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Monsanto **Environmental Services Report**

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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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TABLES (continued)

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 surogate 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 labcratory 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-d_{10} 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-d_5, Cl_2-benzene-d_4, pyrene-d_{10} and chrysene-d_{12} 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 alumuia 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 proceded 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 µg/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 µg/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 µg/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, 1982.

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 instae Daphnia magna (<24 has 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 = 810).

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, 1982 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 0	24	48	Effluent concentration, %
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, µmhos	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
	372			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 approvel EPA anelytical 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 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 quanties (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.

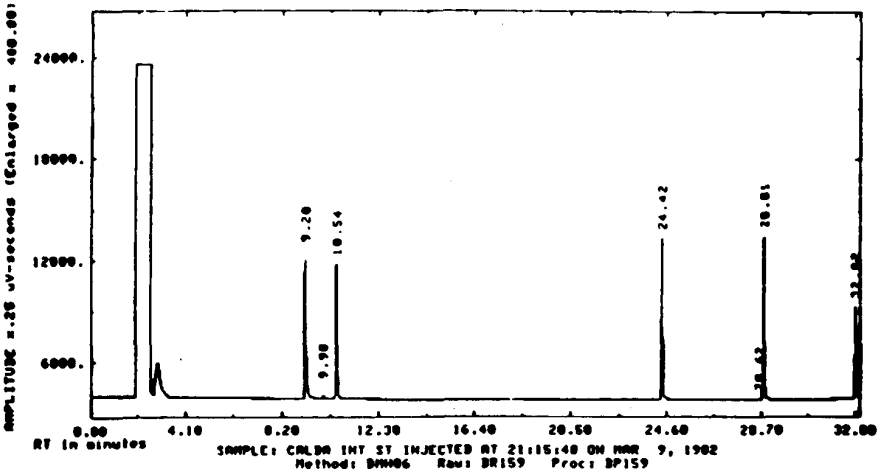

Figure 1. Capillary GC/FID chromatogram of surrogate compound spikeing standard added to each sample before extraction.

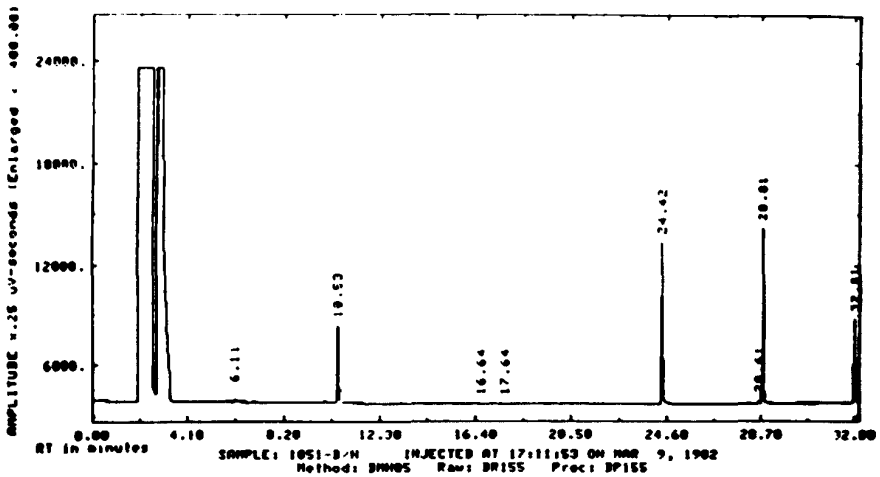

Figure 2. Capillary GC/FID chromatogram of the base/neutral extact of the method blank (Burdick and Jackson Water, distilled in glass).

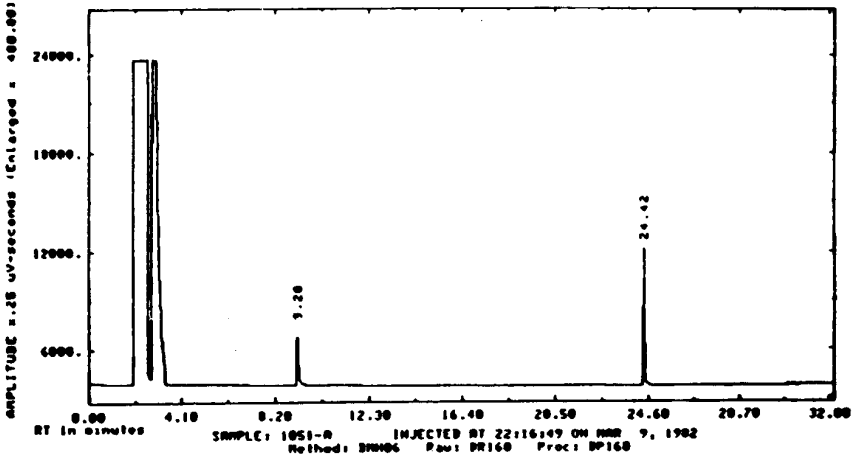

Figure 3. Capillary GC/FID chromatogram of the acid extract of the method blank (Burdick and Jackson Water, distilled in glass).

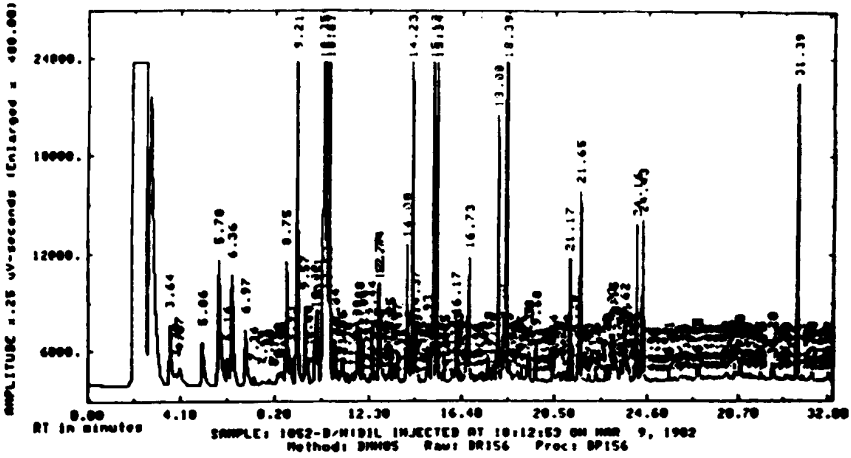

Figure 4. Capillary GC/FID chromatogram of the base/neutral extract of influent sample from Sauget, IL Treatment Plant, diluted ten times before analysis.

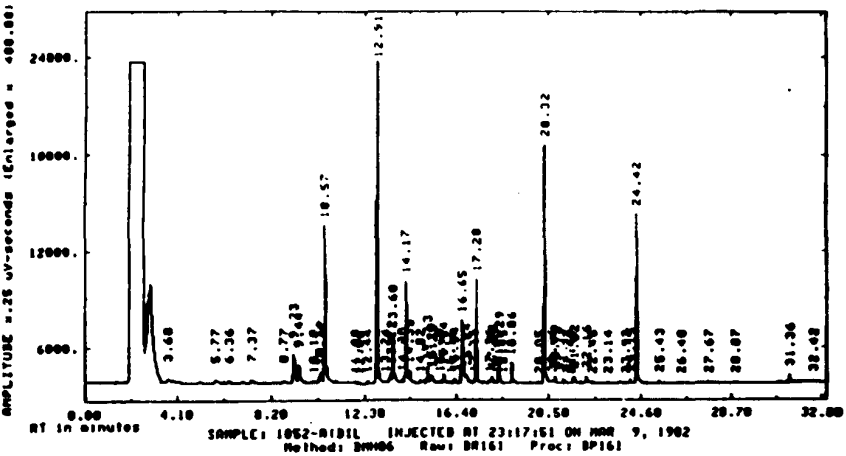

Figure 5. Capillary GC/FID chromatogram of the acid extract of the influent sample from Sauget, IL Treatment Plant, diluted ten times before analysis.

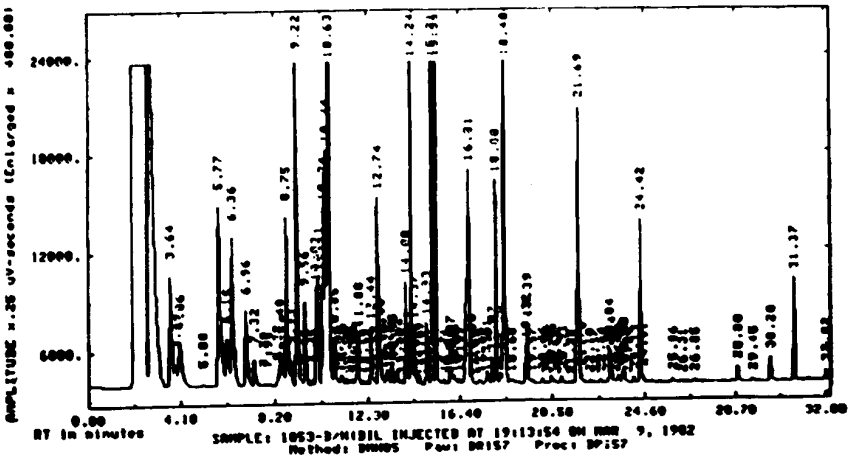

Figure 6. Capillary GC/FID chromatogram of the base/neutral extract of the effluent sample from Sauget, IL Treatment Plant, diluted ten times before analysis.

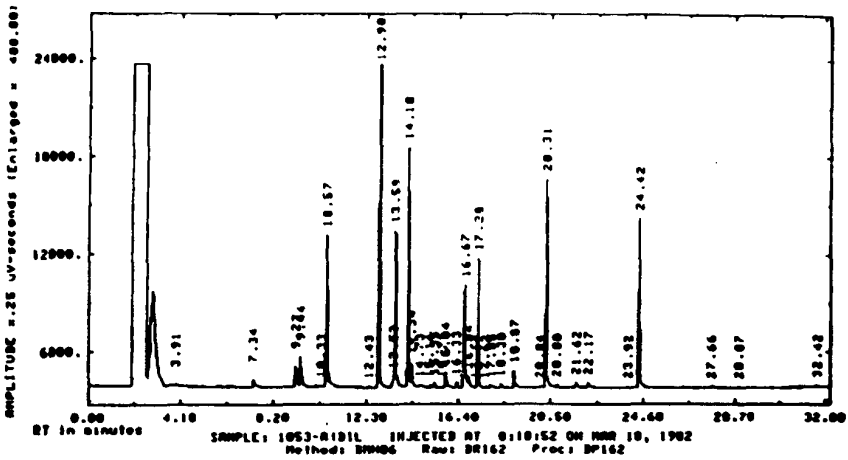

Figure 7. Capillary GC/FID chromatogram of the acid extract of the effluent sample from Sauget, IL Treatment Plant, diluted ten times before analysis.

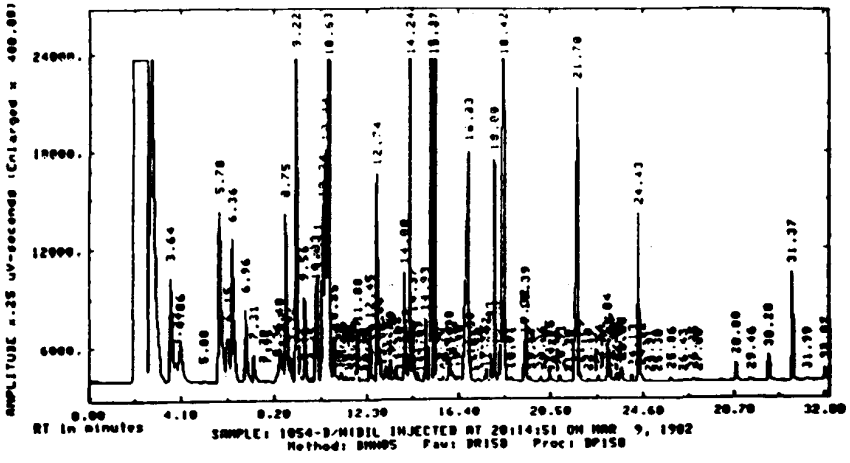

Figure 8. Capillary GC/FID chromatogram of the base/neutral extract of a duplicate effluent sample from Sauget, IL Treatment Plant, diluted ten times before analysis.

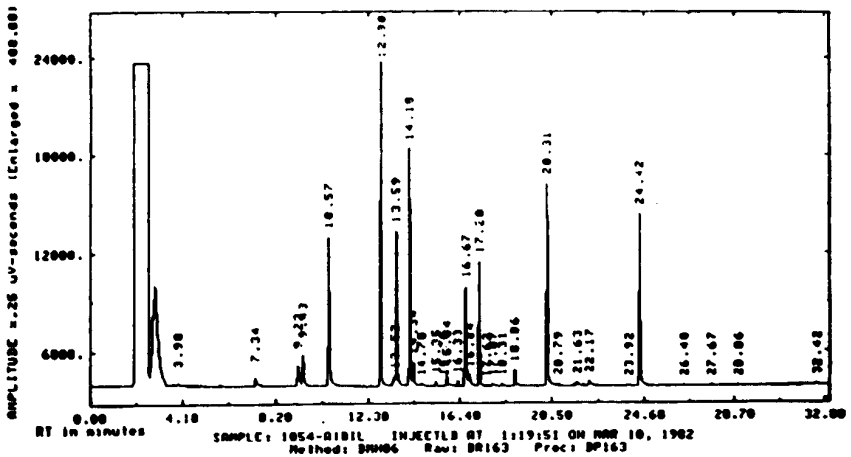

Figure 9. Capillary GC/FID chromatogram of the acid extract of a duplicate effluent sample from Sauget, IL Treatment Plant, diluted ten times before analysis.

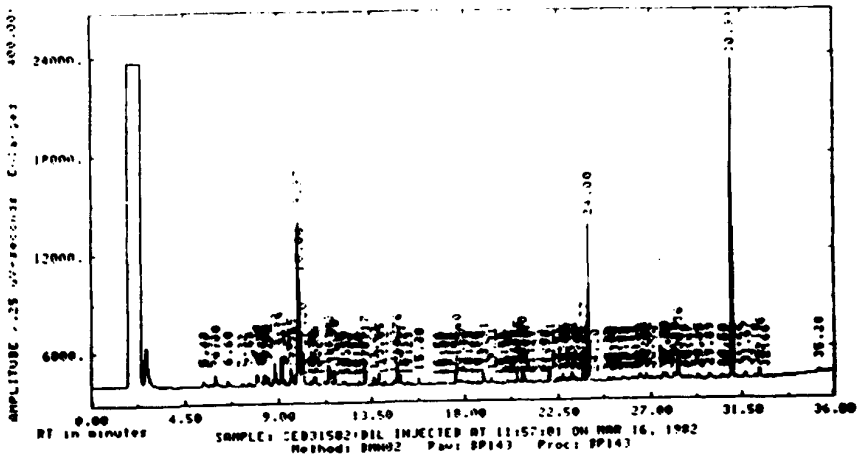

Figure 10. Capillary GC/FID chromatogram of the extract of a 1.07 g sediment sample from Sauget, IL Treatment Plant, diluted ten times before analysis.

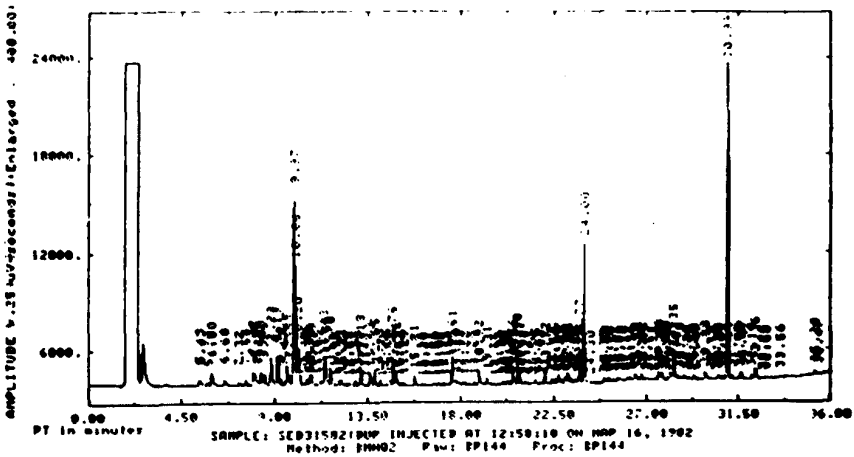

Figure 11. Capillary GC/FID chromatogram of the extract of a 1.38 g duplicate sediment sample from Sauget, IL Treatment Plant, diluted ten times before analysis.

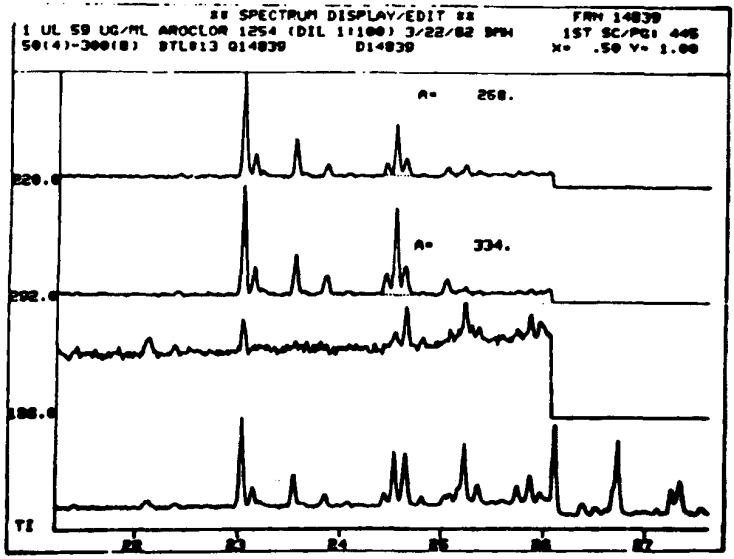

Figure 12. Analysis of 59 µg/mL Aroclor 1254 standard, using $(GC)^2$/MS-SIM analysis techniques.

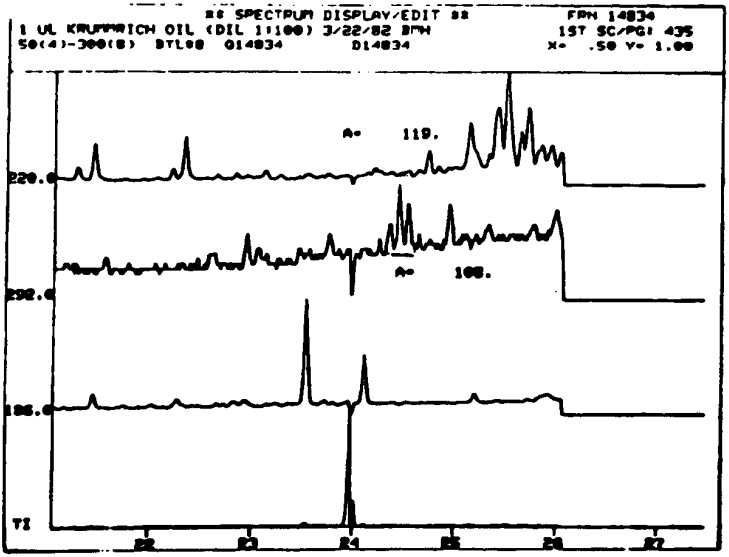

Figure 13. Analysis of an oil sample, using $(GC)^2$/MS-SIM analysis techniques.

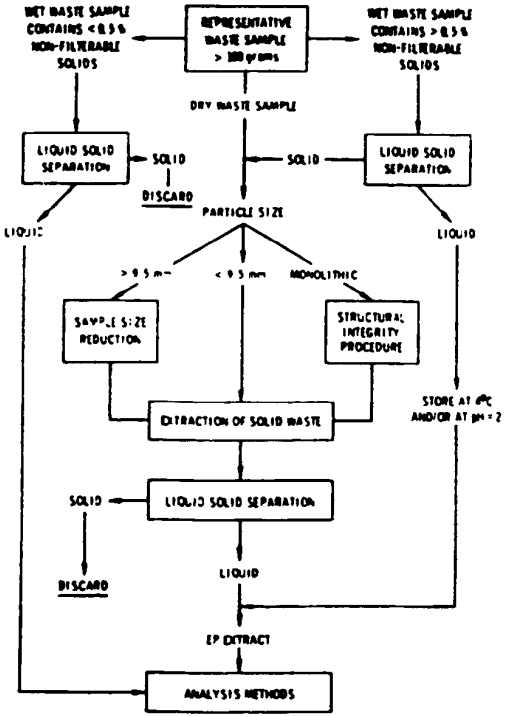

Figure 14. Extraction procedure flowchart.

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

[illegible]	[illegible]		[illegible]	[illegible]
3.64	49354	BV	684.169	
3.89	9660	VV	137.072	
4.07	22465	VB	312.315	
5.0[illegible]	[illegible]	BB	514.380	
5.76	131334	BV	1825.87	
6.1[illegible]	23322	VV	324.231	
6.38	98060	VV	1363.26	
6.57	46741	BV	649.406	
7.34	3873	VB	53.909	
7.69	1037	BB	14.415	
8.07	2842	BV	39.509	
8.15	673	VB	9.352	
8.29	7133	BV	99.171	
8.45	4001	VV	55.619	
8.54	9427	VV	131.053	
8.77	73084	VV	1025.56	
8.91	16506	VV	229.504	
9.21	302706	VV	4047.99	PHENOL-D5
9.57	36402	BV	506.079	
9.6[illegible]	12760	VV	177.399	
9.85	1215	VB	16.657	
10.02	32144	BV	446.887	
10.13	37227	VV	517.698	
10.29	171734	VV	2387.54	
10.44	210532	VV	2726.93	
10.55	20787	VV	319.585	CL2-BENZENE-D4
10.61	219466	VV	3051.13	
10.64	20166	VV	280.352	
10.98	6364	VV	85.469	
11.08	5362	VV	74.550	
11.16	14611	VV	203.132	
11.36	5790	VV	80.495	
11.67	699	BV	9.720	
11.80	15155	VV	210.692	
11.88	19324	VV	268.651	
11.93	1502	VV	20.885	
12.04	13465	VV	187.469	
12.16	2318	VV	32.221	
12.27	1081	VV	15.030	
12.34	1546	VV	21.490	
12.44	21354	VV	297.293	
12.56	4948	VV	68.790	
12.67	1879	VV	26.125	
12.74	25160	VV	349.792	

TABLE 1 (continued)

RT	AREA		ug/L	NAME
12.77	34176	VV	475.163	
12.86	12099	VV	168.210	
12.94	1917	VV	26.657	
13.0[illegible]	340[illegible]	VV	47.399	
13.1[illegible]	1874	VV	26.05[illegible]	
13.17	438[illegible]	VV	60.884	
13.25	1545[illegible]	VV	214.923	
13.38	8070	VV	112.18[illegible]	
13.4[illegible]	2741	VV	38.11[illegible]	
[illegible]	1776[illegible]	V[illegible]	[illegible]	
13.76	115[illegible]	VV	15.993	
13.85	11[illegible]	VV	16.41[illegible]	
14.0[illegible]	[illegible]	VV	74[illegible]	
14.12	6[illegible]47	VV	114.937	
14.[illegible]	12[illegible]	V[illegible]	1[illegible].14	
14.[illegible]	[illegible]	VV	35[illegible].713	
14.44	[illegible]	VV	3[illegible]	
14.4	[illegible]54	V[illegible]	7[illegible]	
14.[illegible]	8[illegible]	V[illegible]	12.044	
14.[illegible]	[illegible]73	VB	5.134	
14.7[illegible]	24[illegible]	BB	3.443	
14.87	2[illegible]14	BV	40.506	
14.[illegible]	15[illegible]	V[illegible]	[illegible]12.42[illegible]	
15.0	[illegible]	VV	3[illegible]	
15.0[illegible]	10[illegible]1	V[illegible]	18.2[illegible]	
15.1[illegible]	[illegible]	V[illegible]	4[illegible].11	
15.[illegible]4	15[illegible]	V[illegible]	[illegible]176.4[illegible]	
15.4[illegible]	4[illegible]	V[illegible]	60.[illegible]	
15.[illegible]	17[illegible]	V[illegible]	2[illegible].0[illegible]	
15.[illegible]	5[illegible]	V[illegible]	73.7[illegible]1	
15.[illegible]	47[illegible]	BB	6.5[illegible]1	
15.9[illegible]	552[illegible]	B[illegible]	7[illegible].[illegible]	
16.1[illegible]	3[illegible]	[illegible]	4[illegible].06[illegible]	
16.1[illegible]	18[illegible]	VV	251.5[illegible]	
16.[illegible]	[illegible]54	VV	87.3[illegible]	
16.3[illegible]	3637	VV	50.557	
16.[illegible]	[illegible]	V[illegible]	36.319	
16.5[illegible]	[illegible]97	VV	31.936	
16.73	10[illegible]47	VV	1399.25	
16.8[illegible]	4940	VV	68.67[illegible]	
17.0[illegible]	[illegible]17	VV	32.205	
17.1[illegible]	1874	VV	26.084	
17.1[illegible]	2295	VV	31.901	
17.24	3016	VV	41.842	
17.34	2760	VV	38.367	
17.47	2115	VV	29.409	
17.58	10313	VV	143.372	
17.71	263	VV	3.653	
17.80	8341	VV	115.960	
18.00	104451	VV	1452.13	
18.20	16022	VV	222.744	
18.39	360249	VV	5006.36	
18.48	7541	VV	104.835	
18.55	2763	VV	38.694	
18.61	2590	VV	36.008	
18.71	964	VV	13.398	

TABLE 1 (continued)

RT	AREA		ug/L	NAME
18.83	1864	BV	26.186	
18.92	700	VV	9.738	
19.05	4840	VV	67.573	
19.19	1121	VV	15.582	
19.31	11130	VV	154.736	
19.35	12158	VV	169.026	
19.54	605	VB	8.467	
19.63	14490	BV	201.449	
19.82	1084	VV	15.072	
19.91	324	VV	4.502	
19.92	276	VP	3.870	
20.06	872	BV	12.117	
20.19	452	VP	6.278	
20.27	[illegible]	PV	[illegible]	
20.3[illegible]	950	VV	13.427	
20.44	7358	VV	103.247	
20.5[illegible]	[illegible]	VV	31.078	
20.65	4875	VV	67.775	
20.7[illegible]	2919	VV	40.577	
20.79	2524	VV	35.09[illegible]	
20.82	1816	VV	25.249	
20.91	4555	VV	63.319	
21.08	4510	BV	62.697	
21.17	[illegible]	VV	492.601	
21.2[illegible]	[illegible]	VV	145.718	
21.36	2481	VV	34.491	
21.4[illegible]	[illegible]	VV	1571.[illegible]	
21.7[illegible]	1245	VV	17.306	
[illegible]	[illegible]	BV	[illegible]	
22.0[illegible]	[illegible]	VV	[illegible]	
22.1[illegible]	[illegible]	VB	[illegible]	
[illegible]	4474	BV	62.205	
22.4[illegible]	[illegible]	VV	21.393	
[illegible]	[illegible]	VB	[illegible]	
22.8[illegible]	[illegible]	BV	63.232	
[illegible]	[illegible]	VV	86.124	
[illegible]	[illegible]	VV	252.117	
23.19	[illegible]	VV	267.474	
23.25	[illegible]	VV	150.862	
23.32	[illegible]	VV	75.365	
23.40	10143	VV	141.090	
23.52	2421	VV	33.661	
23.62	[illegible]	VV	[illegible]	
23.71	9707	VV	134.951	
23.78	7554	VV	105.020	
23.84	4571	VV	63.547	
23.98	309	VB	4.302	
24.07	4543	BV	63.423	
24.16	49078	VV	682.314	
24.24	5491	VV	76.336	
24.32	2193	VV	30.490	
24.43	[illegible]	VV		$ANTHRACENE-D10
24.69	[illegible]	VB	17.257	
24.90	462	PB	6.421	
25.03	1180	BV	16.407	
25.23	543	BB	7.548	
25.42	204	BV	2.833	
25.52	1138	VV	15.815	
25.61	2923	VB	40.638	
25.86	911	BV	12.667	
25.99	292	VB	4.066	

TABLE 1 (continued)

RT	AREA		ug/L	NAME
26.13	1655	BV	23.006	
26.24	964	VB	13.405	
26.35	563	BV	7.829	
26.37	543	VV	7.553	
26.56	1916	BV	26.640	
26.71	388	VB	5.396	
26.75	559	BV	7.776	
26.85	3722	VV	51.749	
26.92	3935	VB	54.705	
27.10	1554	BV	21.629	
27.24	1376	VB	19.132	
27.27	968	B.	13.452	
27.41	992	VB	13.793	
27.47	304	BB	4.224	
27.84	268	VB	3.724	
27.88	255	BB	3.545	
28.0.	289	BB	4.013	
28.12	352	BB	4.894	
28.17	1994	B.	27.715	
28.25	577	VV	6.016	
28.41	1024	VV	14.241	
28.57	4376	VV	60.877	
28.64	3667	VV	50.985	
28.8	9455	VV	131.767	PYRENE-D10
28.94	1477	VV	20.227	
29.03	286	VV	3.973	
29.07	261	VB	3.611	
29.12	1417	B.	19.713	
29.27	1526	VV	20.650	
[illegible]	615	V.	8.556	
29.45	417	VB	5.797	
29.62	796	BV	11.067	
29.77	2261	VV	31.461	
29.88	1256	VB	17.792	
30.25	9255	VB	129.083	
30.74	553	B.	7.690	
30.75	461	BB	6.405	
30.76	654	VB	9.091	
30.94	205	BV	2.781	
31.01	734	VB	10.205	
31.12	1041	BV	14.502	
31.29	120906	BV	1680.92	
31.43	1455	BB	20.263	
31.85	213	BB	2.959	
31.99	516	BB	7.208	
32.09	2035	BB	28.345	
32.23	203	BB	2.822	
32.44	346	BB	4.806	
32.58	984	BV	13.706	
32.69	1767	VV	24.573	
32.82	4646	VV	107.285	CHRYSENE-D12
33.05	567	BB	7.888	
33.26	1642	BV	22.833	
33.34	532	VB	7.390	
33.56	442	BB	6.149	
34.00	466	BB	6.737	
34.22	254	BV	3.538	
34.32	251	VB	3.494	
34.48	647	BB	9.002	
34.60	666	BB	9.263	
35.24	1718	BB	23.888	

TABLE 1 (continued)

27.72	709	BB	9.847
30.02	800	BB	11.297
34.42	652	BB	9.151
37.32	1992	BB	27.697
39.77	343	BB	4.765

TOTAL AREA = 3983967 TOTAL ug/L = 84295.414

PROCESSED DATA FILE: BM154 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 3.11). 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 named or 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 begins. Each spiking compound was added to produce a water concentration of 100 ug/L.

The above analysis includes all chromatographable compounds with retention times between 27 min and 34.000 (which corresponds to C-10 through C-24 normal hydrocarbons) and which can be extracted using the EPA Method 625 Extraction Protocol.

The identification of the priority pollutant components shown to be present in the above analysis at levels greater than 5 ug/L can be verified further by 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 ACID EXTRACT OF THE INFLUENT SAMPLE, DILUTED TEN TIMES

RT	AREA	UG/L	NAME
[illegible]	207 BB	2.776	
5.77	1151 BP	15.467	
6.3[illegible]	736 BB	9.891	
7.[illegible]	546 BB	7.349	
8.77	830 BP	11.156	
[illegible]	17075 BV	185.455	PHENOL-D5
9.44	10[illegible] VB	146.408	
10.1[illegible]	1510 BB	20.293	
[illegible]	[illegible]	[illegible]	
10.42	4[illegible] VV	57.513	
10.[illegible]	[illegible] VB	696.349	CL2-BENZENE-D4
11.[illegible]	42[illegible] BB	5.759	
[illegible]	336 BB	4.543	
12.44	52[illegible] BF	6.9[illegible]	
12.[illegible]	[illegible] BB	4432.68	
13.[illegible]	264 BB	2.757	
13.52	6[illegible] BV	85.42[illegible]	
13.[illegible]	21232 VB	284.594	
14.0[illegible]	909 BB	12.205	
14.17	38774 BV	520.924	
14.2[illegible]	4425 VB	62.195	
14.[illegible]	425 BB	5.710	
15.1[illegible]	6132 BV	82.351	
15.2[illegible]	2655 VV	36.131	
15.[illegible]	2324 VB	31.219	
15.6[illegible]	699 BB	9.396	
15.[illegible]4	3790 BB	50.919	
16.1[illegible]	344 BB	4.912	
16.34	1361 BB	18.281	
16.[illegible]5	[illegible]9417 BV	529.558	
16.64	5634 VB	75.691	
17.18	659 BV	8.850	
17.28	36620 VB	491.985	
17.64	841 BB	11.292	
17.9[illegible]	2152 BB	28.915	
18.20	274 BV	3.676	
18.2[illegible]	8639 VV	116.065	
18.45	219 VB	2.949	
18.[illegible]	7504 BB	100.840	
20.05	772 BB	10.372	
20.32	120942 BB	1624.83	
20.[illegible]	525 BB	7.047	
20.79	2244 BB	30.149	
21.17	1049 BV	14.096	

TABLE 2 (continued)

RT	AREA	ug/L	NAME
21.24	340	VB	4.034
21.40	625	BB	5.715
21.65	3891	BB	31.044
22.16	2609	BB	32.727
22.49	372	BB	4.728
23.14	303	BB	4.064
[illegible]	305	BB	4.067
24.19	1248	BB	16.717
26.41	746.4	BE	ANTHRACENE-D10
27.4	7..	BB	5.77..
27.40	340	BB	4.8.7
27.67	450	BB	6.17.
28.6[illegible]	2.4	BB	3.14.
31.34	2734	BB	36.751
32.42	3..	BB	4.64.
34.91	223	BB	3.0.6

TOTAL AREA = [illegible] TOTAL ug/L = 10,314.775

PROCESSED DATA FILE: BR1:1 RAW DATA FILE: BR1:1

NOTE: The above report is generated from the capillary GC/FID analysis of a methylene chloride extract of an acidic water sample from [illegible]. The retention time units are minutes; the area units are integrator counts and the component concentrations have been 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 spiking compounds which are added to the water sample before extraction begins. Each spiking compound was added to produce a water concentration of 100 ug/L.

The above analysis includes all chromatographable compounds with levels ranging between 7.000 and 36.000 (which corresponds to C7 through C34 normal hydrocarbons) and which can be extracted using the EPA Method 625 Extraction Protocol.

The identification 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 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	91306	BV	1403.01	
3.87	18639	VV	289.476	
4.06	54661	VB	839.653	
5.05	779	BB	11.913	
5.77	171569	BV	2636.65	
6.11	4247	VB	65.267	
6.19	571	BV	8.776	
[illegible]	110317	VV	1695.5[illegible]	
6.56	60419	VV	928.4[illegible]	
[illegible]	[illegible]	VB	[illegible]	
7.7[illegible]	[illegible]	BB	[illegible]	
7.59	236	BB	3.626	
8.37	5669	BV	87.116	
8.41	[illegible]	VV	391.918	
8.6[illegible]	10375	VV	159.445	
8.77	[illegible]	VV	1594.21	
[illegible]	1468[illegible]	VV	222.754	
[illegible]	317182	VV	[illegible]	PHENOL-D5
9.44	[illegible]	VB	7.763	
[illegible]	41408	BV	636.278	
[illegible]	[illegible]	VV	34.202	
10.02	42281	VV	649.715	
[illegible]	5415[illegible]	VV	[illegible]	
10.34	[illegible]	VV	1361.5[illegible]	
10.44	134[illegible]75	VV	2069.47	
[illegible]	22472	VV	[illegible].862	CL2-BENZENE-D4
[illegible]	[illegible]	VV	5451.27	
10.89	[illegible]	VV	327.640	
10.98	2790	VV	42.877	
11.07	1752	VV	26.918	
11.16	5569	VV	85.503	
11.30	2924	VV	44.937	
11.49	226	VB	3.477	
11.59	1405	BV	21.583	
11.63	910	VV	13.985	
11.79	5200	VV	79.900	
11.88	20121	VV	309.184	
12.12	706	BB	10.875	
12.27	604	VV	9.280	
12.34	1071	VV	16.460	
12.44	21884	VV	336.303	
12.58	2933	VV	45.067	
12.74	99770	VV	1533.37	
12.86	17765	VV	272.983	

TABLE 3 (continued)

RT	AREA		ug/L	NAME
13.00	3330	VV	51.262	
13.17	6881	VV	105.279	
13.25	4986	VV	76.149	
13.38	8291	VV	127.393	
13.62	9709	VV	149.182	
13.76	1292	VV	19.854	
13.85	1581	VB	24.299	
14.00	38996	BV	599.216	
14.12	7802	VV	119.881	
14.24	227062	VV	3489.04	
14.37	26382	VV	405.402	
14.59	2899	VB	44.542	
14.70	404	BB	6.204	
14.93	27135	BV	416.955	
15.21	433350	VV	6658.87	
15.34	217321	VB	3339.36	
15.71	2018	BV	31.004	
15.83	373	B.	5.734	
15.97	8204	VV	126.062	
16.11	5292	VV	81.319	
16.22	1914	VV	29.404	
16.31	2411	VB	37.045	
16.61	201594	BV	3097.70	
16.83	8771	VV	134.315	
17.07	392	VB	6.026	
17.23	2654	BB	40.941	
17.55	262	VB	4.023	
17.61	3334	BV	51.173	
17.82	10219	VV	157.030	
18.05	73858	VV	1134.91	
18.24	8938	VV	137.335	
18.44	405452	VV	6291.65	
18.65	2556	VB	39.277	
19.22	14981	BV	230.201	
19.39	22672	VV	348.381	
19.57	1161	VB	17.834	
20.06	3020	BV	46.401	
20.26	294	BB	4.514	
20.45	4617	BV	70.944	
20.64	547	VB	8.405	
20.91	2201	BB	33.820	
21.26	380	BB	5.847	
21.35	316	BB	4.890	
21.69	189673	BV	2914.52	
21.78	4509	VB	69.281	
22.19	824	BB	12.660	
22.37	957	BB	14.713	
22.64	1937	BB	29.768	
22.90	210	BB	3.227	
23.04	13553	BV	208.250	
23.14	5854	VV	89.946	
23.25	1048	VV	16.102	
23.32	446	VB	6.847	
23.44	3370	BV	51.781	
23.62	3331	VV	51.181	
23.66	6455	VV	99.183	
23.98	322	VB	4.944	
24.13	1829	BB	28.099	

TABLE 3 (continued)

RT	AREA		ug/L	NAME
[illegible]	65975	BV		[illegible]ANTHRACENE-D10
24.51	3505	VB	53.856	
25.[illegible]	1651	BB	75.344	
26.31	291	BB	4.472	
26.85	955	BB	14.670	
26.80	7047	BB	106.925	PYRENE-D10
[illegible]	647	BB	9.946	
[illegible]	11755	BB	181.156	
31.37	39456	BB	600.141	
[illegible]	8848	BB	115.524	CHRYSENE-D12

TOTAL AREA = 4154221 TOTAL ug/L = 62744.203

PROCESSED DATA FILE: BR157 RAW DATA FILE: BR157

NOTE: The above report is generated from the capillary GC/FID analysis of a methylene chloride extract of a basic water sample (BR157). 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 spiking compounds which are added to the water sample before a fraction taken. 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 nonpriority 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 4. AMOUNTS OF CHROMATOGRAPHABLE COMPOUNDS DETECTED IN THE ACID EXTRACT OF THE EFFLUENT SAMPLE, DILUTED TEN TIMES BEFORE ANALYSIS

RT	AREA		ug/L	NAME
3.51	215	BB	2.921	
7.54	3974	BB	52.543	
9.22	14[illegible]	BV	191.544	PHENOL-D5
9.44	17407	VB	232.473	
10.[illegible]	335	BB	4.46[illegible]	
10.97	68752	BB	[illegible].034	CL2-BENZENE-D4
11.4	210	BB	2.801	
12.[illegible]	[illegible]145	BB	[illegible]1.[illegible]	
12.[illegible]	[illegible]	B[illegible]	[illegible]	
13.[illegible]	[illegible]	VB	[illegible]	
14.15	84[illegible]	BV	11[illegible].4[illegible]	
14.20	10[illegible]	VB	142.4[illegible]	
14.7[illegible]	111[illegible]	BB	14.[illegible]	
15.14	[illegible]	BB	8.[illegible]	
15.[illegible]	[illegible]	BB	2[illegible].81[illegible]	
15.71	[illegible]	BI	[illegible].84[illegible]	
15.94	5[illegible]71	BB	7[illegible].741	
16.[illegible]	16[illegible]	BB	2[illegible]	
16.[illegible]7	[illegible]47[illegible]	B[illegible]	7[illegible]1.[illegible]	
16.84	[illegible]	VB	[illegible]1.6[illegible]	
17.1[illegible]	[illegible]	B[illegible]	4.4[illegible]1	
17.2[illegible]	47[illegible]	VB	605.[illegible]	
17.4[illegible]	[illegible]	BB	6.941	
17.5[illegible]	1141	BB	15.2[illegible]	
18.[illegible]	154[illegible]	BB	20.691	
18.57	74[illegible]4	B[illegible]	9[illegible].28[illegible]	
20.[illegible]4	27[illegible]	VB	[illegible].472	
20.2[illegible]	97270	BB	12[illegible].06	
20.8[illegible]	857	BB	11.85[illegible]	
21.[illegible]	272[illegible]	BB	36.32[illegible]	
22.17	2[illegible]74	BB	31.70[illegible]	
23.96	[illegible]4[illegible]	BB	4.576	
24.42	74[illegible]77	BB		&ANTHRACENE-D10
27.66	5[illegible]	BB	7.9[illegible]	
28.67	2[illegible]	BB	[illegible].3[illegible]	
32.42	3[illegible]	BB	[illegible].102	

TOTAL AREA = 836072 TOTAL ug/L = 10077.871

PROCESSED DATA FILE: BF162 RAW DATA FILE: BR162

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) [illegible] or 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 EFFLUENT DUPLICATE SAMPLE, DILUTED TEN TIMES BEFORE ANALYSIS

RT	AREA		UG/L	NAME
3.44	86117	BV	1294.52	
3.64	18263	VV	266.361	
4.04	51396	VP	755.042	
5.07	223	BB	3.282	
5.73	179655	BV	2650.42	
6.15	53234	VV	783.515	
6.30	127147	VV	1878.65	
6.51	18657	VV	274.397	
7.31	16729	VB	245.754	
7.72	621	BB	9.131	
7.85	316	BV	4.646	
8.34	1856	BB	27.267	
8.43	17521	BV	257.417	
8.61	6765	VV	99.445	
8.71	100546	VV	1476.49	
8.96	13267	VV	195.029	
9.22	333443	VV	4994.41	PHENOL-D5
9.44	1539	VV	22.604	
9.54	44203	VV	649.373	
9.85	2375	VV	34.855	
10.02	42717	VV	627.544	
10.11	55497	VV	816.219	
10.34	88827	VV	1304.90	
10.44	145022	VV	2130.61	
10.51	19059	VV	309.626	CL2-BENZENE-D4
10.62	377616	VV	5536.62	
10.85	22167	VV	325.456	
10.93	1830	VV	26.898	
11.07	1912	VV	28.095	
11.16	5446	VV	80.037	
11.34	3101	VV	45.550	
11.50	206	VB	3.023	
11.59	227	BB	3.336	
11.65	232	BV	3.414	
11.79	4984	VV	73.222	
11.84	21175	VV	311.078	
12.12	900	BV	13.215	
12.20	293	VV	4.301	
12.27	583	VV	8.645	
12.34	1165	VV	17.109	
12.45	23032	VV	338.354	
12.53	3014	VV	44.272	
12.74	108214	VV	1589.73	
12.86	17192	VV	252.554	

TABLE 5 (continued)

RT	AREA		ug/L	NAME
13.00	3237	VV	47.849	
13.17	6261	VV	91.983	
13.25	4356	VV	64.023	
13.39	7547	VV	110.870	
13.62	5997	BV	86.096	
13.77	918	VV	13.461	
13.85	1403	VB	20.609	
14.06	41905	BV	615.604	
14.12	8543	VV	125.497	
14.26	239525	VV	3524.77	
14.37	29161	VV	428.393	
14.53	3511	VV	51.903	
14.70	802	VB	11.775	
14.91	28222	BV	414.721	
15.21	471529	VV	6927.05	
15.37	236279	VV	3471.05	
15.56	542	VB	7.962	
15.71	2241	BB	32.925	
15.83	432	VV	6.345	
15.95	9385	VV	137.872	
16.13	5840	VV	85.653	
16.27	2249	VV	33.032	
16.47	2771	VB	40.710	
16.61	232417	BV	3414.65	
16.83	8475	VV	124.502	
17.04	425	VB	6.257	
17.27	3165	BB	46.504	
17.59	1395	BV	20.525	
17.62	6254	VV	91.871	
17.82	11795	VV	173.283	
18.06	81438	VV	1196.37	
18.20	9766	VV	143.474	
18.42	459281	VV	6747.12	
18.63	290	VB	4.267	
18.71	1602	BB	23.535	
19.22	17065	BV	250.757	
19.29	26001	VV	381.965	
19.57	1707	VV	25.071	
19.64	727	VB	10.685	
20.06	1373	BB	20.164	
20.26	408	BB	5.993	
20.45	5307	BV	77.965	
20.65	763	VB	11.211	
20.91	2327	BB	34.179	
21.23	404	BB	5.929	
21.35	393	BB	5.775	
21.70	217290	BV	3192.12	
21.79	5215	VB	76.616	
22.19	843	BV	12.378	
22.37	558	BB	8.194	
22.64	2526	BB	37.113	
22.90	214	BB	3.147	
23.04	15629	BV	229.596	
23.15	6809	VV	100.034	
23.25	1419	VV	20.847	
23.32	884	VV	12.984	
23.45	4465	VV	65.587	
23.61	3721	VV	54.671	

TABLE 5 (continued)

RT	AREA		ug/L	NAME
23.63	6659	VB	97.830	
24.13	3047	BB	50.072	
24.47	67471	BV		ANTHRACENE-D10
24.53	4206	VB	61.799	
24.91	296	BB	4.348	
25.50	227	BB	3.337	
25.64	1892	BB	27.787	
26.01	405	BB	5.944	
26.65	1054	BB	15.420	
27.06	207	BB	3.077	
28.00	8915	BB	126.961	PYRENE-D10
[illegible]	971	BB	14.267	
[illegible]	[illegible]	BB	154.999	
31.57	41[illegible]	BB	60[illegible]	
31.93	370	BB	5.437	
32.82	9576	BB	157.453	CHRYSENE-D12

TOTAL AREA = 4485726 TOTAL ug/L = 64778.951

PROCESSED DATA FILE: BF156 RAW DATA FILE: BR156

NOTE: The above report is generated from the capillary GC/FID analysis of a methylene chloride extract of a basic water sample (Run 111). 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 named as E- assuming the unknown compound response is equal to the internal standard- anthracene-d1. Other deuterated compounds identified are surrogate analyte compounds which are added to the water sample before extraction begins. 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 36,000 (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 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	362	BB	4.859	
7.24	3761	BB	50.750	
9.21	15467	BV	146.085	PHENOL-D5
9.8	17611	VB	235.126	
10.57	65955	BB	861.292	CL2-BENZENE-D4
12.5	261497	BB	3507.55	
13.52	6189	BV	85.041	
13.57	[illegible]	VB	736.583	
14.14	[illegible]	BV	1112.26	
14.24	10705	VB	143.657	
14.7	575	BB	15.081	
15.1	1776	BB	25.176	
15.1	644	BB	6.962	
15.84	5612	BB	75.306	
16.1	1670	BB	22.411	
16.47	52755	BV	704.915	
16.54	7408	VB	104.742	
17.1	41655	BB	572.346	
17.41	534	BB	7.111	
17.4	941	BB	12.659	
17.1	847	BB	11.622	
17.5	7166	BB	96.445	
17.1	93155	BB	1254.25	
17.7	814	BB	10.916	
18.6	2102	BB	28.210	
18.17	2593	BB	34.799	
18.1	370	BB	4.966	
24.61	74217	BB		ANTHRACENE-D10
26.4	320	BB	4.294	
27.47	462	BB	6.203	
28.66	275	BB	3.684	
31.42	301	BB	4.034	

TOTAL AREA = 623154 TOTAL ug/L = 9958.256

PROCESSED DATA FILE: BF163 RAW DATA FILE: BA163

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 spiking compounds which are added to the water sample before extraction begins. 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 7. AMOUNT OF CHROMATOGRAPHABLE COMPOUNDS DETECTED IN THE METHYLENE CHLORIDE EXTRACT OF THE SEDIMENT SAMPLE, DILUTED TEN TIMES BEFORE ANALYSIS

RT	AREA		UG/G	NAME
5.4[illegible]	2110	BV	30.020	
[illegible]	924	VP	13.173	
[illegible]	[illegible]	PP	11.460	
[illegible]	[illegible]	BB	[illegible]	
[illegible]	2464	PP	35.[illegible]	
[illegible]	[illegible]	BB	19.33[illegible]	
[illegible]	[illegible]	[illegible]	31.1[illegible]	
[illegible]	5[illegible]66	BV	84.667	
[illegible]	[illegible]	VV	60.764	
[illegible]	[illegible]	[illegible]	[illegible]	
[illegible]	[illegible]	[illegible]	[illegible]	
[illegible]	[illegible]	[illegible]	[illegible]	
[illegible]	[illegible]	VV	[illegible]	
[illegible]	[illegible]	BV	110.455	PHENOL-D5
[illegible]	[illegible]	[illegible]	[illegible]	
[illegible]	2131	VV	30.316	
[illegible]	[illegible]	VP	7.607	
[illegible]	[illegible]	BV	[illegible]	
[illegible]	[illegible]	VV	46.78[illegible]	
[illegible]	62651	VV	891.220	
[illegible]	[illegible]	VV	592.07[illegible]	
[illegible]	[illegible]	VV	[illegible]	CL2-BENZENE-D4
1[illegible].44	[illegible]	VB	5.387	
[illegible]	[illegible]	BV	34.315	
[illegible]	[illegible]	[illegible]	[illegible]	
[illegible]	[illegible]	[illegible]	[illegible]	
[illegible]	[illegible]	[illegible]	[illegible]	
[illegible]	[illegible]	[illegible]	5.70[illegible]	
11.4[illegible]	[illegible]	[illegible]	[illegible]	
11.5[illegible]	6181	VV	87.9[illegible]	
11.7[illegible]	[illegible]	VV	[illegible]	
11.9[illegible]	[illegible]	BB	7.56[illegible]	
11.[illegible]	[illegible]	BV	[illegible]	
12.10	[illegible]	VV	22.687	
12.21	1175	VB	17.006	
12.[illegible]	[illegible]	BB	3.661	
12.4[illegible]	217	BV	3.090	
12.58	260	VP	3.694	
12.64	333	PB	4.737	
12.74	585	VP	8.315	
12.[illegible]	215[illegible]	BV	30.71[illegible]	
[illegible]	[illegible]	VB	7.076	
13.0[illegible]	513	BV	7.300	
13.17	[illegible]	VV	111.03[illegible]	

TABLE 7 (continued)

RT	AREA	[illegible]	[illegible]
13.42	3909 BB	55.686	
13.85	5034 BV	71.638	
14.12	976 BV	13.882	
14.62	2449 VV	34.844	
14.76	8630 VV	122.770	
14.91	4640 VV	66.113	
15.11	460 VB	6.544	
15.28	1006 BB	14.306	
15.80	2484 BB	35.364	
16.04	315 BV	4.484	
16.73	[illegible] VB	3.585	
16.88	423 BB	6.020	
16.97	514 BB	7.[illegible]	
17.0	[illegible] BB	5.0[illegible]	
17.20	415 BB	5.[illegible]	
17.32	200 VB	2.846	
17.42	737 BB	10.4[illegible]	
17.60	8644 BV	122.970	
17.80	1721 VV	24.478	
17.96	487 BB	6.929	
18.15	[illegible] VB	3.394	
18.22	357 BB	5.077	
18.50	215 BB	3.104	
[illegible]	[illegible] VB	13.644	
[illegible]	5171 BB	73.562	
[illegible]	1755 BB	24.971	
[illegible]	34[illegible] BB	4.726	
[illegible]	310 BB	4.410	
[illegible]	410 BB	5.8[illegible]	
20.0[illegible]	1139 BV	16.202	
20.15	384 VB	5.466	
20.25	356 BB	5.057	
20.35	683 BV	9.709	
20.37	272 VV	3.956	
20.45	923 VV	13.135	
20.55	5041 VV	71.766	
20.7[illegible]	632 VB	8.992	
20.80	4760 BV	65.154	
20.91	2271 VV	32.305	
20.9[illegible]	222 VV	3.163	
21.14	306 BB	4.358	
21.2[illegible]	675 BB	9.315	
21.64	1420 BB	20.200	
21.9[illegible]	471 BB	6.920	
21.98	496 BB	7.064	
22.11	3490 BB	49.653	
22.42	1060 BV	15.074	
22.51	1647 VV	23.431	
22.64	1901 VV	27.037	
22.76	2946 VV	41.907	
22.86	1907 VV	27.132	
22.93	1010 VV	14.365	
23.07	2658 BV	37.813	
23.21	5136 VV	73.088	
23.33	1934 VV	27.512	
23.40	998 VV	14.188	
23.45	1329 VV	18.899	
23.5[illegible]	1865 VV	26.535	
[illegible]	864 VV	12.[illegible]93	

TABLE 7 (continued)

23.77	[illegible]	VV	129.001	
23.84	2269	VV	32.980	
24.00	60609	VV		ANTHRACENE-D10
[illegible]	490	VB	6.515	
[illegible]	223	BB	3.172	
25.00	1561	BV	22.205	
25.14	1021	VV	14.517	
25.2[illegible]	1220	VB	17.070	
25.37	348	BB	4.950	
25.47	722	BB	10.776	
[illegible]	395	BB	5.624	
25.7[illegible]	249	BB	3.541	
[illegible]	26[illegible]	BB	3.709	
25.96	394	BB	5.599	
26.15	[illegible]	BB	12.[illegible]	
26.21	40[illegible]	BV	6.[illegible]	
26.31	295	VB	4.204	
26.4[illegible]	1649	BV	23.451	
[illegible]	[illegible]	VV	[illegible]	
26.69	1360	VB	19.341	
26.8[illegible]	2344	BB	33.345	
27.0[illegible]	833	BB	11.853	
[illegible]	[illegible]	BB	3.[illegible]	
[illegible]	2977	BV	42.354	
[illegible]	[illegible]	VV	[illegible].776	
27.7[illegible]	210[illegible]	VV	29.869	
[illegible]	660	VB	9.36[illegible]	
28.1[illegible]	243[illegible]	BV	34.[illegible]	
[illegible]	179[illegible]	VV	[illegible]	
[illegible]	[illegible]	VV	114.406	PYRENE-D10
28.54	792	VV	11.272	
28.71	334	BV	4.75[illegible]	
[illegible]	1127	BV	16.257	
29.3[illegible]	1257	BV	17.898	
[illegible]	4[illegible]	VB	6.[illegible]	
[illegible]	[illegible]	BB	[illegible].276	
[illegible]	1196	BV	17.045	
30.54	746	VV	10.611	
30.61	799	VB	11.364	
30.72	[illegible]	BB	9.940	
[illegible]	[illegible]	BB	2709.74	
31.2[illegible]	1079	BB	15.350	
31.67	1890	BB	26.997	
32.1[illegible]	971	BB	13.809	
32.2[illegible]	[illegible]	BB	89.427	CHRYSENE-D12
[illegible]	261	BB	4.004	
[illegible]	1421	BB	20.216	

TOTAL AREA = [illegible] TOTAL ug/g = 8540.314

PROCESSED DATA FILE: BP143 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, or 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 sample before extraction began. Each spiking compound was added to produce a sediment concentration of 200 ug/g.

The above analysis includes all chromatographable compounds with [illegible] indices between 2,000 and 34,000 (which corresponds [illegible]

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

RT	AREA		ug/g	NAME
5.43	5151	BV	27.474	
5.47	1149	VB	10.676	
5.80	1108	BB	10.113	
6.00	7870	BV	75.154	
6.40	2731	BV	34.874	
7.31	1453	BB	18.542	
7.64	2364	BB	30.190	
[illegible]	6719	BV	86.[illegible]	
[illegible]	514[illegible]	VV	65.766	
8.17	2162	VV	25.885	
8.26	2215	VV	26.341	
8.40	7598	VV	97.050	
8.54	5927	VB	75.707	
8.86	16795	BV	114.963	PHENOL-D5
9.19	10487[illegible]	BV	133.750	
9.[illegible]	2[illegible]	VV	28.[illegible]	
9.49	68[illegible]	VB	8.752	
9.65	10[illegible]	BV	132.732	
9.77	3[illegible]	VV	44.[illegible]	
9.[illegible]	70[illegible]	VV	894.457	
10.[illegible]	46[illegible]	VV	592.095	
10.[illegible]	24[illegible]	VV	310.917	CL2-BENZENE-D4
10.44	[illegible]1	VB	4.[illegible]	
10.[illegible]	2685	BV	34.236	
10.71	2102	VV	26.[illegible]	
10.[illegible]	4[illegible]4	VV	[illegible]	
10.87	1[illegible]	VV	13.[illegible]	
11.[illegible]	1.[illegible]	VB	16.[illegible]	
11.[illegible]	[illegible]1	BB	6.[illegible]	
11.40	8955	BV	113.984	
11.52	7267	VV	93.092	
11.7[illegible]	5461	VB	69.754	
11.[illegible]	[illegible]	BB	7.809	
11.[illegible]	[illegible]	BB	3.[illegible]	
12.11	1840	BV	23.477	
12.21	1473	VV	18.810	
12.45	205	BB	2.556	
12.[illegible]	261	BB	3.[illegible]	
12.[illegible]	374	BB	4.774	
12.91	2685	BV	34.244	
13.01	7[illegible]	VV	10.122	
13.10	751	VV	9.593	
13.[illegible]	7964	VV	101.713	
13.[illegible]	21[illegible]	VB	2.[illegible]	
13.[illegible]	4957	BV	63.309	
13.[illegible]	5939	VB	75.858	

TABLE 8 (continued)

RT	AREA		ug/g	NAME
14.13	1202	BV	15.383	
14.21	205	VB	2.622	
14.46	274	BB	3.382	
14.62	2879	BV	36.771	
14.76	10467	VV	132.688	
14.92	4854	VV	61.992	
15.02	279	VB	3.822	
15.12	555	BB	7.091	
15.24	1242	BB	15.954	
15.61	3222	BB	41.153	
16.14	255	BV	3.254	
16.20	215	VB	2.803	
16.31	327	BB	4.180	
[illegible]	[illegible]	BF	[illegible]	
16.74	265	BV	3.674	
16.86	269	VB	3.440	
[illegible]	527	PB	6.859	
16.97	770	BB	9.829	
17.10	419	BB	5.354	
17.21	534	VB	6.351	
17.32	391	BB	4.996	
17.42	[illegible]	BV	11.231	
17.61	[illegible]	VV	12[illegible].501	
17.86	[illegible]	VV	25.666	
17.97	543	BB	6.941	
18.22	318	BP	4.068	
[illegible]	1073	BB	13.767	
[illegible]	5775	BV	73.760	
[illegible]	1881	BB	24.030	
[illegible]	427	BP	5.430	
19.82	[illegible]	VB	3.941	
19.91	475	BB	6.067	
20.07	1349	BV	17.221	
20.16	[illegible]	VV	6.775	
20.26	455	VB	5.852	
20.34	795	BV	10.189	
20.38	267	VB	3.695	
20.46	1023	BV	13.066	
20.56	5365	VV	68.515	
20.71	733	VV	9.357	
20.80	5126	VV	65.468	
20.91	[illegible]	VV	32.055	
20.97	330	VB	4.221	
21.14	591	BV	7.551	
21.21	859	VB	10.970	
21.56	202	BB	2.586	
21.65	1540	BB	19.674	
21.91	443	BB	5.655	
21.99	516	BB	6.595	
22.12	3486	BB	44.528	
22.42	1113	BV	14.217	
22.52	1784	VB	22.780	
22.65	2024	BV	25.852	
22.77	3089	VV	39.448	
22.87	1947	VV	24.867	
22.93	1084	VV	13.849	
23.00	2791	BV	35.648	
23.21	5367	VV	68.585	
23.33	2008	VV	26.031	

TABLE 8 (continued)

RT	AREA		ug/g	NAME
[illegible]	982	VV	12.544	
23.46	1374	VB	17.552	
23.53	1833	BV	23.413	
23.69	872	VV	11.139	
23.77	9633	VV	123.036	
23.84	2544	VV	32.496	
24.06	56735	VB		ANTHRACENE-D10
24.30	561	BB	7.168	
[illegible]	260	BV	3.325	
[illegible]	1610	VV	20.560	
25.15	1096	VV	14.026	
[illegible]	1326	VB	16.931	
[illegible]	[illegible]	PP	6.750	
25.47	[illegible]	BB	9.444	
[illegible]	377	PP	4.824	
25.71	[illegible]	BV	3.439	
[illegible]	501	VV	6.394	
[illegible]	337	VB	4.302	
[illegible]	464	BB	5.920	
[illegible]	1448	VV	18.494	
[illegible]	719	VP	9.187	
[illegible]	331	BB	4.229	
[illegible]	1826	BV	23.327	
[illegible]	2748	VV	35.111	
[illegible]	1368	VV	17.479	
[illegible]	2564	BV	32.746	
[illegible]	[illegible]	PP	[illegible]	
[illegible]	216	BB	2.762	
[illegible]	[illegible]	VP	9.576	
[illegible]	[illegible]	BP	3.214	
[illegible]	3222	BV	41.146	
[illegible]	[illegible]	VV	24.341	
[illegible]	2347	VV	[illegible]	
[illegible]	677	VB	8.657	
[illegible]	514	BB	6.591	
[illegible]	2644	BV	[illegible]	
[illegible]	2041	VV	26.064	
[illegible]	10717	VV	107.966	PYRENE-D10
[illegible]	1142	VV	14.594	
[illegible]	310	BB	3.956	
[illegible]	1390	BV	[illegible]	
[illegible]	486	VV	6.213	
[illegible]	239	VB	3.052	
[illegible]	285	BB	3.643	
[illegible]	1606	BV	[illegible]	
[illegible]	[illegible]	VP	[illegible]	
[illegible]	4163	PB	53.174	
[illegible]	[illegible]	BB	3.305	
[illegible]	284	BB	3.626	
[illegible]	549	BB	7.016	
30.61	422	BV	5.384	
30.73	803	VB	10.257	
30.99	216876	BV	2770.02	
31.22	1623	VB	20.735	
31.55	626	BB	7.997	
31.67	1974	BV	25.216	
32.19	1562	BV	19.951	
32.34	6124	VB	100.000	CHRYSENE-D12
[illegible]	260	BB	3.322	
32.77	265	BB	3.389	
[illegible]	314	BB	4.007	

TABLE 8 (continued)

32.56	392	BB	5.010
32.78	2026	BB	23.933
35.67	283	BB	3.610

TOTAL AREA = 760471 TOTAL ug/g = 8780.371

PROCESSED DATA FILE: BP144 RAW DATA FILE: BR144

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, or 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 sample before extraction begins. Each spiking 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 C34 normal hydrocarbons) and which can be extracted using methylene chloride as an extracting solvent.

The identification of non-priority pollutant compounds shown to be present in the above analysis at levels greater than 5 ppm can be obtained by further interpretation of the mass spectral data obtained from the capillary GC/FID analysis for priority pollutants. This work is 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 (µg/L for water sampler and µg/g for sediment samples)		
	GC/FID	GC/MS-Total	GC/MS-PP
Influent base/neutal extract	54,000	20,000	5,200
Influent acid extract	10,000	5,500	12,000
Effluent base/neutal extract	63,000	34,000	3,700
Effluent acid extract	10,000	6,800	11,200
Effluent (duplicate) base/neutal 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, µg/L	Compound	Screening level, µg/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
		2-Chloronaphthalene	10
Chloromethane	10	Acenaphthylene	10
Dichlorodifluoromethane	10	Acenaphthene	10
Bromomethane	10	Isophorone	10
Vinyl chloride	10	Fluorene	10
Chloroethane	10	2,6-Dinitrotoluene	10
Methylene chloride	10	1,2-Diphenylhydrazine	10
Trichlorofluoromethane	10	2,4-Dinitrotoluene	10
1,1-Dichloroethylene	10	N-nitrosodiphenylamine	10
1,1-Dichloroethane	10	Hexachlorobenzene	10
Trans-1,2-dichloroethylene	10	4-Bromophenyl phenyl ether	10
Chloroform	10	Phenanthrene/anthracene	10
1,2-Dichloroethane	10	Dimethyl phthalate	10
1,1,1-Trichloroethane	10	Diethyl phthalate	10
Carbon tetrachloride	10	Fluoranthene	10
Bromodichloromethane	10	Pyrene	10
Bis(chloromethyl) ether	10	Di-n-butyl phthalate	10
1,2-Dichloropropene	10	Benzidine	10
Trans-1,3-dichloropropene	10	Butyl benzyl phthalate	10
Trichloroethylene	10	Chrysene	10
Dibromochloromethane	10	Bis(2-ethylhexyl) phthalate	10
Cis-1,3-dichloropropene	10	Benz(a)anthracene	10
1,1,2-Trichloroethane	10	Benzo(b)fluoranthene	10
Benzene	10	Benzo(k)fluoranthene	10
2-Chloroethyl vinyl ether	10	Benzo(a)pyrene	10
Bromoform	10	Indeno(1,2,3-cd)pyrene	25
Tetrachloroethylene	10	Dibenzo(a,h)anthracene	25
1,1,2,2-Tetrachloroethane	10	Benzo(g,h,i)perylene	25
Toluene	10	N-Nitrosodimethylamine	10
Chlorobenzene	10	N-nitrosodi-n-propylamine	10
		4-Chlorophenyl phenyl ether	10
Direct injectables:		3,3'-Dichlorobenzidine	10
		Di-n-octyl phthalate	10
Acrolein	200		
Acrylonitrile	100		

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

Compound	Screens (µg/liter) GC/MS	GC/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

[a] ND - compound not detected.

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

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

Analyte	Sample ID			
	Blank	Influent	Effluent	Effluent duplicate
Base/neutral				
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[b]	530	350	380
Diethyl phthalate	<10	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
Acid				
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

[a] ND - compound not detected.

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

TABLE 14. ANALYTICAL RESULTS FOR EXTRACTABLE ORGANIC PRIORITY POLLUTANTS IN SEDIMENT SAMPLES, µg/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

[a]Spike - 100 µg/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

[a]Spike level

Sediment - 187 µg/g
Sediment duplicate - 145 µg/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]	3	1	ND[d]
13.4	Unknown (58, 43, 41, 39)	ND	4	ND
15.5	Acetone	17	13	24
17.1	Unknown (45, 43, 59)	29	70	35
20.8	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-d_6, internal standard.

[b] Tentative identification, not confirmed with authentic standards.

[c] Major masses of unknown compounds are listed in parentheses according to decreasing intensitives.

[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
		Internal standard[b]				
2	21.28	Anthracene-d_{10}	309007	100	-	-
		Recovery standard[c,d]				
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	28.27	Chrysene-d_{12}	197093	64	100	64.0
		Organics detected				
		None detected				
		TOTAL CONCENTRATION				

[a] Includes 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.

[b] Anthracene-d_{10} internal standard added immediately prior to analysis; 50 mg/L injected equivalent to 100 µg/L 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.

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
		Internal standard[b]				
2	21.20	Anthracene-d_{10}	370013	100	-	-
		Recovery standard[c,d]				
1	6.70	Phenol-d_5	39178	39	100	39
		Organics detected				
		None detected				
		TOTAL CONCENTRATION				

[a] Includes 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.

[b] Anthracene-d_{10} internal standard added immediately prior to analysis: 50 mg/L injected equivalent to 100 µg/L 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.

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, µg/L	Amount spiked, µg/L	Percent recovery
		Internal standard[c]				
29	21.27	Anthracene-d_{10}	353040	1000	-	-
		Recovery standard[d,e]				
		1,2-Dichlorobenzene-d_4		56	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_3-benzene (isomer)	171284	4.9×10^2		
5	6.58	Aniline	411008	1.2×10^3		
6	6.90	C_3-benzene (isomer)	113840	3.2×10^2		
7	7.33	Dichlorobenzene (isomer)	138653	3.9×10^2		

(continued)

TABLE 20 (continued)

Peak No.	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, µg/L	Amount spiked, µg/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	1.7×10^3		
11	7.87	Dichlorobenzene (isomer)	411639	1.2×10^3		
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, µg/L	Amount spiked, µg/L	Percent recovery
		Organics detected (continued)				
27	18.73	Nitroaniline	172253	4.9×10^2		
28	20.28	C_n-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[i]		20.0×10^3		

[a] For background, see method blank analysis, Table 18.

[b] Includes 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 µg/L.

[c] Anthracene-d_{10} internal standard added immediately prior to analysis; 50 mg/L injected equivalent to 1000 µg/L in original sample.

[d] Deuterated recovery standard (spike), added to original sample before extraction.

[e] Quantitated relative to area of major ion for authentic standard.

[f] Estimated concentration based on area of total ion relative to that of anthracene-d_{10}, internal standard.

[g] Tentative identification, not confirmed with authentic standards.

[h] Major masses of unknown compounds are listed in parentheses according to decreasing intensities. Possible molecular ions are underlined.

[i] Summation 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)

TABLE 21 (continued)

Peak No.	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, µg/L	Amount spiked, µg/L	Percent recovery
		Organics detected (continued)				
8	16.02	Cl_2-containing unknown (207,209,97,111)	46427	1.1×10^2		
9	17.63	Nitrophenol isomer	317844	7.4×10^2		
		TOTAL CONCENTRATION[i]		5.5×10^3		

[a] For background, see method blank analysis, Table 19.

[b] Includes 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 µg/L.

[c] Anthracene-d_{10} internal standard added immediately prior to analysis; 50 mg/L injected equivalent to 1000 µg/L in original sample.

[d] Deuterated recovery standard (spike), added to original sample before extraction.

[e] Quantitated relative to area of major ion for authentic standard.

[f] Estimated concentration based on area of total ion relative to that of anthracene-d_{10}, internal standard.

[g] Tentative identification, not confirmed with authentic standards.

[h] Major masses of unknown compounds are listed in parentheses according to decreasing intensities. Possible molecular ions are underlined.

[i] Summation of concentrations of detected compounds, excluding standards.

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
		Internal standard[c]				
29	21.28	Anthracene-d_{10}	375998	1000	-	-
		Recovery standard[d,e]				
		1,2-Dichlorobenzene-d_4		88	100	88.0
		Pyrene-d_{10}		20	100	20.0
		Chrysene-d_{12}		73	100	73.0
		Organics detected[f,g,h]				
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)

TABLE 22 (continued)

Peak No.	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, µg/L	Amount spiked, µg/L	Percent recovery
		Organics detected (continued)				
7	7.33	Dichlorobenzene (isomer)	387403	1.0×10^3		
8	7.50	Unknown (111,125,154,71)	144061	3.8×10^2		
9	7.70	C_4-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	3.6×10^2		

(continued)

TABLE 22 (continued)

Peak No.	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, µg/L	Amount spiked, µg/L	Percent recovery
		Organics detected (continued)				
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}		

[a] For background, see method blank analysis, Table 18.

[b] Includes 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 µg/L.

[c] Anthracene-d_{10} internal standard added immediately prior to analysis; 50 mg/L injected equivalent to 1000 µg/L in original sample.

[d] Deuterated recovery standard (spike), added to original sample before extraction.

[e] Quantitated relative to area of major ion for authentic standard.

[f] Estimated concentration based on area of total ion relative to that of anthracene-d_{10}, internal standard.

[g] Tentative identification, not confirmed with authentic standards.

[h] Major masses of unknown compounds are listed in parentheses according to decreasing intensities. Possible molecular ions are underlined.

[i] Summation 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		30	100	30.0
		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	Dichlorophenol 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, µg/L	Amount spiked, µg/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	235563	5.7×10^2		
		TOTAL CONCENTRATION[i]		6.8×10^3		

[a] For background, see method blank analysis, Table 19.

[b] Includes 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 70 µg/L.

[c] Anthracene-d_{10} internal standard added immediately prior to analysis; 50 mg/L injected equivalent to 1000 µg/L in original sample.

[d] Deuterated recovery standard (spike), added to original sample before extraction

[e] Quantitated relative to area of major ion for authentic standard.

[f] Estimated concentration based on area of total ion relative to that of anthracene-d_{10}, internal standard.

[g] Tentative identification, not confirmed with authentic standards.

[h] Major masses of unknown compounds are listed in parentheses according to decreasing intensities. Possible molecular ions are underlined.

[i] Summation of concentrations of detected compounds, excluding standards.

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, µg/L	Amount spiked, µg/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	Dichlorobenzene (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, µg/L	Amount spiked, µg/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	18.00	Nitroaniline and 5% chloronitroaniline	480195	1.3×10^3		
29	28.13	Butyl benzyl phthalate	324456	9.0×10^2		
		TOTAL CONCENTRATION[i]		35.7×10^3		

[a] For background, see method blank analysis, Table 18.

[b] Includes 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 µg/L.

[c] Anthracene-d_{10} internal standard added immediately prior to analysis; 50 mg/L injected equivalent to 1000 µg/L in original sample.

[d] Deuterated recovery standard (spike), added to original sample before extraction.

[e] Quantitated relative to area of major ion for authentic standard.

[f] Estimated concentration based on area of total ion relative to that of anthracene-d_{10}, internal standard.

[g] Tentative identification, not confirmed with authentic standards.

[h] Major masses of unknown compounds are listed in parentheses according to decreasing intensities. Possible molecular ions are underlined.

[i] Summation of concentrations of detected compounds, excluding standards.

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, µg/L	Amount spiked, µg/L	Percent recovery
		Internal standard[c]				
10	21.28	Anthracene-d_{10}	425158	1000	-	-
		Recovery standard[d,e]				
		Phenol-d_5 1,2-Dichlorobenzene-d_4 Pyrene-d_{10} Chrysene-d_{12}		26	100	26.0
		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, µg/L	Amount spiked, µg/L	Percent recovery
		Organics detected (continued)				
6	14.05	Cl-containing unknown (121,73,66,173)	193047	4.5×10^2		
7	14.52	Trichlorophenol isomer	416595	8.8×10^2		
8	16.02	Cl_2-containing unknown (207,97,133,209)	34361	81		
9	17.62	Nitrophenol isomer	241521	5.7×10^2		
		TOTAL CONCENTRATION[i]		6.0×10^3		

[a] For background, see method blank analysis, Table 19.

[b] Includes 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 70 µg/L.

[c] Anthracene-d_{10} internal standard added immediately prior to analysis; 50 mg/L injected equivalent to 1000 µg/L in original sample.

[d] Deuterated recovery standard (spike), added to original sample before extraction.

[e] Quantitated relative to area of major ion for authentic standard.

[f] Estimated concentration based on area of total ion relative to that of anthracene-d_{10}, internal standard.

[g] Tentative identification, not confirmed with authentic standards.

[h] Major masses of unknown compounds are listed in parentheses according to decreasing intensities. Possible molecular ions are underlined.

[i] Summation 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[a]

Peak No.	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, µg/g	Amount spiked, µg/g	Percent recovery
		Internal standard[b]				
24	21.15	Anthracene-d_{10}	158680	935	-	-
		Recovery standard[c,d]				
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}	18800	99	187	53.0
40	29.10	Chrysene-d_{12}	11207	92	187	49.0
		Organics detected[e,f,g]				
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)-, trans-(-)	11247	66		

(continued)

TABLE 26 (continued)

Peak No.	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, µg/g	Amount spiked, µg/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 (97,69,83,70,139)	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)

TABLE 26 (continued)

Peak No.	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, µg/g	Amount spiked, µg/g	Percent recovery
		Organics detected (continued)				
19	20.02	Phenol,4-(1,1,3,3-tetramethylbutyl)	6362	37		
20	20.13	Nonyl phenol (isomer)	8284	49		
21	20.27	Nonyl phenol (isomer)	9803	58		
22a	20.4	Unknown (135,107,121,134)	2740	16		
22b	20.50	Unknown (91,210,121,85,107)	8279	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		
2h	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)

TABLE 26 (continued)

Peak No.	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, µg/g	Amount spiked, µg/g	Percent recovery
		Organics detected (continued)				
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		

[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 of limit of 10 µg/g.

[b] Anthracene-d_{10} internal standard added immediately prior to analysis; 50 mg/L injected equivalent to 935 µg/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 27. IDENTIFICATION OF MAJOR EXTRACTABLE COMPOUNDS IN THE METHYLENE CHLORIDE EXTRACT OF THE SEDIMENT DUPLICATE SAMPLE, DILUTED 1:10, NOT CORRECTED FOR BACKGROUND[1]

Peak No.	Retention time, min	Compound (major masses)	Peak area, total counts	Amount detected, µg/g	Amount spiked, µg/g	Percent recovery
		Internal standard[b]				
28	21.13	Anthracene-d_{10}	171907	725	-	-
		Recovery standard[c,d]				
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
		Organics detected[e,f,g]				
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, µg/g	Amount spiked, µg/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	24.80		15369	65		
36	25.30		9231	39		
37	25.55		5036	21		
38	25.73		3311	14		
39	26.50		9585	40		
40	26.80		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, µg/g	Amount spiked, µg/g	Percent recovery
		Organics detected (continued)				
42	27.90	(See previous table for compound identifications)	2018	9		
43 &	28.05-		1406794	5.9×10^3		
44	28.08					
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 of limit of 10 µg/g.

[b] Anthracene-d_{10} internal standard added immediately prior to analysis; 50 mg/L injected equivalent to 725 µg/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 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	<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

[a] ND - none detected.

[b] ND (<xx) - none detected (detection limit).

TABLE 29. ANALYSIS OF CHLORINATED DIBENZO-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

[a] ND - none detected.

[b] ND (<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

[a]ND - 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

[a]ND - 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	18.5	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

[a]All analyses by AA.

[b]50 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	1×10^4
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

[a]ND - not detected above lower detection limit.

[b]Duplicate 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	µg/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

[a]Analyses by AA, all others by ICP.

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

	Sample ID, µg/L (ppb)	
	Influent	Effluent[a]
Priority metals		
Arsenic[b]	31.7	4.3
Mercury[b]	<0.8	<0.8
Selenium[b]	14.3	9.95
Thallium	<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
Non-priority metals		
Aluminum	890	<450
Barium	187	77
Boron	388	674
Calcium	2.6×10^5	<210
Cobalt	<70	<70
Iron	6.8×10^3	253
Magesium	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

[a] Average of duplicate analyses.

[b] Analyses 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

[a]Analyses 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

[a]Least observable quality.

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

Analyte	Sample ID, µg/L (ppb) Influent	Effluent
Cyanide	NR[a]	10
Phenol	1384	1898

Quality Control Data

Reference standard	Observed value, µg/L	True value, µg/L	Percent recovery
Cyanide	110	160	69
Phenol	222	250	89

[a]NR - 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 value, mg/L	True value, mg/L	Percent recovery
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

[a]NR - analysis not requested.

APPENDIX A

GC/MS ANALYSES OF EXTRACTABLE ORGANICS

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 x 10^5 to 4 x 10^5 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 µL of the sample extract into 1.5 mL sample vials along with 150 µL of a 100 µg/mL anthracene-d_{10} in methylene chloride internal standard. The vial was capped and placed in the autosampler, which injected approximately 1 µL 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.

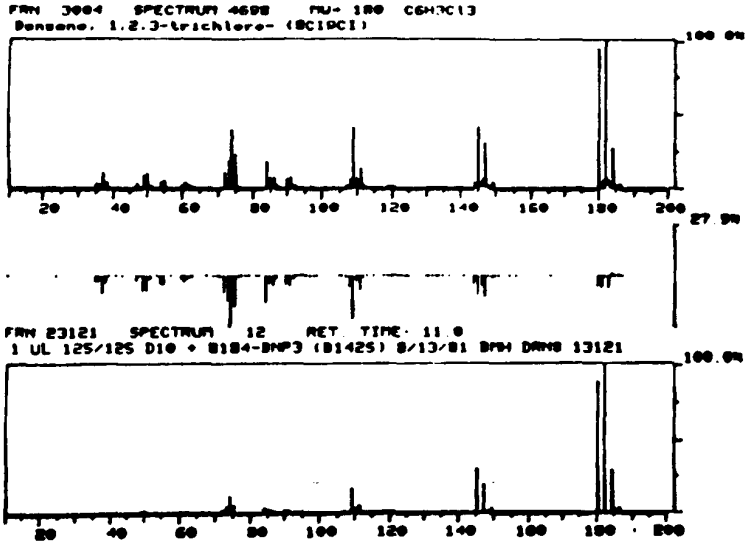


Figure A-1. Comparison of the mass spectrum of the highest matching compound with that of an unknown compound eluting at 11.0 minutes.

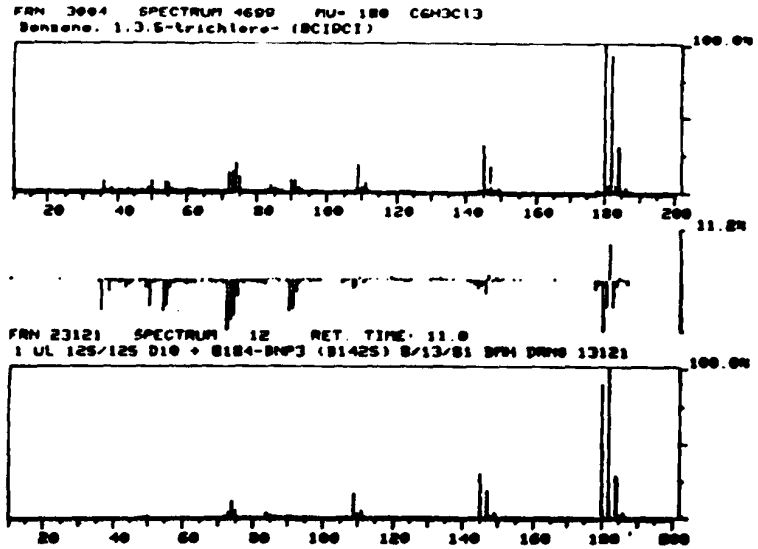


Figure A-2. Comparison of the mass spectrum of the third highest matching compound with that of an unknown compound eluting at 11.0 minutes.

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 occurrance 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-d_{10} 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-d_{10} 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.

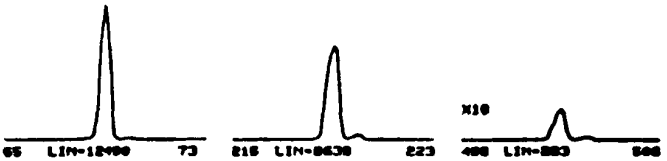
QA/QC for Extractable Organics

MRI 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 AQUIRE 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.

```
* CURRENT PARAMETERS *
(HP5985-1v041M)

DATE (FRM 1400 :: 1/28/91  1:15
REPELLER(V)=10.84          EM VOLTAGE(V)=2200
LAMADJT(V)=26.5            AMU GAIN=115
ION FOCUS(V)=35            AMU OFFSET=113
ENT LENS(MV/AMU)=133       MASS AXIS GAIN=1.00157
X-RAY(V)=154               MASS AXIS OFFSET=-.190491
EMISSION(UA)=300           IONS: POSITIVE
ELECT. ENERGY(EV)=70       ACTUAL SOURCE TEMP =200
LOG AMP OFFSET=1
```



ACTUAL MASS	ABS ABUND	REL ABUND	FWHM	ACTUAL ISO MS	ISO ABUND	ACTUAL ISO RATIO	EFF WIDTH
68.99	12409	100	.84	70	143	1.14	.929
218.97	8638	69.18	.69	218.98	433	5.01	.68
501.98	282	2.25	.52	503	26	9.21	.507

OPTION?

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

DFTPP EPA CRITERION VERIFIED

DRN: 22512
SPECTRUM: 2011

	Mass	Rel. Abund.	Criterion
**	51	9.05206	30-60% MASS 198
	68	0	< 2% MASS 69
	69	29.7239	
	70	0	< 2% MASS 69
**	127	33.4849	40-60% MASS 198
	197	0	< 1% MASS 198
	198	100	BASE PEAK
	199	6.69813	5-9% MASS 198
	275	22.8732	10-30% MASS 198
	365	1.70276	> 1% MASS 198
	441	14.7048	< MASS 443
	442	73.8367	> 40% MASS 198
	443	16.0367	17-23% MASS 442

NOTE: '**' indicates out of range!

PRESS <RETURN> TO RERUN...

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

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 performace standard were: (1) 2,6-dimethylphenol, (2) 2,6-dimethylaniline, (3) decanol, (4) pentachlorophenol, (5) anthracene-d_{10} 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.

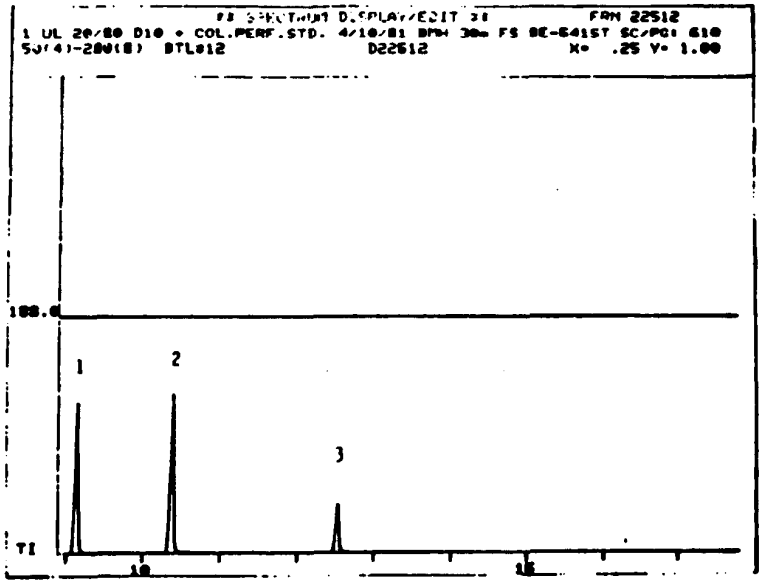


Figure A-5. Chromatogram of the column performance standard with the GC column in average condition. The numbered peaks are identified in the text.

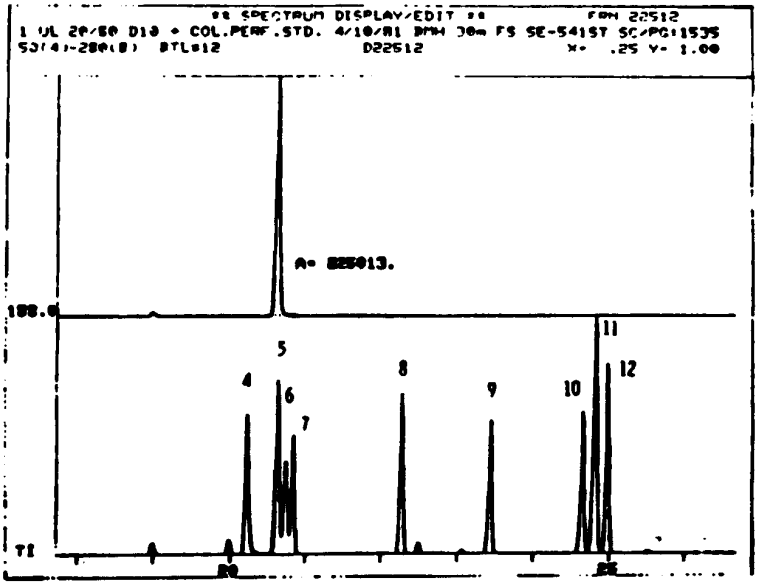


Figure A-5 (continued)

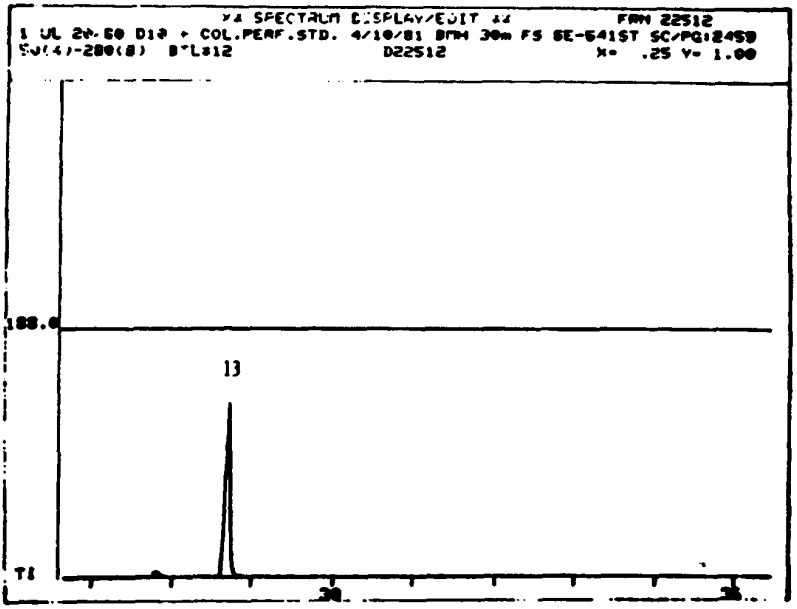


Figure A-5 (continued)

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 ±30%.

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EASC - DAYTON (DEPT./SECTION) ANALYTICAL REPORT (TYPE OF REPORT)

REPORT NO.: MDA-303

JOB/PROJECT NO.: 320.2190 and 482.2210

DATE: May 28, 1982

PERIOD COVERED:

TITLE: ANALYSIS OF TEXAS CITY SOIL SAMPLES FOR POLYCHLORINATED DIBENZO-P-DIOXINS (PCDDs) and POLYCHLORINATED BIPHENYLS (PCBs)

AUTHORS: Frederick D. Hileman
B. Mason Hughes

ABSTRACT: Three soil samples were received from Monsanto Texas City for the analysis of polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated biphenyls (PCBs). The results of these analyses are discussed in this report.

Approved by: Robert F. Ivory
Robert F. Ivory
Environmental Analytical Science Center Service Project Manager

Approval date: 5/28/82

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ACKNOWLEDGEMENT

The contributions of the following Dayton Laboratory employes are gratefully acknowledged:

Sample Preparation: D. Kirk

[redacted]

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MONS 022502

Three soil samples labeled TP-1 (Sample A); TP-5 (Sample B), and TP-5;SW (Sample C) were received for the analysis of polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated biphenyls (PCBs). These samples were assigned MRC Log number 1-82-05-07-01. Table 1 shows the results of the dioxin analyses, while Table 2 shows the results of the analyses for PCBs.

SAMPLE PREPARATION

The samples were prepared for analyses by weighing 5 grams of soil into a 250 mL bottle. Forty mL of petroleum ether was added to the bottle which was then sealed and shaken for 15 minutes. The petroleum ether was decanted out of the bottle into a centrifuge tube. Fifteen mL of methylene chloride was added to the bottle and then shaken for 5 minutes. The methylene chloride was also decanted into the centrifuge tube with the petroleum ether and these extracts were concentrated to 2 mL.

The extract was applied to a column containing 4 grams of silica gel which had been preeluted with 15 mL of hexane. This extract was then eluted through the column with 45 mL of methylene chloride and the eluant solvent exchanged to hexane concentrate and this concentrate was then placed onto a combination column containing silica gel, silica gel plus H_2SO_4 and silica gel plus KOH. This column was eluted with 45 mL of hexane and this eluant was concentrated to 1mL.

The final chromatographic separation was carried out on a 2.5 grams column of Woelm B alumina. The first elution consisted of 10 mL of 2% methylene chloride/hexane (v/v) which was concentrated to 1 mL and submitted for PCB analysis. A second elution of this column was made using 15 mL of 50% methylene chloride/hexane (v/v) to which was added 300 µL of dodecane keeper and the eluant was then concentrated to 300 µL and submitted for GC/MS analysis for PCDDs.

Quality control was carried out by spiking Sample B with both PCBs and PCDDs. This sample was selected for spiking since it appeared to be the most contaminated and would thus give a worst case situation for the recovery studies. A replicate and a method blank were also prepared and analyzed.

DISCUSSION OF PCDD ANALYSIS

The GC/MS analyses for PCDDs were carried out on a Hewlett Packard 5985B GC/MS system using a 30 meter SE-54 capillary column for the separation prior to mass spectral detection. The mass spectrometer was operated in the selected ion monitoring mode following m/z values characteristic of monochloro through octachlorodibenzo-p-dioxin.

No PCDDs were detected in any of the samples at the detection limits given in Table 1. Results from the quality control samples are also given. Note that the detection limit for dichlorodibenzo-p-dioxin were higher than the spike levels in both the low level and even the high level spike. Therefore no recovery information could be given for these dichlorodibenzo-p-dioxins. For the other PCDDs the recoveries are not particularly good but in light of how contaminated the samples were and the short time constraints, these results were considered adequate.

DISCUSSION OF PCB ANALYSIS

Three sample extracts, one replicate and two spiked samples were analyzed for Aroclor formulations. Eight PCB isomers were quantified and from these levels of Aroclors 1248, 1254, and 1262 were determined.

The low Aroclor 1248 and 1254 concentrations reported for Sample A may be due to instrument contamination from the the higher concentrations of these Aroclor formulations present in Samples B and C. The Sample B plus 0.16 µg/g can be treated as a replicate of Sample B, since the spiking level is ten times less the amount of Aroclor 1248 present in Sample B. Recovery of Aroclor 1248, added to Sample B at a concentration of 1.6 µg/g, was measured to be 75%. The levels of Aroclor 1254 and 1262 are only order of magnitude estimates, due to the large number of interfering compounds present.

TABLE 1. ANALYSIS OF SOIL SAMPLES FOR CHLORINATED DIBENZO-P-DOXINS

Sample ID	ng Dioxin/gm Soil (ppb)							
	Cl_1	Cl_2	Cl_3	Cl_4	Cl_5	Cl_6	Cl_7	Cl_8
Sample A	ND*	ND	ND	ND	ND	ND	ND	ND
Sample B	ND	ND	ND	ND	ND	ND	ND	ND
Sample B (rep)	ND	ND	ND	ND	ND	ND	ND	ND
Sample C	ND	ND	ND	ND	ND	ND	ND	ND
Blank	ND	ND	ND	ND	ND	ND	ND	ND
Method Detection Limit	2	50	2	2	2	2	2	2
Spike Level	4	3	4	3	3	3	4	3
B (spiked)	3	ND	2	3	2	2	3	2
Recovery %	75	-	50	100	67	67	75	67
Spike Level	35	34	38	28	33	32	35	28
B (spiked)	22	ND	15	20	15	20	23	12
Recovery %	63	-	39	71	45	63	66	43

*ND = none detected at the method detection limit

TABLE 2. LEVELS OF AROCLOR FORMULATIONS MEASURED IN SAMPLE EXTRACTS

Sample ID	Concentration[a] (µg/g) Aroclor 1248	Aroclor 1254	Aroclor 1262
Sample A	0.058[a,b]	0.028[b]	<0.02
Sample B	2.2	0.069	0.075
Sample B (rep)	2.2	0.047	0.045
B + 0.16 µg/g Aroclor 1248	2.2	0.062	0.061
Sample C	1.6	0.15	0.37
B + 1.6 µg/g Aroclor 1248	3.4[c]	0.10	0.09

[a] µg Aroclor formulation/g sample. Eight PCB isomers were used to identify the Aroclor formulation and estimate the relative amounts.

[b] Possible instrument contamination from analysis of Samples B & C.

[c] This value for Aroclor 1248 concentration represents a recovery of 75% of Aroclor 1248 added to Sample B.