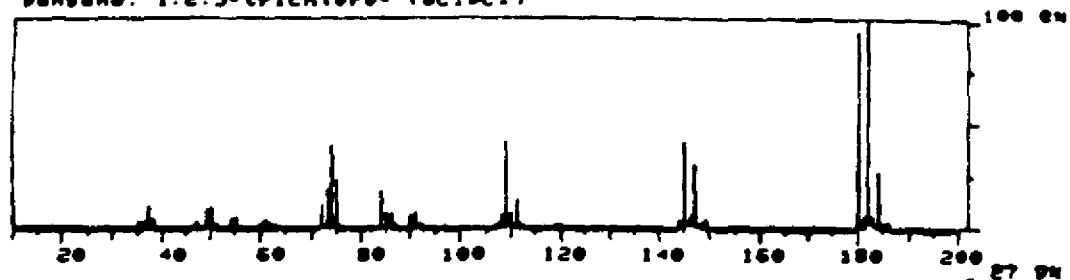
Figures A-1 and A-2 show spectral comparison plots which were generated from those "best fit" candidate compounds identified in the a computerized search of some typical GC/MS data. These figures show comparisons of two different trichlorobenzene isomers with the unknown spectrum. From the difference of each of the two spectra plotted in these figures, it can be seen that less than 30% deviations with the two standard spectra were measured. Since the two standard spectra of trichlorobenzene isomers are very similar, the identification of the peak at 11.0 minutes would be listed in the effluent table for as "Benzene, trichloro-(isomer)."

These comparative mass spectral plots were reviewed by an expert mass spectroscopist who based on experience in mass spectrometry and mass spectral interpretation, accepted or rejected the computer-generated identification. In the example, trichlorobenzene (isomer) was determined to be the proper identification for the compound in question. It should be emphasized that this identification is considered to be tentative until confirmed by the measurement of its relative retention time of an authentic standard of isomers of the identified compound using similar analytical conditions as those used for the original sample extract analysis.

Some considerations that go into making decisions on the identity of components in effluent and sediment extracts include:

- 1) The unknown spectrum must contain all peaks with intensities greater than 10% of the base peak in the standard spectrum.

FRN 3004 SPECTRUM 46DB PU- 180 C6H3Cl3
 Benzene, 1,2,3-trichloro- (BC19C1)



FRN 23121 SPECTRUM 12 RET TIME: 11.0
 1 UL 125/125 D10 + 0184-BMP3 (B1425) 8/13/81 BPH DANE 13121

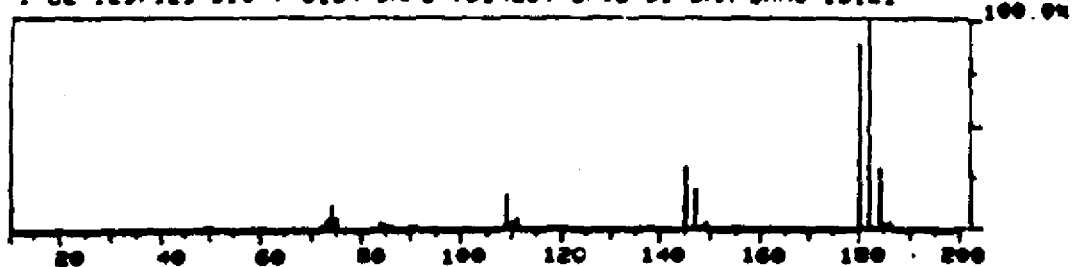


Figure A-1. Comparison of the mass spectrum of the highest matching compound with that of an unknown compound eluting at 11.0 minutes.

MONS 220708

FRN 3004 SPECTRUM 4699 MU-180 C6H3Cl3
Benzene, 1,3,5-trichloro- (BCIDCI)



FRN 23121 SPECTRUM 12 RET. TIME: 11.0
1 UL 125/125 D10 + 8184-BMP3 (81425) 8/13/81 BPH DMS 13121

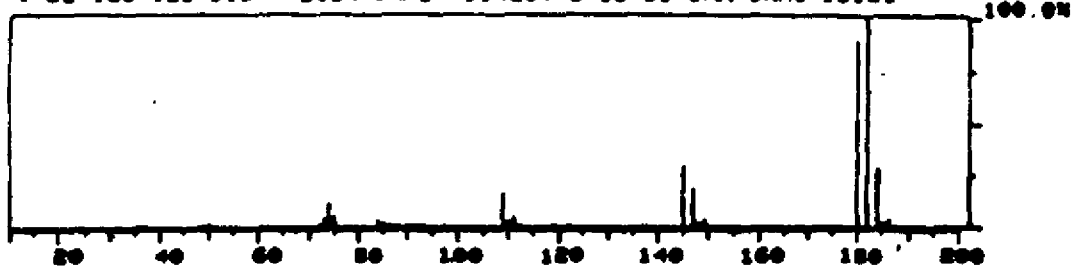


Figure A-2. Comparison of the mass spectrum of the third highest matching compound with that of an unknown compound eluting at 11.0 minutes.

- 2) All mass intensities of multiplets present in the standard spectrum must also be present in the unknown spectrum with similar relative intensities.
- 3) Since the DFTPP spectrum in the low mass region is lower than that given in the EPA criterion for this compound quantitative agreement of ion intensities was not required for masses below m/e 60.
- 4) If peaks are present additional to those in the standard spectrum of a compound, then it is assumed that the additional peaks are due to the presence of an unknown co-eluting compound. In obvious cases of mixed spectra, the PBS SEARCH program identified the presence of a mixture of spectra with a "+" in the column following the match factor. Where mixed spectra are confirmed using the spectral comparison program, the major identified compound was listed along with an estimate of the percent of the unknown compound present in the mixed spectrum.

Attempts were made to give additional information such as the major masses found in the unknown, the molecular weight if the spectrum appears to contain a molecular ion, and the presence of nonhydrocarbon atoms. However, the identification of the presence of chlorine, sulfur, or silicon in unknown compounds is based solely upon the occurrence of multiplet ion patterns which are consistent with the naturally-occurring abundances of isotopes for these species, and is used only to give more information about the unknown compound's spectrum.

Quantitation of GC/MS Data

The calculations used for determining the concentration of organics in sample extracts were performed in two ways. All levels of deuterated spiking compounds were determined using the response

of the molecular ion of each of the compounds, relative to the anthracene-d₁₀ molecular ion. Therefore, all recovery data were calculated using authentic standards.

The amounts of all other compounds were determined assuming the total ion response of each compound to be equal to the response of the anthracene-d₁₀ internal standard. This latter calculation was made using the following relationship:

$$\text{Concentration of X} = \text{concentration Anthracene-d}_{10} \frac{\text{Total ion area X in unknown}}{\text{Total ion area anthracene-d}_{10}} \times (\text{sample concentration factor})$$

The sample concentration factor takes into account the extraction and concentration of the 1-liter sample to a 1-mL extract or the extraction of approximately 30 g sediment and final volume of 1 mL in the sediment extract.

QA/QC for Extractable Organics

MRC uses a routine procedure for assessing the condition of the mass spectrometer and the capillary column in the gas chromatograph. The mass spectrometer is tuned approximately daily using perfluorotributylamine (PFTBA) which is the basis for the Hewlett-Packard AUTOTUNE program. The output from an average tune is shown in Figure A-3.

Samples were run in the automated BATCH ACQUIRE mode overnight, and with each batch of samples a system performance standard was analyzed. This included the compound decafluorotriphenylphosphine (DFTPP) which was analyzed by the Hewlett-Packard program: DFTPP EPA Criterion Verifier. The output from a typical analysis by this program is shown in Figure A-4.

A-9

* CURRENT PARAMETERS *
 LVS985-140414J
 DATE (FRM 1990 11 1/26/81 1115
 ACCELER(V)=10.84 EM VOLTAGE(V)=2200
 WADJOUT(V)=26.5 AMU GAIN=115
 ION FOCUS(V)=35 AMU OFFSET=113
 ENT LENS(FV/AMU)=133 MASS AXIS GAIN=1.00157
 X-RAY(V)=154 MASS AXIS OFFSET=-.190491
 EMISSION(IU)=300 IONS: POSITIVE
 ELECT. ENERGY(EV)=70 ACTUAL SOURCE TEMP =200
 LOG AMP OFFSET=1

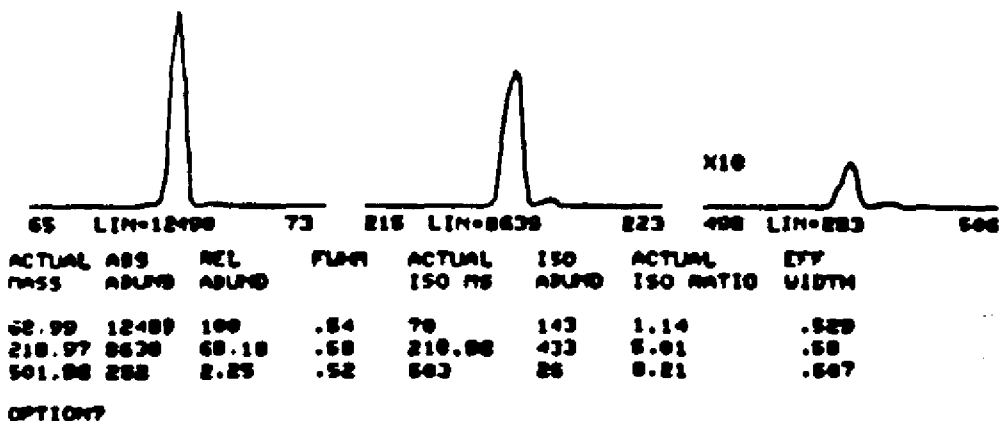


Figure A-3. A typical result from tuning the Hewlett-Packard 5985 mass spectrometer with PFTBA.

DFTPP EPA CRITERION VERIFIER

DRN: 22512
SPECTRUM: 2011

Mass	Rel. Abund.	Criterion
xx 51	9.95206	30-60% MASS 198
68	0	< 2% MASS 69
69	29.7239	
70	0	< 2% MASS 69
xx 127	33.4849	40-60% MASS 198
197	0	< 1% MASS 198
198	100	BASE PEAK
199	6.60613	5-0% MASS 198
275	22.8732	10-30% MASS 198
365	1.70276	> 1% MASS 198
441	14.7049	< MASS 443
442	73.8387	> 40% MASS 198
443	16.0367	17-23% MASS 442

NOTE: 'xx' indicates out of range!

PRESS RETURN TO RERUN...

Figure A-4. Computer analysis of mass spectrum of DFTPP.

MONS 220713

Typically, the m/e 51 and 127 on our instrument fell somewhat below the normal range specified by the program. This fact was taken into account in interpreting mass spectra. If other peaks fell outside the ranges, the tuning of the mass spectrometer was re-examined.

The substances present in the system performance standard were: (1) 2,6-dimethylphenol, (2) 2,6-dimethylaniline, (3) decanol, (4) pentachlorophenol, (5) anthracene-d₁₀ added prior to analysis, (6) octadecene, (7) octadecane, (8) DFTPP, (9) eicosane, (10) heneicosane, (11) pyrene, (12) methyl stearate, and (13) chrysene. Figure A-5 shows a typical chromatogram obtained from the standard. By examining the resolution of the peaks ~20.7 min and ~24.7 min, and by looking at the tailing of certain peaks such as decanol and pentachlorophenol, we visually determined the condition of the column. When the resolution and tailing were substantially worse than in a new column, the injection port insert was replaced and the front end of the column was broken off or the column replaced.

In order to assess the accuracy of GC/MS quantitation, two standards were compared with each other. A Supelco phenols standard was analyzed on 8 February 1981, and a freshly prepared MRC standard containing many of the same phenols was analyzed on 4 April 1981. In both standards the concentrations of the components were close to 100 mg/L. Taking the concentrations in the MRC standard to be correct, the concentrations of the components in the Supelco standard were calculated and compared with their stated values. The substances analyzed included 2-nitrophenol, 2,4-dinitrophenol, 2,4-dichlorophenol, 4-chloro-m-cresol, 2,4,6-trichlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-o-cresol, and pentachlorophenol.

A-12

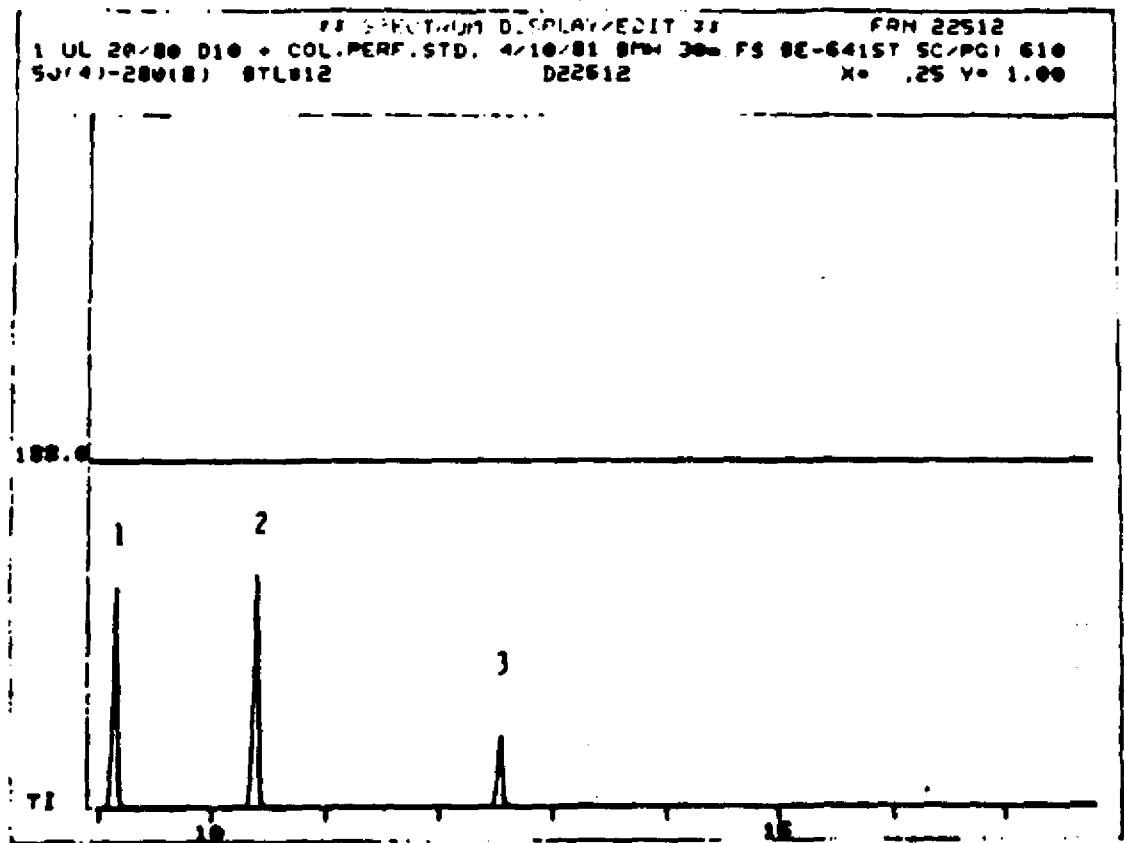


Figure A-5. Chromatogram of the column performance standard with the GC column in average condition. The numbered peaks are identified in the text.

MONS 220715

A-13

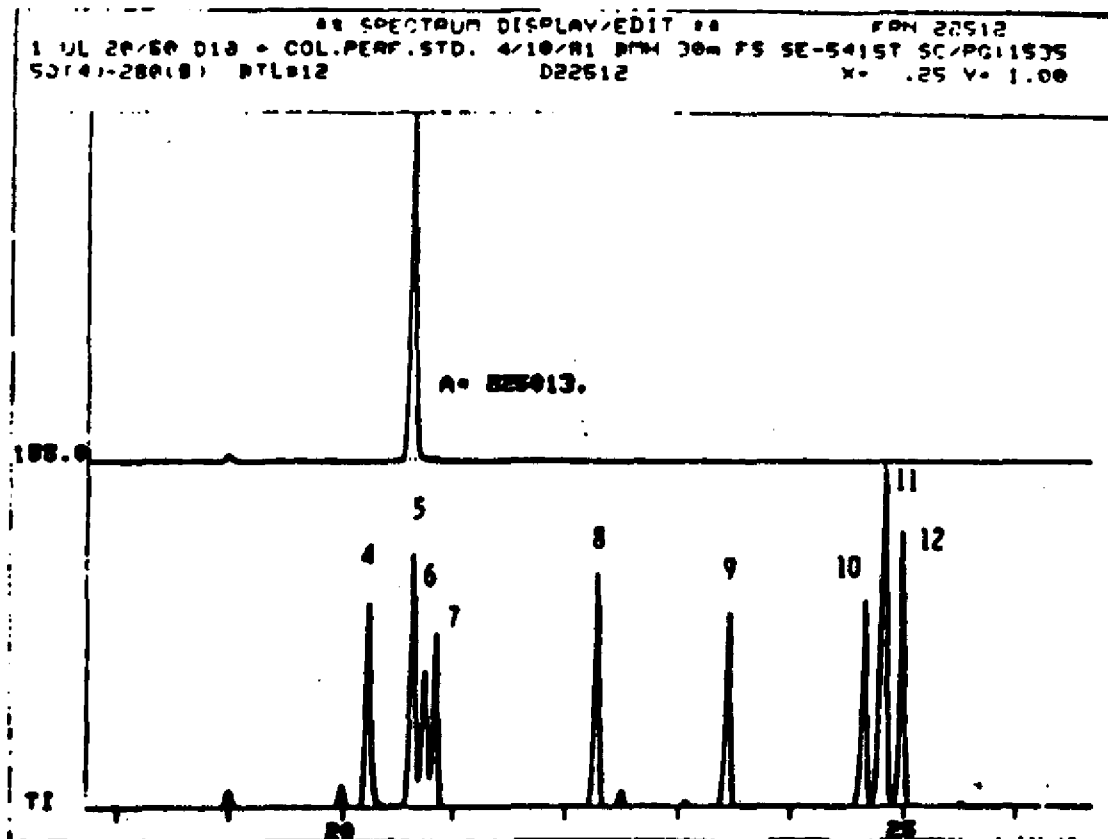


Figure A-5 (continued)

NONS 220716

A-14

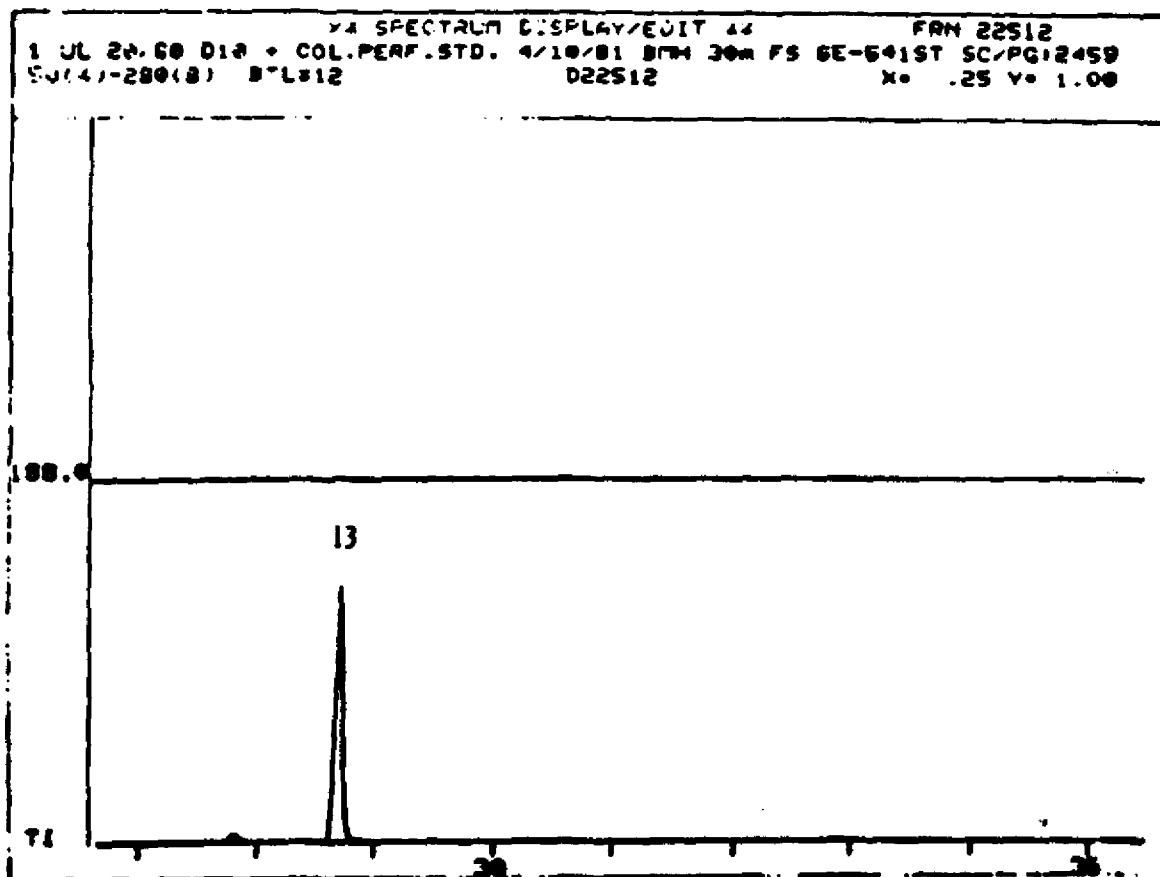


Figure A-5 (continued)

Based on MRC standards the average percent deviation of the values found from the stated values was 28%, excluding 4-nitrophenol, where 7 mg/L was found vs. the stated 100 mg/L.

The quantitation of 4-nitrophenol has been a source of difficulty previously, as it appears to diminish in concentration with storage time of the standard. The indication from the comparison of standards is that except for a particularly troublesome compound such as the 4-nitrophenol, the average uncertainty in the quantitation of clean samples in which the unknown component is of comparable concentration to the standard, is approximately $\pm 30\%$.