Presence of Priority Pollutants in Sewage and their Removal in Sewage Treatment Plants

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EXECUTIVE SUMMARY OF THE PRESENT STUDY

The present study, "Presence of Priority Pollutants in Sewage," developed methodology to monitor the removal of organics and heavy metals in fullscale publicly-owned wastewater treatment works (POTW). The plants sampled as of now are: Seattle (Renton), Atlanta (Clayton), Oakland (EBMUD), Modesto, Peoria, Rockford, and Kenosha. The plants were elected based on the percentage of the population working in different industrial categories, and percentage of industrial waste discharged to the POTW.

The analyses to date, covering the first three plants, found 67 priority pollutants, i.e. 37 base/neutral organics, 21 volatile organics, and 9 phenolics. Compounds found in the present study that have not been detected in the parallel 40 city survey (city A,B) are: 1,1,2,2-tetrachloroethane, bis(2-chloroethyl)-ether, bis(2-chloroisopropyl)ether, n-nitro-di-n-propylamine, nitrobenzene, dipheylhydrazine, n-nitrosodiphenylamine, benzo(B)-fluorathene, and benzo(K)fluoranthene. While individual removals show a substantial variation, some tentative conclusions can already be made.

The volatile compounds found in highest concentration to date were: 1,1,1-trichloroethane, tetrachloroethene, and ethylbenzene. Moderate removals were observed for chlorinated C_1 and C_2 compounds during primary and secondary treatment for dichloromethane (35%), trichloromethane (16%), trichloroethene (23%), 1,1,1-trichloroethane (71%), and tetrachloroethene (56%). Higher removals were noted for the volatile aromatics, such as benzene (71%), methylbenzene (79%), chlorobenzene (96%), and ethylbenzene (97%). The greater removals for the volatile benzenes seem to be partially due to a greater absorption onto the solids, as only these volatile compounds accumulate in the digested sludge. The removal of the extractable chlorinated benzenes thereafter declined with further increasing molecular weights for 1,3-dichlorobenzene (70%), 1,4-dichlorobenzene (85%), 1,2-dichlorobenzene (50%), and 1,2,4-dichlorobenzene (43%). High removals were noted for nitrobenzene (97%) and 2,6-dinitrotoluene (greater than 99%). The removal of the polynuclear aromatics increases with increasing molecular weight, or with an increasing GC retention time for napthalene (82%), acenaphthylene (84%), and fluorene (95%), while it is generally greater than 99%% for higher molecular weight PAH's. The increased removal is generally reflected by increased presence of the PAH's in the digested sludge. Especially high sludge values have been noted for fluorene, pyrene, phenanthene, and fluoranthene. High phthalate removals are observed for the lower molecular weight homologs, such as dimethylphthalate (99%) and diethylphthalate (93%), which is probably due to biologicl degradation as they do not accumulate in the sludge. The higher molecular weight phthalates, such as butylbenzylphthalate and bis(2-ethylhexyl)phthalate accumulate in

the digested sludge, presumably due to their higher absorptive capacity.

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SECTION 1

INTRODUCTION

The usage and subsequent discharge of water carries the potential that trace pollutants enter the environment. Currently, the projected industrial water usage for 1980 is 75,000 million gallons per day (mgd); the municipal water use is expected to reach 33,600 mgd (Steele, 1968). While past efforts have primarily focussed on the removal of general organics, as measured with the biochemical or chemical oxygen demand (BOD and COD) test, current concern exists with regard to removal of organic and inorganic trace pollutants that are potentially toxic to humans.

Several recent studies have noted the presence of trace organics in sewage and secondary effluent (Table 1-1) indicating that biological treatment processes do not remove all trace pollutants. It was further noted that chlorination of secondary effluent resulted in formation of potentially hazardous organics (Glaze et. al., 1976). The above data indicate that classes of potentially hazardous organics, such as phenols, volatile chlorinated hydrocarbons, pesticides, and polynuclear aromatic hydrocarbons, are likely to be present in the discharge of sewage treatment plants. The toxic pollutant section, 307a, of Public Law 95-500, as redefined in the Consent Decree, specifically limits the discharge of the above mentioned classes of "priority" pollutants (Ward, 1977). While previous studies have analyzed for one or two classes of toxic pollutants, none encompassed a comprehensive range of the defined priority pollutants. The present study will conduct such a comprehensive study by determining volatile chlorinated hydrocarbons, Cl/Alkyl/N/OH-benzenes, biphenyls, alkanes, ethers, nitrosamines, polynuclear aromatic hydrocarbons, pesticides, and heavy metals in sewage, primary, and secondary effluent at 25 large sewage treatment plants located in different regions of the United States.

Since many toxic trace organics are of a hydrophobic nature, they adsorb readily onto bacterial solids. Substantial concentrations are therefore found in digested sludge. Since the disposal of sludges containing hazardous substances is also regulated by EPA, the current study will also determine the presence of the priority pollutants in waste activated sludge and anaerobic digester sludge.

During the initial year of effort, 9 months of method development have produced an integrated scheme for the analysis of priority compounds, superior sensitivity and quality assurance are indicated from developmental results. These methods have been tested against real samples comprising three sewage treatment plants: Renton (Seattle), Washington; Atlanta, Georgia; and Oakland, California. This report describes the method develop-

Table 1-1 Detection of trace organics in sewage treatment plants.

Author	Sample	Organics
Reichert et al(1971)	Sewage	Polynuclear aromatic hydrocarbons
Schmidt et al (1971)	Sewage	PCB
Timofeeva & Stom (1976)	Sewage	phenol, catechol, quinol
Lawrence & Tosine (1976)	Sewage	PCB
Methews (1976)	Sewage	Cyclohexarol & other solvents
Jensen & Peterson (1971)	Sewage	2,5 di(benzoxazole-2-yl) thiophene, optic brightener
Chian & DeWalle (1977)	Secondary effluent	55 Compounds, 17 priority pollutants
Glaze & Henderson (1975)	Secondary effluent	30 chlorinated organics
Glaze et al (1976)	Secondary effluent	39 chlorinated organics
Jolley et al (1976)	Secondary effluent	60 chlorinated aromatic compounds
Manka et al (1974)	Secondary effluent	Aromatic hydrocarbons
Painter (1973)	Secondary effluent	Pesticides & phthalates
Burlingame et al (1976)	Secondary effluent	28 organics: chlorinated benzenes, phenyl ethers, phthalates
Andrade et al (1975)	Anaerobic dig. sludge	Mirex & metabolites
Mattsson et al (1975)	Anaerobic dig. sludge	Penta bromotoluene, fire retardant
Ayanaba & Alexander (1974)	Anaerobic dig. sludge	nitrosamines

ment phase and initial results from the sampling phase of this project.

SECTION 2

PURGEABLES

The research has focussed on the evaluation of three techniques, i.e., the headspace displacement flask as shown in Figure 2-1a (Kuo et al, 1977), the stripping bubbler (Bellar and Lichtenberg, 1974) as shown in Figure 2-1b, and the purge and trap sampler combined with cryogenic trapping and capillary column chromatography.

2-1 Evaluation of Headspace Displacement Flask

The headspace displacement flask (HDF) was selected for the initial evaluations using the conditions, listed in Table 2-1. A major advantage of the method is that the purging gas does not have to pass through the liquid, which circumvents potential foaming of the 36 compounds listed under the purgeable category, three of them (acrolein, acrylonitrile, and methylene chloride) are merged with the solvent peak methanol used for dissolution of spiked compounds, six of them (chloroethane, dichlorofluoromethane, methyl bromide, methyl chloride, trichlorofluoromethane, and vinyl chloride) are gases at room temperature, while four of them (1,2-dichlorobenzene, 1,3-dichlorobenzene, 1,4-dichlorobenzene, and hexachloroethane) have too long detention times for packed columns.

The percentage recovery from purgeable-free water of 18 volatile organics spiked at 20 ppb ranged from 56% for carbon tetrachloride to 100% for tetrachloroethylene and dichloropropane (Table 2-2). The standard deviation ranged from 3% for trichloroethylene to 23% for 1,1,1-trichloroethane. The percentage recovery tended to decrease slightly with increasing concentration, as shown in Figure 2-2. This is likely due to some overloading of the Tenax GC trap.

The effect of matrix organics was tested in sewage and digested sludge of the sewage treatment plant in Urbana, Illinois. The results in Table 2-3 indicate that the recoveries in sewage and sludge are lower than those obtained in distilled water, due to organics present in the sample matrix that compete for adsorption sites in the Tenax. The recoveries after 16 hour equilibration are considerably lower for about six of the 17 spiked compounds, especially those with aromatic rings, due to extensive adsorption onto the bacterial solids.

The matrix effect is most clearly demonstrated in Figures 2-3 and 2-4, where increasing amounts of sewage and digested sludge decrease the recovery of the individual organics. Most organics tend to follow a linear function on a log-normal graph. This relationship can be used in a serial dilution method to determine the recoveries at an infinite dilution, which protocol

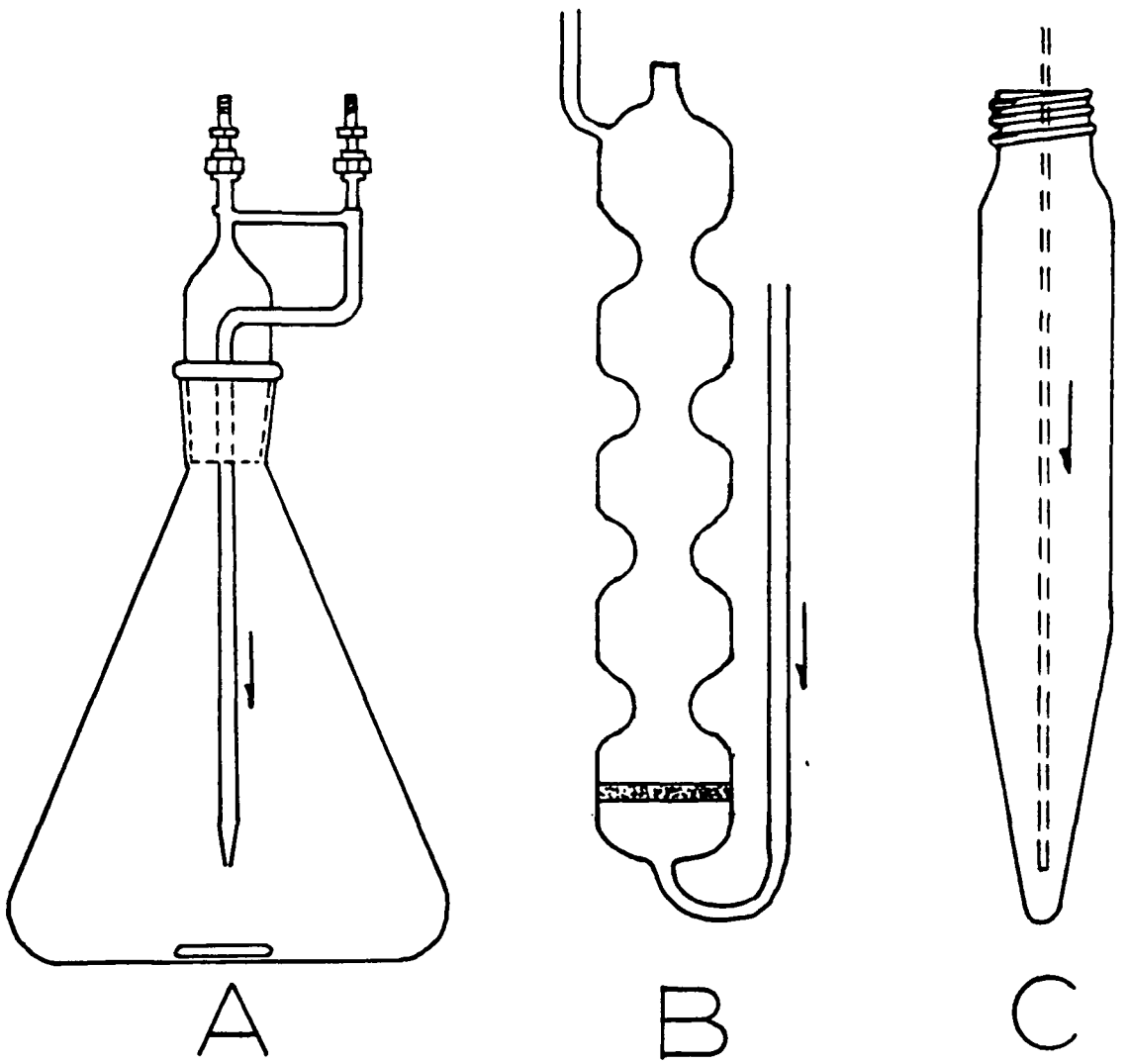


Figure 2-1 Different Stripping Vessels Evaluated During the Study; (A) Headspace Displacement Flask of 1000 ml, (B) Stripping Bubbler of 10 ml, (C) Purging Tube of the Purge and Trap Module

Table 2-1. Instrumental conditions during purge and trap method to determine low molecular weight volatile organics.

100 ml sample in 100 ml volumetric flask sealed with wetted ground glass stopper

↓

transfer to 1& stripping flask (Kuo and Chian) spike with standards and stripped for 20 minutes, at 60°C with 200 ml/min He purified with molecular seive and extra tenax trap

transfer 5 ml to 10 ml to the bubbler, spike with standards and bubble gas through sample at room temp. for 12 minutes at 40 ml/min at 40 ml/min (Bellar and Lichtenberg)

↓

organics adsorb on Tenax GC 60/80 mesh in 7", 1/4" OD stainless steel tube, initially baked out at 250°C with He flushing

↓

trap connected to GC, flushed with He for 2 minutes; heating of adsorbant column for 2 minutes at 200°C

↓

organics flushed into GC column (12 Ft. 2 mm ID with 0.2% Carbowax 1500 on Carbopack C 60/80 mesh) for 4 minutes at 30 ml/min

↓

column temperature from 30°C to 200°C at 8°/min

↓

organics introduced in MS through jet separator

↓

MS with 70eV electron energy; 500 ua emission current; 6V ion energy; -100V lens voltage, 8V extractor voltage; 20-300 amn mass range, 17 msec integration/amn; MS calibrated with decal fluorotriphenyl-phosphine

Table 2-2 Recovery of 18 compounds from purgeable-free water using headspace displacement flask spiked at 20 ppb as run in triplicate

Compound	Relative Retention Time	Percentage Recovery	Standard Deviation
1,1-DICHLOROETHANE	0.85	85	14
1,2-TRANSDICHLOROETHYLENE	0.93	99	N/A
CHLOROFORM	1.00	91	17
1,2-DICHLOROETHANE	1.10	89	4
CARBON TETRACHLORIDE	1.25	56	N/A
1,1,1-TRICHLOROETHANE	1.29	64	23
DICHLOROBROMOMETHANE	1.31	97	N/A
TRICHLOROETHYLENE	1.57	89	3
BENZENE	1.63	91	5
1,2-DICHLOROPROPANE	1.68	100	N/A
DIBROMOCHLOROMETHANE	1.89	100	N/A
TETRACHLOROETHYLENE	2.13	100	N/A
2-BROMO 1-CHLOROPROPANE	2.16	88	6
BROMOFORM	2.32	95	17
CHLOROBENZENE	2.35	94	10
ETHYL BENZENE	2.54	95	21
1,1,2,2-TETRACHLOROETHANE	2.71	87	12
TOLUENE	3.04	100	N/A

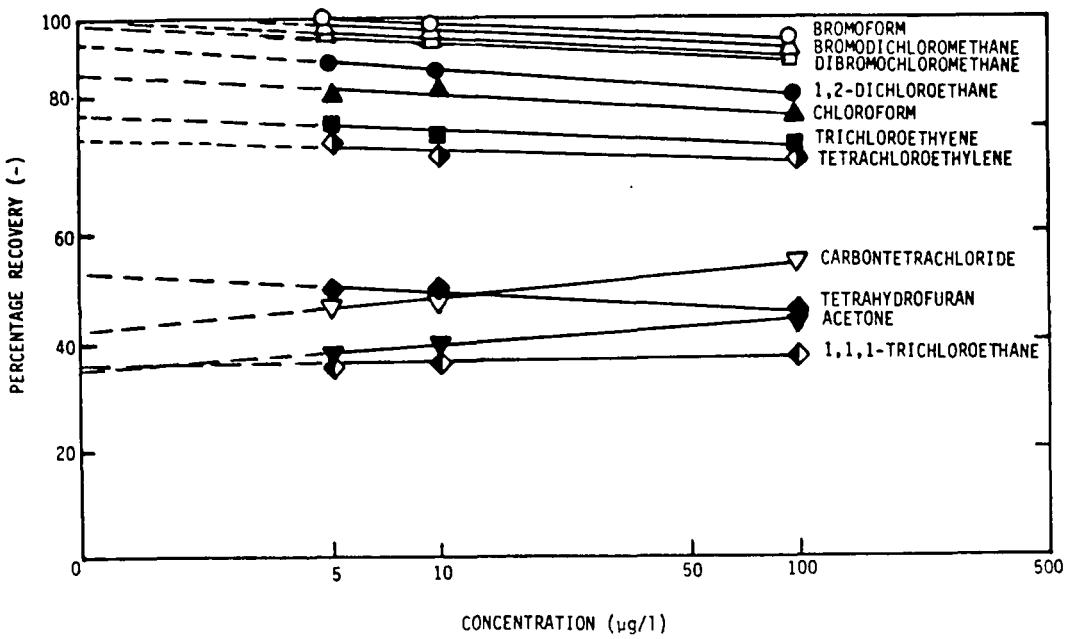


Figure 2-2 Percentage Recovery of Volatile Compounds Spiked at Different Concentrations in Distilled Water.

Table 2-3. Effect of Matrix Organics on Recovery of Volatile Organics Spiked at 20 ppb using the Headspace Displacement Flask

Compound	Relative Retention Time	Distilled Water	Raw Sewage (1)	3% Raw Sludge (2)	1.5% Digested Sludge (2)	Equilibrated Digested Sludge (16 hr) (2)
1,1-DICHLOROETHANE	0.85	85		97	93.1	92
CHLOROFORM	1.00	100	100	67	89.0	79
1,2-DICHLOROETHANE	1.10	89	88	90	93.4	93
CARBON TETRACHLORIDE	1.25	56		87.4	89.7	65*
1,1,1TRICHLOROETHANE	1.29	64	66	55	54.2	39*
DICHLOROBROMOMETHANE	1.31	97	78	95	95.4	84
TRICHLOROETHYLENE	1.57	89		98	86.7	80
BENZENE	1.63	91	73	94	87.1	66*
1,2-TRICHLOROETHANE	1.68	100	98	94	92.1	90
DIBROMOCHLOROMETHANE	1.89	100	89	95	98	91
TETRACHLOROETHYLENE	2.13	100	100	91	90.3	88
2-BROMO-1-CHLOROPROPANE	2.16	88	78	100	98.3	95
BROFORM	2.32	95	88	100	97	89
CHLOROBENZENE	2.35	94	75	99	89.6	61*
ETHYLBENZENE	2.54	95	87	100	89.5	43*
1,1,2,2-TETRACHLOROETHANE	2.71	87	78	100	100	93
TOLUENE	3.04	100	100	100	96.5	70*
1,1,2-TRICHLOROETHAM		100	100			

*Decreased recovery after equilibration

(1) Sewage diluted 25 ml to 100 ml

(2) Sludge diluted 10 ml to 100 ml

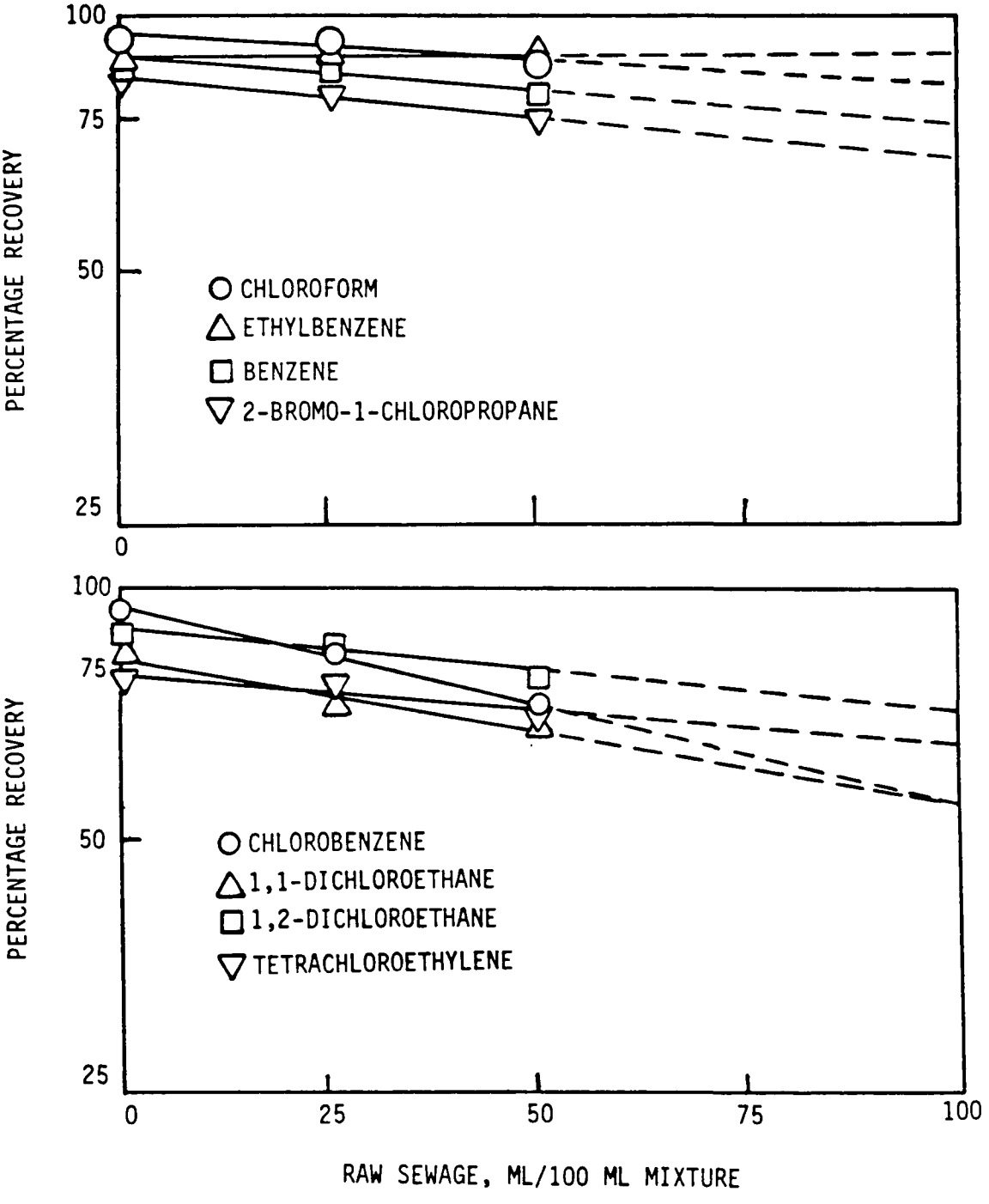


Figure 2-3 Recovery of Volatile Compounds Added to Diluted Sewage Samples at a Concentration Level of 20 ppb.

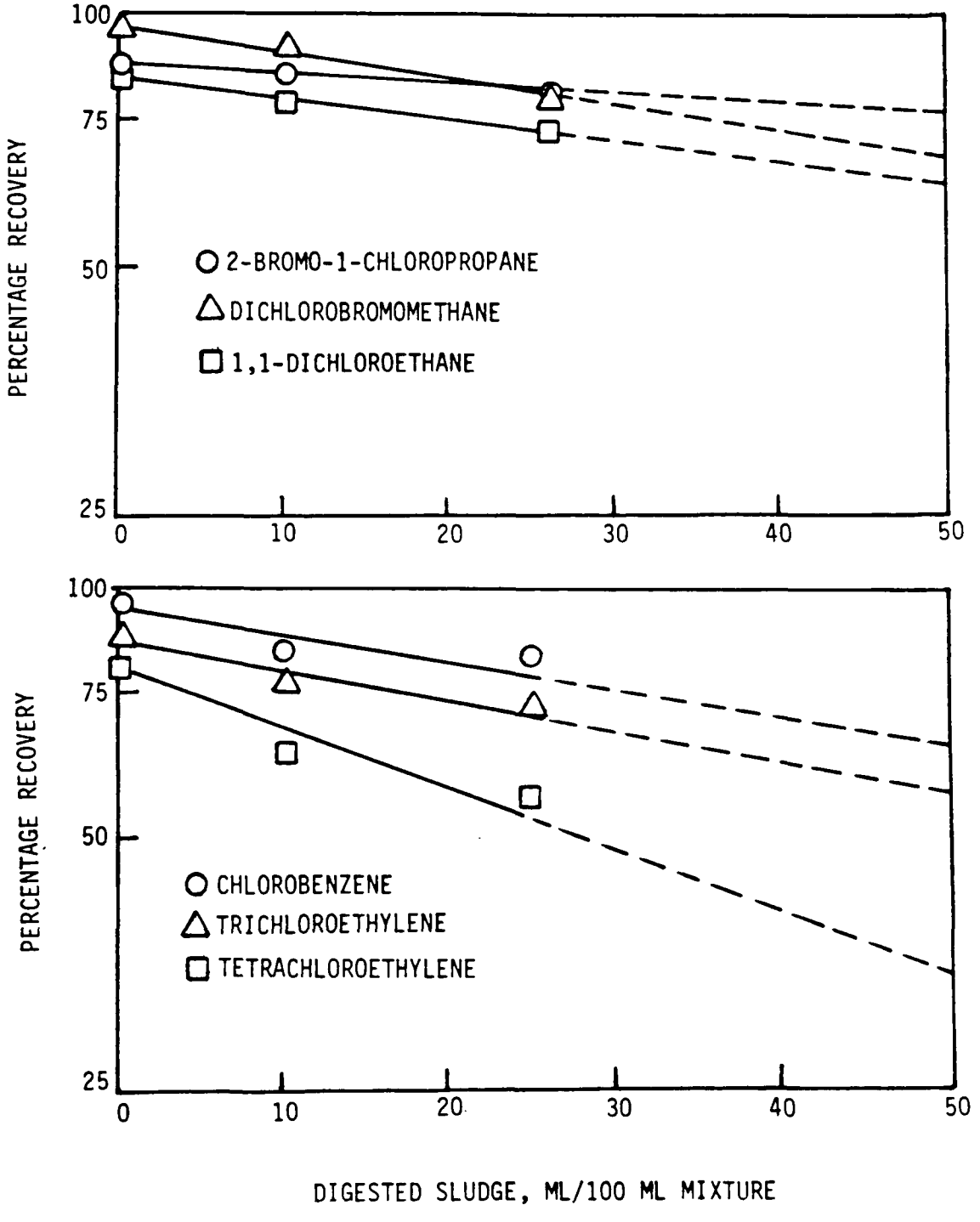


Figure 2-4 Recovery of Volatile Compounds Added to Diluted Digested Sludge Samples at a Concentration Level of 20 ppb.

is given in Table 2-4.

2-2 Evaluation of the Stripping Bubbler

In subsequent runs, the 10 ml stripping bubbler was evaluated. The vessel holds 10 ml and inert gas is sparged through the fritted glass bottom. The recoveries and standard deviation shown in Table 2-5 indicate that higher deviations are experienced for the first and last eluting compounds, while lower deviations are noted for compounds eluting in the middle. Similar to the HDF experiments, it was noted that the recoveries decreased slightly with increasing levels of the spiked compound, as shown in Figures 2-5 and 2-6. The decrease in recovery for ethylbenzene and tetrachloroethylene tended to be greater than for other compounds. A typical gas chromatogram of Atlanta sewage is shown in Figure 2-7.

2-3 VOA Determination by Glass Capillary GC

The principal shortcomings of the packed Column GC/FID and purge and trap method are: 1) poor detection of compounds more volatile than methylene chloride, 2) multiple experiments being required to determine matrix effects, and 3) potential false identification of GC peaks, especially from high background samples. An alternative method, utilizing glass capillary GC/MS has been devised at the University of Washington. For this purpose, a Hewlett-Packard 7675A Purge and Trap module has been interfaced with the GC/MS/DS system. The interface consists of a swagelok connectoer that has been tapped and welded to a steel tube to serve as a splitter vent. The column end is passed through this fitting and inserted approximately 1/4" into the steel line emerging from the purge and trap assembly. The conditions found to be optimum in operating the analysis successfully are presented in Table 2-6. The system is shown schematically in Figure 2-8.

In order to obtain acceptable chromatographic separation of the sample, on-column cryogenic trapping is essential. For this purpose, approximately 10 cm of the first coil of the column are inserted in metal jackets of various design. The first model consisted of a notched copper tube that has been prebent to conform to the column shape. Liquid nitrogen is introduced from the exit side of the cryotrap and essentially fills the trap and bathes the column during desorption from the Tenax trap. In GC/MS analysis where MS acquisition is routinely begun with the initiation of desorption and cold trapping, it is observed that allowing sufficient time to chill the transfer lines and cold trap itself (5 - 10 minutes) results in essentially perfect cold trapping so long as the sample loading is reasonable (no more than micrograms of the sample). The first peak observed to chromatograph after cessation of cold trapping is carbon dioxide, followed rapidly by other gaseous and highly volatile components

Table 2-4. Proposed Protocol for Measuring Purgeables in Raw Sewage with HDF (Headspace Displacement Flask)

1. Establish the percent recovery of the purgeable priority pollutants at a concentration of 50 ppb (v/v) in purgeable-free water under the experimental conditions available at individual laboratory.

2. Measure the level of these compounds at two to three dilutions (use 10, 25, and 50% sewage addition for 100-ml sample or 25, 50, 100% for 10-ml sample as with the Bellar and Lichtenberg's apparatus).

3. Normalize all concentration determined based on the highest dilution employed and plot on a semilog graph paper with the abscissa in logarithmic scale for concentration.

4. Draw a least square line through these points and extrapolate to zero sewage addition. The value obtained represents the concentration of the given compounds to be determined in purgeable-free water.

5. Divide the above value at zero sewage addition by the percent recovery of the corresponding compounds determined at 50 ppb (v/v). The actual value can be obtained by multiplying the value by the number of the highest dilution made with the run.

6. Use the EPA internal standard, 2-bromo-1-chloropropane, in all quantitation studies to minimize variations in FID responses and injection sample size.

7. Concentrations are to be reported on a weight basis by multiplying the above values by the density of each compound.

Table 2-5. Reproducibility of Bellar and Lichtenberg System spiked with eleven purgeable organics 100µg/l in raw sewage and digested sludge based on triplicate. Sewage and sludge were obtained from Atlanta, Georgia, Clayton Plant.

		Raw Sewage (1)		Digested Sludge (2)	
Compound	Relative Retention Time	Recovery (%)	Standard Deviation (%)	Recovery (%)	Standard Deviation (%)
1,1-DICHLOROETHANE	0.85	73	6.6	81	4.8
CHLOROFORM	1.00	54	3.0	75	9.5
1,2-DICHLOROETHANE	1.10	76	7.2	83	3.7
CARBONTETRACHLORIDE	1.25	65	1.5	78	5.3
DICHLOROBROMOMETHANE	1.31	67	4.8	83	4.8
BENZENE	1.63	78	7.8	83	4.4
TETRACHLOROETHYLENE	2.13	65	7.7	85	16.3
2-BROMO1-CHLOROPROPANE	2.16	82	8.9	83	6.5
CHLOROBENZENE	2.35	68	10.1	75	13.4
ETHYLBENZENE	2.54	72	17.1	63	12.8

(1) 5 ml sewage diluted to 10ml

(2) 2.5 ml sludge diluted to 10 ml

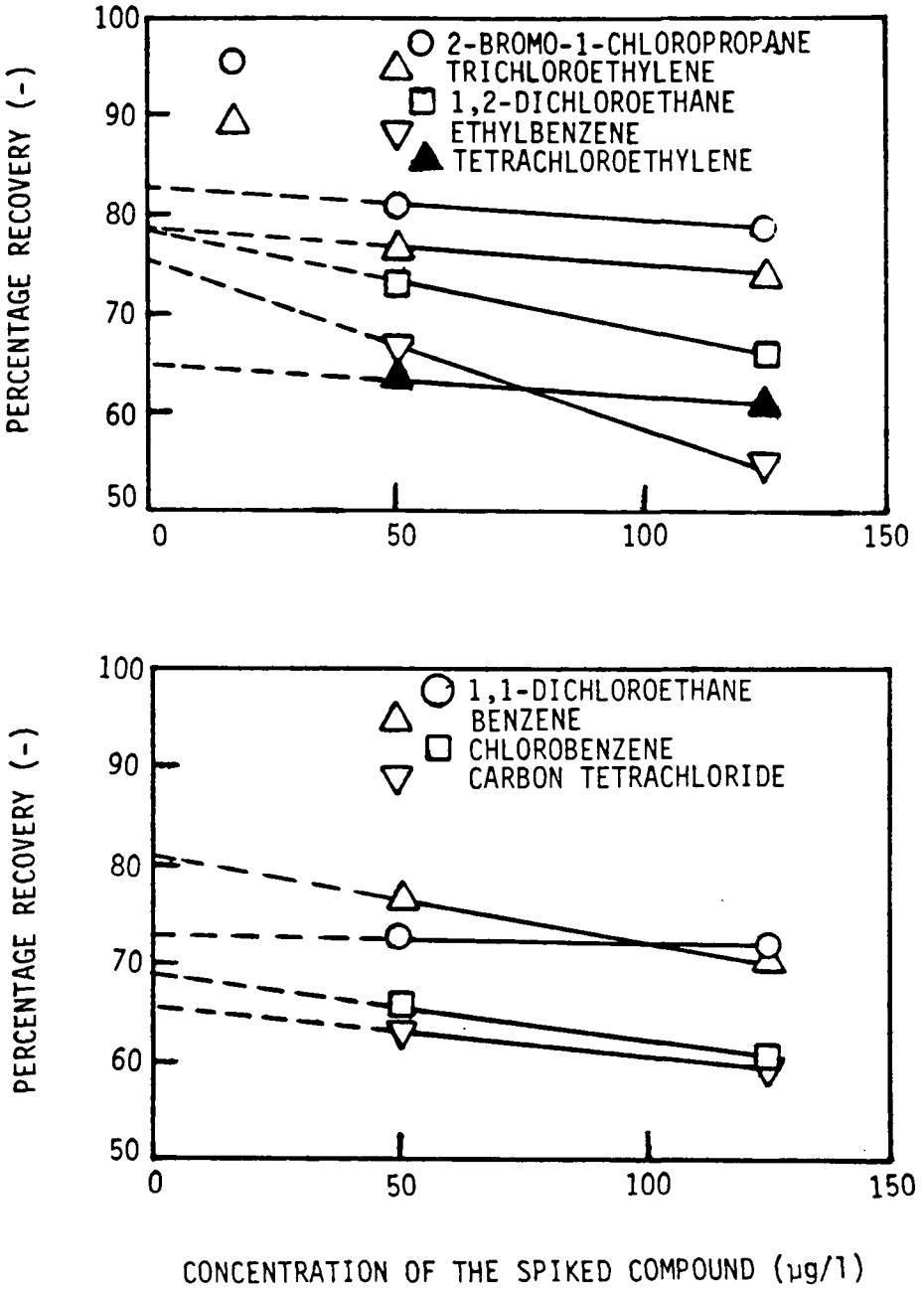


Figure 2-5 Recovery of Volatile Organics from a 50% Diluted Champaign Sewage Spiked at Different Concentration Levels in a Stripping Bubbler.

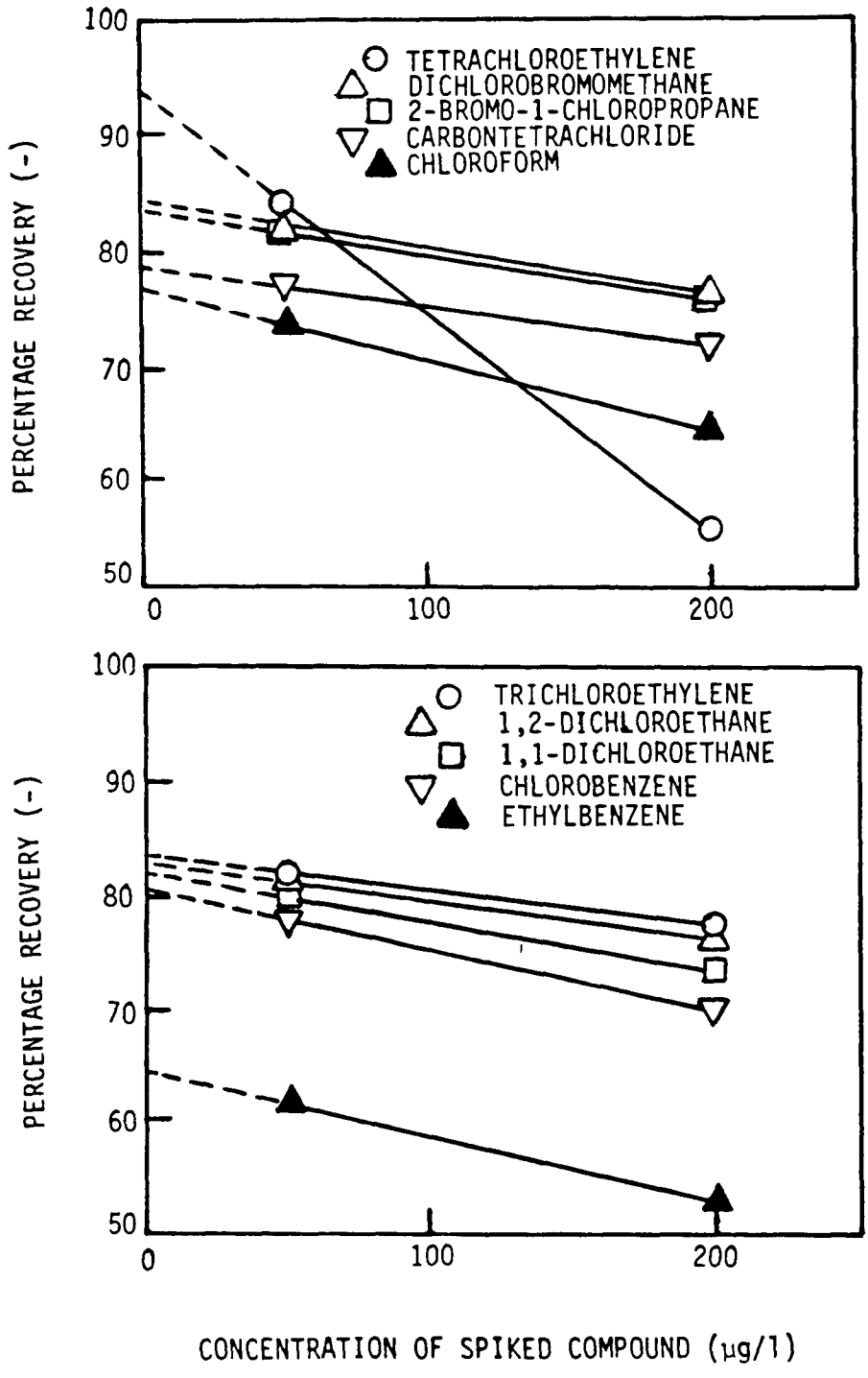


Figure 2-6 Recovery of Volatile Organics from a 25% Diluted Champaign Anaerobic Digested Sludge Spiked at Different Concentration Levels in a Stripping Bubbler.

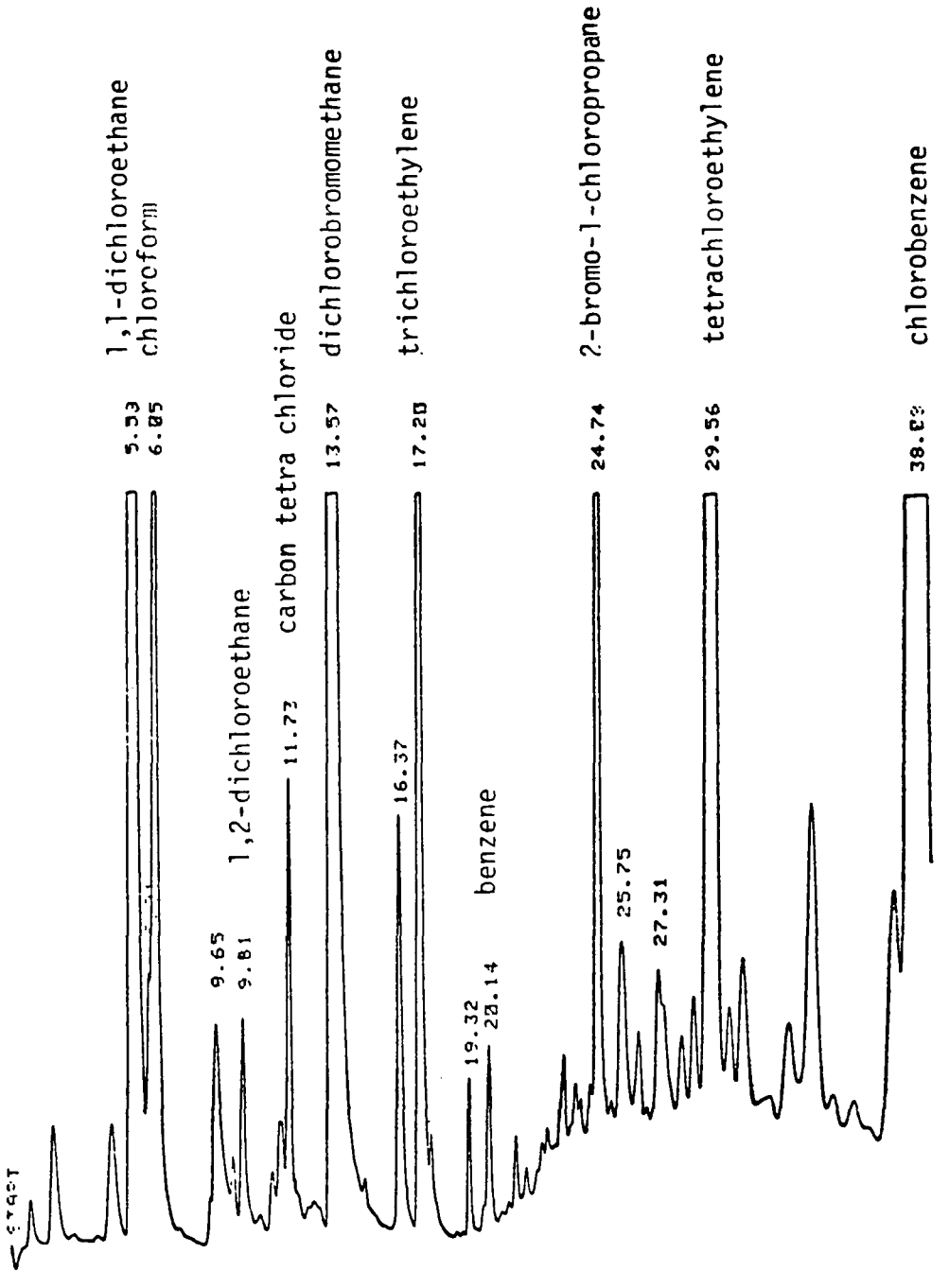


Figure 2-7 Chromatogram of 25% Diluted Atlanta Sewage Purged in the B-L Bubbler and Separated on a 0.2% Carbowax GC Column

Table 2-6 GC and GC/MS Conditions for the VOA Analysis

Purge time	15 minutes
Purge rate	20 ml/min
Purge temperature	ambient
Purge Gas	organic free nitrogen purified through catalyst
Desorption time	10 minutes
Desorption temperature	200°C
Desorption flow rate	1 ml/min
Cryo temperature	liquid nitrogen temperature (-198°C)
Trap condition time	10 minutes
GC column	30 m SE-54
GC program	0-10 min cryotrap 10-14 min isothermal 14-18 min 4°/min 18-22 min 8°/min
MS conditions	34-443 mass range 0.45 sec scan time 0.5 sec scan rate

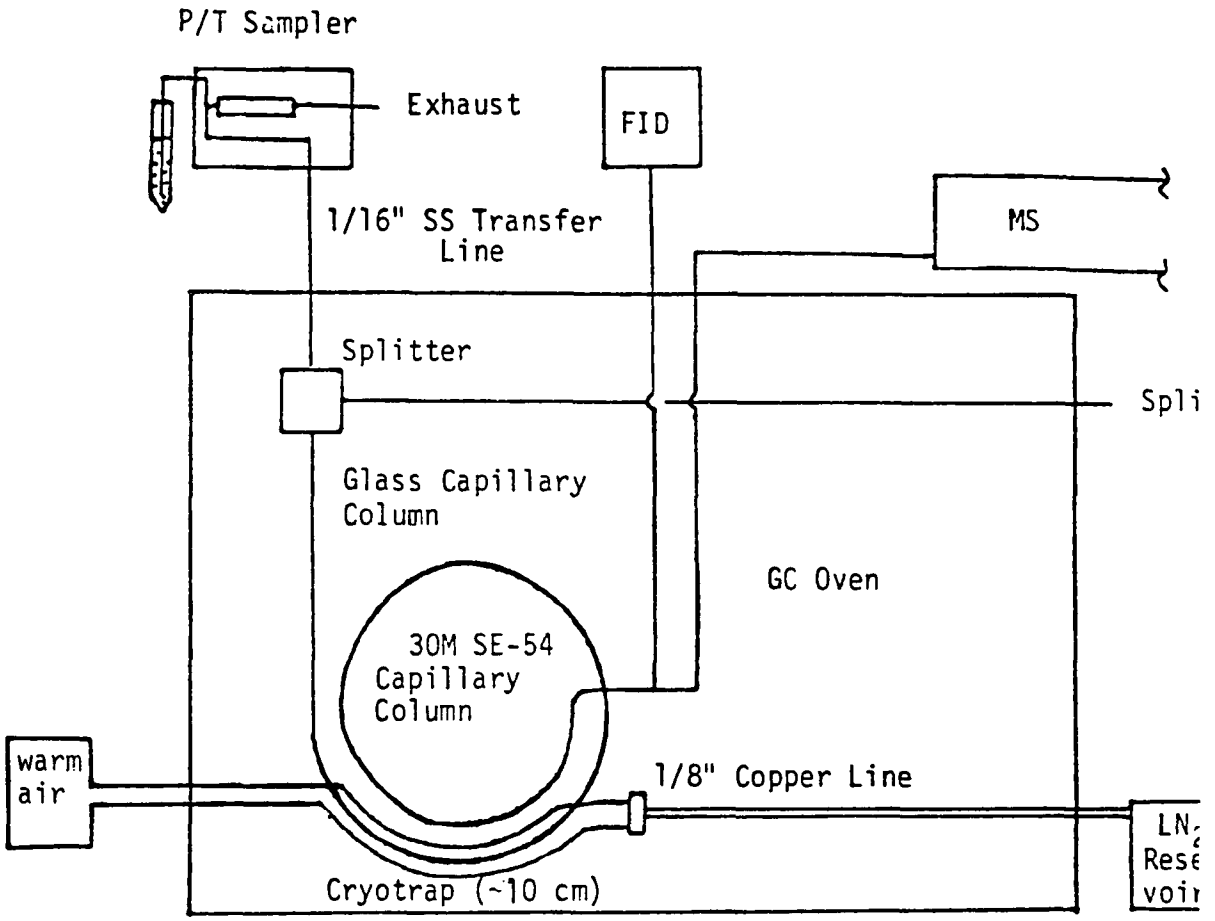


Figure 2-8 Schematic of VOA Analysis Instrumentation Using Cryogenic Trapping and Capillary GC/MS Separation.

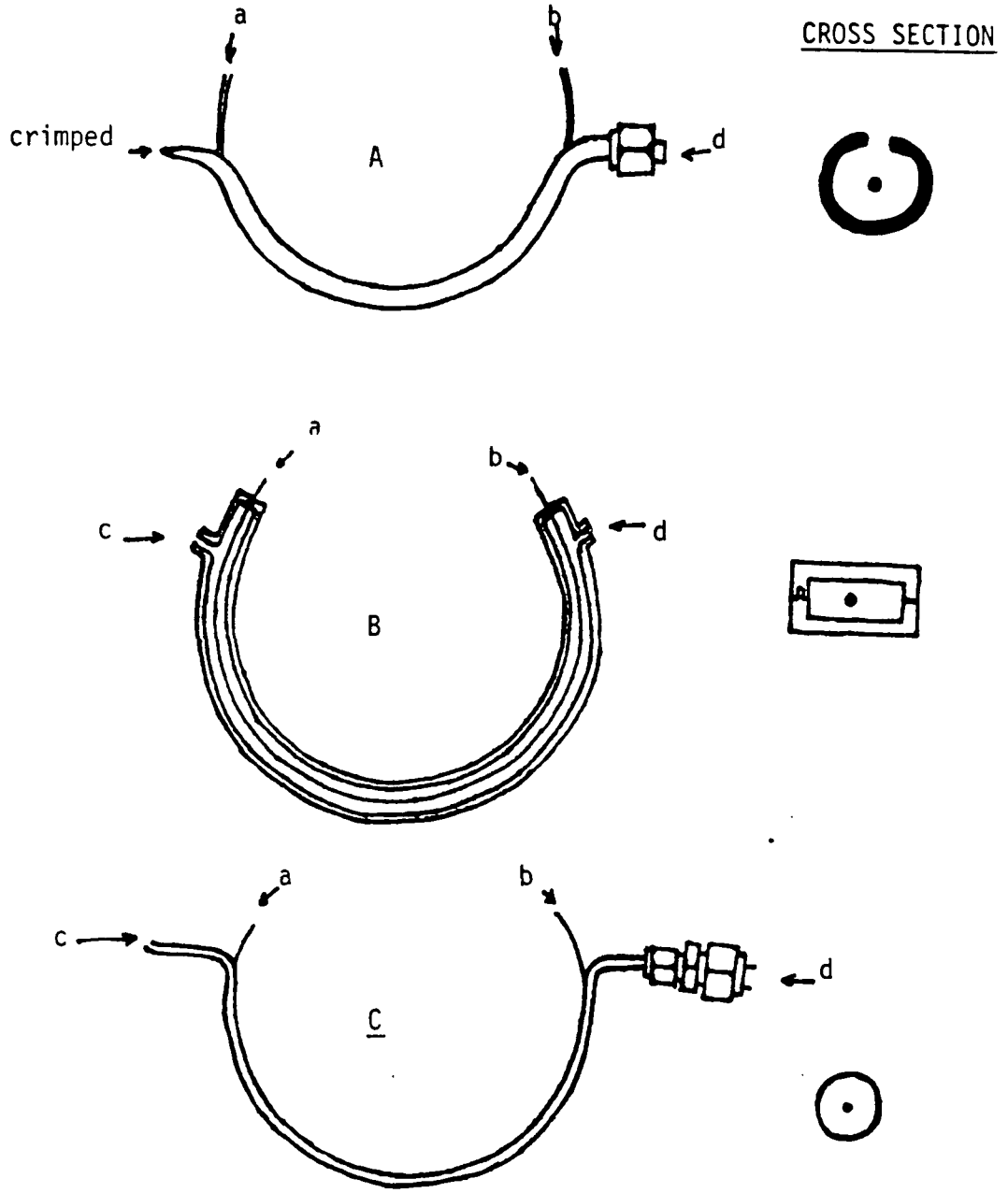
such as pentane, halomethanes, vinyl chloride, etc.

The cryogenic trapping thermally focusses the sample during desorption from the Tenax trap. It is desirable to warm the cold trap rapidly following desorption to improve chromatographic banding. The initial notched-tube trap was deficient in that it warmed too slowly. Two additional designs have shown much improved chromatographic performance -- a two piece design and a steel tube, shown in Figure 2-9. Both of these permit warm air to be blown over the cryotrap in a direction opposite to the coolant flow. After some experimentation, the configuration found to be most desirable is to have the coolant enter the trap near the column exit, and the warm air enter near the GC column entrance. Other configurations produced split peaks and peak spreading. Subsequent conversations with the Hewlett-Packard applications scientist working on this same approach have provided independent confirmation of these observations. As a matter of convenience, the warm air is run from a tank through a coil of copper placed inside the transfer oven to the MS, kept at 240°. This provides 40-80°C air for warm up.

Initial chromatography of purgables was carried out with the Tenax-Chromosorb layered trap recommended by EPA in the BAT protocols. Unfortunately, continued operation at temperatures at or below 200°C results in significant trap background from apparent thermolysis of the polystyrene Chromosorb. While these components do not appear to actually obscure the priority compounds to a large degree, the implications with regard to stable and reproducible behavior of the trap are not encouraging. Operating with Tenax alone provides a very clean background analysis, at the expense of poor trapping for compounds more volatile than methylene chloride. For the present, it appears that no fully satisfactory trapping medium has been found.

For sludge or foamy samples, agitation of the sampling vessel with an ultrasonic bath is recommended. This disperses sparging gas efficiently and provides better mixing within the sample. In extreme cases, a surface sparging technique recently reported by Hewlett-Packard could be employed.

Recovery of spiked compounds in a variety of sample types are reported in Table 2-7. Using this method of capillary chromatography, increased sensitivity and lower detection limits relative to packed column analyses are observed. Figure 2-10 shows the chromatogram of a 0.4 PPb standard purged from water and trapped on Tenax plus Chromosorb 102. The sample size was 5 ml (10% of the recommended upper limit for the purge and trap sampler) and the attenuation was "8".



a FROM CAPILLARY INJECTOR
b TO REST OF GC COLUMN, MASS SPECTROMETER
c WARM AIR INLET
d COOLANT INLET (FROM LIQUID NITROGEN RESERVOIR)

Figure 2-9 Cryotrap Designs : A) Notched Tube; B) Aluminum Sandwich; C) Stainless Steel Tube

Table 2-7. Recovery of volatile organics from different matrices using the cryogenic trapping technique and the P/T module.

	PERCENTAGE DETECTION OF SPIKED COMP.					SEDIMENT		SEWAGE		SLUDGE	
	SEDIMENT	SEDIMENT	SEWAGE	SEWAGE	SLUDGE	PERCENTAGE RECOV.	STANDARD DEVIATION	PERCENTAGE RECOV.	STANDARD DEVIATION	PERCENTAGE RECOV.	STANDARD DEVIATION
LEVEL OF SPIKING (µG/ L)	25	10	25	10	10						
NUMBER OF DETERMINATIONS	6	13	5	5	2						
2,2-DICHLOROPROPANE	100	100	100	100	100	QS**		QS		QS	
CHLOROETHANE	100	92	100	60	50	286%	420	159%	152	12%	NA
ACRYLONITRILE	50	62	60	100	50	129	79	153	129	539	NA
CHLOROFORM	100	100	100	100	100	108	14	117	21	103	19
TRICHLOROETHANE	100	100	100	80	100	104	6	103	5	98	11
BENZENE	83	100	100	100	100	98	12	103	20	92	18
TOLUENE	*83	100	100	100	100	QS		QS		QS	
BROMOFORM	*83	100	100	100	100	QS		QS		QS	
Cl_2-BENZENE	67	77	75	80	50	84	22	150	92	563	NA

**Quantitation Std.

*data searching error

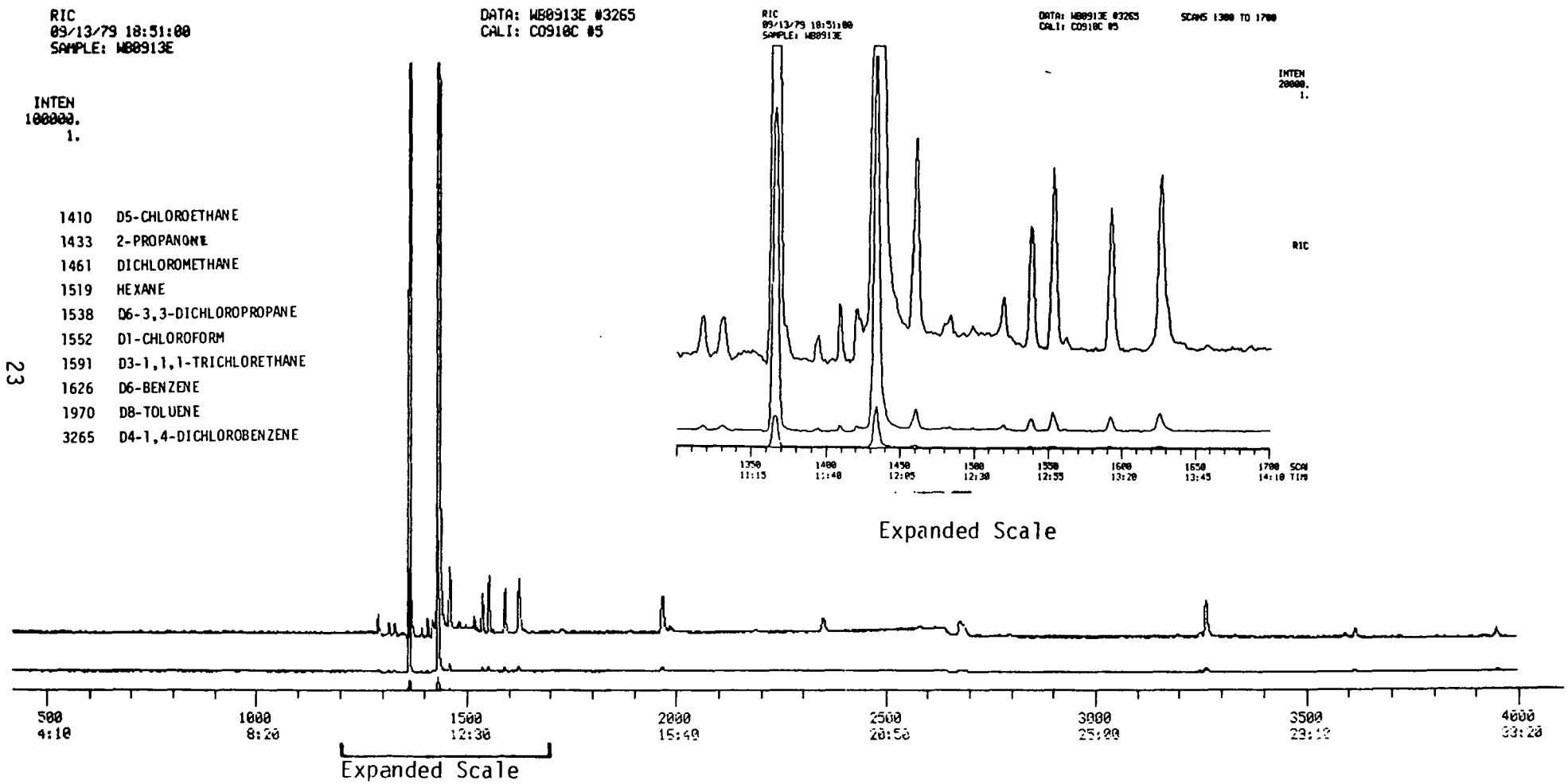


Figure 2-10 Reconstructed Ion Current of a 0.40 ppb Volatile Organic Mixture in Distilled Water.

In addition to increased sensitivity, capillary analysis has permitted the detection of more volatile compounds than are typically resolved in packed column VOA work. Figure 2-11 shows the purge and trap GC/MS analysis of a standard containing all of the priority purgables, except dichlorodifluoremethane, acrolein, and acrylonitrile. In only a few instances are priority compounds found to co-elute and these would be readily quantified in the MS/DS analysis regardless of resolution. Initial analyses were performed using a 30-meter SE-54 capillary column; the initial temperature for the analysis has varied between -10° and 30° C. Although these temperatures are below the nominal operating range for this column, acceptable performance has been observed.

Analysis of sewage effluent samples to determine the degree of resolution is illustrated in Figure 2-12. For the most part, the chromatography is qualitatively improved over the packed column results reported in the previous quarterly reports.

Remaining areas of refinement for the capillary VOA technique include: improved Tenax trapping to retain the most volatile priority compounds efficiently, development of alternative methods for sparging foamy samples, and improvement of techniques to analyze VOAs from sludges. Plates I, II, and III show the instrumental configuration.

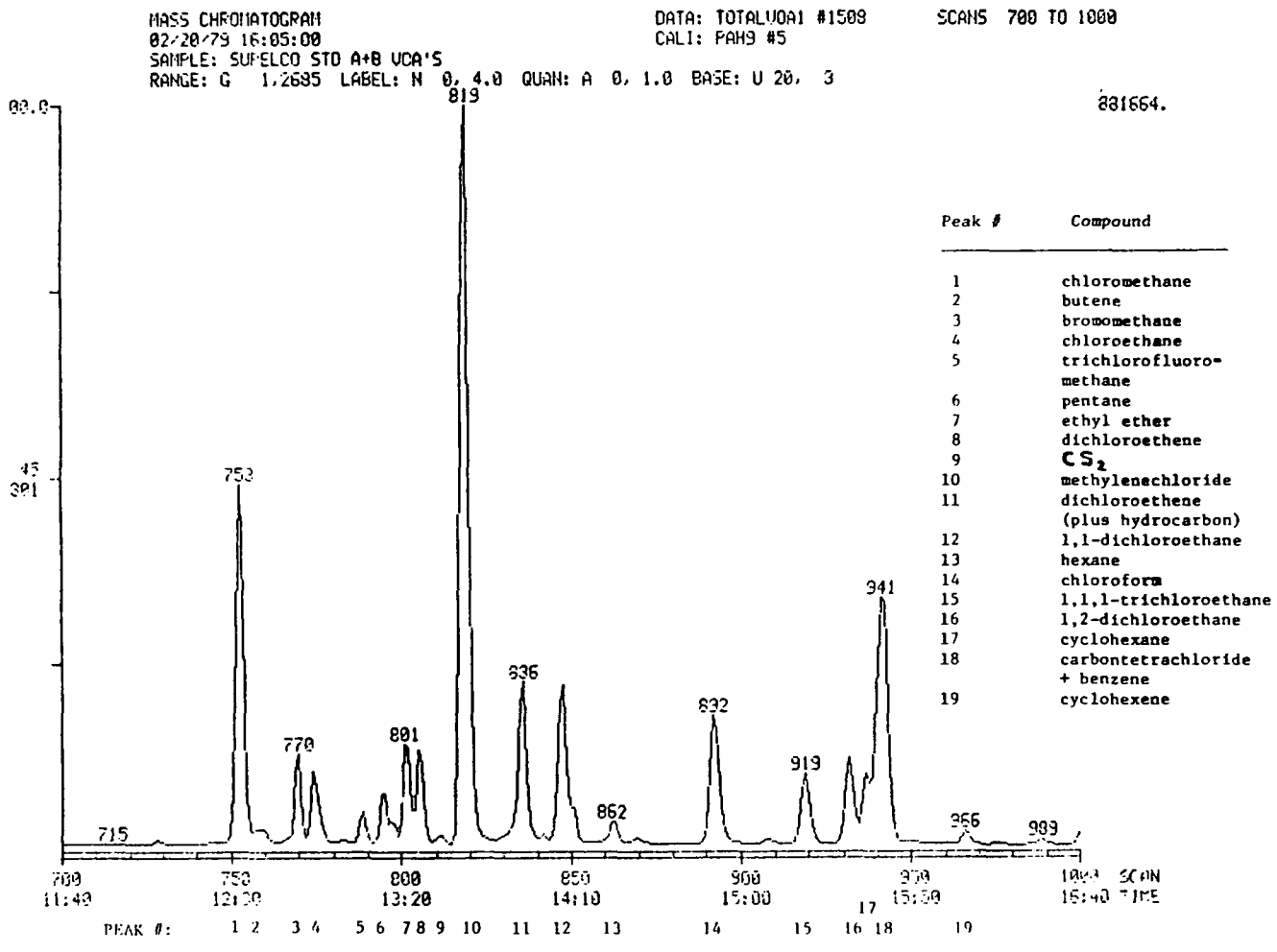


Figure 2-11 Reconstructed Gas Chromatogram of the most volatile organics of the VOA standard

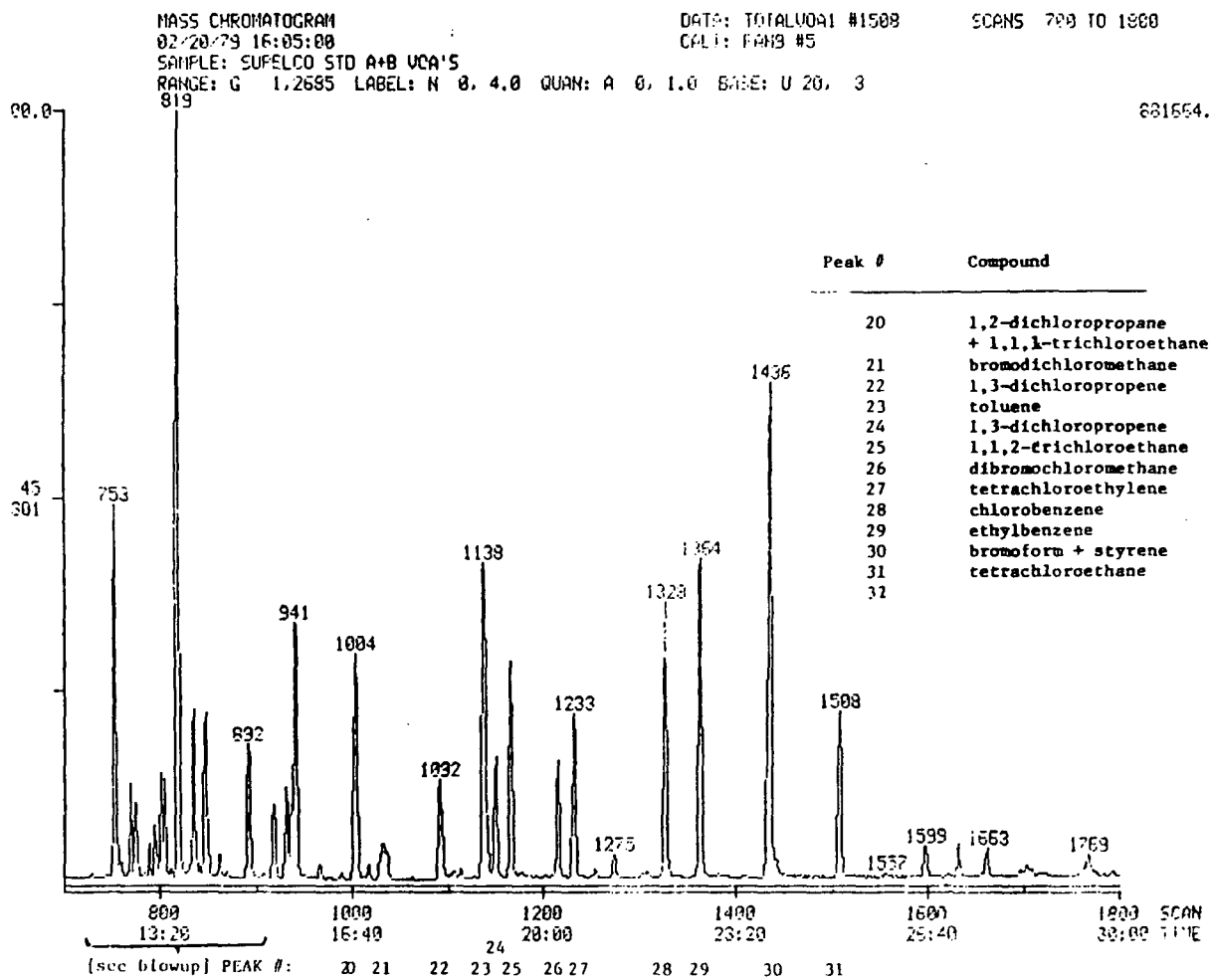


Figure 2-11 Reconstructed Gas Chromatogram of a VOA standard

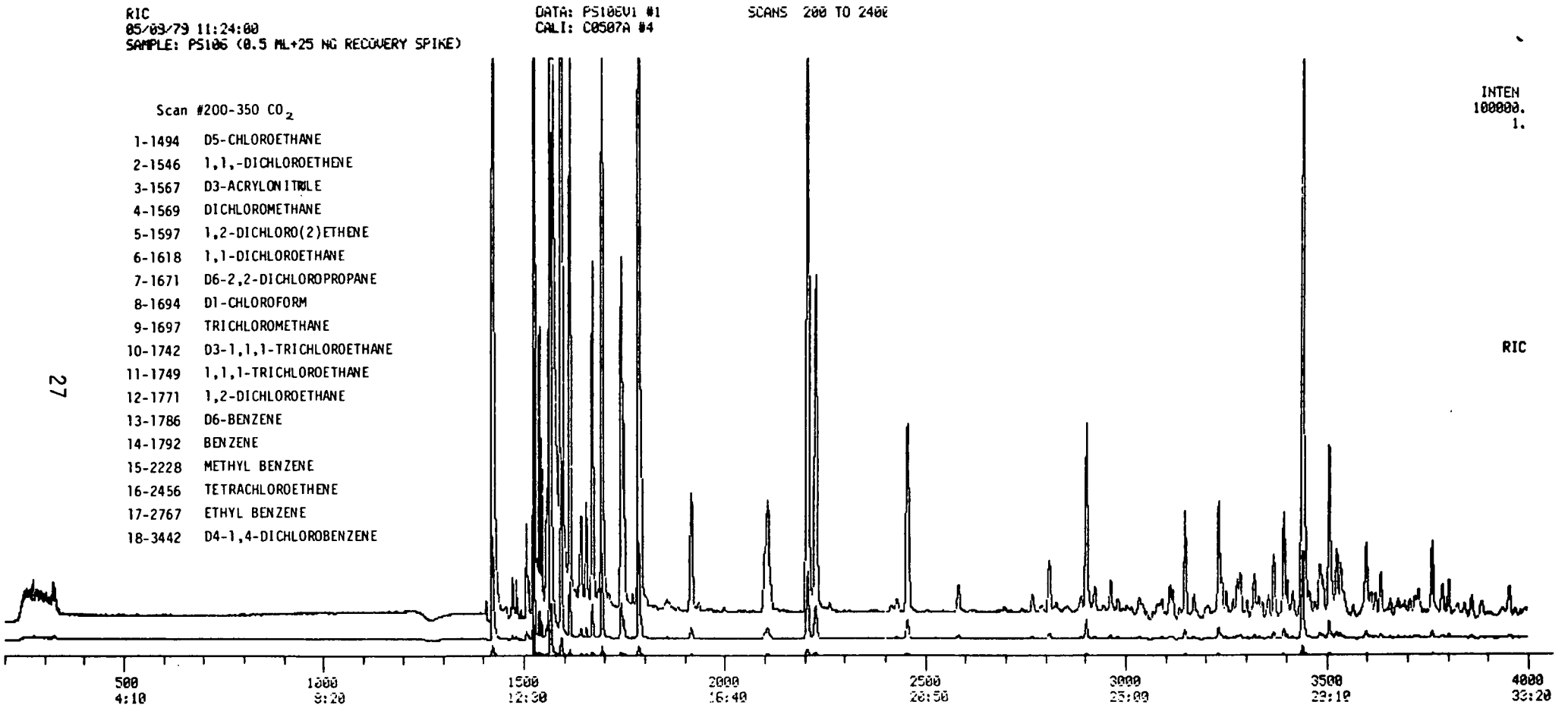
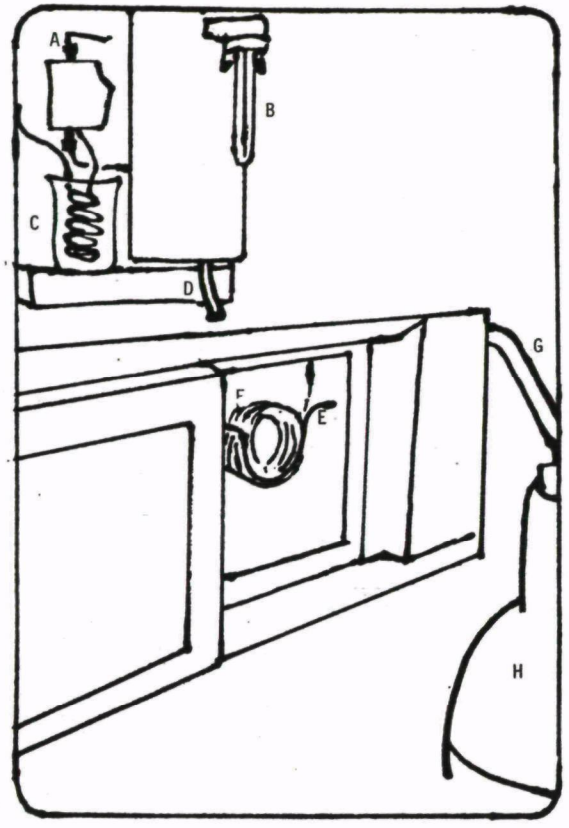


Figure 2-12 Reconstructed Ion Current of Renton-Seattle Raw Sewage.



A TENAX TRAP
B SAMPLE TUBE AND PURGE TUBE
C COOLANT COILS FOR TRAP
D VOA TRANSFER LINE TO GC COLUMN
E CRYOTRAP FOR COLUMN
F GC CAPILLARY COLUMN
G LIQUID NITROGEN TRANSFER LINE
H LIQUID NITROGEN DEWAR

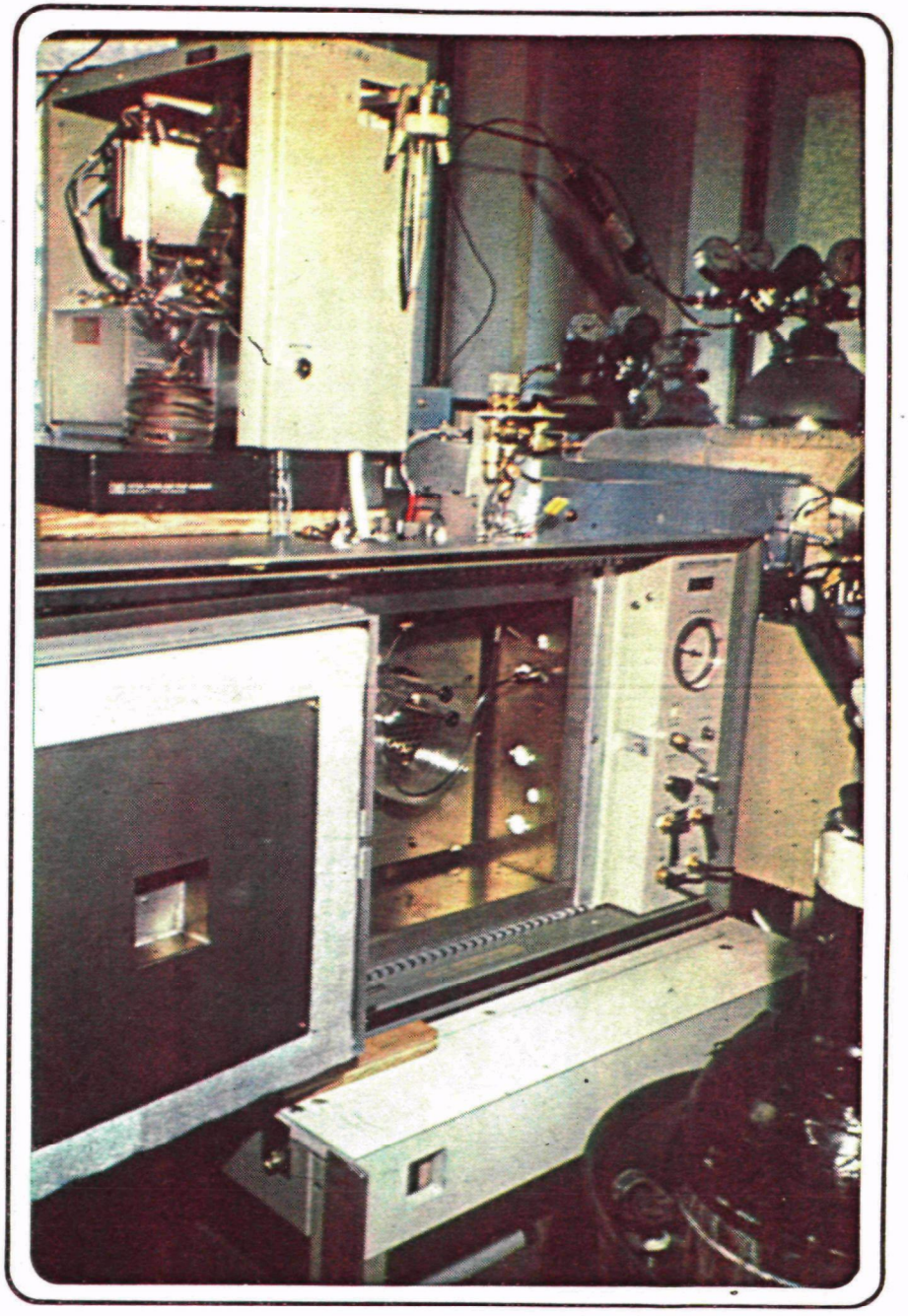
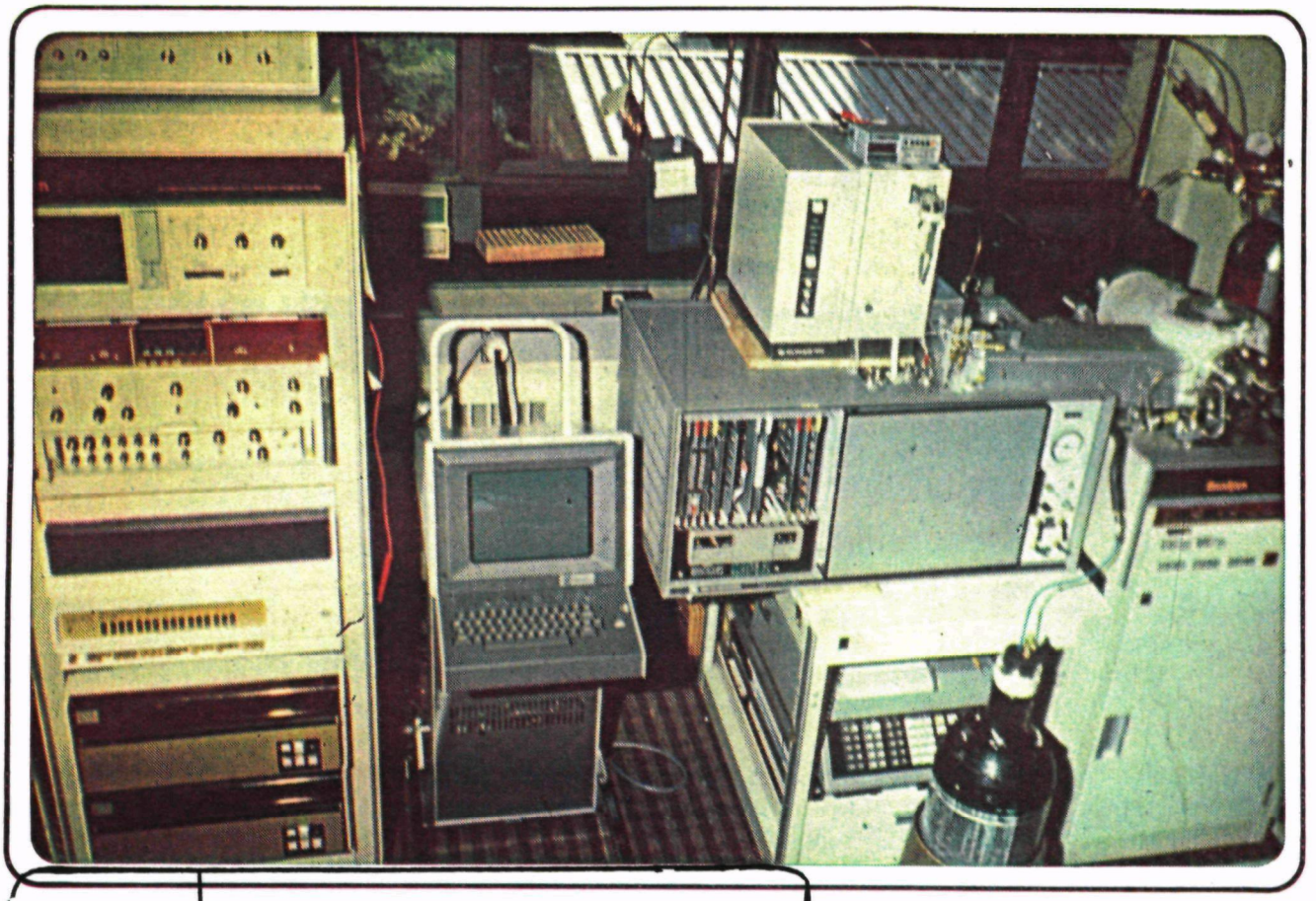


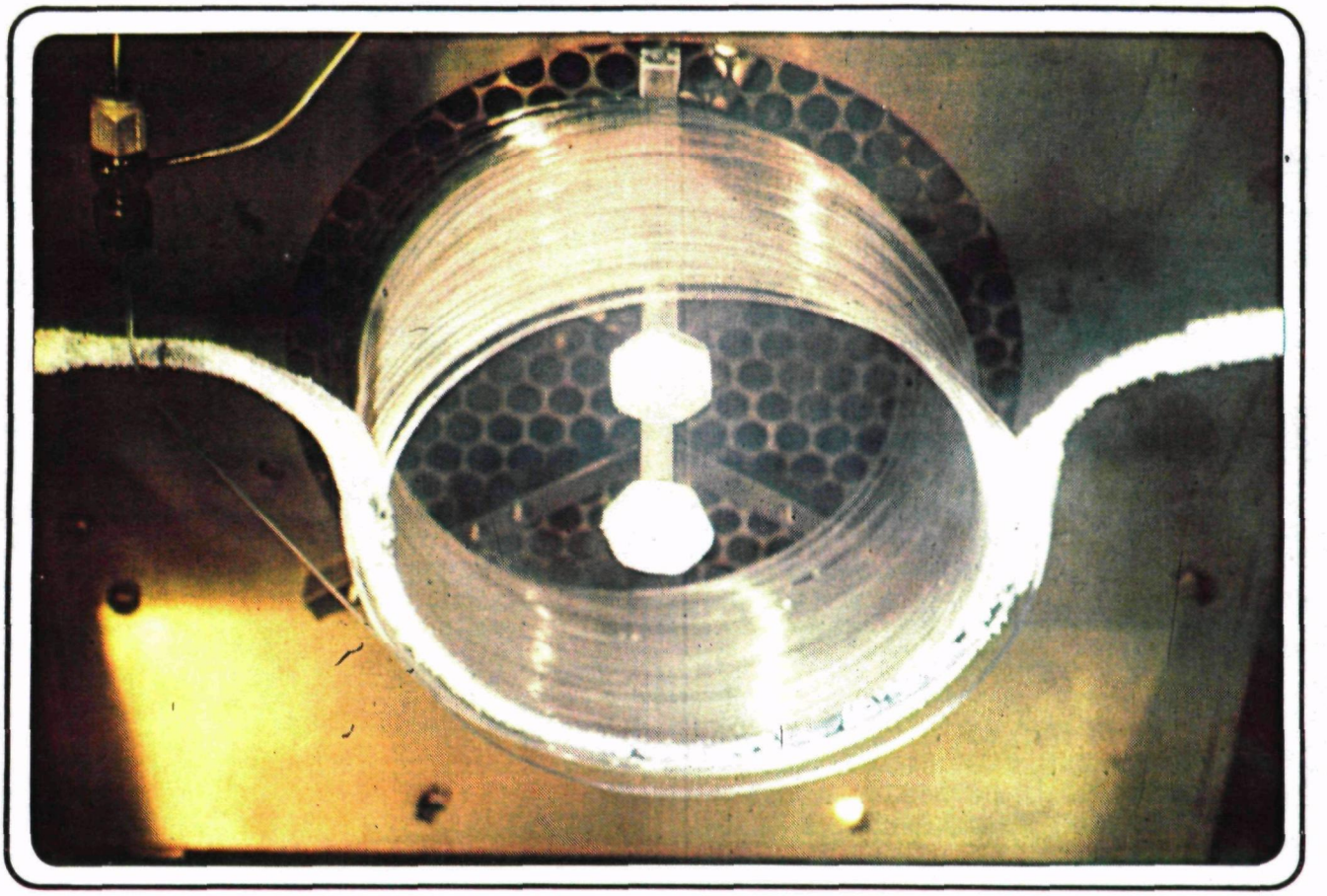
PLATE I PURGE AND TRAP SAMPLER, GAS CHROMATOGRAPH AND CRYOTRAP SYSTEM

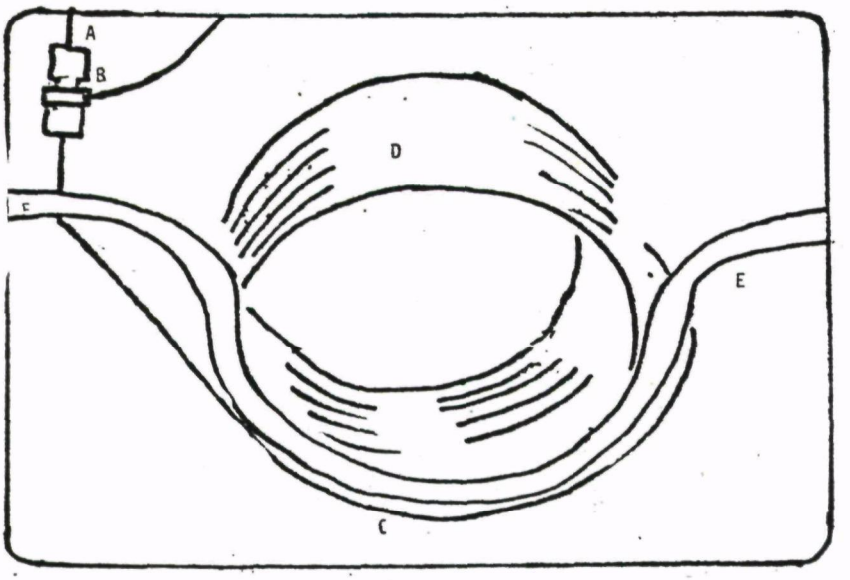


A MASS SPECTROMETER CONTROLLING ELECTRONICS AND DATA SYSTEM
B PURGE GAS PURIFIER
C VOA UNIT
D VOA TRAP TEMPERATURE MONITOR
E TRANSFER OVEN BETWEEN GC AND MS
F TERMINAL FOR MASS SPECTROMETER
G GC OVEN
H LIQUID NITROGEN DEWER
I GC TERMINAL

PLATE II

PURGE AND TRAP SYSTEM
INTERFACED WITH
MASS SPECTROMETER





A INLET FROM VOA UNIT
B VOA CAPILLARY SPLITER
C FIRST COIL OF CAPILLARY COLUMN
D CAPILLARY COLUMN
E CRYOTRAP INLET FOR LIQUID NITROGEN
F CRYOTRAP INLET FOR WARM AIR

PLATE III STEEL CRYOTRAP IN OPERATION

SECTION 3

THE EXTRACTION PROCEDURE

Different methods of extraction have been compared for water, sewage, sludge, sediment, and tissue samples. For water, sewage and sludge, the methods considered were: 1) standard EPA separatory funnel separation; 2) homogenation of sludge or sewage plus solvent and brine with a tissumizer followed by centrifugation; 3) a stirred liquid-liquid continuous extractor and, preliminarily, steam distillation/vapor extraction.

3-1 Separatory Funnel Separation

The study initially evaluated the conventional separatory funnel in which 100/50/50 ml portions of methylene chloride were used to extract the organics at pH 2. The extraction efficiencies of 26 neutral compounds spiked at 20 ppb were 95% and 56% on duplicate samples respectively, while it was 18% and 16% for the 15 acid extractables. Extraction of sludge samples, however, resulted in an emulsion which could not be broken. The funnel separation was therefore abandonned for further use.

However, the recovery of priority compounds by this method from distilled water was determined in duplicate (Table 3-1), and may be considered to represent an effective upper limit to recoveries by separatory funnel extraction for real samples.

3-2 Sample Homogenation

For sludge samples, embodying perhaps the most difficult separation problems, a 3 way comparison was made at the GIT laboratory of different homogenation procedures.

3-2.1 Extraction using filtration

A 200 ml 10% aqueous solution of calcium chloride (w/v), and 200 ml methylene chloride was added to sludge (50 g) in a 1-liter Erlenmeyer flask. This suspension was stirred with a stirring bar (3" long, solvent cleaned) at fast speed for 20 minutes at room temperature. The resulting emulsion was vacuum filtered through a sintered glass disk into a separatory funnel. Another extraction was performed on the retained layer with 100 ml methylene chloride. The recovered total extract was 220 ml, representing a 73% solvent recovery.

To counter the thick emulsion formed during stirring, 1 liter of distilled water was added to 50 grams of sludge in a 4 liter Erlenmeyer flask. A 10% aqueous solution of calcium chloride (100ml) was added, followed by 200 ml methylene chloride. The suspension was magnetically stirred at fast speed for 20 minutes at room temperature. The solution was vacuum filtered in a

Table 3-1. Percent recovery of 20 ppb acids and neutrals spiked into distilled water (500 ml) and extracted by separatory funnel according to EPA standard protocol.

Compound	% Recovery	Δt	% Recovery	Δt
NAPHTHALENE	45	00	62	-09
HEXACHLOROBUTADIENE	92	00	44	-06
ACENAPHTHENE	59	-01	35	-06
2,4-DINITROTOLUENE	0	+37	35	-06
FLUORENE	250	-08	11	-
DIETHYLPHTHALATE	350	-10	300	-05
ANTHRACENE	370	-03	190	-05
PYRENE	34	+04	0	-33
BENZO (A) ANTHRACENE/CHRYSENE	0		54	-08
DIBENZO (A,H) ANTHRACENE	0		0	-
HEXACHLOROETHANE	54	-03	36	-04
N-NITROSO-DI-N-PROPYLAMINE	61	-02	0	-35
1,2,4-TRICHLOROBENZENE	83	-01	56	-12
2-CHLORONAPHTHALENE	84	-00	0	-18
AZPBENZENE (DIPHENYLHYDRAZINE)	38	+01	46	-07
N-NITROSODIPHENYLAMINE	48	+00	01	+20
PHENANTHRENE	116	+04	31	-09
FLUORANTHENE	37	+04	16	-1
HEXAMETHYLBENZENE (1.S.)	100	00	100	00
D-10 ANTHRACENE	70	00	250	-01
1,3-DICHLOROBENZENE	96	-14	66	-02
1,2-DICHLOROBENZENE	88	-16	41	-07
BUTYLBENZYLPHTHALATE	70	-03	28	-01
N-NITROSODIMETHYLAMINE	0	00	0	00
DI-N-OCTYLPHTHALATE	290	+12	22	00
HEXACHLOROBENZENE	80	+02	40	-04
HEXAMETHYLBENZENE (10)	100	00	100	00
2-CHLOROPHENOL	56	-08	0(OR)	-26
PHENOL	0	-	0	-
2,4-DIMETHYLPHENOL	0	-	0	-
4-CHLORO-3-METHYLPHENOL	26	+05	92	-10
ANISOLE	0	-	0	-
D-10-ANTHRACENE (20)	95	-02	60	-06
2-CHLOROMETHOXYBENZENE	-	-	-	-
4-CHLORO-3-METHYLMETHOXYBENZENE	-	-	<0.1	+05
2,4-DICHLOROMETHOXYBENZENE	<0,1	-05	0	-
2,4,6-TRICHLOROMETHOXYBENZENE	-	-	-	-
2-NITROMETHOXYBENZENE	-	-	-	-
4-NITROMETHOXYBENZENE	-	-	-	-
4,6-DINITRO-2-METHYLMETHOXYBENZENE	0	-	1	+03
PENTACHLOROMETHOXYBENZENE	0	-	0	-

separatory funnel through a sintered glass disk. The lower solvent layer was collected, the upper aqueous layer and emulsion was again extracted with 100 ml methylene chloride and vacuum filtered. The lower layer was combined with the first extract. The total combined methylene chloride extract was 160 ml, representing an 80% solvent recovery.

This method was found to be unsuitable for this analysis. The time required in extracting sludge and waiting for the emulsion to break before filtration was frequently very long, usually 3-4 hours. The viscous emulsion also made filtration difficult. When vacuum was applied, particulates were trapped in the frit pores and made subsequent filtration difficult. Different salts at different concentrations were used in an attempt to break up the extract emulsions. A high salt (sodium chloride) concentration, 30% (w/v) was tried, and found to be comparable to 10% (w/v) of calcium chloride, in terms of time required for the emulsion to settle.

3-2.2 Extraction-centrifugation

A 50 ml, 10% (w/v) aqueous solution of calcium chloride and 100 ml methylene chloride were added to 100 grams of well mixed sludge in a 500 ml Erlenmeyer flask. The suspension was homogenized with a Tekmar Tissumizer for 1 minute at 10,000 rpm. The suspension was transferred to four 100 ml glass centrifuge tubes and centrifuged at 2000 XG for 5 minutes to separate the emulsion. The lower solvent layer was taken up with a 30 ml syringe and transferred to a 250 ml Kuderna-Danish flask. The sludge cake and supernatant were combined and 50 ml fresh methylene chloride was subsequently added. This suspension was again homogenized with the Tekmar Tissumizer for 1 minute at 10,000 rpm, centrifuge separated, and the lower solvent layer combined with the first extract. This extraction was repeated a third time with 50 ml methylene chloride. The combined methylene chloride extract of 160 ml, representing an 80% solvent recovery was evaporated to 2 ml. The coffee-colored thick extract was passed through a 5-3/4" disposable pipett packed with 3" baked anhydrous sodium sulfate. The microcolumn was rinsed with fresh methylene chloride and the volume was adjusted to 5 ml. The total extraction time required, including solvent evaporation time, was less than one and a half hours.

An alternative procedure for extraction efficiency was also tested. A 50 ml aqueous solution of 10% calcium chloride (w/v) and 100 ml methylene chloride was added to 100 grams of sludge in a 1 liter Erlenmeyer flask. The suspension was stirred by magnetic stirrer at fast speed for 10 minutes at room temperature instead of a Tissumizer. The resulting emulsion was placed in four centrifuge tubes and centrifuged for 5 minutes at 2000 XG. The methylene chloride layer was taken up with a 30 ml syringe. The sludge cake and aqueous phase was returned to the flask and

extracted with 50 ml methylene chloride two more times. The combined methylene chloride was placed in a Kuderna-Danish evaporator and evaporated to 2 ml. The concentrated extract was passed through a micro column packed with 3" baked anhydrous sodium sulfate. The final volume was increased to 5 ml with fresh methylene chloride. When 100 ul of the above extract was transferred onto a weighed aluminum pan and solvent evaporated, duplicate residues weighed 11.2 mg and 11.0 mg. When 100 ul of the extract, which was homogenized with the Tissumizer, was similarly weighed, the duplicate residues weighed 12.5 mg and 13.2 mg. The above two extraction methods therefore do not show much difference in extracting efficiency. Mixing using Tissumizer has the advantage of saving time, since only 1 minute was needed for complete mixing.

The final extract volume was decided by placing 100 ul of the extract onto a tared aluminum pan, and letting the solvent evaporate. The residue was weighed and the solution concentration was adjusted to 100 mg/ml. This concentration is very critical for the subsequent gel permeation column chromatography because the gel column has a fixed capacity. Overloading the column will reduce the resolution and shorten the column life.

3-3 Liquid-Liquid Extraction

In an attempt to improve recoveries in the extraction of organics from water, a new extractor was designed and evaluated at the University of Washington.

The present EPA analytical protocol for the screening phase of the BAT program calls for simple liquid-liquid extraction of aqueous samples with 450 ml of solvent (in portions of 250, 100, 100 ml) by means of a separatory funnel. In cases where emulsions prevent the recovery of 85% of the extracting solvent volume, the emulsions are to be broken or continuous extraction used at the analyst's discretion. Bulk organics in the sample matrix cause emulsification, which varies greatly with the levels and nature of these compounds. The actual efficiency of partitioning between the solvent and the sample, consisting of a wide range of compounds such as the priority pollutants, also varies considerably with the matrix. For accurate and precise determinations of these compounds in water samples, an extraction technique that is relatively insensitive to matrix effects and is applicable to a broad range of compounds is desirable. Conventional liquid-liquid extraction generally satisfies these requirements but is impractical because long extraction times (24-48 hours or longer) are required. The rate of extraction for liquid-liquid continuous extractors can be greatly improved by gentle stirring of the phases to increase the rate of mass-transport within each phase, while having little or no effect on the contact area or area/volume ratio for the phases. The presumptions that the slow process in the extraction is diffusion of solute to the aqueous/

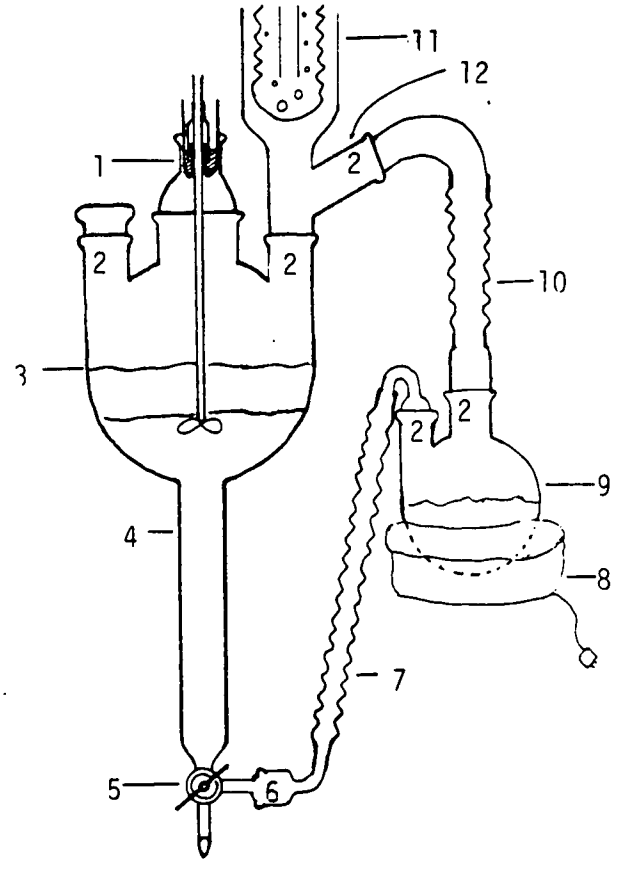
organic interface, and that crossing the interface is relatively rapid, are in accordance with the observations of other investigators (Dunges, 1978). Slow nonturbulent stirring without breaking the solvent and sample up into droplets has a minimal tendency toward emulsion formation, but can substantially increase the rate of extracton by moving solutes to the interface rapidly relative to their diffusion rates in water.

The extractor is illustrated schematically in Figure 3-1. Its design is modular and is constructed of stock laboratory glassware. The main extraction chamber is a 2000 ml 3-neck round bottom flask, modified by the addition of a stem at the bottom, terminating in a teflon stopcock and ground glass connector. The concentrator vessel is a standard 500 ml round bottom flask. A standard Friedrichs condenser was modified by the addition of a distillate inlet below the cold finger; this modification is not essential but is convenient for the spatial arrangement of the components. The connections between condenser, extraction vessel, and concentration vessel are of corrugated teflon tubing, which is flexible enough to permit the concentrator to be raised or lowered relative to the extractor chamber. The stirrer shaft is introduced through the center neck of the extractor chamber via a liquid-seal; the stirrer itself is teflon coated. The stem of the extractor chamber is filled with silanized glass beads to aid in breaking any slight emulsions that may form and to retain and separate aqueous droplets. All interior surfaces of the apparatus are of teflon or silanized glass. For this extractor, a special non-vortexing stirrer head (Bel-Art catalog #H37218) was used.

The extractor was evaluated in three ways: 1) the general performance characteristics, such as reflux rates, degree of stirring possible, and efficiency of solvent/water separation, were observed; 2) recovery profiles of total extractables from raw sewage samples were measured gravimetrically for several sets of extractor conditions; and 3) individual recoveries of representative priority pollutants from spiked distilled water were measured in a preliminary experiment. The gravimetric studies utilized untreated, unfiltered sewage that had been stored at 5 C. The total recoveries of extractable matter varied from experiment to experiment, presumably because of varying amounts of suspended particulates in each sample. Samples were taken at one hour intervals by the following procedure: the reflux was stopped, the volume in the concentrator vessel was adjusted to 200 $\pm$ 5 ml, and a 10 ml aliquot was withdrawn in a class A pipette. This sample was air dried in a tared pan, then dried in a dessicator over P_2O_5 to constant weight. In addition to hourly samples, one "infinity" sample was taken at 12 or 24 hours.

1 24/40 ⧖ LIQUID STIRRER SEAL
2 24/40 ⧖ JOINT
3 2000 ML 3-NECK RB FLASK
4 GLASS NECK FILLED WITH SILANIZED BEADS
5 TEFLON 3-WAY STOPCOCK
6 14/20 ⧖ JOINT
7 1/4 INCH CORRUGATED TEFLON
8 HEATING MANTLE
9 500 ML 2-NECK RB FLASK
10 3/4 INCH CORRUGATED TEFLON
11 FRIEDRICHS CONDENSER
12 MODIFICATION (VAPOR INLET)

1. ⧖ 24/40 CONNECTION TO ADDITIONAL CONDENSOR
2. OUTLET COLD FINGER
3. INLET COLDFINGER
4. ⧖ 34/40 JOINT FOR COLD FINGER
5. CONDENSOR OUTLET
6. CONDENSOR OPENING
7. CONDENSOR INLET
8. NOTE: ONE ARM IS HIGHER
9. ⧖ 24/40 FLASK WITH SAMPLE
10. ⧖ 24/40 FLASK WITH METHYLENE CHLORIDE



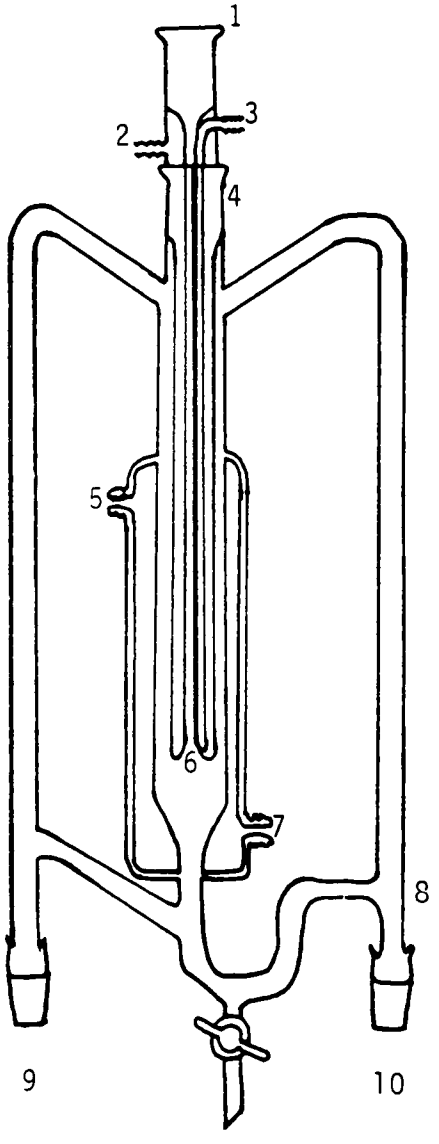


Figure 3-1 Stirred Liquid-Liquid Continuous Extractor and Steam Distillation Vapor Extractor Evaluated in the Present Study

The spiked distilled water experiment utilized a standard mixture containing approximately 10 micrograms each of 53 compounds in 10 ml of methanol, diluted in 1200 ml with distilled water and left 48 hours before extraction. Prior to extraction the pH was adjusted to 1.9 with dilute HCl. A six hour and a 12 hour extract were taken, comprising the entire extract up to those respective points (plus a rinse) after which the pH was raised to 11.9 with NaOH solution, and two further 6 hour samples were taken. The samples were concentrated to 25 mls by evaporation at room temperature, then dried by standing over anhydrous sodium sulfate for 18 hours. They were then concentrated to 1 ml and a 10 microgram spike of anthracene solution was added as an external standard. The samples were analyzed by GC-FID on an SE-54 WCOT capillary column with GC/MS verification. Individual compounds were quantified by comparison with the anthracene external standard. The phenols were analyzed on a SP-1000 WCOT capillary column and quantified in the same manner. All extractions were performed using methylene chloride as the extracting solvent.

The mechanical stirrer used (Gerald Heller GT 21) provided good control of speeds between 100 and 400 rpm. No vortexing was observed at any of these speeds, nor was any significant emulsification seen during the subsequent experiements using sewage samples. The transport of water from the extractor to the separator was minimal, and only in microdroplet form as evidenced by cloudiness in the extract. The reflux rate was limited by the rate of solvent return through the glass bead bed. Too fast a reflux rate resulted in solvent buildup in the extractor chamber side. Refluxing at 1L/hour was possible without significantly depleting the reservoir in the concentrator vessel.

Table 3-2 presents the experimental conditions and results of the gravimetric experiments. The results have been corrected for the effect of removing successive aliquots from the concentrator while the extraction was in progress and are expressed as a percentage of the "infinity" value. The results are presented graphically in Figure 3-2. In general, the observed trends bear out intuitive expectations: increasing the stirring rate (4<3<2=1) increased extraction efficiency, while positioning the stirrer nearer the interface (1,3,4 vs 2) also shortened the extraction time. The degree to which intra-phase mixing can improve extraction rate is seen most dramatically by comparing experiment 1 with experiment 4. In the first case, with moderate gentle stirring, 70% extraction is achieved in 2.5 hours. With the slowest stirring possible, 70% extraction requires approximately 8 hours. With no stirring at all (as in a conventional liquid-liquid continuous extractor), 70% extraction would presumably take longer still. Since there was no substantial interphase mixing observed in any of these experiments, the improvement may be attributed to intra-phase transport rate enhancements. The gravimetric results suggest that with optimization of

Experiment:		1	2	3	4
Volume, aq.	(ml)	1100	1100	1200	1200
Volume, org.	(ml)	650	550	550	550
Stir speed	(rpm)	104	104	16+	16-*
Placement (distance from interface)	(cm)	0.5	2.5	0.5	0.5
Recovered weight: mg ± mg (% of infinity value)**					
1 hour		1.7 (42.5)	1.3 (35.7)	1.1 (31.9)	0.6 (21.1)
2		2.5 (62.1)	2.1 (56.7)	1.8 (49.4)	0.9 (32.7)
3		3.1 (77.5)	2.4 (65.0)	2.1 (62.1)	1.3 (44.8)
4		3.4 (83.8)	2.8 (76.2)	2.2 (65.0)	--
5		--	--	2.4 (70.8)	1.9 (68.0)
6		--	3.2 (87.9)	--	--
8		--	--	--	2.4 (85.2)
12		--	3.6 (100)	3.4 (100)	2.5 (89.1)
24		4.0 (100)	--	--	2.8 (100)

*Lowest speed attainable with mechanical stirrer.

**Inifinity values are underlined. A blank experiment under conditions of experiment 3 yielded less than 0.1 mg after 12 hours.

Table 3-2 Gravimetric Liquid-Liquid Extractor Experiments

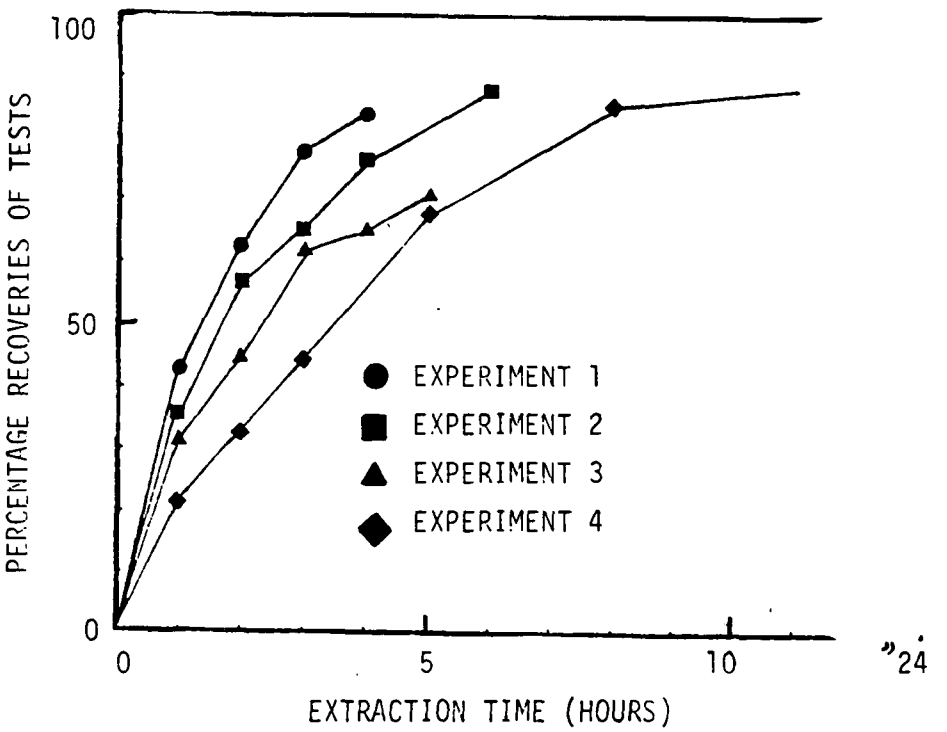


Figure 3-2 Extraction Efficiencies of the Modified Stirred Liquid-Liquid Extractor.

extractor stirring, total extractables may be recovered to 80+% within six hours.

The recoveries of 48 priority pollutants, spiked at 20 ppb, are listed in Table 3-3. The average recovery of 46% was lower than the gravimetric results, although 95% of all extraction occurred within the first six hours. The standard deviation of 21% was considerably lower than observed for the separatory funnel.

Further experiments have been carried out on real matrices. In these cases, separate recoveries for extraction are not possible since the extracts require cleanup and prefractionation prior to quantitation. However, system recoveries determined with water and spiked sewage samples show higher recoveries, (as will be discussed in future sections) resulting from improved quantification methods. A comparison of extraction performance from real samples for this extractor involved gravimetric studies of extracts of influent and sludge obtained by 3 methods: stirred liquid-liquid continuous (SLLC), tissumizer and centrifuge (TC), and sepfunnel and centrifuge (Table 3-4). In the first series, the effect of additions of methanol or brine ($CaCl_2$) to the sludge were evaluated, with no significant difference observed. In a second and third series, the three methods were compared using conditions as shown. The TC conditions were those developed at GIT, with the total volume of solvent increased in the second experiment to match that used in the other two methods. Based on total extractable mass, a significant increase for the SLLC is observed.

The principal shortcomings of the SLLC are its complicated nature and the difficulty of setting it up and operating it in a steady state. Also, its very efficiency increases the loading of interference in the sample. For these reasons, a steam distillation vapor extractor, as shown in Figure 3-1, was investigated. In this procedure, the aqueous sample is boiled at ambient pressure and the condensate and vapor mixed with organic vapor/condensate. The combined distillates fall into a separation chamber and each phase is returned to its vessel. Initial experiments recovering 35 extractable pollutants spiked in methanol from distilled water at the 20 ppb level showed 89.2 $\pm$ 19.9% recovery using methylene chloride as the solvent in a 12 hour extraction, and this unit is therefore currently the method of choice. Plate V shows the stirrer in operation. Plate VI shows an alternative apparatus currently under evaluation.

Recovery: %

#	Compound		0-6 hrs	6-12 hrs	0-12 hrs
1	1,4-DICHLOROBENZENE 1,2-DICHLOROBENZENE 2-UNRESOLVED PHENOLS	(1) (1) (1)	25.5 27.1	4.1 4.2	29.6 31.3
2	HEXACHLOROETHANE DIISOPROPYL N-NITROSAMINE	(4,6) (4,6)			
3	NITROBENZENE		45.2	3.9	49.1
4	TRICHLOROBENZENE	(5)	23.9	2.9	26.8
5	DIMETHYLPHENOL		31.2	--	31.2
6	NAPHTHALENE		31.2	5.0	36.2
7	TRICHLOROBENZENE	(5)	33.8	3.8	37.7
8	4-CHLORO,3-METHYLPHENOL		23.8	--	23.8
9	2-CHLORONAPHTHALENE		43.0	3.1	46.1
10	1-CHLORONAPHTHALENE		44.6	4.1	48.7
11	ACENAPHTHYLENE HEXAMETHYL BENZENE	(4,6) (4,6)			
12	2,4-DINITROTOLUENE		75.4	--	75.4
13	ACENAPHTHENE		54.3	4.2	58.5
14	2,6-DINITRO TOLUENE		87.6	--	87.6
15	FLUORENE		54.7	3.1	57.8
16	DIETHYLPHTHALATE		89.5	3.1	92.6
17	DIPHENYLHYDRAZINE DIPHENYLDIAZINE (AZOBENZENE)	(3,6) (3,6)			
18	4-BROMODIPHENYL ETHER		54.4	2.2	56.6
19	α-BHC		62.3	3.1	65.4
20	HEXACHLOROBENZENE		12.4	--	12.4
21	DIBENZOTHIOPHENE		57.0	2.2	59.2
22	β-BHC γ-BHC (LINDANE)	(4) (4)			
23	PHENANTHRENE		62.3	2.7	65.0
24	ANTHRACENE		Internal Standard		
25	δ-BHC		87.2	--	87.2
26	HEPTACHLOR		19.9	--	19.9
27	ALDRIN		24.1	--	24.1
28	HEPTACHLOR EXPOXIDE		61.8	--	61.8
29	FLUORANTHENE		50.6	1.8	52.4
30	PYRENE		43.4	1.6	45.0
31	P,P' DDE		20.6	--	20.6
32	DIELDRIN		57.7	--	57.7
33	ENDRIN ALDEHYDE (Δ-KETO ENDRIN)	(3)			
34	P,P' DDD		33.7	--	33.7
35	P,P' DDT		16.7	--	16.7
36	BENZ (A) ANTHRACENE		22.7	--	22.7
37	CHRYSENE		12.9	--	12.9
38	DICHLOROBENZIDINE	(4)			
39	BIS-ETHYL HEXYL PHTHALATE		71.8	--	71.8
40	MIREX		23.1	--	23.1
41	DIOCTYL PHTHALATE		46.2	--	46.2
42	BENZO (E) PERYLENE	(4)			
43	DIBENZO (A) ANTHRACENE	(4)			
44	BENZO (GHI) PERYLENE	(4)			
45	PHENOL	(1)	38.4	2.2	40.6
46	O-CHLOROPHENOL	(1)	36.7	1.9	38.6
47	DICHLOROPHENOL	(1)	48.0	2.3	50.3
48	TRICHLOROPHENOL	(1)	51.9	1.4	53.3

Notes:

1. Quantified in additional GC analysis.
2. Numbers correlate with Figures 5-4 and 5-5.
3. The analysis of this compound is discussed in "Instrumental" section
4. Compound not quantitated in this experiment.
5. Isomer not assigned in this experiment.
6. Not resolved by GC/FID

Table 3-3 Recoveries From Spiked Water Using Stirred Liquid-liquid Continuous Extractor.

Sample:	Sewage	Sewage	Sewage	1^{o} Sludge	1^{o} Sludge	1^{o} Sludge
Volume, aqueous (ml)	500	500	500	200	200	200
Volume, $MeCl_2$ (ml)	500	500	500	(500;500)	(500;200)	(500;500)
Volume, $CaCl_2$	-0-	50 grams	-0-	(-0-;-0-)	(100ml 10%; 100 ml)	(-0-;-0-)
Volume, MeOH	-0-	-0-	200 ml	(200;200)	(100 ml;-0-)	(200;200)
Extraction Method	Stirred C.L.L. 12 hrs, pH2 12 hrs, pH12	Stirred C.L.L. 16 hrs, pH2 8 hrs, pH12	Stirred C.L.L. 16 hrs, pH2 8 hrs, pH12	Stirred C.L.L. 12 hrs, pH2	Tissumizer & centrifuge, 3 portions 3:1:1	Separatory funnel & centri- 3 portions 3:1:1
Residue weight from extraction:						
Acid	6.1 mg	6.6 mg	6.2 mg	(143.1;113.5)	(36.0;44.0)	(43.6;45.7)
Base	1.3 mg	1.45 mg	1.4 mg	---	---	---

Table 3-4 Effect of experimental conditions of recovery of gravimetric residue using continuous liquid-liquid extractor and comparison with tissumizer and separatory funnel

A STIRRER MOTOR
B CONDENSER
C MERCURY SEAL
D METHYLENE CHLORIDE VAPOR TRANSFER TUBE
E STIRRER PADDLE AND SAMPLE EXTRACTION FLASK
F METHYLENE CHLORIDE LIQUID TRANSFER TUBE
G METHYLENE CHLORIDE EXTRACT
H HEATING MANTLE

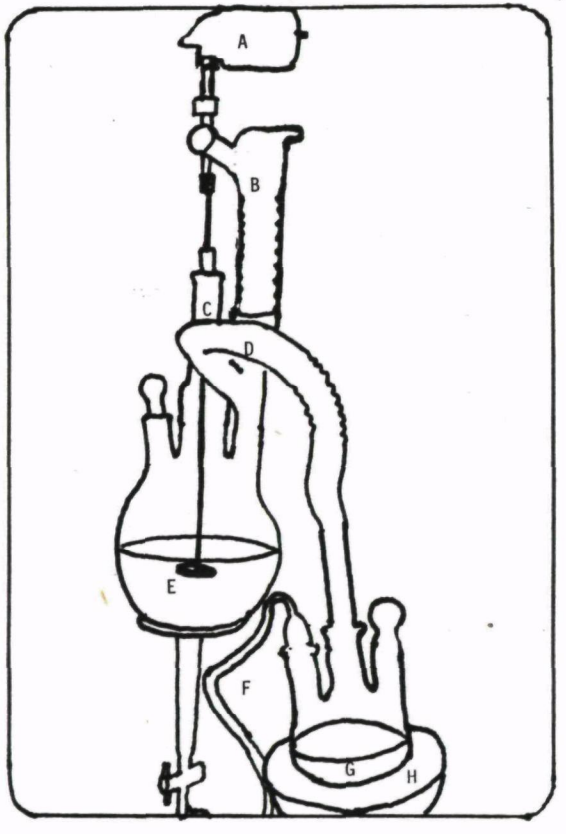
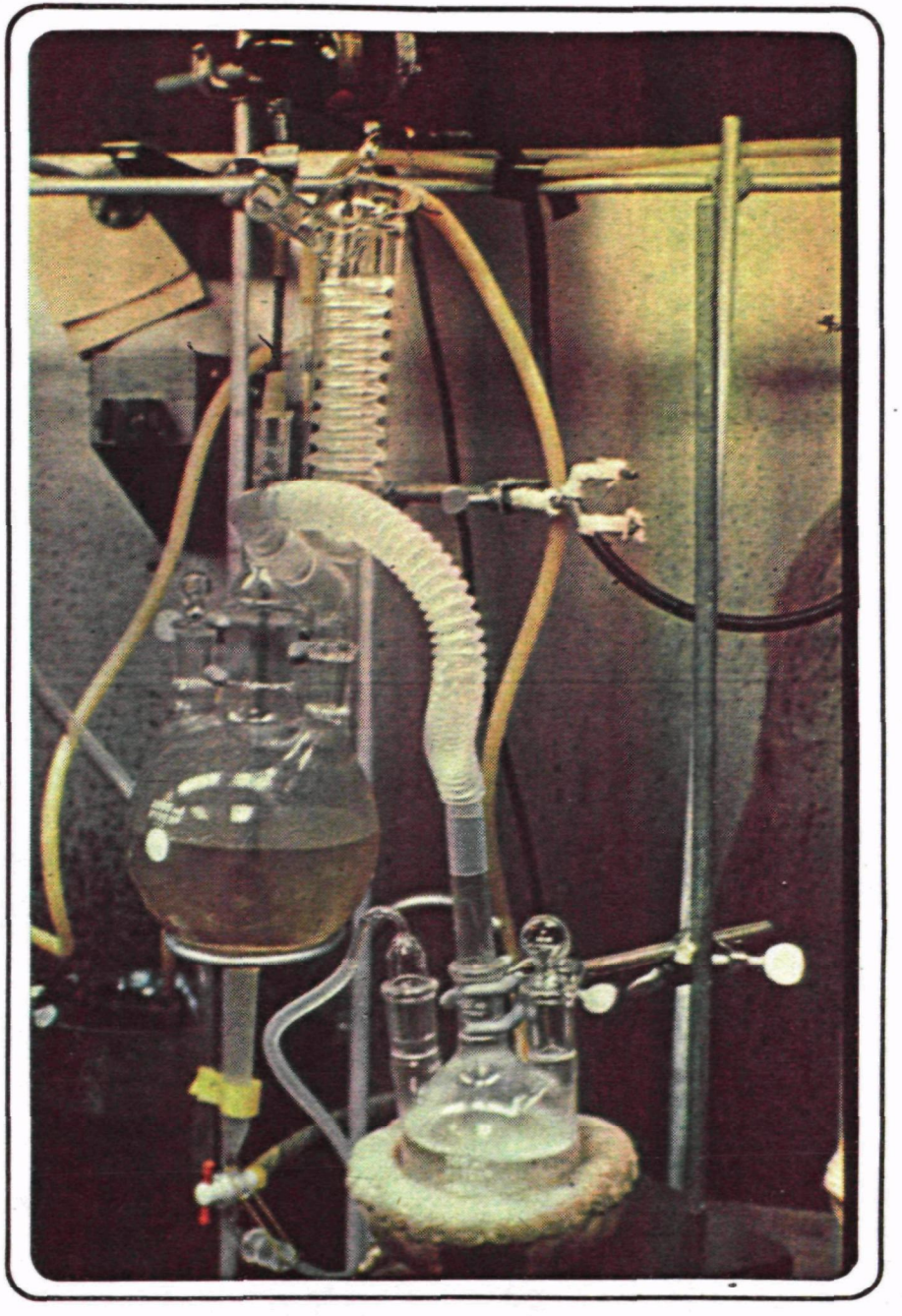
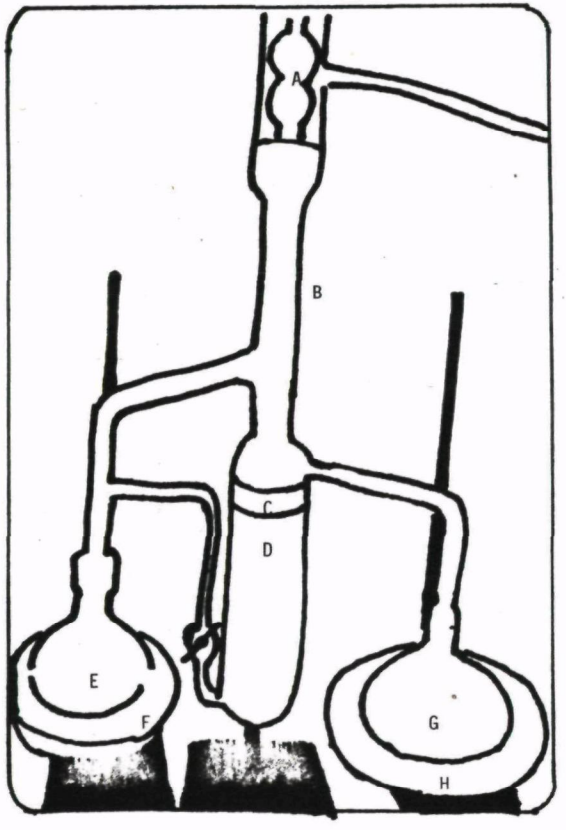


PLATE IV STIRRED LIQUID-LIQUID EXTRACTOR IN OPERATION



A CONDENSER AND WATER INLET HOSE
B CONDENSATION OF WATER-$MECL_2$ IN EXTRACTOR
C WATER LAYER
D METHYLENE CHLORIDE LAYER
E METHYLENE CHLORIDE FLASK
F HEATING MANTLE FOR METHYLENE CHLORIDE
G SAMPLE FLASK
H HEATING MANTLE FOR SAMPLE



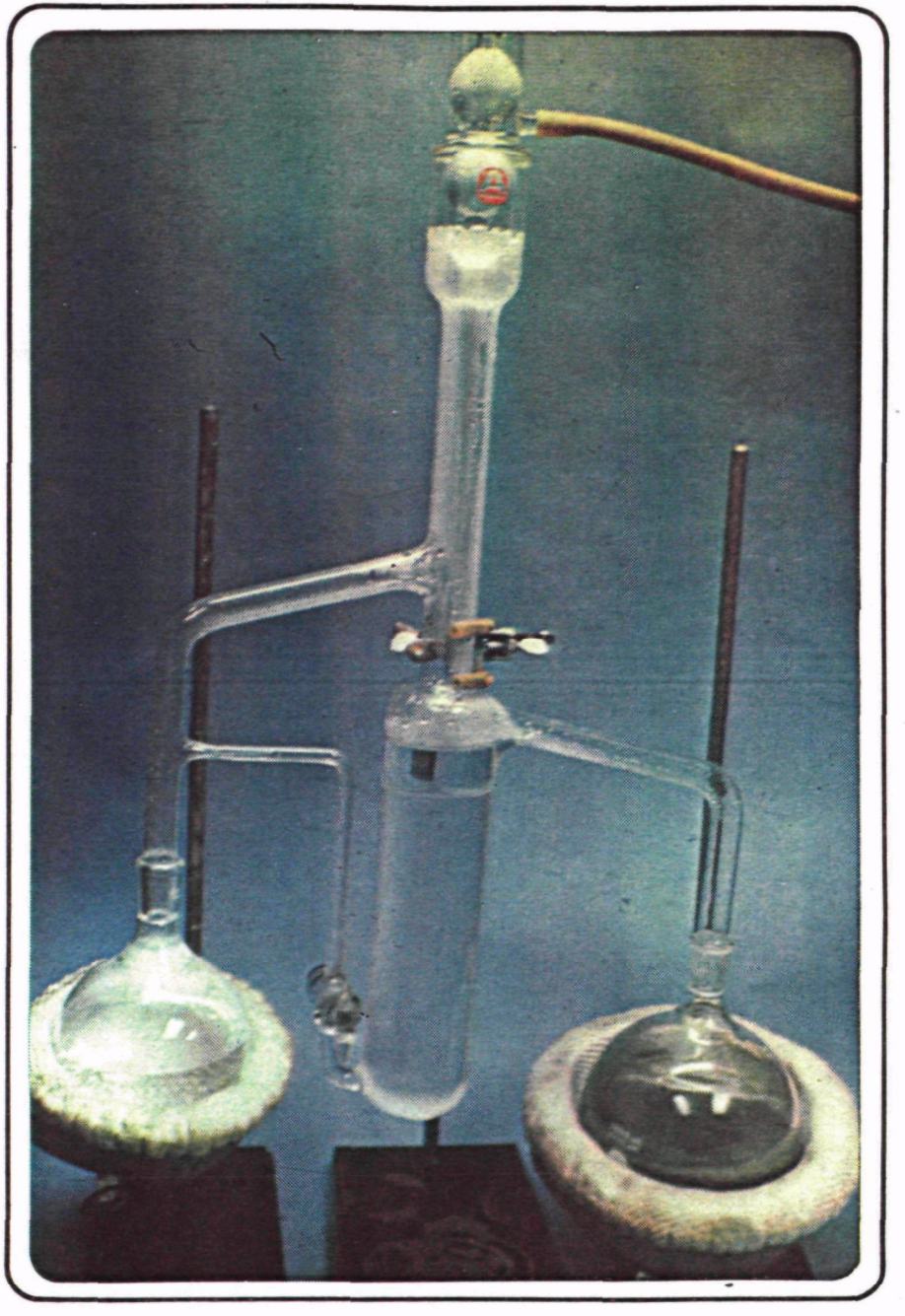


PLATE V EXTRACTIVE STEAM DISTILLATION APPARATUS IN OPERATION

SECTION 4

PREFRACTIONATION AND CLEANUP

4-1 Gel Permeation Chromatography

Based on the reports of Stalling (1972) and others, studies were undertaken to determine the feasibility of using gel permeation chromatography on BioBeads (BioRad Corp) for the removal of lipid and other gross biological contaminants from concentrated sample extracts. The initial experiments, indicated that significant reductions of interfering compounds seen in analyses of the acidic fraction could be achieved with this method, rather than by using the standard EPA procedure. Initially, it was observed that, for the analysis of phenols in particular, the most troublesome compounds present in the matrix were free fatty acids. In order to determine whether separation of the fatty acids from phenols is obtainable on BioBeads, and to characterize the relative elution order and separation of interfering compounds from representative acidic, neutral, and basic priority pollutants, a series of chromatography experiments were conducted. Four types of BioBeads (SX-2, 3, 8, and 12) with nominal exclusion limits of 2700, 2000, 1000, and 400, respectively, were evaluated using three different solvent mixtures (cyclohexane/methylene chloride: 75/25, 50/50, 35/65). The experiments were conducted using a synthetic sample mixture containing representatives of the chemical classes of priority pollutants, plus model interferences such as fatty acids, cholesterol, and bile acids, as listed in Table 4-1.

The BioBeads were pre-equilibrated in the selected chromatography solvent system prior to gravity packing into 2 x 50 cm silanized glass columns with silanized glass wool plugs. The packed columns were rinsed with approximately two column volumes of solvent and the surface of the gel bed was covered with a 1 cm layer of silanized 10 micron glass beads to protect the bed from being disturbed during solvent additions. The samples were applied by addition of a 1 ml solution pre-adjusted to the same solvent composition as the eluting solvent, with minimal dilution during sample application. Flow rates were then determined and sample collection time adjusted to give approximately 10 ml fractions, which were collected in silanized glass test tubes. Some shrinkage of the gel was observed upon changing from 50/50 cyclohexane/methylene chloride (solvent "A") to 75/25 (solvent "B"). Solvent "C" was increased in cyclohexane from 25% to 35% because the former solvent mixture was dense enough to float the BioBeads, creating experimental difficulties.

In the analyses of the fractions from the original 12 experiments (SX-2, 3, 8, 12 with solvents A, B, and C) difficulties were encountered in the GC analysis of the acidic compounds. Poor and inconsistent column performance from packed columns of

PHENANTHRENE	DIETHYLPHTHALATE	CAPRIC ACID
PYRENE	DI-N-OCTYLPHTHALATE	MYRISTIC ACID
CHRYSENE	P,P'-DDT	CAPROIC ACID
BENZ(E)PYRENE	P,P'-DDE	CAPRYLIC ACID
NAPTHALENE	DIELDRIN	LITHOCHOLIC ACID
P-DICHLOROBENZENE	AROCHLOR 1254	CHOLIC ACID
1,2,3-TRICHLOROBENZENE	HEPTACHLOR	DEOXYCHLOLIC ACID
HEXACHLOROBENZENE	LINDANE	CHLOESTEROL
4-CHLORO,3-METHYLPHENOL	BENZO(GHI)PERYLENE	LAURIC ACID
2,4,6-TRICHLOROPHENOL	P-NITROPHENOL	
2,4-DIMETHYLPHENOL	2,4-DINITROPHENOL	
NITROBENZENE		
O-CHLOROPHENOL		

Table 4-1 Mixture of chemicals tested during the gel permeation studies

SP 1240DA, low derivatization yields, and contamination of some samples by leaching of vial cap, prevented the detection of several of the fatty acid and phenol components. An additional series of experiments were subsequently carried out, focusing on the compounds of interest (phenols, fatty acids phthalates, and lipid) using the columns representing the extremes of performance (SX-2 and 12). The solvent for this series ("D", "E", and "F") was 50/50 pentane/methylene chloride with the same column dimensions as for A, B, and C. Series D determined the elution behavior of a lipid mixture (as modeled by corn oil) by gravimetric analysis (for SX-2 only), while series E used a selective sample (phthalates, phenols, and fatty acids) and series F used the same sample as series A, B, and C, respectively. These were analysed by GC on SE 54 and SP 1000 WCOT capillary columns. Again, fractions were collected at time intervals and selected to give 10 ml fractions. Table 4-2 summarizes the BioBead experimental conditions.

The data from the GC analyses of the original BioBeads experiments are presented in Figures 4-1 through 4-13. These figures show the elution pattern for generalized groups of compounds, with elution behaviors for selected specific compounds indicated in the bar-graph portion. In no case was total class fractionation (lipid from phthalates from fatty acids from phenols, etc.) achieved. Also, to obtain a more complete picture of the elution profile, the experimental results for series A, D, E, and F should be superimposed, as the conditions were practically the same. Inspection of the entire set of data shows a certain insensitivity of the elution profile to solvent composition and BioBeads exclusion limit. The differences observed are significant, however, and can be best addressed by placing the gel permeation procedure in its proper context in an overall preparatory scheme.

The gel permeation chromatography of crude extract concentrates serves two purposes: 1) the removal of gross lipid and other large neutral organic interferences from the sample and 2) fractionation of the sample in a way that permits convenient subsequent treatment, such as direct instrumental analysis or further prefractionation, derivatization, or other necessary procedures. In the context of priority pollutant analysis, highly critical separations are the lipid contaminants and fatty acids from the sample, particularly from the phenolic priority compounds. In general, the separation of acidic from neutral chemical compounds can be readily accomplished by a variety of techniques. Ion exchange is one commonly used method, and experimental results and at the University of Washington suggest that coupled chromatography on potassium or cesium silicate is successful at differentiating organic acids from neutral compounds. Since this type of separation is readily accomplished after the GPC step, the more limited goal of separating neutral interferences from neutral priority compounds, and acidic inter-

Experiment:	A	B	C	D	E	F
Solvent (% in methylene chloride)	50,cyclohexane	75,cyclohexane	35,cyclohexane	50,pentane	50,pentane	50,pentane
Sample	Standard Mix	Standard Mix	Standard Mix	Lipid Mixture	Phenols, phthalates, fatty acids	Standard Mix
BioBeads:						
SX-2	x	x	x	x	x	x
SX-3	x	x	x			
SX-8	x	x	x			
SX-12	x	x	x		x	x

Table 4-2 Summary of gel permeation experiments

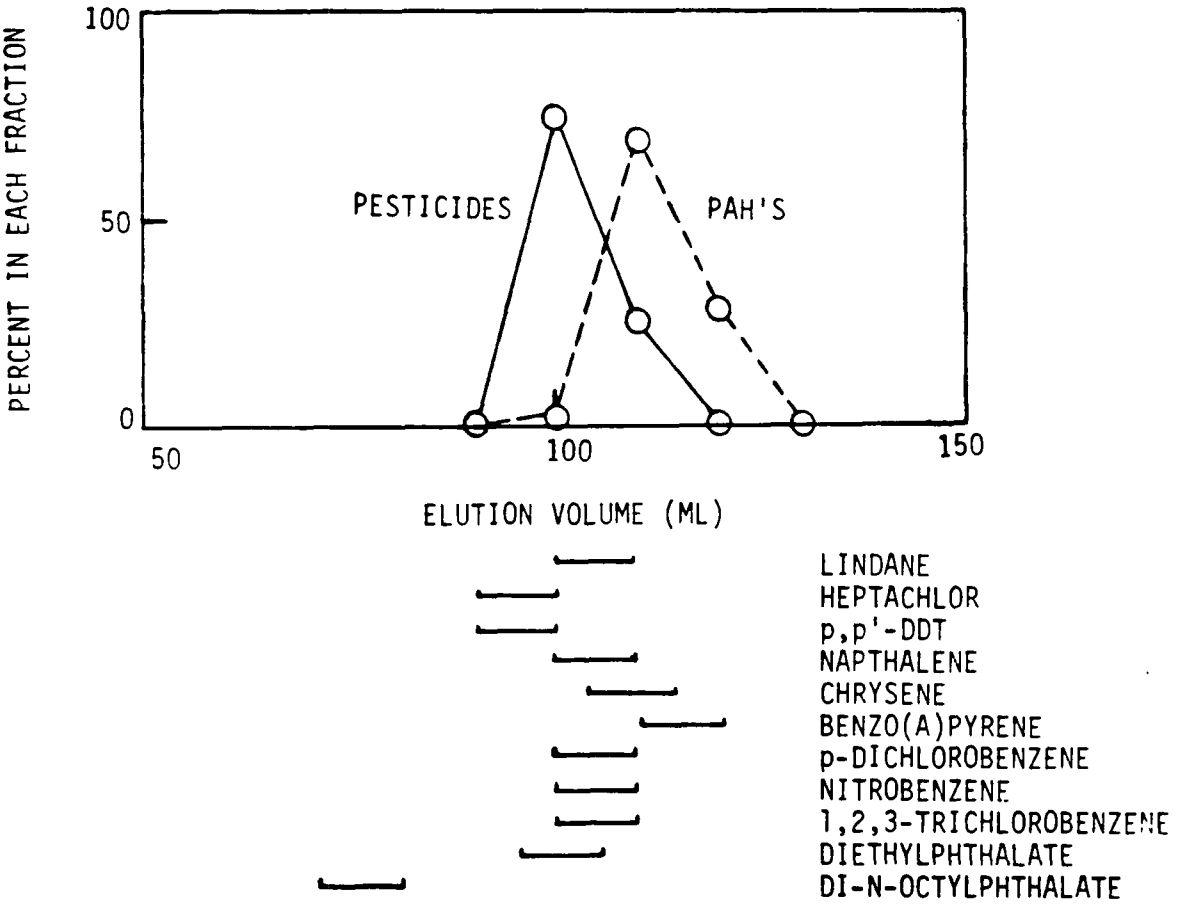


Figure 4-1 Elution Profile on BioBeads S-X2A System

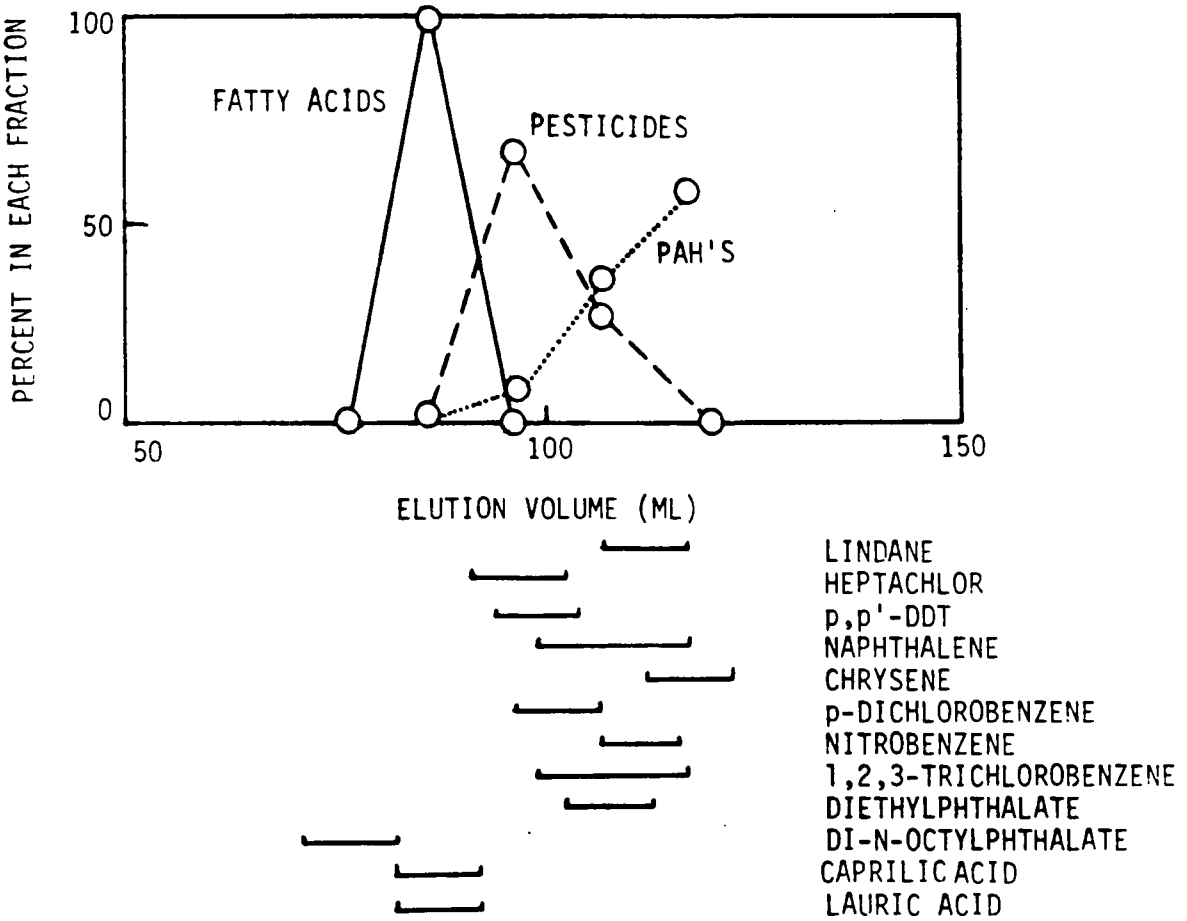


Figure 4-2 Elution Profile on BioBeads S-X2B System.

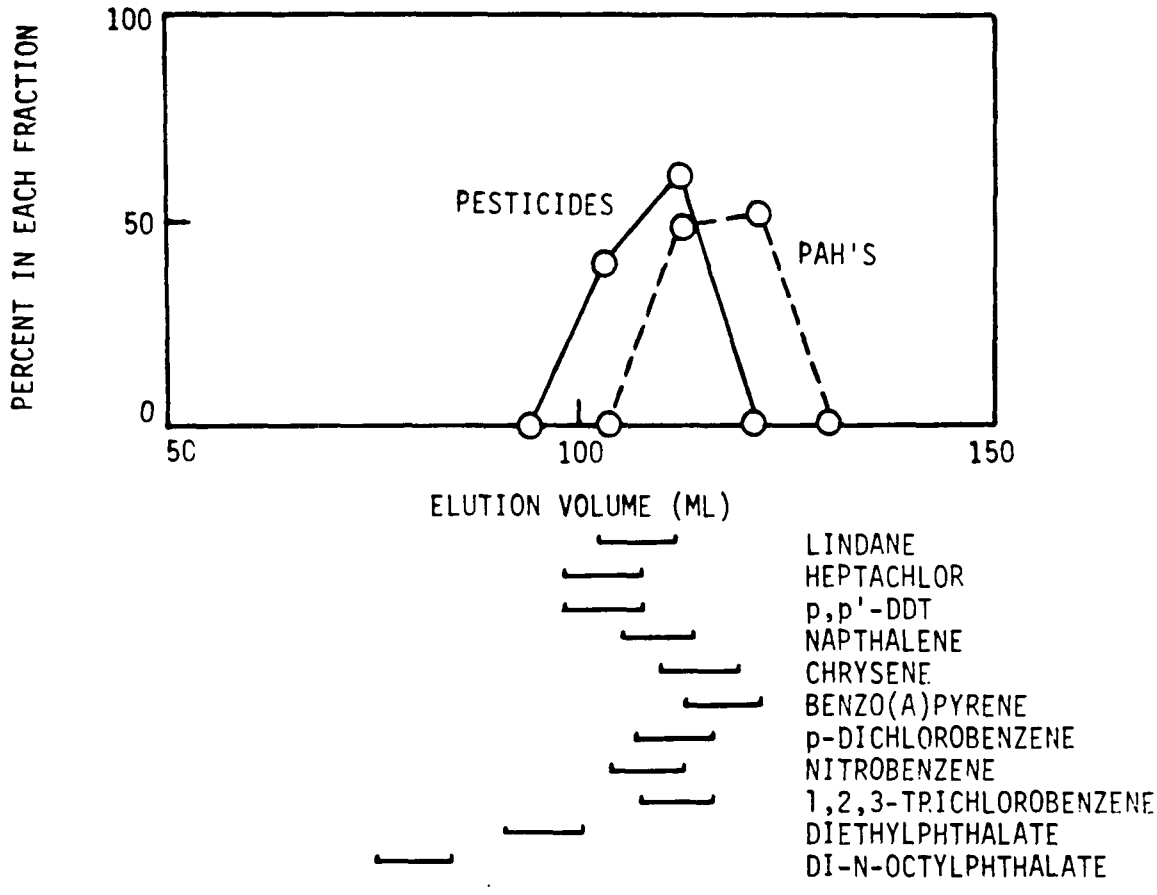


Figure 4-3 Elution Profile on S-X2C System.

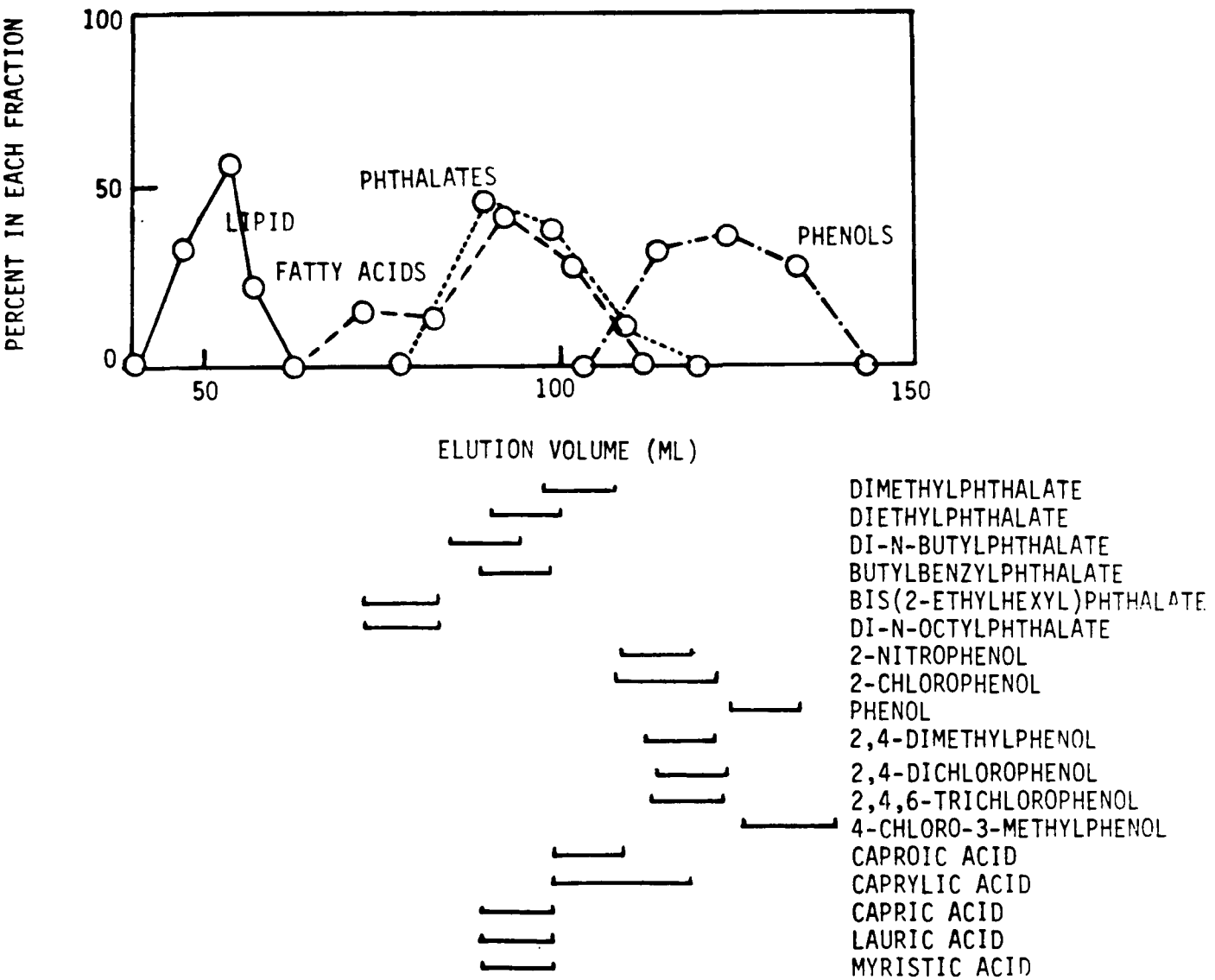


Figure 4-4 Elution Profile on BioBeads S-X2E System

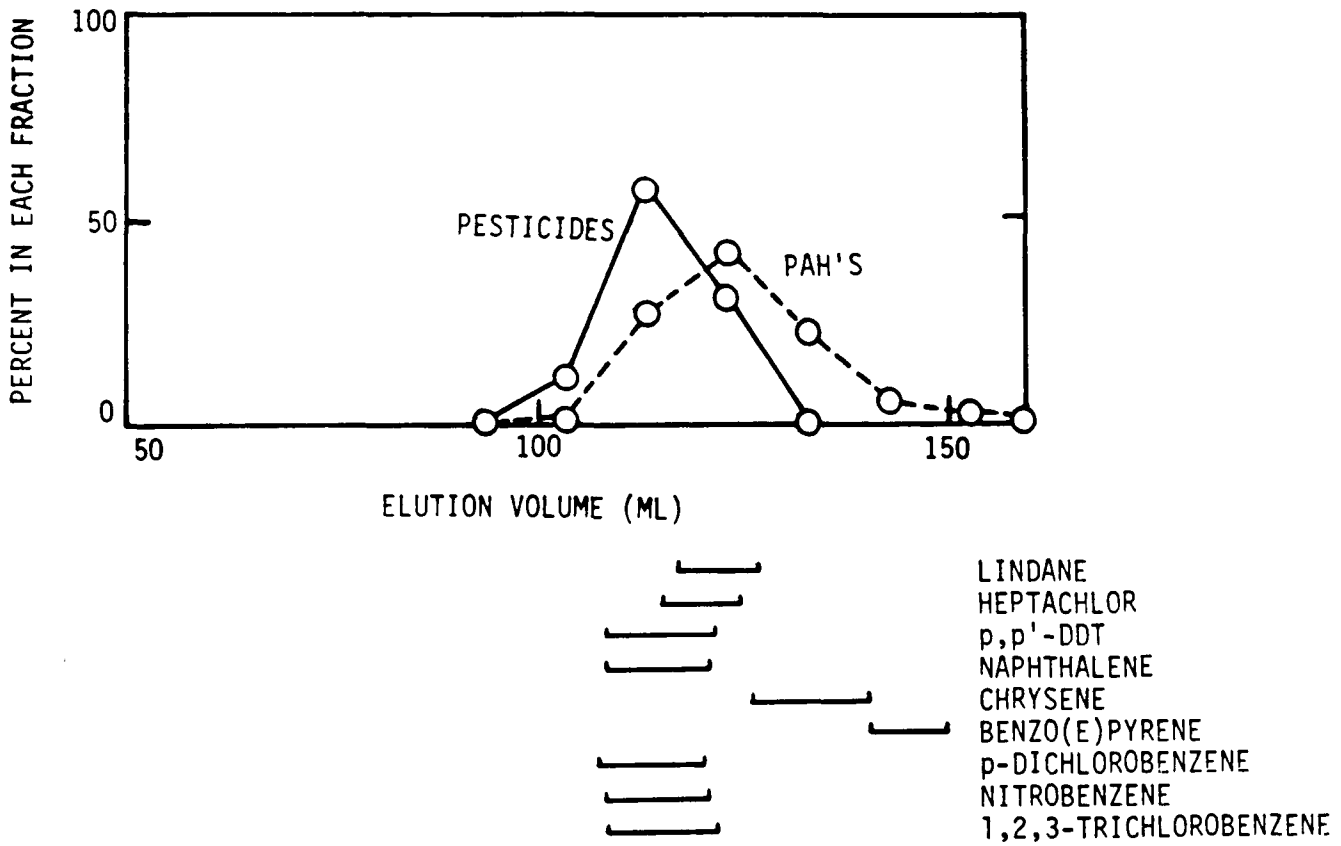


Figure 4-5 Elution Profile on BioBeads S-X2F System.

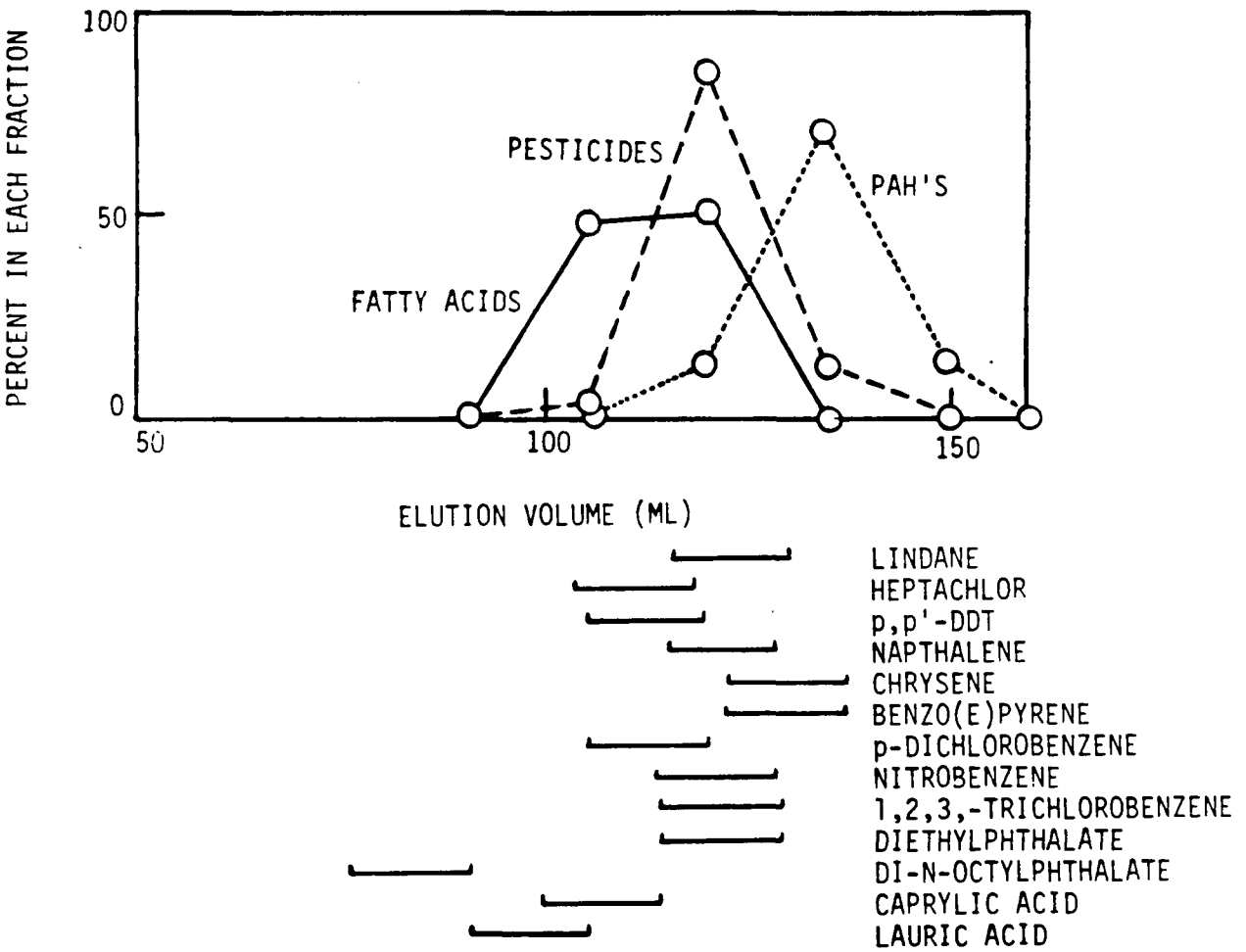


Figure 4-6 Elution Profile on BioBeads S-X3A System.

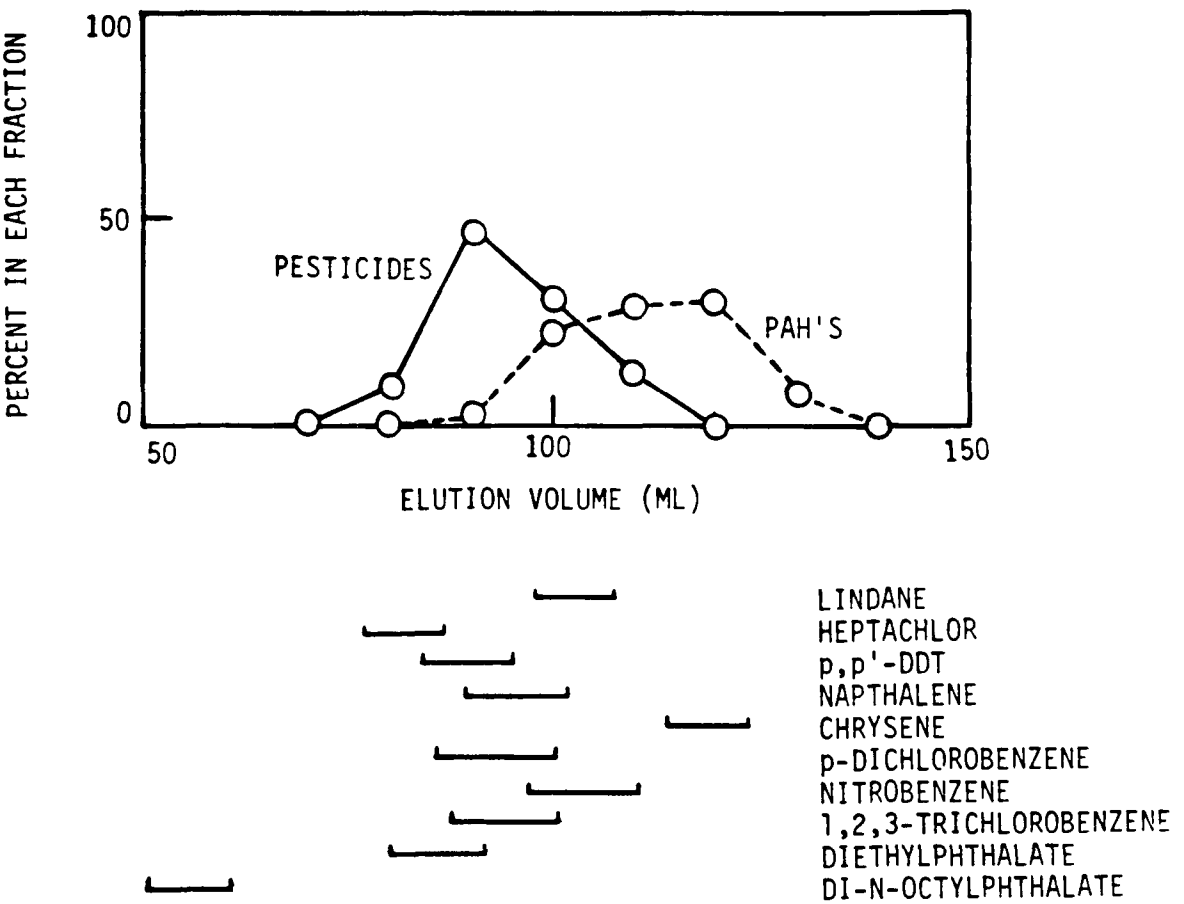


Figure 4-7 Elution Profile on BioBeads S-X3B System.

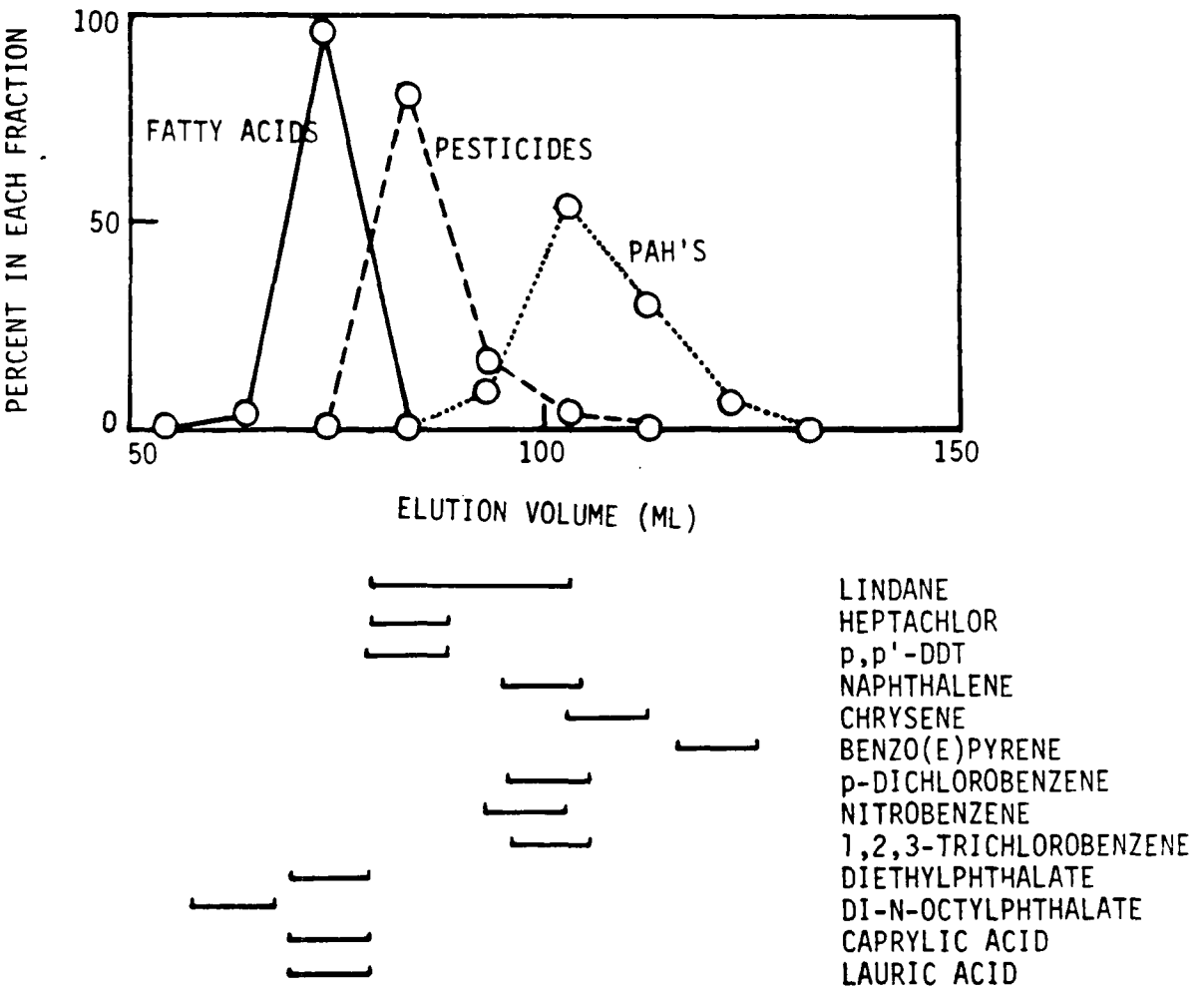


Figure 4-8 Elution Profile on BioBeads S-X8A System.

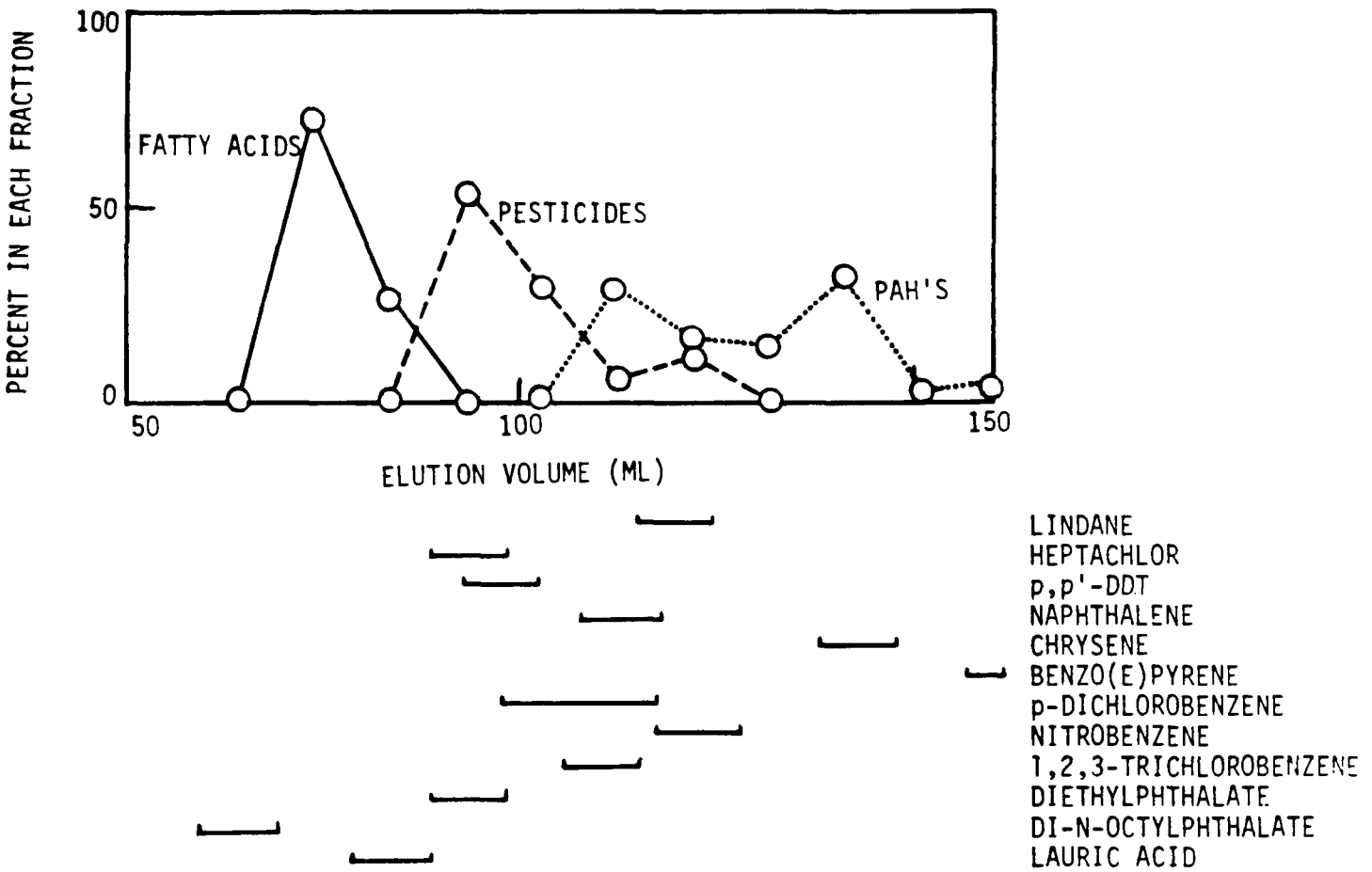


Figure 4-9 Elution Profile on BioBeads S-X8B System.

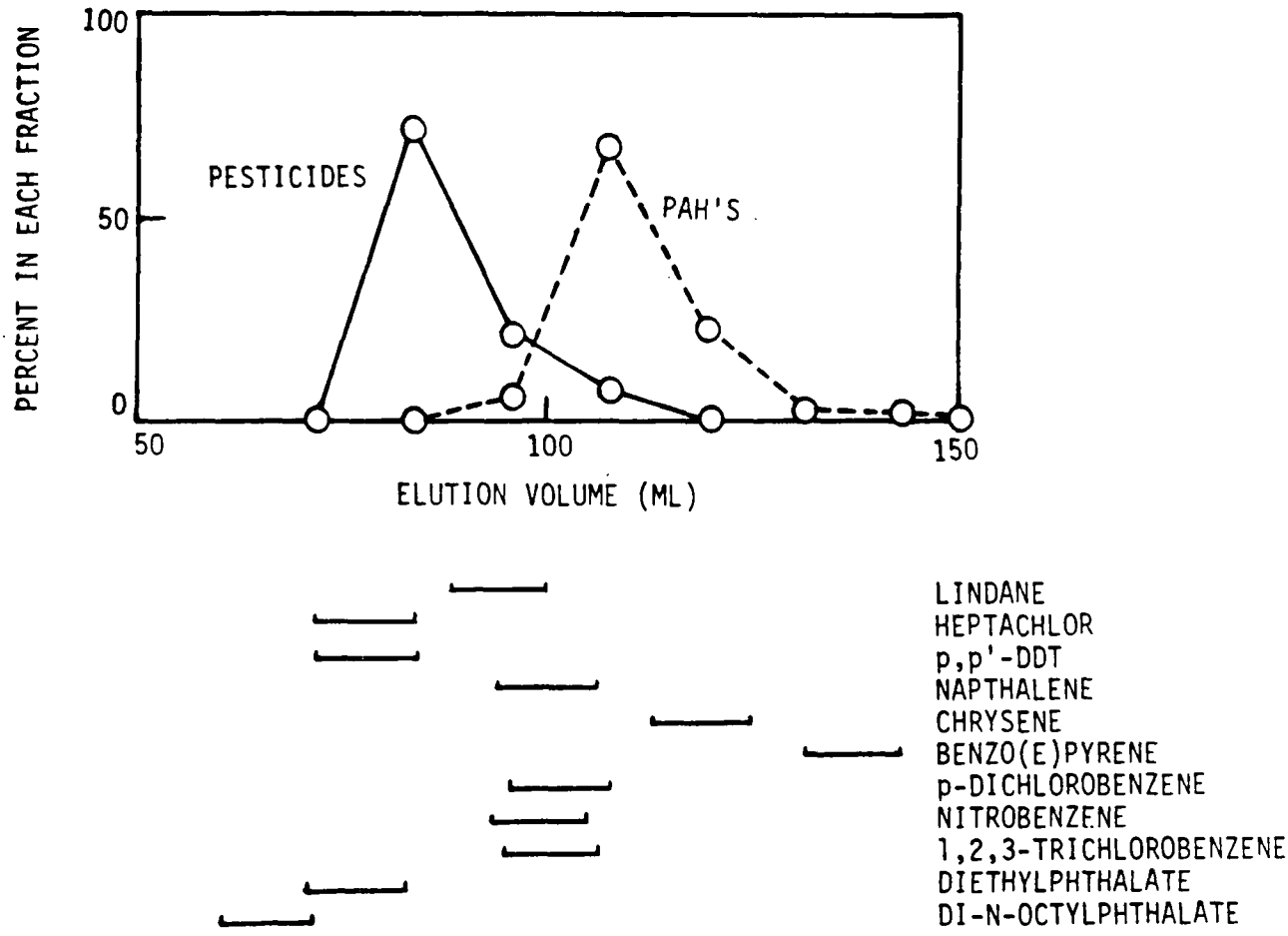


Figure 4-10 Elution Profile on BioBeads S-X12A System.

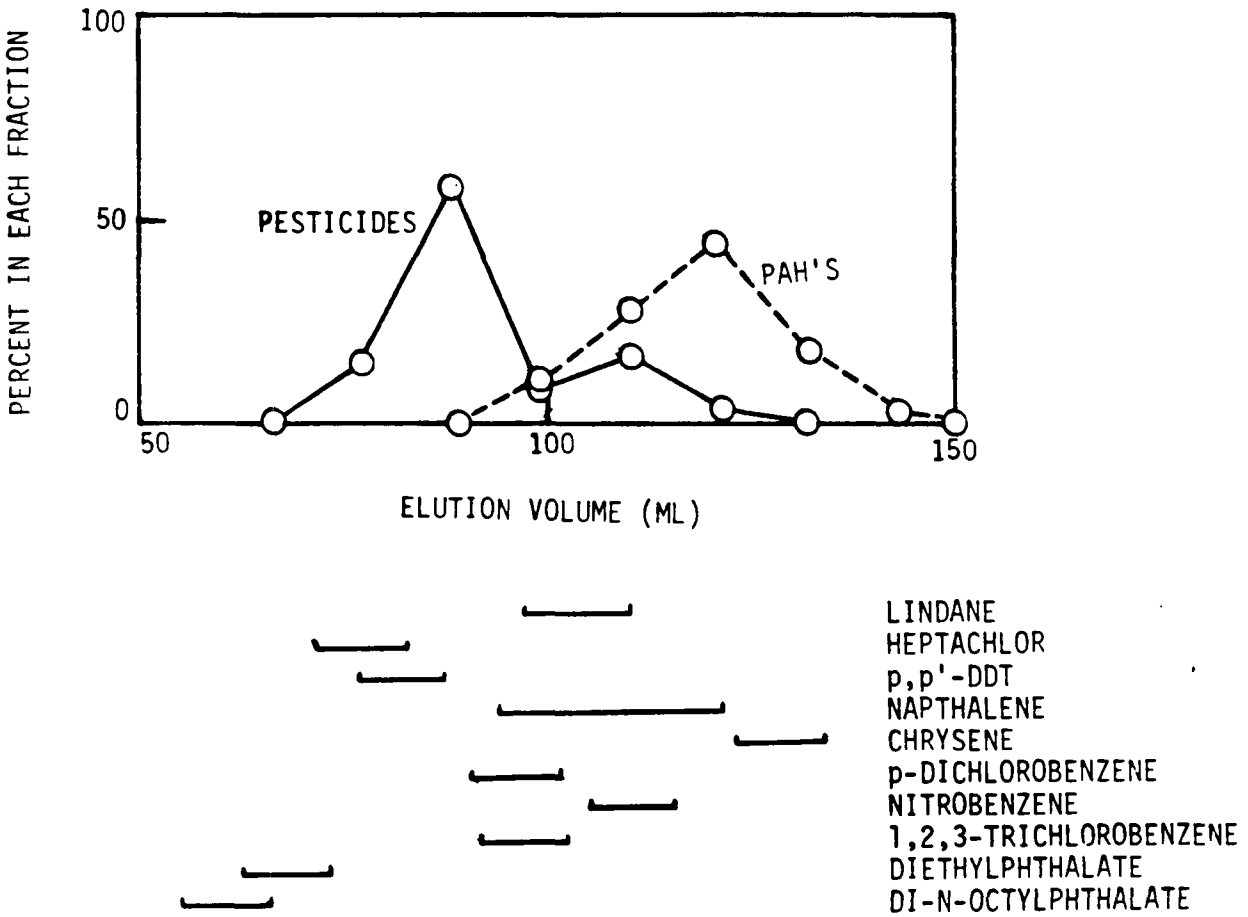


Figure 4-11 Elution Profile on BioBeads S-X12B System.

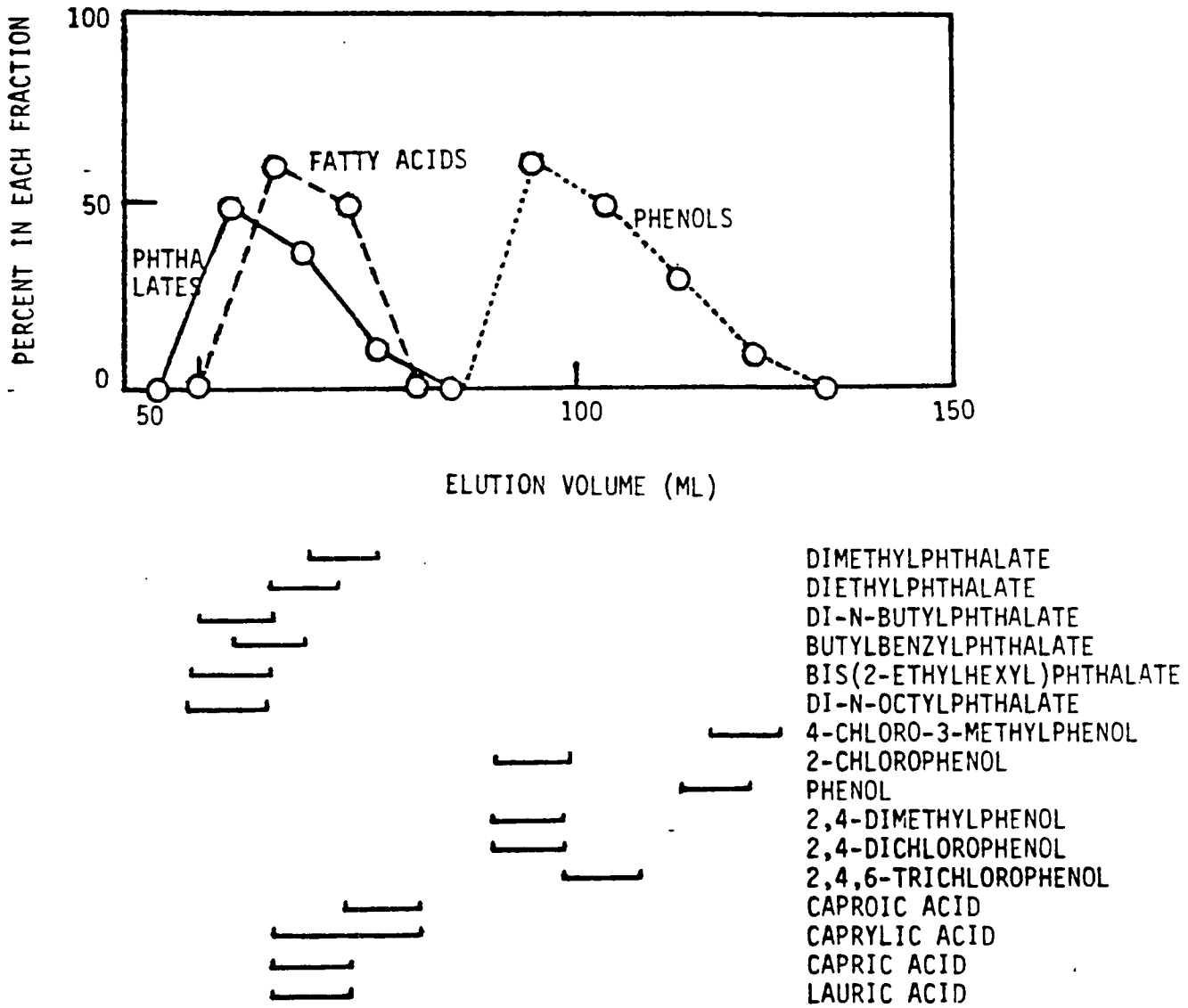


Figure 4-12 Elution Profile on BioBeads S-X12E System.

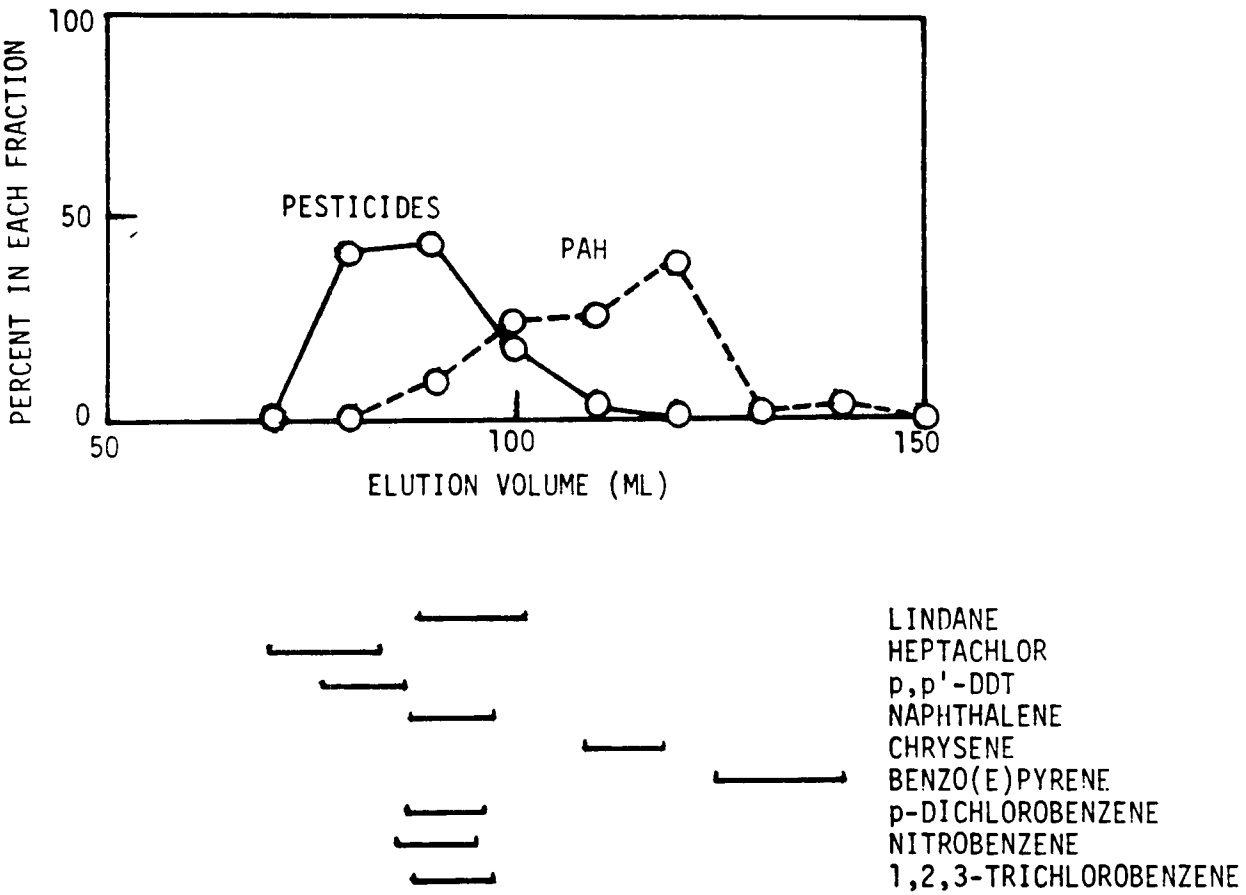


Figure 4-13 Elution Profile on BioBeads S-X12F System.

ferences from acidic priority compounds, will constitute a successful gel permeation chromatographic cleanup step. Therefore, the BioBeads data should be used for the separation of lipid from neutral priority pollutants and fatty acid components from phenolic priority compounds.

The separation of fatty acids from phenols is demonstrated in Figure 4-12 for BioBeads SX-12. This grade of gel showed the best separation of acidic compounds in all experiments. The fatty acids used in the study do not represent the entire molecular weight range that might be encountered, and shorter-chain homologs in particular are expected to elute later, and therefore closer, to the phenols.

The separation of lipid and other neutral interferences, as modeled by corn oil, from the neutral priority compounds is best achieved by SX-2 BioBeads. These interfering compounds are uniformly the earliest eluting and present separation difficulties only with the earliest eluting priority neutrals, such as the phthalate esters and possibly some of the pesticides. Among the compounds in the experimental mixture, diocytyl phthalate elutes the earliest in every case. Previous studies (Stalling, 1972) characterize the separation observed between lipid (fish extracts) and DDT for a variety of BioBeads grades and solvents. The results obtained in the present set of experiments correlate well with the observations of Stalling: the separation of lipid and pesticides decreases with decreasing exclusion limit of the BioBeads. Figures 4-4 and 4-5 present the best example of this separation. In the SX-2 experiments, we find that there is significant overlap between the fatty acids and the phenols, phthalates, pesticides, and PAH compounds.

In general, the data presented in these figures show that solvent mixtures with a greater proportion of cyclohexane or pentane exhibit increased selectivity on a molecular size basis, possibly with a superimposed electronic effect for aromatic molecules. The effect of changing the grade of BioBeads is a complex one. Empirically, the effect is increased separation of fatty acids from phenols, smaller elution volumes, and increased sorting within compound groups as the exclusion limit decreases.

The variation of elution behavior seen among the different classes of compounds with changes in solvent and exclusion limit reflects the interaction of two basic chromatographic effects in this type of gel permeation chromatography: 1) adsorption effects and 2) steric or molecular sieving effects. The apparent exclusion limits assigned to grades of BioBeads will vary with the solvent.

A more detailed analysis of the elution behavior of most of the neutral and acidic extractable priority pollutants has been obtained for SX-2 and SX-8 BioBeads, as shown in Table 4-3. The latter grade was emphasized over SX-12 because of its use by Battelle and GIT in treating crude sludge extracts. These elution profiles, and subsequent experiments, suggest that for removal of gross biological contaminants, SX-2 is superior to SX-8. Plate VI and VII show the columns in use.

4-2 Florisil Chromatography

The desired separation for florisil LC cleanup consists of removal of saturated hydrocarbon material and removal of biological contaminants, including fatty esters, glycerides and sterols. Initial experiments yielded poor recoveries of phthalates and isophorone, even upon elution with 100% ether. Various levels of aqueous deactivation were therefore examined to improve chromatographic behavior. Table 4-4 presents the results of several such experiments. The Florisil columns were prepared by overnight activation at 140^0 C, deactivation in a sealed bottle with the appropriate weight percent of distilled water, followed by at least 24 hours equilibration with periodic shaking. The separations were run on a 0.8 cm x 9" column, dry-packed with the florisil and rinsed with pentane prior to sample loading. The samples were loaded in hexane and eluted with 40 mls pentane, followed by ether/pet. ether step gradients in increments of 200 mls.

The results presented above indicate that substantial amounts of pesticide (in particular Aldrin and Hexachlorobenzene) appear in the first pentane fraction. This fraction contains the saturated hydrocarbon components of the sample. Only in the case of 0.75% and 0.50% deactivation, with some volume reductions in the pentane eluate, could a clean separation of pesticide and saturated hydrocarbon be achieved. For this reason, 0.75% deactivated florisil was selected for further studies. Figure 4-14 illustrates the elution behavior of this activity of florisil with a prechromatographed extract of anaerobic digested sludge plus spike sample. The spike consists of 100 ul each of hexochlorobenzene, lindane, aldrin, DDT, naphthalene, and all phthalates. From this figure, it is clear that during the florisil separation, some interfering material will co-elute along with priority compounds, and that further subdivision of the eluent will not permit complete separation of those contaminants. These contaminants contain some steroidal material, as identified by GC/MS/DS analysis. Depending upon the levels of these compounds, the use of florisil may not be sufficient to provide an adequately cleaned sample for all sludges. However, the florisil procedure is adequate for cleanup of sewage samples. The data shown later indicate that the majority of neutral priority compounds are divided between the florisil-treated fraction and the silicate-treated fraction. For this reason, quantitation of most

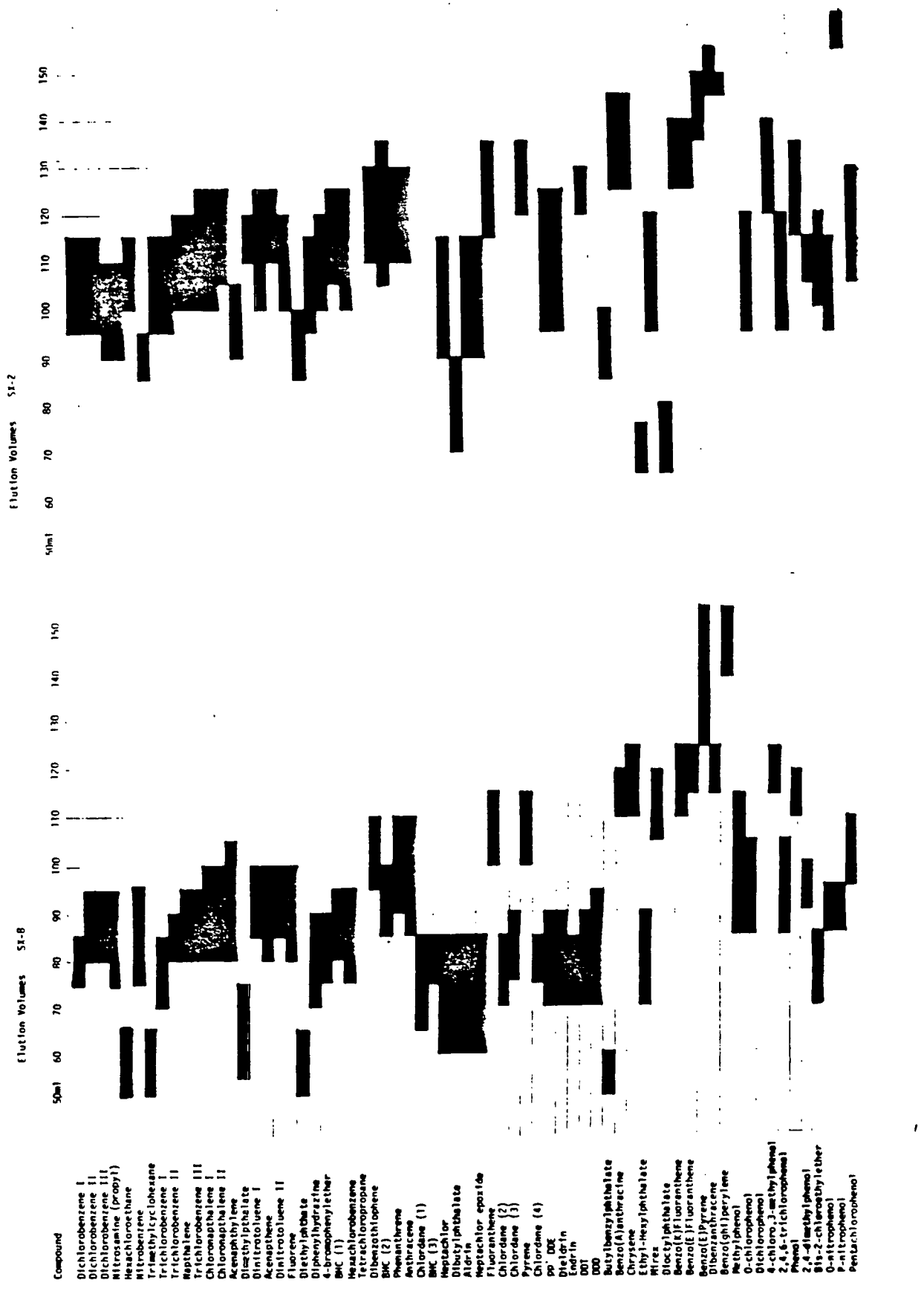


Table 4-3 Elution Behavior of Priority Compounds During Gel Permeation

PLATE VI BIOBEADS GEL PERMEATION CHROMATOGRAPHY

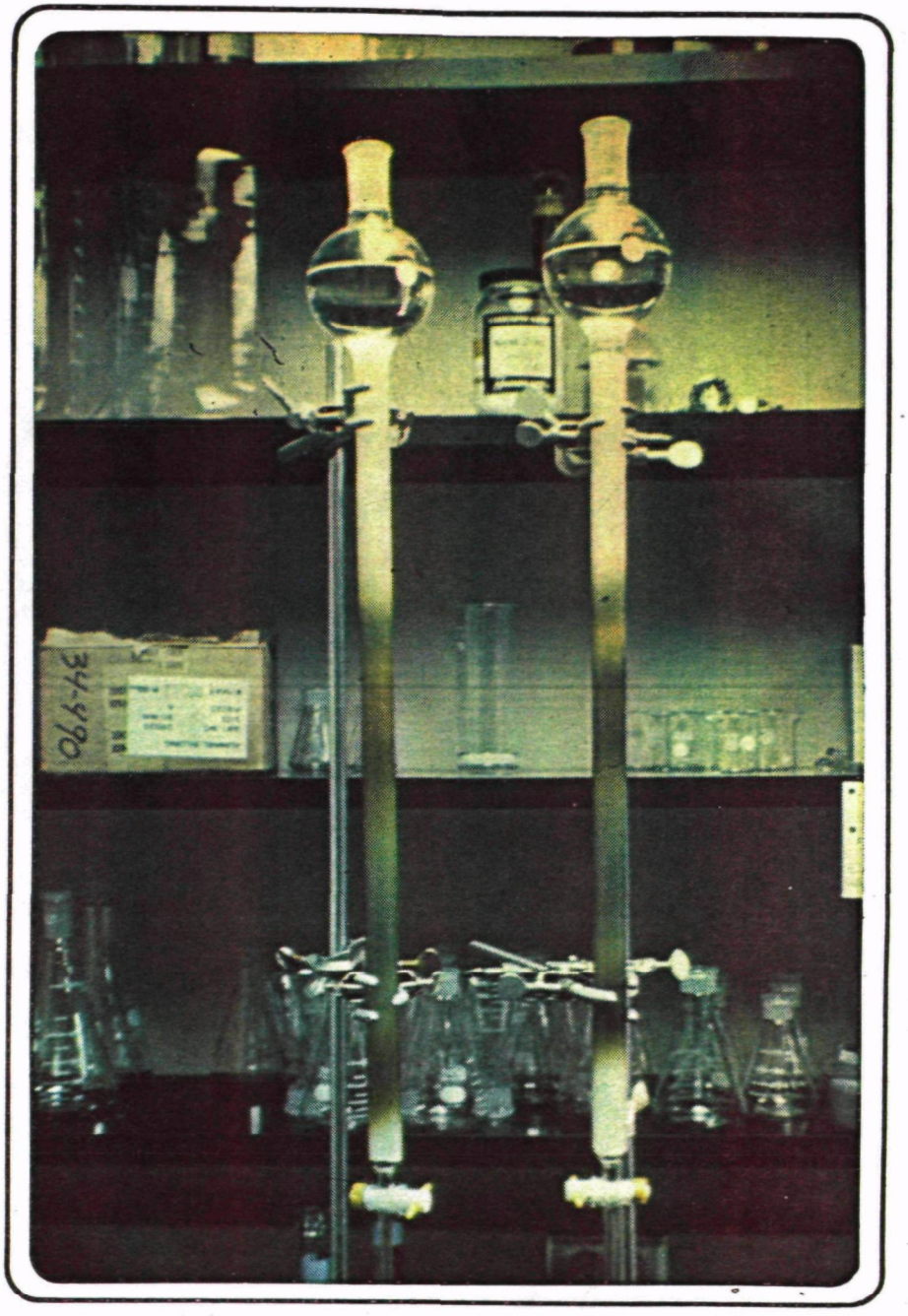


PLATE VII BIOBEADS GEL PERMEATION CHROMATOGRAPHY (LONG WAVE UV LIGHT)

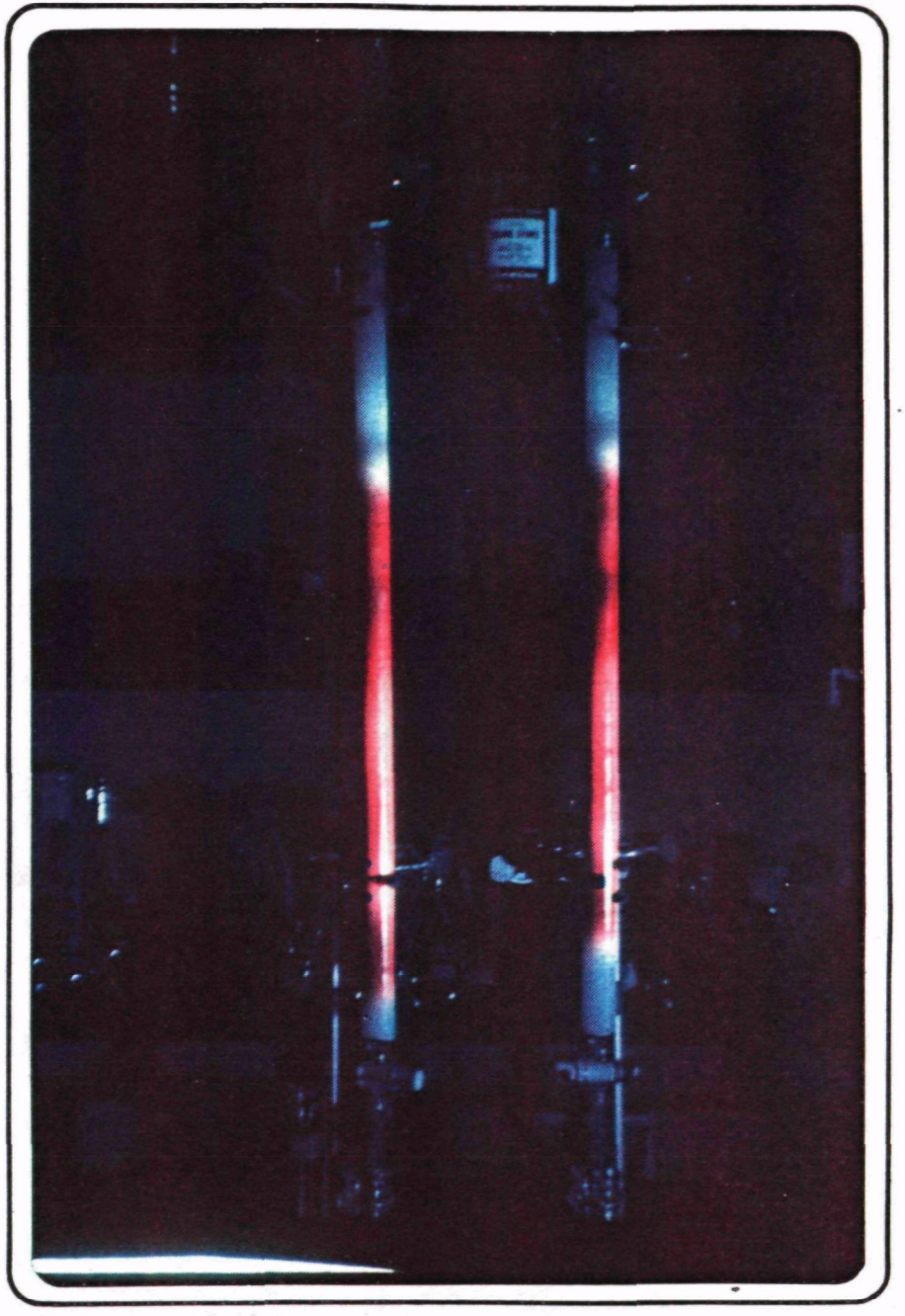


Table 4-4 Elution behavior of selected compounds during florisil chromatography

Percent Deactivation	Eluant	Isophorone	Napthalene	HMB	Dimethyl phthalate	Hexa-chloro-benzene	Hepta-chlor	Aldrin	Diethyl phthalate	Dibutyl phthalate	Heptachlor epoxide	pp' DDT	Butyl-benzyl-phthalate	Ethyl-hexyl-phthalate	Dioctyl phthalate
5%	Pentane		+	+		+	+	+				+			
	15% ether	+	+	+	+	+	+	+	+	+	+	+	+	+	+
	25% ether														
	100% ether														
3%	Pentane		+	+		+	+	+							
	15% ether		+	+		+	+	+	+	+	+	+	+	+	+
	25% ether	+			+										
	100% ether														
1.5%	Pentane					+	+	+							
	15% ether		+	+		+	+	+		+	+	+	+	+	+
	25% ether	+			+				+	+			+	+	+
	100% ether														
0.75%	Pentane					+		+							
	15% ether		+	+		+	+	+		+	+	+		+	+
	25% ether				+				+	+			+	+	+
	100% ether	+			+										
0.5%	Pentane					+		+							
	15% ether		+	+		+	+	+		+	+	+			
	25% ether	+			+				+				+	+	+
	100% ether														

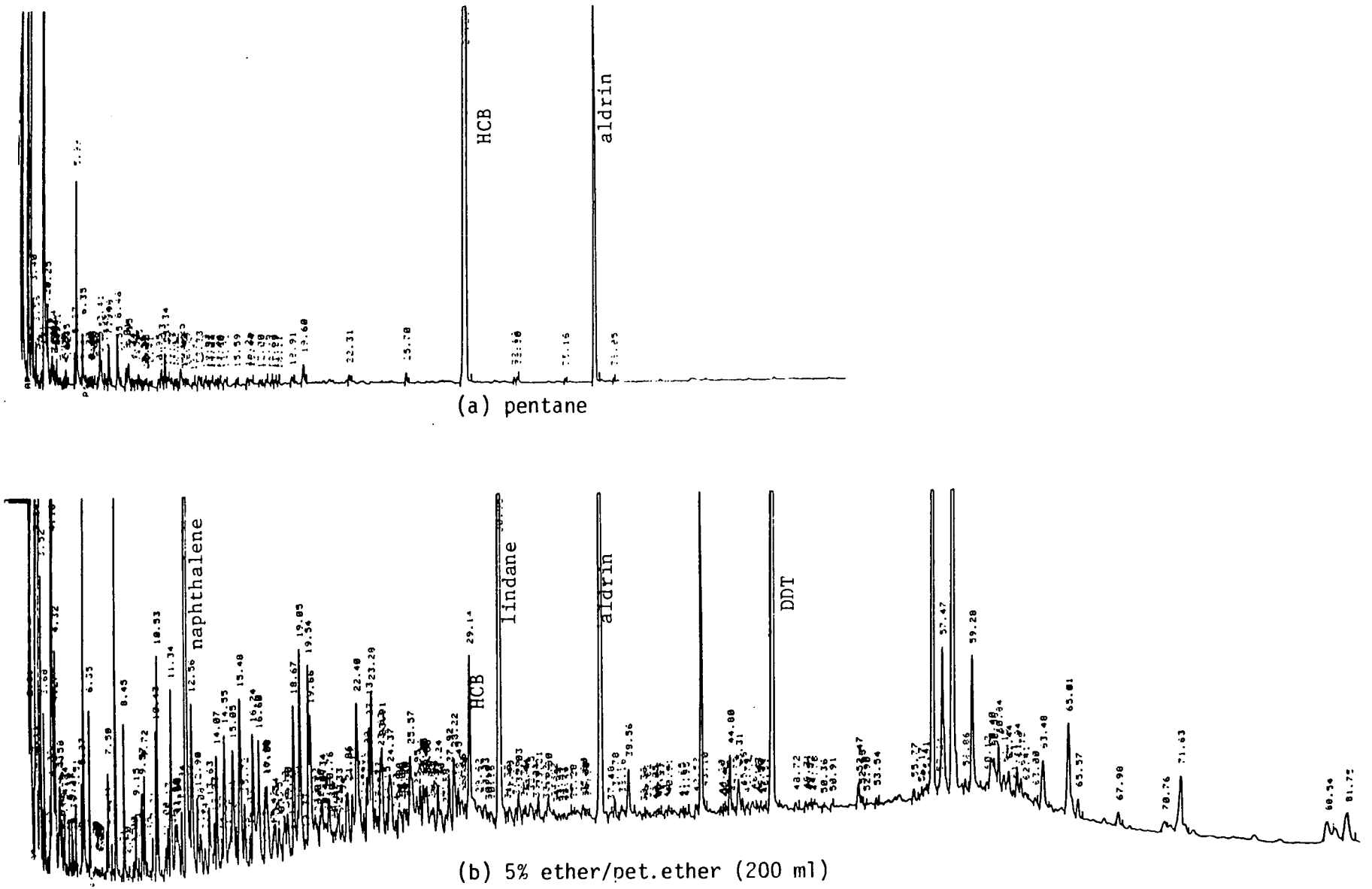
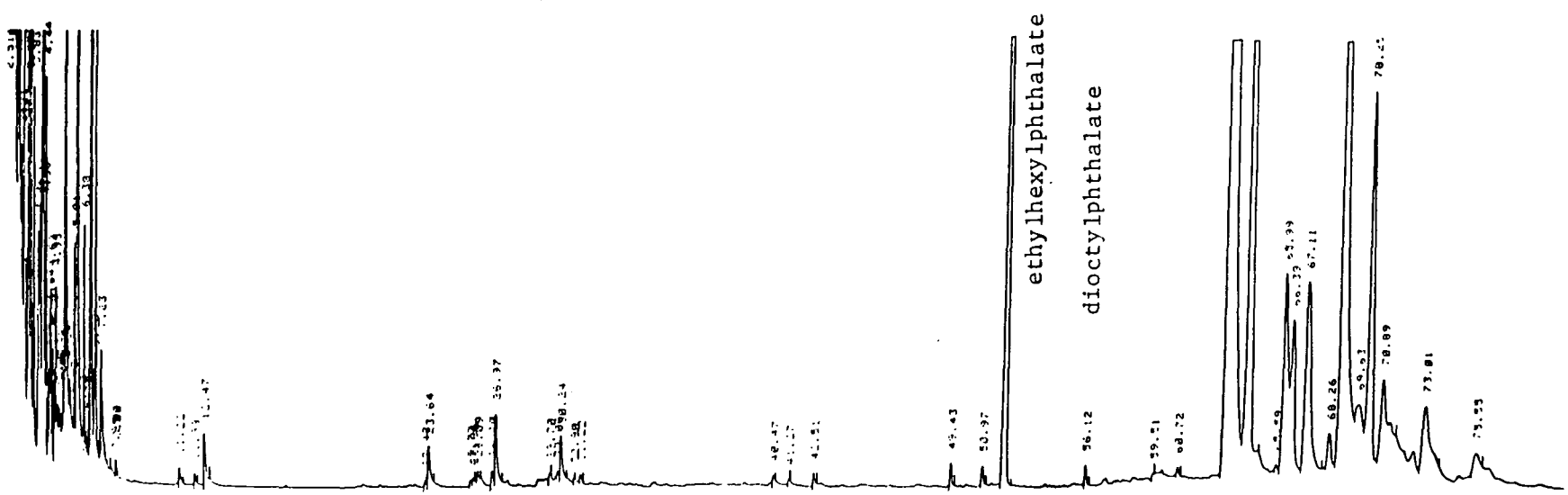


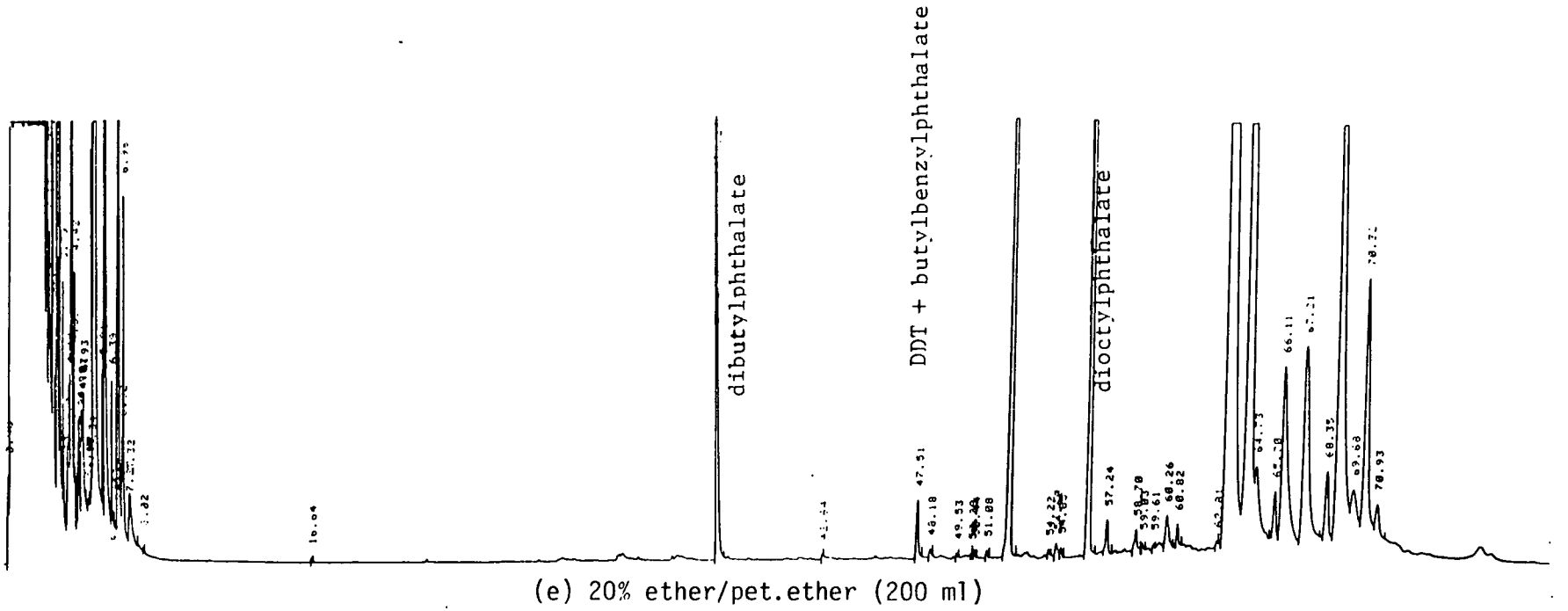
Figure 4-14 Elution of Priority Pollutants Spiked in Prefractionated Anaerobic Digested Sludge Extract During Florisil Clean-up.

(c) 10% ether/pet.ether (200 ml)

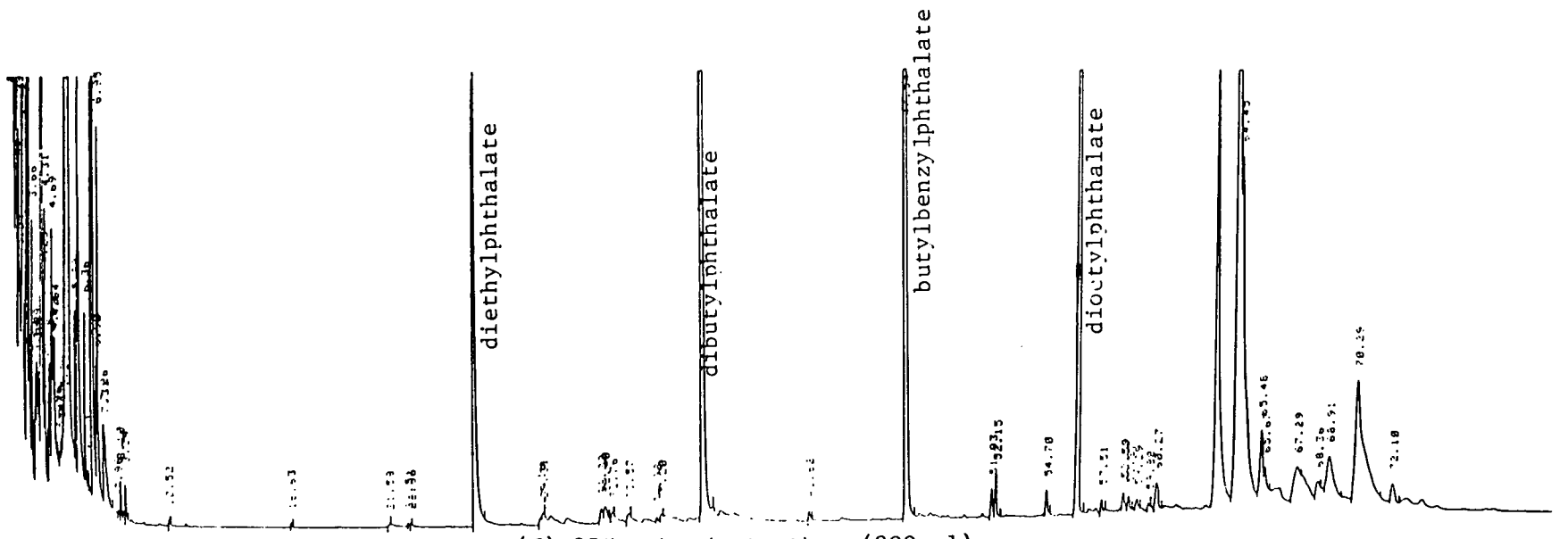


(d) 15% ether/pet.ether (200 ml)

Figure 4-14 Continued

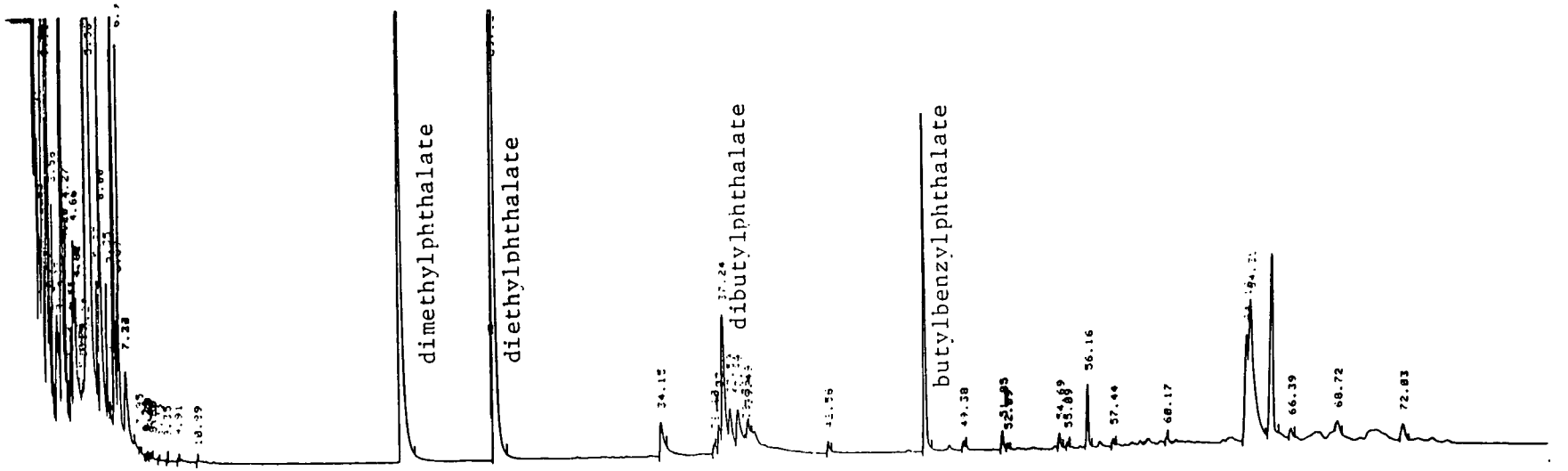


(e) 20% ether/pet.ether (200 ml)



(f) 25% ether/pet.ether (200 ml)

Figure 4-14 Continued.



(c) 35% ether/pet.ether (200 ml)

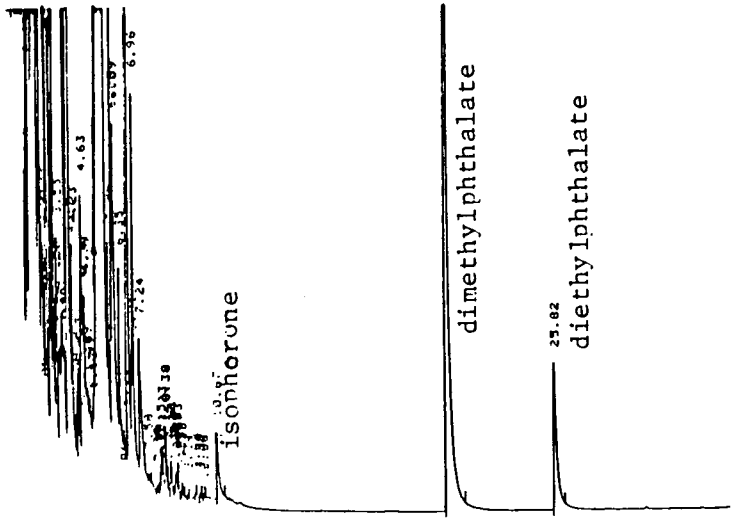
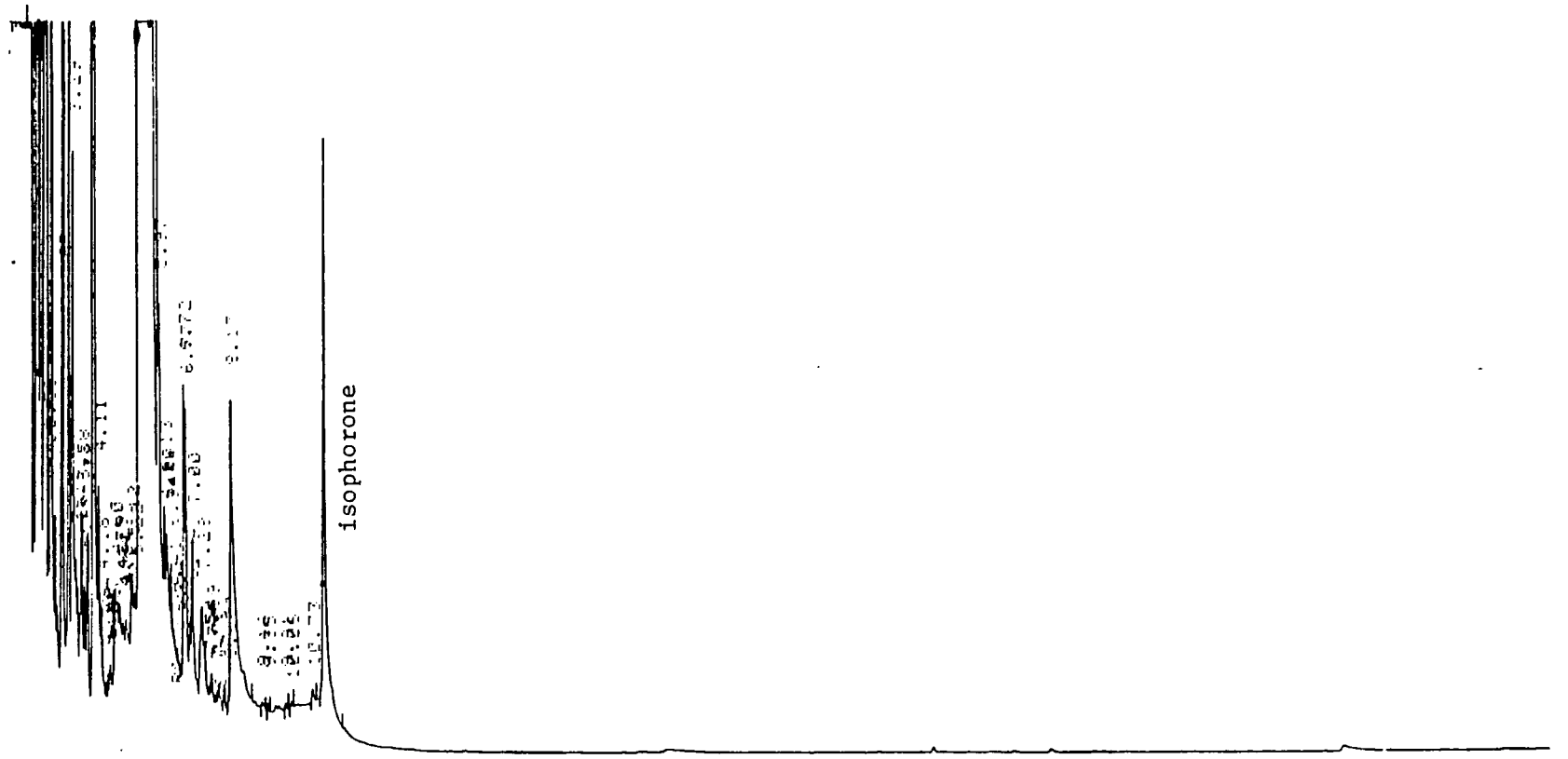


Figure 4-14 Continued.



(i) 100% ether (140 ml)

Figure 4-14 Continued.

of these compounds will require improved cleanup for sludge samples and heavily polluted waste water samples.

SECTION 5

DETERMINATION OF PHENOLIC COMPOUNDS

Phenolic compounds present particular difficulties among the priority pollutants for several reasons: a) they are highly susceptible to chemical degradation, b) they have a high affinity for water relative to most priority compounds, c) they are susceptible to adsorbtive losses, d) they chromatograph poorly, and e) they exhibit nonuniform efficiencies for most derivatizing methods.

5-1 Cleanup Protocols for Phenolic Compounds

The analysis of phenols from extracts in the intergrated scheme will therefore be discussed in three sections---isolation, derivatization, and GC analytical conditions. It should be born in mind that these stages are not separable in a single experiment, as recovery values of necessity depend on successful negotiation of all three steps.

5-2 Isolation of Phenols

The eleven phenols of the so-called "acid fraction" represent 9% of the compounds on the priority pollutant list. The analysis of phenols in sewage is complicated by the considerable abundance of non-phenolic acidic compounds in the sample matrix. These compounds are co-extracted with the phenols at low pH and appear to be primarily fatty acids, and possibly some bile acids and fulvic acids. The much higher relative concentrations of these compounds, compared to the phenols, obscure the detection and quantitation of several of the latter by gas chromatographic methods. Initial developmental work was directed toward sample cleanup and isolation of the phenols from matrix interferences. Two different preparatory schemes were selected for comparison with the standard EPA wastewater analysis method. One scheme utilized ion exchange on Sephadex QAE as adapted from Renberg (1974). The other scheme utilized gel permeation chromatography on BioBeads SX-3 (Stalling, 1972) coupled with flow-through adsorbtion chromatography on potassium silicate (Ramljack, 1977). Based on results reported in the literature, the latter method was investigated because it offered the potential for removal of interfering acidic compounds as well as base-neutral interferences.

Following initial development of chromatographic conditions with standard phenols, experiments were conducted with raw sewage as the sample matrix. A concentrated standard solution of the eleven "priority" phenols in methanol was spiked into 800 ml aliquots of both sewage and blank water samples to give a final spike concentration of 30 ppb per phenol. The samples were analyzed in parallel experiments using the three methods and quanti-

fied with GC (without derivatization except as noted), and detailed in Table 5-1.

The standard EPA method, i.e. adjustment of pH to 12, removal of base-neutral organics by repeated extraction with methylene chloride, acidification to pH 2, and removal of phenols and other organic acids by further methylene chloride extraction. This was accomplished for the sewage and blank samples using silanized separatory funnels and centrifugation where necessary to break emulsions. The acid extractable fractions were concentrated, dried over Na_2SO_4, and analyzed without further prefractionation.

The anion-exchange experiments were conducted using a total organic extract obtained by direct multiple extraction of pH 2 sample with methylene chloride + 10% methanol with centrifugation as necessary. Sephadex QAE, a strong anion exchange gel, was used to isolate acids in the total extract by batch extraction, after Renberg et. al. The acids were subsequently recovered from the rinsed gel by shaking with pH 2 aqueous acid and diethyl ether. The ether extracts were dried over $Na_2 SO_4$ and concentrated to 1 ml for GC analysis.

The gel permeation coupled chromatography experiments used a total organic extract obtained similarly to that used in the ion exchange experiments. The total organic extracts were dried with anhydrous Na_2SO_4, and the solvent replaced with neat methylene chloride by multiple partial evaporation. The extracts were concentrated to 2.5 ml and diluted to 5 ml with cyclohexane. The samples were applied to BioBeads SX-3 columns and eluted with 1:1 methylene chloride/cyclohexane, according to the method of Stalling et. al. After elution of the first fraction (containing much of the lipid and fatty acid components) the effluents were allowed to pass directly through columns of potassium silicate. The latter were prepared from silica gel and KOH-saturated isopropanol according to the method of Ramljack et. al. The latter column retains acidic compounds and phenols while allowing neutral components to pass through. After complete elution of the phenol fractions from the SX-3 onto the potassium silicate, the columns were uncoupled and the potassium silicate rinsed with three column-volumes of methylene chloride. The adsorbed acids were then eluted with 100% methanol. The methanol extracts were concentrated, acidified with 1 N aqueous HCl, and extracted with methylene chloride to recover the phenols. The final extracts were dried over Na_2SO_4 and concentrated for GC analysis.

All final extracts were reduced to 1 ml volume in methylene chloride and 2-secbutyl phenol was added as an external standard. GC analyses were carried out on a Hewlett/Packard 5840A gas chromatograph, using a SP 1240 column. The 1% SP 1240 DA column (3 feet in length and 2 mm in diameter) was held at 70^0C for 1 minute and programmed at a rate of 10^0C/mm till 190^0C, then held

EPA Method (I)

800 ml sewage
extract at pH 12 with
100, 50, 50 ml CH_2Cl_2
↓
extract aqueous phase
at pH 2 with 100, 50,
50 ml CH_2Cl_2
↓
pass methylene chloride
through Na_2SO_4 column
↓
K-D to 10 ml
↓
Microsnyder to 1 ml
↓
GC with SP 1240 DA

Ion Exchange Method (II)

800 ml sewage
extract at pH 2 with
100, 50, 50 ml CH_2Cl_2
↓
K-D to 5 ml
↓
extract into 2, 2, 2 ml
of pH 12 NaOH
↓
equilibrate base with
3 ml of swollen gel
Sephadex QAE
↓
rinse resin with H_2O
↓
shake at pH 2 with HCL/
KCl buffer and 5, 5 ml
diethylether
↓
pass diethylether through
Na_2SO_4 column
↓
Microsnyder to 2 ml, add
5 ml methylenechloride
and reduce to 1 ml
↓
GC with SP 1240 DA

GPC/Adsorption Method (III)

800 ml sewage
extract at pH 2 with
100, 50, 50 ml CH_2Cl_2
↓
pass methylenechloride
through Na_2SO_4 column
↓
K-D to 10 ml
↓
Microsnyder to 2.5 ml
↓
add 2.5 ml cyclohexane
and pass through Biobeads
SX-3 column (1.9 cm diam.,
40 cm length)
↓
elute with 1:1 cyclohexane/
methylene chloride and
discard fraction 0-75 ml
↓
pass fraction 75-125 ml
through 10 g K_2SiO_4
(1 cm diam., 15 cm length)
rinse with two bedvolumes
methylene chloride and elute
with 20 ml methanol
↓
add cyclohexane and
K-D to 2 ml methanol
↓
add 4 ml 1N HCl and extract
with 2, 2, 2 ml methylene-
chloride
↓
pass methylene chloride
through Na_2SO_4 column
↓
Microsnyder to 1 ml
↓
GC with SP 1240 DA

Table 5-1 Different extraction and clean-up procedures evaluated during the present study.

for 10 minutes. The gas flow rate was 30 ml/minute N with an injector temperature of 225°C and a detector temperature of 325°C. The attenuation was 25 with a chart speed of 1 cm/minute.

The EPA scheme presented particular difficulty in the primary extraction phase due to the formation of very stable emulsions at pH 12. This problem is clearly matrix-related, and was not observed for blank extractions. Figures 5-1 and 5-2 present the GC results for the EPA sewage sample and blank, respectively. Although high recoveries of phenols are observed (70-140% where quantifiable), the EPA method is least selective in excluding interfering acids and polar organics.

Figure 5-3 presents the results obtained for QAE ion exchange isolation of phenols from sewage sample. The recoveries, where quantifiable, were generally low (30-90%), presumably due to interfering organic acids overloading the resin. Again, high levels of interfering acidic organics were noted.

The results from the coupled-chromatography isolation scheme are shown in Figure 5-4. Recoveries were determined to be 60-110%. Subsequently, this sample was also treated with methanol/boron trifluoride to derivatize any carboxylic acids present (Figure 5-5). Some interference is again noted.

Problems common to the analysis of phenolics from sewage samples by all three methods are variable recoveries among the different phenols, and quantitation in the presence of high relative concentrations of interfering compounds. Among the methods studied, the EPA method and the coupled chromatography method gave higher recoveries, but in all cases the nitro phenols in particular showed poor recovery. Refinement of extraction and chromatographic procedures (especially in the quantitation phase) is clearly needed.

The selection of methods are based on several criteria for desirable cleanup/prefractionation procedures. First, the procedures should reflect the known chemistry of ubiquitous contaminants in sewage samples. Secondarily, procedures suitable for a unified fractionation scheme for all extractable priority pollutants is desirable. The majority of interfering materials observed has a carboxylic acid functionality and consists predominately of fatty acids. This can be discerned from comparison of Figures 5-4 and 5-5. The interfering contaminant peaks in Figure 5-4 (retention times 9.28, 10.53, 11.73, and 13.10) show the regularity of a homologous series. After derivatization to methyl esters, a uniform shift of these compounds to shorter retention times (3.82, 5.68, 7.21, and 8.81) was observed. This is behavior prescriptive of homologous carboxylic acids. Since these compounds are the dominant interferences separation methods relying solely on acid/base properties will not remove them. Further, although crude extracts containing carboxylic interfer-

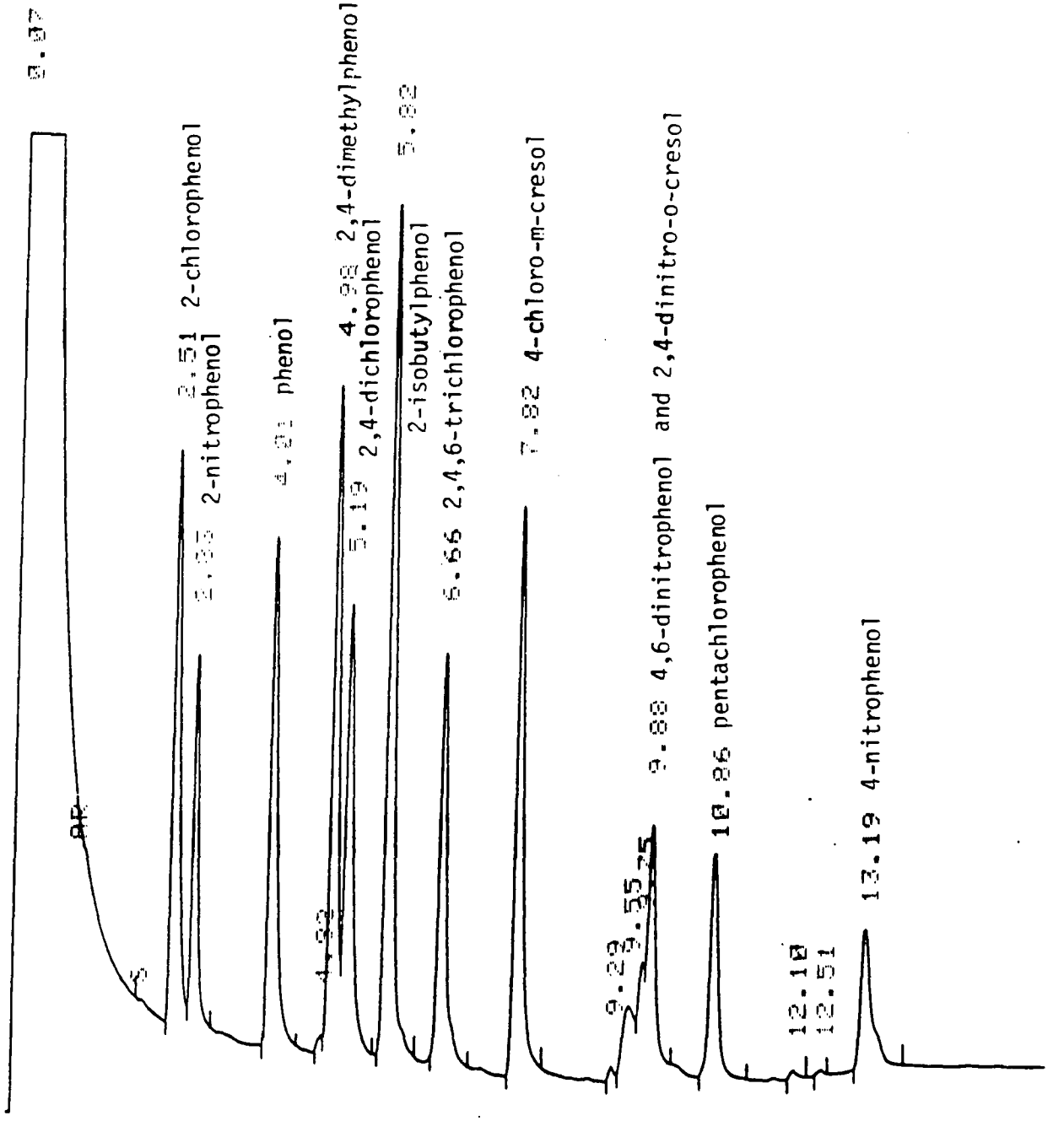


Figure 5-1 Chromatogram of distilled water spiked at 30 ppb phenolics and extracted according to the EPA method.

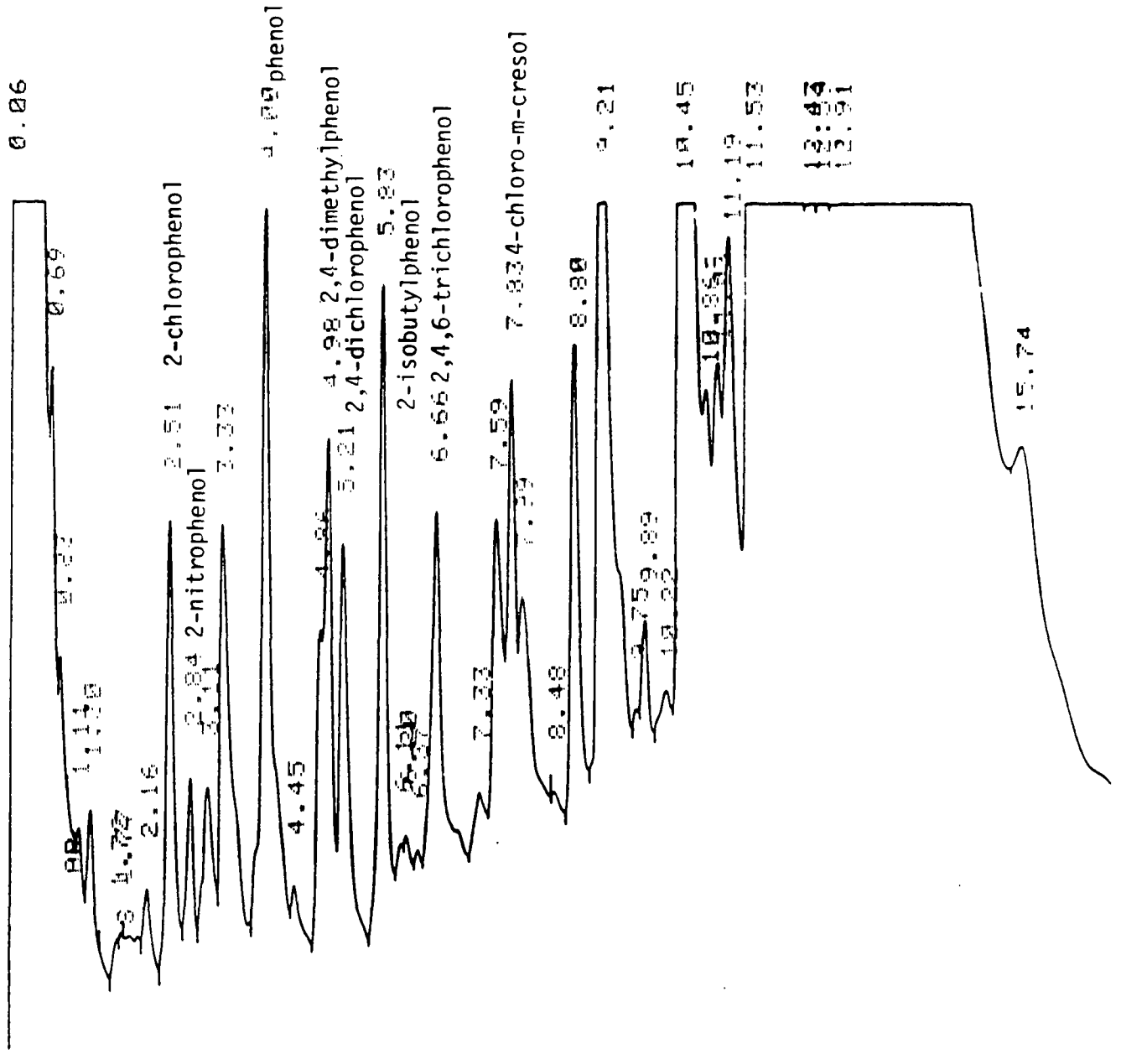


Figure 5-2 Chromatogram of sewage sample spiked with 30 ppb phenolics and extracted according to the EPA method.

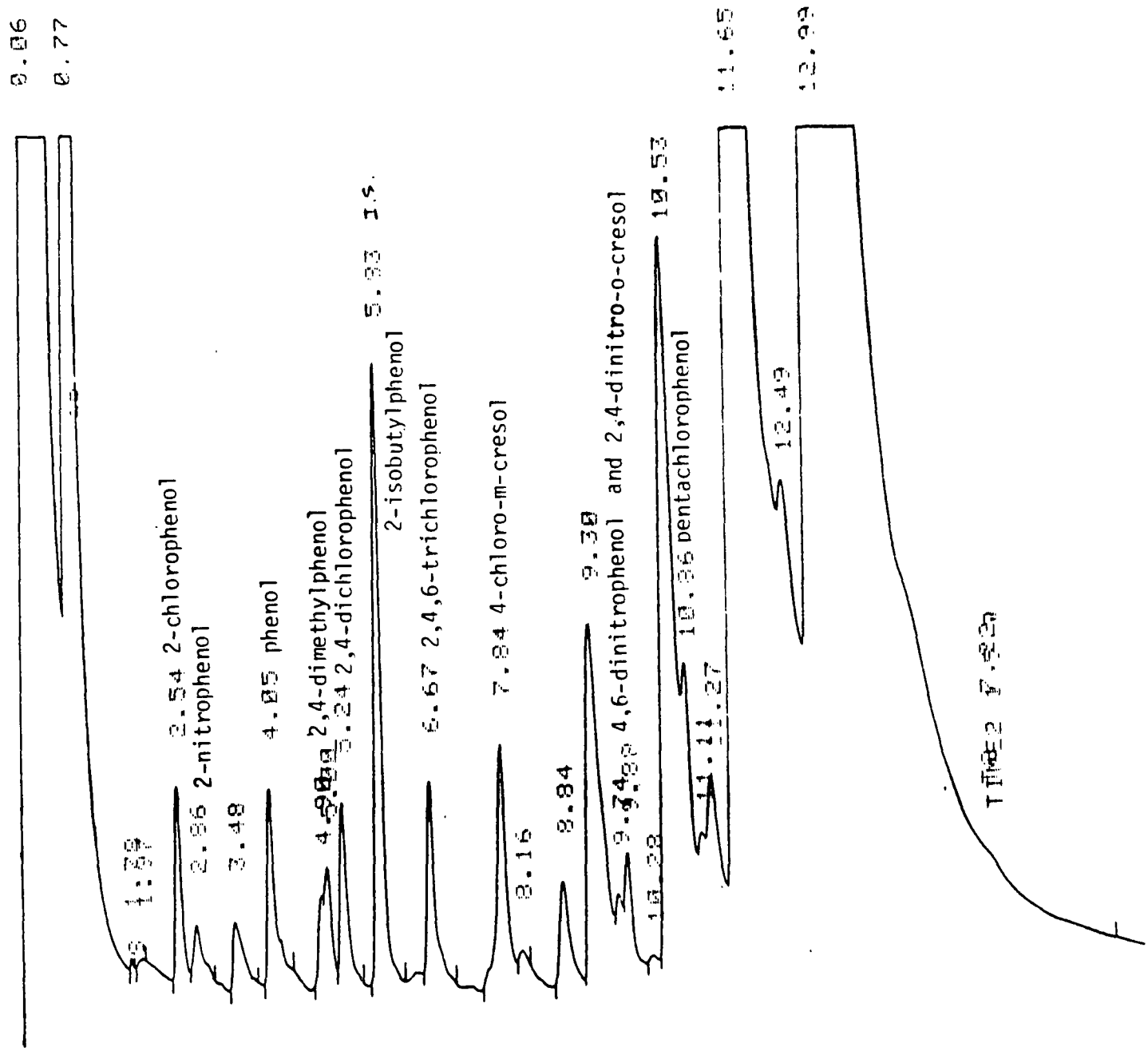


Figure 5-3 Chromatogram of sewage sample spiked with 30 ppb phenolics and extracted and cleaned-up according to the ion-exchange method.

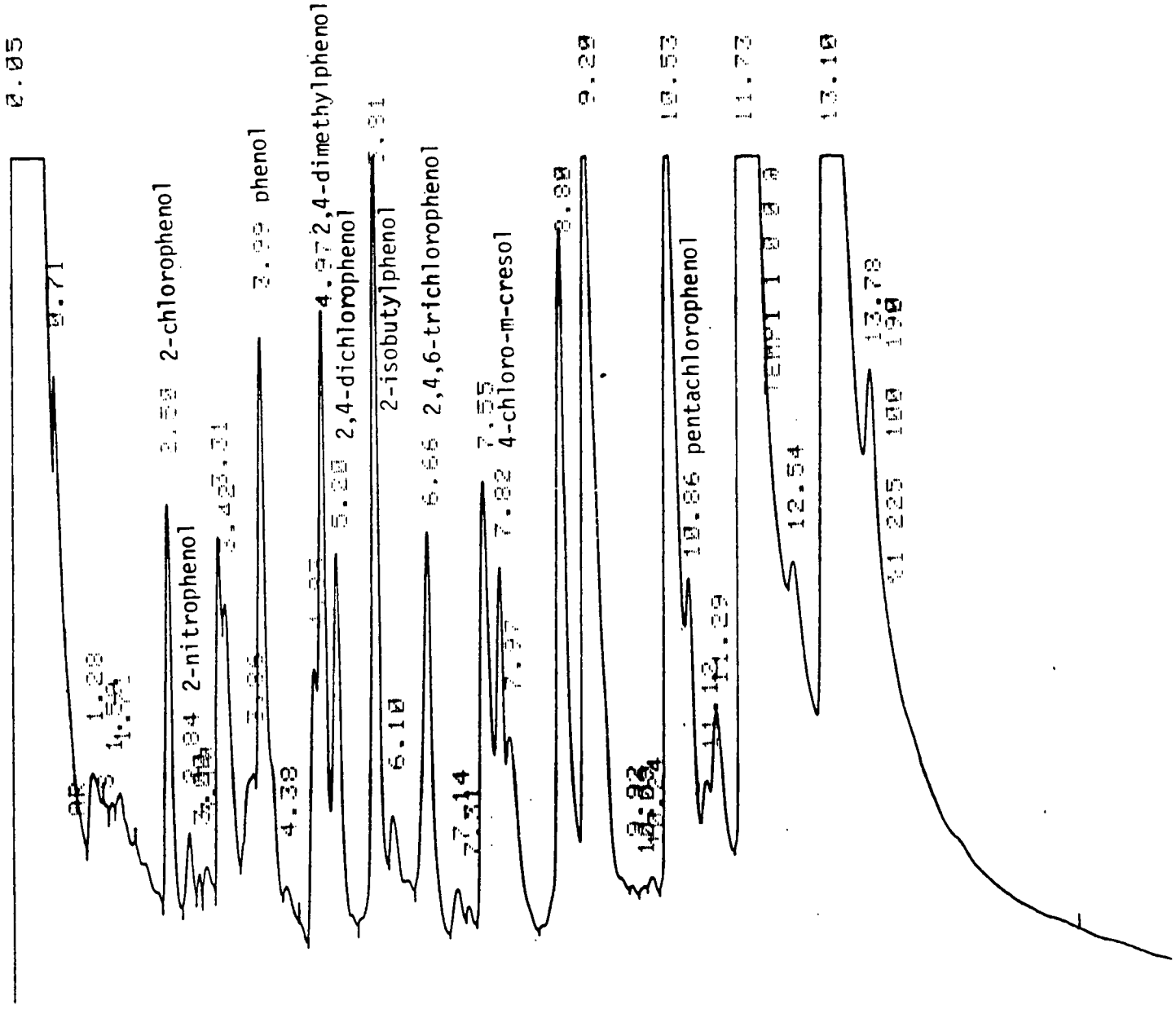


Figure 5-4 Chromatogram of sewage sample spiked with 30 ppb phenolics and extracted and cleaned-up according to the gel permeation method.

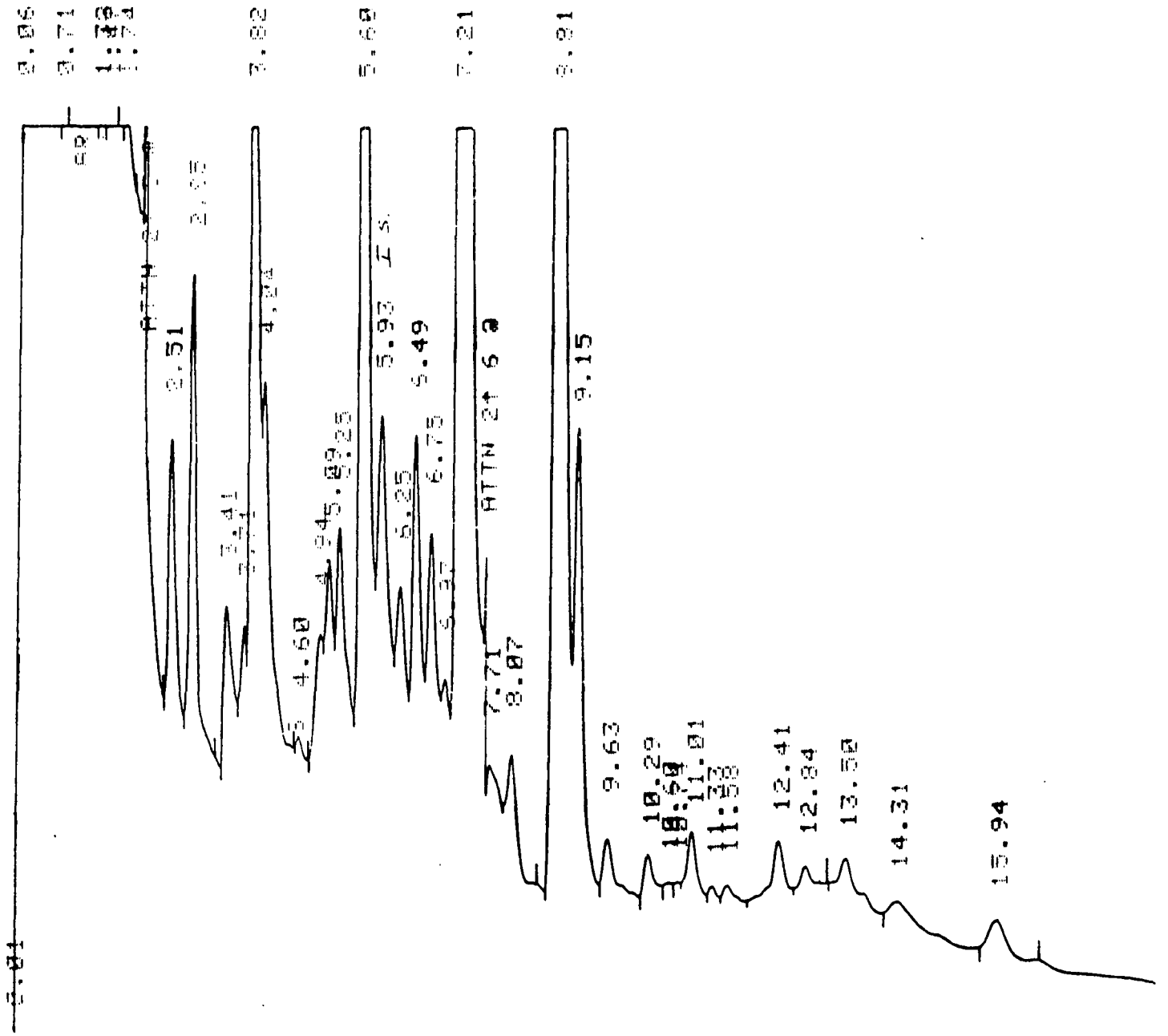


Figure 5-5 Chromatogram of sewage sample spiked with 30 ppb phenolics and extracted and cleaned-up according to the gel permeation method followed by derivatization with BF_3 in methanol.

ences can sometimes be quantified by GC/MS techniques, the high background of unwanted compounds more rapidly contaminates the mass spectrometer and increases the difficulty of data interpretation. Also, these carboxylic interferences adversely affect GC column performance and lifetime.

The results indicate that significant reductions in concentrations of carboxylic and lipid interferences, without great sacrifices in phenol recoveries, are possible using gel permeation chromatography in combination with acid-selective adsorption. Both EPA and coupled column experiments gave good recovery and quantitation for the less acidic phenols (i.e. those eluted early from the GC). The recovery of nitrophenols by either method is not yet satisfactory and requires further development. The removal of carboxylic interferences is substantially better for the gel permeation-adsorbtion method, although not yet optimum. The QAE ion exchange method yields poor recoveries and intermediate (but high) levels of interfering compounds.

Coupled chromatography using base-treated silica gel after GPC showed the best results for separating acidic compounds from neutral ones. In the developed integrated cleanup scheme, most of the lipid-type interferences are removed by gel permeation chromatography from the compounds of interest prior to the silica gel step.

Results of the initial GPC experiments indicate that some neutral priority pollutants are removed by the silica gel and co-elute with fatty acids when these are recovered from the silica gel.

The initial experiments also revealed that binding of the phenols on potassium hydroxide modified silica gel was not complete for the most weakly acidic ones. An additional problem encountered was precipitation of salts in the eluates from the silicate column upon solvent exchange. Substitution of cesium for potassium in the modified silicate has been reported to improve retention and subsequent release of phenolics, so this material was also evaluated.

Cesium silicate was prepared by stirring 20 grams silica gel (MCB 100-200 mesh) for 1 hour with 50 ml of a saturated solution of cesium hydroxide in methanol. After filtration, the cesium silicate was soxhlet extracted for 1 hour with methanol followed by 4 hours extraction with methylene chloride. The solid was then dried at room temperature. Fifteen grams of the cesium silicate was slurry-packed in methylene chloride into a 0.8 x 8cm silanized glass column plugged with silanized glass wool. The solvent was replaced with 50/50 cyclohexane/methylene chloride and the sample applied in the same solvent. Samples containing free fatty acids plus phenols and fatty acid methyl esters plus

phenols were applied to the columns. The excess solvent was drained and the compounds eluted with methylene chloride containing a step-gradient increase of methanol, as indicated in Figure 5-6. Ten twenty-five ml fractions were collected, concentrated with the K/D evaporator, washed with pH 2 saline solution, and dried over anhydrous sodium sulfate prior to GC analysis.

The results for the cesium silicate experiments are shown in Figure 5-6. It was not possible to separate underivatized fatty acids from phenols with an elution gradient using cesium silicate. Prior derivatization of the fatty acids to methyl esters allowed complete separation of these from phenols (as previously mentioned in the BioBeads discussion). Some variability in the preparation of cesium silicate from silica gel and cesium hydroxide is indicated by the differences in the elution of phenols in the two column experiments.

Direct comparison of potassium and cesium silicate with a spike of priority phenols in methylene chloride showed good retention of phenols using cesium silicate, together with high recoveries upon elution with methanol. Since derivatization is required to permit GC analysis and quantitation, recoveries for the LC procedure are not separable from derivatization recoveries. In the case of potassium silicate, some break-through of phenol was observed, while poor recoveries were noted upon elution with methanol for all phenols while virtually no recovery of dinitrophenol was observed using potassium silicate.

Based upon the foregoing observations, cesium silicate is the material of choice for use in separation of phenolics from the neutral compounds. Its preparation is similar to that for initial experiments: 100g silica gel (unactivated) is placed in a 600 ml beaker, to which 300 ml of a saturated solution of CsOH in methanol is added. The mixture is stirred for 1 hour, allowed to settle, and decanted. The solids are washed with fresh methanol, filtered in a sintered glass funnel, and rinsed with 200 ml methanol, followed by 300 ml methylene chloride. The solids are then allowed to air dry at slightly elevated temperatures by placing them on top of an oven. The dried material is used without activation. Initial procedures called for extensive rinsing of the cesium-modified solids by soxhlet extraction, but this was subsequently abandoned when the simplified preparation gave satisfactory and reproducible results.

The observation that the methanol eluate from both potassium and cesium modified silica gel produced a white precipitate upon concentration and solvent exchange into methylene chloride prompted the soxhlet extraction procedure employed initially. The solids were clearly inorganic, and may have resulted from poor removal of free cesium or potassium salts in the silicate preparation. However, silica gel itself displays a limited solubility in neat methanol (especially hydrous methanol). The modi-

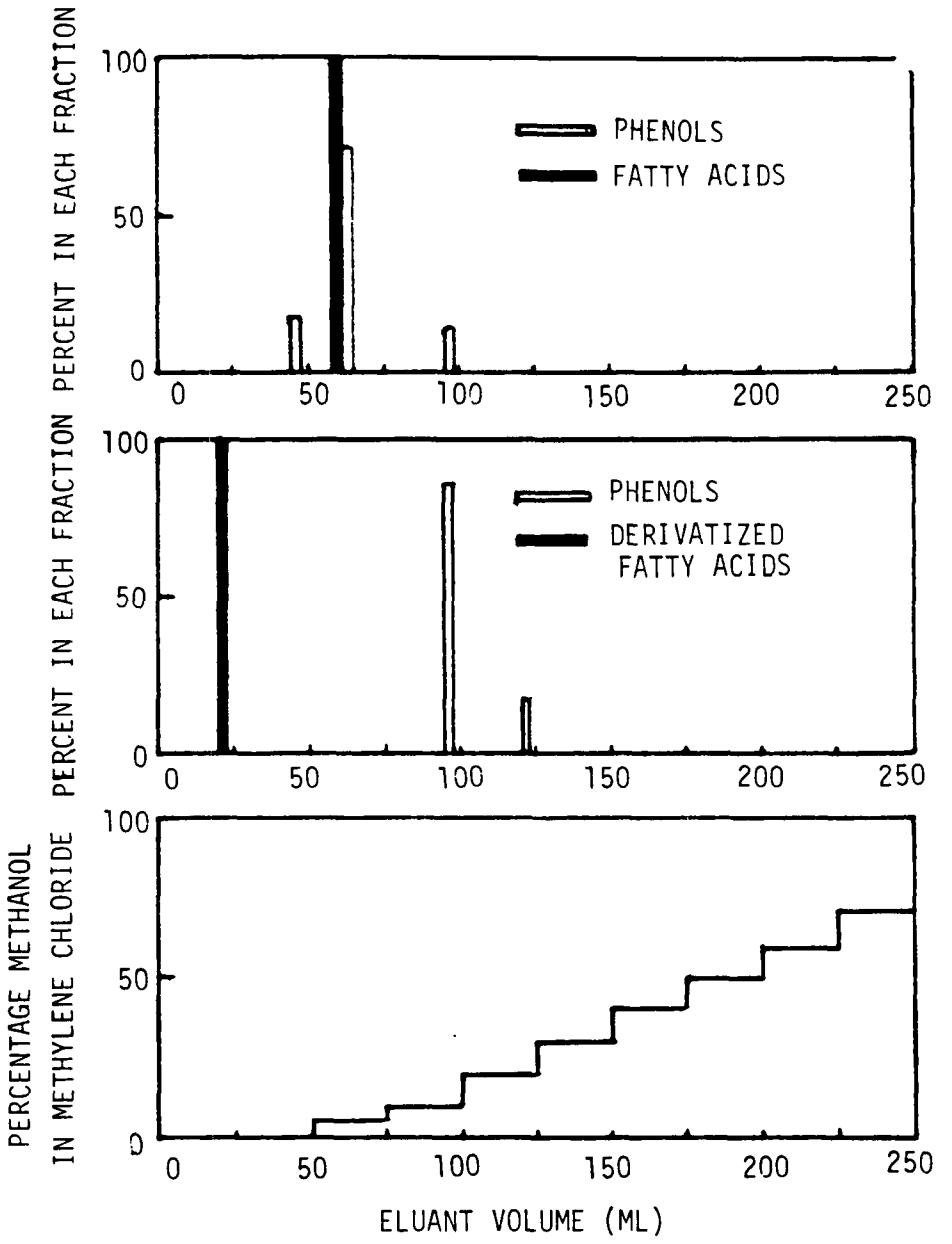


Figure 5-6 Elution Profile of Phenols and Fatty Acids on Cesium Silicate.

fied silicates are therefore expected to dissolve to a greater extent in the methanol eluant. Additional experiments showed that transfer of eluted phenols into a suitable solvent for derivatization and GC analysis, such as methylene chloride, can be accomplished by concentrating the methanol eluate and partitioning it between acidic water (pH 2) and methylene chloride. This procedure avoids the precipitation which could potentially remove phenols by inclusion or adsorption. When a 200 ug standard of phenols was dissolved in 2 ml methanol and partitioned between 10 ml pH 2 water and 10 ml methylene chloride, followed by two additional extractions with methylene chloride, the recoveries from the combined extracts were virtually quantitative. Additional extraction with methylene chloride produced no further detectable phenol. Plates VIII, IX, and X illustrate the separation.

5-3 Derivatization Studies of Phenols and Fatty Acids

Improvement of the chromatographic behavior of acidic compounds by chemical derivatizaton has been extensively investigated . Among the methodologies available are: 1) methylation with BF_3/methanol (to give methyl esters of fatty acids in high yield, but no derivatization of phenols), 2) methylation with diazomethane (to give methyl esters of fatty acids and methyl esters of some phenols, but with notably poor yields for phenol and alkyl phenols), and 3) methylation with base/dimethyl sulfate or other alkylating agent and a suitable catalyst to give methyl ethers of phenols but low to no yields of fatty acids esters. Several experiments to survey the ease and efficacy of various derivatization procedures have been carried out.

Table 5-2 summarizes the experimental conditions and results for the derivatization experiments. The samples were dried over sodium sulfate, solvent exchanged into cyclohexane, and analyzed by GC and GC/MS.

The most satisfactory results have been obtained by methylation with diazomethane. High yields can be obtained for all but phenol and the alkyl-substituted phenols for standard mixtures. Some encouraging results have been obtained with dimethyl sulfate alkylation. These observations are in agreement with other investigators (Drozd, 1975).

To optimize derivatization yields, several experiments with diazomethane were conducted. The effect of solvent composition was investigated in different combinations. All samples were bubbled with diazomethane until a persistent yellow color appeared, followed by a two minute wait and a subsequent volume reduction to 1 ml. The highest derivatization yields were seen for pentane/methylene chloride. The influence of reaction time was also investigated for times between 2 and 15 minutes, with the increase in yields leveling off after 7 minutes.

PLATE VIII CESIUM SILICATE (LEFT)
AND FLORISIL (RIGHT)
CHROMATOGRAPHY

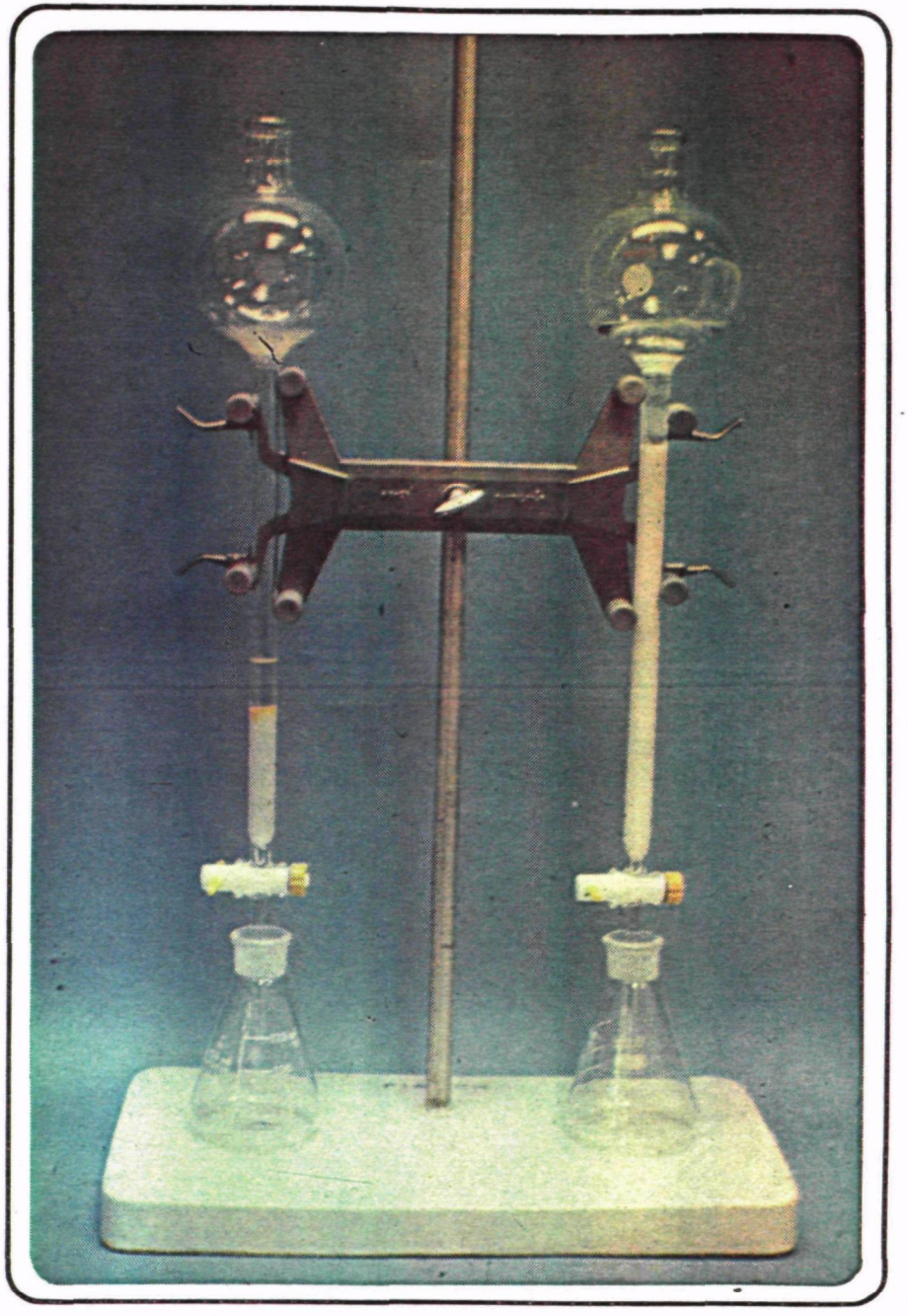


PLATE IX PHENOLS TRAPPED ON CESIUM SILICATE COLUMN

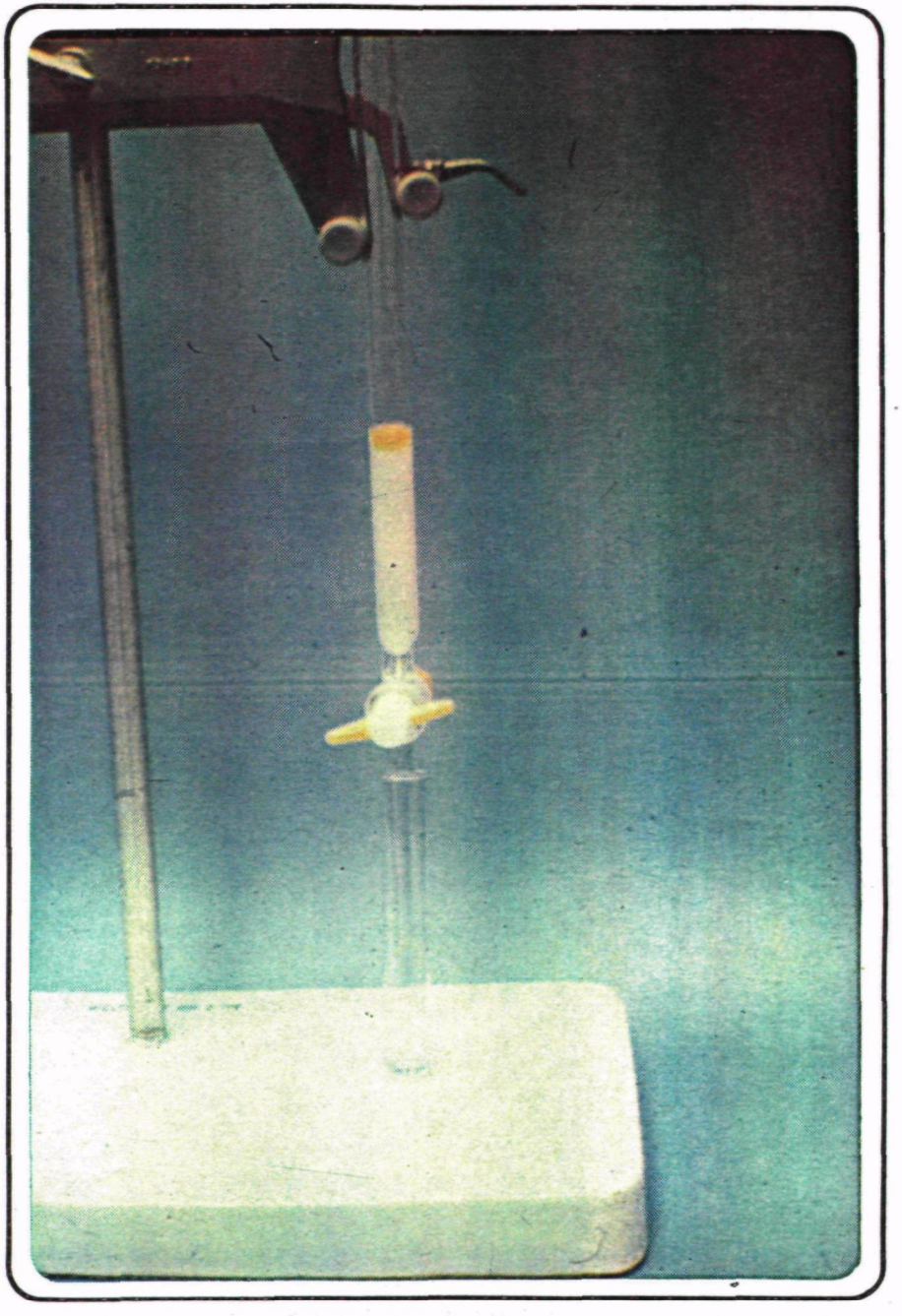
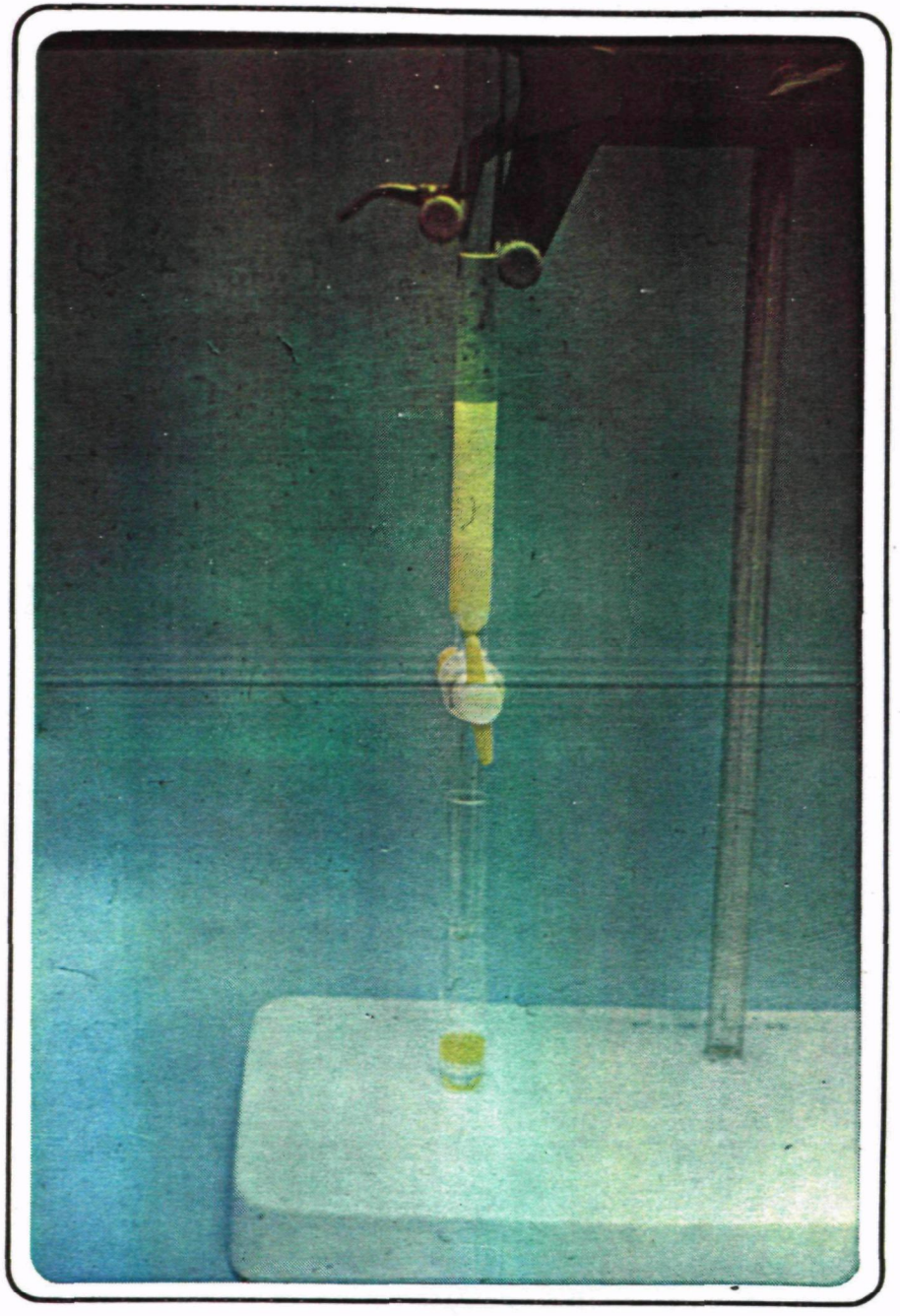


PLATE X PHENOLS ELUTED FROM CESIUM SILICATE WITH METHANOL



EXPERIMENT:	I	II	III	IV	V	VI	VII
Conditions:							
solvent	Et_2O	CH_2Cl_2	CH_2Cl_2	Et_2O	Et_2O	acetone	acetone
base	none	"proton sponge"*3	OH^-	OH^-	OH^-	OH^-	OH^-
catalyst	10% MeOH	Bu_4N^+	Bu_4N^+	Bu_4N^+	Bu_4N^+	Bu_4N^+	Bu_4N^+
alk. agent	CH_2N_2 gas	Me_2SO_4	Me_2SO_4	Me_2SO_4	MeI	Me_2SO_4	MeI
temp.	ambient	reflux	reflux	reflux	reflux	reflux	reflux
min. time	2 min	1 hr	15 hrs	2 hrs	2 hrs	2 hrs	2 hrs
Derivative Yields From:							
Phenol	x	o	x		o	x	+
2,4-dimethyl-	o	o	+	o	o	o	o
2,4,6 trichloro-	+	x	+	x	o	o	x
4-chloro,3-methyl-	x	x	+	o	o	o	o
2-chloro-	+	x	*1	*1	*1	*1	*1
2,4-dinitro	+	o	o	-	o	o	-
4-nitro	+	x	+	o	o	o	o
pentachloro	+	*1	+	-	o	o	-
4,6-dinitro, 3-methyl	+	*1	+	-	o	o	-
2,4 dichloro	+	*1	*1	*1	*1	*1	*1
2-nitro	+	*1	*1	*1	*1	*1	*1

Key: *1 - compound not present
*2 - not detected by GC
*3 - "proton sponge" - N,N,N',N'-tetramethyl, 1,8-diamino naphthalene, is a trademark of Aldrich Chemical Co.

+ high yield
x some yield
- trace yield
o not detectable yield

Table 5-2 Experimental Conditions and Results for the Derivatization Study.

A recovery experiment using the optimized methods of liquid chromatographic separation of the phenols, elution from silicate, solvent exchange, and derivatization was conducted on a spiked water sample. This experiment determined recoveries from silicate chromatography through derivatization, and compared the standard methanol eluate with methanol plus 1% formic acid eluate. Table 5-3 presents the results obtained, and shows an overall recovery of 66% using methanol and 69% using methanol and formic acid, indicating that no major improvement is realized by the addition of formic acid.

Recent advances in glass capillary technology, discussed in Section 5-4, have permitted the more complete evaluation of diazomethane derivatization for the analysis of phenols from spiked raw sewage and digested sludge samples. Based upon this new information, the recoveries of phenols are clearly dependent on time elapsed between derivatization and instrumental analysis. The lack of stability resulting from diazomethane treatment is discussed more fully in Section 10-4, but should be understood before this method is used.

5-4 Instrumental Analysis

Initial experiments utilized a packed SP 1240 DA column which has recently been recommended to replace Tenax GC. Figure 5-7 was obtained from a new column and Figure 5-8 shows performance of a different column after substantial use. At present, this type of column is the only one capable of eluting all eleven priority phenols without derivatization. Comparison of these figures illustrates that: a) resolution varies between columns and b) recoveries of the more acidic phenols declines with column use, particularly in 4,6-dinitrophenol and 2,4-dinitro-o-cresol. However, this column still gives superior resolution as suggested by Table 5-4, which compares the relative GC retention times we observe for SP-1240 DA with literature values for several other GC liquid phases.

During extensive use of several columns prepared from this material, however, serious shortcomings have been observed. No two columns prepared from the same bottle of packing material have given the same separation even though all methods used were identical. A more serious reproducibility problem was encountered from one injection to the next on the same column. Using identical GC conditions, reproducible peak area ratios for some phenols could not be obtained from sequential injections of the same standard phenol mixture. Ortho-, chloro-, and nitro- phenols suffered especially from this phenomenon. A further problem with the SP 1240 DA column, and packed columns in general, has been insufficient resolution of phenol isomers and other compounds present in actual sample extracts. Finally, column performance degraded rapidly and dramatically with modest use, especially when real extracts were analyzed. Attempted regeneration

Phenol	Derivative	Recovery (%) Methanol	Methanol + Formic Acid
phenol	anisole	79	84
phenol	*underivatized	59	40
o-chlorophenol	*underivatized		
o-chlorophenol	o-chloroanisole	69	109
4-chloro, 3-methylphenol	4-chloro, 3-methylanisole	68	23
4-chloro, 3-methylphenol	*underivatized	66	187
2,4 dimethylphenol	2,4-dimethylanisole	59	59
2,4-dichlorophenol	2,4-dichloroanisole	64	50
2,4,6-trichlorophenol	2,4,6-trichloroanisole	69	128
2-nitrophenol	2-nitroanisole	66	23
4-nitrophenol	4-nitroanisole	65	53
4,6-denitro 2-methylphenol	4,6-dinitro, 2-methylanisole	55	10
pentachlorophenol	pentachloroanisole	70	67
2,4-dinitrophenol	2,4-dinitroanisole	66	67

*not resolved by gc

Table 5-3 Overall Recovery of Phenols in Spiked Distilled Water During Column Chromatography and Derivatization

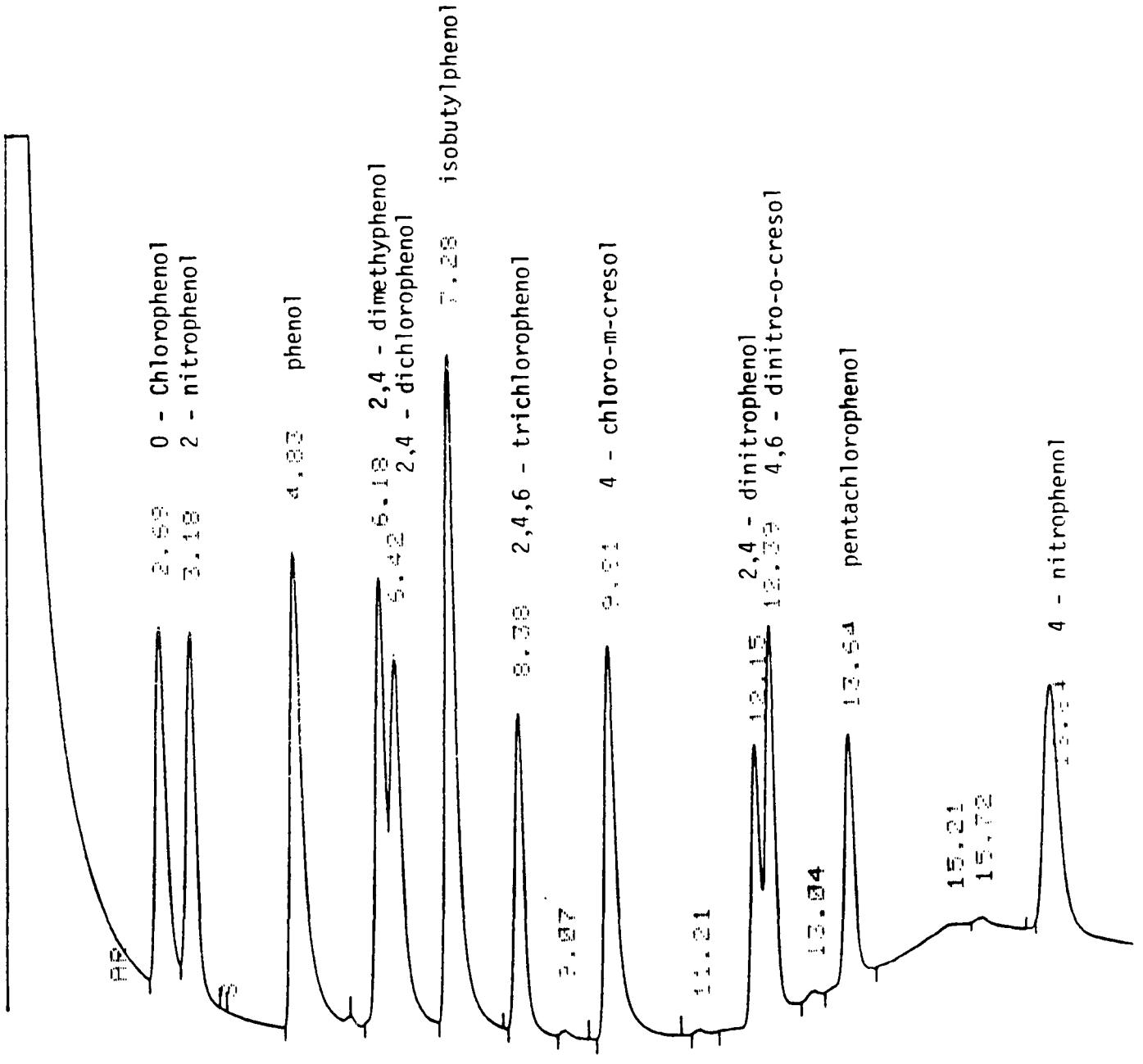


Figure 5-7 Chromatogram of Phenolic Standard on a new 3' SP-1240DA packed column.

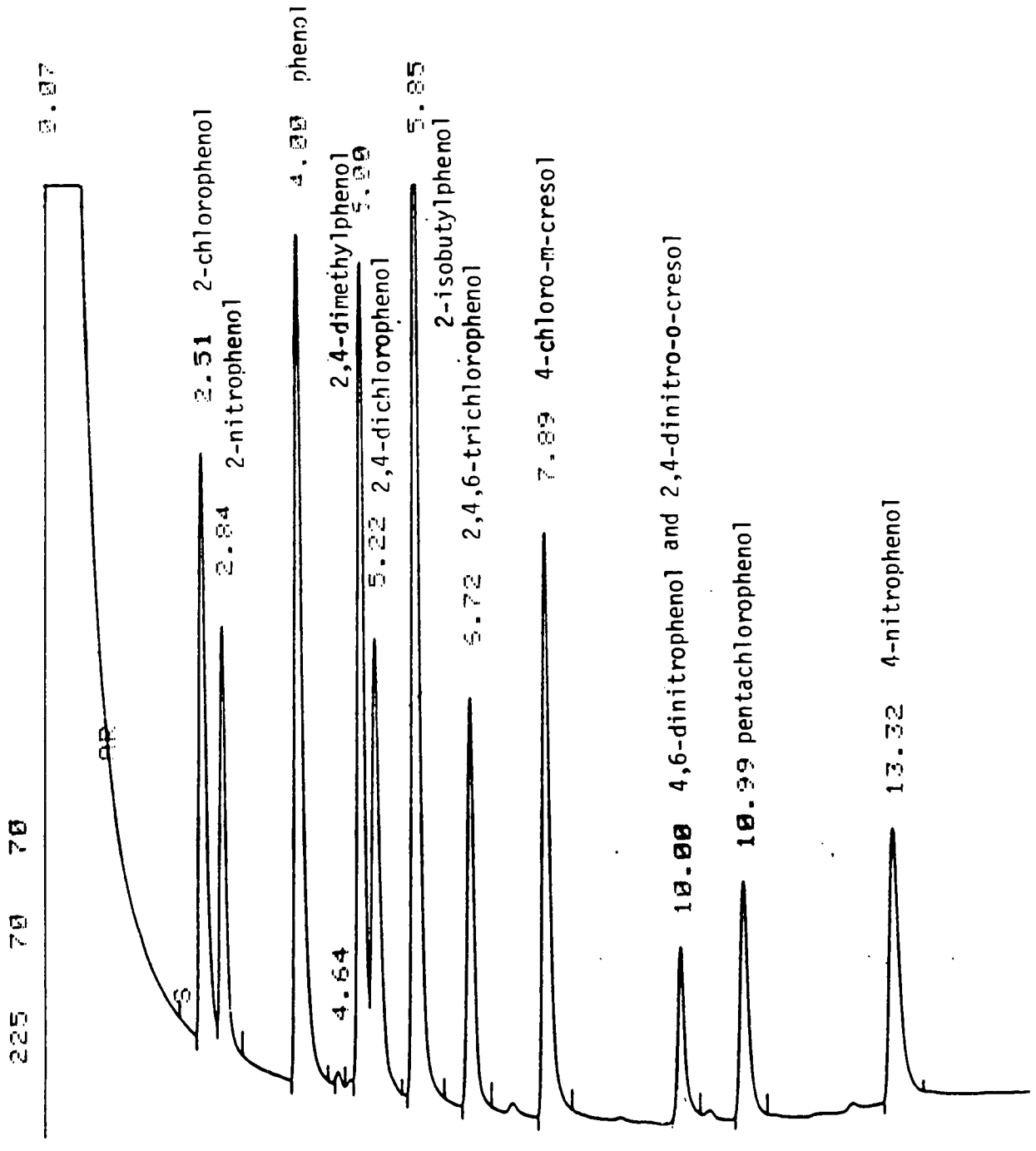


Figure 5-8 Chromatogram of phenolic standard after degradation of SP-1240 DA performance

Compound	Tenax-GC	SP-2250	OV-17	SP-1240 DA
2-chlorophenol	0.63	0.66	0.64	0.88
phenol	0.66	1.26	0.76	1.41
2,4-dichlorophenol	0.96	1.00	1.11	1.83
2-nitrophenol	1.00	1.00	1.00	1.00
p-chloro-m-cresol	1.05	1.38	1.44	2.75
2,4,6-trichlorophenol	1.14	1.34	1.51	2.34
2,4-dimethylphenol	1.32	0.98	-*	1.75
2,4-dinitrophenol	1.34	-*	-*	3.47
4,6-dinitrocresol	1.42	-*	-*	3.61
4-nitrophenol	1.45	-*	-*	4.63
pentachlorophenol	1.60	2.09	3.23	3.82

Table 5-4 Relative retention times of phenolic priority pollutants on four different GC colums.

of the column by injection of phosphoric acid solution, as per the manufacturer's instruction, failed to restore stable and acceptable column performance. EPA-Cincinnati has reported similar observations to EPA contractors: good separations can be obtained with the SP 1240 DA column under ideal conditions, but column lifetimes are short.

Other commercially available GC columns require chemical derivatizaton of the phenolics if more sensitive and reliable results have to be obtained. Capillary columns give better separation but can only be used with a derivatized or partially derivatized phenol sample. A capillary column coated with SP-1000 (also known as FFAP) was therefore chosen for further analysis. A ten meter column of this type is able to chromatograph 7 of the 11 priority phenols without derivatization and with superior resolution compared to the packed column (Figure 5-9). Only the very acidic phenols: p-nitrophenol, pentachlorophenol, dinitrocresol, and dinitrophenol, are not eluted from this column. Many of the preparatory chromatography experimental fractions were analyzed on the SP-1000 capillary column.

Reaction with diazomethane has been shown to quantitatively produce methyl ethers of these four most acidic compounds. Other priority phenols are also derivatized, but not all quantitatively (and very poorly in the case of phenol and dimethyl phenol). Analysis of a partially methylated standard on the SP-1000 capillary column is illustrated in Figure 5-10 and appears to be a satisfactory alternative if other derivatization methods are unsuccessful. Of course, quantitative analysis by this approach requires locating and quantitating two separate peaks (compounds) utilizing two different retention times and response factors each for many of the priority phenols. There are still significant advantages to a general and quantitative chemical derivatization method for all of the compounds as opposed to the above alternative. In particular alkyl ethers of the phenols are readily chromatographed on the same capillary column (SE-54) used for the base-neutral priority compounds as shown in Figure 5-11. Also some workers have reported that phenols derivatized to pental fluoro benzyl ethers can be detected with very high sensitivity by GC/ECD and negative-ion GC/CIMS.

The variable performance of SE-54 and SP 1000 glass capillary columns is reflected in the fact that sequential analyses of the same sample reveal variable chromatography for the underivatized phenols. This problem is more serious for real samples than stan-dards, as derivatization yields are frequently adversely affected by the presence of background. The poor behavior of these compounds on glass capillaries is a reflection of the nonideal deactivation of the glass itself. Recent advances in glass deactivation techniques, discussed in Section 7, provide one solution. However, a new material for the construction of capillary columns, fused silica (synthetic quartz), is now avail-

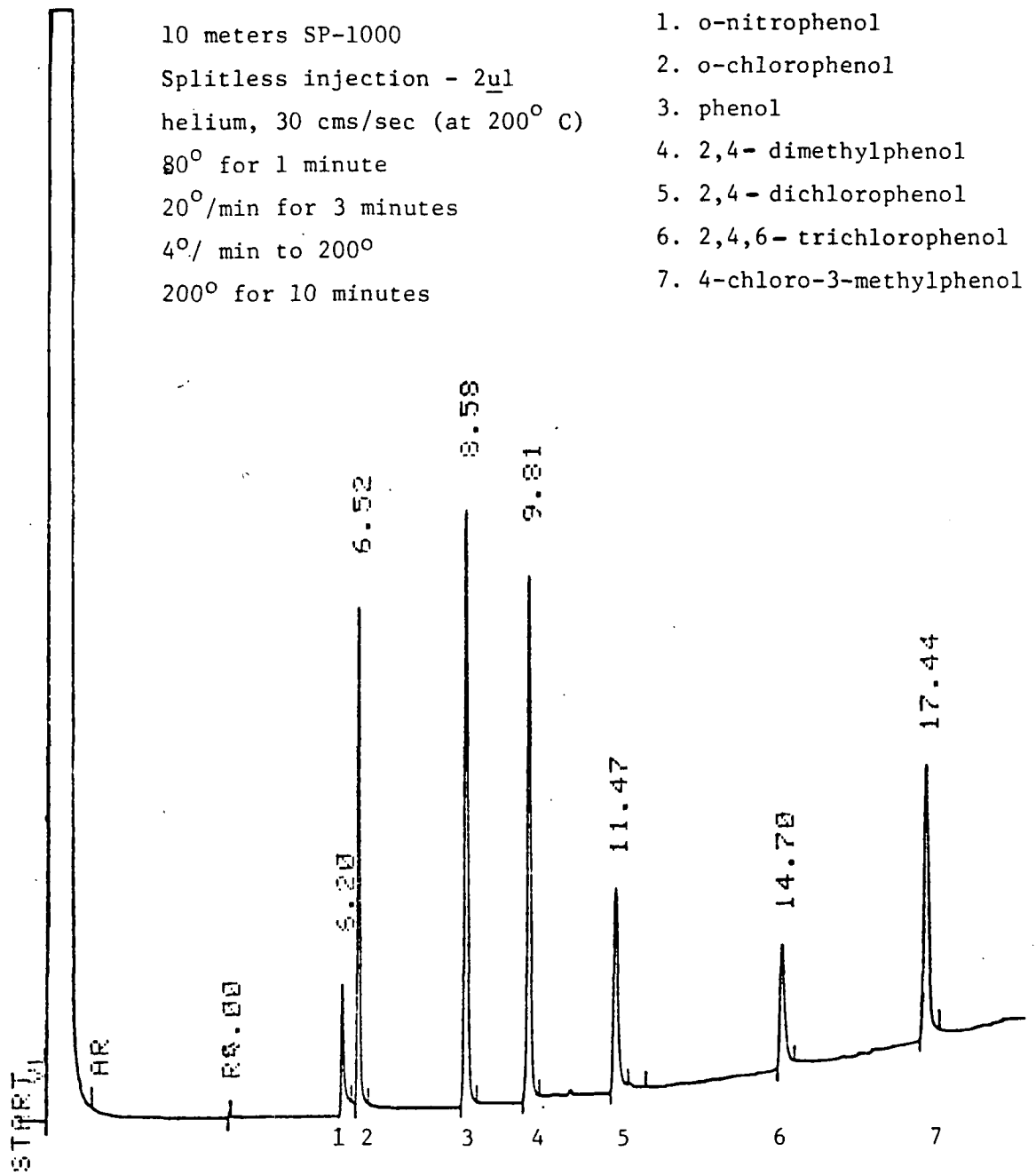


Figure 5-9 Analysis of Phenol Standard on SP-1000

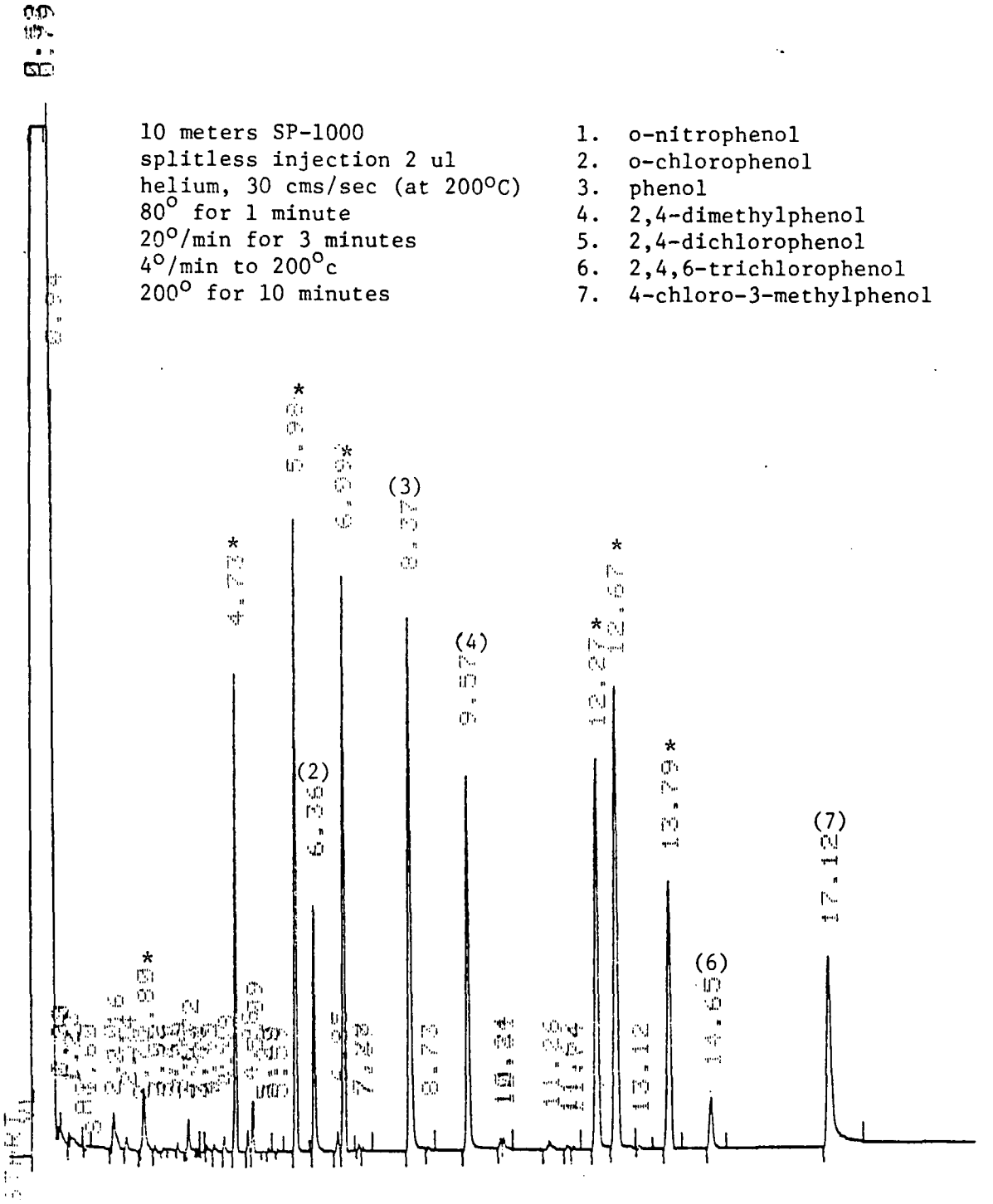


Figure 5-10 Analysis of Partially Derivatized Phenols on SP-1000

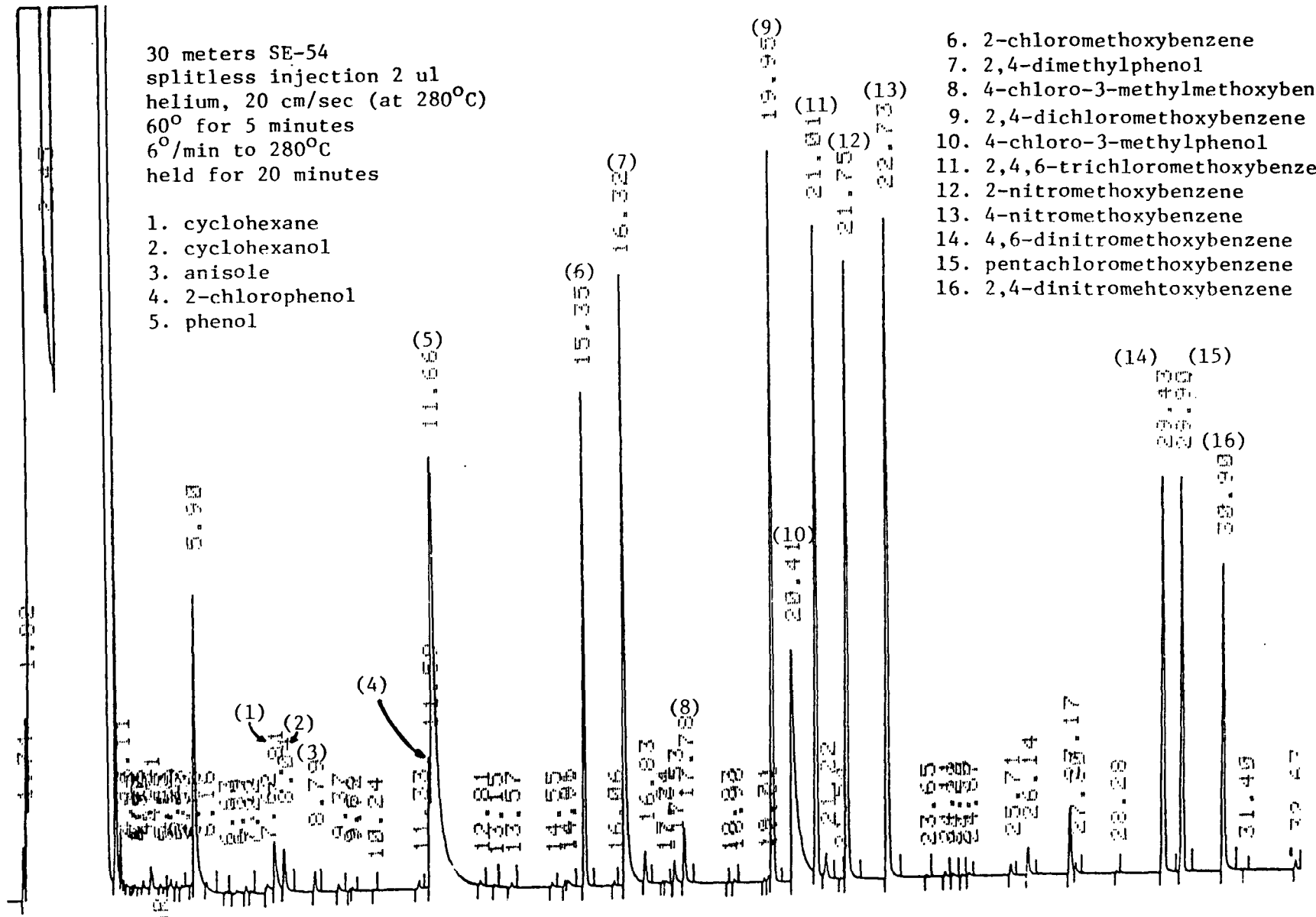


Figure 5-11 Analysis of Partially Derivatized Phenols on SE-54

able with SP 2100 stationary phase from Hewlett-Packard Corporation. These columns display superior inertness and are preferable to any previous glass capillary for this group of compounds. Further improvements in this area are imminent, offering the immediate liklihood of a satisfactory analysis of phenols in their underivatized form.

SECTION 6

PRIORITY POLLUTANT INTEGRATED ANALYTICAL SCHEME

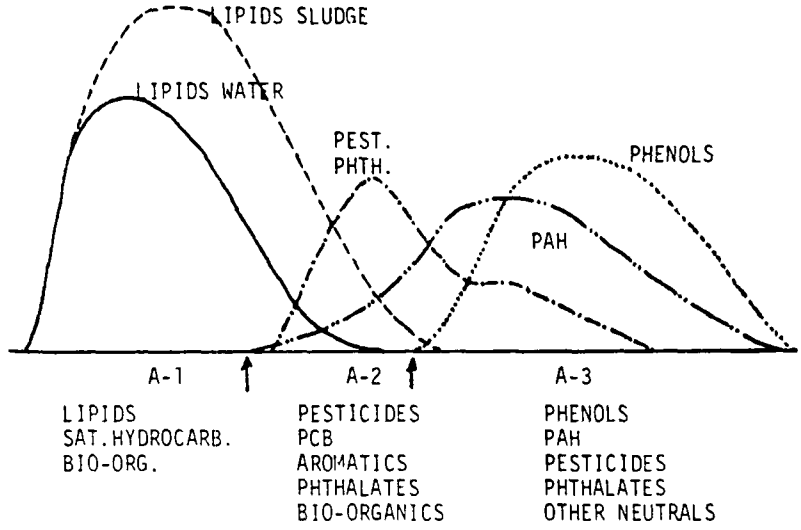
The results obtained in Sections 2, 3, 4, and 5 were subsequently utilized to develop an integrated analytical scheme for the recovery and determination of priority pollutants in wastewater and sludges. The scheme uses gel permeation chromatography, florisil cleanup, and silicate column chromatography.

The gel permeation chromatography of crude extract concentrates serves two purposes: 1) the removal of gross lipid and other large neutral organic interferences from the sample and 2) fractionation of the sample in a way that permits convenient subsequent treatment. The separation of lipid and other neutral interferences from the neutral priority compounds is best achieved by SX-2 BioBeads. These interfering compounds are uniformly the earliest eluting and present separation difficulties only with the earliest eluting priority neutrals, i.e. dioctyl phthalate and some of the pesticides. Solvent mixtures with a greater proportion pentane exhibited increased selectivity on a molecular size basis, possibly with a superimposed electronic effect for aromatic molecules. Empirically, the effect is increased separation of fatty acids from phenols, smaller elution volumes, and increased sorting within compound groups as the exclusion limit decreases.

Florisil chromatography is utilized following the BioBeads to remove interferences that co-elute along with phthalates, pesticides, and PAHs in the GPC step. Desired separation for florisil LC cleanup consisted of removal of saturated hydrocarbon material, and removal of biological containants including fatty esters, glycerides and sterols. It was found that a 0.75% deactivated florisil was able to separate saturated hydrocarbons and pesticides. A spike of priority phenols in methylene chloride shows good retention of phenols using cesium silicate, together with high recoveries upon elution with methanol followed by derivatization.

The results of the chromatography experiments and the proposed analytical scheme are shown in Figure 6-1, while the detailed steps are listed in Figure 6-2. An average system recovery of 113%, using a standard mixture of the neutral fraction in methylene chloride, is presented in Table 6-1.

Initial evaluations of the separation scheme using real samples were carried out using BioBeads, florisil, and silicate chromatography. It was found that the silicate fraction (which begins with the early-eluting phenols) was remarkably free of gross amounts of interfering compounds. Figure 6-3 shows a typical reconstructed gas chromatogram from an analysis of West Point primary sewage effluent. The florisil chromatography, in its



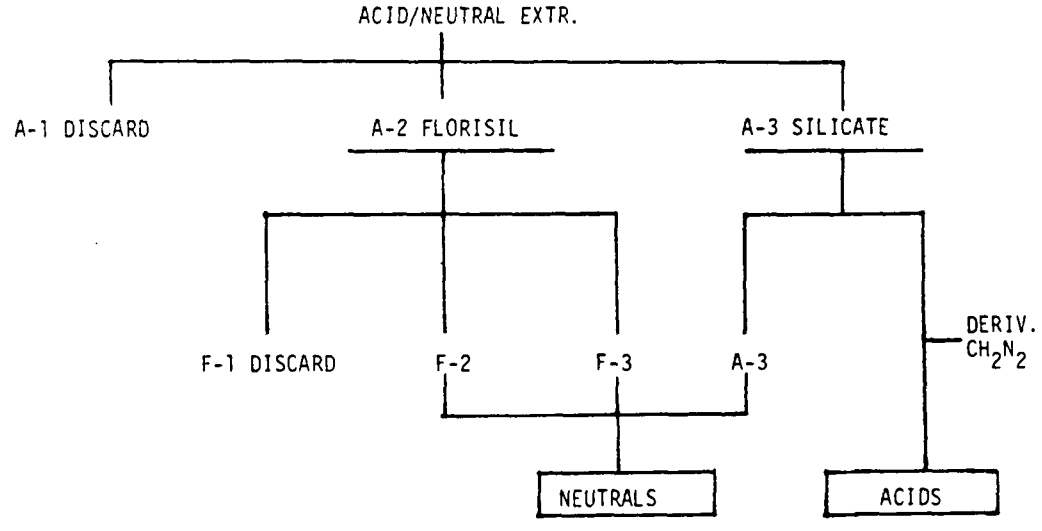


Figure 6-1 Schematic Elution of Priority Pollutants During Column Chromatography.

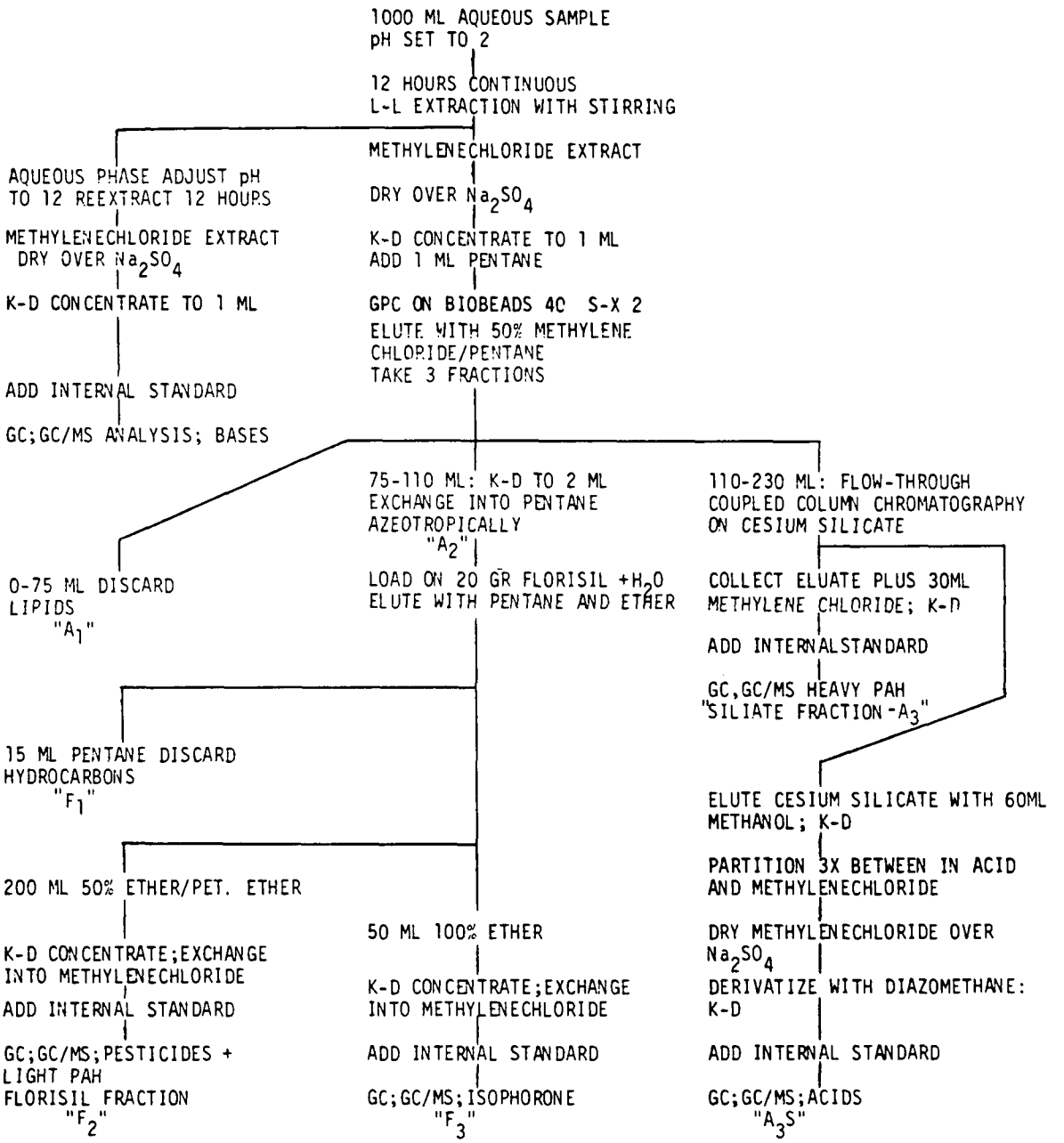


Figure 6-2 Modified Cleanup Scheme for Acid and Neutral Organics

COMPOUND	% RECOVERY: FLORISIL FRACTION	SILICATE FRACTION	TOTAL
HEXAMETHYLBENZENE	**	**	**
N-NITROSODIMETHYLAMINE	*	*	*
BIS (2-CHLOROETHYL) ETHER	65.6	5.2	70.7
(1,3-) DICHLOROBENZENE	81.5	11.2	92.7
1,4-DICHLOROBENZENE	61.6	23.0	84.6
(1,2-) DICHLOROBENZENE	58.5	26.8	35.4
BIS(2-CHLOROISOPROPYL)ETHER	*	*	*
HEXACHLOROETHANE	137.9	0	137.9
N-NITROSO-DI-N-PROPYL AMINE	69.1	0	69.1
NITROBENZENE	24.3	64.7	89.0
BIX(2-CHLOROETHOXY)METHANE	*	*	*
1,2,4-TRICHLOROBENZENE	66.6	43.3	109.9
NAPHTHALENE	28.0	41.0	69.0
HEXACHLOROBUTADIENE	*	*	*
2-CHLORO-NAPHTHALENE	15.6	95.8	111.4
ACENAPHTHYLENE	3.1	81.7	84.8
DIMETHYLPHTHALATE	94.3	0	94.3
2,6-DINITROTOLUENE	0	83.3	83.3
ACENAPHTHENE	16.6	62.3	78.9
2,4-DINITROTOLUENE	0	74.9	74.4
FLUORENE	18.7	55.9	74.6
DIETHYLPHTHALATE	94.4	0	94.4
AZOBENZENE (FROM DIPHENYLHYDRAZINE)	56.8	8.5	65.4
N-NITROSODIPHENYLAMINE	*	*	*
4-BROMODIPHENYL ETHER	46.6	72.1	118.7
HEXACHLOROBENZENE	51.4	106.7	158.1
PHENANTHRENE	0	65.0	65.0
DIO-ANTHRACENE (I.S.)	118.4	86.9	**
ANTHRACENE	0	59.8	59.8
DI-N-BUTYLPHTHALATE	89.5	0	89.8
FLUORANTHENE	0	85.7	85.7
PYRENE	0	104.2	104.2
BUTYLBENZYLPHTHALATE	87.5	0	87.5
BENZO(A)ANTHRACENE	0	122.7	122.7
CHRYSENE	0	217.2	217.2
3,3'-DICHLOROBENZIDENE	*	*	*
BIS(2-ETHYLHEXYL)PHTHALATE	87.5	0	87.5
DI-N-OCTYL PHTHALATE	86.1	0	86.1
BENZO(B)FLUORANTHENE	0	182.2	182.2
BENZO(K)FLUORANTHENE	0	183.3	183.3
BENZO(A) PYRENE	0	192.6	192.6
INDENO(1,2,3-CD)PYRENE	*	*	*
DIBENZO(A,H)ANTHRACENE	0	263.4	263.4
BENZO(GHI)PERYLENE	0	267.8	267.8

* Not present in spike
** Internal standard

Table 6-1 Recovery of Neutral Priority Pollutant Standard During Column Chromatography.

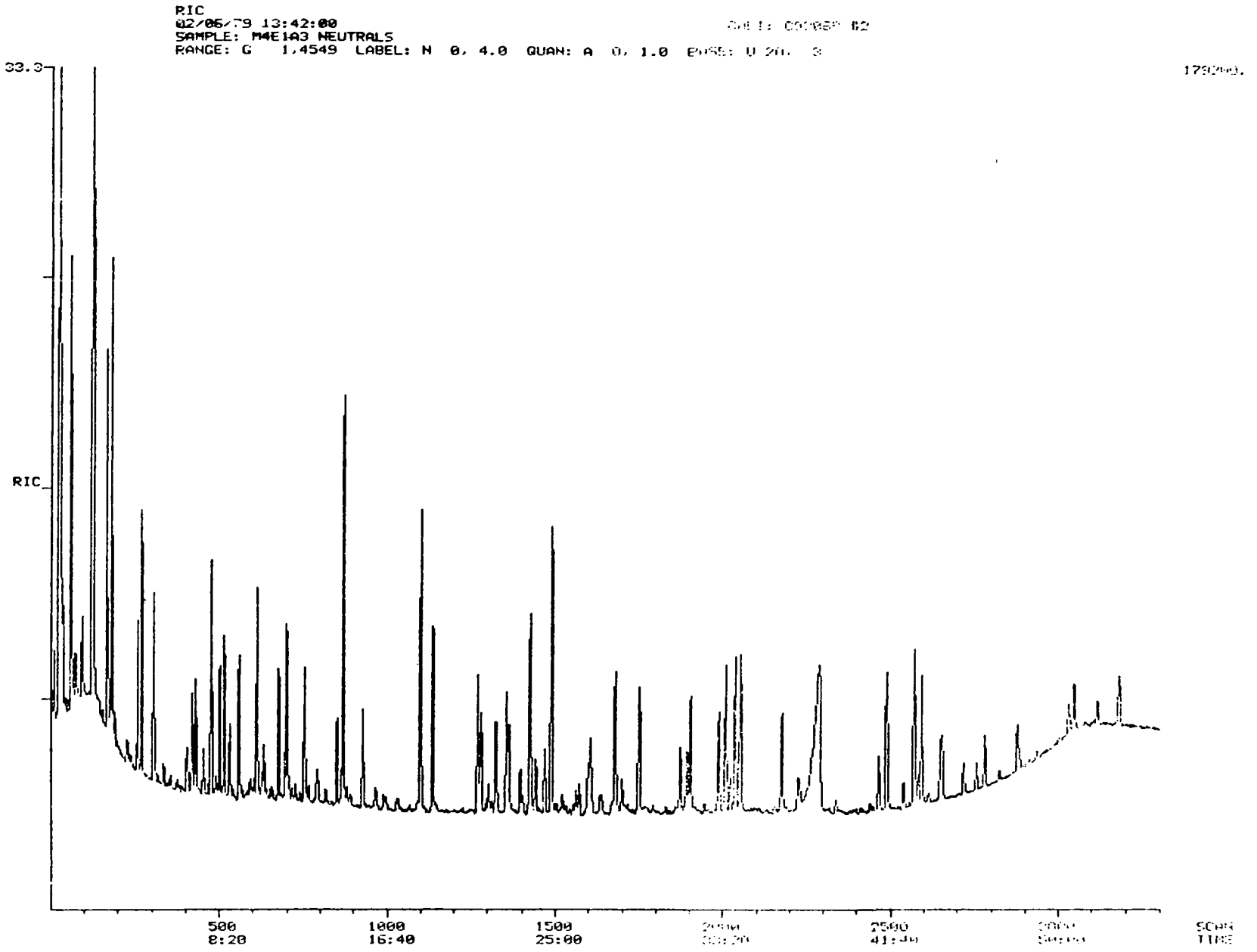


Figure 6-3 Reconstructed Gas Chromatogram of Silicate Fraction of West Point Primary Effluent.

present state of refinement, was used to prepare sewage and sludge extracts for instrumental analyses. The extract fractions generated by the cleanup scheme were screened by GC-FID and are presented for one representative raw sewage and one waste activated sludge sample (both spiked with approximately 45 priority compounds) in Figures 6-4 through 6-8. The priority compounds appear in the silicate, florisil, and derivatized acid fractions (Figures 6-5, 6-6, and 6-7). Note that for the case of the raw sewage extract, the cleanup scheme is highly successful in removing contaminants (Figure 6-5) while in the case of the sludge extract, the florisil fraction is insufficiently purified of interferences.

Based on these screening analyses, the sewage sample was analyzed for neutral priority compounds and recoveries computed based on comparison of the spiked and unspiked results. Table 6-2 presents the computed recoveries. For the sludge samples, recoveries could only be determined for those compounds eluted entirely within the silicate fraction, i.e. the 12 heavy polynuclear aromatics. The florisil fractions of these sludges were not analyzable by GC/MS without further cleanup. Recoveries are presented in Table 6-3.

The sludge and sewage samples were spiked prior to extraction (a 1-liter sample for the sewage, 500 mls for the sludge). The extract obtained could only be handled by the cleanup chromatographic columns as an aliquot; a 10% aliquot of the primary sludge extract gave 221 mg residual weight. Using 10% aliquots of the sludge extracts meant a 10-fold dilution in the spike concentration down to 1 ug per compound. Imperfect recoveries from the extraction and cleanup steps further reduced the spike level. In future experiments, either smaller sludge samples are used, or higher levels of spike are employed.

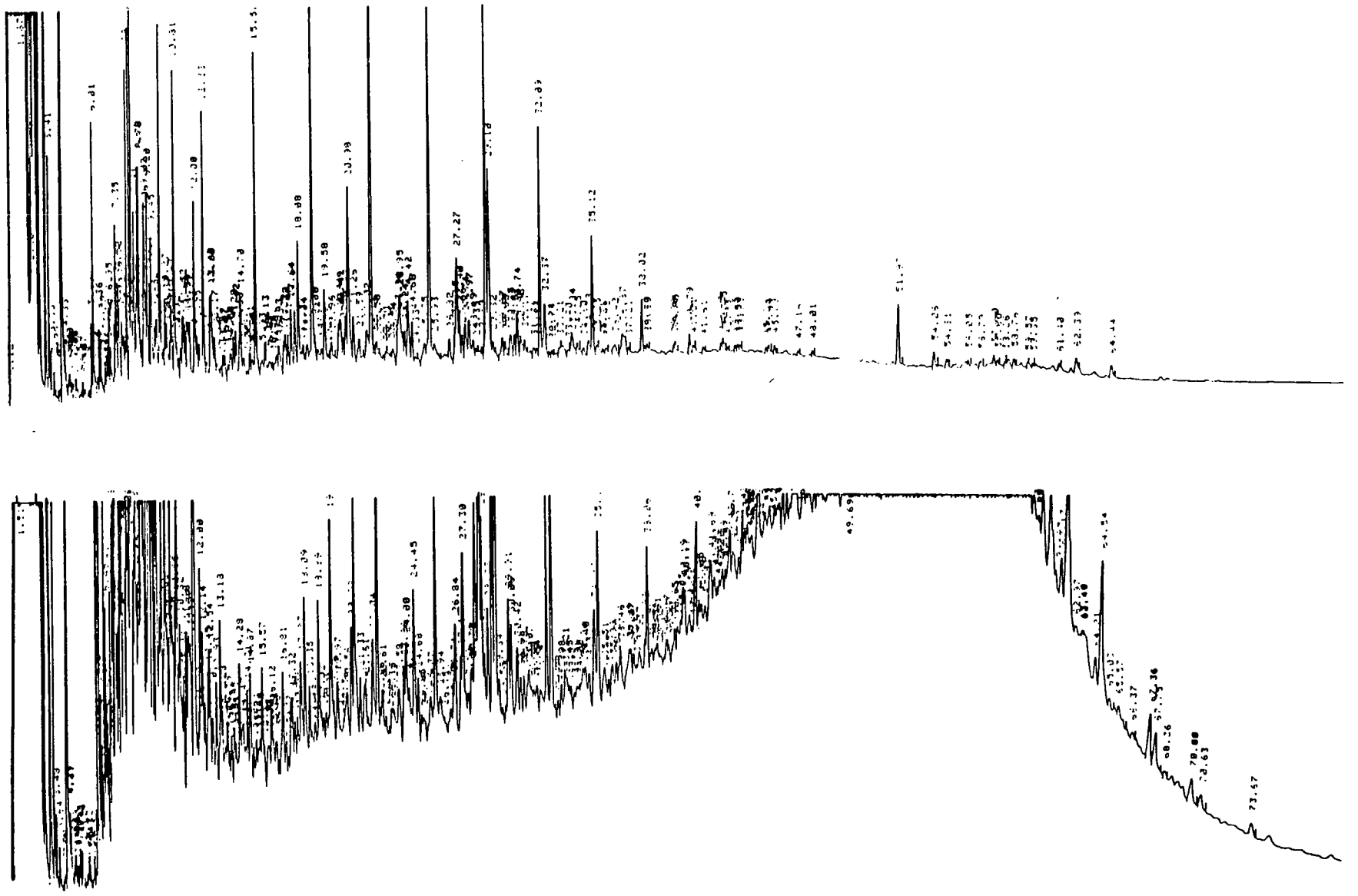


Figure 6-4 Hydrocarbon (discard) Florisil Fraction for Spiked Raw (a) and Waste Activated Sludge (b) Sample.

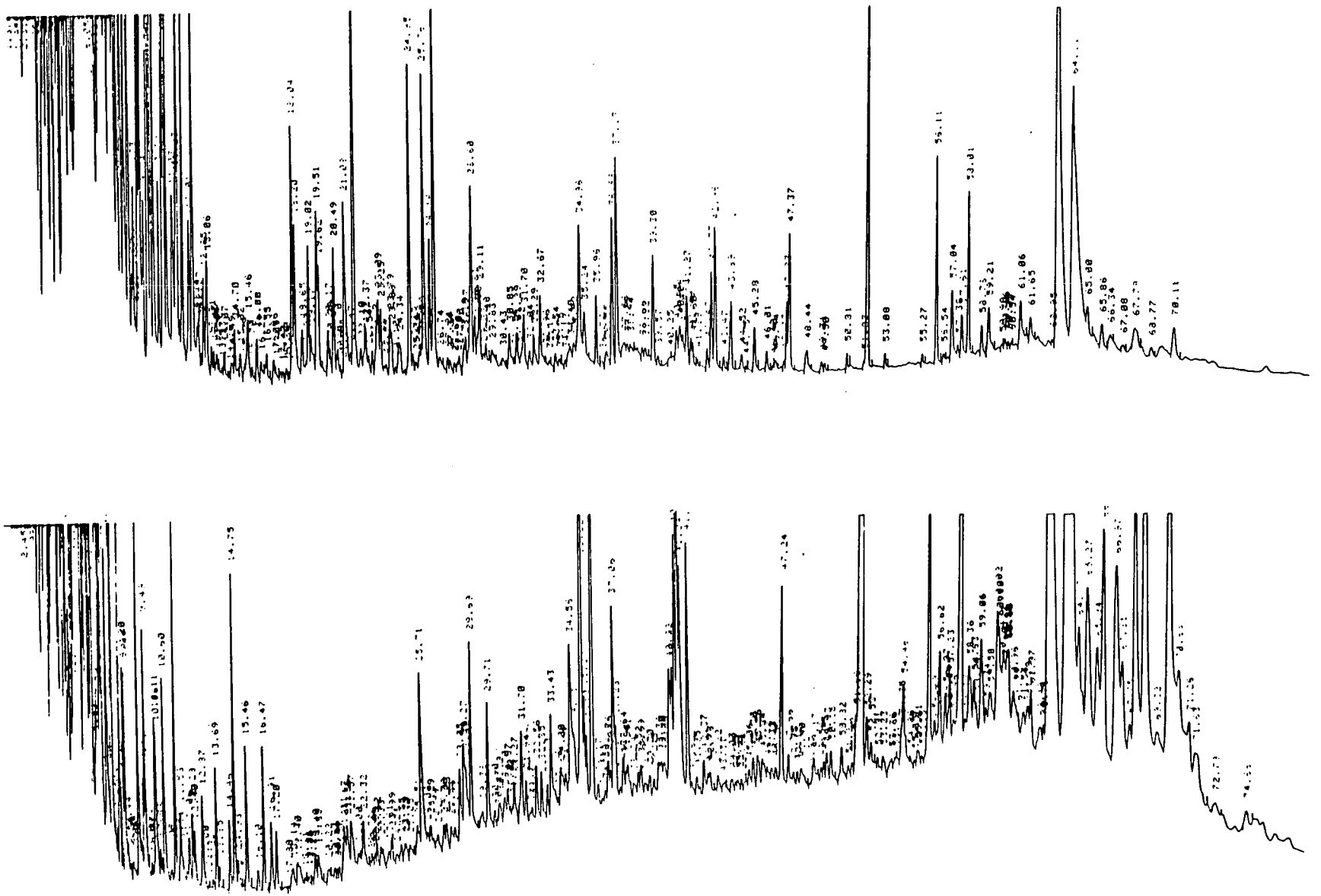


Figure 6-5 Florisil Neutral Fraction of Spiked Sewage and Sludge Sample.

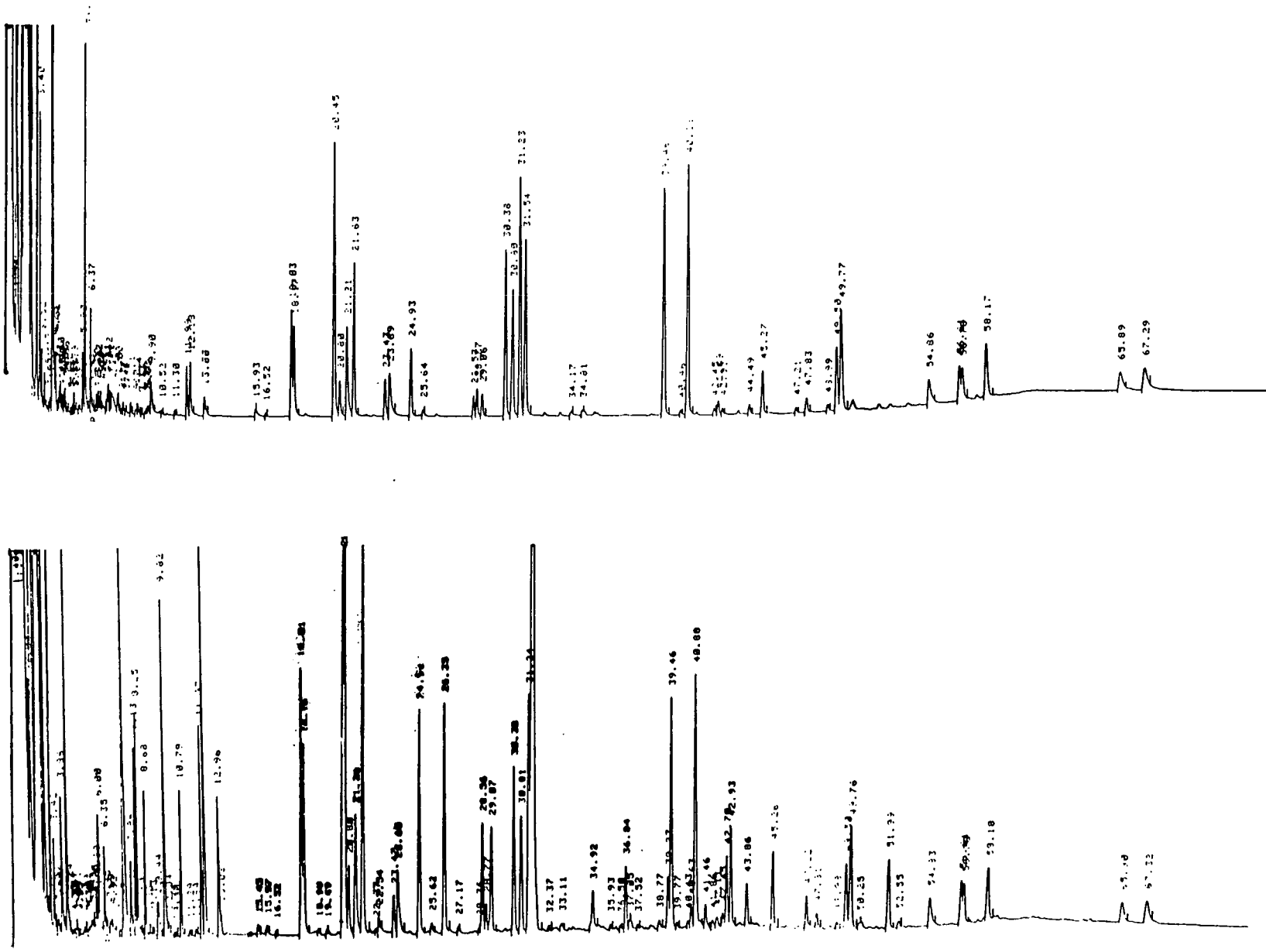


Figure 6-6 Silicate Neutral Fraction for Spiked Sewage (a) and Sludge (b) Sample.

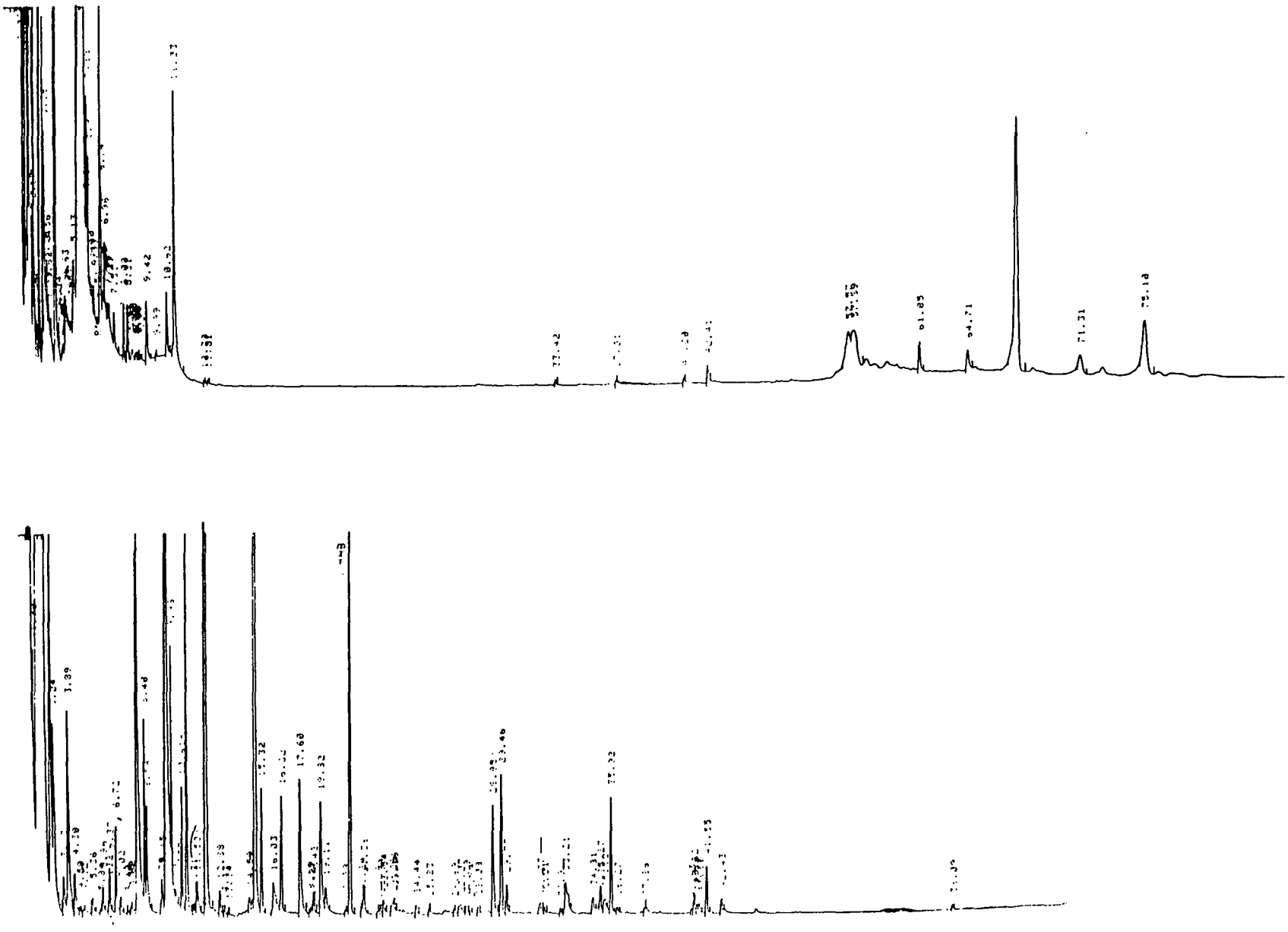


Figure 6-7 Third Ether (a) Florisil Fraction of a Spiked Sludge Sample and (b) Derivatized Phenol Fraction of a Spiked Sludge Sample

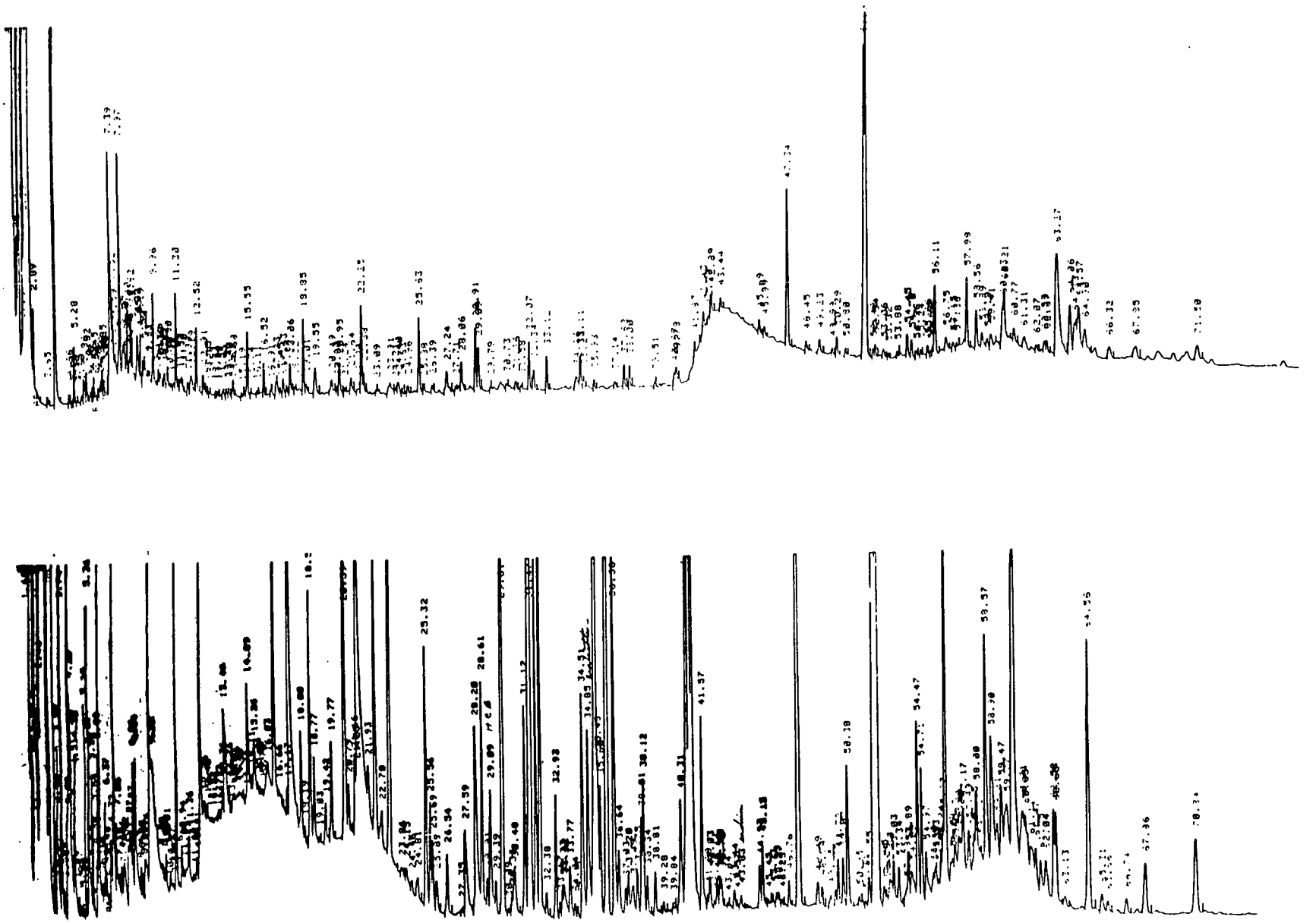


Figure 6-8 Base Extracts of Spiked Sewage (a) and Spiked Sludge Sample (b).

COMPOUND	% RECOVERY
AZOBENZENE (FROM DIPHENYLHYDRAZINE)	102.32
N-NITROSODIPHENYLAMINE	*
4-BROMODIPHENYL ETHER	125.14
HEXACHLOROBENZENE	58.94
PHENANTHRENE	122.40
DIO-ANTHRACENE (I.S.)	**
ANTHRACENE	***
DI-N-BUTYLPHTHALATE	137.39
FLUORANTHENE	107.37
PYRENE	101.52
BUTYLBENZYLPHTHALATE	130.36
BENZO(A)ANTHRACENE	***
CHRYSENE	85.86
3,3'-DICHLOROBENZIDENE	***
BIX(2-ETHYLHEXYL)PHTHALATE	*
DI-N-OCTYL PHTHALATE	152.04
BENZO(B)FLUORANTHENE	85.65
BENZO(K)FLUORANTHENE	***
BENZO(A) PYRENE	89.79
INDENO(1,2,3-CD)PYRENE	*
DIBENZO(A,H)ANTHRACENE	84.75
BENZO(GHI)PERYLENE	85.14
HEXAMETHYLBENZENE	**
N-NITROSODIMETHYLAMINE	*
BIS (2-CHLOROETHYL) ETHER	109.19
(1,3-) DICHLOROBENZENE	50.96
1,4-DICHLOROBENZENE	2.01
(1,2-) DICHLOROBENZENE	50.66
BIS(2-CHLOROISOPROPYL)ETHER	*
HEXACHLOROETHANE	***
N-NITROSO-DI-N-PROPYL AMINE	103.13
NITROBENZENE	93.91
BIS(2-CHLOROETHOXY)METHANE	***
1,2,4-TRICHLOROBENZENE	29.83
NAPHTHALENE	131.56
HEXACHLOROBUTADIENE	*
2-CHLORO-NAPHTHALENE	173.74
ACENAPHTHYLENE	113.95
DIMETHYLPHTHALATE	138.98
2,6-DINITROTOLUENE	135.95
ACENAPHTHENE	117.84
2,4-DINITROTOLUENE	107.67
FLUORENE	123.18
DIETHYLPHTHALATE	146.63

* not present in spike ** internal standard *** detected but not quantitated

Table 6-2 Recoveries of Compounds Spiked in Raw West Point Sewage.

Table 6-3. Recoveries of Heavy PAH & Phenol fraction is Spiked Sludge Extracts.

COMPOUND	Secondary Sludge	Anaerobic Digested Sludge
2, 6-DINITROTOLUENE	21.6	68.4
2, 4-DINITROTOLUENE	152.1	***
PHENATHRENE	127.4	0
FLUORANTHENE	141.7	65.6
PYRENE	137.6	63.4
BENZO(A)ANTHRACENE	140.0	58.4
CHRYSENE	127.2	104.1
BENZO(B)FLUORANTHENE	187.0	20.3
BENZO(K)FLUORANTHENE	***	142.1
BENZO(E)PYRENE	166.7	20.6
DIBENZO (A,H)ANTHRACENE	196.2	27.9
BENZO (G,H,I)PERYLENE	189.5	43.3

*** not quantitated

SECTION 7

CAPILLARY GC ANALYSIS

The qualitative and quantitative evaluation of priority pollutant chlorinated pesticides and PCBs mixture (AROCHLOR 1242) was performed at Georgia Tech by high resolution gas chromatography after a preliminary cleanup step by GPC. Glass capillary columns coated by 5 different stationary phases were evaluated in order to establish the best performance in terms of separation capability toward pesticide and PCBs. The gas chromatographic system was equipped with an Electron Capture Detector and Liquid Automatic Sampler. It was possible to resolve organochlorine pesticides from PCBs in sludge extract by means of glass capillary columns coated by different stationary phases (SP-2401 and CARBOWAX-20M).

7-1 Preparation of glass capillary columns for GC analysis

Generally, the preparation of a glass capillary column can be defined in the following steps:

a) column material selection
b) surface treatment
c) deactivation
d) coating
e) evaluation

a. Soda-lime borosilicate glass tubes were used as starting material. The tubes were washed with a detergent solution, rinsed with water and acetone, and finally dried by means of heat-gun and flow of nitrogen. Capillaries were drawn using a Shimadzu GDM-1 glass-drawing machine. Operating at drawing ratio 100, it was possible to obtain capillaries with I.D. 0.35 mm and about 90 m long from tubes with O.D. 7 mm, I.D. 5 mm and 120 cm long. After drawing, each capillary was sealed at both ends if not used immediately.

b. Relatively polar stationary phases exhibit surface tension values different from that of a non-treated glass surface. As a result of this, nonuniform spreading of the phase, and in some circumstances the formation of droplets on the inner wall of the capillary, can occur. Capacity, temperature stability, and deactivation of the surface complete the list of requirements that define suitable surface treatments for the successful preparation of glass capillary columns. Table 7-1 gives summaries of all the methods of glass treatment available in literature, together with the type of glass being used and the effects produced. We have been using three of the reported methods. Soda-lime glass capillaries

Table 7-1 SURFACE TREATMENT FOR GLASS CAPILLARY COLUMNS

METHOD AND CONDITION	GLASS TYPE	EFFECT AND REMARKS
DEPOSITION OF EXTERNAL MATERIAL		
CARBONIZATION: CH_4Cl_2 Pyrolysis	Soft + Borosilic	Thin layer of carbon. Suitable for moderately polar phases; difficult to reproduce
COLLOIDAL SUSPENSION	Soft + Borosilic	Layer of: siliceous material, graphitized carbon black. High capacity columns.
CRYSTALLIZATION: IN SITU REACTION OF $Ba(OH)_2 + CO_2$	Soft + Borosilic	Stable $BaCO_3$ crystals
CHEMICAL BONDING: ORGANOSILICONE, GRIGNARD'S REAGENTS Gas phase 260°C	Soft + Borosilic	Replacement of hydroxyl groups by organic monolayer of polymer.
IMPREGNATION WITH NON-IONIC SURFACTANT	Soft + Borosilic	Thin, physical held layer of polar substance. Low temperature stability. Retention behavior
DIRECT REACTION WITH COMPONENTS OF GLASS		
GAS PHASE HCl ETCHING (300°-350°C)	Soft	High density NaCl crystals. Easy and fast
ORGANIC DECOMPOSITION AND DEVELOPMENT OF HCl or HF (300°C)	Soft + Borosilic	Silicate and NaCl crystals and thin carbon layer. Very mild etching
STRONG ALKALI HYDROXYDE LIQUID PHASE ETCHING	Soft	Leaching Process. Phases may deteriorate. Low column efficiency
FLUOROETHER ETCHING (400°C)	Borosilic	Silicate Wiskers. Very large surface area; suitable for wide range in phase polarity. Active surface.

were dynamically etched by gaseous dry HCl at high temperatures (350^0-380^0C). Borosilicate glass capillaries were treated in two different ways. The first method consisted of the deposition of very fine particulate layers of silica on the inner wall by pushing a plug (10% entire length of column) of diluted colloidal suspension of silicic acid through the capillary at a constant speed of 1-2 cm/sec, as reported by Shulte (1976). The second method consisted of the growth of silica whiskers on the inner wall of the glass capillary by the action of HF developed by decomposition of 1,2-dichloro 1,2,2-trifluoroethyl methyl ether at high temperature (360^0-400^0C), as reported by Pretorius (1975).

c. The presence on the glass surface of silanol groups and "Lewis acid active sites" (Ca++, AP+++, Mg++, B+++) requires that a suitable deactivation be performed prior to the coating of the glass-capillary by the stationary phase. Glass capillary columns coated by relatively non-polar and medium polarity stationary phases (SE-30, OV-101, OV-17, SE-54), if not adequately deactivated, will exhibit partial or complete adsorption of polar compounds on the surface. Glass capillary columns coated by relatively polar stationary phases (CARBOWAX 20M, EMULPHOR ON-870) may not require prior deactivation since the polar groups of the phase itself neutralize the active sites of the glass surface. Diluted solutions of CARBOWAX-20M and EMULPHOR ON-870 (0.05% w/v in methylene chloride) have been used for the deactivation of (soda-lime) HCl-etched and (borosilicate) silic acid-treated glass capillaries. The method used follows Grob's instructions for the deactivation of $BaCO_3$ treated surface (1978). Whisker columns presenting higher "activity" were deactivated by polymerisation on the surface of N-cyclogexyl-3-azetidinol (CHAZ), as reported by Verzele (1977).

d. Glass capillary columns were coated following two different methods. CARBOWAX-20M and EMULPHOR ON-870 phases were coated by the dynamic Hg plug method, as reported by Schomburg (1974). 15 - 20% of the entire length of the column was filled with 10% (w/v) solution of the phase in methylene chloride. A Hg plug (about 3 - 4 cm long) immediately followed the solution; any air bubble between the two phases must be avoided. A nitrogen carrier-gas line, regulated by a fine pressure regulator, was used to push the solution at a constant speed of 0.5 cm/second. In order to avoid temperature gradients that could result in nonuniform film thickness of the stationary phase, the column was placed in a water bath. A dummy column was connected to the open end of the glass capillary. Since the length of the glass capillary col-

Table 7-2 **Relative Retention Time of Chlorinated Pesticides (Relative to Aldrin)**

Compound	SE-30	SE-52	OV-17	SP-2401	CW-20M
α-BHC	0.69	0.75	0.80	0.89	0.95
β-BHC	0.75	0.81	0.93	0.99	1.42
Y-BHC	0.76	0.82	0.88	0.95	1.12
Heptachlor	0.93	0.94	0.94	0.96	0.98
*Aldrin	1.00	1.00	1.00	1.00	1.00
Heptachlor epoxide	1.07	1.09	1.11	1.16	1.31
Endosulfan	1.15	1.16	1.18	1.22	1.34
Dieldrin	1.21	1.23	1.24	1.27	1.47
pp'-DDE	1.20	1.19	1.23	1.18	1.49
pp'-DDT	1.20	-	-	1.35	1.49
pp'-DDD	1.27	1.28	1.35	1.32	1.84
Endrin	1.23	-	-	-	1.50

SE-30: L = 30 m; I.D. = 0.35 mm; $d_f \simeq 0.2$ μm (film thickness)
Working temperature: 100°- (3°C/m) - 230°C; carrier-gas = He/H_2

SE-52: L = 50 m; I.D. = 0.35 mm; $d_f \simeq 0.2$ μm
Working temperature: 100°-(3°C/m) - 230°C; carrier-gas: He/H_2

OV-17: L = 45 m; I.D. = 0.35 mm; $d_f \simeq 0.2$ μm
Working temperature: 100° -(3°C/m) - 240°C; carrier-gas: He/H_2

SP-2401: L = 40 m; I.D. = 0.35 mm; $d_f \simeq 0.2$ μm
Working temperature: 70°- (3°C/m) - 210°C; carrier-gas: He/H_2

CW20M: L = 40 m; I.D. = 0.35 mm;
Working temperature: 100°C - (3°C/m) - 210°C; carrier-gas: He/H_2

umns prepared did not exceed the length of 50 meters, all the relatively non-polar and medium-polar stationary phases were coated with the static method for reasons as reported elsewhere. Each column was conditioned in the GC, under a flow of He (1 - 2 ml/m) at a temperature program rate of $0.5^{o}C/m$.

e. The set of glass capillary columns used for chlorinated pesticides and PCBs analysis was evaluated in terms of efficiency, (expressed as number of effective plates and separation number, with respect to chlorinated pesticides). A more extensive evaluation of the glass capillary columns will be performed in order to establish their suitability for the priority pollutants analysis. A standard qualitative and quantitative evaluation test, recently introduced by Grob (1977), will be treated in detail.

7-2 Investigation of Stationary Phases and GC Conditions

The selectivity of different stationary phases and the high separation power of glass capillary columns have been exploited for the resolution of chlorinated pesticides and PCBs eluting in the same gel permeation fraction. The stationary phases were selected in order to cover a wide range in relative polarity in an attempt to optimize the resolution of chlorinated pesticides. The five glass capillary columns selected for this study are listed in Table 7-2.

The evaluation of all the samples was performed by means of a Hewlett/Packard 5830A gas-chromatograph, keyboard controlled and equipped with a H/P split-splitless capillary injection system, Electron Capture Detector (model 18803-A), and Liquid Automatic Sampler (model 7672-A). He/H_2 (91.5%/8.5%) has been used as carrier-gas, while Ar/Me (95%/5%) has been used as make-up gas in order to satisfy the required flow conditions for the ECD.

The samples were analyzed under temperature program conditions at average carrier-gas velocity of 40 - 45 cm/second. All the components of each sample were eluted within 1 hour of analysis time. Standard solutions of chlorinated pesticides, AROCHLOR 1242 and chlorinated pesticides + AROCHLOR 1242 were evaluated on each glass capillary column in order to establish the separation capability. Figures 7-1 to 7-6 show the chromatograms of each standard solution for the SP-2401 and CARBOWAX-20M stationary phases. The results relative to the resolution of organochlorine pesticide from PCBs and their relative retention times are reported on Tables 7-3 and 7-4, respectively.

RT	Compound
22.59	α-BHC
24.47	γ-BHC
24.91	Heptachlor
25.85	β-BHC + Aldrin
30.87	Heptachlor epoxide
31.53	pp'-DDE
32.69	Endosulfan
33.23	op'-DDT
34.39	Dieldrin
35.95	pp'-DDD
36.76	pp'-DDT

1 µℓ splitless injection (30")
SP-2401; L ≈ 40 m; I.D. = 0.35 mm
carrier gas He/H_2 (91.5% + 8.5%); P = 1.5 atm

Temperature program: 50° -(4°C/m) - 200°C

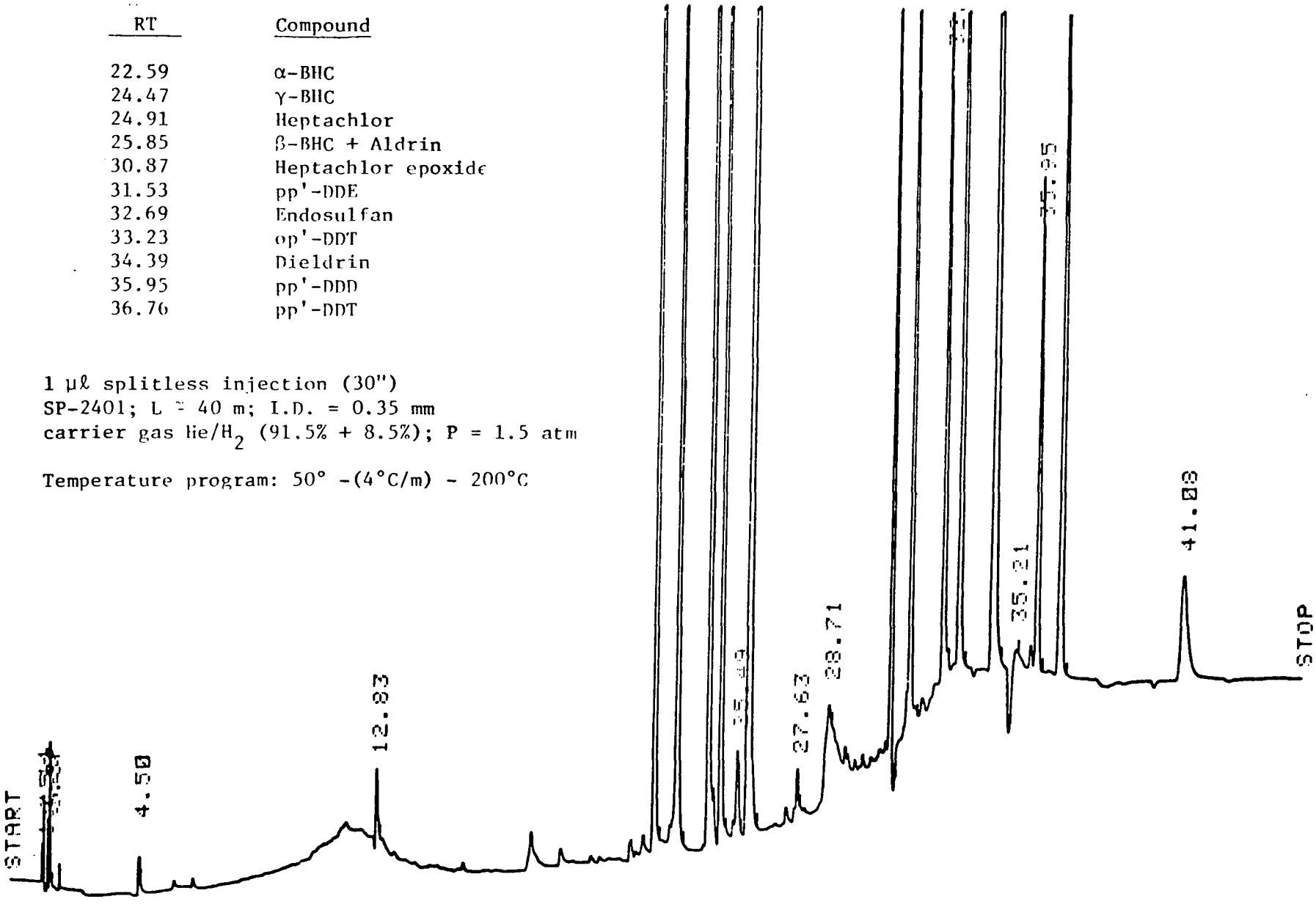


Fig. 7-1 Pesticide standard solution in Hexane (200 ppb)

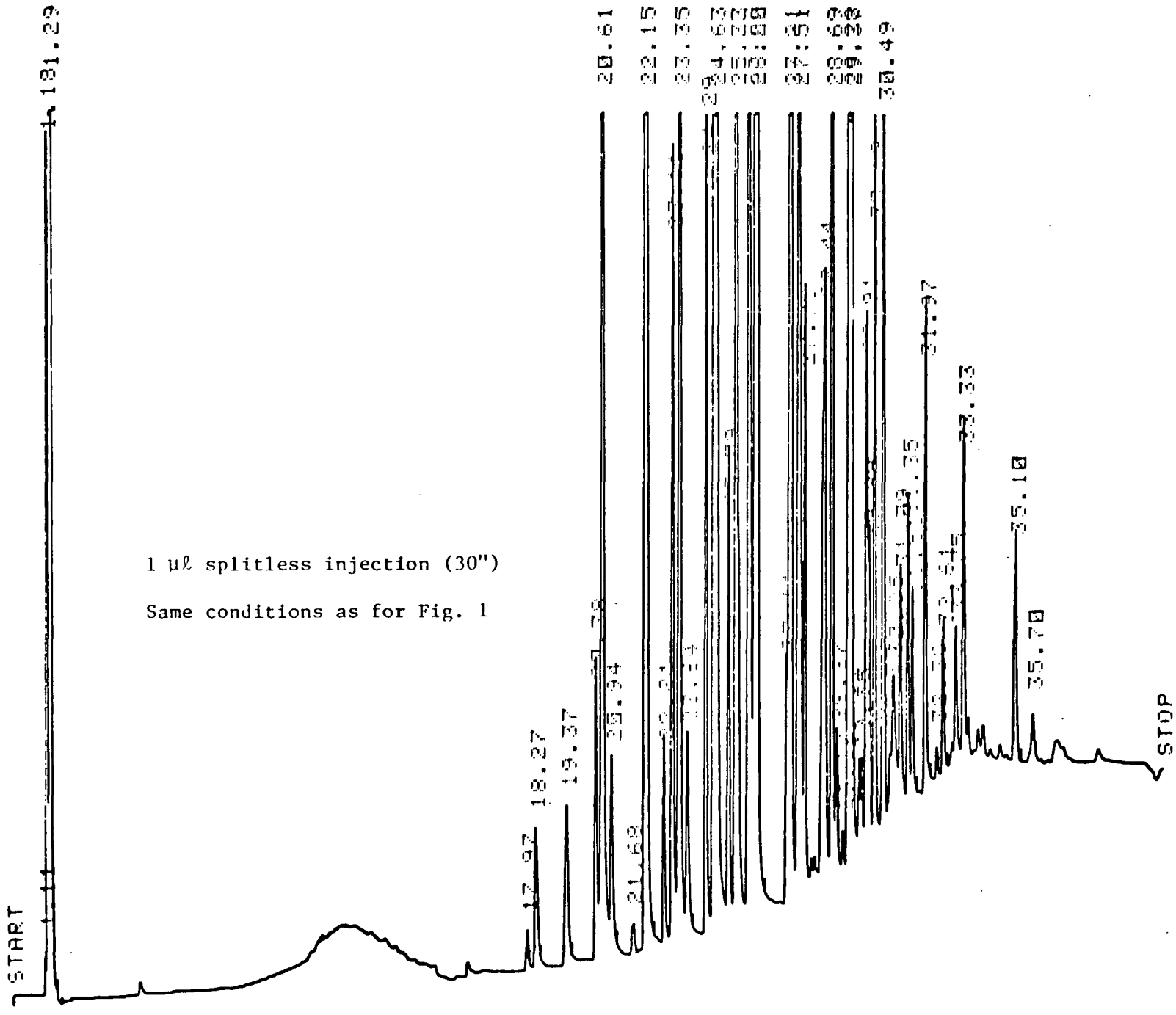


Fig. 7-2 PCBs (AROCHLOR 1242) standard solution in Hexane (2 ppm)

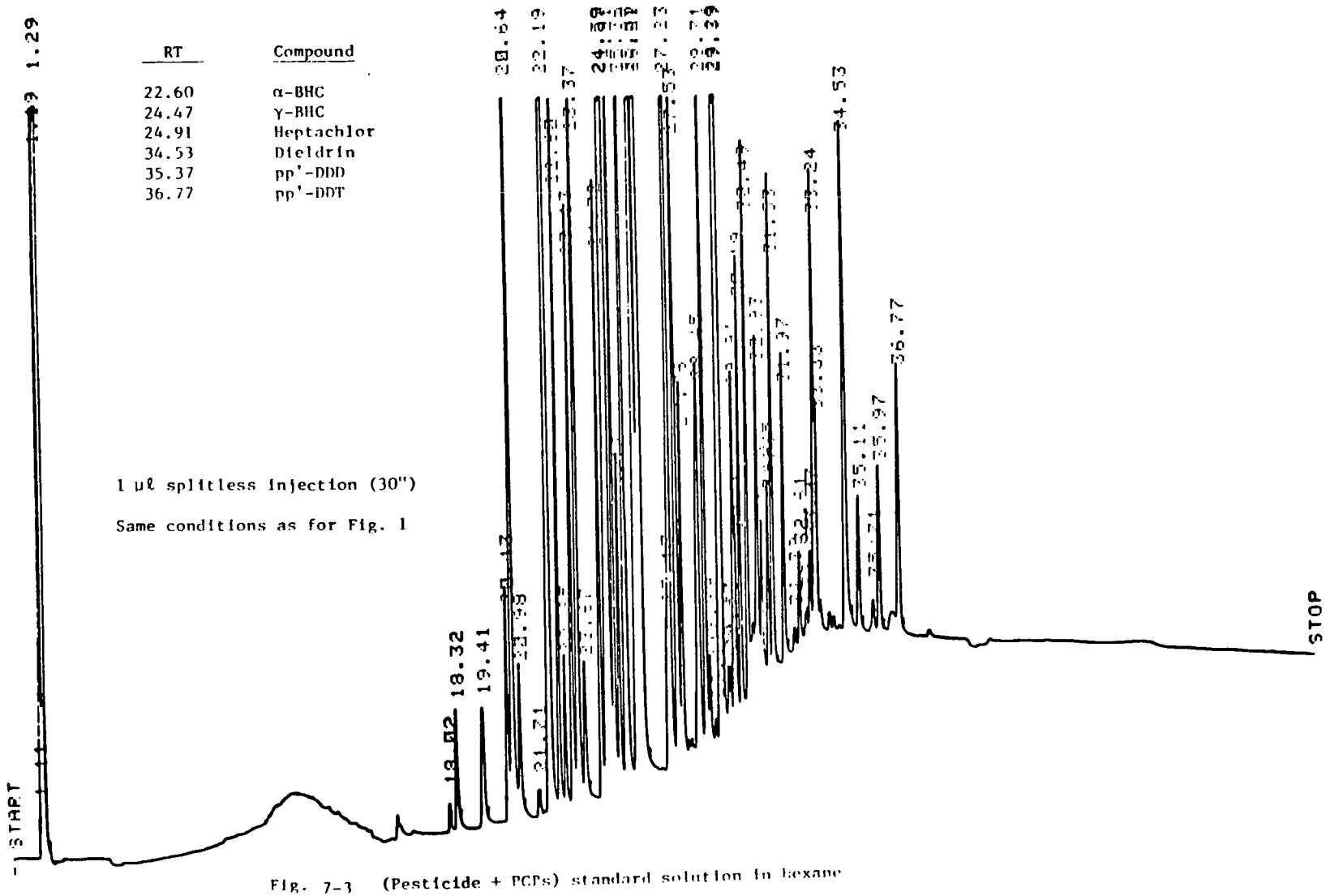


Fig. 7-3 (Pesticide + PCPs) standard solution in hexane

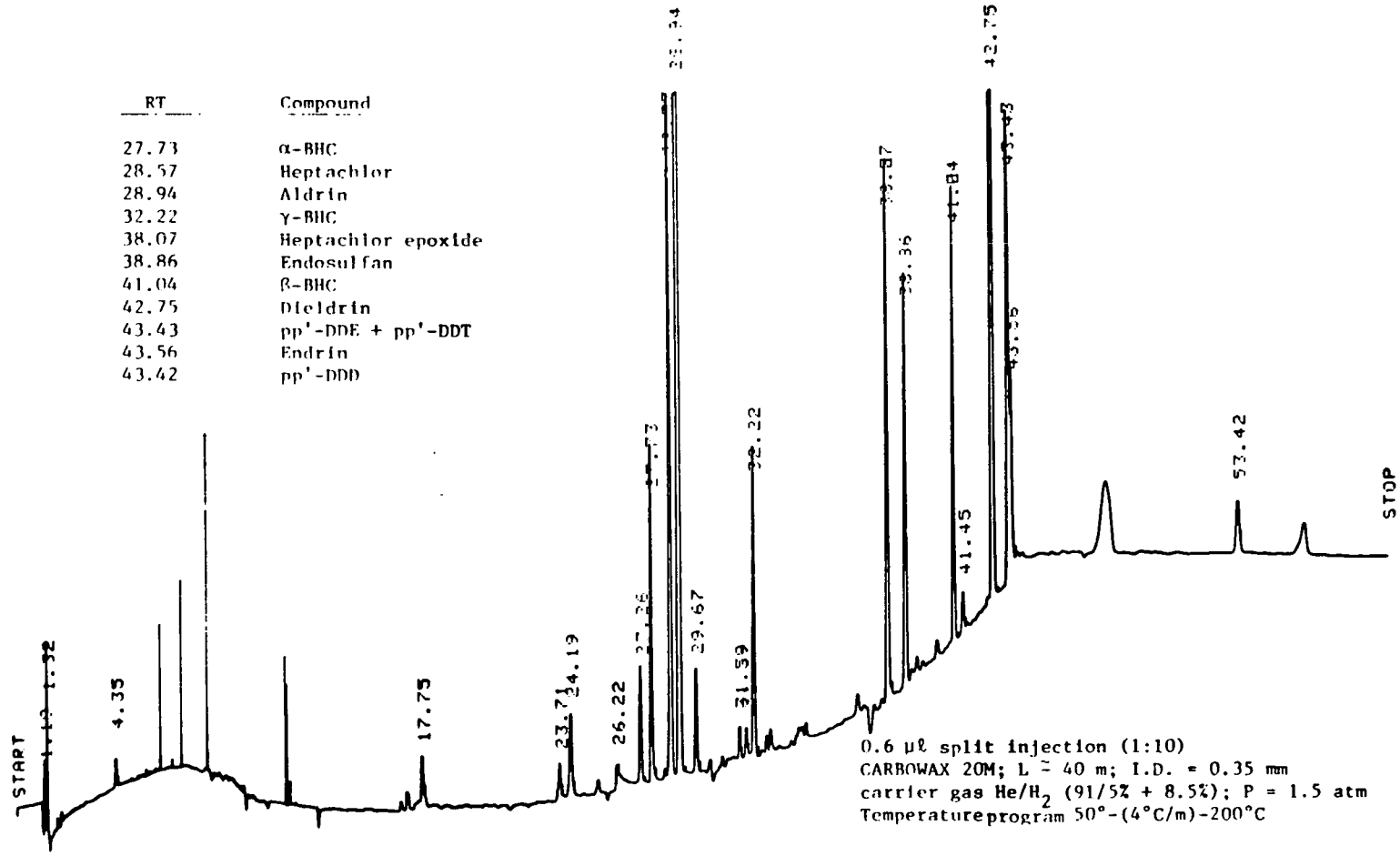


Fig. 7-4 Pesticide standard solution in Hexane

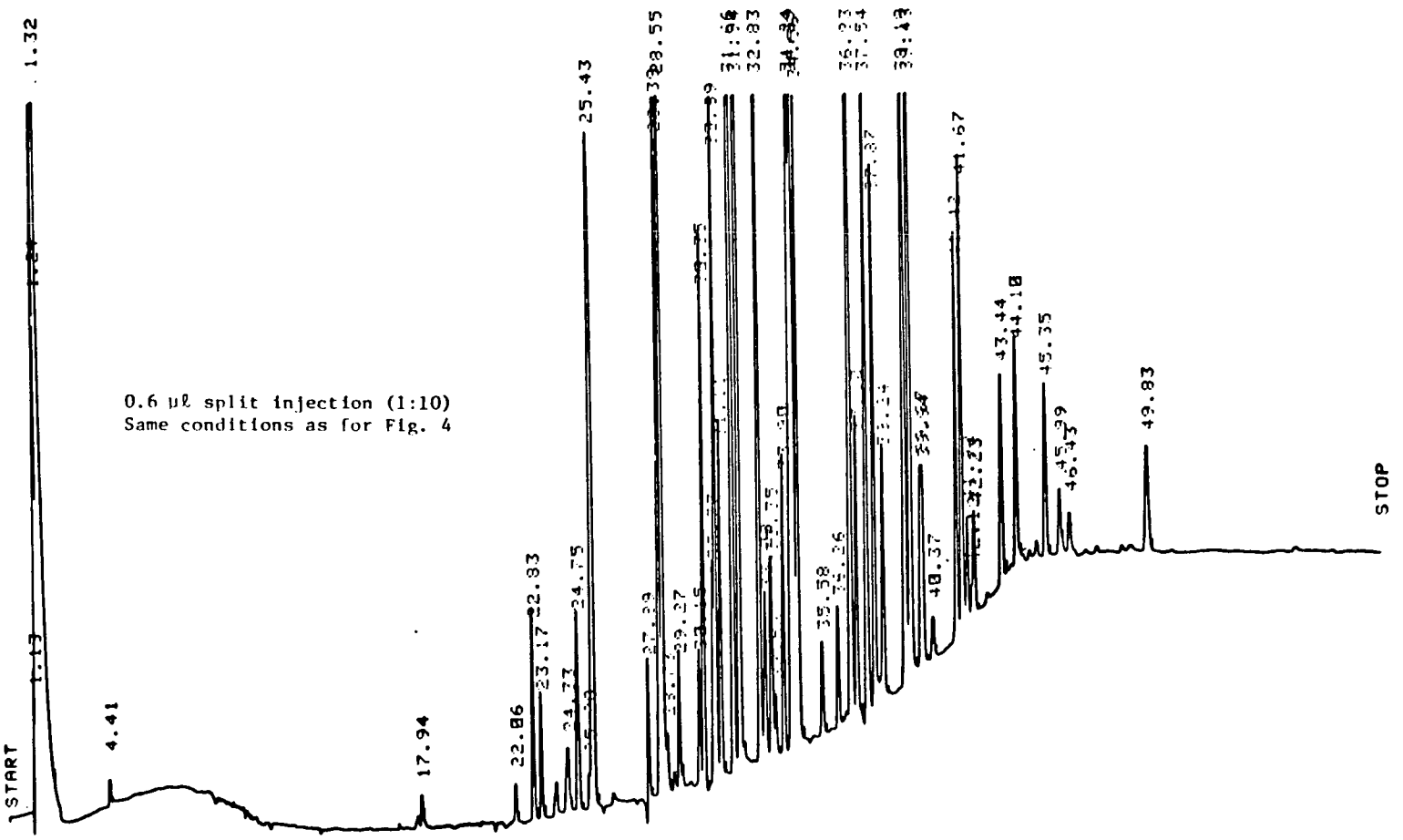


Fig. 7-5 PCBs (AROCHLOR 1242) standard solution in Hexane

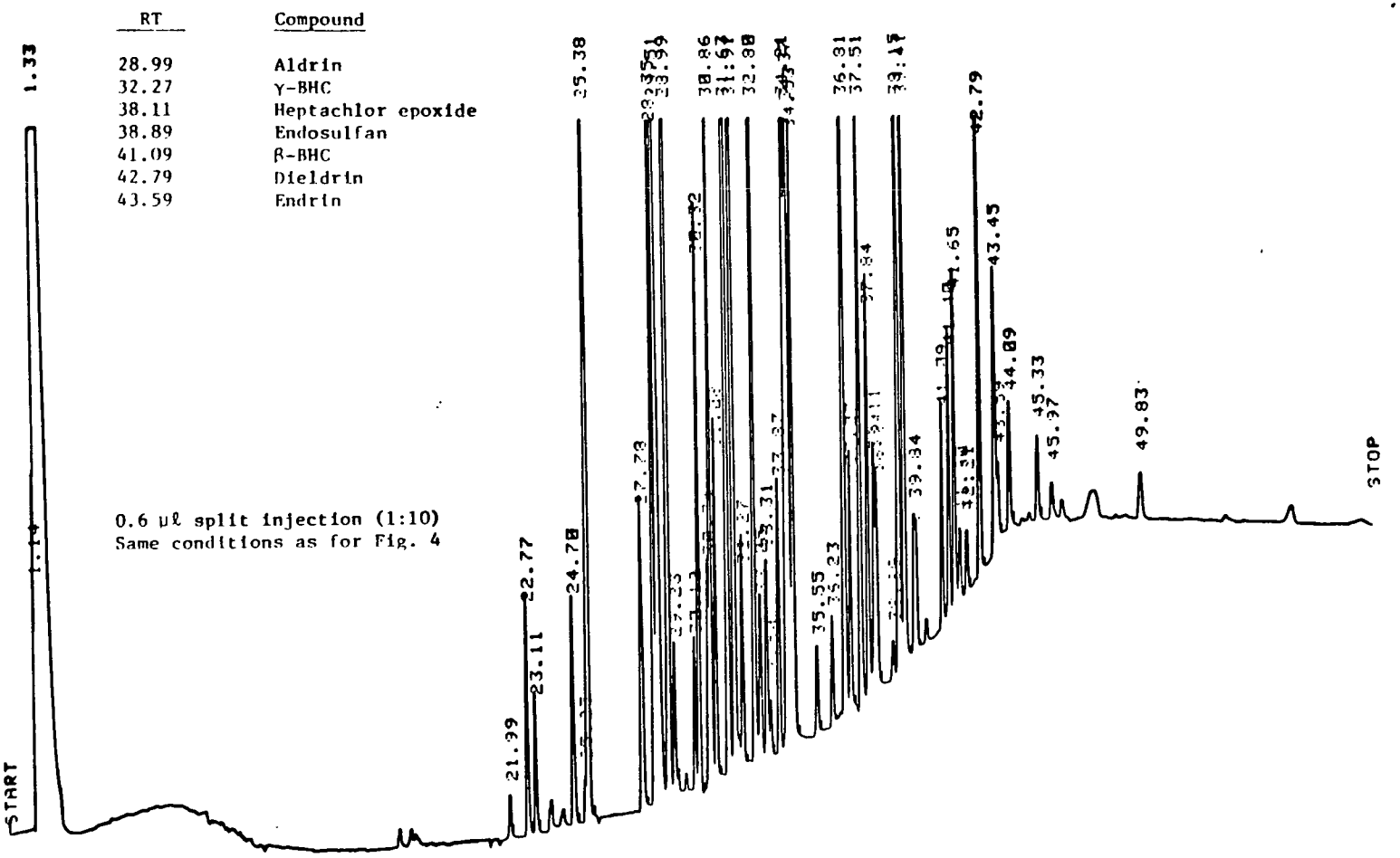


Fig. 7-6 (Pesticide + PCBs) standard solution in Hexane

Table 7-3 **Glass Capillary Columns Selected**

Stationary Phase	Etching	Deactivation	Coating	L(m)	I.D. (mm)
SE-30	dry HCl	CW-20M + Emulphor	Static	30	0.35
SE-52	"	"	"	50	0.35
OV-17	"	"	"	45	0.35
SP-2401	"	"	"	40	0.35
Carbowax-20M	"	-	Dynamic	40	0.35

Table 7-4 Organochlorine Resolved from PCBs

Compound	SE-30	SE-52	OV-17	SP-2401	CW-20M
α-BHC	X		X	X	
β-BHC	X	X	X		X
γ-BHC	X	X	X	X	X
Aldrin	X	X			X
Heptachlor			X	X	
Heptachlor epoxide			X		X
Endosulfan		X			X
pp'-DDE		X			
Endrin					X
Dieldrin	X			X	X
pp'-DDD	X	X	X	X	X
pp'-DDT				X	

In order to identify any major interference related to the matrix, both spiked distilled water extracts and spiked sludge extracts were evaluated. Figures 7-7 to 7-12 show the EC traces from analyses of pesticide spiked sludge, pesticide + AROCHLOR 1242 spiked sludge and, pesticide spiked distilled water, respectively, on CW-20M stationary phase. One conclusion, suggested from a survey of the results, is that it is possible to evaluate organochlorine pesticides and PCBs eluting in the same gel permeation fraction by means of two glass capillary columns coated by different stationary phases and Electron Capture Detection. The correlation of results from the use of different stationary phases will also improve the possibility of resolution and identification of any major interference related to the matrix itself. From the results obtained in our evaluation, it was possible to determine that some minor peaks present in the non-spiked sludge extract were themselves organochlorine pesticides and PCBs.

Some difficulties were encountered in the evaluation of pp'-DDT and Endrin. As reported in the literature, these compounds may undergo decomposition under the gas chromatographic conditions. Temperature is the main factor, but active sites in the system may catalize the degradation process. Since we observed evidence of decomposition that was particularly severe when using glass capillary columns coated by non-polar stationary phases (OV-17, SE-52), the responsibility of this effect was ascribed to the presence of residual activity in the column, due to insufficient deactivation of the surface.

The development of the gas chromatographic system will be directed to three areas: 1) better deactivation of columns, 2) shorter analysis time, and 3) evaluation of other stationary phases. In light of the negative results for nonpolar glass capillary columns, more complete deactivation of the surface will be sought. Shorter capillaries with smaller internal diameters will improve the analysis time; this will also imply less contact time between the labile compound and the system, and therefore less chance to undergo decomposition, at the expense of some resolution and column capacity. SE-54 and 52 are among the preferred stationary phases.

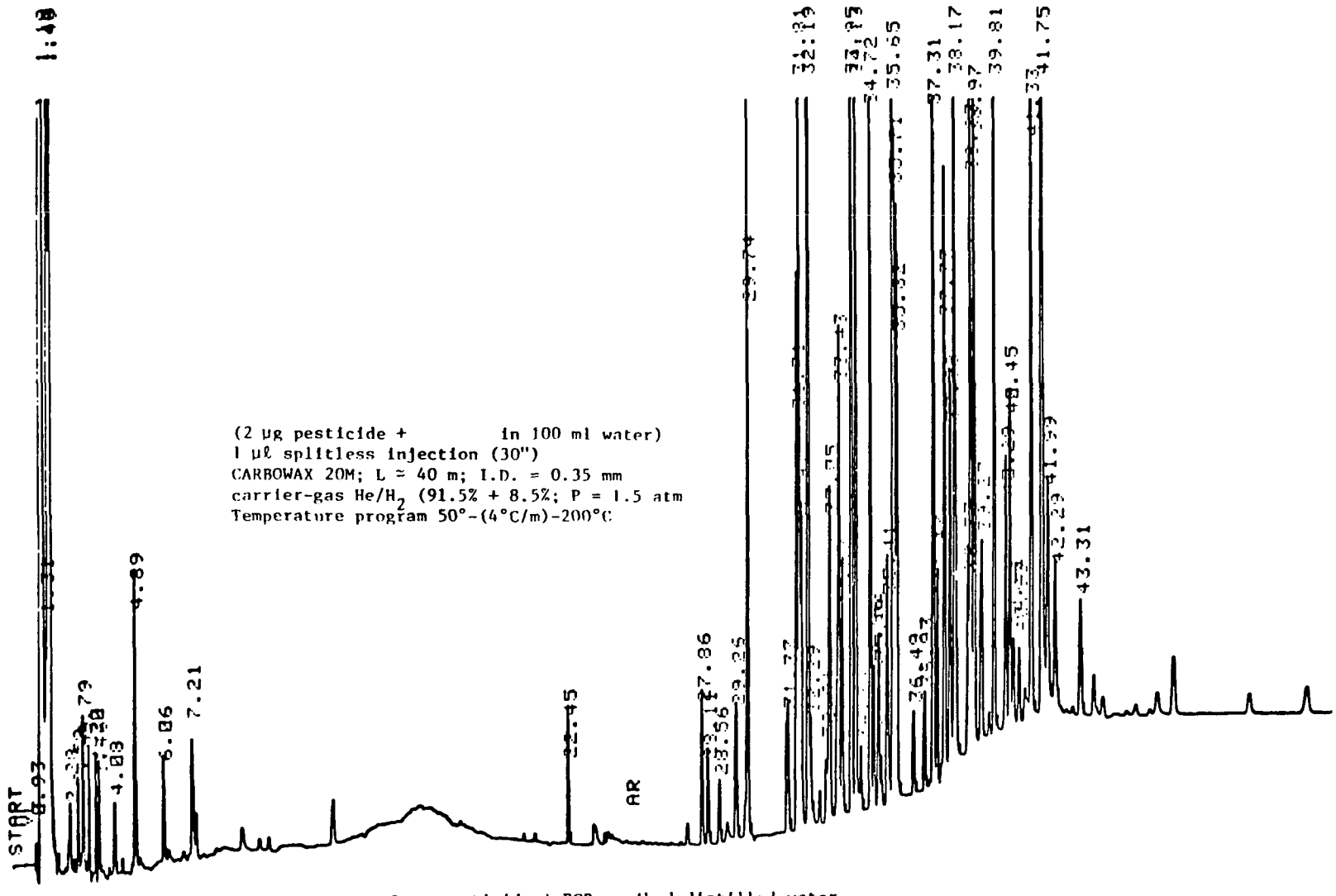


Fig. 7-7 Extract from pesticide + PCBs spiked distilled water

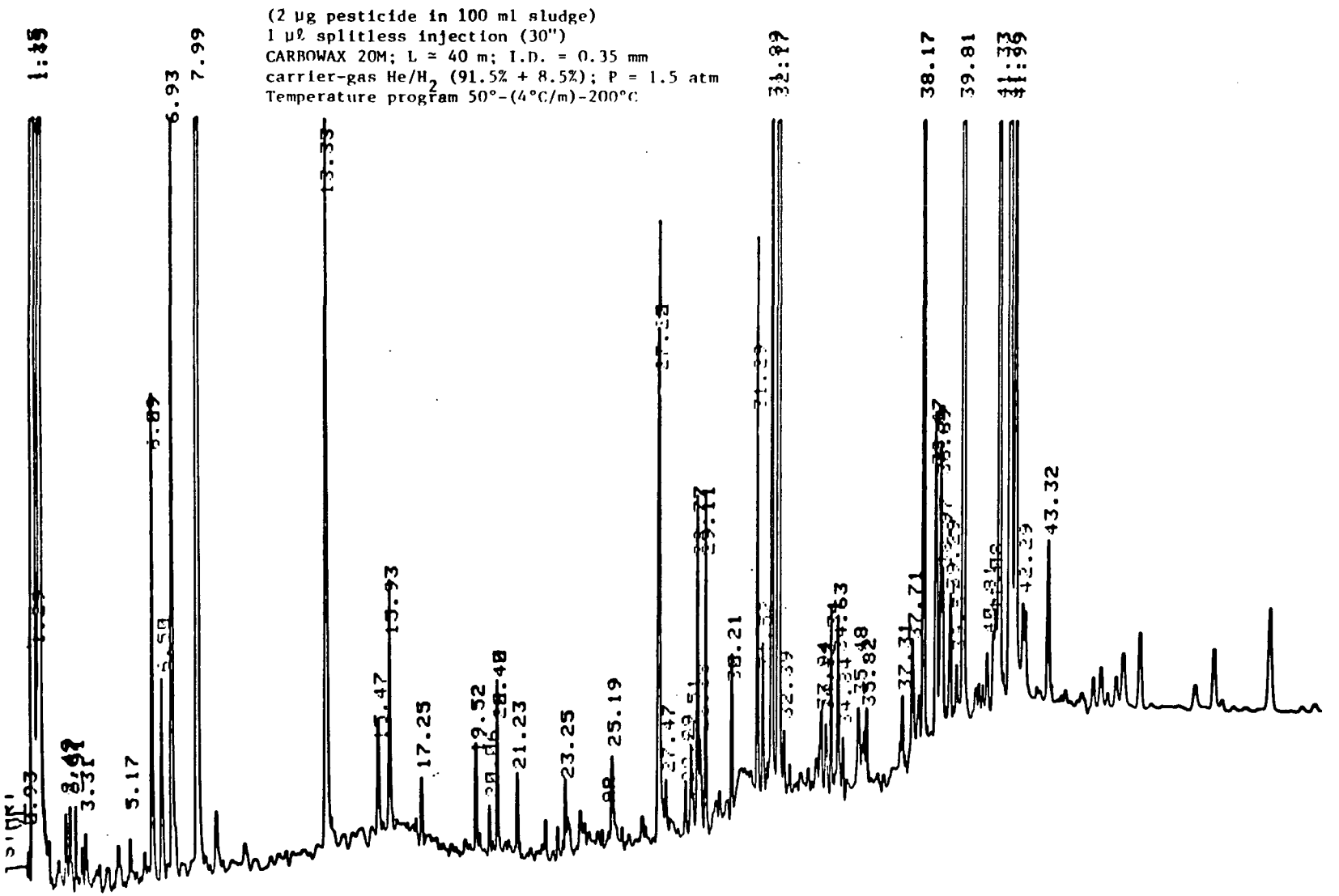


Fig. 7-8 Extract from pesticide spiked sludge

Fig. 7-9 Extract from pesticide + PCBs spiked sludge

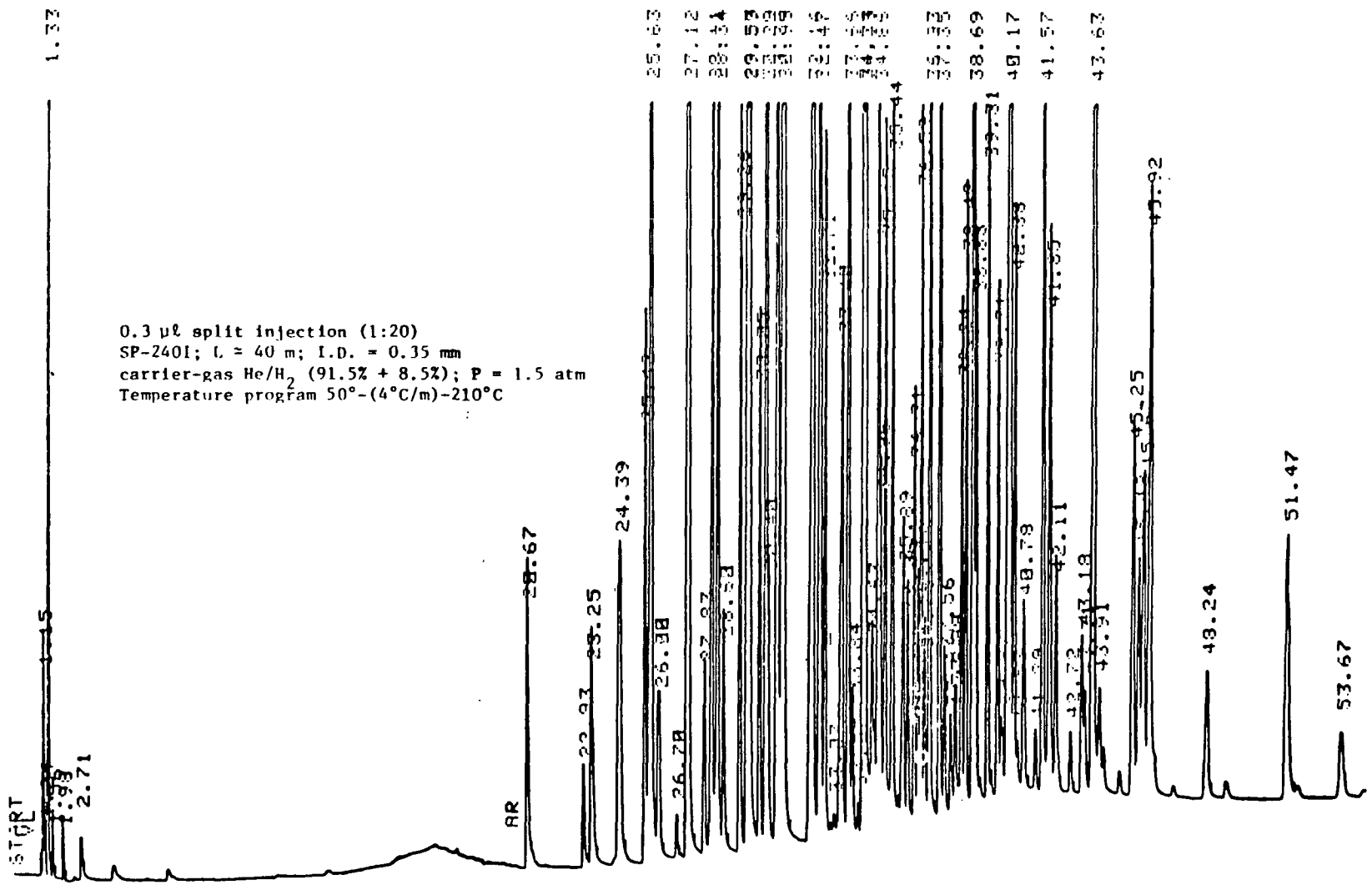


Fig. 7-10 PCBs mixture #2 SUPELCO (AROCHLOR 1221 + 1242 + 1254)

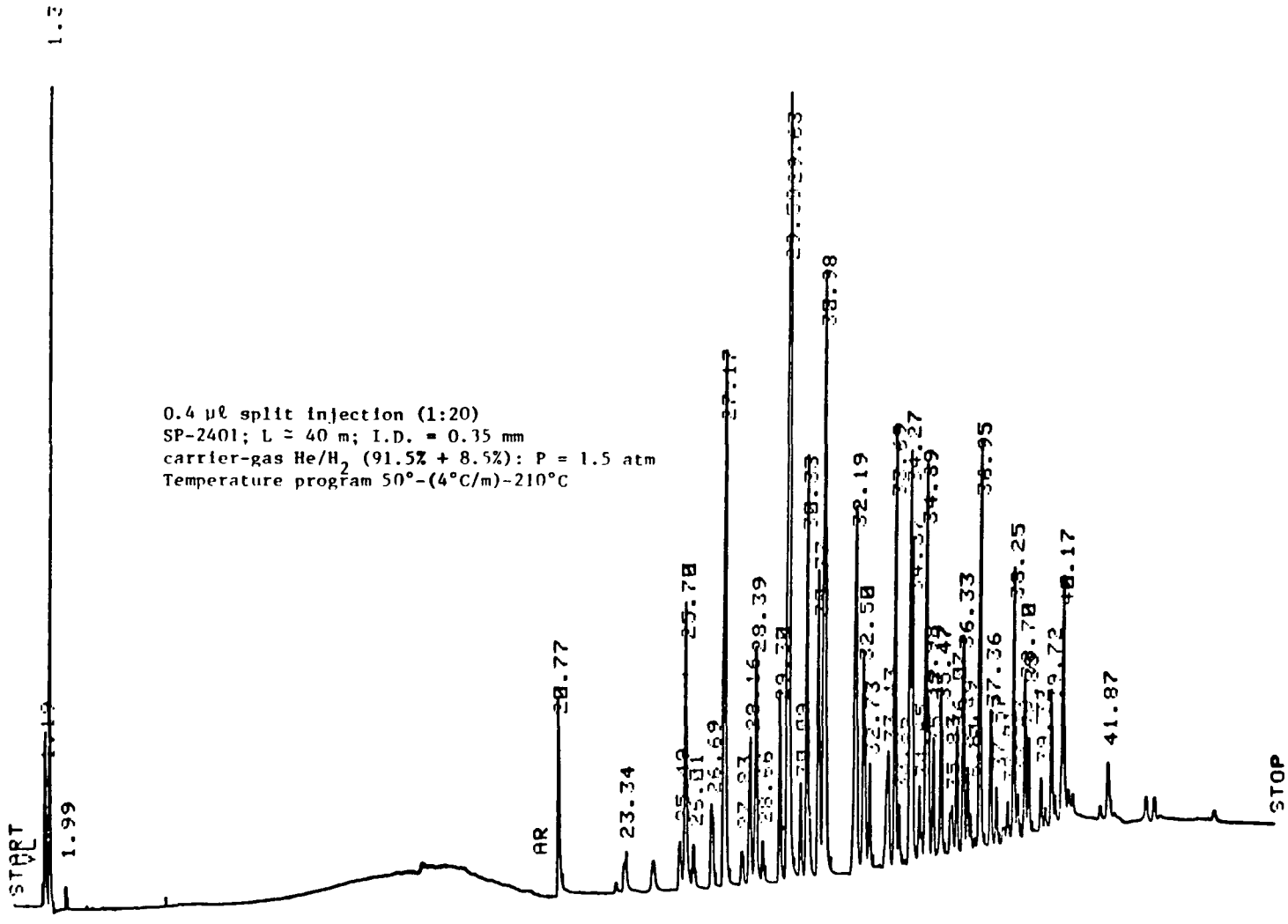


Fig. 7-11 PCBs mixture #1 SUPELCO (AROCHLOR 1016 + 1232 + 1248 + 1260)

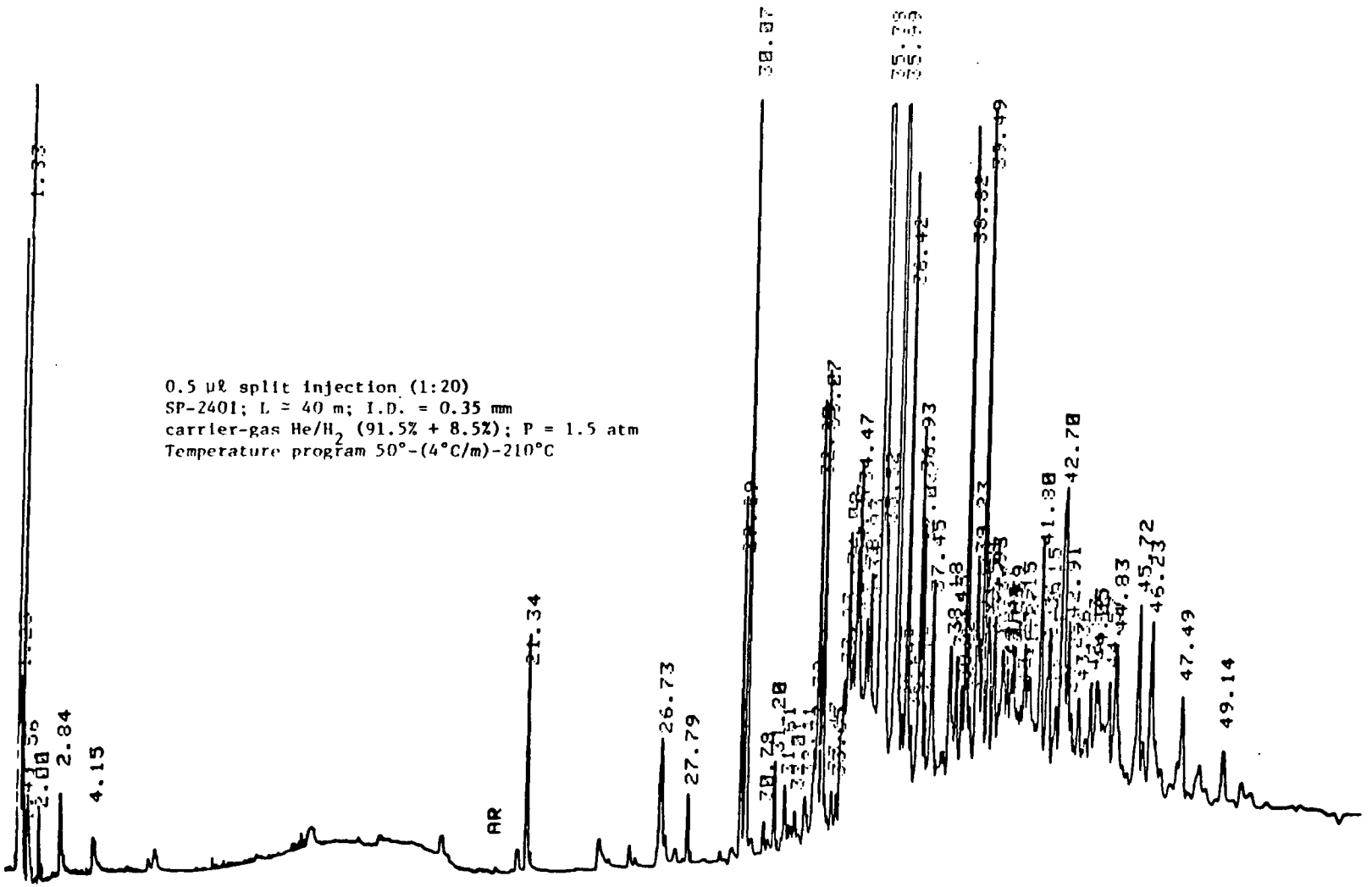


Fig. 7-12 **Toxaphene + Chlordane mixture SUPELCO**

SECTION 8

GC/MS/DS ANALYSIS OF PRIORITY POLLUTANTS

8-1 Chromatography of Organics

The base-neutral and pesticide priority compounds exhibit varying chemical properties. These compounds can be chromatographed successfully on glass capillary columns coated with different liquid phases. Among the different types of columns, two have proven to be distinctly superior to most others for routine analysis of these compounds. The phases SE-52 and especially SE-54 (10% phenyl and 5% phenyl + 5% vinyl methyl silicones, respectively) are preferred in our laboratory and in others, including the National Analytical Facility of NOAA, the Woods Hole Oceanographic Institute, and the Swiss Federal Institute for Water Resources and Water Pollution Control (EAWAG). While the resolution obtained with these two phases is not significantly different from that achieved by the commonly used methyl silicones, SE-30 and OV-101 (AP-2100), the durability, stability, and longevity of the phenyl substituted phases is generally superior. These characteristics have been most strikingly observed during the analysis of polynuclear aromatic hydrocarbons.

A standard mixture of acids, bases, neutrals, and pesticides, as used in the liquid/liquid extractor experiments, was analyzed by both GC/FID and GC/EI-MS. The chromatograms from these runs are shown in Figures 8-1 and 8-2, respectively. The analyses were done on two different 30 meter SE-54 glass capillary columns using identical GC conditions without stream splitting. The resolution obtained in these two runs is essentially identical. It should be noted that several compounds are still unresolved even though the overall resolution is much greater than that achieved on packed columns. Examples of this are the hexachloroethane + di-n-isopropyl-n-nitrosamine (peak 2), and the acenaphthylene + hexamethyl benzene (peak 11). In the first case, no separation is observed, even of selected masses on the GC/MS run. The GC/MS data system, however, is still frequently able to identify the components of mixed peaks correctly, as is shown in the library search report of the mixed spectrum obtained from peak #2 (Figures 8-3 and 8-4). The severely tailing peaks, #1, #5, and #8, in these chromatograms, represent or contain phenols which show significant adsorption problems on this column type. In contrast, the phenols when derivatized to their methyl ethers, chromatograph very readily on this column, as was indicated earlier.

Two of the compounds in the test mixture have shown some degradation problems in their GC analyses, either from thermal pyrolytic conditions during analysis, or other reaction conditions on standing in solution, or both. Diphenyl hydrazine (peak #17) is converted to diphenyl diazine which co-chromatographs

Figure 8-1 Analysis of Standard Mixture by GC/FID.

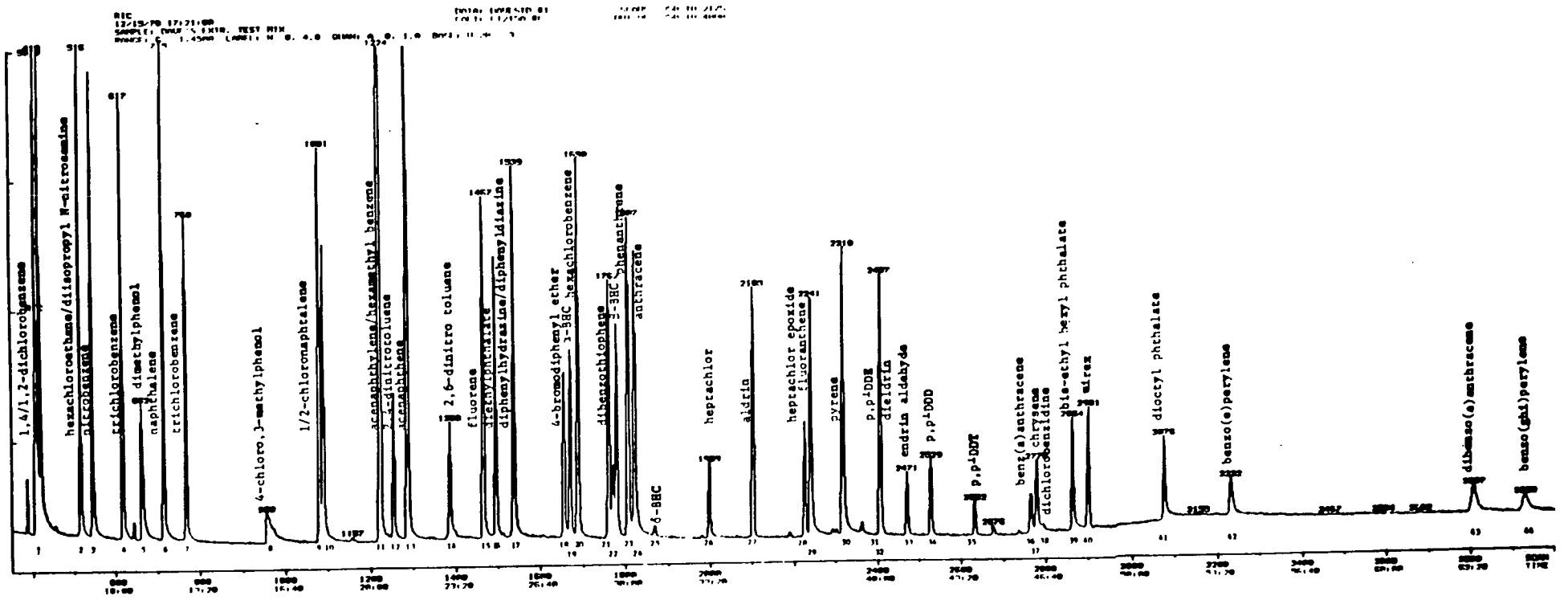


Figure 8-2 Analysis of Standard Mixture by GC/EIMS.

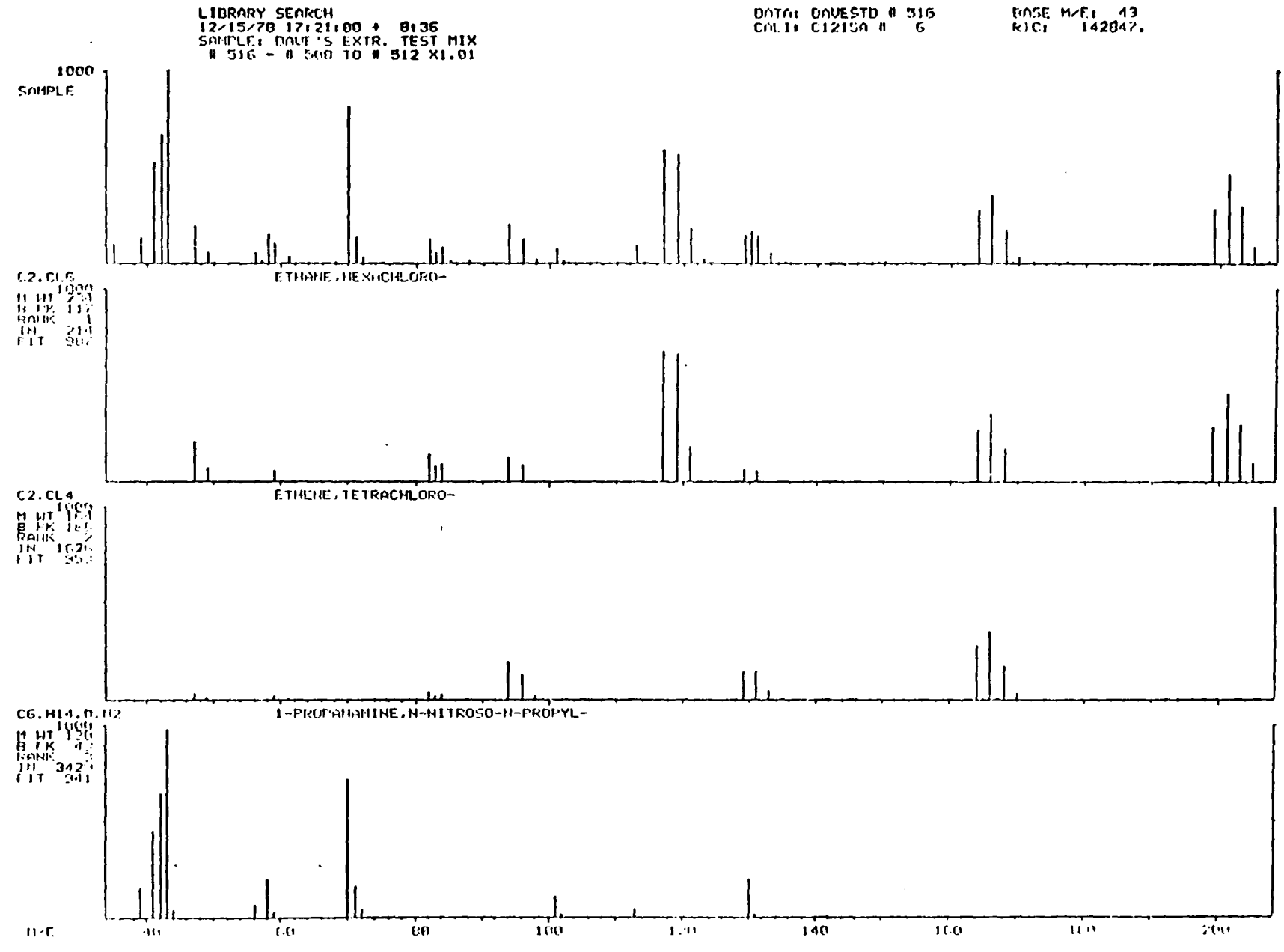


Figure 8-3 Spectra of Unresolved GC/MS peak No.2

```
LIBRARY SEARCH                  DATA: DAVESTD # 516        BASE M/E:    43
12/15/78 17:21:00 +  8:36       CAL1: C1215A #   6         RIC:      142847.
SAMPLE: DAVE'S EXTR. TEST MIX
 # 516 - # 508 TO # 512 X1.01

25410 SPECTRA IN LIBRARYNB SEARCHED FOR MAXIMUM FIT
  280 MATCHED AT LEAST 4 OF THE 16 LARGEST PEAKS IN THE UNKNOWN
REDUCTION:    PKS/100 AMU:      40;  WINDOWS:   50,  7
PRE-SEARCH:   ENTRIES TO PASS: 100;  SAMPLE PEAKS: 16
MAIN SEARCH:  MASSES:   34 - 534;  NORM INT:   25;  RATIO FACTORS:    2.0,  1.0

RANK  IN  NAME
1     214 ETHANE, HEXACHLORO-
2    1626 ETHENE, TETRACHLORO-
3    3429 1-PROPANAMINE, N-NITROSO-N-PROPYL-
4    9145 1,4-DIOXANE-2,5-DIONE, 3,3,6,6-TETRAMETHYL-
5    1569 PYRROLIDINE
6    1773 L-PROLINE
7    3235 2-PROPANAMINE, N-(1-METHYLETHYL)-N-NITROSO-
8    5560 CYANICACID, 1-METHYLETHYLESTER
9     315 PROPANENITRILE, 2-HYDROXY-2-METHYL-

RANK  FORMULA          M.WT  B.PK  PURITY  FIT  RFIT
1  C2.CL6               234   117     701  987   701
2  C2.CL4               164   166     280  953   283
3  C6.H14.O.N2          130    43     262  941   264
4  C8.H12.O4            172    43     188  925   195
5  C4.H9.N               71    43     167  862   176
6  C5.H9.O2.N           115    70     159  849   173
7  C6.H14.O.N2          130    43     237  847   243
8  C4.H7.O.N             85    70     166  833   184
9  C4.H7.O.N             85    43     170  831   185
```

Figure 8-4 . Library Search of Unresolved GC Peak #2.

with its unaltered parent. Up to 1-2 second separation is observed in the reconstructed mass chromatographs from the GC/MS analysis. Endrin (peak #33) is also partially or completely oxidized to Δketoendrin or endrin aldehyde in most analyses thus far.

As mentioned previously, one of the primary objectives of the development effort is to be able to prepare and analyze samples for neutrals and pesticides in one single fraction, with the bases added prior to analysis. Even though certain compounds in these groups are not resolvable by our chromatographic system using non-specific GC detectors, the GC/MS is still able to easily distinguish them based on their individual mass spectra. In addition, the following compounds have been reported to be unstable for GC analysis: 3,3-dichlorobenzidene, diphenylnitrosamine, dimethylnitrosamine, and bischloromethylether.

8-2 GC/MS Data Processing

A. Initialization

Prior to unknown sample analyses, the Supelco standard priority pollutant mixtures were diluted in methylene chloride and analyzed using the identical GC/MS setup and conditions later used for unknowns. The compound peaks in these runs were identified by computer comparison of their spectra with NBS library and other reference spectra. The best spectra obtainable from these data, after careful background subtraction, were then used to create a new "quantitation" library. During this process, insignificant mass peaks were deleted and additional information was added for each library entry, such as the mass to be used for quantitation and the concentration in the standard mixture. The quantitation library used for the majority of unknown analyses contains spectra of all base/neutral priority compounds.

The relative retention times and relative response factors for each compound in the library were then computed by an initialization program, using a GC/MS analysis of the complete standard mixture run under identical conditions.

The internal standard chosen for these studies was arrived at after some experimentation. Initially, hexamethylbenzene was used, as well as D10 anthracene. In the course of early analyses, it was found that multiple internal standards, spanning a range of retention times, would greatly facilitate data processing by allowing closely set retention time windows. Further, in several environmental samples, D10 anthracene was used as one component of a multiple standard mixture. It was found to attenuate over time relative to the other internal standards. Because of this instability, D10 phenanthrene is substituted as the medium retention-time standard. D8 naphthalene and D12 perylene complete the internal standards. In addition, several surrogate

recovery compounds and other standards are included during sample preparation. The analysis of these spiked compounds provides a useful index of chromatographic behavior for a particular gas chromatogram, which is a significant aid in automated data analysis.

B. Analysis of Unknowns

The initalized quantitation library was utilized by a computer program, called SINUNK, to analyze unknown samples. Only those compounds present in the library are searched for and, when found, are quantitated relative to the internal standard. The essential features of this program are described as follows:

1. The internal standard is searched for in a two minute time window around the expected retention time. If it is located, the program continues.

2. The area of the mass chromatogram peak for the standard's base peak is calculated and compared to an expected value. If it is within acceptable limits, the program continues.

3. The actual retention time of the authenticated internal standard peak is then stored for use in relative retention time calculations.

4. The first library entry is retrieved and the expected retention time calculated. If this value plus 30 seconds exceeds the length of the data file, the program stops searching and reports the quantitation results up to that point. If the file length is not going to be exceeded in subsequent steps, the program continues.

5. The data file is then reverse searched against the library spectrum in a window -/+ 30 seconds around the expected retention time. If no peak is found with a statistical FIT of 750, a "NOT FOUND" is reported for that compound. A FIT of 1000 is a perfect match. If one or more peaks are found in this window, the program continues and the scan number corresponding to the peak of highest FIT is stored.

6. If the FIT is less than 850 but over 750, the spectrum is added to a list of questionable identifications to be forward searched against a library containing all of the priority com-

pounds.

7. The expected retention time is then compared with this best fit scan number. If the "best" retention time is -10, -5, +5, +10, +15, or +20 seconds in error relative to the expected retention time, then the size of the quantitation window is adjusted. This window is normally -/+ 7 seconds around the expected retention time. For example, if an error of +12 seconds is calculated, the window becomes -7 to +17 seconds.

8. The mass chromatogram for the base peak mass is generated over a one minute window around the expected retention time. The peak areas are then computed for only those peaks falling within the quantitation window calculated above and are stored along with other information for the final report.

9. The next library entry is retrieved and steps 4 - 8 are repeated for the entire library.

10. The amounts of each compound found are calculated using the peak areas, relative response factors, and the data output in a tabular format.

The expected retention time, best retention time, PURITY, FIT, library entry number, number of peaks found by sesarch, and number of peaks quantitated are also output for each compound.

8-3 Complications with GC/MS Analysis

1. Certain compounds, such as bischloroethoxymethane, produce poorly diagnostic EI mass spectra and sometimes are falsely identified by reverse library searches.

2. Some isomeric pairs of compounds, such as phenanthrene/anthracene, may not both be detected if the retention times are greatly different from expected values and the wrong member of the pair in the same search window gives a better FIT than the other.

3. Single mass quantitation is not very sensitive for many of the compounds. This is especially serious for those that undergo extensive fragmentation, such as the pesticides. Also interferences at the same mass may cause significant errors in some cases.

By selection of window sizes and FIT/PURITY criteria, one can bias the data analysis program in favor of false negatives or false positives. As a matter of choice, we have chosen the latter. This produces an output which must be edited to remove artifacts. These occur when multiple peaks are assigned the same identity (not infrequent for isometric or homologous compounds) and when the spectrum being searched for contains significant components in common with the background. The latter case can produce a positive where no peak is discernable. Compounds prone to this latter error include the chloroethers, di-nitrotoluenes, and dimethyl nitrosamine. For these compounds, FIT criteria must be stricter and should not be accepted blindly. Chromatographic behavior must be relied on to a greater extent in deciding whether to accept or reject a positive identification.

Examples of potential and actual problems in automated data analysis will be discussed in conjunction with actual analytical results.

SECTION 9

PROPOSED QA/QC PROGRAM FOR POTW SAMPLES

The elements of this proposal are derived from the following key EPA documents: a November 1978 results summary of the Cincinnati POTW sources pilot study QA/QC program (conducted by Arthur D. Little, Inc.); an Arthur D. Little Company handout prepared for an EPA contractors' meeting on method development March 9, 1979; "Recommended Sediment and Sludge Sample Collection Procedures for Priority Pollutant Analysis," Addendum to Sampling and Analysis Procedures for Screening of Industrial Effluents for Priority Compounds (March 1979, EPA - Cincinnati ENSL documents); and BAT Screening Protocols.

QA/QC consists of the following elements: contamination control, containment/recovery of sample, and instrumental calibration. For each element, one phase of the QA/QC program is needed. Contamination control is addressed by carefully defining materials and cleaning procedures for all sampling and preparation materials, and is monitored by blank determinations. Field blanks consist of distilled, organic free water samples exposed to the same glassware and manipulations as a real sample. Lab blanks consist of an aqueous blank run through the extraction/cleanup steps. Containment/recovery is also dependent on sampling and handling technique and is monitored by recovery standards spiked into actual or blank samples. The recovery standards may be model compounds ("surrogates") or mixtures of the actual priority compounds. Instrumental calibration is ensured by the use of external standards to tune instruments uniformly and internal standards to provide cross checks---detecting losses during injection or in procedures occurring between addition of separate standards, as well as monitoring the integrity of each internal standard.

Figure 9-1 shows a schematic representation of the use of various types of standards to monitor QA/QC for different segments of the analysis. The inclusion of specific, noninterfering compounds to provide QA/QC information requires only minimal effort for GC/MS analysis, and is therefore a preferable alternative to parallel analyses of samples unspiked and spiked with priority pollutants.

The actual structure of a QA/QC program has two logical phases -- initiation and maintenance. In the initiation phase, the analysis method is verified by repeated determinations and is shown to be statistically reliable. Laboratories just beginning to follow EPA methods, or applying alternative procedures not yet in routine use, will determine what, if any, experimental problems exist during the initiation phase.

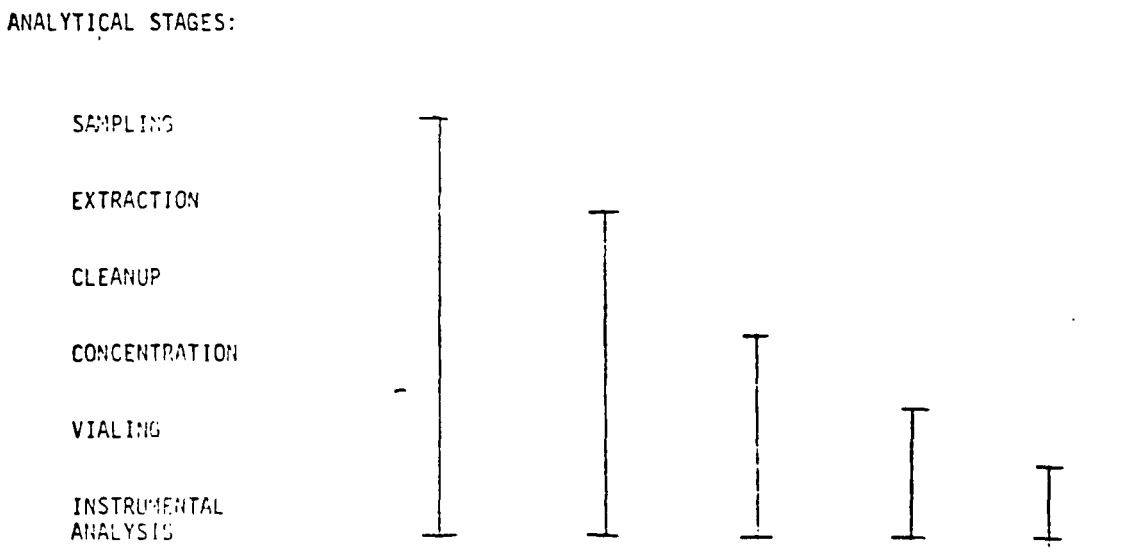


Figure 9-1 Use of Standards and Spiked Compounds

EPA has suggested a 12-fold initiation program involving six sets of replicate determinations of surrogate spiked and priority pollutant spiked samples, with three concentration levels of the spikes. This program provides enough repetition for statistical validation, but is inconvenient in that it does not permit many real analyses to take place concurrently. A reasonable modification would be to perform the different spike concentration replicates on different (but preferably similar) samples. This would permit three real analyses rather than one. The necessity of determining recoveries at three concentration levels, rather than two, is also debatable. It is the extremely low concentration range where QA/QC is most important, and most difficult. Determinations at 1 ppb and 100 ppb might be acceptable in lieu of the prescribed 2x, 10x, and 100x spikes.

Maintenance of QA/QC involves specific analyses to "measure" known spike concentrations. The EPA proposal for a routine QA/QC maintenance sample is: 1) the unspiked sample, 2) a spiked sample, 3) a replicate spiked sample, 4) a spiked distilled water sample, and 5) a system blank. If one sample in 10 is a QA/QC sample, then 14 analyses will be required to produce 10 sets of real data (40%). The following modification is proposed: each sampling event needs a field blank and, if 5 samples are taken on one day or at one time or with one set of glassware or reagents, a blank should accompany this set. The need for replicate analyses to demonstrate reproducibility could be met with suitable recovery compounds in the (sample and sample + spike) pair of analyses. The choice of what type of sample to use for QA/QC (influent, sludge, etc.) will significantly effect the final recoveries seen, since background varies substantially among sample types. Therefore, the importance in appropriate model compounds to provide recovery QA/QC information on each sample cannot be over-emphasized. At present, the choices of available surrogates is limited. Table 9-1 presents the EPA recommendations, as well as those in use at the University of Washington. A satisfactory set of model compounds should represent the range of chemical and physical behavior of the priority pollutants, and may need to contain specific models for difficult (reactive) compounds as well. Perfluorinated compounds have substantially different physical properties from their perproteo counterparts but, together with a limited number of deuterated and C-13 labelled priority compounds, presently are all that is available. Specific classes of priority compounds, such as phthalates and halogenated ethers, have no model currently available. As better sets of model compounds not present in the environment are developed, appropriate reductions in the number of spiked samples could be achieved, with an increase in QA/QC.

One example of the use of multiple compounds for recovery and quantitation is the volatile recovery standard currently in use. This standard consists of nine stable isotope-substituted priority compounds: chloroethane-d5; 2,2-dichloropropane-d6;

PURPOSE: COMPOUND	MS Tuning	GC Perform.	Internal Quan.Std VOA	Internal Quan.Std Extract	Recovery Standard VOA	Recovery Standard Extract	Concent. Recovery Standard	Reference Internal Standard
COMPOUND: (EPA)	$F_{10}Ø_3P$; $F_5BrØ$ (VOA)	Benzidine + Aldrin	$BrClCH_2$	D_{10}-Anthracene	none	Acids: $F_5ØOH$ o-FØOH Neutrals: F_{10}-biphenyl o-Fluoro-Aniline	none	none
COMPOUND: (U of W)	$F_{10}Ø_3P$; $F_5BrØ$	Priority Pollutants	D_6Cl_2-propane D_8toluene Bromoform-D_1	D_8Naphthalene D_{10}Phenanthrene D_{12}Perylene	D_5Chloroethane D_1Chloroform D_6Benzene D_3Acetonitrile D_4Dichlorobenzene	EPA COMPOUNDS+ others as obtained	D_4Dichlorobenzene	Hexamethylbenzene

Table 9-1 Standard Compounds for QA/QC

chloroform-d; benzene-d6; toluene-d8; acrylonitrile d3; bromoform-d; and dichlorobenzene-d4. (The underlined compounds are GC quantitation standards; the rest are included for recovery data and to cross check the quantitation standards).

In the sampling phase for the current project R806102-01, the proposed QA/QC program consists of: a) one field blank per sampling occasion (per plant sampled), b) one QA/QC sample per two plants (approximately 1 in 10 samples), c) recovery compounds to be used in every sample, d) use of recovery compounds to provide replicate verification rather than a second spiked sample (the second spiked sample will be taken and extracted, and the extracts archived), e) an expanded set of standards/recovery standards for VOA as shown in Table 9-1, f) one blank per each VOA analysis, g) replicate VOA analyses for those samples indicated by poor recovery of spiked standards, and h) expansion of recovery standard list and eventual reduction of frequency of spiking to 1 in 20 samples.

SECTION 10

ANALYTICAL RESULTS OF POTW SAMPLES

10-1 Purgable Organics

Of the different analyses, the VOA analysis is less prone to false identifications, and to losses in general. This is because 1) the sample handling is minimal, 2) the purge and trap process is fairly selective, so the complexity of the chromatogram is less than for an injected sample, and 3) the compounds themselves are generally more stable. An exception to the last could possibly be the alkenes (vinyl chloride, acrolein, and acrylonitrile) if the pH of the sample is extreme.

The analysis conducted at the University of Washington was somewhat altered from the standard Bellar/Lichtenberg procedure. As has been reported, the inclusion of additional trapping media along with Tenax has been unsatisfactory. Chromosorb 102 produced a substantial alkylated aromatic background when heated even modestly (not above 150°C in the University of Washington experience, and even up to 100°C as reported by GIT). Although this does not pose a problem for analysis of priority purgables, it does interfere with priority aromatics, which are detected in both the VOA and base/neutral extract. Silica gel increases the detained water from purging to the point where analysis by capillary GC is impossible. Therefore, the data from the Seattle, Atlanta, and Oakland plants was obtained using Tenax GC alone as the trapping medium. The most volatile organics, under the experimental conditions used are very inefficiently trapped, and 2 - 5% of the most volatile recovery standard, deuterated chloroethane is usually recovered. This data will therefore not accurately reflect the presence of dissolved gases. Beginning with dichloropropane, the trapping efficiency of Tenax GC, combined with the improved sensitivity of the of the capillary analysis, gives consistantly good results. A recent improvement developed at the University of Washington will substantially increase the sensitivity for the most volatile compounds, giving at least 50% recovery of vinyl chloride by slightly chilling the Tenax GC alone by cold air. Initial tests with closed-loop stripping shows that late eluting compounds are better recovered.

The data analysis for VOA is performed by dividing the priority purgables into three groups, according to retention time on SE-54. Three of the nine deuterated standards are selected to be internal standards (used to compute expected retention times for the analyte compounds based on relative retention of the compound vs standard in calibration analyses). The three standards normally selected are: d4-2,2-dichloropropane (early eluting), d8-toluene, and dl-bromoform (late eluting). Because the standards are purged and trapped along with the sample, they too are subject to matrix effects and efficiencies of purging and trap-

ping. Thus they are recovered with varying efficiency. A single analyte quantitated against two internal standards will vary by 5 - 10% ordinarily, and by more if one or the other standard is recovered more or less than usual. As the table shows, variability is greatest for extremely low boiling compounds (d5-chloroethane) and extremely late eluting compounds (di-bromoform and d4-dichlorobenzene). Presumably, the former trap poorly while the latter purge poorly (and chromatograph poorly). All quantitation values are therefore connected to reflect a single quantitation standard (2,2-dichloropropane). The full data, as reported in the Appendix, reflects the reproducibility of this analysis. The increased MS scan rate means that ΔT is reported in increments of 0.5 seconds. Typical RIC traces for both raw sewage and digested sludge are shown in Figures 2-12 and 10-1.

10-2 Neutral Extractables

The particular problems of extractable analysis are of several types: 1) chemical loss or degradation of the particular compounds in the environment of a particular sample, 2) losses in handling due to chromatographic adsorption, evaporation, and handling errors; 3) contamination; 4) chemical reactions associated with the analysis (upon acidification, in the GC injection port, or in the instrument); and 5) errors in data processing. Of these, the majority must be considered nonsystematic and therefore not addressed by standard analyses, spiked analyses, etc. Large numbers of analyses will indicate the statistical frequency of these problems, but do not allow one to detect them in an individual sample. Therefore, the first line of defense in discovering artifacts is careful scrutiny of the data. An example of 1) is the chemical attenuation of d10-anthracene. When this compound is used as an internal (quantitation) standard in some samples, the quantities of compounds found appear to rise as the sample ages. When other reference compounds are also present (hexamethyl benzene, for example) the d10-anthracene is seen to diminish relative to them over time. This can also be observed in samples that contain phenanthrene, but almost no anthracene itself. Since these compounds arise from the same sources and are usually found together, the absence of anthracene is probably a sign that it is reacting. One further example is the dinitrotoluenes. When recovery of these compounds from spiked samples dropped off, the MS response factors for the last several months were examined and found to decrease steadily, unlike other compounds in the calibration standard. If the compounds undergo reaction in a sample mixture in organic solvent at -10° C over time, then by implication the same process can occur in environmental samples.

Examples of handling losses will be found in the QA/QC data for Atlanta. Losses occurring prior to extraction will only be indicated by unusual appearance in the chromatogram (and only in extreme cases). Adsorption losses will discriminate against the

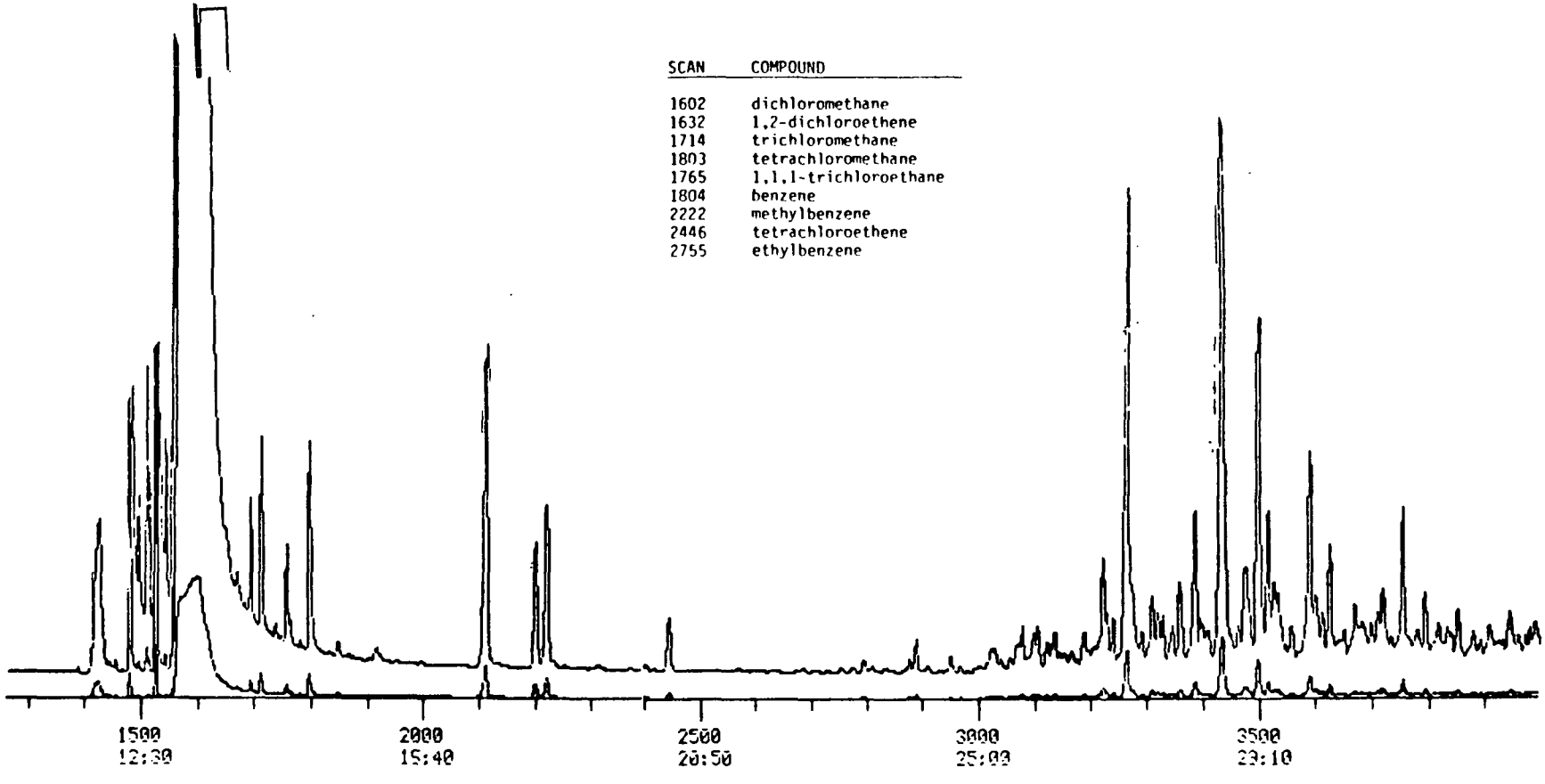


Figure 10-1 RIC Trace of Purgables in Seattle-Renton Primary and Waste-Activated Sludge

high retention time peaks; evaporative losses will show attenuation of early eluting peaks relative to later eluting ones. At the time of extraction, surrogates and stable isotope marker compounds are introduced into the sample; recoveries of these and later standards (as indicated in the preceding QA/QC section) also indicate losses in handling. For the first three plants, these standards were not yet available.

Contamination is a continuing problem. Blank analyses and regular analyses indicate intermittent phthalate contamination and occasionally dichlorobenzene contamination. Blanks have been run on all chromatography materials, solvents, glass wool, glassware, etc., and chromatographic materials as well as drying agent are soxhlet extracted prior to use. Intermittent contamination still occurs and is recognizable primarily by 1) observable quantities of phthalates and 2) the relative levels of the different phthalates present. System blanks run with each batch of samples also can identify some contamination problems.

Data processing errors are infrequent for most compounds and are ultimately recoverable when they do occur, provided that the problem is identified. In terms of the data from the first three POTW sites, data analysis errors are primarily confined to 1) false positives for haloethers and dimethyl nitrosamine and 2) misidentification of isomeric pairs of PAHs. Familiarity with retention behavior of the neutral organics helps to correct the latter problem. Typical RIC traces of the neutral fraction of raw sewage and digested sludge are shown in Figure 10-2 and Figure 10-3.

10-3 Pesticides

The pesticides have been quantitated by two separate analyses: GC/MS and GC/EC. However, the response for these compounds by EI-MS is such that the electron capture analysis was the only one to find these compounds, except in the cases of spiked samples. The quantitation by EC was performed using absolute instrument response, although future analyses will employ decafluorobiphenyl as an internal standard. The significant difficulties with this analysis are those associated with any GC method: qualitative identification and proper integration. The data reported in the appendix for these compounds, therefore, includes retention time differences between standard and sample (within 2 seconds) and also indicates unresolved peaks or other integration difficulties. Typical traces for pesticides in raw sewage and digested sludge are shown in Figures 10-4 and 10-5.

10-4 Phenolics

The acidic priority compounds are the most poorly handled in the initial set of analyses. The possible difficulties here were: 1) poor extraction (matrix effects), 2) variable efficacy

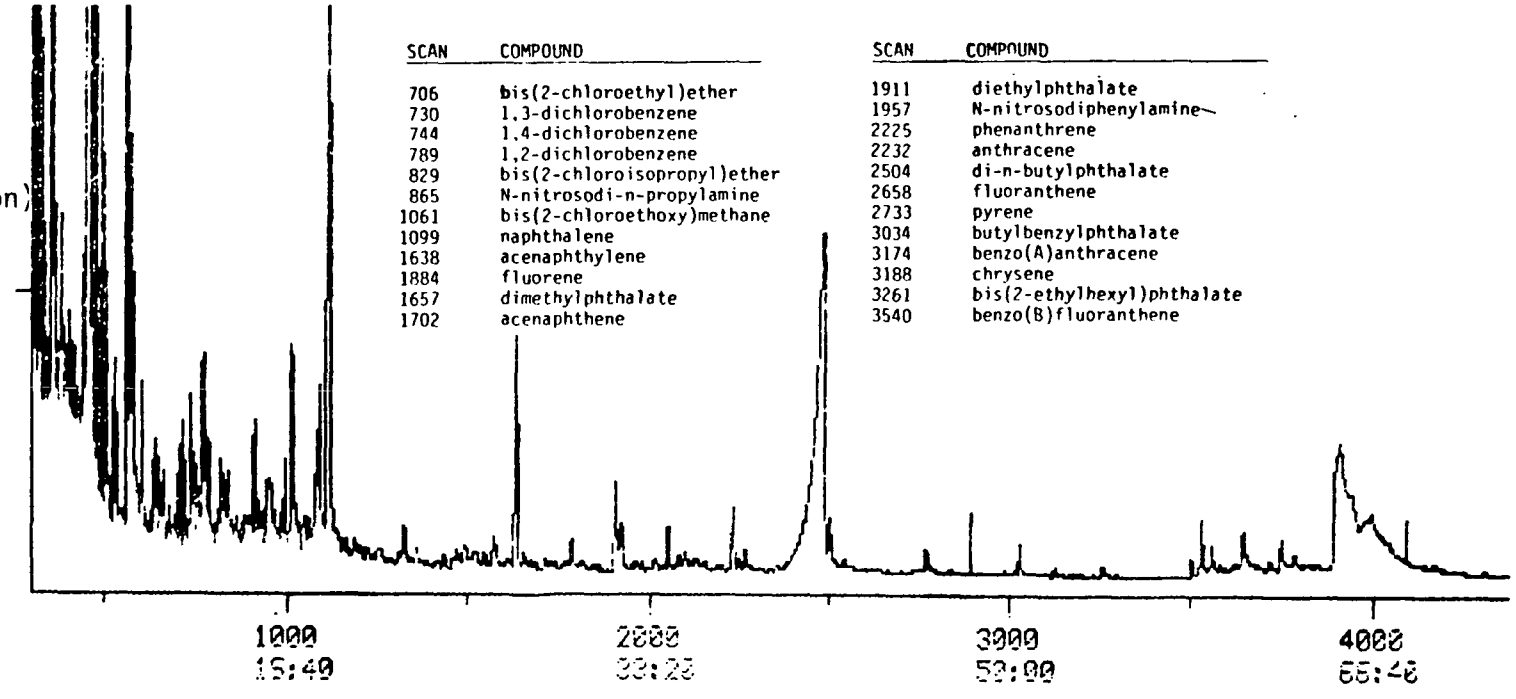


Figure 10-2
RIC Trace of
Seattle(Renton)
Raw Sewage

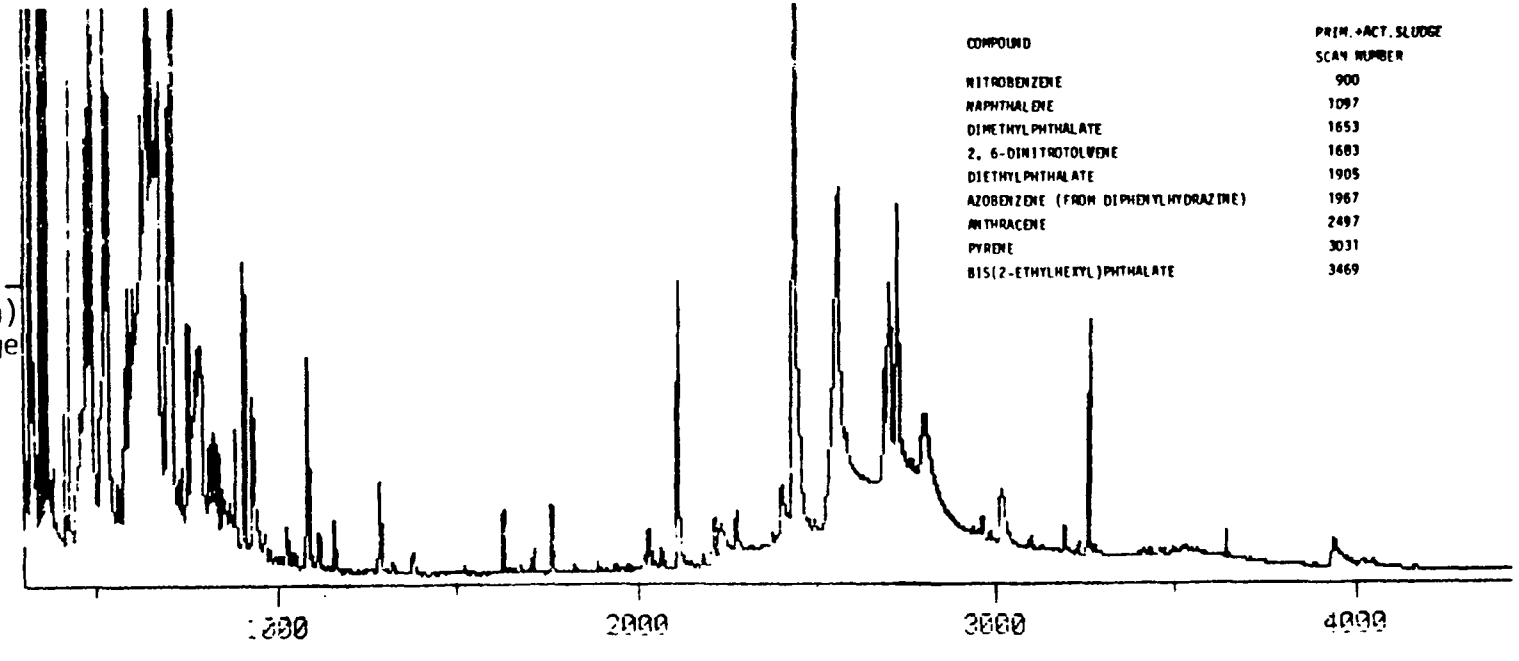
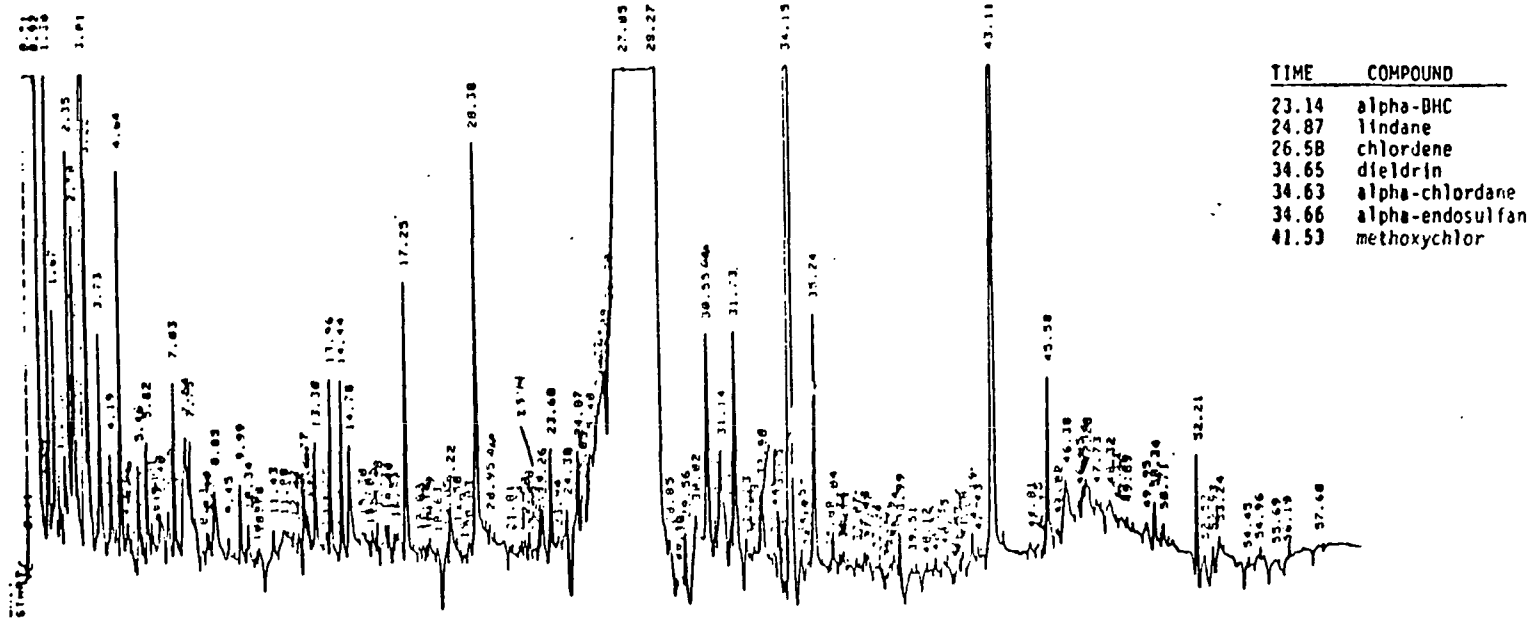
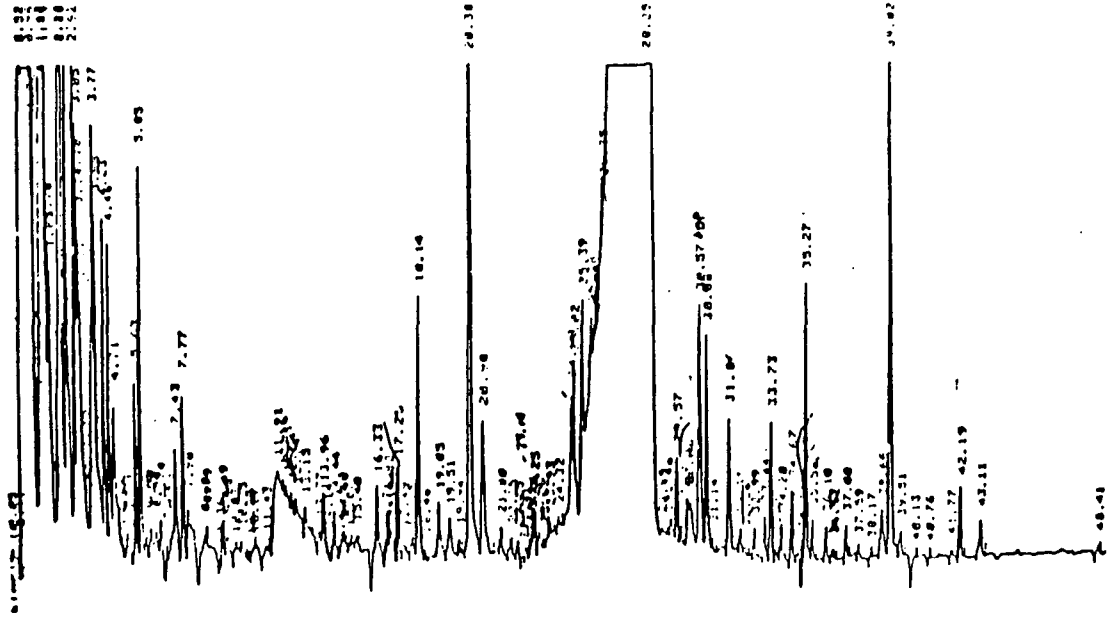


Figure 10-3
RIC Trace of
Seattle(Renton)
Combined Sludge



TIME	COMPOUND
23.14	alpha-BHC
24.87	lindane
26.58	chlordene
34.65	dieldrin
34.63	alpha-chlordane
34.66	alpha-endosulfan
41.53	methoxychlor

Figure 10-5 GC Trace of Pesticide Fraction, Renton (Seattle) Sludge



TIME	COMPOUND	TIME	COMPOUND
23.14	alpha-BHC	33.73	alpha-chlordane
24.88	lindane	34.67	dieldrin
25.02	beta-BHC	35.14	p,p' DDE
28.25	heptachlor	35.58	endrin
31.86	heptachlorepoxide	37.08	p,p' DDD
32.99	gamma-chlordane	38.66	p,p' DDT
33.44	alpha-endosulfan	42.19	mirex

Figure 10-4 GC Trace of Pesticide Fraction, Renton (Seattle) Raw Sewage

of derivatization, 3) poor GC behavior, and possibly 4) chemical instability. Application of advanced columns materials available after October 1, 1979 have revealed that poor gas chromatography and chemical instability were the principal causes. Figures 10-6 and 10-7 present analysis of Renton raw sewage analyzed by SE-54 glass and fused-silica SP 2100, respectively. The data reported for recovery in Table 10-7 (Section 10-5) are presented as SE-54 values in parentheses with the later SP 2100 values following. Because of the recent availability of these new GC columns, approximately 4 months elapsed between derivatization and final analysis by SP 2100, during which time the samples were stored at -10 degrees centigrade. The probability that chemical instability is responsible for the uniform but low recovery is indicated by the fact that three subsequent QA/QC studies on raw sewage (spiked at 20 ppb) and one on activated sludge (spiked at 200 ppb) showed recoveries for the priority phenols of 91.8%, 88.5%, 91.4%, and 81.6%, respectively, with a relative standard deviation of no more than 25%. The elapsed time between derivatization and analysis for these samples did not exceed 3 weeks.

The results reported in Table 11-11, 11-12, 11-13, and pages 338-352 in Appendix 2 are based on data from earlier techniques (SE-54). These samples hve been re-analyzed and the improved results will be reported in a subsequent quarterly research report.

10-5 QA/QC Data

Tables 10-1 to 10-3 list individual QA/QC studies for base/neutral recovery studies for each of the first three POTW sites. The recovery data for these and three other samples (two sediments and one tissue) are summarized in Tables 10-4 and 10-5. The per compound results are presented in Table 10-6. Among these, the Atlanta sample clearly suggests evaporative losses due to overheating, while the tissue sample shows high molecular weight discrimination, probably due to matrix effects in extraction and/or to adsorprive losses. The variability on a per sample and on a per compound basis is higher than desirable (an average relative standard error of 59% per sample and 43% per compound, computed without including zero recovery values). The initiation of the recovery monitoring program discussed earlier should help to substantially reduce this variability.

The recovery samples for pesticides are presented in Tables 10-6 to 10-10 and are summarized in Table 10-11. The values are rather consistent with the base/neutral recovery data but a bit better (probably due to the selectivity of EC for these compounds). The average relative standard deviation was 38%. Those compounds left blank are analyzed but not presently quantitated; they will be added retroactively.

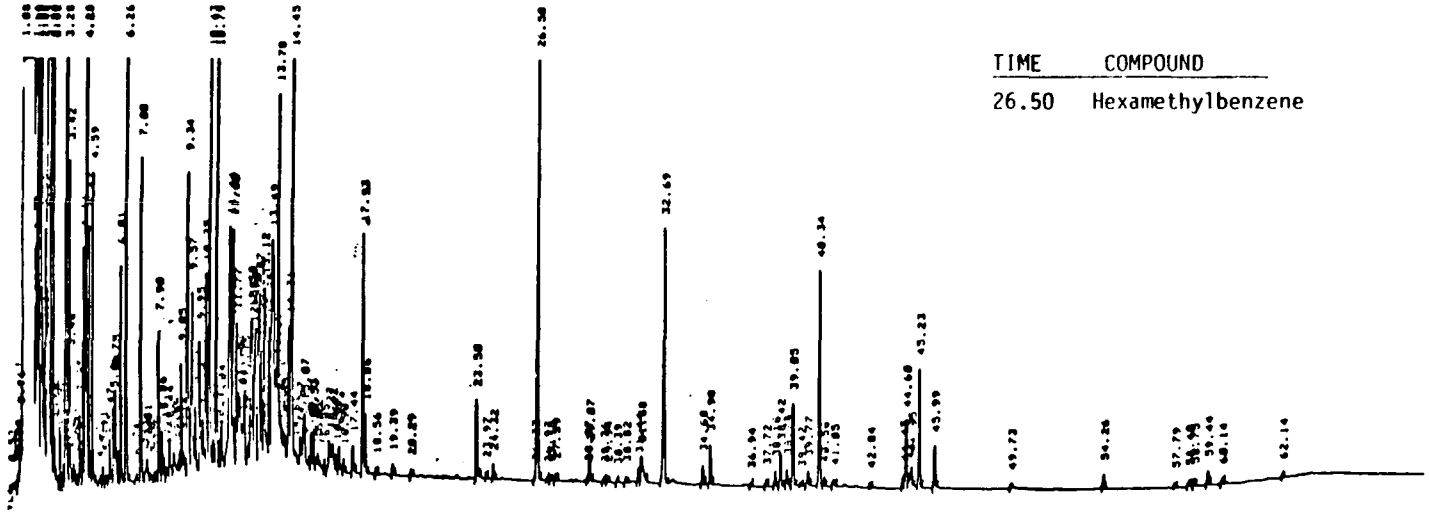


Figure 10-6 Phenolics in Renton-Seattle Raw Sewage.

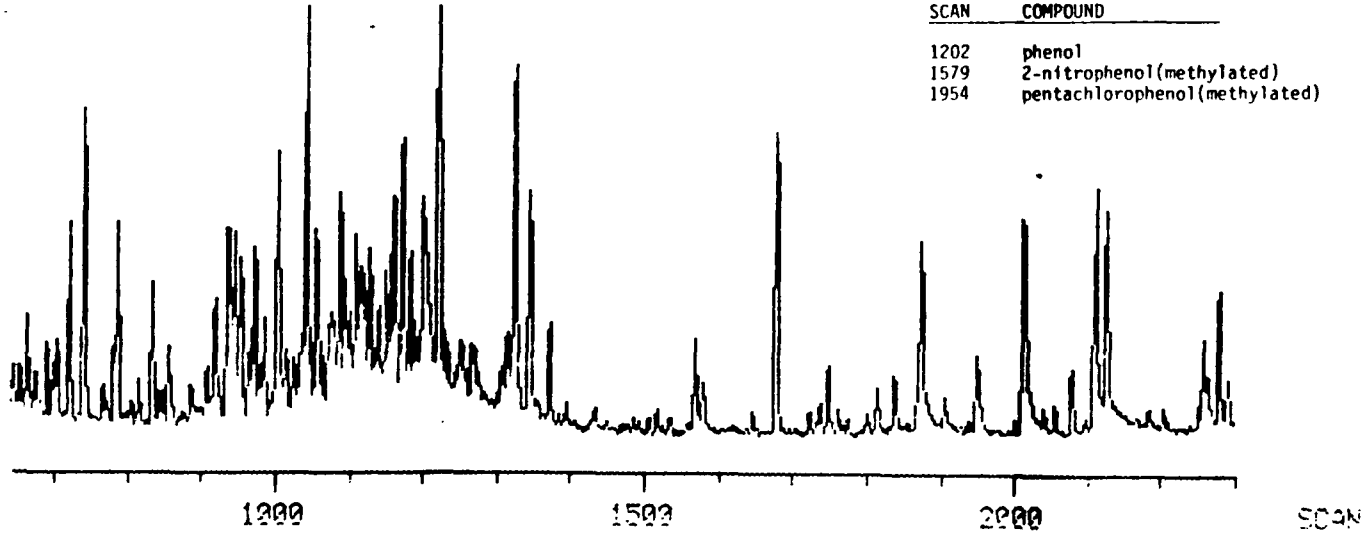


Figure 10-7 Phenolics in Renton-Seattle Raw Sewage, Reanalyzed With Fused-Silica GC/MS

QA/QC RENTON

Table 10-1 BASE & NEUTRAL EXTRACTABLES

QA/QC
ANALYSIS REPORT
Sample: Renton-Seattle Raw Sewage
Lab:
Sample Size: spiked at 10 ppb
Date Sampled:

Quantitation Standard:
Quantitation Method:

COMPOUND	BLANK	UNSPIKED	SPIKED	SPIKED	STD	% REC E1	% REC E2
N-NITROSODIMETHYLAMINE		---	---	---	---		
BIS(2-CHLOROETHYL)ETHER		0.069	6.91	7.09	8.16	84	86
(1,3-)DICHLOROBENZENE		0.557	15.38	13.35	13.74	108	93
1,4-DICHLOROBENZENE		3.66	19.12	16.77	12.18	127	108
(1,2-)DICELOROBENZENE		4.83	14.83	13.21	7.71	130	109
BIS(2-CHLOROISOPROPYL)ETHER		0.321	---	---	---	--	--
HEXACHLOROETHANE		---	8.96	8.88	8.36	107	106
N-NITROSO-DI-N-PROPYL AMINE		---	6.33	7.46	9.96	64	75
NITROBENZENE		0.046	20.44	21.62	19.04	107	113
BIS(2-CHLOROETHYOXY)METHANE		0.126	---	0.24	---	--	--
1,2,4-TRICELOROBENZENE		0.207	10.49	10.35	9.45	109	107
NAPHTHALENE		1.180	13.36	13.65	12.04	101	104
HEXACHLOROBUTADIENE		---	---	---	---	--	--
2-CHLORO-NAPHTHALENE		---	10.90	9.08	12.94	84	70
ACENAPHTHYLENE		0.003	12.97	13.62	19.08	68	71
DIMETHYLPHTHALATE		0.515	6.57	6.15	5.68	108	100
2,6-DINITROTOLUENE		0.182	9.58	14.34	11.06	85	128
ACENAPHTHENE		0.063	15.60	18.37	20.80	75	88
2,4-DINITROTOLUENE		---	---	13.76	11.07	0	124
FLUORENE		---	---	10.75	11.61	0	93
DIETHYLPHTHALATE		7.44	13.71	14.30	12.93	48	53
AZOBENZENE (FROM DIPHENYLHYDRAZINE)		---	4.04	7.07	22.98	18	31
N-NITROSODIPHENYLAMINE		---	---	---	---	--	--
4-BROMODIPHENYL ETHER		---	4.80	6.32	7.16	67	88
HEXACHLOROBENZENE		---	13.18	14.66	14.77	89	99
PHENANTHRENE		0.14	6.98	8.97	13.05	52	68
ANTHRACENE		0.01	4.36	4.87	11.47	38	42
DI-N-BUTYLPHTHALATE	1.53	3.01	7.69	9.38	9.84	48	65
FLUORANTHENE		0.02	6.45	9.58	11.604	55	82
PYRENE		0.01	8.99	13.42	13.44	67	100
BUTYLBENZYLPHTHALATE		2.59	---	10.40	8.30	NQ	94
BENZO(A)ANTHRACENE		---	11.11	9.24	6.33	176	146
CHRYSENE		---	---	7.99	2.78	NQ	287
3,3'-DICHLOROBENZIDENE			---	---	---	--	--
BIS(2-ETHYLHEXYL)PHTHALATE			---	36.93	11.33	--	376
DI-N-OCTYL PHTHALATE			0.62	12.99	10.21	65	127
BENZO(B)FLUORANTHENE			2.37	3.23	1.355	175	238
BENZO(K)FLUORANTHENE			---	2.39	1.25	NQ	191
BENZO(A)PYRENE			---	---	2.40	0	0
INDENO(1,2,3-CD)PYRENE			---	---	---	--	--
DIBENZO(A,H)ANTHRACENE			---	8.73	2.26	--	386
BENZO(GHI)PERYLENE			---	7.38	1.15	--	642
CHLOROPHENYL, PHENYL ETHER							
HEXACHLOROCYCLOPENTADIENE							

blank=not found; NQ=detected but not quantitated; NA=not analyzed for..

Table 10-2QA/QC ATLANTA

BASE & NEUTRAL EXTRACTABLES

QA/QC
ANALYSIS REPORT
Sample: Clayton-Atlanta Raw Sewage
spiked at 10 ppb
Lab:
Sample Size:
Date Sampled:

Quantitation Standard:
Quantitation Method:

COMPOUND	BLANK	UNSPIKED	SPIKED	EXPECTED	% REC
N-NITROSODIMETHYLAMINE		--	--	--	
BIS(2-CHLOROETHYL)ETHER	(0.1)	--	4.03	7.06	57
(1,3-)DICHLOROBENZENE		3.149	3.63	16.37	(3)
1,4-DICHLOROBENZENE		13.939	10.21	14.89	(<0)
(1,2-)DICHLOROBENZENE	(.05)	43.858	44.23	8.50	(4)
BIS(2-CHLOROISOPROPYL)ETHER		--	.71	--	
HEXACHLOROETHANE				12.46	(0)
N-NITROSO-DI-N-PROPYL AMINE		--	3.61	6.67	(34)
NITROBENZENE		0.120	6.76	21.11	(31)
BIS(2-CHLOROETHOXY)METHANE		--	.12	--	--
1,2,4-TRICHLOROBENZENE		.613	2.64	10.53	(19)
NAPHTHALENE	(.16)	10.941	5.21	12.28	(<0)
HEXACHLOROBUTADIENE		--	--	--	--
2-CHLORO-NAPHTHALENE	(.02)	--	7.45	14.45	52
ACENAPHTHYLENE		.252	9.47	21.36	43
DIMETHYLPHTHALATE	(.05)	--	4.83	6.13	79
2,6-DINITROTOLUENE		--	--	14.66	--
ACENAPHTHENE	(.04)	.884	13.48	21.60	58
2,4-DINITROTOLUENE		--	--	14.93	--
FLUORENE		1.720	11.18	11.24	84
DIETHYLPHTHALATE	(.55)	1.36	12.87	11.25	102
AZOBENZENE (FROM DIPHENYLHYDRAZINE)	(.01)	--	7.05	16.03	44
N-NITROSODIPHENYLAMINE		--	--	--	--
4-BROMODIPHENYL ETHER		--	3.73	7.75	48
HEXACHLOROBENZENE		--	7.21	19.44	37
PHENANTHRENE	(.33)	3.24	16.41	13.49	98
ANTHRACENE		2.92	4.73	NQ	--
DI-N-BUTYLPHTHALATE	3.77	12.91	31.88	--	--
FLUORANTHENE	(.07)	0.06	7.91	13.66	57
PYRENE	(.11)	0.13	10.74	18.73	57
BUTYBENZYLPHTHALATE	(.95)	17.88	44.42	8.45	314
BENZO(A)ANTHRACENE		--	5.71	12.85	44
CHRYSENE		--	5.97	10.48	57
3,3'-DICHLOROBENZIDENE		--	--	--	--
BIS(2-ETHYLHEXYL)PHTHALATE		--	41.71	15.75	265
DI-N-OCTYL PHTHALATE		--	10.28	19.43	53
BENZO(B)FLUORANTHENE		--	1.53	3.53	43
BENZO(K)FLUORANTHENE		--	1.13	2.13	53
BENZO(A)PYRENE		--	--	6.67	--
INDENO(1,2,3-CD)PYRENE		--	0.09	--	--
DIBENZO(A,H)ANTHRACENE		--	2.57	6.94	37
BENZO(GHI)PERYLENE		--	2.86	6.61	43
CHLOROPHENYL, PHENYL ETHER					
HEXACHLOROCYCLOPENTADIENE					

blank=not found; NQ=detected but not quantitated; NA=not analyzed for.

Table 10-3 QA/QC OAKLAND

BASE & NEUTRAL EXTRACTABLES

QA/QC
ANALYSIS REPORT
Sample: EBMUD Oakland Second. Effluent
Lab: spiked at 10 ppb
Sample Size:
Date Sampled:

Quantitation Standard:
Quantitation Method:

COMPOUND	BLANK	UNSPIKED	SPIKED	EXPECTED	% REC
N-NITROSODIMETHYLAMINE	---	0.40*	---	---	
BIS(2-CHLOROETHYL)ETHER	0.006	---	5.78	8.16	71
(1,3-)DICHLOROBENZENE	0.003	0.41	6.13	13.74	42
1,4-DICHLOROBENZENE	0.005	2.75	7.74	12.18	41
(1,2-)DICHLOROBENZENE		1.34	5.28	7.71	51
BIS(2-CHLOROISOPROPYL)ETHER		0.27	---	---	---
HEXACHLOROETHANE		---	2.86	8.36	34
N-NITROSO-DI-N-PROPYL AMINE		0.10	5.87	9.96	58
NITROBENZENE		---	15.41	19.04	81
BIS(2-CELOROETHOXY)METHANE		---	---	---	---
1,2,4-TRICHLOROBENZENE		0.32	5.33	9.45	53
NAPHTHALENE		---	8.38	12.04	70
HEXACHLOROBUTADIENE		---	---	---	---
2-CHLORO-NAPHTHALENE		---	9.16	12.94	71
ACENAPHTHYLENE		---	11.77	19.08	62
DIMETHYLPHTHALATE		---	3.06	5.63	54
2,6-DINITROTOLUENE		---	8.58	11.06	78
ACENAPHTHENE		---	14.29	20.80	69
2,4-DINITROTOLUENE		---	4.51	11.07	41
FLUORENE		---	8.27	11.61	71
DIETHYLPHTHALATE			3.51	12.93	27
AZOBENZENE (FROM DIPHENYLHYDRAZINE)			7.93	22.98	35
N-NITROSODIPHENYLAMINE			---	---	---
4-BROMODIPHENYL ETHER			4.37	7.16	61
HEXACHLOROBENZENE			8.19	14.77	55
PHENANTHRENE			5.72	13.05	44
ANTHRACENE			3.74	11.47	33
DI-N-BUTYLPHTHALATE	6.24	5.99	4.71	9.84	*
FLUORANTHENE			6.53	11.604	56
PYRENE			8.94	13.44	67
BUTYBENZYLPHTHALATE			0.61	8.30	07
BENZO(A)ANTHRACENE			4.49	6.33	71
CHRYSENE			4.29	2.78	154
3,3'-DICHLOROBENZIDENE			---	---	
BIS(2-ETHYLHEXYL)PHTHALATE			7.09	11.33	63
DI-N-OCTYL PHTHALATE			4.17	10.21	41
BENZO(B)FLUORANTHENE			1.11	1.355	82
BENZO(K)FLUORANTHENE			1.13	1.25	90
BENZO(A)PYRENE			---	2.40	---
INDENO(1,2,3-CD)PYRENE			0.11	---	---
DIBENZO(A,H)ANTHRACENE			2.28	2.26	101
BENZO(GHI)PERYLENE			2.26	1.15	197
CHLOROPHENYL, PHENYL ETHER					
HEXACHLOROCYCLOPENTADIENE					

blank=not found; NQ=detected but not quantitated; NA=not analyzed for.
*probable artifact.

	OTHER:			POTW:			
	SED 1	SED 2	TISSUE	RENTON 1	RENTON 2	ATLANTA	OAKLAND
D8-NAPHTHALENE D4-1,4-DICHLOROBENZENE HEXAMETHYLBENZENE D10-ANTHRACENE	STDS						
N-NITROSODIMETHYLAMINE	--	--	--	--	--	--	--
BIS(2-CHLOROETHYL)ETHER	81	99	84	84	86	57	71
(1,3-)DICHLOROBENZENE	60	34	68	108	93	3	42
1,4-DICHLOROBENZENE	67	46	77	127	108	<0	41
(1,2-)DICHLOROBENZENE	79	71	77	130	109	4	51
BIS(2-CHLOROISOPROPYL)ETHER	--	--	--	--	--	--	--
HEXACHLOROETHANE	48	7	43	107	106	0	34
N-NITROSO-DI-N-PROPYL AMINE	74	122	108	64	75	54	58
NITROBENZENE	85	93	72	107	113	31	81
BIS(2-CHLOROETHOXY)METHANE	--	--	--	--	--	--	--
1,2,4-TRICHLOROBENZENE	71	54	81	109	107	19	53
NAPHTHALENE	162	107	84	101	104	0	70
HEXACHLOROBUTADIENE	--	--	--	--	--	--	--
2-CHLORO-NAPHTHALENE	81	94	68	84	70	52	71
ACENAPHTHYLENE	25	0	73	68	71	43	62
DIMETHYLPHTHALATE	82	96	77	108	100	79	54
2,6-DINITROTOLUENE	162	84	52	85	128	--	78
ACENAPHTHENE	57	102	79	75	88	58	69
2,4-DINITROTOLUENE	67	70	49	0	124	--	41
FLUORENE	71	103	82	0	93	84	71
	79.5 ± 35.6	73.9 ± 36.3	73.4 ± 15.6	84.8 ± 38.2	98.4 ± 17.4	37.23 ± 29.9	59.2±14.6

Table 10-4 Summary of Neutral QA/QC Date

Table 10-5 Summary of Neutral QA/QC Data.

	OTHER:			POTW:			
	SED 1	SED 2	TISSUE	RENTON 1	RENTON 2	ATLANTA	OAKLAND
D10-ANTHRACENE D4-1,4-DICHLOROBENZENE D8-NAPHTHALENE HEXAMETHYLBENZENE	STDS						
ACENAPHTHENE	57	79	40	54	64	58	50
2,4-DINITROTOLUENE	67	68	35	47	90	0	29
FLUORENE	71	107	60	54	68	84	52
DIETHYLPHTHALATE	81	103	66	48	53	102	27
AZOBENZENE(FR.DIPHENYLHYDRAZINE)	45	95	48	18	31	44	35
N-NITROSODIPHENYLAMINE	--	--	--	--	--	--	--
4-BROMODIPHENYL ETHER	71	116	67	67	88	88	61
HEXACHLOROBENZENE	72	99	66	89	99	37	55
PHENANTHRENE	77	105	60	52	68	98	44
ANTHRACENE	--	--	--	38	42	--	33
DI-N-BUTYLPHTHALATE	54	--	--	48	65	--	contain *
FLUORANTHENE	73	114	73	55	82	57	56
PYRENE	60	114	75	67	100	57	67
BUTYLBENZYLPHTHALATE	110	53	<0	*NQ	94	314	07
BENZO(A)ANTHRACENE	21	167	89	176	146	44	71
CHRYSENE	--	164	91	NQ	287	57	154
3,3'-DICHLOROBENZIDENE	--	--	--	--	--	--	--
BIS(2-ETHYLHEXYL)PHTHALATE	contain *	195	0	0	326	265	63
DI-N-OCTYL PHTHALATE	contain *	173	0	65	127	53	41
BENZO(B)FLUORANTHENE	160	275	0	175	238	43	82
BENZO(K)FLUORANTHENE	--	291	0	NQ	191	53	90
BENZO(A)PYRENE	197	211	97	0	0	0	0
INDENO(1,2,3-CD)PYRENE	--	--	--	--	--	--	--
DIBENZO(A,H)ANTHRACENE	73	200	0	0	(386)	37	101
BENZO(GHI)PERYLENE	398	155	10	0	(642)	43	197
$\bar{x} \pm \sigma$	99 ± 86	144.2 ± 65.2	46.2 ± 34.7	55 ± 49.7	113 ± 85	75 ± 78	63 ± 45

BASE + NEUTRAL EXTRACTABLES

Table 10-6 QA/QC SUMMARY

COMPOUND	% Recovery	Detection Rate
N-NITROSODIMETHYLAMINE	Not Spiked	
BIS(2-CHLOROETHYL)ETHER	80.4 ± 13	7 of 7
(1,3-)DICHLOROBENZENE	58.3 ± 35.8	7 of 7
1,4-DICHLOROBENZENE	77.7 ± 34.1	6 of 7
(1,2-)DICHLOROBENZENE	74.4 ± 44.3	7 of 7
BIS(2-CHLOROISOPROPYL)ETHER	Not Spiked	
HEXACHLOROETHANE	57.5 ± 40.5	6 of 7
N-NITROSO-DI-N-PROPYL AMINE	79.3 ± 25.9	7 of 7
NITROBENZENE	83.1 ± 27.1	7 of 7
BIS(2-CHLOROETHOXY)METHANE	Not Spiked	
1,2,4-TRICHLOROBENZENE	70.6 ± 32.0	7 of 7
NAPHTHALENE	104.7 ± 31.4	6 of 7
HEXACHLOROBUTADIENE	Not Spiked	7
2-CHLORO-NAPHTHALANE	74.3 ± 13.5	7 of 7
ACENAPHTHYLENE	57.0 ± 19.1	6 of 7
DIMETHYLPHTHALATE	85.1 ± 18.0	7 of 7
2,6-DINITROTOLUENE	80.7 ± 27.0	6 of 7
ACENAPHTHENE	75.4 ± 16.1	7 of 7
2,4-DINITROTOLUENE	70.2 ± 32.4	5 of 7
FLUORENE	84.0 ± 12.5	6 of 7
DIETHYLPHTHALATE	68.6 ± 28.5	7 of 7
AZOBENZENE (FROM DIPHENYLHYDRAZINE)	45.1 ± 24.3	7 of 7
N-NITROSODIPHENYLAMINE	Not Spiked	7
4-BROMODIPHENYL ETHER	79.7 ± 19.2	7 of 7
HEXACHLOROBENZENE	73±9 ± 23.4	7 of 7
PHENANTHRENE	72.0 ± 22.9	7 of 7
ANTHRACENE	38.3 ± 5.5	3 of 7
DI-N-BUTYLPHTHALATE	55.7 ± 8.6	3 of 7
FLUORANTHENE	72.9 ± 20.9	7 of 7
PYRENE	77.1 ± 21.6	7 of 7
BUTYLBENZYLPHTHALATE	115.6 ± 117.9	5 of 7
BENZO(A)ANTHRACENE	102 ± 61.5	7 of 7
CHRYSENE	150.6 ± 88.2	5 of 7
3,3'-DICHLOROBENZIDENE	Not Spiked	
BIS(2-ETHYLHEXYL)PHTHALATE	212.3 ± 113.0	4 of 7
DI-N-OCTYL PHTHALATE	91.8 ± 56.2	5 of 7
BENZO(B)FLUORANTHENE	153.8 ± 87.1	6 of 7
BENZO(K)FLUORANTHENE	156.3 ± 107.1	4 of 7
BENZO(A)PYRENE	168.3 ± 62.2	3 of 7
INDENO(1,2,3-CD)	Not Spiked	
DIBENZO(A,H)ANTHRACENE	102.8 ± 69.9	4 of 7
BENZO(GHI)PERYLENE	160.6 ± 153.5	5 of 7
CHLOROPHENYL,PHENYL ETHER	NA	
HEXACHLOROCYCLOPENTADIENE	NA	5 of 7

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=probable artifact

QA/QC
ANALYSIS REPORT
Sample:

Lab:
Sample Size:
Date Sampled:

Quantitation Standard:
Quantitation Method:

Table 10-7
ACID-EXTRACTABLES (PHENOLS)

COMPOUND	Atlanta % Recovery (SE-54)	Atlanta % Recovery SP2100	Oakland % Recovery (SE-54)	Oakland % Recovery SP2100
PHENOL (M)				
2-CHLOROPHENOL	(0)	14.5	(0)	13.7
PHENOL	(179)	76.5	(0)	32.4
2-CHLOROPHENOL (M)				
2,4-DIMETHYLPHENOL	(129)	12.5	(0)	2.9
4-CHLORO-3-METHYLPHENOL (M)		27.2		26.0
2,4-DICHLOROPHENOL	(68)	25.4	(0)	31.06
4-CHLORO-3-METHYLPHENOL	(0)	NA	(0)	NA
2,4,6-TRICHLOROPHENOL (M)	(41)	22.0	(64)	20.2
2-NITROPHENOL (M)	(0)	8.2	(0)	9.9
4-NITROPHENOL (M)	(102)	NA	(29)	NA
4,6-DINITRO-2-METHYLPHENOL (M)	(16)	6.4	(40)	12.1
PENTACHLOROPHENOL (M)	(3188)	104.8	(67)	21.3
2,4-DINITROPHENOL (M)	(45)	NQ	(11)	14.4

blank=not found; NQ=detected,not quantitated; NA=not analyzed for
(M)=derivatized with diazomethane

Table 10-8 WORKSHEET-QA/QC
ANALYSIS REPORT RENTON QA/QC-1 PESTICIDES + PCB's

Sample: PS92E1 Neutrals

Lab: RENTON
Sample Size:
Date Sampled

Quantitation Standard: absolute
Quantitation Method: GC/EC

COMPOUND	BLANK	PS90 SAMPLE	SAMPLE SPIKE	EXPECTED	% RECOV.
α-BHC		0.125	4.45	5.33	81.1
β-BHC + γ-BHC (LINDANE)			19.041	24.69	77.1
δ-BHC			10.985	12.44	88.3
P,P'-DDD			6.614	12.75	51.9
P,P'-DDE			3.701	6.75	54.8
P,P'-DDT			11.086	19.53	56.8
DIELDRIN			4.607	9.00	51.2
ENDRIN			8.143	14.07	57.9
ENDRINALDEHYDE					
α-ENDOSULFAN			12.29	7.43	165.4
β-ENDOSULFAN			12.16	7.477	162.6
ENDOSULFANE SULFATE					
HEPTACHLOR			NQ	12.08	
HEPTACHLOR EPOXIDE		.1757	8.61	16.60	50.8
PCB 1242	NA				
PCB 1254					
PCB 1221					
PCB 1232					
PCB 1248					
PCB 1260					
PCB 1016					
ALDRIN			2.566	5.81	44.2
METHOXYCHLOR					
MIREX			8.99	17.58	51.1
CHLORDENE			4.683	2.303	203.3
OXYCHLORDANE*					
γ-CHLORDANE			3.61	2.49	135.1
α-CHLORDANE			2.79	2.61	107.1

blank=not found; NQ=detected but not quantitated; NA=not analyzed for; M=merged peak; D=detected but not integrated; O=area obscured; I=improper integration.
*coelutes with heptachlor epoxid

ANALYSIS REPORT RENTON QA/QC (2) Table 10-9 PESTICIDES + PCB's

Sample: PS92E2 Neutrals

Lab:
Sample Size:
Date Sampled

Quantitation Standard: absolute
Quantitation Method: GC/EC

COMPOUND	BLANK	SAMPLE	SPIKE SAMPLE	EXPECTED	% RECOVERY
α-BHC		0.125	5.649	5.33	122.4
β-BHC +					
γ-BHC (LINDANE)			28.091	24.69	113.8
δ-BHC			20.218	12.44	162.5
P,P'-DDD			12.911	12.75	101.3
P,P'-DDE			6.96	6.75	103.1
P,P'-DDT			20.74	19.53	106.2
DIELDRIN			8.45	9.00	93.9
ENDRIN			3.88	14.07	27.6
ENDRINALDEHYDE	NA				
α-ENDOSULFAN			23.01	7.43	309.7
β-ENDOSULFAN			25.56	7.48	341.8
ENDOSULFANE SULFATE					
HEPTACHLOR		0.18	17.42	12.08	142.8
HEPTACHLOR EPOXIDE			16.04	16.60	96.6
PCB 1242	NA				
PCB 1254	NA				
PCB 1221	NA				
PCB 1232	NA				
PCB 1248	NA				
PCB 1260	NA				
PCB 1016	NA				
ALDRIN			4.21	5.81	72.5
METHOXYCHLOR	NA				
MIREX			14.70	17.58	83.6
CHORDENE			8.59	2.30	373.0
OXYCHLORDANE	NA				
γ-CHLORDANE			7.20	2.49	289.5
α-CHLORDANE			5.31	2.61	203.8

blank=not found; NQ=detected but not quantitated; NA=not analyzed for; M=merged peak; D=detected but not integrated; O=area obscured; I=improper integration.

ANALYSIS REPORT ATLANTA QA/QC Table 10-10 WORKSHEET-QA/QC PESTICIDES + PCB's

Sample:

Lab:
Sample Size:
Date Sampled

Quantitation Standard: Absolute
Quantitation Method: GC/EC

COMPOUND	BLANK	SAMPLE	SPIKE SAMPLE	EXPECTED	% RECOV.
α-BHC			2.93	2.67	109.9
β-BHC +					
γ-BHC (LINDANE	0.13	0.14	11.16	12.35	89.2
δ-BHC			5.139	6.22	82.6
P,P'-DDD		0.17	6.80	6.38	103.9
P,P'-DDE			3.15	3.38	92.8
P,P'-DDT	NQ		5.39	9.77	55.2
DIELDRIN	0.07	NQ	4.38	4.50	97.3
ENDRIN	0.19		7.76	7.04	110.3
ENDRINALDEHYDE					
α-ENDOSULFAN	0.03		13.313	7.43	179.2
β-ENDOSULFAN	0.02		8.85	7.48	118.3
ENDOSULFANE SULFATE NA					
HEPTACHLOR	NQ	NQ	4.07	6.04	67.3
HEPTACHLOR EPOXIDE	NQ		8.26	8.30	99.5
PCB 1242 NA					
PCB 1254 NA					
PCB 1221 NA					
PCB 1232 NA					
PCB 1248 NA					
PCB 1260 NA					
PCB 1016 NA					
ALDRIN			1.97	2.91	67.8
METHOXYCHLOR NA					
MIREX	NQ		4.26	8.79	48.5
CHORDENE	0.06		3.25	2.30	141.0
OXYCHLORDANE					
γ-CHLORDANE		0.33	3.82	2.49	153.6
α-CHLORDANE	0.09	0.10	3.55	2.60	135.2

blank=not found; NQ=detected but not quantitated; NA=not analyzed for; M=merged peak; D=detected but not integrated; O=area obscured; I=improper integration.

ANALYSIS REPORT OAKLAND QA/QC Table 10-11 WORKSHEET-QA/QC PESTICIDES + PCB's

Sample:

Lab:
Sample Size:
Date Sampled

Quantitation Standard: Absolute
Quantitation Method: GC/EC

COMPOUND		BLANK	SAMPLE	SAMPLE SPIKE	EXPECTED	% RECOV.
α-BHC		0.13		4.09	2.67	153.3
β-BHC +						
γ-BHC (LINDANE)			0.16	15.54	12.35	124.6
δ-BHC				5.40	6.22	86.8
P,P'-DDD		.16	.15	4.78	6.38	72.6
P,P'-DDE				2.30	3.38	68.1
P,P'-DDT		.40		7.75	9.77	79.3
DIELDRIN		NQ		4.04	4.50	89.8
ENDRIN		0.83		7.28	7.04	103.4
ENDRINALDEHYDE	NA					
α-ENDOSULFAN		0.01	0.04	11.67	7.43	156.5
β-ENDOSULFAN		0.04	0.14	9.20	7.48	121.2
ENDOSULFANE SULFATE						
HEPTACHLOR				4.14	6.04	68.6
HEPTACHLOR EPOXIDE		NQ	.40	7.65	8.3	87.3
PCB 1242	NA					
PCB 1254	NA					
PCB 1221	NA					
PCB 1232	NA					
PCB 1248	NA					
PCB 1260	NA					
PCB 1016	NA					
ALDRIN			NQ	1.635	2.91	56.2
METHOXYCHLOR						
MIREX				4.95	8.79	56.3
CHORDENE			0.06	2.56	2.30	108.5
OXYCHLORDANE						
γ-CHLORDANE			0.221	2.46	2.49	90.1
α-CHLORDANE				2.76	2.61	105.7

blank=not found; NQ=detected but not quantitated; NA=not analyzed for; M=merged peak; D=detected but not integrated; O=area obscured; I=improper integration.

ANALYSIS REPORT QA/QC SUMMARY- FIRST THREE PLANTS

Table 10-12 WORKSHEET QA/QC PESTICIDES + PCB's

Sample:

Lab:
Sample Size:
Date Sampled

Quantitation Standard: Absolute
Quantitation Method: GC/EC

COMPOUND		RENTON 1	RENTON 2	ATLANTA	OAKLAND	$\bar{x}$ (σ)
α-BHC		81.1	122.4	153.3	109.9	116.7(29.9)
β-BHC +						
γ-BHC (LINDANE		77.1	113.8	124.6	89.2	101.2(21.8)
δ-BHC		88.3	162.5	86.8	82.6	105.1(38.4)
P,P'-DDD		51.9	101.3	72.6	103.9	82.4(24.8)
P,P'-DDE		54.8	103.1	68.1	92.8	79.7(22.2)
P,P'-DDT		56.8	106.2	79.3	55.2	74.4(23.9)
DIELDRIN		51.2	93.9	89.8	97.3	83.1(21.5)
ENDRIN		57.9	27.6	103.4	110.3	74.8(39.1)
ENDRINALDEHYDE	NA					
α-ENDOSULFAN		165.4	309.7	156.5	179.2	202.7(71.9)
β-ENDOSULFAN		162.6	341.8	121.2	118.3	185.9(105.8)
ENDOSULFANE SULFATE						
HEPTACHLOR		NQ	142.8	68.6	67.3	69.7(58.3)
HEPTACHLOR EPOXIDE		50.8	96.6	87.3	99.5	83.6(22.4)
PCB 1242	NA					
PCB 1254	NA					
PCB 1221	NA					
PCB 1232	NA					
PCB 1248	NA					
PCB 1260	NA					
PCB 1016	NA					
ALDRIN		44.2	72.5	56.2	67.8	60.18(12.7)
METHOXYCHLOR	NA					
MIREX		51.1	83.6	56.3	48.5	59.9(16.2)
CHORDENE		203.3	373.0	108.5	141.0	206.5(117.8)
OXYCHLORDANE						
γ-CHLORDANE		135.1	289.5	90.1	153.6	167.0(85.9)
α-CHLORDANE		107.1	203.8	105.7	136.2	125.6(11.2)
$\bar{x}$ (σ)		84.6(53.3)	161.4(103.8)	95.8(301.1)	103.1(35.2)	110.5(42.6) overall

The major cause of poor recovery for phenols (Table 10-12) is apparently matrix-related. When spiked water recoveries are run, the results are consistently higher than obtained in wastewater. The prospects for improvement in this analysis are: 1) use of new chromatographic columns which are described as eliminating the need for derivatization, 2) a prompt, direct analysis of phenols alone, probably by HPLC. This latter method is under investigation at GIT, the former is being pursued at the University of Washington.

SECTION 11

SELECTION OF SAMPLING SITES, SAMPLING PROCEDURES, AND POTW REMOVAL EFFICIENCIES

The present study selected the municipal sewage treatment plants in order to obtain a representative picture of the efficiency of the plants to remove priority pollutants from raw sewage.

The amount of priority pollutants in municipal sewage is expected to be influenced by the amount and type of industrial discharge into the network. An initial compilation was therefore made of the nature of the industrial water usage.

11-1 Water Use in Industry

Large industrial users, i.e. those using more than 38 gpm of process and cooling water, utilized a total of 15,024 billion gallons of water per year (U.S. Census, 1972). About half of the water (49%) was subjected to different types of treatment to make it suitable for subsequent use. Most of the water (60%) was taken in through company water treatment systems employing fresh surface water from rivers or estuaries, with 19% taken from salt or brackish water, primarily for cooling purposes. The industries withdrew 11% from public water supplies and 10% from ground water. The most common water treatment was softening, practiced by 59% of the plants, followed by chlorination (36%), and ion exchange (30%). While softening and ion exchange will remove some of the priority pollutants already present, the chlorination step will increase the number and concentration of low molecular weight chlorinated hydrocarbons. Chlorination is most widely used in the food industry, Standard Industrial Code (SIC)-20, and the paper industry (SIC-26) where 52% and 46% of the plants practice it. Most of the intake water is used for direct cooling purposes (36% of intake water) followed by process water (25%), steam-electric (13%), and boiler feed water (4%), as listed in Table 11-1. Industries that use a high percentage of their intake for process water, and therefore produce a more polluted wastewater, are paper (SIC-26), fabricated metals (SIC-34), and lumber (SC-24) with 58%, 48%, and 48% of the intake used for process water.

The water is reused on the average of 2.9 times before being discharged, thus increasing the possibility of pollutant accumulation. A high degree of reuse is practiced by SIC-36 (10 fold), SIC- 37 (8.1 fold), and SIC- 28 (6.4 fold).

Most of the industrial wastewater is discharged (Table 11-2) to streams and rivers (64%) and smaller percentages to bays and estuaries (16%), lakes and ponds (9%), and municipal sewers (7%). The electrical and fabricated metal industries discharge as much

Table 11-1 Percentage Use of Water for Different Purposes

	Fresh Water for Process. Water	Fresh Water for Steam Electric.	Fresh Water for Air Conditioning	Fresh Water for Other Cooling Purposes	Fresh Water for Sanitary Service	Fresh Water for Boiler Feed	Bracklsh Water for Process.	Salt Water for Process.
All industries	25	13	2	36	1	4	1	1
20. Food	29	4	1	33	4	7	--	--
21. Tobacco	35	4	15	20	13	13	--	--
22. Textile	--	--	--	--	--	--	--	--
24. Lumber	48	20	1	11	1	12	--	--
25. Furniture	--	--	--	--	--	--	--	--
26. Paper	58	21	1	12	1	4	1	--
28. Chemicals	9	5	2	50	1	4	1	--
29. Petroleum	4	--	1	33	--	9	--	--
30. Rubber	--	--	--	--	--	--	--	--
31. Leather	--	--	--	--	--	--	--	--
32. Stone	27	21	1	25	2	5	--	--
33. Prim. Metal	24	22	2	39	1	2	--	--
34. Fabr. Metal	48	3	3	27	9	10	--	--
35. Machinery	16	12	14	38	7	5	--	--
36. Electrical	39	1	12	27	13	6	--	--
37. Transportation	42	1	10	20	8	10	--	--
38. Instruments	41	14	11	15	9	10	--	--
39. Miscellaneous	33	--	--	33	8	9	--	--

as 62% and 61% to municipal sewers, while petroleum, primary metals, and paper industries rarely discharge to POTW's.

Less than half of the wastewater (43%) is subjected to treatment (Table 11-3) before being discharged. Among the different waste streams, the highest degree of treatment is given to the process water with 74% of that stream treated, with less treatment given to used air conditioning water and boiler water (32% and 32% respectively). Several industries, such as petroleum and primary metals, treat as much as 100% and 91% of their process water, while transportation equipment and fabricated metals treat only one third of that waste stream. The greatest degree of treatment is given to wastes discharged to bays and estuaries and to transferred water (65%), while the lowest treatment is given to waste discharged to oceans (24%), POTW's (30%), and ground water (30%). Some industrial categories provide very little pretreatment, while others, such as petroleum and primary metals, provide a higher degree of pretreatment (56% and 55% of that waste stream).

The most commonly used treatment processes are primary settling, used by 50% of the plants (Table 11-4), followed by pH control (44%) for neutralization. Some industries almost all use certain processes, such as surface skimming and flotation by 88% of the petroleum industries, and pH control by 83% of the electrical manufacturing plants. The highest degree of biological treatment is given to wastes from textile, petroleum, and tobacco industries. Additional solids removal by secondary settling or sand filtration is not widely used, while chlorination of the wastewater is half as often practiced as chlorination of the intake water.

11-2 Priority pollutants in industrial wastewater discharged to POTW's

Data compiled by the U.S. EPA Effluent Guidelines Division in Washington, D.C. showed that the compounds most often found in industrial effluents are bis(2-ethylhexyl), phthalate, chloroform, and dichloromethane with 42%, 40%, and 34% of the analyzed samples reporting positive identification. The phthalate is one of the most commonly used compounds and is present in most plastics, chemicals, and finished products. The chloroform and dichloromethane are likely the result of chlorination processes practiced with the municipal water, intake water, or wastewater. The next most common compounds, such as benzene (29%), toluene (29%), and phenol (26%) are used as feedstock for numerous other chemicals. More than half of the wastewater samples contained copper and chromium. The largest number of priority pollutants were found in the effluent of organic and plastic industries (106), followed by pesticide (71), iron and steel (57), petroleum refining (55), and auto manufacturing (54).

Table 11-2

Percentage of Industrial Waste Water Discharged to Different Categories

	Public Sewer	Streams Rivers	Lakes Ponds	Bays Estuaries	Ocean	Ground	Transferral	TOTAL UNTREATED (BGY)	TOTAL DISCHARGED (BGY)
All Industries	7	64	9	16	1	1	1	7987.2	14,144
20. Food	30	34	5	5	--	6	5	497.7	745
21. Tobacco	--	--	--	--	--	--	--	----	4
22. Textile	53	41	4	--	--	<1	--	91.4	160
24. Lumber	3	67	13	6	--	5	<1	84.6	123
25. Furniture	--	--	--	--	--	--	--	----	6
26. Paper	5	74	2	17	1	<1	<1	1050.6	2,301
27. Chemical	4	62	6	25	--	<1	1	2333.7	3,911
29. Petroleum	1	52	--	7	--	<1	2	464.6	1,159
30. Rubber	18	78	--	<1	--	2	--	115.8	143
31. Leather	57	42	--	--	--	--	--	4.8	8
32. Stone	10	50	20	8	6	4	1	157.8	192
33. Prim. Metal	2	72	15	8	--	2	<1	2701.5	4,757
34. Fabr. Metal	61	33	1	--	--	3	<1	73.7	101
35. Machinery	29	43	6	19	--	1	--	124.5	165
36. Electrical	62	34	1	1	--	3	--	65.8	97
37. Transportation	36	45	7	6	2	4	<1	184.1	227
38. Instruments	31	61	1	--	--	<1	--	18.4	35
39. Miscellaneous	41	56	--	--	--	--	--	9.2	12

Table 11-3

Percentage Treatment of Waste Streams Discharged to Different Receptors

	Public Sewer	Streams Rivers	Lakes Ponds	Bays Estuaries	Ocean	Ground	Transferral	TOTAL
All Industries	30	40	45	65	24	30	53	43
20. Food	33	31	58	8	100	53	0	33
21. Tobacco	--	--	--	--	--	--	--	--
22. Textile	20	67	87	--	--	--	--	43
24. Lumber	0	38	0	--	--	26	--	31
25. Furniture	0	--	--	--	--	--	--	0
26. Paper	40	56	67	52	46	89	15	54
28. Chemicals	34	28	56	67	--	0	100	40
29. Petroleum	56	50	--	0	--	100	26	60
30. Rubber	34	21	--	100	--	7	--	19
31. Leather	38	39	--	--	--	--	--	38
32. Stone	17	27	3	0	0	21	59	18
33. Prim. Metal	55	41	41	76	--	4	3	43
34. Fabr. Metal	19	39	--	--	--	54	0	27
35. Machinery	10	38	78	1	--	11	--	25
36. Electrical	28	41	33	100	--	14	--	32
37. Transportation	19	25	3	0	0	6	0	19
38. Instruments	14	61	0	--	--	0	--	47
39. Miscellaneous	15	28	--	--	--	--	--	21

Table 11-4. Percentage of Plants Using Certain Wastewater Treatment Processes

	Total	Surface Skimming	PH Control	Coagulation	Flotation	Primary Settling	Biological Oxidation	Secondary Settling	Sand Filtration	Chlorination	Other
All Industries	5320	35	44	18	11	50	36	21	5	18	21
Food	1125	38	18	9	18	36	35	15	3	15	32
Tobacco	2	50		50		100	100	100		50	
Textiles	292	12	30	9	4	34	60	30	41	25	20
Lumber	99	18	21	5	9	52	47	14	6	16	12
Furniture	21	24	62	14	10	48	24	24		24	24
Paper	445	22	21	23	11	71	44	24	3	14	20
Chemical	631	40	62	17	7	61	43	26	6	20	19
Petrols	235	88	43	18	28	71	56	40	6	13	14
Rubber	152	28	34	9	3	38	28	14	7	13	11
Leather	31	32	36	13	13	64	29	19		3	23
Stone	294	24	19	11	3	50	30	18	6	10	10
Primary Metals	520	44	51	28	7	61	29	24	8	20	15
Fabricated Metals	357	30	71	19	6	43	19	17	5	25	21
Mechanical	225	42	58	23	11	39	20	16	7	17	14
Electrical	365	18	83	23	3	40	14	19	6	21	20
Transportation	216	48	70	41	22	55	25	19	9	23	21
Instrument	71	24	73	25	4	44	27	21	6	25	28
Miscellaneous	39	8	69	31	10	35	13	22	8	28	8

The publicly owned municipal treatment plants receive varying amounts of industrial wastewaters as shown in the 1978 EPA Need Survey which inventoried plants above 5 mgd. For example, the 31 mgd St. Helens plant in Pennsylvania receives more than 95% of its flow from industrial contributors, as does the 8.5 mgd Lyman plant in South Carolina and the 10 mgd Grand Rapids plant in Michigan. For most plants, however, the percentage industrial contribution does not exceed 15%. The surveyed plants ranged in size from 5 mgd to the 800 mgd WSW plant near Chicago while the median is around 12 mgd, as shown in Figure 11-1. Some plants have high contributions of specific industries such as the Modesto, California plant having high contributions of food wastes; the Greensville, South Carolina plant having high textile contributions; the Appleton, Wisconsin plant with high paper waste contributions; and the Wilmington, Delaware plant with large chemical waste contributions.

Plants receiving large amounts of industrial chemicals usually do not meet the 20/20 or 30/30 effluent requirement. However, there are several examples of discharging plants meeting these requirements using advanced secondary or tertiary treatment techniques. For example, the 25 mgd Decator, Illinois plant, receiving 50% industrial waste, meets to 20/20 requirement by usinmg pure oxygen activated sludge and a polishing lagoon.

11-3 City Selection for POTW Sampling

Data from the U.S. Department of Labor were used to determine which cities had a high percentage of the working population in the different SIC categories. The data in Table 11-5, for example, indicate that as many as 28% work in transportation equipment manufacturing in Kenosha, Wisconsin and 28% work in primary metals in Gary, Indiana. In order to obtain a balanced selection of twenty-five POTW's for the present study, one to two cities were chosen for each industrial code category. The POTW in the city with the highest percentage workers was then contacted to determine whether the major industries were indeed discharging into the municipal or county sewer system. Often, it was noted that the major industries had their own treatment plants, while the smaller industries in that code discharged into the POTW. If the treatment plant did not receive the waste of the major industries, the next city on the list in Table 11-5 was selected and contacted.

The POTW selection also sought to obtain cities with varying death rates as such cities with high incidence of cancers and lung disease are expected to have a high number of priority pollutants in their wastewater. The death rates have to be adjusted for the age effect, as cities with a high proportion of older people have a high death rate. The death ratio was therefore developed representing the number of deaths per thousand inhabitants per year divided by the percent of the population above 65

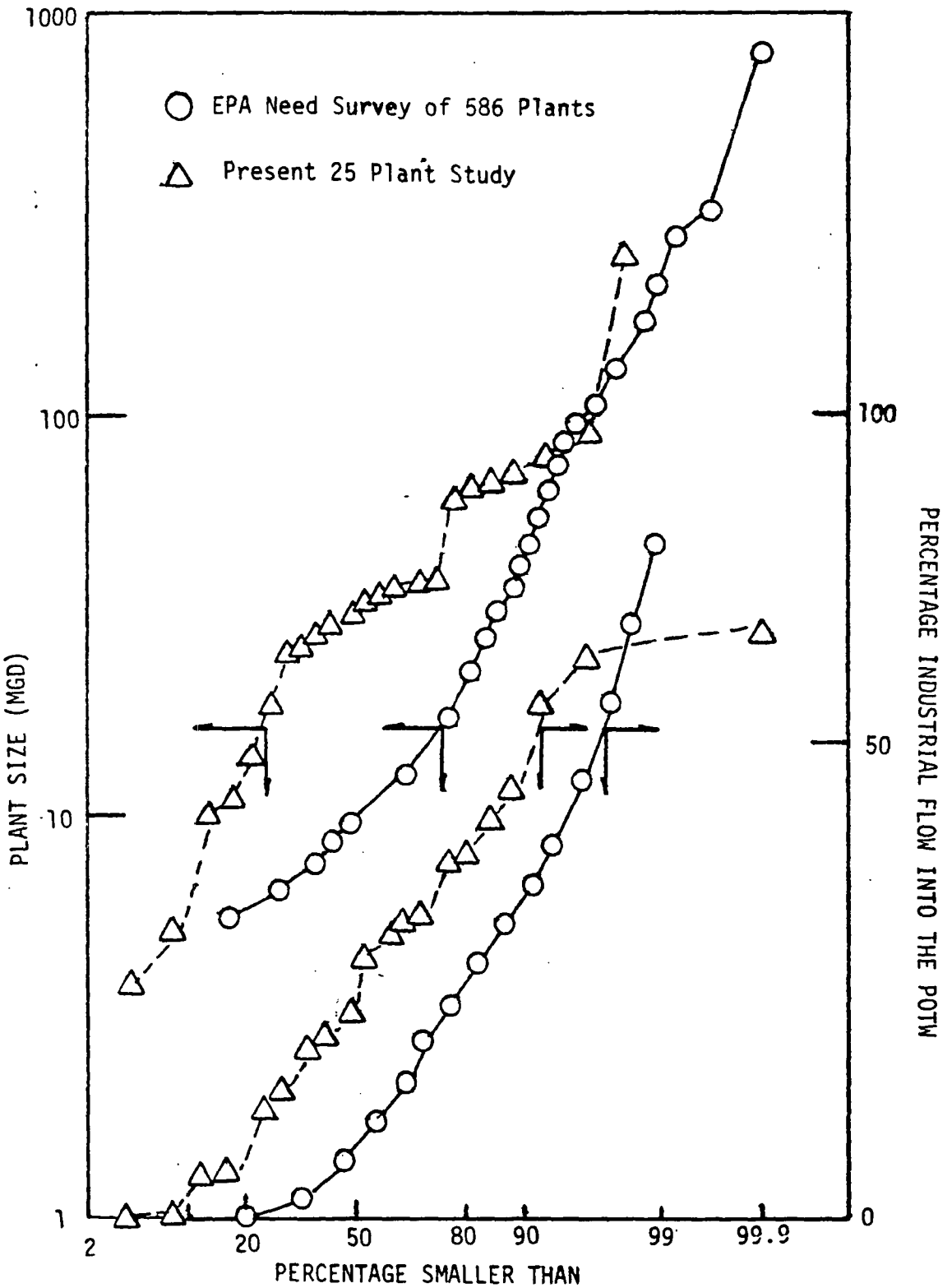


Figure 11-1 Frequency Distribution Plot of Size and Percentage Industrial Inflow into POTW'S

Table 11-5 Percentage of Employed Population Working in Specific Industries

SIC		
20	Food	Modesto, CA 16.1%; Sioux City, IA 13.5%; Battle Creek, MI 12.2%; Fayetteville, N.C. 11.9%; Cedar Rapids, IA 8.0%; Green Bay, WI 7.3%; Salem, OR 6.5%
22	Textiles	Greenville, S.C. 19%; Columbus, GA 12.3%; Charlotte, N.C. 11.1%; Augusta, GA 10%; Fall River, MA 9.1%
23	Textile Manfact.	Fall River, MA 20.2%; Eugene, OR 15.6%; Allentown, PA 9.3%; NE Pennsylvania Scranton, PA 9.1%
26	Paper	Appleton, WI 21.7%; Savannah, GA 6.6%; Greenbay WI 5.3%
28	Chemical	Wilmington, DE 17.3%; Charleston, W.V. 12.8%; Baton Rouge, LA 12.1%; Parkersburg W.V. 9.7%; Waterbury, CT 8.9%; Pennsacola, FL 8.8%; Perth Amboy , N.J. 8.5%; Terre Haute, IN 4.9%; Chattanooga, TN 4.7%
29	Petroleum	Lake Charles, LA 6%
30	Rubber	Akron, OH 14%; Tuscaloosa, Al 3.9%; Erie, PA 3.1%
31	Leather	Lawrence/Haverhill, MA/N.H. 5%; Brockton, MA 4.9%
33	Primary Metals	Gary, IN 28.2%; Youngstown, OH 19.4%; New Britain, CT 18.3%; Saginaw, MI 13.1%; Canton, OH 11.8%; Pittsburgh, PA 11.4%; Muskegon, MI 10.8%
34	Fabricated Metals	Hartford, CT 13.5%; Rockford, IL 10.5%; Waterbury, CT 8.2%; Flint, MI 6.4%; Akron, OH 6.3%
35	Machinery	Peoria, IL 23.2%; Cedar Rapids, IA 22.7%; Davenport, IA 17.2%; Racine, WI 16.2%; Muskegon, MI 13.5%; Erie, PA 13.4%; Battle Creek, MI 13%; Rockford. IL 12.4%; Dayton, OH 11.3%; Milwaukee, WI 9.7%; York, PA 9.4%
36	Electricity	New Britain, CT 10.8%; Fort Wayne, IN 8.1%; Anaheim, CA 7.2%
37	Transportation Equipment	Kenosha, WI 28.1%; Wichita, KS 18.3%; Newport, R.I. 18.3%; Saginaw, MI 14.7%; New Orleans, LA 14.2%; Lansing, MI 14.2%; Detroit, MI 13.4%; Ann Arbor, MI 12.4%; Seattle, WA 10.4%

Table 11-5 Continued

SIC	
38 Instruments	Rochester, N.Y. 19.8%; Waterbury, CT 6.1%
39 Mining	Wheeling, W.V. 10.8%; Johnstown, PA 9.8%; Bakersfield, CA 7.3%; Providence RI 7.8%

years. Cities with high death ratios are: Fayetteville, North Carolina (1.24) with major food processors; Norfolk, Virginia (1.0) with shipbuilding; Columbia, Georgia (1.01) with textiles; and Fall Rivers, Massachusetts (1.01) also with textiles. Cities with low death death ratios are Fort Lauderdale, Florida (0.50); Palm Beach, Florida (0.53); Fort Meyer, Florida (0.51); and Bradenton, Florida (0.45).

The POTW selection was also based on size of the plants, percentage industrial contribution into the network, and geographical location in order to obtain a representative range for each. The size plants in the present study range from 3.75 mgd in Lock Haven, Pennsylvania to 276 mgd in Washington, D.C. with a median size of 35 mgd, which is about three times larger than the national medium size. The percentage industrial flow ranged from 0% in Fort Meyers, Florida to 73% in Muskegon, Michigan.

The POTW selection was finally based on the type of treatment processes in order to determine the efficiency of each type. While the majority of the plants used activated sludge, three use trickling filters, while two use aerated lagoons or algal ponds. Two of the POTW's use land spreading to meet zero discharge limits. Twelve of the twenty-five plants have combined sewers and are expected to receive more PAH priority pollutants from street runoff. The final selection of plants is listed in Table 11-6 and reflect the above discussed characteristics.

11-4 Sampling Procedures of POTW's

The sampling gear utilized in the present study is depicted in Figure 11-2. The unit was custom made by Mr. D. Tigwell, instrument maker, and consists of a flow measuring component and a sampling component. The flow is measured by an ultrasonic probe which measures the height of the water level near an overflow weir. The flow meter converts the signal into a measure of flow depending on the pipe configuration. The signal then enters the computer, where it can be stored. The computer is programmed to initial the sampling process sequentially, with the first raw sewage sampler starting at t1 and the second primary effluent sampler starting at t1 + D1 with D representing the detention time in the primary sedimentation basin, etc. The samplers are also shut-off in the same sequential fashion. The computer can also be programmed to activate the sampler when the combined storm flow, as a result of a rainstorm, exceeds a certain preset flow change in a given time period. An ion selective probe (Cl, NH_4) can be connected to the computer via a potentiometer to calculate total mass flow (volume times concentration) in a given time period and to allow sampling according to mass instead of the less accurate flow. The data in the computer can be displayed on the video screen. The computer signals are transmitted by air to a receiver present at each of the four sampling stations to collect the raw sewage, primary effluent, secondary

Table 11-6 Cities Selected in the Present 25 City Survey.

CITY	POPULATION	D.R.	I.E.%	%SIC	MGD	%IND	SEWER	TREAT.FAC.	COMMENTS
Fayetteville,NC	226,000(152)	1.24	16. (-)	NA	11.5 (2)	25	SEP	Tert. O_2 and Cl_2 Filters (Cross Creek) WTP	Textiles,Rubber (Kelly) Cargill. Borden Chem.Plant Feed Processing High Death Ratio
Chattanooga, NC	392,000(92)	.80	36.2 (38.3)	8.3(22) 6.0(34) 4.7(28)	39 (5)	65	COMB	Sec.,Act.Sludge	Moccasin Bend WWTP Lots of Chem.Plants
Fort Meyers,FL	154,000(195)	.51 Low	5.3 (-)	NA Low	5.0 (1)	0.0	SEP	Sec.,Trickling, Pure Oxygen,Act.Sludge	Only Laundry and Restaurants; Lowest Death Ratio
Hammond (Gary), IN	643,000(57)	.96	44.2 (36.2)	28.2(33) 3.0(34)	35.7 (1)	26	COMB	Adv. Secondary	Steel
Fort Wayne,IN	373,000(96)	.78	35.1 (39.9)	8.26(37) 8.0(36) 4.56(35)	36.7 (1)	14	COMB	Adv.Sec.,Filters,Chem. Add.	GE,Magnavox,Essex. Lagooning of Lard Oil for Wire Drawing
Akron, OH	667,000(55)	.81	39.3 (36.9)	6.3(34) 14.0(30) 4.7(35)	78 (3)	13	COMB	Sec.,Act. Sludge	Goodyear
Atlanta, GA	1,790,000(18)	.85	21.6 (41.0)	3.07(37) 2.07(30) 9 other ground (1)	90	6	COMB	Adv.Prim.,Act.Sludge, Bio.Nitrification	R M Clayton WPCP
Winston Salem,NC	764,000(49)	.81	41.3 (42.9)	11.2(22) 2.5(23)	25.8 (5)	54	SEP	Sec. Trickling,Act. Sludge,Single Stage Nitrification	Archie Elledge Plant., R.J.Reynolds (Food & tobacco),Schlitz has 2 pretreat. lagoons(1/10 of flow)
Lock Haven, PA	15,000(-)	--	--	-----	3.75	38%	SEP	Sec.,Aeration	BOD,SS,Cyanide, and chemical violations.
Greenville,SC	525,000(70	.85	41.4	19.0(22) 4.9(35) 4.1(23)	25 (5)	23	SEP	Tert.Trickling Filter	Paper Mill Input; Textiles
Providence, RI	904,000(38)	.75	36.5 (39.3)	7.8(39) 3.2(22) 3.0(34) 3.0(35)	65 (5)	38%	COMB	Sec.,Act.Sludge	E.Providence, Jewelry, high Ni,Zn, Cd.
Modesto, CA	223,000(154)	.74	19.4 (31.3)	16.1(20) 2.8(34)	36	Seasonal	SEP	Sec.,Lagoons, Land Treatment Anaerobic Digestion	Canning from Aug.-Sept. Gallo Vineries
Peoria, IL	354,000(100)	.75	34.1 (40.9)	23.2(35) 4.5(33) 4.0(20)	28 (5)	21%	COMB	Sec.,Act.Sludge, Anaerobic Digestion Building RBC's and Polishing/Holding Ponds	Hiram Walker, Catapillar Tractor

Table 11-6 Continued.

CITY	POPULATION	D.R.	I.E.%	%SIC	MGD	%IND	SEWER	TREAT.FAC.	COMMENTS
Rochester,NY	970,000(37)	.73	45.3 (39.5)	19.8(38) 3.7(35) 2.97(36)	69 (2)	22	SEP	Adv.Sec.,Filters or Additions, Lagoons	Frank E Van Lare Plant, Kodak,Xerox Brewery,Auto Support $25million in pretreatment since '72
Springfield, MA (Chicopee-Holyboke)	550,000(68)	.76	33 (28.2)	3.5(26) 3.1(35) 2.7(34)	32 (2)	38 50 by loading	COMB	Sec.,Act. Sludge	Paper,Monsanto, Textile,Drop Forges Monsanto has settling,acid neutral. Bondits
Lawrence,Haverhill, MA	270,000(132)	--	-- (35)	4.96(31) 2.5(30) 2.3(35)	10.2 (2)	33	COMB	Sec.,Act.Sludge	Tanning,Dyers Paperboard;pretreats for fiber recovery
Oakland,CA (SF-Berk)	3,140,000(7)	.75	16.7 Non-Agric Employ. (44.)	.42(30) 1.41(27)	60.8 (2)	16.	SEP	Sec.,Act.Sludge Oxygen fed Act.	East Bay Mud
Seattle,WA Everett,WA	1,406,000(24)	.76	24.2 (40.4)	10.4(37) 1.34(35) 1.53(30)	29.4	.06	COMB	Adv.Sec.,Act. Sludge	Renton WWTP
Allentown,PA Bethelehem- Easton,PA,NJ	624,000(59)	.75	45.7 (40)	11.2(34-36) 9.4(23) 3.9(35) 3.8(36)	30 (4)	40	SEP	Adv.Sec.with NH_3-NO_3 Tertiary	Kline Is. WWTP Pretreatment 2MGD Dyes; 3MGD Kraft,1.5 MGD Brewery,Foods,Chem. Plants,Batteries
Wilmington,DE	518,000(72)	.92	32.4 (39.3)	17.3(28)	70 (4)	50 32 manf. 18 chem.	COMB	Adv.Sec.,Act.Sludge	Dupont does some pretreatment
Washington,D.C.	3,021,000(7)	.91	6.6 (44)	1.4(27) .4(30)	276.4 (2)	0	SEP	Act.Sludge Sec. Chem.Addition $FeCl_3$	Blue Plains has a sludge program Low industry
Rockford, IL	272,000(131)	.73	45.3 (41.0)	12.5(35) 10.5(34) 8.9(37)	39 (1)	45%	SEP	Sec.,Act.Sludge Aerobic Digestion Vacuum filtration	ASMA Study Member,cyanide problems
Appleton, WI	284,000(124)	.70	36.2 (37.3)	12.7(26) 5.7(35) 3.6(27)	14 (5)	35%	SEP	Sec.,Act.Sludge; Polymer,Ferric Chloride addition, Zimmerman Process, Vacuum filtration	Paper Mill Canning
Kenosha, WI	123,000(228)	.74	42.8 (34.3)	28.1(37) 5.0(34) 3.8(33)	19 (1)	46%	COMB	Sec.,Act.Sludge Chem. Addition (Pickling liquor) AnaerobicDigestion	AMC,Eaton Transmission Anaconda Brass Ocean Spray
Muskegon, MI	178,000	.86	43.8 (31.8)	5.29(34-36) 2.65(20) 3.57(31)	35 (1)	73	SEP	Aeration,Lagoons Land Application to grow corn.	High organic waste, have GC/MS system

Symbols Used for Headlines of Chart:

Population (Ranking, 1977)

D.R. = Death Ratio $\frac{\text{Death per 1000 per year}}{\text{\% Population above 65 years}}$

I.E.% = Industrial Employment (1970)
( ) = (Non-Agricultural Employment)

%SIC = % of workers in SIC
( ) = SIC number

MGD = Plant Size Million Gallon/Day

(Effluent Category): 1 = 20/20 4 = 60/60
2 = 30/30 5 = 100/100
3 = 45/45

%IND = % Industrial Input (Usually by Flow)

SEWER: SEP = Separate COMB = Combination

TREAT.FAC. = Treatment Facility

Trickling Filters

Greenville, S.C. Fort Meyers, FL
Winston Salem, N.C.

Combined Storm Sewers

Hammond, IN	Providence, RI
Fort Wayne, IN	Perth Amboy, NJ
Akron, OH	Wilmington,DE
Springfield, MA	Chattanooga, TN
Haverhill, MA	Seattle, WA
Kenosha, WI	Atlanta, GA

Lagoons and Ponds

Modesto, CA Muskegon, MI
Rochester, NY

Land Spreading (Effluent)

Modesto, CA Muskegon, MI

SIC: Numbers in Parenthesis represent:

20 - Food and kindred products	29 - Petroleum and coal products
21 - Tobacco	30 - Rubber and plastic products,NEC
22 - Textile mill products	31 - Leather and its products
23 - Apparel and other textile	33 - Primary metal industries
26 - Paper and allied products	34 - Fabricated metal-products
27 - Printing and publishing	35 - Machinery,except electrical
28 - Chemical and allied products	36 - Electrical equipment and supplies
	37 - Transportation equipment
	38 - Instruments and related products
	39 - Mining

Number of States in Survey (18)

California (2)	Illinois (2)	North Carolina (2)
Delaware	Indiana (2)	Ohio
District Of Columbia	Massachusetts (2)	Pennsylvania (2)
Florida	Michigan	Rhode Island
Georgia	South Carolina	Tennessee
	New York	Washington
		Wisconsin (2)

Table 11-6 Continued.

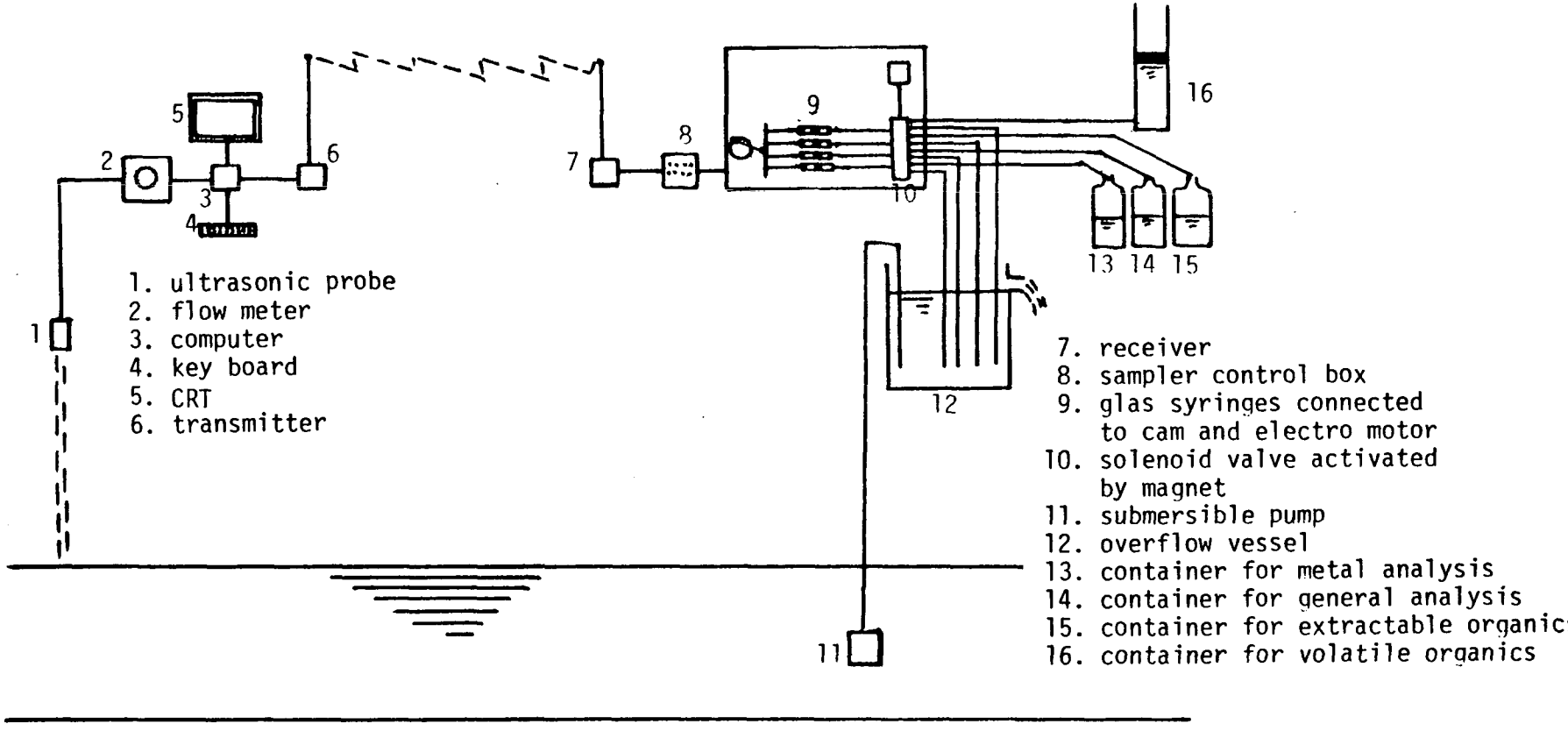


Figure 11-2 Schematic of Sampling Units Employed in the Present Study.

effluent, and secondary effluent after chlorination or dechlorination. The fifth sample, the digested sludge is collected as a grab sample.

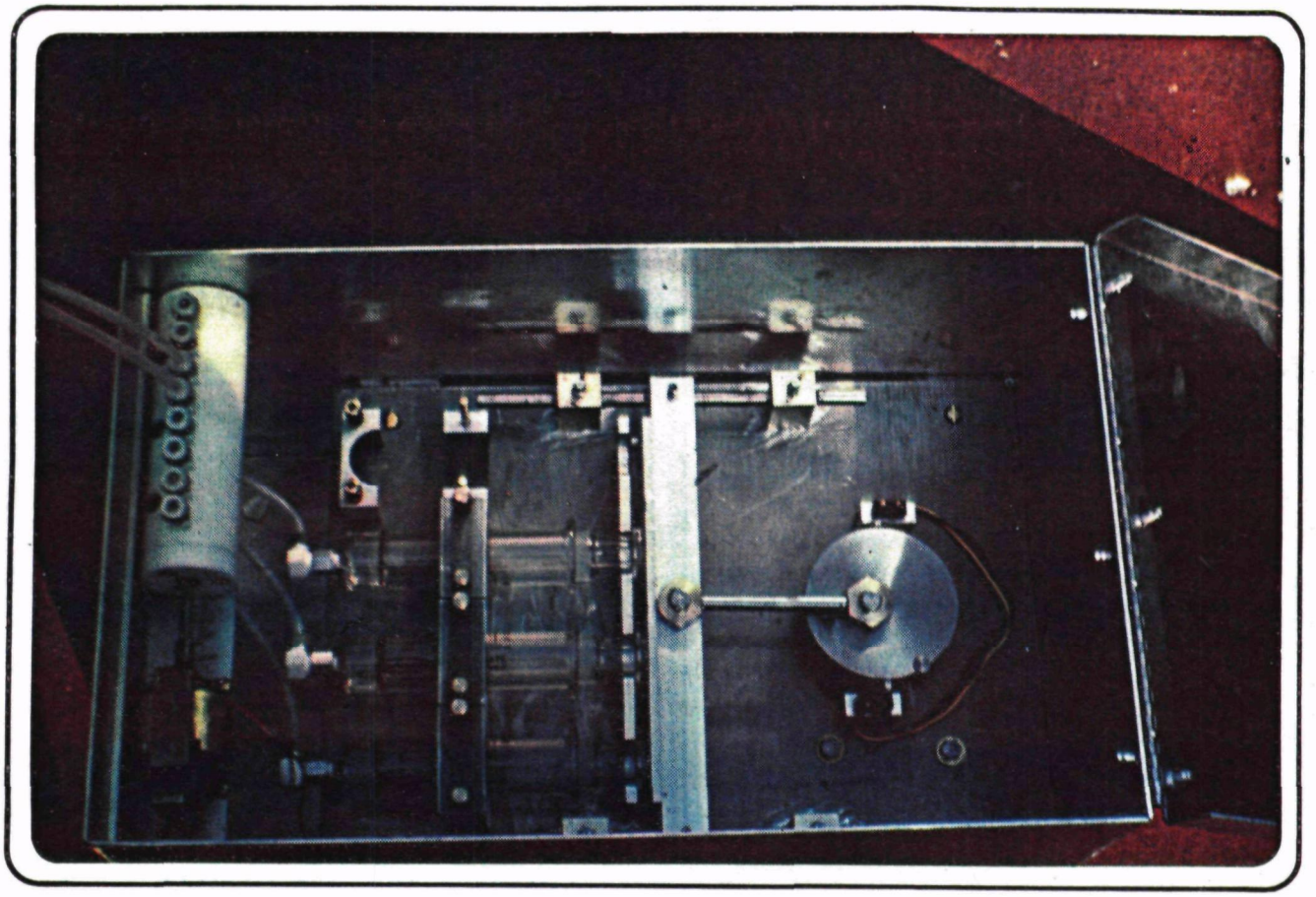
The receiver gives the signal to the sampler control box, which activates the electrometer that drives the syringes of the four sampling lines. The liquid is sucked into the teflon lines from the overflow vessel by the negative pressure of the syringes At that point, the teflon solenoid volume switches, and liquid is pressurized by the syringes into each of the four collection vessels. The vessel used for the volatile organic analysis contains a floating teflon plunger to prevent any volatilization of the organics. All glass vessels for extractable organics are silanized to prevent adsorption of organics on the wall of the vessel, while the polyethylene containers for the other analysis are nitric acid rinsed. The containers are icechilled during collection and shipped by express air freight to the laboratory. The collection system is depicted in plates XI - XIII.

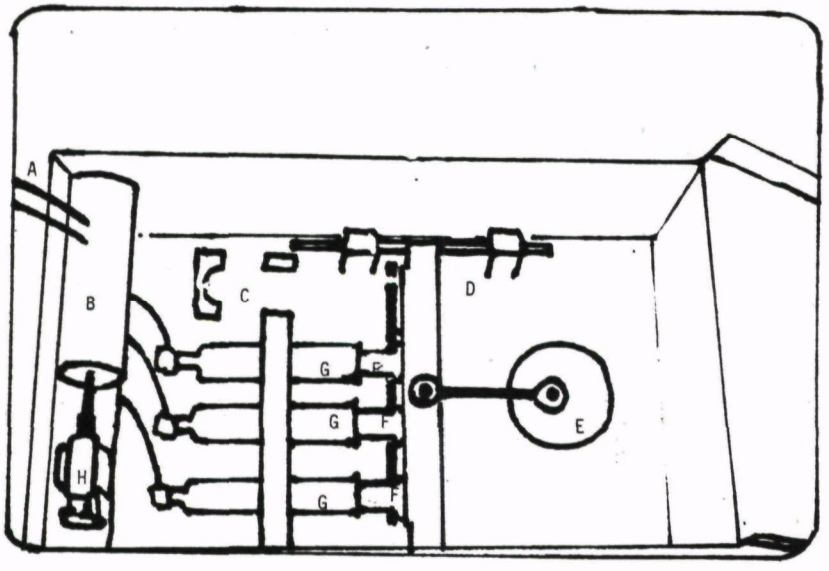
11-5 Removal Efficiencies at POTW's

The tabulation of priority pollutant concentrations in each of the first three POTW's is presented in this section. The information in Table 11-7 shows where each of the five 24 hour composite samples were collected. A description of each treatment plant is given in Tables 11-8, 9, and 10.

The data reported in this and subsequent sections are actual analytical results, uncorrected for QA/QC recoveries. As the body of QA/QC information grows, estimates of actual concentrations will be made and attention given to those compounds with recoveries sufficiently low that the measured values might be misleading indicators of actual levels present.

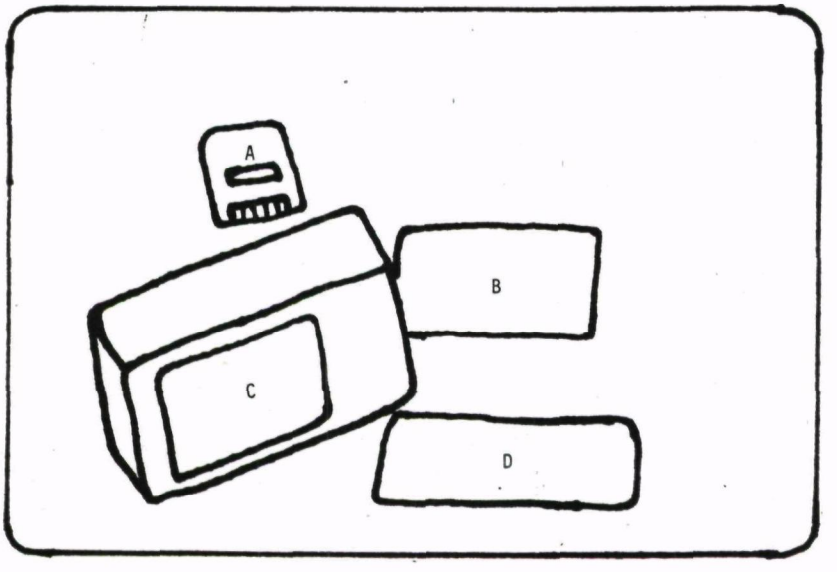
The actual data on Renton-Seattle (Table 11-11); Clayton, Atlanta (Table 11-12); and EBMUD, Oakland (Table 11-13) shows that removals tend to increase with increasing molecular weight or increasing GC column retention. While phthalates and PAH's tended to increase in the sludge, no such accumulation was noted for chlorinated Cl compounds. A tabulation of individual compounds in Table 11-14 shows that a wide range of compounds were detected. A typical presentation of the total number of compounds in each of the groups for Oakland is shown in Figure 11-3 while the accumulation of the different fractions in sludge is shown in Figure 11-4.





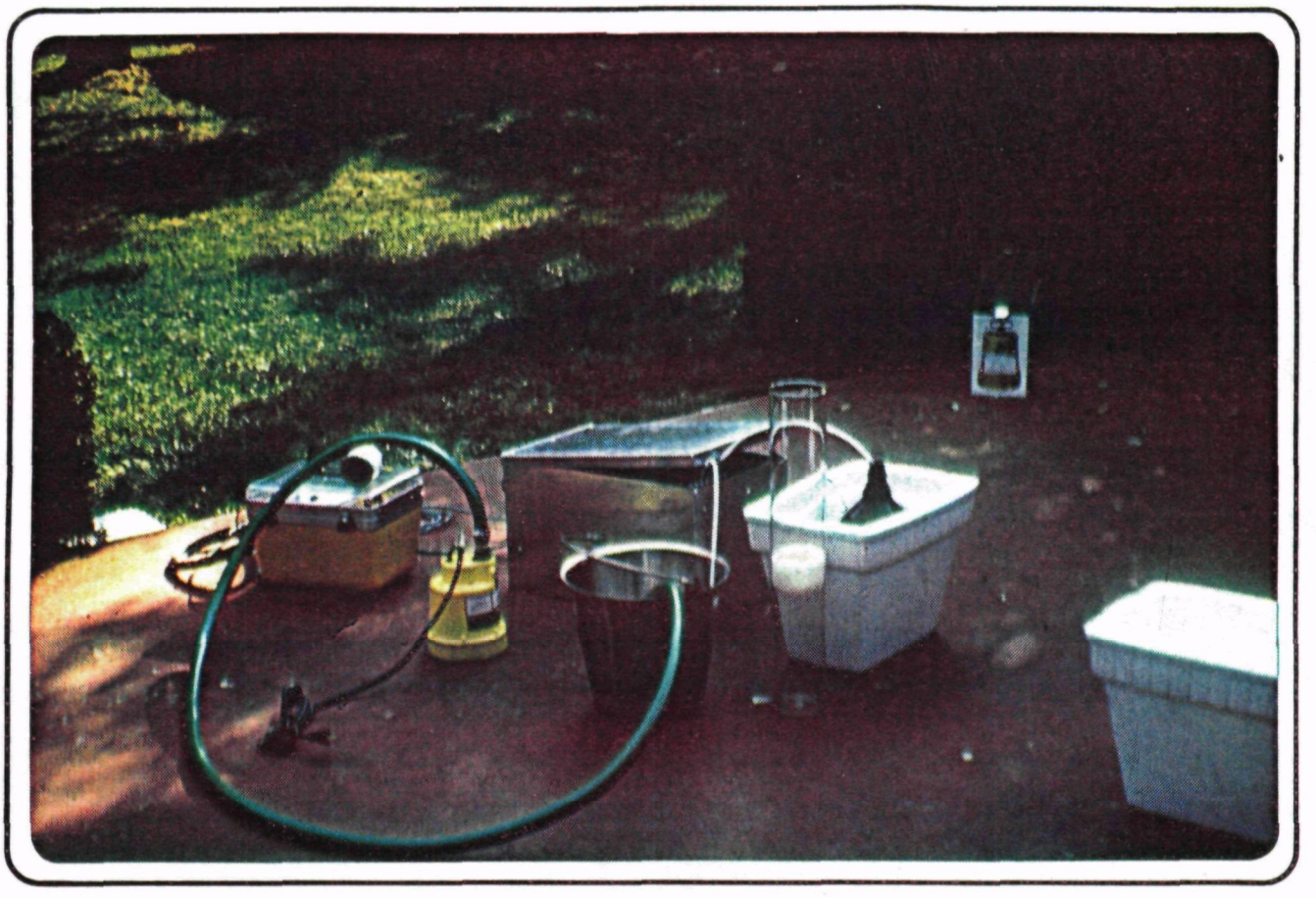
A INLET AND OUTLET LINES
B DIVERTING VALVES FOR INLET AND OUTLET LINES
C SYRINGE MOUNTING BRACKET
D PISTON GUIDE
E MOTOR
F SYRINGE PLUNGER
G SYRINGE BARREL
H VALVE ACTUATING SELINOID

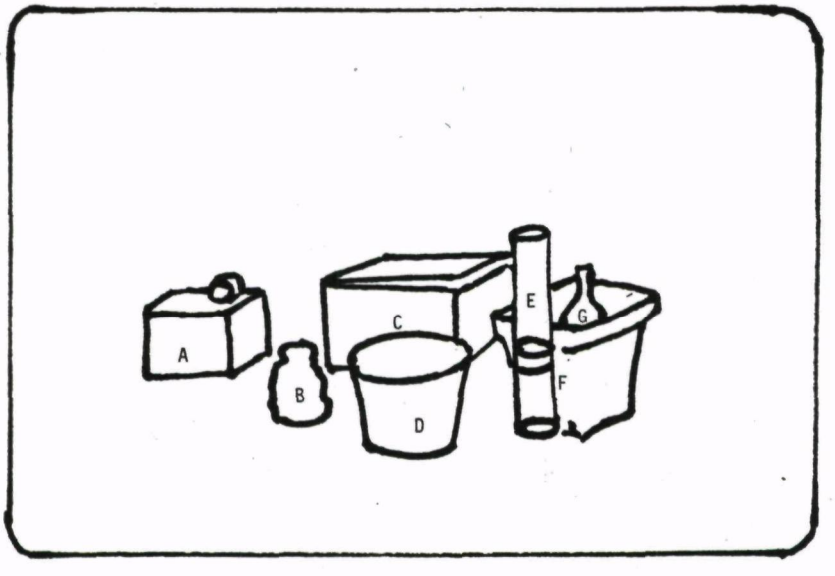
PLATE XI DRIVEN - SYRINGE SAMPLER
FOR AUTOMATED SAMPLING SYSTEM



A CASETTE TAPE DECK
B MICRO-COMPUTER ELECTRONICS
C CRT TERMINAL
D KEYBOARD

PLATE XII AUTOMATED SAMPLING SYSTEM
CONTROLLING ELECTRONICS





A ULTRASONIC FLOW METER
B PUMP
C AUTOMATIC SAMPLER
D STAINLESS STEEL BUCKET
E VOA SAMPLER
F TEFLON FLOAT IN VOA SAMPLER
G SAMPLE COLLECTION BOTTLE

PLATE XIII AUTOMATED SAMPLING SYSTEM

Table 11-7

Description of Sampling Sites

	Renton-METRO	Atlanta(Clayton)	Oakland(EBMUD)
	April 9	June 14	July 3
Raw Sewage	after prechlorination, before grit removal.	Before grit & prechlorinated	after sludge dewatering supernatant return influent & after prechlorination
Primary Sewage	sample at outflow from primary sedimentation basin in tunnel	"Channel" open (2 hrs) between activated sludge and primary	Main Channel to Oxygenation tanks
Secondary effluent before chlorination	from second sediment tank outflow	Before Chlorination (5 hrs) "weir box" entire plants	Deep Channel right before chlorination box.
Secondary effluent after chlorination	from sample at dechlorination station	weir box after 15 minutes	Outfall several miles from plant
Primary and Secondary Sludge	WAS from open chambers and primary Sludge combined	After digestion	After digestion

Table 11-8
Renton Treatment Plant Information

System Characteristics

Population (indicate year and source)	198,000 (May 1978)
No. of Businesses	NA
Surface area	66 square miles
No. of waste discharge permits	37
Miles of combined sewers	none

Plant Characteristics

Maximum flow (m^3/sec; MGD)	72 mgd
Dry weather flow	36 mgd
Percentage Industrial Flow	5%
Treatment Sequence: (Indicate details)	screenings ground and recycled, aerated grit channel, grit dewatered, trucked to landfill. Primary, activated sludge, secondary, chlorination, SO_2 dechlor.
Solids handling (Include disposal)	Raw sludge, screen & WAS is pumped via forced main to the West Point Treatment Plant to be digested, dewatered & dumped into Puget Sound.
Screened debris volume (cubic feet/time)	
Grit Volume (cubic feet/time)	160 cu ft/day
Floating oil from Skimmers (cubic feet/time)	unknown
Sludge cake (dry tons/year)	18,993 metric tons primary ; 5,072 metr.
Oxygen production (metric tons/year)	none tons secondary.
Chlorine usage (metric tons/year)	137.66 metric tons/year
Primary sludge stream (volume)	.509 MGD
Secondary sludge stream (volume)	.99 MGD
Digested sludge stream (volume)	Goes to Westpoint

Plant Operation

Activated sludge loading (lbs.BOD/lbs. Volatile SS/day)	.929
Digestor loading (lbs. Total Solids/1,000 cubic feet/day)	No Digestors
Activated sludge age (days)	2.03 days
Digestor detention time (days)	No digestors
Chlorine contact time (minutes)	35 minutes
Residual Chlorine (mg/l)	0.26

Wastewater Characteristics (mg/l)

BOD Influent	273 mg/e
Effluent	12.5 mg.e total, 8.9 mg.1 carbonaceons
SS Influent	351 mg/l
Effluent	12.5 mg/l
Total Nitrogen Influent	33.5 mg/l
Effluent	17.3 mg/l
Total Phosphorus Influent	10.4 mg/l
Effluent	4.9 mg/l

Table 11-9
Atlanta Treatment Plant Information

System Characteristics

Population (indicate year and source) 367,000
No. of Businesses not available
Surface area 142 square miles
No. of waste discharge permits 122
Miles of sewers 390 total miles of sewers (combined & uncombined)(8"-54" sewers)

Plant Characteristics

Maximum flow (m^3/sec; MGD) 150 MGD (180 MGD design)
Dry weather flow 65 MGD
Percentage Industrial Flow not available
Treatment Sequence:
(Indicate details) coarse screening, grit removal, primary clarification, activated sludge chlorination, discharge.

Solids handling
(include disposal) Flotation thickening to Waste Activated Sludge. WAS and primary sludge to Anaerobic Digestion. Digested sludge dewatered by centrifugation, incinerated and put on landfill.

Screened debris volume (cubic feet/time) not available
Grit Volume (cubic feet/time) not available
Floating oil from Skimmers (cubic feet/time) not available
Sludge cake (dry tons/year) 21,000
Oxygen production (metric tons/year) 365,000
Chlorine usage (metric tons/year) 990
Primary sludge stream (volume) 288,000 gallons/day
Secondary sludge stream (volume) 1.1 million gallons/day
Digested sludge stream (volume) not available

Plant Operation

Activated sludge loading (lbs.BOD/lbs. Volatile SS/day) Not available
Digestor loading (lbs. Total Solids/1,000 cubic feet/day) Not available
Activated sludge age (days) 2
Digestor detention time (days) 30-40
Chlorine contact time (minutes) 20
Residual Chlorine (mg/l) 1

Wastewater Characteristics (mg/l)

BOD Influent 122
Effluent 31
SS Influent 141
Effluent 40
Total Nitrogen Influent
Effluent
Total Phosphorus Influent 6.2
Effluent 4.0

Table 11-10

Oakland Treatment Plant Information

System Characteristics

Population (indicate year and source)	575,000
No. of Businesses	7,000
Surface area	215 km^2 (83 sq. miles)
No. of waste discharge permits	2,300 km (1,400 miles)
Miles of combined sewers	of combined sewers

Plant Characteristics

Maximum flow (m^3/sec; MGD)	7.4 m^3/second (168 mgd)
Dry weather flow	2.7 m^3/second (62 mgd)
Percentage Industrial Flow	15%

Treatment Sequence:

(Indicate details) prechlorination, screening, grit chamber, aerated grit chamber, primary sedimentation, pure oxygen activated sludge, chlorination, dechlorination, outfall.

Solids handling

(include disposal) anaerobic digestors, vacuum filters, sanitary landfill

Screened debris volume (cubic feet/time)	3,500 cubic feet/month
Grit Volume (cubic feet/time)	8,500 cubic feet/month
Floating oil from Skimmers (cubic feet/time)	270 cubic feet/month
Sludge cake (dry tons/year)	605 dry ton/year
Oxygen production (metric tons/year)	226 metric tons/day (250 tons/day)
Chlorine usage (metric tons/year)	2,446 metric tons/year (2,700 tons/year)
Primary sludge stream (volume)	4.6 MGD
Secondary sludge stream (volume)	3.34 MGD
Digested sludge stream (volume)	1.26 MGD

Plant Operation

Activated sludge loading (lbs.BOD/lbs. Volatile SS/day)	.42
Digestor loading (lbs. Total Solids/1,000 cubic feet/day)	97
Activated sludge age (days)	6.6
Digestor detention time (days)	15
Chlorine contact time (minutes)	120
Residual Chlorine (mg/l)	0 (dechlorinated)

Wastewater Characteristics (mg/l)

BOD	Influent	330 mg/l
	Effluent	8 mg/l
SS	Influent	470 mg/l
	Effluent	21 mg/l
Total Nitrogen	Influent	52.7 mg/l
	Effluent	37.9 mg/l
Total Phosphorus	Influent	12 mg/l
	Effluent	8.1 mg/l

THIS DATA UNCORRECTED FOR RECOVERY

Table 11-11 Priority Pollutants in Renton, Seattle POTW

	Sewage	Primary Effluent	Secondary Effluent	Secondary Effluent Chlorinated	Sludge
TRICHLOROFLUOROMETHANE	1.54				
1,1-DICHLOROETHENE			0.52	3.87	
DICHLOROMETHANE	292.8	62.3	303.5	327.1	NQ
1,2-DICHLOROETHENE			0.28	0.17	1.62
1,1-DICHLOROETHANE			0.45	1.78	
TRICHLOROMETHANE	4.2	2.8	4.0	7.5	4.5
1,2-DICHLOROETHANE	1.5	0.87	0.53	1.7	
TETRACHLOROMETHANE		0.08			0.08
1,1,1-TRICHLOROETHANE	9.1		11.2	32.6	12.1
BENZENE	8.77	4.5	3.0	8.9	5.2
METHYLBENZENE	0.85		8.4	11.0	35.8
TETRACHLOROETHENE	6.4	5.8	19.8	24.5	38.2
ETHYLBENZENE	0.18		0.17	1.20	1.51
Cl_n-C_1	298.5	265.2	307.5	334.6	4.6
Cl_n-C_2	18.6	9.1	17.3	66.3	65.5
Cl_n-AROM.	9.4	4.3	10.1	86.2	42.8

THIS DATA UNCORRECTED FOR RECOVERY

Table 11-11 Cont. RENTON-SEATTLE

	Sewage	Primary Effluent	Secondary Effluent	Secondary Effluent Chlorinated	Sludge
BIS(2-CHLOROETHYL)ETHER	0.06	0.07			
1,3-DICHLOROBENZENE	0.71	9.56		NQ	
1,4-DICHLOROBENZENE	5.02	3.57	0.74	1.93	
1,2-DICHLOROBENZENE	4.79	4.83	2.95	3.88	
BIS(2-CHLORISOPROPYL)ETHER	0.16	0.32	0.06		
N-NITROSO-DI-N-PROPYL AMINE	0.49		0.06	0.31	59.8
NITROBENZENE		0.05			
BIS(2-CHLOROETHOXY)METHANE	0.31	0.13	0.02		
1,2,4-TRICHLOROBENZENE		0.21			
NAPHTHALENE	1.27	1.18	0.21		11.0
ACENAPTHYLENE	0.04	NQ			
DIMETHYLPHTHALATE	0.72	0.52			4.2
2,6-DINITROTOLUENE		0.+8			42.6
ACENAPHTHENE	0.09	0.06			
FLUORENE	0.15				
DIETHYLPHTHALATE	6.57	7.44	0.16		6.0
N-NITROSODIPHENYLAMINE	0.24				
PHENANTHRENE	0.37	0.14	0.40		
ANTHRACENE	NQ	0.01	NQ		
DI-N-BUTYLPHTHALATE	5.58	3.01	1.79	5.41	119.2
FLUORATHENE	0.16	0.02			
PYRENE	0.16				
BUTYLBENZYLPHTHALATE	6.69	2.59			20.0
BENZO(A)ANTHRACENE	0.06				
CHRYSENE	0.09				
BIS(2-ETHYLHEXYL)PHTHALATE	1.33		0.92	12.75	40.09
DI-N-OCTYL PHTHALATE					124.0
BENZO(B)FLUORANTHENE	0.07				
ETHER	0.43	0.52	0.08		
AMINE	0.73		0.06	0.31	59.8
BENZENE	10.5	9.4	3.7	5.8	42.6
AROMATIC HYDROCARBONS	2.43	1.46	0.61		11.0
PHTHALATES	20.9	13.56	2.87	18.2	293.5

THIS DATA UNCORRECTED FOR RECOVERY

Table 11-11 Cont. RENTON-SEATTLE

Compound	Sewage	Primary Effluent	Secondary Effluent	Secondary Effluent Chlorinated	Sludge
α-BHC	0.130	0.125			0.159
LINDANE	0.579				0.323
β-BHC	0.226				
Δ-BHC			0.895	0.889	
HEPTACHLOR	37.02			0.012	
HEPTACHLOR EPOXY	0.154	0.176	0.084	0.085	
DIELDRIN	0.022			0.001	0.017
p,p'-DDE	0.183				
p,p'-DDD	0.181				
p,p-DDT	0.525				
METHOXYCHLOR					0.468
MIREX	0.019				
ENDRIN	0.191			0.188	
α-ENDOSULFAN	0.101		0.009		0.042
CHLORDENE		0.224	0.097		0.535
γ-CHLORDANE	0.214	0.185	0.198	0.066	
α-CHLORDANE	0.207	0.126	0.078		0.174
PESTICIDES	40.1	0.84	1.36	1.24	1.68

Compound	Sewage	Primary Effluent	Secondary Effluent	Secondary Effluent Chlorinated	Sludge
4-CHLORO-3-METHYLPHENOL		1.26			
2-NITROPHENOL		23.0			1.44
4,6-DINITRO-2-METHYLPHENOL		1.62	5.35		7.7
PENTACHLOROPHENOL		4.68	11.8	14.7	
PHENOLICS	-	10.46	17.2	14.7	9.14

THIS DATA UNCORRECTED FOR RECOVERY

Table 11-11 Cont. Seattle, Washington Plant (#1)

Inorganic Priority Pollutants (ppb)

Metal	1-1 (A/RS1)	1-2 (A/PE1)	1-3 (A/SEBC)	1-4 (A/SEAC)	1-5 (A/S1)
Antimony	4	3(82)	4.5	4	40(90)*
Arsenic	12.3	21.3	4.4	4.5	396
Beryllium	0.2	0.2(71)	0.2	0.1	3.4(95)
Cadmium	22	20	23	14	400
Chromium	170	156	35	30	2225
Copper	100	72.5	<1	<1	2080
Lead	12	10	14	6	190
Mercury	11	12(80)	2	1	85(70)
Nickel	105	80	80	70	9,490
Selenium	1	1(89)	1	1.8	9(20)
Silver	173	7.9(56)	1	1	213(270)
Thallium	ND	ND	ND	ND	ND
Zinc	125	80(190)	50	25	3125(112)
Cyanide	29	23	8	0	122

*% recovery of spiked sample is given in the parenthesis.

THIS DATA UNCORRECTED FOR RECOVERY

Table 11-12 Priority Pollutants in Clayton, Atlanta POTW

CLAYTON-ATLANTA

Compound	Sewage	Primary Effluent	Secondary Effluent	Secondary Effluent Chlorinated	Sludge
DICHLORODIFLUOROMETHANE	Data wiped out from disc	9.2			Data wiped out from disc
TRICHLOROFLUOROMETHANE		58.8	5.9		
1, 1-DICHLOROETHENE		43.2	1.2	0.65	
DICHLOROMETHANE		647.1	182.5	140.6	
1, 2-DICHLOROETHENE		0.89			
1, 1-DICHLOROETHANE		6.7	0.56		
TRICHLOROMETHANE		17.1	0.38	0.56	
TRICHLOROETHANE			2.5	1.8	
1, 2-DICHLOROETHANE		8.4	1.3	1.1	
1,1,1-TRICHLOROETHANE		1791.5	36.1	30.8	
BENZENE		7.7	0.65	1.08	
BROMODICHLOROMETHANE		0.67			
METHYLBENZENE		70.1	.68	1.51	
TETRACHLOROETHENE		566.0	5.3	4.37	
ETHYLBENZENE				1.27	
$C1\text{-}C_1$		732.9	188.8	141.2	
$C1\text{-}C_2$		2416.7	47.0	40.0	
C1-AROMATICS		76.9	1.4	2.6	

THIS DATA UNCORRECTED FOR RECOVERY

Table 11-12 Cont. CLAYTON-ATLANTA

	Sewage	Primary Effluent	Secondary Effluent	Secondary Effluent Chlorinated	Sludge
BIS(2-CHLOROETHYL)ETHER		0.36	0.09	NQ	
1,3-DICHLOROBENZENE	3.15	3.8	0.45	1.32	17.0
1,4-DICHLOROBENZENE	13.94	14.26	2.04	5.74	89.4
1,2-DICHLOROBENZENE	43.86	52.87	1.22	10.12	312.4
BIS(2-CHLOROISOPROPYL)ETHER			0.34	0.13	
N-NIRTOSO-DI-N-PROPYL AMINE		1.38		0.11	
NITROBENZENE	0.12		0.01		
BIS(2-CHLOROETHOXY)METHANE		0.89			
1,2,4-TIRCHLOROBENZENE	0.61		0.09	0.34	
NAPHTHALENE	10.94	14.55	4.06	0.37	318.6
ACENAPHTHYLENE	0.25	0.23	0.04		19.6
DIMETHYLPHTHALATE		0.94	0.12	0.12	
2,6-DINITROTOLUENE		0.35			
ACENAPHTHENE	0.88	0.63	0.05	0.04	21.50
FLUORENE	1.72	1.05	0.12		51.27
DIETHYLPHTHALATE	1.36	5.50	0.26	0.25	
DIPHENYLHYDRAZINE			0.06		3.6
N-NITROSODIPHENYLAMINE				0.20	122.6
PHENATHRENE	3.24	1.56	0.28	0.28	101.0
ANTHRACENE	NQ	NQ	0.07	NQ	9,6
DI-N-BUTYLPHTHALATE	12.91	22.92	4.97	1.82	200.2
FLUORANTHENE	0.06		0.05	0.06	27.2
PYRENE	NQ		0.14	0.18	39.6
BUTYLBENZYLPHTHALATE	17.88	26.07	1.63	3.68	3115.8
BENZO(A)ANTHRACENE					28.8
CHRYSENE					23.0
BIS-(2-ETHYHEXYL)PHTHALATE	50.0	26.21	11.17		3794.8
DI-N-OCTYL PHTHALATE			0.54		181.8
BENZO(B)FLUORANTHENE					14.6
BENZO(K)FLUORANTHENE					13.6
BENZO(A)PYRENE					8.4
ETHERS		1.14	0.43	0.13	
AMINES		1.38	0.06	0.31	126.2
BENZENES	61.7	71.4	3.81	17.5	418.8
AROMATIC HYDROCARBONS	17.2	17.81	4.8	0.97	652.3
PHTHALATES	81.5	81.64	18.7	5.87	7292.6

THIS DATA UNCORRECTED FOR RECOVERY

Table 11-12 Cont. CLAYTON-ATLANTA

Compound	Sewage	Primary Effluent	Secondary Effluent	Effluent Chlorinated	Sludge
α-BHC		0,123	0.126		
LINDANE	0.140	0.171	0.147	0.165	0.130
δ-BHC				0.437	
HEPTACHLOR		0.012			
HEPTACHLOR EPOXY				0.361	0.263
DIELDRIN		0.016	0.048	0.036	0.060
ENDRIN				0.391	
p,p'-DDE			0.048	0.187	
p,p'-DDD	0.173	0.180	0.143	0.169	0.203
p,p'-DDT		0.569	0.401	0.456	
METHOXYCHLOR		0.484		0.501	
α-ENDOSULFAN		0.038	0.009		0.219
β-ENDOSULFAN		0.050	0.128	0.045	0.049
ENDOSULFAN SULFATE				4.837	
CHLORDENE		0.078	0.197	0.106	
γ-CHLORDANE	0.327	0.349	0.249	0.823	0.710
α-CHLORDANE	0.100	0.324	0.325	0.609	0.470
PESTICIDES	0.74	2.39	1.82	9.12	2.1

Compound	Sewage	Primary Effluent	Secondary Effluent	Secondary Effluent Chlorinated	Sludge
2,4-DIMETHYLPHENOL					11.4
2,4-DICHLOROPHENOL					9.4
4,6-DINITRO-2-METHYLPHENOL					0.14
PENTACHLOROPHENOL	29.6	13.3		13.5	
PHENOLICS	29.6	13.3	-	13.5	20.9

THIS DATA UNCORRECTED FOR RECOVERY
Table 11-12 Contin. R. M. Clayton, Georgia Plant (#2)

Inorganic Priority Pollutants (ppb)

Metal	2-1	2-2	2-3	2-4	2-5
Antimony	<1	<1	<1	<1	<1
Arsenic	60	96	30	30	4,950
Beryllium	4	6.3	5.1	2.9(73)*	23(240)
Cadmium	1.1	1.3	<1	<1 (110)	770
Chromium	56	42	16	15(187)	23,500
Copper	21	16	31	31	2470
Lead	117(90)	62	47	22(90)	12,860
Mercury	9.6	6.4	0.7	1.6	475
Nickel	565	1965	1365	865	98,975
Selenium	3.8	2.5	3.8	7	<1
Silver	9.6	7.6	7.1	7.1(44)	395
Thallium	<1	<1	<1	<1	<1(100)
Zinc	1,700	900	500	300	337,000
Cyanide	28	45	7	4	19,300

* % recovery of spiked samples is given in the parenthesis.

THIS DATA UNCORRECTED FOR RECOVERY

EBMUD-OAKLAND

Table 11-13 Priority Pollutants in EBMUB, Oakland POTW

	Sewage	Primary Effluent	Secondary Effluent	Secondary Effluent Chlorinated	Sludge
DICHLORODIFLUOROMETHANE				3.5	
CHLOROETHANE	0.89				
TRICHLOROFLUOROMETHANE	34.5				
1,1-DICHLOROETHENE	1.8				1.1
DICHLOROMETHANE	9.0	1.6	4.6	7.3	0.24
1,1-DICHLOROETHANE	1.7		1.6	1.9	2.3
TRICHLOROMETHANE	37.5	15.7	19.1	34.2	0.5
TRICHLOROETHENE	11.2	6.2	21.1	65.1	
1,2-DICHLOROETHANE	0.46		2.0	6.0	
TETRACHLOROMETHANE	0.59				
1,1,1-TRICHLOROETHANE	88.4	11.7	26.9	36.3	
BENZENE	10.5	3.1			12.4
1,2-DICHLOROPROPANE			1.2		
BROMODICHLOROMETHANE	1.5			0.89	
METHYLBENZENE	112.4	13.7	0.95	0.99	126.2
TETRACHLOROETHENE	49.3	12.4	10.5	25.2	
CHLOROBENZENE	177.3	15.1	0.17	0.46	22.8
ETHYLBENZENE	148.3	34.5	0.33	0.81	160.6
TRIBROMOMETHANE		3.1	1.3		7.6
1,1,2,2-TETRACHLOROETHANE		2.6			NO
$Cl\text{-}C_1$	83.8	20.4	25.0	42.4	8.3
$Cl\text{-}C_{2,3}$	241.4	82.1	62.8	138.4	2.9
Cl-AROM.	438	65.4	6.6	2.3	309.6

THID DATA UNCORRECTED FOR RECOVERY

Table 11-13 Cont. EBMUD-OAKLAND

	Sewage	Primary Effluent	Secondary Effluent	Secondary Effluent Chlorinated	Sludge
N-NITROSODIMETHYLAMINE			0.40		
BIS(2-CHLOROETHYL)ETHER	0.12	0.70	<0.01	0.01	
1,3-DICHLOROBENZENE	0.22	1.29	0.41		5.5
1,4-DICHLOROBENZENE	1.52	6.88	2.75	0.30	37.0
1,2-DICHLOROBENZENE	1.78	5.90	1.34	0.23	15.8
BIS(2-CHLOROISOPROPYL)ETHER			0.27	0.23	
N-NITRO-DI-N-PROPYL AMINE	6.73		0.10		
NITROBENZENE	0.29	1.97			
BIS92-CHLOROETHOXY)METHANE					
1,2,4-TRICHLOROBENZENE			0.32		13.5
NAPHTHALENE	6.85	10.70			76.0
ACENAPHTHYLENE		0.27			
DIMETHYLPHTHALATE	0.38	0.31	NQ	0.05	0.75
2,6-DINITROTOLUENE	2.61	1.35			
ACENAPHTHENE	0.28			2.87	
FLUORENE	0.57	0.58			5.8
DIETHYLPHTHALATE	9.77	5.18		0.11	
DIPHENYLHYDRAZINE	NQ			0.02	0.01
N-NITROSODIPHENYLAMINE	1.28	0.70			
PHENATHRENE	1.21	1.43			27.3
ANTHRACENE	0.05				NQ
DI-N-BUTYLPHTHALATE	11.07	12.87	5.99	4.90	1.8
FLUORANTHENE	3.10	3.48			42.31
PYRENE	0.80	1.52			24.8
BUTYLBENZYLPHTHALATE	126.34	154.7		0.38	
BENZO(A)ANTHRACENE					
CHRYSENE	0.66	1.42			14.0
BIS(2-ETHYLEXYL)PHTHALATE	275.0	309.5		3.35	7429.
DI-N-OCTYL PHTHALATE	10.60	10.79			237.0
BENZO(B)FLUORANTHENE	NQ				NQ
BENZO(K)FLUORANTHENE	0.38	0.77			8.0
BENZO(A)PYRENE	0.08	NQ			4.8
ETHERS	0.12	0.70	0.28	0.26	
AMINES	8.01	0.70	0.50	0.02	0.01
BENZENES	6.42	17.4	4.8	0.53	71.8
AROMATIC HYDROCARBONS	14.74	21.6			223.5
PHTHALATES	433.1	493.4	6.0	8.8	313.84

THIS DATA UNCORRECTED FOR RECOVERY

Table 11-13 Cont. EBMUD-OAKLAND

Compound	Sewage	Primary Effluent	Secondary Effluent	Secondary Effluent Chlorinated	Sludge
LINDANE	0.414	0.572	0.155	0.139	0.122
Δ-BHC				0.867	
HEPTACHLOR EPOXY	4.974	0.645	0.405		
DIELDRIN	0.050	0.123			0.149
p,p,'-DDD	0.290	0.201	0.151		0.273
p,p'-DDT					0.550
METHOXYCHLOR	0.632	0.550			
MIREX					0.021
α-ENDOSULFAN	0.277	0.515	0.039		0.386
ϒ-ENDOSULFAN	0.228		0.141		0.403
ENDOSULFAN SULFATE					1.327
CHLORDENE	1.141	1.636	0.060	0.071	0.101
ϒ-CHLORDANE	0.510	1.152	0.221		0.72
α-CHLORDANE	0.444..	0.692			
PESTICIDES	8.96	6.09	1.17	1.08	4.05

Compound	Sewage	Primary Effluent	Secondary Effluent	Secondary Effluent Chlorinated	Sludge
PHENOL	164.9				
2-CHLOROPHENOL		479.3			
2,4-DIMETHYLPHENOL	58.2	42.6			
2,4,6-TRICHLOROPHENOL	14.3	12.1			
PHENOLICS	237.4	534			

THIS DATA UNCORRECTED FOR RECOVERY

Table 11-13 Cont. EBMUD, Oakland California

Inorganic Priority Pollutants (ppb)

Metal	3-1 (PS 180)	3-2 (PS 190)	3-3 (PS 200)	3-4 (PS 210)	3-5 (PS 220)
Antimony	3.8	2.5	2.5	<1	120
Arsenic	13.8	13.8	11.3	11.3	1,375
Beryllium	<1	<1	<1	<1	14
Cadmium	34	14	<1	<1	3,000
Chromium	390	155	41.5	66.5	1,900
Copper	140	130	140	70	15,000
Lead	96	58.4	13	25	4,000
Mercury	17.5	10	13	10	275
Nickel	640	460	290	152.5	35,000
Selenium	<1	<1	<1	<1	85
Silver	9.3	11	2.1	<1	250
Thallium	<1	<1	<1	<1	<1
Zinc	1650	850	150	200	120,000
Cyanide	126	112	65	77	

Table 11-14 Summary of Priority Pollutants Found in 3 POTW'S

DICHLORODIFLUOROMETHANE
TRICHLOROFLUOROMETHANE
DICHLOROMETHANE
TRICHLOROMETHANE
TETRACHLOROMETHANE
BROMODICHLOROMETHANE
TRIBROMOMETHANE

1,2-DICHLOROPROPANE
BENZENE
METHYLBENZENE
CHLOROBENZENE
ETHYLBENZENE

CHLOROETHANE
1,1-DICHLOROETHENE
1,1-DICHLOROETHANE
TRICHLOROETHENE
1,2-DICHLOROETHANE
1,1,1-TRICHLOROETHANE
TETRACHLOROETHENE
1,1,2,2-TETRACHLOROETHANE

BIS(2-CHLOROETHYL)ETHER
BIS(2-CHLOROISOPROPYL)ETHER
BIS(2-CHLOROETHOXY)METHANE
N-NITROSODIMETHYLAMINE
N-NITROSO-DI-N-PROPYL AMINE
DIPHENYLHYDRAZINE
N-NITROSODIPHENYLAMINE
1,3-DICHLOROBENZENE
1,4-DICHLOROBENZENE
1,2-DICHLOROBENZENE
NITROBENZENE
1,2,4-TRICHLOROBENZENE
2,6-DINITROTOLUENE

NAPHTHALENE
ACENAPHTHYLENE
ACENAPTHENE
FLUORENE
PHENANTHRENE
ANTHRACENE
FLUORANTHENE
PURENE
BENZO(A)ANTHRACENE
CHRYSENE
BENZO(B)FLUORANTHENE
BENZO(K)FLUORANTHENE
BENZO(A)PYRENE
DIMETHYLPHTHALATE
DIETHYLPHTHALATE
DIBUTYLPHTHALATE
BUTYLBENZYLPHTHALATE
BIS(2-ETHYLHEXYL)PHTHALATE
DI-N-OCTYL PHTHALATE

α-BHC
LINDANE
β-BHC
ΔBHC
HEPTACHLOR
HEPTACHLOR EPOXY
DIELDRIN
p,p-DDE
p,p'-DDD
p,p'-DDT
METHOXYCHLOR
MIREX
CHLORDANE α,γ
CHLORDENE
ENDOSULFAN SULFATE
ENDOSULFAN α,β

4-CHLORO-2-METHYL PHENOL
2-NITROPHENOL
4,6-DINITRO-2-METHYL PHENOL
PENTACHLOROPHENOL
2,4-DIMETHYLPHENOL
2,4-DICHLOROPHENOL
PHENOL
2-CHLOROPHENOL
2,4,6-TRICHLOROPHENOL

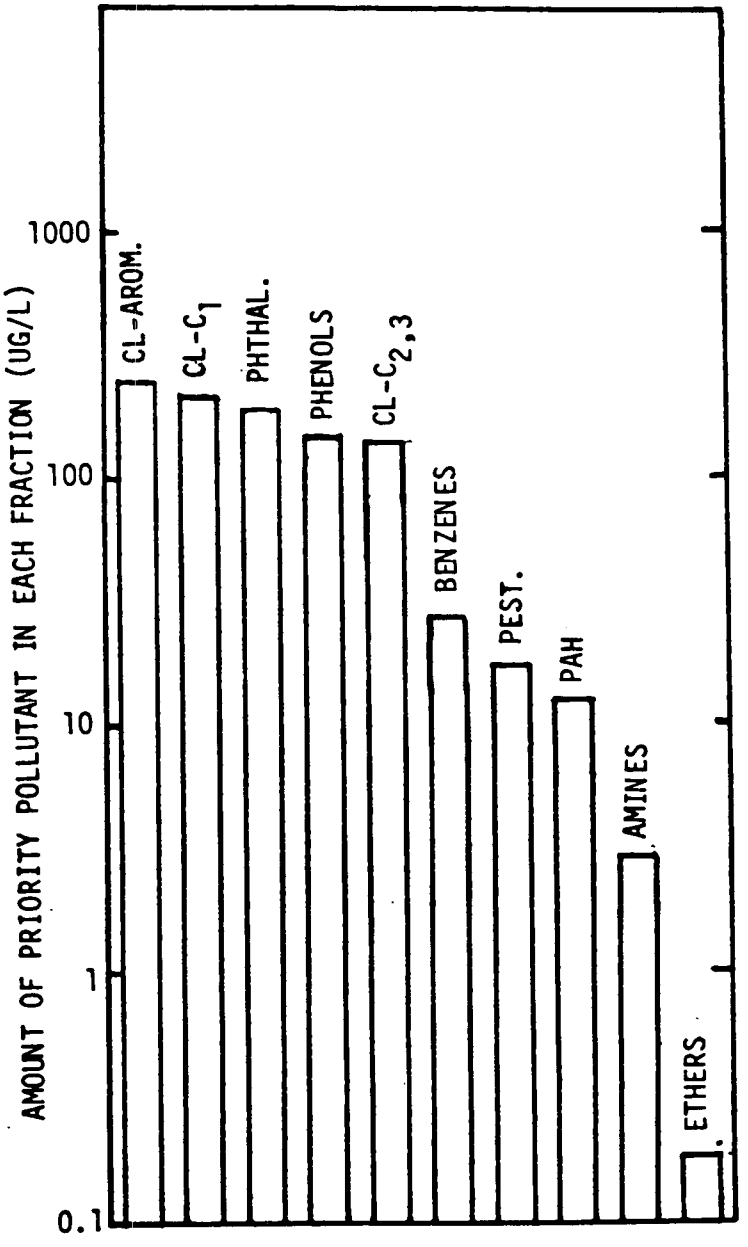


Figure 11-3 Representation of Compunds Found in Different Fractions

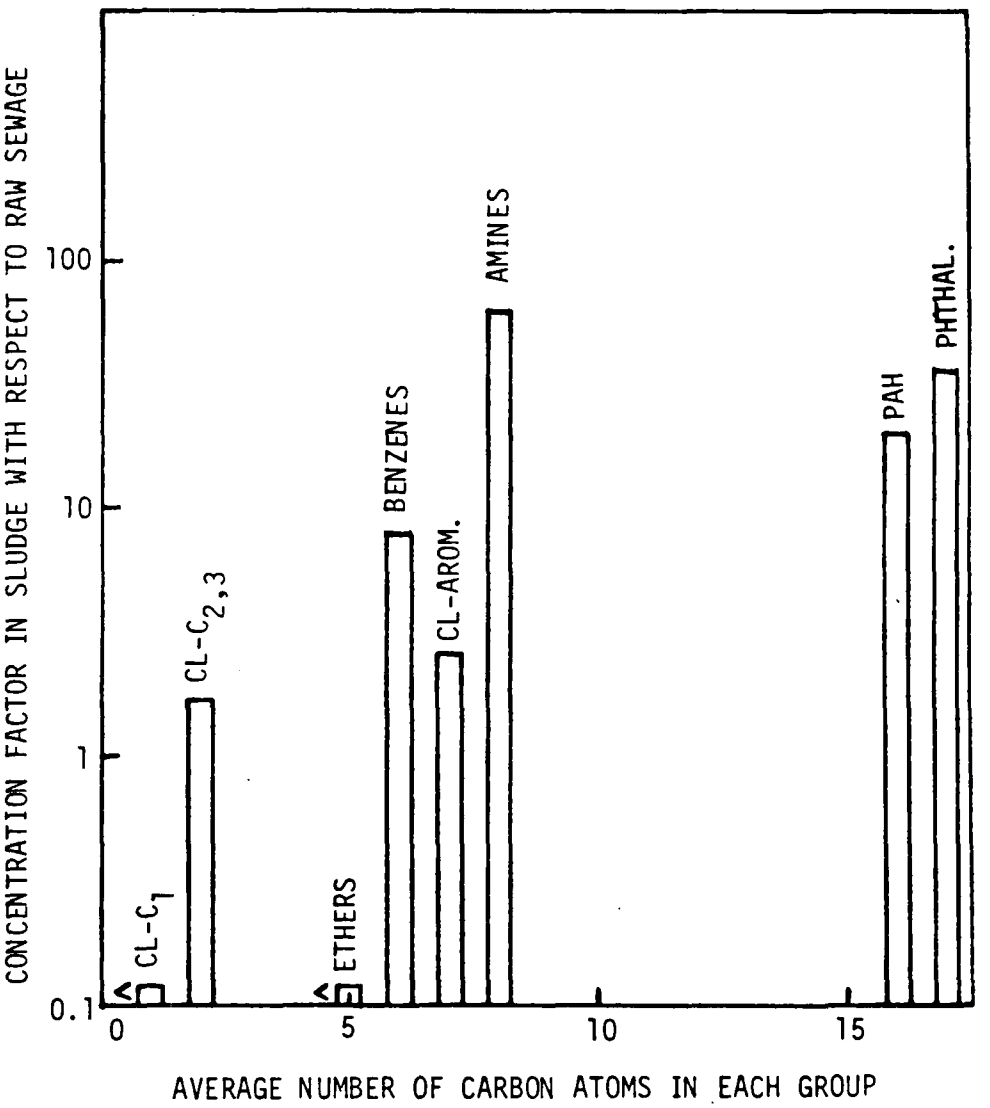


Figure 11-4 Bioaccumulation of Compounds in Different Fractions

SECTION 12

MUTAGENIC ACTIVITY OF POTW SAMPLES

12-1 Introduction

Two sewage samples, i.e. digested sludge (PS140) and secon-effluent before chlorination (PS 170) from the Clayton, Atlanta plant, each consisting of six different fractions, were examined for mutagenic activity by *in vitro* microbiological assays with five strains of *Salmonella typhimurium* (TA1535, TA1537, TA1538, TA98, and TA100). An Aroclor 1254-stimulated rat-liver homogenate metabolic activation system was included in the assay procedure to provide metabolic steps that the bacteria either are incapable of conducting or do not carry out under the assay conditions. The purpose of this study was to determine whether the samples elicited a mutagenic response in microorganisms.

The assay procedure with *S. typhimurium* has proven to be 85 to 90% reliable in detecting carcinogens as mutagens, and it has about the same reliability in identifying chemicals that are not carcinogenic (McCann et. al., 1975).

12-2 Methods

The *Salmonella typhimurium* strains used in this study are TA1535, TA1537, TA1538, TA98, and TA100. These *Salmonella typhimurium* strains are all histidine auxotrophs by virtue of mutations in the histidine operon. When these histidinedependent cells are grown on a minimal media petri plate containing a trace of histidine, only those cells that revert to histidine independence (his+) are able to form colonies. The small amount of histidine allows all the plated bacteria to undergo a few divisions; in many cases, this growth is essential for mutagenesis to occur. The his+ revertants, resulting from their mutation in the presence of the organic mutagen, are easily counted as colonies against the slight background growth. The spontaneous mutation frequency of each strain is relatively constant, but when a mutagen is added to the agar, the mutation frequency is increased 2 - 100 fold.

Our *S. typhimurium* strains were obtained indirectly from Dr. Bruce Ames of the University of California at Berkeley via Dr. Kristien Mortelmans of SRI International in Menlo Park, California. In addition to having mutations in the histidine operon, all the indicator strains have a mutation (rfa-) that leads to a defective lipopolysaccaride coat; they also have a deletion that covers genes involved in the synthesis of biotin (bio-) and in the repair of ultraviolet(uv)-induced DNA damage (uvrB-). The rfa- mutation makes the strains more permeable to many large aromatic molecules, thereby increasing the mutagenic effect of these molecules. The uvrB- mutation decreases repair of some types of

chemically or physically damaged DNA and thereby enhances the strains' sensitivity to some mutagenic agents. Strain TA1535 is reverted to his+ by many mutagens that cause base-pair substitutions. TA100 is derived from TA1535 by the introduction of the resistance transfer factor plasmid pKM101. This plasmid is believed to cause an increase in error-prone DNA repair that leads to many more mutations for a given dose of most mutagens. TA100 can detect mutagens such as benzyl chloride and 2-(2-furyl)-3-(5-nitro-2-furyl)acrylamide (AF2), which are not detected by TA1535. The presence of this plasmid also makes strain TA100 sensitive to some frameshift mutagens such as benzo-(a)pyrene, aflatoxin B1, and 7,12-dimethylbenz(a)anthracene. Strains TA1537 and TA1538 are reverted by many frameshift mutagens. Strain TA98 is derived from TA1538 by the addition of the plasmid pKM101, which makes it more sensitive to some mutagenic agents.

All the indicator strains are routinely checked for their genotypic characteristics. For each experiment, an inoculum from the stock cultures is grown overnight at 37°C in nutrient broth. After stationary overnight growth, the cultures are shaken for 3 hours to ensure optimal growth.

The Aroclor 1254-simulated metabolic activation system was used in the present study to activate the organics in each fraction. Some carcinogenic chemicals, either of the aromatic amino type or polycyclic hydrocarbon type, are inactive unless they are metabolized to active forms. In animals and man, an enzyme system in the liver or other organs (e.g., lung or kidney) is capable of metabolizing a large number of these chemicals to carcinogens. Some of these intermediate metabolites are very potent mutagens in the S. typhimurium test. Ames has described the liver metabolic activation system that we use (Ames et. al., 1975). The metabolic activation mixture for each experiment consists of, for 10 ml:

1.00 ml of S-9 fraction of the rat liver homogenate
0.20 ml of $MgCl_2$ (0.4M) and KCl (1.65M)
0.05 ml of glucose-6-phosphate (1M)
0.40 ml of NADP (0.1M)
5.00 ml of sodium phosphate (0.2M, pH 7.4)
3.35 ml of H_2O

Assays in Agar

The assays in agar are conducted in the following manner: to a sterile 13x100 mm test tube placed in a 43°C heating block, the following are added:

1) 2.00 ml of 0.6% agar containing a trace of histidine and biotin
2) 0.05 ml of indicator organism
3) 0.50 ml of metabolic activation mixture (optional)
4) 0.05 ml of a solution of the test sample

For negative controls, 0.05 ml of the solvent used for the test sample is added. For positive controls, specific mutagens known to revert each strain are used.

The mixture is stirred gently and then poured onto minimal agar plates. After the top agar has set, the plates are incubated at 37 ^{0}C for approximately 48 hours. The number of his+ revertant colonies is counted.

12-3 Results and Discussion

Two sewage samples (140 and 170), each consisting of six fractions, were examined in a set of preliminary experiments using the two most sensitive S. typhimurium strains - TA98 and TA100. Each test vial contained a dry residue of known weight to which 1.0 ml of either dimethyl sulfoxide (DMSO) or water had been added. Tables 12-1 and 12-2 present the results of an experiment in which all the fractions were tested at similar dilutions regardless of their varying residue weights. The volume of each dilution added per plate was consistently 0.05 ml. The highest dose was 0.05 ml of the undiluted fraction. No mutagenic activity was present in these preliminary tests, either in the presence or in the absence of metabolic activation. Although the number of histidine-positive revertants per plate is somewhat higher than that observed on the control plates, no dose-related increase is evident. Toxicity, however, is clearly present at the higher doses of the 140 series of fractions. While all fractions of the 140 sample produced some signs of killing, the toxic effect was most pronounced with the PS140A3S (phenols) and PS140A2 +A3 (neutrals) fractions. In the 170 series of fractions, toxicity was present only at the highest dose of PS170 (water from the extractor).

Tables 12-3 and 12-4 present the results of a subsequent experiment, in which the dose range for some of the fractions was narrowed in order to better focus on those dose levels immediately preceding a toxic effect. With fractions in which no toxicity had been observed in the previous experiment, the highest dose was increased by adding more volume of the undiluted test solution to the plate. Only S. typhimurium strains TA98 and TA100 were used. No mutagenic response was observed with any fraction. Toxicity, however, was evident with all the fractions in the absence of metabolic activation.

Table 12-1

IN VITRO ASSAYS WITH SALMONELLA TYPHIMURIUM - 140 Series

Compound	Metabolic Activation	Micrograms of Compound Added per Plate	Histidine-Positive Revertants per Plate TA98	TA100
Negative Control	- *	0	15,19	121,138
	+	0	28,36	126,124
DMSO Control (dimethyl sulfoxide)	-	50,000	20,18	123, 103
	+	50,000	32,25	110, 117
Water Control	-	50,000	22,19	130,117
	+	50,000	30,23	142,140
Positive Controls:				
Sodium Azide	-	1.0		542
2-Nitrofluorene	-	10.0	960	
2- Anthramine	-	2.5	29	133
	+	2.5	356	1150

* - = in the absence of metabolic activation, + = in the presence of metabolic activation

Table 12-1 (cont)

IN VITRO ASSAYS WITH SALMONELLA TYPHIMURIUM - 140 Series

Compound	Metabolic Activation	Percent Conc. of Solution Added per Plate	Volume of Solution Added per Plate	µg of Residue Added per Plate	His+ Revertants per Plate TA98	TA100
PS140B (unfractionated base extract)	-	0.5	0.5 ml.	4.6	20	136
	-	1.0	0.5	9.2	18	120
	-	5.0	0.5	46.0	19	121
	-	10.0	0.5	92.0	25	107
	-	20.0	0.5	184.0	10	107
	-	100.0	0.5	920.0	11 + pp.	85 + pp.
	+	0.5	0.5	4.6	44	172
	+	1.0	0.5	9.2	25	148
	+	5.0	0.5	46.0	34	170
	+	10.0	0.5	92.0	38	194
	+	20.0	0.5	184.0	39	183
	+	100.0	0.5	920.0	41	99
PS140 (water from extractor)	-	0.5	0.5	0.0	35	201
	-	1.0	0.5	0.0	18	124
	-	5.0	0.5	0.0	16	152
	-	10.0	0.5	0.0	24	122
	-	20.0	0.5	0.0	16	197
	-	100.0	0.5	0.0	9 + pp.	92
	+	0.5	0.5	0.0	34	142
	+	1.0	0.5	0.0	31	152
	+	5.0	0.5	0.0	45	177
	+	10.0	0.5	0.0	35	204
	+	20.0	0.5	0.0	35	207
	+	100.0	0.5	0.0	32 + pp.	173

* pp. = pinpoints, which is a sign of toxicity

Table 12-1 (cont.)

IN VITRO ASSAYS WITH SALMONELLA TYPHIMURIUM - 140 Series

Compound	Metabolic Activation	Percent Conc. of Solution Added per Plate	Volume of Solution Added per Plate	µg of Residue Added per Plate	His^+ Revertants per Plate TA98	TA100
PS140A2 +	-	0.5	0.5 ml.	2.5	10	133
PS140 A3	-	1.0	0.5	5.0	16	110
(neutrals)	-	5.0	0.5	25.0	8	79 + pp.
	-	10.0	0.5	50.0	16	100 + pp.
	-	20.0	0.5	100.0	13 + pp.	74
	-	100.0	0.5	500.0	6 + pp.	0
	+	0.5	0.5	2.5	41	158
	+	1.0	0.5	5.0	37	133
	+	5.0	0.5	25.0	29	126
	+	10.0	0.5	50.0	32	124
	+	20.0	0.5	100.0	20	142
	+	100.0	0.5	500.0	16 + pp.	130
PS140A3S	-	0.5	0.5	0.8	19	124
(phenols)	-	1.0	0.5	1.5	26	173
	-	5.0	0.5	7.5	20	138
	-	10.0	0.5	15.0	13	57
	-	20.0	0.5	30.0	1 + pp.	0
	-	100.0	0.5	150.0	0	1
	+	0.5	0.5	0.8	23	137
	+	1.0	0.5	1.5	35	134
	+	5.0	0.5	7.5	42	142
	+	10.0	0.5	15.0	42	124
	+	20.0	0.5	30.0	27	140
	+	100.0	0.5	150.0	0	11

* pp. = pinpoints, which is a sign of toxicity

Table 12-1 (cont.)

IN VITRO ASSAYS WITH SALMONELLA TYPHIMURIUM - 140 Series

Compound	Metabolic Activation	Percent Conc. of Solution Added per Plate	Volume of Solution Added per Plate	µg of Residue Added per Plate	His+ Revertants per Plate TA98	TA100
PS140A	-	0.5	0.05 ml.	96.3	18	182
(unfractionated	-	1.0	0.05	192.5	24	107
acid/neutral	-	5.0	0.05	962.5	18	77
extract)	-	10.0	0.05	1925.0	14 + pp.	126
	-	20.0	0.05	3850.0	4 + pp.	39 + pp.
	-	100.0	0.05	19250.0	pp.	20 + pp.
	+	0.5	0.05	96.3	38	147
	+	1.0	0.05	192.5	37	135
	+	5.0	0.05	962.5	40	155
	+	10.0	0.05	1925.0	36	189
	+	20.0	0.05	3850.0	30	164
	+	100.0	0.05	19250.0	35	129
PS140A1	-	0.5	0.05	8.3	15	131
(throw away	-	1.0	0.05	16.5	23	157
fraction of	-	5.0	0.05	82.5	21	150
acid/neutral	-	10.0	0.05	165.0	29	122
extract)	-	20.0	0.05	330.0	18	158
	-	100.0	0.05	1650.0	22	105 + pp.
	+	0.5	0.05	8.3	41	165
	+	1.0	0.05	16.5	43	129
	+	5.0	0.05	82.5	25	166
	+	10.0	0.05	165.0	35	165
	+	20.0	0.05	330.0	38	165
	+	100.0	0.05	1650.0	19 + pp.	150

* pp. = pinpoints, which is a sign of toxicity

Table 12-2

IN VITRO ASSAYS WITH SALMONELLA TYPHIMURIUM - 170 Series

Compound	Metabolic Activation	Micrograms of Compound Added per Plate	Histidine-Positive Revertants per Plate TA98	TA100
Negative Control	- *	0	15,19	121,138
	+	0	28,36	126,124
DMSO Control (dimethyl sulfoxide)	-	50,000	20,18	123, 103
	+	50,000	32,25	110, 117
Water Control	-	50,000	22,19	130,117
	+	50,000	30,23	142,140
Positive Controls: Sodium Azide	-	1.0		542
2-Nitrofluorene	-	10.0	960	
2-Anthramine	-	2.5	29	133
	+	2.5	356	1150

* - = in the absence of metabolic activation, + = in the presence of metabolic activation

Table 12-2 (cont.)

IN VITRO ASSAYS WITH SALMONELLA TYPHIMURIUM - 170 Series

Compound	Metabolic Activation	Percent Conc. of Solution Added per Plate	Volume of Solution Added per Plate	µg of Residue Added per Plate	His^+ Revertants per Plate TA98	TA100
PS170B	-	0.5	0.5 ml.	3.8	23	183
(unfractionated	-	1.0	0.5	7.5	30	143
Base)	-	5.0	0.5	37.5	21	123
	-	10.0	0.5	75.0	16	159
	-	20.0	0.5	150.0	19	152
	-	100.0	0.5	750.0	15	187
	+	0.5	0.5	3.8	26	157
	+	1.0	0.5	7.5	25	102
	+	5.0	0.5	37.5	33	163
	+	10.0	0.5	75.0	31	194
	+	20.0	0.5	150.0	33	179
	+	100.0	0.5	750.0	33	143
PS170	-	0.5	0.5	0.0	22	137
(water from	-	1.0	0.5	0.0	30	123
extractor)	-	5.0	0.5	0.0	18	137
	-	10.0	0.5	0.0	16	126
	-	20.0	0.5	0.0	20	135
	-	100.0	0.5	0.0	1	0
	+	0.5	0.5	0.0	47	201
	+	1.0	0.5	0.0	46	203
	+	5.0	0.5	0.0	36	186
	+	10.0	0.5	0.0	38	163
	+	20.0	0.5	0.0	37	161
	+	100.0	0.5	0.0	0	161 + pp.*

* pp. = pinpoints, which is a sign of toxicity

Table 12-2 (cont.)

IN VITRO ASSAYS WITH SALMONELLA TYPHIMURIUM - 170 Series

Compound	Metabolic Activation	Percent Conc. of Solution Added per Plate	Volume of Solution Added per Plate	µg of Residue Added per Plate	His^+ Revertants per Plate TA98	TA100
PS170A2 + PS170A3 (neutrals)	-	0.5	0.05 ml.	0.5	20	129
	-	1.0	0.05	1.0	19	123
	-	5.0	0.05	5.0	24	154
	-	10.0	0.05	10.0	31	150
	-	20.0	0.05	20.0	17	152
	-	100.0	0.05	100.0	22	96
	+	0.5	0.05	0.5	29	169
	+	1.0	0.05	1.0	58	158
	+	5.0	0.05	5.0	53	149
	+	10.0	0.05	10.0	50	163
	+	20.0	0.05	20.0	37	168
	+	100.0	0.05	100.0	40	142
PS170A3S (phenols)	-	0.5	0.05	1.0	22	184
	-	1.0	0.05	2.0	29	145
	-	5.0	0.05	10.0	20	162
	-	10.0	0.05	20.0	25	160
	-	20.0	0.05	40.0	22	161
	-	100.0	0.05	200.0	32	155
	+	0.5	0.05	1.0	35	127
	+	1.0	0.05	2.0	41	156
	+	5.0	0.05	10.0	44	149
	+	10.0	0.05	20.0	43	168
	+	20.0	0.05	40.0	38	208
	+	100.0	0.05	200.0	42	198

Table 12-2 (cont)

IN VITRO ASSAYS WITH SALMONELLA TYPHIMURIUM - 170 Series

Compound	Metabolic Activation	Percent Conc. of Solution Added per Plate	Volume of Solution Added per Plate	µg of Residue Added per Plate	His^+ Revertants per Plate TA98	TA100
PS170A (unfractionated acid/neutral extract)	-	0.5	0.05 ml.	12.5	27	124
	-	1.0	0.05	25.0	17	126
	-	5.0	0.05	125.0	23	154
	-	10.0	0.05	250.0	18	136
	-	20.0	0.05	500.0	19	134
	-	100.0	0.05	2500.0	29	165
	+	0.5	0.05	12.5	39	197
	+	1.0	0.05	25.0	43	131
	+	5.0	0.05	125.0	32	155
	+	10.0	0.05	250.0	47	130
	+	20.0	0.05	500.0	45	163
	+	100.0	0.05	2500.0	45	140
PS170A1 (throw away from acid/ neutral)	-	0.5	0.05	0.0	21	173
	-	1.0	0.05	0.0	24	149
	-	5.0	0.05	0.0	30	175
	-	10.0	0.05	0.0	29	178
	-	20.0	0.05	0.0	22	149
	-	100.0	0.05	0.0	28	150
	+	0.5	0.05	0.0	59	136
	+	1.0	0.05	0.0	54	147
	+	5.0	0.05	0.0	40	130
	+	10.0	0.05	0.0	43	137
	+	20.0	0.05	0.0	28	120
	+	100.0	0.05	0.0	21	142

Table 12-3

IN VITRO ASSAYS WITH SALMONELLA TYPHIMURIUM - 140 Series

Compound	Metabolic Activation	Micrograms of Compound Added per Plate	Histidine-Positive Revertants per Plate TA98	TA100
Negative control	- *	0	23,13	163,169
	+	0	44,40	144,144
DMSO control (dimethyl sulfoxide)	-	50,000	26,29	177,152
	+	50,000	39,45	163,178
Water control	-	50,000	22,18	176,178
	+	50,000	47,50	178,178
Positive controls:				
Sodium azide	-	1.0		488
2-Nitrofluorene	-	10.0	1113	
2-Anthramine	-	2.5	31	178
	+	2.5	481	497

* - = in the absence of metabolic activation, + = in the presence of metabolic activation

Table 12-3 (cont.)

IN VITRO ASSAYS WITH SALMONELLA TYPHIMURIUM - 140 Series

Compound	Metabolic Activation	Percent Conc. of Solution Added per Plate	Volume of Solution Added per Plate	µg of Residue Added per Plate	His^+ Revertants per Plate TA98	TA100
PS140B (unfractionated base extract)	-	2.0	0.5 ml.	18.4	28	88
	-	5.0	0.5	46.0	35	116
	-	10.0	0.5	92.0	32	121
	-	20.0	0.5	184.0	31	101
	-	50.0	0.5	460.0	23	103
	-	100.0	0.5	920.0	22 + pp.*	67 + pp.
	+	2.0	0.5	18.4	40	172
	+	5.0	0.5	46.0	34	172
	+	10.0	0.5	92.0	49	159
	+	20.0	0.5	184.0	48	144
	+	50.0	0.5	460.0	54	148
	+	100.0	0.5	920.0	48	147
PS140 (water from extractor)	-	2.0	0.5	0.0	29	124
	-	5.0	0.5	0.0	28	157
	-	10.0	0.5	0.0	31	131
	-	20.0	0.5	0.0	31	142
	-	50.0	0.5	0.0	33	162
	-	100.0	0.5	0.0	17 + pp.	93 + pp.
	+	2.0	0.5	0.0	55	178
	+	5.0	0.5	0.0	45	157
	+	10.0	0.5	0.0	49	149
	+	20.0	0.5	0.0	35	168
	+	50.0	0.5	0.0	32	171
	+	100.0	0.5	0.0	43	139 + pp.

* pp. = pinpoints, which is a sign of toxicity

Table 12-3 (cont.)

IN VITRO ASSAYS WITH SALMONELLA TYPHIMURIUM - 140 Series

Compound	Metabolic Activation	Percent Conc. of Solution Added per Plate	Volume of Solution Added per Plate	µg of Residue Added per Plate	His^+ Revertants per Plate TA98	TA100
PS140A2 +	-	0.1	0.5 ml.	0.7	21	144
PS140A3	-	0.2	0.5	1.0	24	136
(neutrals)	-	0.4	0.5	2.0	30	150
	-	1.0	0.5	5.0	32	109
	-	4.0	0.5	20.0	19	105
	-	20.0	0.5	100.0	21	185
	+	0.1	0.5	0.7	46	187
	+	0.2	0.5	1.0	48	141
	+	0.4	0.5	2.0	42	182
	+	1.0	0.5	5.0	56	138
	+	4.0	0.5	20.0	55	156
	+	20.0	0.5	100.0	44	107
PS140A3S	-	0.2	0.5	0.3	28	168
(phenols)	-	1.0	0.5	1.5	27	152
	-	2.0	0.5	3.0	27	178
	-	4.0	0.5	6.0	28	121
	-	10.0	0.5	15.0	21	89
	-	20.0	0.5	30.0	pp.	pp.
	+	0.2	0.5	0.3	48	170
	+	1.0	0.5	1.5	51	116
	+	2.0	0.5	3.0	48	162
	+	4.0	0.5	6.0	38	171
	+	10.0	0.5	15.0	37	143
	+	20.0	0.5	30.0	45	148

*pp. = pinpoints, which is a sign of toxicity

Table 12-3 (cont.)

IN VITRO ASSAYS WITH SALMONELLA TYPHIMURIUM - 140 Series

Compound	Metabolic Activation	Percent Conc. of Solution Added per Plate	Volume of Solution Added per Plate	µg of Residue Added per Plate	His^+ Revertants per Plate TA98	TA100
PS140A	-	0.2	0.5 ml.	38.0	22	134
(unfractionated	-	1.0	0.5	192.5	25	110
acid/neutral	-	2.0	0.5	385.0	30	95
extract)	-	10.0	0.5	1925.0	31	100
	-	50.0	0.5	9625.0	17	19
	-	100.0	0.5	19250.0	6	31
	+	0.2	0.5	38.0	50	199
	+	1.0	0.5	192.5	47	158
	+	2.0	0.5	385.0	57	155
	+	10.0	0.5	1925.0	57	119
	+	50.0	0.5	9625.0	28	100
	+	100.0	0.5	19250.0	20	75
PS140A1	-	5.0	0.5	82.5	27	123
(throw-away	-	10.0	0.5	165.0	22	109
fraction of	-	20.0	0.5	330.0	19	87
acid/neutral)	-	50.0	0.5	825.0	20	98
	-	100.0	0.5	1650.0	19	78
	-	100.0	1.0	3300.0	20	72 + pp.
	+	5.0	0.5	82.5	33	161
	+	10.0	0.5	165.0	50	171
	+	20.0	0.5	330.0	41	115
	+	50.0	0.5	825.0	41	114
	+	100.0	0.5	1650.0	42	118
	+	100.0	1.0	3300.0	37	117

*pp. = pinpoints, which is a sign of toxicity

Table 12-4

IN VITRO ASSAYS WITH SALMONELLA TYPHIMURIUM - 179 Series

Compound	Metabolic Activation	Micrograms of Compound Added per Plate	Histidine-Positive Revertants per Plate TA98	TA100
Negative control	-*	0	23,13	163,169
	+	0	44,40	144,144
DMSO control (dimethyl sulfoxide)	-	50,000	26,29	177,152
	+	50,000	39,45	163,178
Water control	-	50,000	22,18	176,178
	+	50,000	47,50	178,178
Positive controls:				
Sodium azide	-	1.0		488
2-Nitrofluorene	-	10.0	1113	
2-Anthramine	-	2.5	31	178
	+	2.5	481	497

* - = in the absence of metabolic activation, + = in the presence of metabolic activation

Table 12-4 (cont.)

IN VITRO ASSAYS WITH SALMONELLA TYPHIMURIUM - 170 Series

Compound	Metabolic Activation	Percent Conc. of Solution Added per Plate	Volume of Solution Added per Plate	µg of Residue Added per Plate	His+ Revertants per Plate TA98	TA100
PS170B	-	20.0	0.05 ml.	150.0	32	163
(unfractionated	-	50.0	0.05	375.0	23	145
base extract)	-	100.0	0.05	750.0	37	124
	-	100.0	0.10	1500.0	20 + pp.*	0**
	-	100.0	0.15	2250.0	3	-
	+	20.0	0.05	150.0	42	157
	+	50.0	0.05	375.0	44	172
	+	100.0	0.05	750.0	45	147
	+	100.0	0.10	1500.0	-	-
	+	100.0	0.15	2250.0	-	-
PS170	-	2.0	0.05	0.0	24	156
(water from	-	5.0	0.05	0.0	22	124
extractor)	-	10.0	0.05	0.0	25	86
	-	20.0	0.05	0.0	23	115
	-	50.0	0.05	0.0	15 + pp.	89
	-	100.0	0.05	0.0	1	12 + pp.
	+	2.0	0.05	0.0	58	145
	+	5.0	0.05	0.0	57	162
	+	10.0	0.05	0.0	43	137
	+	20.0	0.05	0.0	41	138
	+	50.0	0.05	0.0	35 + pp.	148
	+	100.0	0.05	0.0	16 + pp.	95 + pp.

*pp. = pinpoints, which is a sign of toxicity

** - indicates that no plate count is available at the specific dose listed. This is due to the limited volume of sample.

Table 12-4 (cont.)

IN VITRO ASSAYS WITH SALMONELLA TYPHIMURIUM - 170 Series

Compound	Metabolic Activation	Percent Conc. of Solution Added per Plate	Volume of Solution Added per Plate	µg of Residue Added per Plate	His^+ Revertants per Plate TA98	TA100
PS170A2 +	-	10.0	0.05 ml.	10.0	3	190
PS170A3	-	20.0	0.05	20.0	7	152
(neutrals)	-	50.0	0.05	50.0	14	163
	-	100.0	0.05	100.0	19	137
	-	100.0	0.10	200.0	19 + pp.*	152
	-	100.0	0.20	400.0	-**	7
	+	10.0	0.05	10.0	29	166
	+	20.0	0.05	20.0	42	131
	+	50.0	0.05	50.0	49	127
	+	100.0	0.05	100.0	24	156
	+	100.0	0.10	200.0	37	123 + pp.
	+	100.0	0.20	400.0	-	-
PS170A3S	-	20.0	0.05	40.0	31	136
(phenols)	-	50.0	0.05	100.0	20	140
	-	100.0	0.05	200.0	20	155
	-	100.0	0.10	400.0	27	51
	-	100.0	0.15	600.0	-	6
	+	20.0	0.05	40.0	41	156
	+	50.0	0.05	100.0	45	176
	+	100.0	0.05	200.0	39	151
	+	100.0	0.10	400.0	-	-
	+	100.0	0.15	600.0	-	-

*pp. = pinpoints, which is a sign of toxicity

** - indicates that no plate count is available at the specific dose listed. This is due to the limited volume of sample.

Table 12-4 (cont.)

IN VITRO ASSAYS WITH SALMONELLA TYPHIMURIUM - 170 Series

Compound	Metabolic Activation	Percent Conc. of Solution Added per Plate	Volume of Solution Added per Plate	µg of Residue Added per Plate	His+ Revertants per Plate TA98	TA100
PS170B (unfractionated base extract)	-	20.0	0.05 ml.	150.0	32	163
	-	50.0	0.05	375.0	23	145
	-	100.0	0.05	750.0	37	124
	-	100.0	0.10	1500.0	20 + pp.*	0**
	-	100.0	0.15	2250.0	3	-
	+	20.0	0.05	150.0	42	157
	+	50.0	0.05	375.0	44	172
	+	100.0	0.05	750.0	45	147
	+	100.0	0.10	1500.0	-	-
	+	100.0	0.15	2250.0	-	-
PS170 (water from extractor)	-	2.0	0.05	0.0	24	156
	-	5.0	0.05	0.0	22	124
	-	10.0	0.05	0.0	25	86
	-	20.0	0.05	0.0	23	115
	-	50.0	0.05	0.0	15 + pp.	89
	-	100.0	0.05	0.0	1	12 + pp.
	+	2.0	0.05	0.0	58	145
	+	5.0	0.05	0.0	57	162
	+	10.0	0.05	0.0	43	137
	+	20.0	0.05	0.0	41	138
	+	50.0	0.05	0.0	35 + pp.	148
	+	100.0	0.05	0.0	16 + pp.	95 + pp.

*pp. = pinpoints, which is a sign of toxicity

** - indicates that no plate count is available at the specific dose listed. This is due to the limited volume of sample.

Table 12-4 (cont.)

IN VITRO ASSAYS WITH SALMONELLA TYPHIMURIUM - 170 Series

Compound	Metabolic Activation	Percent Conc. of Solution Added per Plate	Volume of Solution Added per Plate	µg of Residue Added per Plate	His^+ Revertants per Plate TA98	TA100
PS170A	-	20.0	0.05 ml.	500.0	20	138
(unfractionated	-	50.0	0.05	1250.0	29	169
acid/neutral	-	100.0	0.05	2500.0	36	164
extract)	-	100.0	0.10	5000.0	17 + pp.*	110
	-	100.0	0.15	7500.0	pp.	82 + pp.
	+	20.0	0.05	500.0	48	208
	+	50.0	0.05	1250.0	40	183
	+	100.0	0.05	2500.0	41	144
	+	100.0	0.10	5000.0	-**	-
	+	100.0	0.15	7500.0	-	-
PS170A1	-	20.0	0.05	0.0	29	147
(throw-away from	-	50.0	0.05	0.0	26	162
acid/neutral	-	100.0	0.05	0.0	23	124
extract)	-	100.0	0.10	0.0	7 + pp.	90
	-	100.0	0.15	0.0	-	4
	+	20.0	0.05	0.0	36	161
	+	50.0	0.05	0.0	33	145
	+	100.0	0.05	0.0	44	128
	+	100.0	0.10	0.0	-	-
	+	100.0	0.15	0.0	-	-

* pp. = pinpoints, which is a sign of toxicity.

** - indicates that no plate count is available at the specific dose listed. This is due to the limited volume of sample.

The results of an experiment using all five strains of S. typhimurium -- TA 1535, TA1537, TA1538, TA98, and TA100 -- are shown in Tables 12-5 and 12-6. Only three of the six fractions of each sample were tested. However, these three fractions contain all the potential mutagenic pollutants in the sewage sample. They are the fractions PS140A and PS170A (the unfractionated acid/neutral extract), PS140B and PS170B (the base extract), and PS140 and PS170 (the water from the extractor). The dose ranges were varied in order to best utilize the limited volumes of sample, while at the same time reaching for the highest doses. In some cases, only four doses were used. No mutagenic activity was evident with any of the strains, in the presence or in the absence of metabolic activation.

None of the experiments conducted indicate that the fractions of the sludge sample 140, or the fractions of the secondary effluent sample 170, are mutagenic in the standard Ames Salmonella/microsome procedure, either with or without metabolic activation.

Because of the toxicity at higher doses, it was difficult to determine if these fractions possessed mutagenic activity at very high doses. However, the fact that bacterial toxicity was evident is of interest in itself. An evaluation of the cytotoxic actions of these samples is shown in Figures 12-1 and 12-2 and correlate the amount of residue weight (dose) with the decrease in spontaneous revertants. The data show that the bacterial toxicity of the fractions did qualitatively demonstrate a log dose-response relationship for both TA98 and TA100 strains of S. typhimurium. A critical dose of about 10 ug of residue per plate for the phenol fraction in digested sludge was suggested from this plot (Figure 12-1). Other fractions which produced bacterial toxicity, observed a lesser decrease in the number of spontaneous revertants over the control. The least toxic was the A-1 throw away fraction containing the lipids. A different picture emerged for the secondary effluent (Figure 12-2) where the non-extractable organics containing fulvic and humic substances were the most toxic followed, as in the sludge, by the neutrals and phenols. The unfractionated extract was the least toxic, possibly indicating that the extracted compound exerts less effect in a complex mixture than do the individual fractions. While the current study is primarily concerned with the purgeable and extractable organics, the non-extractable organics may in fact be more toxic and should receive further study.

Table 12-5

IN VITRO ASSAYS WITH SALMONELLA TYPHIMURIUM - 140 Series

Compound	Metabolic Activation *	Micrograms of Residue Added per Plate	Histidine Revertants per Plate TA1535	TA1537	TA1538	TA98	TA100
Negative control	-	0	22,17	4,4	11,9	24,21	115,121
	+	0	14,19	6,6	24,26	30,26	116,113
DMSO control (dimethyl sulfoxide)	-	50,000	16	6	12	25	121
	+	50,000	20	11	13	31	106
Water control	-	50,000	31	4	14	23	129
	+	50,000	18	9	16	28	110
Positive controls:							
Sodium azide	-	1.0	312				595
9-Aminoacridine	-	100.0		1022			
2-Nitrofluorene	-	10.0			472	967	
2- Anthramine	-	2.5	21	18	15	30	156
	+	2.5	759	939	876	868	903

* - = in the absence of metabolic activation, + = in the presence of metabolic activation

Table 12-5 (cont.)

IN VITRO ASSAYS WITH SALMONELLA TYPHIMURIUM - 140 Series

Compound	Metabolic Activation	Micrograms of Residue Added per Plate	Histidine Revertants per Plate TA1535	TA1537	TA1538	TA98	TA100
PS140A (unfractionated acid/neutral extract)	-	19.3	11	5	13	31	110
	-	38.5	20	4	12	18	138
	-	192.5	25	5	18	25	116
	-	385.0	30	6	9	19	85 + pp.*
	-	1925.0	22	6	17	21	89
	-	9625.0	31	6	14	15	39
	+	19.3	10	6	12	25	109
	+	38.5	18	8	18	21	123
	+	192.5	14	6	21	26	105
	+	385.0	12	7	18	24	122
	+	1925.0	7	8	19	30	133
	+	9625.0	5	8	17	18	40
PS140B (unfractionated base extract)	-	16.4	10	6	6	38	136
	-	41.0	12	4	9	25	126
	-	82.0	14	5	6	21	137
	-	164.0	12	4	10	30	98
	-	410.0	12	9	8	24	151
	-	820.0	14	10	18	21	96
	+	16.4	16	9	23	29	141
	+	41.0	12	7	22	30	99
	+	82.0	16	7	23	25	101
	+	164.0	15	8	22	23	95
	+	410.0	5	6	34	18	96
	+	820.0	7	12	19	21	106

* pp. = pinpoints, which is a sign of toxicity

Table 12-5 (cont.)

IN VITRO ASSAYS WITH SALMONELLA TYPHIMURIUM - 140 Series

Compound	Metabolic Activation	Micrograms of Residue Added per Plate	Histidine Revertants per Plate TA1535	TA1537	TA1538	TA98	TA100
PS140	-	0.0 (2.0%)*	9	6	12	34	120
(Water from extractor)	-	0.0 (5.0%)	11	5	7	28	105
	-	0.0 (10.0%)	18	10	19	21	80
	-	0.0 (20.0%)	12	5	14	31	130
	-	0.0 (50.0%)	10	3	15	25	69
	+	0.0	8	9	17	28	94
	+	0.0	10	7	21	25	81
	+	0.0	9	8	15	22	96
	+	0.0	10	8	21	** 19	64
	+	0.0	5	5	17 + pp.	23	85

* The percent concentration of solution added per plate is listed in the parentheses. In each case, 0.05 ml. was the volume added.

** pp. = pinpoints, which is a sign of toxicity

Table 12-6

IN VITRO ASSAYS WITH SALMONELLA TYPHIMURIUM - 170 Series

Compound	Metabolic Activation	Micrograms of Residue Added per Plate	Histidine Revertants per Plate				
			TA1535	TA1537	TA1538	TA98	TA100
Negative control	-*	0	22,17	4,4	11,9	24,21	115,121
	+	0	14,19	6,6	24,26	30,26	116,113
DMSO control (dimethyl sulfoxide)	-	50,000	16	6	12	25	121
	+	50,000	20	11	13	31	106
Water control	-	50,000	31	4	14	23	129
	+	50,000	18	9	16	28	110
Positive controls:							
Sodium azide	-	1.0	312				595
9-Aminoacridine	-	100.0		1022			
2-Nitrofluorene	-	10.0			472	967	
2-Anthramine	-	2.5	21	18	15	30	156
	+	2.5	759	939	876	868	903

* - = in the absence of metabolic activation, + = in the presence of metabolic activation

Table 12-6 (cont.)

IN VITRO ASSAYS WITH SALMONELLA TYPHIMURIUM - 170 Series

Compound	Metabolic Activation	Micrograms of Residue Added per Plate	Histidine Revertants per Plate TA1535	TA1537	TA1538	TA98	TA100
PS170A	-	250.0	19	5	18	30	115
(unfractionated	-	625.0	27	10	9	28	112
acid/neutral	-	1250.0	39	8	5	21	105
extract)	-	2500.0	30 + pp.*	0	5 + pp.	0	109
	+	250.0	13	9	34	35	114
	+	625.0	8	12	21	24	107
	+	1250.0	11	12	25	30	105
	+	2500.0	15	5	20	21	103
PS170B	-	75.0	38	4	7	24	93
(unfractionated	-	188.0	24	10	9	28	103
base extract)	-	375.0	31	8	9	20	89
	-	750.0	36	pp.	8 + pp.	7 + pp.	0
	+	75.0	11	7	26	23	88
	+	188.0	18	7	20	31	94
	+	375.0	22	10	31	35	98
	+	750.0	17	4	13	28	115

* pp. = pinpoints, which is a sign of toxicity

Table 12-6 (cont.)

IN VITRO ASSAYS WITH SALMONELLA TYPHIMURIUM - 170 Series

Compound	Metabolic Activation	Micrograms of Residue Added per Plate	Histidine Revertants per Plate TA1535	TA1537	TA1538	TA98	TA100
PS170	-	0.0 (2.0%)*	18	11	10	28	109
(Water from extractor)	-	0.0 (5.0%)	29	7	15	23	104
	-	0.0 (10.0%)	24	8	10	20	109
	-	0.0 (20.0%)	33	4	12	31	95
	-	0.0 (50.0%)	34	10	5 +	** pp. 18	113
	+	0.0 (2.0%)	40	5	26	35	112
	+	0.0 (5.0%)	22	10	16	34	119
	+	0.0 (10.0%)	14	6	33	28	92
	+	0.0 (20.0%)	18	13	21	30	99
	+	0.0 (50.0%)	26	6	14	21	119

* The percent concentration of solution added per plate is listed in the parentheses. In each case, 0.05 ml. was the volume added.

** pp. = pinpoints, which is a sign of toxicity

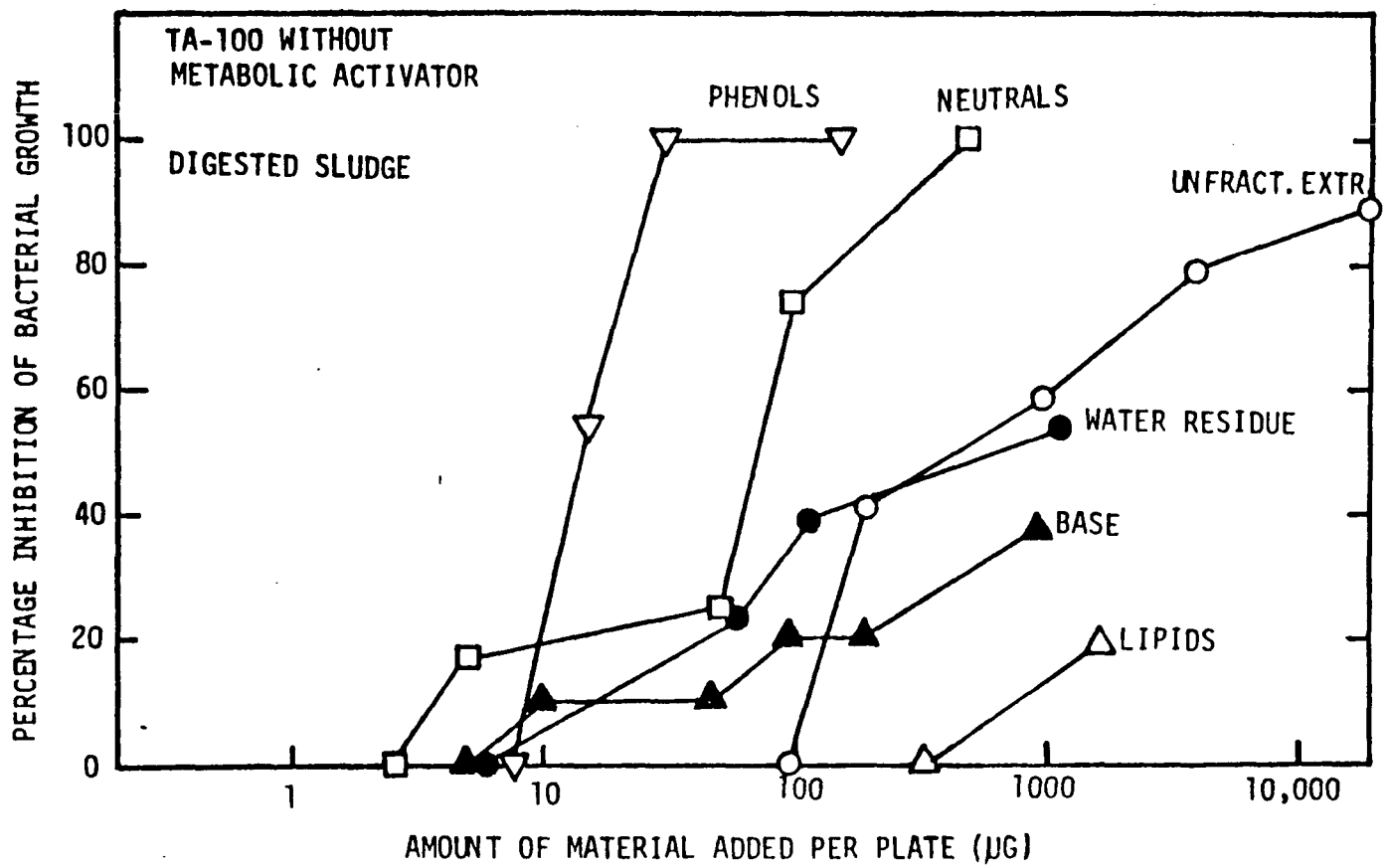


Figure 12-1 Toxicity of Different Fractions Present in Digested Sludge.

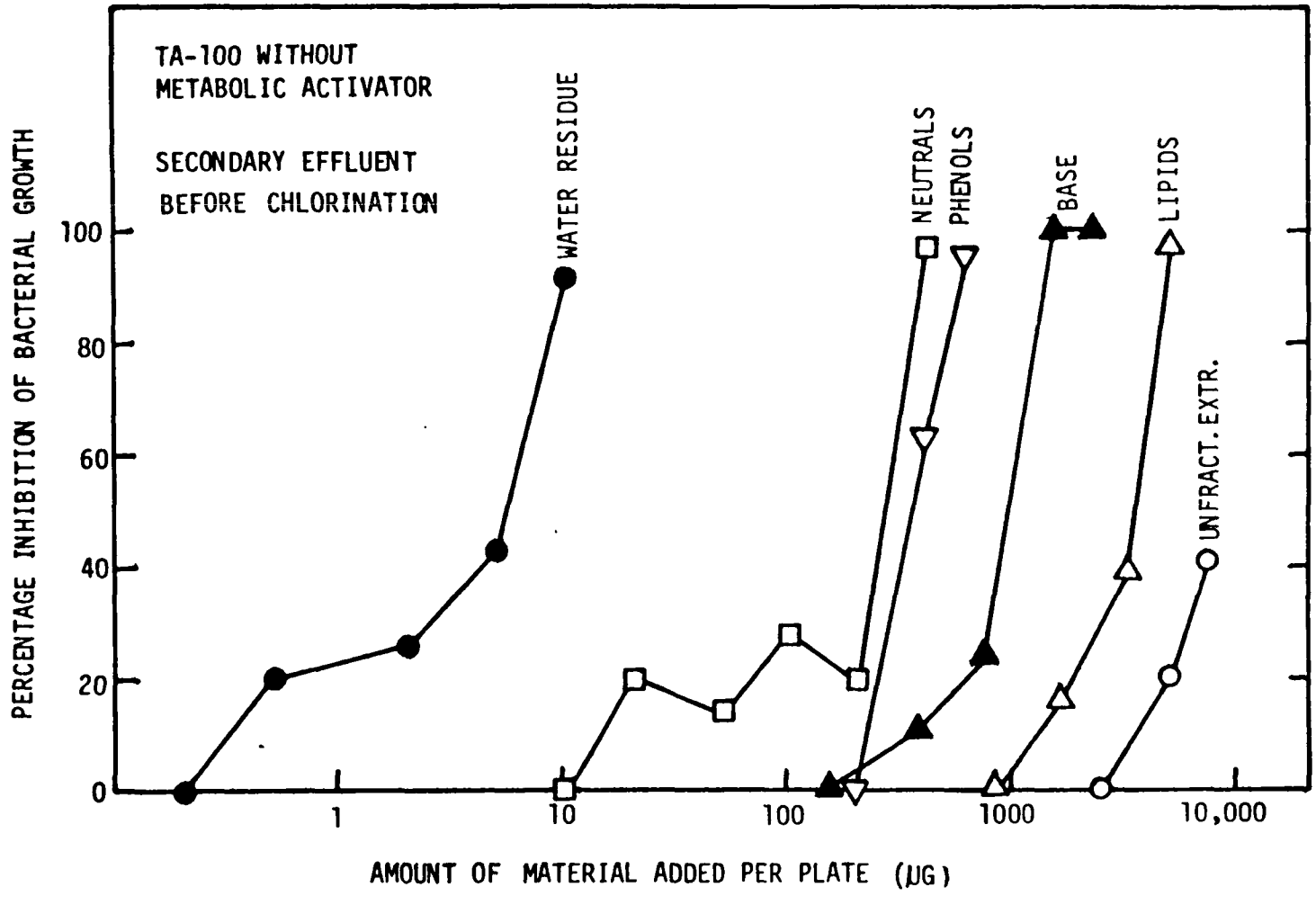


Figure 12-2 Toxicity of Different Fractions Present in Secondary Effluent Before Chlorination.

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Appendix I

Protocols for Integrated Analysis of Priority Pollutants

Method 100 - Purgeable Organics by Purge and Trap/Capillary GC/MS

110 Scope and Application

111 This method allows the determination of low molecular weight organics contained in a water, sewage, or sludge sample. It has also been utilized for sediment samples. Table 100-1 lists the compounds appropriate for this method. Among these, those indicated by an asterisk are poorly retained in the trapping stage and therefore quantitate with a lower sensitivity than commonly seen for the majority of these compounds.

120 Summary

121 This method is a modification of the standard Bellar-Lichtenberg technique developed by EPA. It has been devised using a Hewlett-Packard 7675A Purge and Trap Sampler, but is applicable to any system capable of the purge and trap technique. In this method, the aqueous sample is spiked with a recovery/quantitation standard, diluted if necessary with purgeable-free water, and purged with a stream of organic-free helium. The gas stream is then passed through an inert adsorbent trap at room temperature where the entrained organics are

Consent Decree Number		Compound
		Volatiles
2	*	Acrolein
3	*	Acrylonitrile
4		Benzene
18		Bis(2-chloroethyl)-ether
17		Bischloromethylether
48		Bromodichloromethane
47		Bromoform
6		Carbontetrachloride
7		Chlorobenzene
51		Chlorodibromomethane
16	*	Chloroethane
19		2-chloroethylvinylether
23		Chloroform
25		1,2-Dichlorobenzene
26		1,3-Dichlorobenzene
27		1,4-Dichlorobenzene
13		1,1-Dichloroethane
10		1,2-Dichloroethane
29		1,1-Dichloroethylene
32		1,2-Dichloropropane
33		1,2-Dichloropropylene
50	*	Dichlorodiflouramethane
38		Ethylbenzene
12		Hexachloroethane
46		Methylbromide
45	*	Methylchloride
44		Methylenechloride
15		1,1,2,2-Tetrachloroethane
85		Tetrachloroethylene
86		Toluene
30		1,2-Transdichloroethylene
11		1,1,1-Trichloroethane
14		1,1,2-Trichloroethane
87		Trichloroethylene
49		Trichloroflouromethane
88	*	Vinylchloride

Table 100-1 Compounds Detected by Capillary GC Purge and Trap Analysis

retained. Upon completion of the purging, the trap is heated and back-flushed with organic-free helium to desorb the trapped organics. This sample stream is passed directly into a capillary GC column where it is cryotrapped at liquid nitrogen temperatures. When all of the sample has been desorbed and cryotrapped, the coolant is turned off and the cryotrap warmed with a flush of hot air. The GC or GC/MS analysis then ensues.

122 This method is recommended for use only by an analyst experienced in purge and trap analysis, or under the close supervision of such a person.

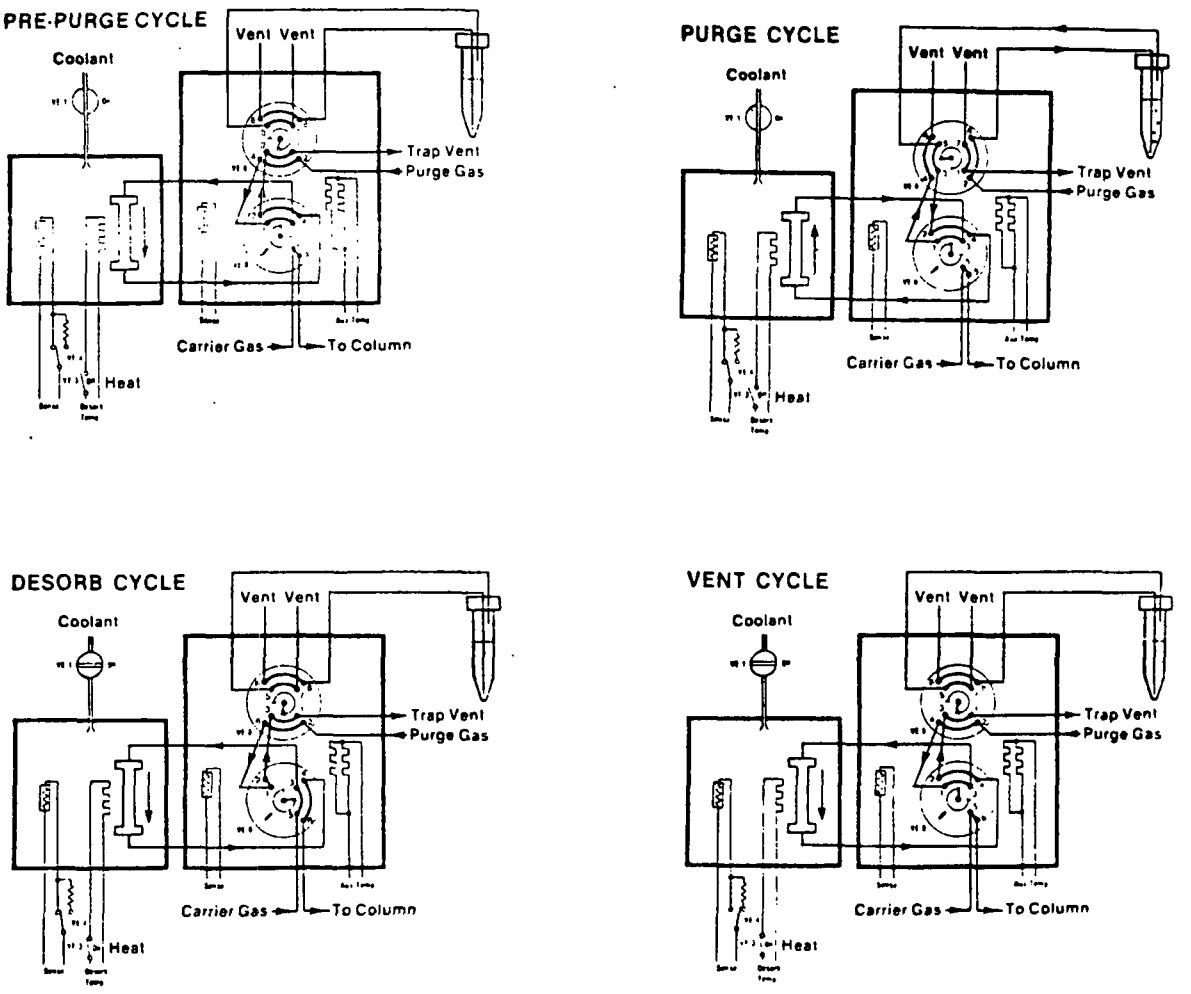
130 Apparatus and Reagents

131 For sample preparation, see section 140.

132 Purging system, Figure 100-1

a. Hewlett-Packard 7675A Purge and Trap Sampler, or equivalent
b. Replacement glass-filled teflon gaskets
c. Steel 1/4" tube traps packed with Tenax-GC (0.4 g) and pre-conditioned at 250 degrees centigrade.

FIGURE 100-1 Purge and Trap System Schematic



133 Glassware -- all purgeable-free and stored in an oven at 150 degrees centigrade prior to use. No chemicals or solvent vapors should be permitted in the oven. No open containers of solvent should be allowed in the room where this analysis is performed.

a. Sample thiefs -- cut off 10 ml pipettes with at least 1 ml graduations showing.
b. Sample tube -- threaded top centrifuge tubes (15, 70 ml)
c. Syringe -- 10 microliters with teflon-tipped plunger

134 Organic-free water and holding vessel, Figure 100-2

a. Magnetic stirrer
b. Teflon stir bar

135 Soap bubble flowmeter, 25 ml

136 Purging gas -- ultrapure helium

a. Catalytic scrubber for gas -- Aadco #173 or equivalent

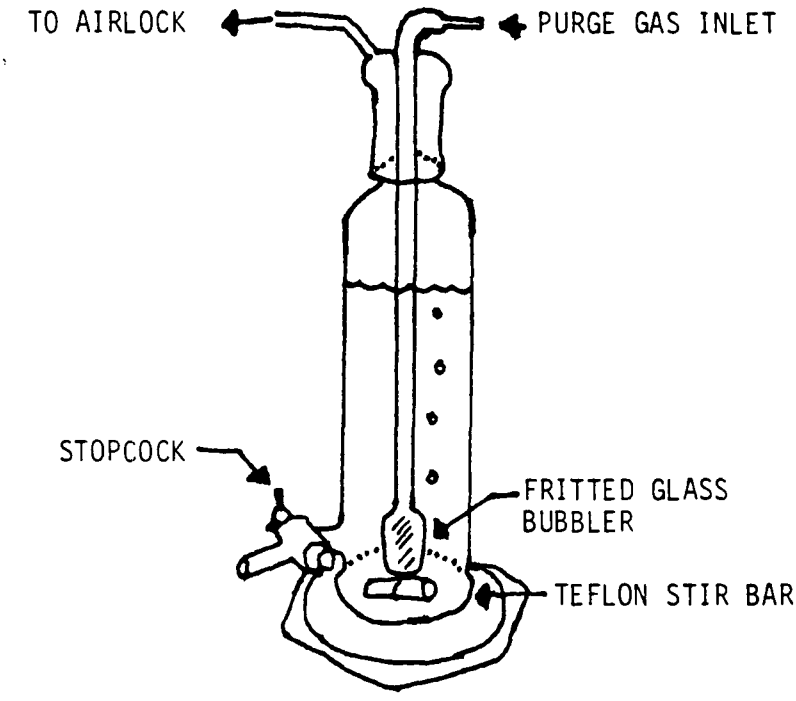


FIGURE 100-2 Vessel For Purgable-Free Water

137 Cryotrap system, Figure 100-3

a. Thin wall stainless steel trap, Figure 100-4
b. Swage or millitorr high vacuum connectors
c. Liquid nitrogen reservoir with pressure delivery

138 GC/MS System

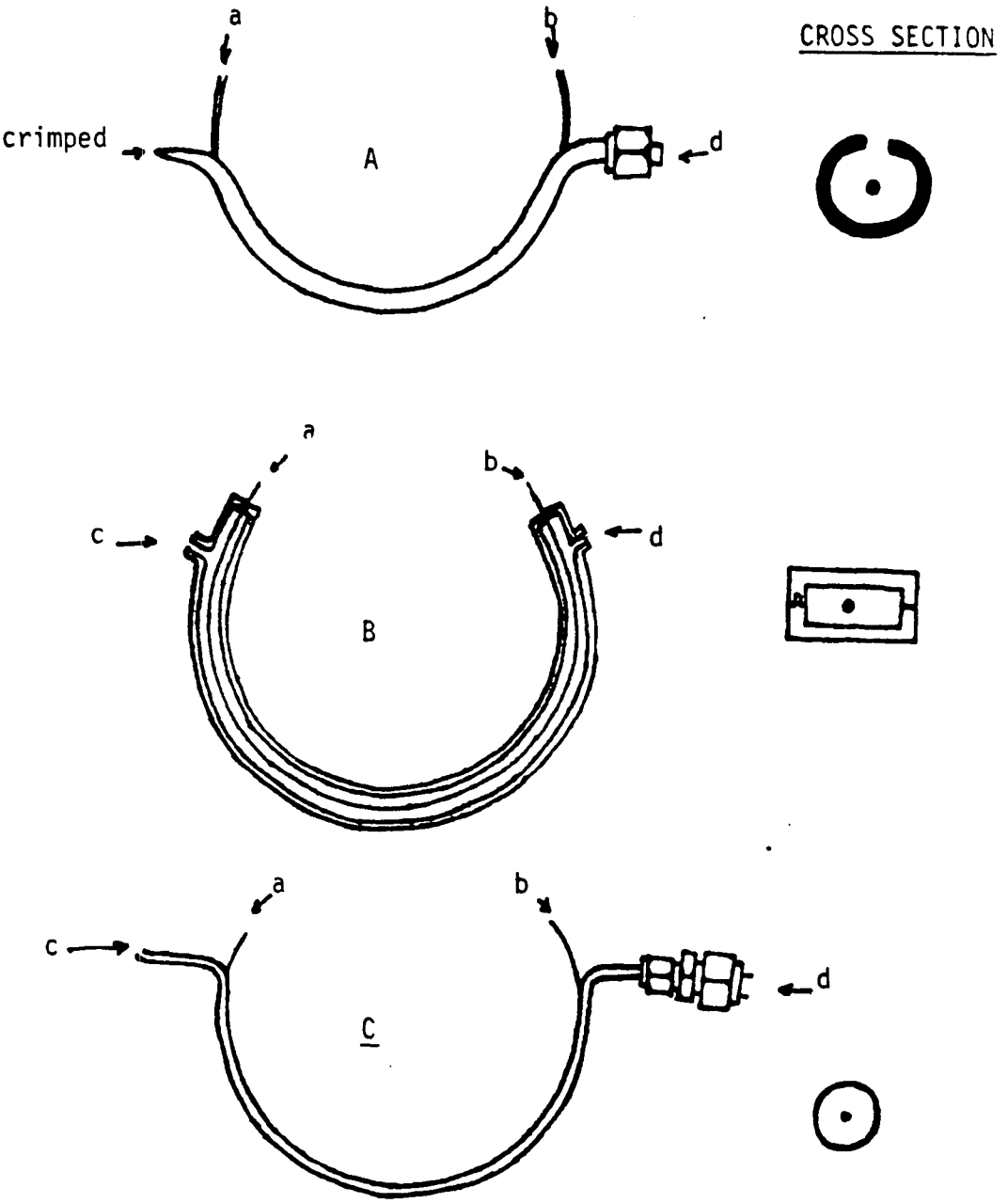
a. Capillary-capable GC with split/split less injector
b. SE-54 (J&W Scientific) 30 M capillary column, or equivalent
c. MS/DS System or FID Detector

139 Recovery/Quantitation Internal Standard

a. Solution in water prepared daily from a methanol stock

140 Sample Preparation and Preservation

141 Sample must be collected in a screw-cap vial with zero headspace, sealed with a teflon cap liner or face. The sample bottle should be muffled in an oven or kiln at 450 degrees for at least 1 hour and then sealed prior



a FROM CAPILLARY INJECTOR
b TO REST OF GC COLUMN, MASS SPECTROMETER
c WARM AIR INLET
d COOLANT INLET (FROM LIQUID NITROGEN RESERVOIR)

Figure 100-4 Cryotrap Designs : A) Notched Tube; B) Aluminum Sandwich; C) Stainless Steel Tube

to use. Caps and teflon liners should be boiled in organic free water immediately prior to sealing the muffled vials.

142 Samples should be refrigerated immediately after collection, stored at 5 degrees centigrade and analyzed prior to the formation of bubbles or within a week, whichever is sooner.

143 Sludge and sediment samples should have a dry weight determination, according to Standard Methods.

150 Sample Purging

151 Prior to an analysis, the Tenax trap must be conditioned and the analytical system shown to be contamination-free. Standard analyses of reference compounds, including the actual priority volatiles, are needed to supply quantitation and chromatographic behavior indices.

152 The Tenax trap should be replaced periodically.

153 The sample tube with 3 ml of organic-free water is shown to be background-free by blank analysis.

154 A recovery spike for that day's analyses is prepared as follows:

a. 50 ml of tested organic-free water is placed in an organic-free 50 ml volumetric.
b. A 10 microliter syringe is rinsed several times with boiling water.
c. After a few minutes cooling, 10 microliters from a 1 mg/ml methanol stock solution is injected into the water. The volumetric is now immediately capped and the spike dispersed with sonication or gentle swirling. This spike is now 2 ng/ul.

155 Using an organic-free sample thief, 1 or 2 ml of mixed sample (with solids resuspended prior to opening) is transferred to the 3 ml organic-free water. The sample is then immediately spiked with 5 ul of the recovery standard using a boiling-water rinsed syringe.

156 The sample is sealed to the purge and trap system. The purge is begun and carried out at a gas flow rate of 20 ml/minute for 15 minutes.

157 Five minutes prior to the end of the purge period, the coolant for the cryotrap is activated to bring the

cryotrap to a stable cold condition prior to initiation of the desorbtion.

158 The desorbtion occurs at a flow rate of 1 ml/minute for 10 minutes, with a trap temperature of 200 degrees centigrade. During this time the splitter should be closed. Typically, the Mass Spectrometer analysis is initiated at the beginning of the desorb cycle in order to detect any failure of the cryotrap.

159 Breakthrough of components of masses higher than 45 during the desorb phase invalidates the analysis.

160 GC/MS/DS Analysis

161 Upon the successful completion of cryotrapping, the coolant flow is stopped and warm air is blown over the column in the reverse direction for 60 seconds. These events should be synchronized with the cessation of desorb flow from the Tenax trap and with the opening of the splitter.

162 The GC analysis is then carried out according to the parameters presented in Table 100-2.

163 The data reduction of raw MS results should utilize the

INSTRUMENT PARAMETERS:		MASS PARAMETERS:	
INTERFACE NUMBER	0	MINIMUM PEAK WIDTH	3
SUB-INTERFACE	0	MIN FRAG WID AS % PEAK WID	80
2ND ACQU INTERFACE	-1	BASELINE TO SUBTRACT	0
SUB-INTERFACE	-1	MINIMUM AREA AFTER MERGE	1
NUMBER OF ACQU BUFFERS	16	MERGE TOL AS % PEAK WIDTH	80
		AREA TOL (DELTA SQRT AREA)	20
INSTRUMENT TYPE	Q	ALIGN TOL AS % PEAK WIDTH	80
FULL SCALE MASS	1024	MAX # OF PEAKS TO READ	2000
ZERO SCALE MASS	1	MASS DEF AT 100 AMU (MMU)	30
INTENSITY/ION	2	NOISE REJ FOR ENHA (0-4 S.D.)	2
ACQUISITION DIRECTION	UP	TAIL REJ FOR ENHA (-1=NONE)	0
SAMPLES/PEAK - CENTROID	10	BKGND MULTIPLIER IN ADD	1.003
SAMPLES/PEAK - FRAGMENT	10	ADD OR AVERAGE IN ADD	AVER
PEAK WIDTH (MMU)	1000.		
OFFSET AT LOW MASS (MMU)	0		
OFFSET AT HIGH MASS (MMU)	0		
VOLTAGE SETTLING TIME(MS)	4		

LOW MASS:	34	UP:	0.45 L*	TOP:	0.00
HIGH MASS:	334	DOWN:	0.00 L	BOTTOM:	0.05
CENT S/P:	10 ACTUAL: 10	SAMP INT (MS):	0.150	PEAK WIDTH:	1000.
FRAG S/P:	10 ACTUAL: 10	SAMP INT (MS):	0.150	INTEN/ION:	2
MIN PEAK WIDTH:	3	MIN FRAG WIDTH %:	80	MIN AREA:	20
A/D THRESHOLD:	2	BASELINE:	0		

MODE: CENTROID POSITIVE ION

GC Parameters

TEMP1	255	30	30
TIME1	14.00		
RATE	2.00		
TEMP2	255	200	
TIME2	10.00		
INJ TEMP	400	0	27
FID TEMP	400	250	250
AUX TEMP	400	250	107
CHT SPD	0.20		
ZERO	5.0		
ATTN 2†	2		
FID SGNL	+8		
SLP SENS	0.50		
AREA REJ		25.	
FLOW A	0.0	48.7	
FLOW B	0.0	0.0	

Table 100-2 Instrumental Conditions for VOA Analysis

internal reference compounds for quantitation and/or recovery standards. The sensitivity of the method will depend on the detection limit for the individual GC/MS system, but ppb analyses should be achievable by using an appropriate sample size. With initial calibration of the instrument in an appropriate concentration range (to give a signal/noise of no less than 50), both quantitation and recovery information can be obtained from the reference standards.

164 Recalibration with standards should be performed frequently, at least every 15 analyses.

170 Control of Contamination

171 Organic-free water is prepared by boiling distilled water under a purge of organic-free gas. The purging system for organic-free water should be equipped with an air lock, or otherwise isolated from the atmosphere. No solvents should be permitted in open containers anywhere in the room where these analyses are performed.

172 Between samples, the purge tube that actually contacts the sample should be heat-gunned or replaced. Care should be taken to avoid introduction of the sample into the lines, or on exposed surfaces of the

instrument or purging system.

173 To document the integrity of the analysis, blank determinations must be made between every pair of real analyses. These water blanks also can be used as dilution water for the following analysis.

174 The gaskets, or other sealing devices used to connect the sample tube, should be inspected for wear or contamination. The effluent flow from the Tenax trap can be monitored during purging to assure a closed system with no pressure losses.

175 The stock solutions used to make the recovery standard should be stored at sub-zero temperatures and protected from losses and contamination.

176 The EPA EMSL recommendations for method verification and quality analysis assurance for volatiles are applicable to this method.

Method 200 -- Purge and Trap Method for Volatile (Alternative)

210 Scope and Application

211 This method covers the determination of various low-molecular weight hydrocarbons, including most of the halogenated C1 and C2 compounds. The complete list of compounds included in this procedure is provided in Table 200-1.

212 This method is applicable to the measurement of these compounds in municipal sewages and sludges. It can be used to meet the monitoring requirement of the National Pollutant Discharge Elimination System (NPDES). As such, it presupposes a high expectation of finding, quantitatively, specific compounds of interest. If the user is attempting to screen samples for all of the compounds in Table 200-1, he should refer to Section 282 for general guidance.

220 Summary

221 The method offers several procedural alternatives, dependent on the availability of specific purging apparatus and the nature and extent of interferences. The

TABLE 200-1 Volatile Low Molecular Weight Compounds Detectable with the Purge and Trap Method

Compound
Cl/Br/Methanes
methylchloride
methylenechloride
chloroform
carbontetrachloride
methylbromide
bromoform
chlorodibromomethane
bromodichloro methane
dichlorodifluoramethane
trichlorofluoromethane
Cl Ethanes
chloroethane
1,1 - dichloroethane
1,2 - dichloroethane
1,1,1 - trichloroethane
1,1,2 - trichloroethane
1,1,2,2 - tetrachloroethane
hexachloroethane
Alkenes
acrolein
acrylonitrile

Compound
Cl Ethylenes, Propane
vinylchloride
1,1 - dichloroethylene
1,2 - transdichloroethylene
trichloroethylene
tetrachloroethylene
1,2 dichloropropane
1,2 dichloropropylene
Ethers
bischloromethylether
2-chloroethylvinylether
bis (2-chloroethyl) ether
bis (2-chloroisopropyl) ether
Cl-Benzenes
benzene
chlorobenzene
1,3-dichlorobenzene
1,2-dichlorobenzene
1,4-dichlorobenzene
toluene
ethylbenzene

procedure describes the use of purging with a purified inert gas, and trapping with a solid adsorbent or a combination of two solid adsorbents to efficiently recover the materials with a minimum of contamination. Chromatographic conditions are suggested for the accurate measurement of the compounds.

222 This method is recommended for use only by an analyst experienced in the analysis of trace organics at ppb level, or under the close supervision of such qualified persons.

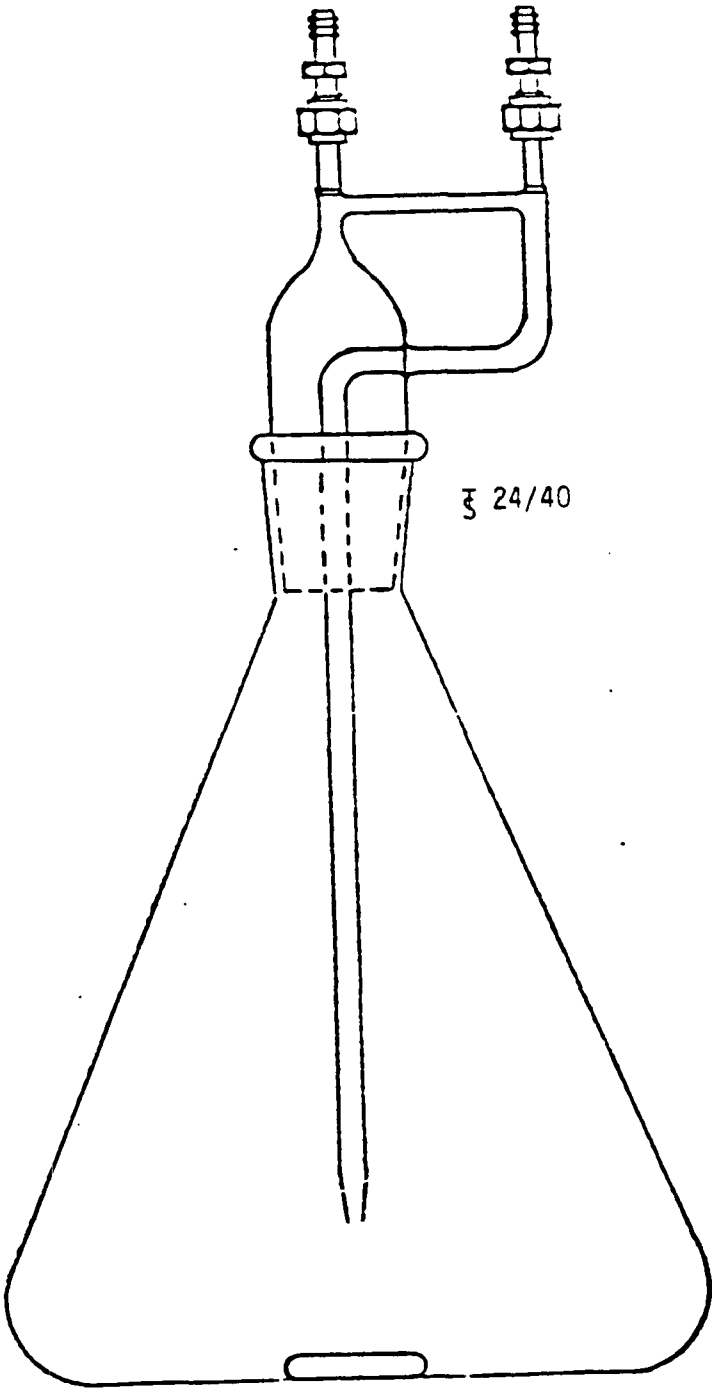
230 Apparatus and Reagents

231 For sample preparation, Section 250

231.1 Purging system, Figure 200-1

a. Erlenmeyer purging flasks -- 1000 ml with 24/40 ground joint
b. Purging head piece with 24/40 ground joint
c. Glass covered magnetic stirring bar (approximately 50 mm long x 6 mm OD).
d. Water bath -- electric frying pan
e. Magnetic stirring table with heater

FIGURE 200-1 1000 ml Erhlenmeyer Purging Flask



f. Thermometer -- glass, 0-100@C

232.2 Trap, Figure 200-2

a. Stainless steel traps (approximately 220 mm long x 6.35 mm OD) containing 0.4 g of Tenax GC, followed by 0.2 g of Chromosorb 102, and marked by an arrow on the swagelok for the direction of gas flow

b. Stainless steel trap guards (approximately 120 mm long x 6.35 mm OD) containing approximately 0.2 g of Tenax GC

c. Stainless steel plugs (6.35 mm)

231.3 Volumetric flasks -- 100 ml with glass stopper

231.4 Purgeable-free water glassware, Figure 200-3

a. Erlenmeyer flasks -- 4000 ml with 40/50 joint

b. Purging head pieces with 40/50 joint

c. Teflon-coated magnetic stirring bar (762 mm long x 12.7 mm)

FIGURE 200-2 Tenax-GC Trap and Guard

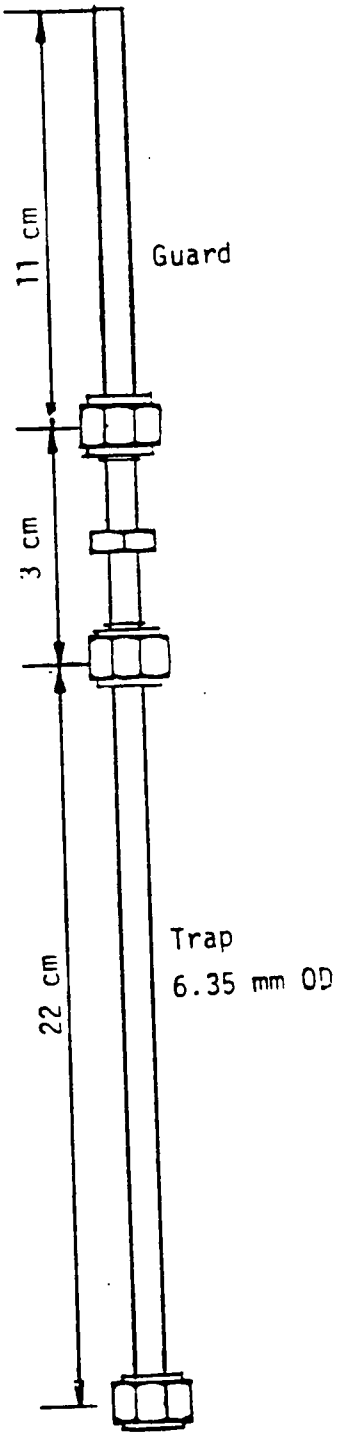
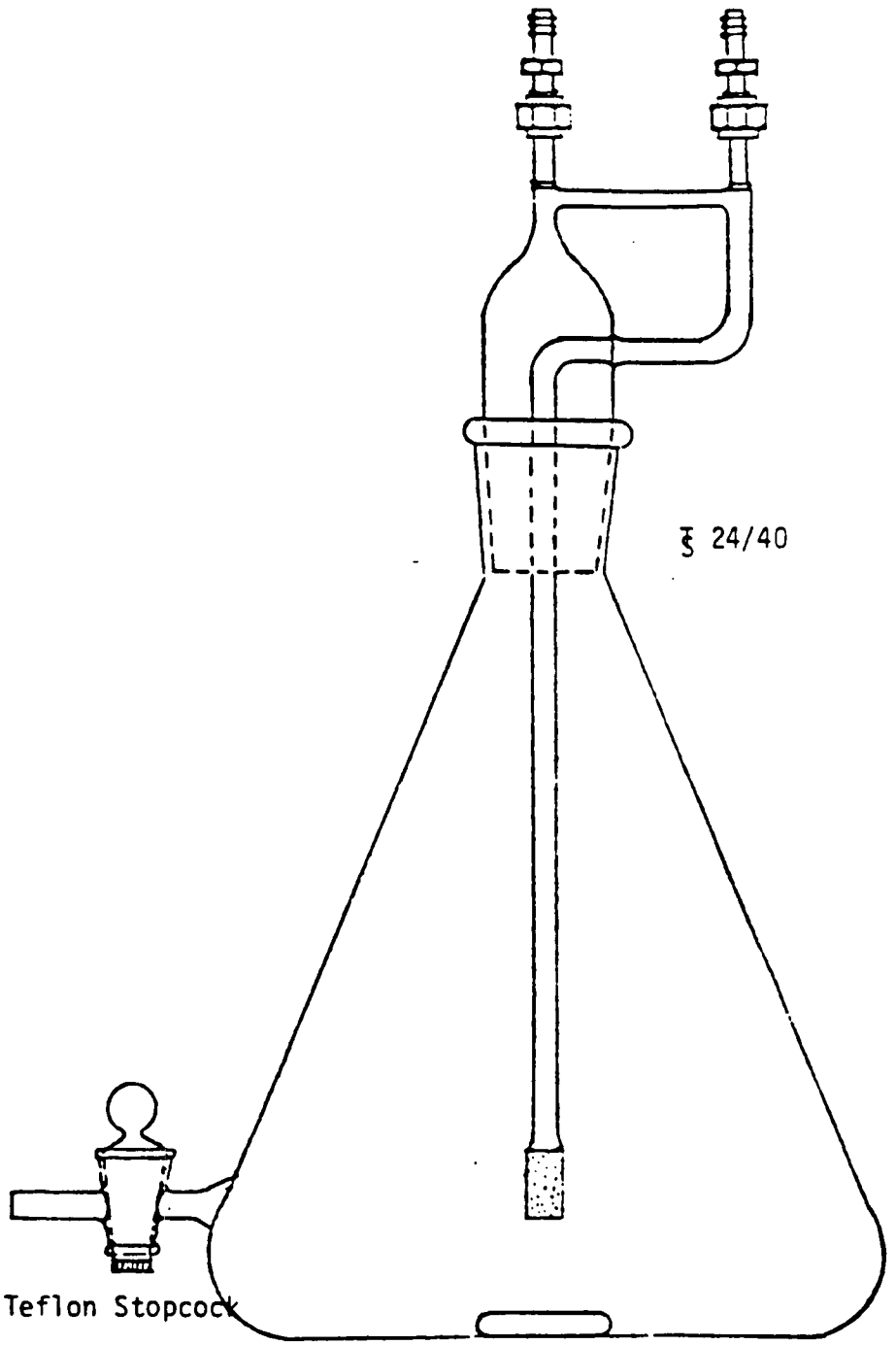


FIGURE 200-3 4-liter Flask for Purgable-Free Water



231.5 Torch -- propane

231.6 Soap bubble flowmeter -- 100 ml

231.7 Springs for fastening the purging vessels

231.8 Purging gas -- N2, water compressed

231.9 Catalytic gas purifier

a. Tubular heater -- 1000^{0}C
b. Quartz tubing (approximately 460 mm long x 19 mm OD)
c. Catalyst for TOC analysis -- Beckman
d. Molecular sieve (approximately 460 mm long x 13 mm OD)

231.10 Rotameter -- 0 to 500 ml/minute of air

231.11 Muffled furnace or pottery kiln -- 450^{0} C

231.12 Trap conditioning oven (drying) -- 275^{0} C

231.13 Glass tubing (approximately 610 mm long x 25 mm OD) with one end sealed and packed with 10 mm thick wool at the end

231.14 Screw cap septum vials with Teflon-lined septum: 7 ml, 25 ml, 40 ml

231.15 Aluminum tares

231.16 Distilled water

232 For quantitation, Section 260

232.1 Gas chromatograph -- equipped with a 4-way valve gas injection system. The recommended detectors and columns are discussed in Section 260.

232.2 Heating tape (approximately 500 mm long)

232.3 Portable tubular heater (approximately 250 mm x 25 mm ID) -- 250°C

232.4 Variac

232.5 Recorder -- potentiometer strip chart (10 inch) compatible with the recorder. Preferable with the microdata processor for integrating detector readings.

232.6 Reference materials -- assay quantity of compound(s) of interest, and purgeable-free water, Section 250.

240 Sampling and Preservation

241 Sample must be collected in screw cap vials with zero head space and sealed with an inert liner (teflon) incorporated into the septum. The number of samples required is discussed in Procedures 272 and 281. The vials should be muffled at 450 ^{0}C overnight and the septum pre-washed with methanol, boiled in distilled water, and dried at 103^{0}C to minimize contaminations and loss of volatiles. Conventional sampling practices should be followed to collect representative samples.

242 Preservation requirements can be found in EPA BAT screening protocols. As a general guideline, refrigerate or ice the sample within 7 days, and complete the analysis within 30 days of collection. Procedures for storing the sample in the trap are provided in Section 250.

243 Dry solids of the sludge sample should be determined according to Standard Methods.

250 Sample Purging

251 Condition the traps with the guard attached at 250°C overnight and adjust N2 gas flow at 20 ml/minute for each trap.

252 Set the temperature of the catalytic gas purifier at 900°C one day before purging the sample and check for flow of N2 through the purifier.

253 Install on a daily basis a freshly conditioned trap on the purified purging gas line so that residual impurities in the purge gas can be further removed.

254 Place the muffled 1000 ml Erlenmeyer flask in the water bath at 60°C. Attach the purging head to the flask and secure with springs. Connect a well-conditioned trap along with the guard and the N2 purging gas line to the head piece, and adjust the N2 flow to 200 ml/minute by measuring the flow with the soap bubble flowmeter connected to the end of the guard. Transfer 100 ml purgeable-free water (Procedure 259) into the flask and purge under stirring for 20 minutes.

255 Analyze the trap by GC for system and purgeable-free water blank, Section 260.

256 Quantitatively transfer the entire amount of sample from the sample vial into a muffled 100-ml volumetric flask and fill with the purgeable-free water to mark. Transfer the diluted sample to the 1000 ml purging flask with a well-conditioned trap along with the guard connected to the purging head and purge under stirring for 20 minutes.

257 Analyze the trap on GC immediately after purging, Section 260.

258 If the purged sample cannot be analyzed immediately, the trap should be capped and placed in a muffled glass tubing with one end sealed and padded with glass wool. The glass tubing is pull sealed with the aid of a propane torch long with a stream of N2 gas blowing into the tube. This is necessary to prevent contamination of the trap from the air trapped in the glass tubing.

259 Preparation of purgeable-free water

259.1 Boil vigorously 2-3 liters of good quality distilled water in a 4-liter flask for 15 minutes.

259.2 Allow to cool gradually under positive pressure as supplied by a stream of N2 gas purging con-

tinuously through the water under stirring.

259.3 Fill the muffled 100 ml volumetric flask full to overflow with the purged warm distilled water. Place the muffled stopper tightly on the flask with no dead volume, and let it cool down further to room temperature. In this way, diffusion of volatiles from ambient air into the water can be minimized.

259.4 Use the purgeable-free water from the 100 ml volumetric flask. Prepare purgeable-free water on a daily basis.

260 Quantitation

261 Quantitative measurements with this method are made by gas chromatography. While the choice of detector and column packing is left to the discretion of the analyst, recommendations are made below based on general applicability of all of the parameters included in this test procedure. A flame ionization detector is recommended for routine analysis. The detector of the gas chromagraphic system must be operated within its linear response range and the noise level should be less than 2% of fullscale deflection. Standards prepared from

the assayed reference materials must be injected frequently as a check on the stability of operating conditions. It is suggested that the concentration of stock standards be prepared on a volumetrical basis. A volumetric concentration of 50 ul/ml for the stock standards is suggested. Serial dilution can be prepared in methanol until a concentration is reached that is suitable for calibration of the instrument. The weight concentration can be computed by multiplying the density of the specific reference materials.

262 The 38 volatile organics listed in Table 211A can be determined by this procedure. A 12 ft x 2 mm ID Pyrex glass column packed with 0.2% Carbowax 1500 on Carbopack C, 60/80 mesh, is suggested. The sample is injected by desorbing from the trap at 250^{o}C for 4 minutes with the aid of a tubular heater (250^{o}C) and a 4-way valve wrapped around with a heating tape (80^{o}C). After flushing the sample into the column for 4 minutes, the temperature of the column is programmed to increase from 40^{o}C to 200^{o}C at a rate of 8^{o}C/minute and hold at 200^{o}C for 16 minutes. Table 200-2 lists the relative retention times of these volatile organics under the conditions given above. The procedures have good general utility for overcoming interferences in the analysis of volatile organics in sewage and sludge.

TABLE 200-2 Relative Retention Times of Volatile Organics on Packed GC Column (0.2% Carbowax 1500 on Carbopack C)

Compound	Relative Detention Time Chloroform	2-Bromo-1-chloropropane
1,1-dichloroethane	0.85	0.37
1,2-transdichloroethylene	0.93	0.43
Chloroform	1.00	0.46
1,2-dichloroethane	1.10	0.53
Carbon tetrachloride	1.25	0.58
1,1,1-trichloroethane	1.29	0.60
Dichlorobromomethane	1.31	0.61
Trichloroethylene	1.57	0.71
Benzene	1.63	0.75
1,2-dichloropropane	1.68	0.77
Dibromochloromethane	1.89	0.87
1,1,2-trichloroethane	1.89	0.87
Tetrachloroethylene	2.13	0.98
2-bromo-1-chloropropane	2.16	1.00
2-chloroethyl vinyl ether	2.18	1.01
Bromoform	2.32	1.07
Chlorobenzene	2.35	1.08
Ethylbenzene	2.54	1.17
1,1,2,2-tetrachloroethane	2.71	1.25
Toluene	3.04	1.40
bis(2-chloroethyl) ether	3.31	1.53

270 Removal of Interferences

271 The dilution procedures in this section have proven utility in wastewater and sludge analysis. When this procedure is followed, recoveries of standards prepared from the assayed reference materials at a concentration of approximately 50 ppb (v/v) in purgeable-free water must be established. If use of this procedure fails to show the need for the dilution technique to eliminate interferences with a specific purge and trap glassware, wastewater, or sludge, the analyst may elect to delete this procedure with proper justification.

272 Determine the level of compounds of interest, Section 260.

272.1 Measure the level of these compounds in wastewater or sludge at two dilutions -- 7 and 25% for sludge or 25 and 40% for sewage. Section 260.

272.2 Normalize all concentrations determined on the basis of the higher dilution employed (7% for sludge or 25% for sewage), and plot on semilog graph paper with the ordinate in logarithmic scale for concentration.

272.3 Draw a straight line with a negative slope through these points and extrapolate to zero sewage or sludge addition. The value obtained at the intercept on the ordinate represents the concentration of a given compound to be determined in purgeable-free water.

272.4 Divide the above value obtained at zero sewage or sludge addition by the recovery of these compounds of interest determined at approximately 50 ppb (v/v) in purgeable-free distilled water. This gives the amount of these compounds in 100 ml of clean water. The actual concentration of these compounds in the original sewage and sludge can be obtained by dividing by 25% and 7%, respectively. Section 290.

272.5 Use the EPA internal standard, 2-bromo-1-chloropropane, in all quantitation studies to minimize variations in FID responses.

280 Quality Assurance (EMSL Protocol)

281 Standard quality assurance practices should be used with this method. Field replicates should be collected to validate the precision of the sampling technique.

Laboratory replicates should be analyzed to validate the precision of the analysis. Fortified samples should be analyzed to validate the accuracy of the analysis. The use of an internal standard for computing the relative FID response (Procedure 272.5) would improve the accuracy of the analysis by minimizing the effect of instability of the instrument. Where doubt exists over the identification of a peak on the chromatogram, confirmatory techniques, such as mass spectroscopy, should be used.

282 If the sample contains a large number of compounds, so that the peaks on the gas chromatogram are too close to each other, the verification of identifications by the relative retention times becomes extremely unreliable by GC retention times. A GC-mass spectrometer becomes the detector of choice.

290 Calculation and Reporting

291 Determine the concentration in the sample according to the following formula:

291.1 Sewage

$$\text{Micrograms/liter} = \frac{(A)\ (D)}{(B)\ (C)}$$

A = Concentration of compounds of interest in ppb (v/v), obtained by extrapolating to zero addition of sewage

B = Recovery from purgeable-free water

C = Percentage of the highest dilution made, 25% for sewage

D = Density of the compound of interest

291.2 Sludge

$$\text{Micrograms/gram} = \frac{(D)\,(E)}{(B)\,(F)\,(G)}$$

E = Concentration of compounds of interest in ppb (v/v)

F = Percentage of the lowest dilution made, 7% for sludge

G = Percentage of dry solids in the sludge

Method 300 -- Determination of Extractable Organics by Integrated Analysis

310 Scope and Application

311 This method allows the determination of the acid, neutral, and base fractions of the EPA Priority Pollutants (Table 300-1) and for non-priority compounds of similar structure and behavior. The detection of compounds in the ppb range or below is frequently possible with this method, depending on the specific compound and on the sample type.

312 This method has been developed primarily for POTW industry samples, and is demonstrably effective for sewage, effluent, and water samples. It has also been applied to sediment, sludge, and tissue samples with some success. It is anticipated that other types of samples could be handled by this method, with or without modifications.

320 Summary

321 This method consists of three stages: extraction, cleanup/prefractionation, and analysis. Some varia-

	Neutral/Base Extractable
1	Acenaphthene
77	Acenaphthylene
78	Anthracene
5	Benzidine
72	Benzo(a)anthracene
73	Benzo(a)pyrene
74	Benzo(b)fluoranthene
79	Benzo(ghi)perlyene
75	Benzo(k)fluoranthene
43	Bis(2-chloroethoxy)methane
42	Bis(2-chloroisopropyl)ether
66	Bis(2-ethylhexyl)phthalate
41	4-Bromophenylphenylether
67	Butylbenzylphthatlate
40	4-Chlorophenylphenylether
20	2-Chloronaphthalene
76	Chrysene
82	Dibenzo(a,q)anthracene
29	3,3-Dichlorobenzidine
70	Diethylphthalate
1	Dimethylphthalate
35	Dinitrotoluene
36	2,6-Dinitrotoluene
68	Di-n-butylphthalate
69	Di-n-octylphthalate
37	1,2-Diphenylhydrazine
39	Fluoranthene
80	Fluorene
9	Hexachlorobenzene
52	Hexachlorobutadiene
53	Hexachlorocyclopentadiene
12	Hexachloroethane
83	Indeno(1,2,3-cd)pyrene
54	Isophorone
55	Naphthalene
56	Nitrobenzine
61	N-Nitrosodimethylamine
62	N-Nitrosodiphenylamine
63	N-Nitrosodipropylamine
84	Pyrene
81	Phenanthrene
8	1,2,4-Trichlorobenzene

	Acid Extractable
24	2-Chlorophenol
31	2,4-Dichlorophenol
34	2,4-Dimethylphenol
60	4,6-Dinitro-o-cresol
59	2,4-Dinitrophenol
57	2-Nitrophenol
58	4-Nitrophenol
22	2,2-Parachlorometacresol
64	Pentachlorophenol
65	Phenol
21	2,4,6-Trichlorophenol

	Pesticides/PCB*
102	a-BHC-Alpha
103	b-BHC-Beta
104	γ-BHC-(lindane)Gamma
105	δ-BHC-Delta
91	chlordane
93	4,4'-DDD
94	4,4'-DDE
92	4,4'-DDT
90	Dieldrin
98	Endrin
95	a-endosulfan-Alpha
96	b-endosulfan-Beta
97	endosulfan-sulfate
99	endrinaldehyde
100	heptachlor
101	heptachlorepoxy
106	PCB 1242
107	PCB 1254
108	PCB 1221
109	PCB 1232
110	PCB 1248
111	PCB 1260
112	PCB 1016

Table 300-1 Compounds Detected in Extractables Analysis

tions in the details of the method will exist, depending on what type of sample is handled.

a. Extraction consists of a stirred liquid-liquid continuous extraction for 12 hours at pH 2, followed by 12 hours at pH 12 for most samples. Sediment, tissue, and sludges too heavy in particulates to be handled by this method are extracted by alternative methods. The extracts are dried and concentrated, and residue weights determined on an aliquot.

b. Cleanup/prefractionation consists of size-exclusion chromatography followed by secondary chromatography steps to deal with specific interference problems using a variety of techniques: adsorption, ion-exchange, and reverse-phase chromatography. The acidic components in the acid/neutral extract are separated at this point for derivatization and special analysis.

c. Analysis consists of screening of each fraction by capillary GC-FID, subsequent recombination of the neutral components, capillary GC/MS analysis of the neutral and derivatized acid fractions, and GC/EC analysis of the neutral fraction to pick up low levels of PCB's and pesticides. The GC/MS data is

analyzed by an automated data searching routine capable of applying both spectrum matching and GC retention time criteria for qualitative identification, and capable of either single-ion or multiple-ion quantitation.

330 General Preparation

331 Glassware

a. All glassware, including sampling vessels, beakers and flasks, chromatography columns, glass beads, etc., are cleaned by immersion in a pH sulfuric acid solution of tetramethylammonium peroxydisulfate ("no chromix") followed by a distilled water rinse. The glassware is then pyrolyzed in a pottery kiln at 450 degrees centigrade for 1 hour. Upon cooling, the glassware is silanized by treatment with a 5% solution of dichlorodimethyl-silane in pet ether for 5 minutes, followed by successive rinses with methanol and methylene chloride.

b. Teflon stopcocks and other teflon parts are acid washed and water-rinsed, followed by a methylene chloride solvent rinse.

c. An exception to the above is vessels used to collect and contain samples for VOA analysis. In this case, the glassware is muffled and sealed without silanization.

332 Preparation of chromatographic materials

a. All common materials in contact with the sample: teflon boiling stones, sodium sulfate, silica gel, florisil, glass wool, etc. are soxhlet-extracted for 6-8 hours with methylene chloride.

b. The florisil and silica gel are screened to remove fines and lumps.

c. Florisil: After extraction and drying, the florisil is activated in an oven at 140 degrees centigrade. In a narrow mouth cleaned jar, a weighed amount of the activated florisil is deactivated by adding 0.75 wt % distilled water, and rolling the sealed bottle on a jar mill overnight. The deactivated florisil must be carefully protected from any further contact with moisture.

d. Cesium silicate: Silica gel (after extraction, drying and screening) is slurried in methanol satu-

rated with cesium hydroxide (100 grams silica gel in 300 mls methanol solution). After stirring vigorously for 1 hour, the suspension is allowed to settle and is decanted. 100 ml fresh methanol is added as a rinse and the solids are separated by filtration through fritted glass. The filtered solids are washed with 200 ml methanol followed by 300 ml methylene chloride. The solids are air dried at 40-50 degrees centigrade and stored in a dessiccator. (Note: This material is used <u>without</u> activation).

e. SX-2 BioBeads: After extraction, the beads are air-dried and 40 grams are swollen in 50% methylene chloride/hexane for several hours prior to use. Upon packing into a column, several column-volumes of solvent are run through the material prior to use. After this column is thoroughly rinsed, it must be calibrated prior to use. Table 300-2 presents the mixture used for column calibration in order of elution from SX-2 BioBeads. The cuts taken for the fractions are indicated. The calibration run is made similarly to a real sample run, but with 60 ml followed by eighteen 5 ml fractions taken and analyzed upon conclusion by GC-FID.

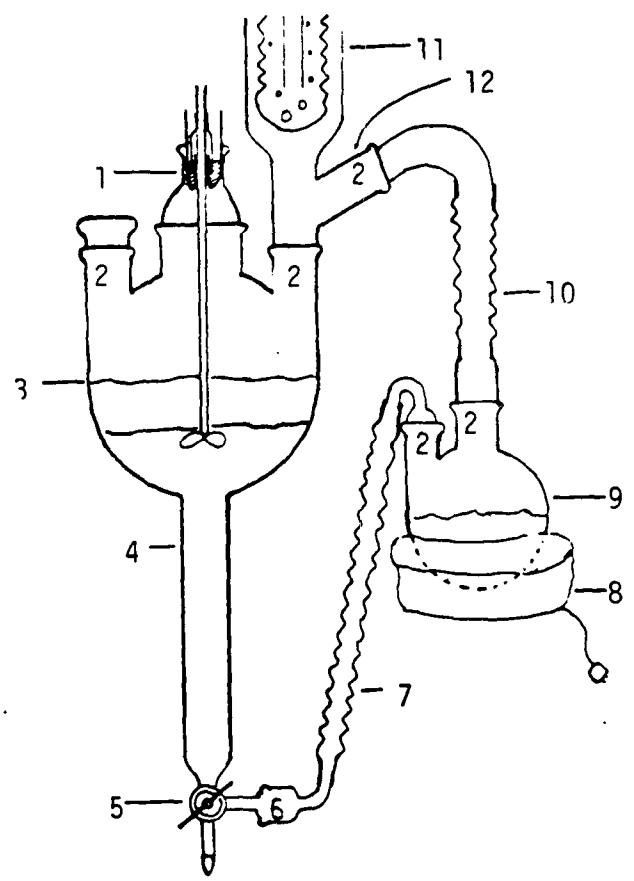
TABLE 300-2 Calibration Mixture for GPC

COMPOUND	FRACTION
2,4,6-trichlorophenol	A3
2-chlorophenol*	A3
Di-n-octylphthalate*	A2
Di-ethylhexylphthalate	A2
Heptachlor Epoxide	A2
Aldrin	A2
Heptachlor	A2
Hexanoic Acid	A1

* indicates compounds marking the start of the fraction

1 24/40 ⅄ LIQUID STIRRER SEAL
2 24/40 ⅄ JOINT
3 2000 ML 3-NECK RB FLASK
4 GLASS NECK FILLED WITH SILANIZED BEADS
5 TEFLON 3-WAY STOPCOCK
6 14/20 ⅄ JOINT
7 1/4 INCH CORRUGATED TEFLON
8 HEATING MANTLE
9 500 ML 2-NECK RB FLASK
10 3/4 INCH CORRUGATED TEFLON
11 FRIEDRICHS CONDENSER
12 MODIFICATION (VAPOR INLET)

1. ⅄ 24/40 CONNECTION TO ADDITIONAL CONDENSOR
2. OUTLET COLD FINGER
3. INLET COLDFINGER
4. ⅄ 34/40 JOINT FOR COLD FINGER
5. CONDENSOR OUTLET
6. CONDENSOR OPENING
7. CONDENSOR INLET
8. NOTE: ONE ARM IS HIGHER
9. ⅄ 24/40 FLASK WITH SAMPLE
10. ⅄ 24/40 FLASK WITH METHYLENE CHLORIDE



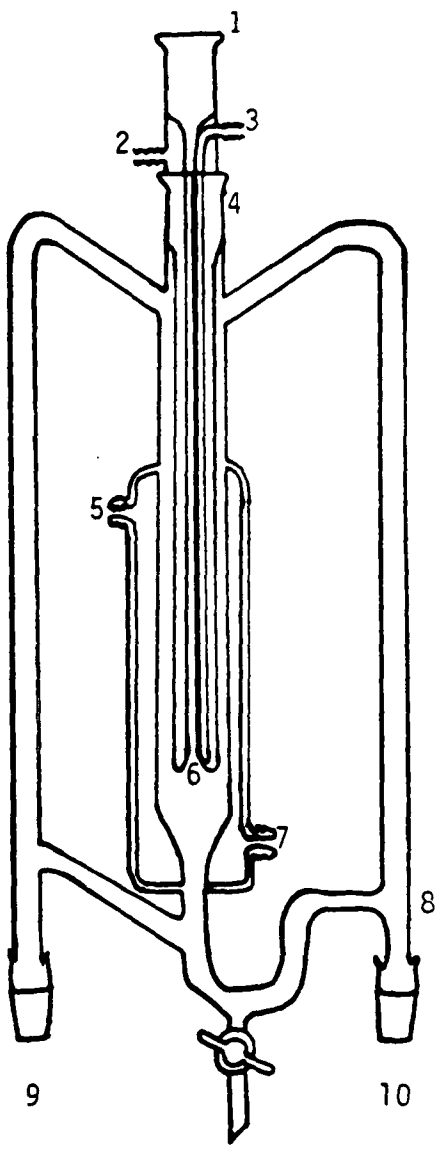


Figure 300-1 Stirred Liquid-Liquid Continuous Extractor and Steam Distillation Vapor Extractor Evaluated in the Present Study

f. Preparation of glass chromatographic columns: After columns are silanized and dry, the stopcock is reassembled. Tamp a small pug of silanized glass wool into the throat of the column using a glass stirring rod. Add 5 ml silanizing solution and run through to cover glass wool. After 5 minutes, drain into methanol and rinse column with 60 ml methanol. Disassemble stopcock and rinse barrel and glass with methanol from a squeeze bottle. Reassemble stopcock and rinse column thoroughly with methylene chloride, or pentane in the case of a florisil column.

340 Extraction

341 Apparatus and materials

a. Stirred liquid-liquid continuous extractor (Figure 300-1)
b. pH meter
c. Silanized glass beads
d. Variac
e. 500 ml heating mantle
f. Stirring motor
g. Toploading balance
h. Graduated cylinder

i. Distilled-in-glass methylene chloride and methanol

j. Reagent HCL and NaOH solutions

342 Sample handling and preparation sampling techniques appropriate to trace organics analysis must be used: sampling apparatus and bottles must be constructed of glass or teflon, glass surfaces must be clean (muffled at 450 degrees if possible), and containers to hold samples for extraction should be silanized or be of teflon. Refrigeration and prompt analysis is important for sample integrity, particularly for phenolic compounds.

343 Extraction of water and sewage samples

a. This procedure is the recommended alternative for all samples excepting those with physical properties such that this technique is impossible.

b. The components of the extractor apparatus are cleaned and muffled; the glass parts are silanized.

c. The neck of the extractor is filled with clean silanized glass beads. 1000 ml of a mixed (solids resuspended) sample is measured out and the pH

adjusted to 2.0 with hydrochloric acid solution.

d. The extractor is filled with approximately 200 ml distilled-in-glass methylene chloride to cover the glass beads. The sample is poured in to float above the methylene chloride. The side vessel is charged with about 300 ml methylene chloride.

e. The stopcock connecting the extractor vessel and the side vessel is opened and the relative heights of the two components adjusted to position the aqueous-organic interface about 2 cm above the top of the neck.

f. The stirrer is positioned in place and sealed in with the mercury seal. It is turned on and set to a moderate rate, sufficient for rapid mixing, but gentle enough to prevent emulsification. (For most samples this is between 50 and 200 rpm).

g. The side vessel is heated to reflux with a mantle heater. The rate of reflux is adjusted to equal the maximum flow rate through the glass bead bed. This requires periodic checking to readjust relative height of the side-arm vessel and the variac setting. The extractor should be in a stable con-

dition for at least an hour before being left unattended.

h. At the end of 12 hours reflux, the solvent in the side-arm vessel is removed and the pH of the aqueous phase is raised to 12. A fresh 300 ml of solvent is placed in a new side-arm vessel and step g is repeated.

i. The extraction is stopped after 12 hours and the base extract removed. Both the acid and the base extracts are dried over or by passage through anhydrous sodium sulfate (soxhlet-extracted prior to reactivation), and are K/D concentrated to between 1 and 2 mls.

j. A 10% aliquot of the extract is transferred to a tared weighing boat, dried at 100 degrees centigrade for 3 hours and weighed on an analytical balance to ±0.5 mg. The residue is then heated for an additional period and reweighed. The final weight, after no further change is seen, is used to compute the extracted residue in the remainder of the extract.

350 Size-exclusion Chromatography

351 Apparatus and materials

a. 40 gram SX-2 BioBeads column, pre-equilibrated and calibrated according to protocols (Section 330)
b. Distilled-in-glass pentane, methylene chloride
c. Vortex mixer
d. Precleaned and silanized pasteur pipettes
e. Graduate cylinder, 3, 100 ml, precleaned and silanized
f. K/D concentration apparatus

352 Preparation

a. The sample (acid or base extract preconcentrated to 1 ml in methylene chloride, residue weight determined) is diluted 50% with pentane to a find volume of not more than 3 ml. The sample is mixed by vortexing. If acid and base extracts are to be combined, further concentration may be required. For either separate or combined extracts, the total residue weight should not exceed 200 mg.

b. The column should be rinsed with at least 100 ml 50% pentane/methylene chloride. Care must be taken to assure the proper equilibration of the column. If the column has been stored for several hours or more under a reservoir of solvent, the solvent should be pipetted off and replaced, rather than eluted through. This is necessary as evaporation will change the solvent composition, even if the column is stoppered.

353 The Elution

a. The solvent head is allowed to diminish to less than 2 ml by eluting from the column.

b. The sample is applied with a pasteur pipette and the flow restarted. Starting at this point, the eluent is collected in a graduated cylinder. The container that held the sample is rinsed with 3 successive 2 ml portions of 50% pentane/methylene chloride which are each gently added to the column with the pipette as the solvent level approaches the gel bed.

c. The rinses are allowed to drain into the column until the top of the gel is nearly exposed. The

solvent reservoir over the column is charged with 250 ml of 50% pentane-methylene chloride.

d. Elution continues with 3 fractions being taken ("A1, A2, A3") according to the volumes determined in the calibration of the particular column used, of approximately 80, 40, and 130 mls for a 40 g column.

e. After the second fraction has eluted, the last fraction is eluted through a coupled column of cesium modified silica gel, as described in Section 350.

f. After last fraction is eluted, 100 ml 50% pentane/ methylene chloride is added as a rinse.

g. After rinsing, the column is inspected by UV light for uneluted polycyclic aromatics. It is also inspected visually for cracks, bubbles, and residue at the top of the column.

h. The fractions are K/D concentrated to 1 ml. If a concentration standard (such as D4-dichlorobenzene) is to be used, it is added prior to K/D concentration .

360 Coupled Cesium-Silica Gel Chromatography

361 Apparatus and materials

a. Cesium-modified silica gel
b. Glass chromatography column, 8 x 20 mm ID, prepared as indicated in Section 230.
c. Distilled-in-glass solvents: pentane, methylene chloride, and methanol
d. Prepared glass containers -- 50, 100 ml Erlenmeyer flasks
e. Graduated cylinder, 100 ml

362 Preparation

a. The column is prepared by slurrying 5.0 grams of the cesium silicate in 10 ml 50% pentane-methylene chloride and pouring the suspension into a prepared column. The beaker used to slurry the silica is rinsed with more of the same solvent to quantitatively transfer the silica gel.

b. The silica is allowed to settle and is rinsed from the sides of the column. The excess solvent is allowed to drain until the top is basely covered.

363 Coupled column chromatography

a. The prepared column is inserted under the BioBeads SX-2 column immediately after the collection of the second fraction from the BioBeads. A small head of eluent is allowed to accumulate over the silicate, then the elution rates are matched between the columns by use of the stopcocks on the columns. The flow-through eluent from the silicate is collected as fraction A3.

b. After the entire fraction from the BioBeads has eluted onto the silica column, a 30 ml rinse of the pentane-methylene chloride solvent is run through the silica gel and combined with A3.

c. The retained components are eluted into a separate contained following this rinse by application of 60 ml methanol.

d. If dinitrophenol is present in the sample, or has been spiked into the sample, it will indicate the proper functioning of the silicate column. This compound forms a bright yellow band at the top of the column, which eluted as a yellow solution in methanol. Only upon acidification does this

material (presumably cesium dinitrophenoxide) turn colorless.

e. The A3 eluent is concentrated as described in Section 240. The A3S (methanol eluent) fraction is treated with 140 ml hexane and K/D evaporated to 2 ml. After cooling, this concentrate is quantitatively transferred to a 60 ml separatory funnel. 10 mls each of methylene chloride and 1 M HCl are added and the separatory funnel is shaken vigorously. The mixture is allowed to settle, and the methylene chloride is carefully drained into a 50 ml Erlenmeyer flask. The aqueous phase is extracted twice more with 10 ml portions of methylene chloride, and the combined extracts dried over prepared sodium sulfate for at least one hour, with intermittent agitation.

f. The sample is concentrated to 1 ml prior to derivatization.

370 Florisil Chromatography

371 Apparatus and materials

a. Florisil, 0.75% H2O deactivated (Section 230)

b. Glass column, 8 x 20 mm ID, prepared according to Section 230.

c. Glass pipette, cleaned and silanized

d. Distilled-in-glass solvents: pentane, pet ether, ethyl ether, and methylene chloride

e. Graduate cylinder, 100 ml

f. K/D apparatus

372 Preparation

a. The A2 fraction from the BioBeads column is K/D concentrated to 1 ml. 2 ml hexane is added and the fraction further concentrated to a final volume of 1 ml.

b. The column is prepared by slurry-packing 10 g prepared florisil in pentane into the column using a long-stemmed funnel. The column should be prepared as described in Section 230. The florisil is washed into the column with a hexane wash bottle and 50 ml hexane eluted through as a rinse.

c. If bubbles or cracks develop, resuspend the florisil by shaking the stoppered column with a solvent head of hexane and allow to resettle. After preparation, the solvent head is allowed to drain to

barely cover the florisil.

373 Florisil chromatography

a. The prepared fraction is applied to the top of the column with a pasteur pipette and allowed to drain as a 1 ml pentane rinse is used to effect quantitative transfer of the sample to the column.

b. The collection of the eluent begins with the sample application and may be collected directly in a K/D concentrator tube.

c. 14 ml pentane is applied to the column and eluted. The eluent from this rinse and the sample loading is collected as fraction "F1".

d. When the solvent head approaches the florisil bed, the collection vessels are changed and 200 ml 50% ethyl ether/pet ether is added. This solvent should be stored in a sealed container, or else prepared freshly. The eluent from this solvent is collected as fraction "F2".

e. Upon elution of this solvent, 50 ml 100% ethyl ether is added and its eluent collected as fraction

"F3".

f. All three fractions are K/D concentrated to 1 ml, treated with 5 ml methylene chloride, further concentrated to 1 ml, and vialed for GC analysis.

380 Derivatization of Fraction A3S with Diazomethane

381 Apparatus and materials

a. Diazomethane generator, see Figure 300-2
b. N-methyl,N-nitro,N-nitrosoguanidine (MNNG)
c. Wipes
d. Small spatula
e. Solvents: diethyl ether, methanol, glacial acetic acid, pentane, and methylene chloride
f. SN NaOH solution
g. Aluminum foil
h. K/D concentration apparatus
i. Gloves
j. Face shield
k. Fume hood

382 Preparation (in a fume hood)

a. Setting up the diazomethane generator:

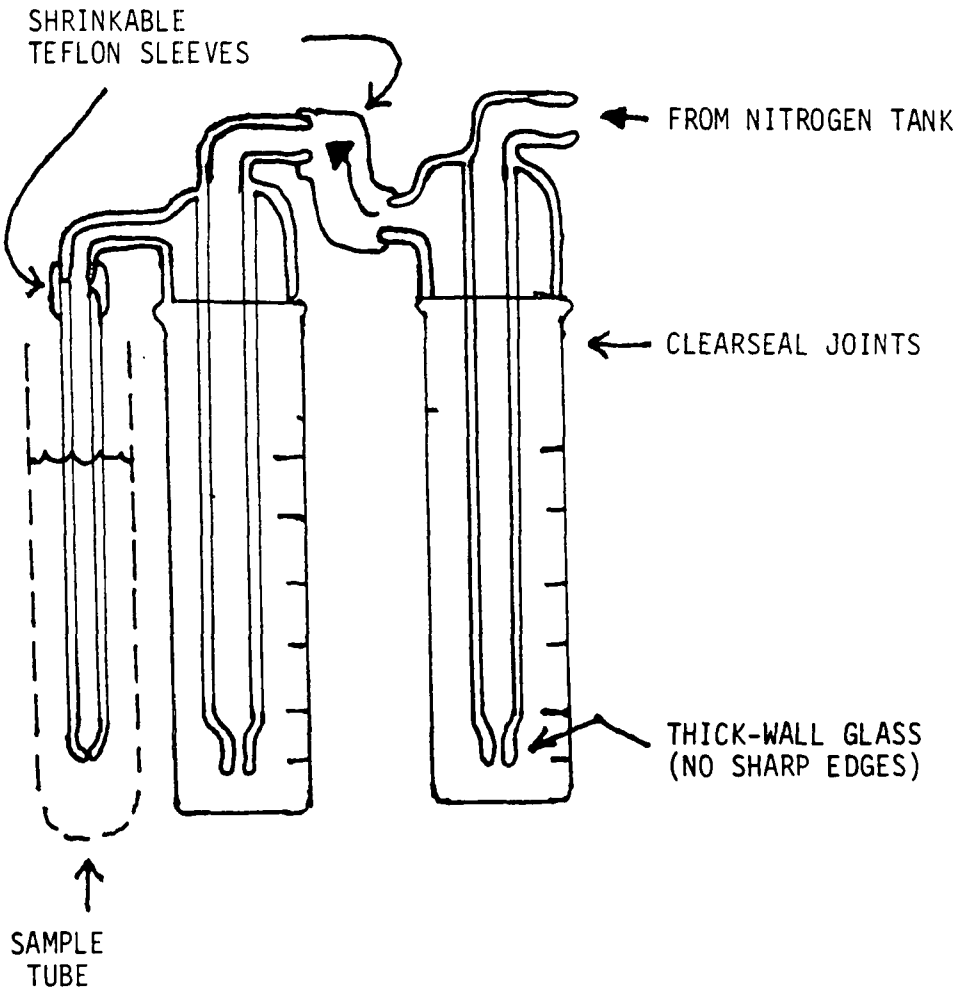


FIGURE 300-2 DIAZOMETHANE GENERATOR

1) Check the nitrogen tank for pressure.

2) Spread aluminum foil under the apparatus.

3) Place a small amount of MNNG in tube 1. A spatula tip will do. Weighing of this material or other unnecessary manipulations are to be avoided. More can be added, if necessary, during derivatization.

4) Any MNNG on outside of apparatus or other exterior surfaces should be immediately wiped up with a tissue which is disposed of in a plastic bag.

5) 30 ml diethylether is added to each tube, rinsing the MNNG down the sides and into the bottom of the first tube.

6) Ready samples, connect nitrogen purge line, and reseal both tubes.

7) 1 ml 5N NaOH is added to tube 1 with a pipette. This tube is immediately resealed and the nitrogen flow begun by cautiously opening the needle valve.

8) Diazomethane and its precursors are extremely hazardous materials. Gloves and face shield should be worn and a good fume hood used to contain vapors.

b. Sample preparation: The 1 ml concentrates are treated with 1 ml pentane and 0.2 ml methanol. The actual derivatization is very fast, so the samples can be arranged in a rack to permit rapid switching from one to another.

383 Derivatization

a. The bubbler tip is inserted into the first sample tube, and the gas stream containing the diazomethane is bubbled through the sample until formation of a yellow color that persists upon removal from the bubbler. If the sample is already yellow prior to bubbling, bubble for 90 seconds.

b. Set aside and let stand at room temperature for 7 minutes.

c. 9 ml pentane and 2 ml methylene chloride is added and the sample K/D concentrated to 1 ml. It can then be vialed for GC analysis.

d. When all of the samples have been derivatized, the gas is shut off and the generator is carefully disassembled. The excess diazomethane and/or MNNG is deactivated by addition of a few mls of glacial

acetic acid (added dropwise). The parts of the apparatus are rinsed with methanol and then with methylene chloride. All waste is carefully sealed in plastic bags for disposal.

390 Instrumental Analysis

391 Apparatus and materials

a. Internal standard solutions
b. Gas chromatograph (H/P 5840 or equivalent) equipped with FID and GC
c. GC/MS (Finnigan 4023 or equivalent)
d. Syringes (gas tight, 10 ul)

392 Preparation

a. The readied fractions: A1, A3, A3S (derivatized), F1, F2, and F3 are vialed in 1 ml methylene chloride solvent. Appropriate 10 ug hexamethylbenzene is added to each as a preliminary standard.

393 Instrumental analysis

a. GC/FID Screening: Each of the fractions except A1 is screened on a 15 meter SE-54 glass capillary

using a temperature program between 30 and 280 degrees centigrade. Fractions F1, F2, and F3 are screened using split injections with a split ratio of 20:1. Fractions A3 and A3S may be screened using a splitless injection.

b. If the results from the GC screening indicate adequate cleanup, fractions F2, F3, and A3 may be combined prior to GC/MS quantitation.

c. GC/EC analysis: The response and linearity of the priority pesticides and arochlors between 1 and 1000 picograms must be predetermined in standard runs and checked with standards periodically. An appropriate internal standard, such as decafluorobiphenyl, must be added to the sample. A 10% aliquot of the combined sample is spiked with a quantitation standard, translated into hexane azeotropically, and analyzed by GC/EC using a 15 meter SE-54 column with splitless injection. The analysis is temperature-programmed between 30 and 280 degrees centigrade. Priority compounds peaks are identified by retention time, reported to 0.01 minutes, and are tabulated along with integration values and expected retention times. Analysis of standards must show retention behavior to within 2

seconds of expected values.

e. GC/MS analysis: The instrument must be pretuned with FC-43 and decafluorotriphenylphosphine reference spectra specifications met. The sample is spiked with a multiple internal standard including the DFTPP MS standard. Injections are splitless with a 30 meter SE-54 column. Data is acquired at 1 second intervals for 5000 seconds to a final temperature of 280 degrees centigrade. The mass range and other acquisition praameters are as presented in Table 300-3.

400 Data Analysis

401 Apparatus and materials

a. This procedure is developed for an INCOS Data System operating on quadruple low resolution mass spectral data (Finnigan 4023 or equivalent).

b. Authentic standards of all EPA priority compounds, standards for quantitation and recovery, and instrument tuning are required.

402 Preparation

INSTRUMENT PARAMETERS:		MASS PARAMETERS:	
INTERFACE NUMBER	0	MINIMUM PEAK WIDTH	3
SUB-INTERFACE	0	MIN FRAG WID AS % PEAK WID	80
2ND ACQU INTERFACE	-1	BASELINE TO SUBTRACT	0
SUB-INTERFACE	-1	MINIMUM AREA AFTER MERGE	1
NUMBER OF ACQU BUFFERS	16	MERGE TOL AS % PEAK WIDTH	80
		AREA TOL (DELTA SQRT AREA)	20
INSTRUMENT TYPE	Q	ALIGN TOL AS % PEAK WIDTH	80
FULL SCALE MASS	1024	MAX # OF PEAKS TO READ	2000
ZERO SCALE MASS	1	MASS DEF AT 100 AMU (MMU)	30
INTENSITY/ION	2	NOISE REJ FOR ENHA (0-4 S.D.)	2
ACQUISITION DIRECTION	UP	TAIL REJ FOR ENHA (-1=NONE)	0
SAMPLES/PEAK - CENTROID	10	BKGND MULTIPLIER IN ADD	1.003
SAMPLES/PEAK - FRAGMENT	10	ADD OR AVERAGE IN ADD	AVER
PEAK WIDTH (MMU)	1000.		
OFFSET AT LOW MASS (MMU)	0		
OFFSET AT HIGH MASS (MMU)	0		
VOLTAGE SETTLING TIME(MS)	4		

LOW MASS:	34	UP:	0.95 L*	TOP:	0.00
HIGH MASS:	534	DOWN:	0.00 L	BOTTOM:	0.05
CENT S/P: 10	ACTUAL: 10	SAMP INT (MS):	0.200	PEAK WIDTH:	1000.
FRAG S/P: 10	ACTUAL: 10	SAMP INT (MS):	0.200	INTEN/ION.	2
MIN PEAK WIDTH:	3	MIN FRAG WIDTH %:	80	MIN AREA:	20
A/D THRESHOLD:	2	BASELINE:	0		

MODE: CENTROID POSITIVE ION

GC Parameters

TEMP1	285	30	33
TIME1	4.00		
RATE	15.00		
TEMP2	285	280	
TIME2	30.00		
INJ TEMP	400	0	92
FID TEMP	355	250	249
AUX TEMP	285	250	243
CHT SPD	0.10		
ZERO	5.0		
ATTN 2↑	3		
FID SGNL	+B		
SLP SENS	0.50		
AREA REJ		0	
FLOW A	0.0	47.0	
FLOW B	0.0	0.0	

Table 300-3 Instrumental Conditions for Extractables Analysis

a. Data acquired in a GC/MS analysis, as described in Section 270 and available on magnetic disc, is needed.

b. Initialization: Standard response factors, retention times, and mass spectra for the priority compounds must be obtained from calibration analysis of standards. The actual retention times and spectra for each compound must be obtained by hand manipulation of the data and stored in a library or libraries on the system. Once this is done, the response and retention of each compound can be updated using the programs SININ and UPDATE. These are modified versions of a proprietary program of the Finnigan Corporation. The calibration analysis treated with SININ to generate a quantitation list which is then hand-edited. When it is complete and correct, UPDATE is used to add this information to the quantitation libraries. The formats for using the programs are WQ DATAFILE,CALFILE,SININ (return), and UPDATE (return), respectively, where DATAFILE is the name of the calibration analysis data file. In order for UPDATE to run properly, the MSDS variable table must show the appropriate files (i.e., DATAFILE in variable 1 and DATAFILE.QL in variable 8).

c. Data analysis: The program SINUNK is used to reduce the data. It is begun by typing WQ DATA-FILE,CALFILE,SINUNK, (return). Prior to this, the library list of the libraries to be searched for in the data must be entered in variable 6 of the MSDS table, and the total library to be used for forward searching of questionable identifications must be entered in the program Other critical parameters to be set or changed as the data requires are the window for the internal standards search, the window for the unknown search, and the fit/purity test threshold. The proper set of these parameters will determine the frequency of false positive/negative identifications, and must be arrived at by experience on a per-compound basis. Initially, it is wise to use larger windows and lower fit criteria, to bias the analysis toward false positives. These can be edited out by hand. This program has been modified to allow for additional analysis for compounds with a fit below an arbitrarily high value (850 in this version) but above a minimum fit (750 here). These marginal compounds are forward searched against a total library. If the fit from the forward search is not above 800, the identification can be safely rejected (based on analysis of standards and spiked samples).

d. Quantitation is carried out on a single-ion basis for most compounds, although a several component abbreviated spectrum could be used for quantitation in cases of low response if desired.

e. The data analysis report is reduced to a summary (an example is included) which includes retention behavior, statistical quality of the identification, quantitation and integration information, and the analysis of several spiked compounds to provide QA/QC information on each sample.

f. Figure 300-3 shows a sample data analysis Figure 300-4 shows a sample data summary sheet.

```
PARAMETERS                    DATA: PS600V2.TI (PS600V2.MI)
10/03/79 11:46:00 + 33:20     CALI: C0917A              SCANS     1 TO 4000
SAMPLE: PS600V2 10UL PS606 + 10UL RECOV STD FORT WAYNE
CONDS.: C0926A.CT
FORMULA: WB1003A              INSTRUMENT: FINN          WEIGHT:     0.000
SUBMITTED BY: COLDTRAP        ANALYST: RD               ACCT. NO.:

ACC. VOL.:    8000            THRESHOLD:        2       INTEN/ION:        2
A/D S.I.:    0.025            CENT S.I.:    0.150       FRAG S.I.:    0.150
PEAK WIDTH:   1000.           CENT SAMP/PK:    10       FRAG SAMP/PK:     1
MIN. WIDTH:      3            MIN. FRAG. WIDTH (%): 80  MIN AREA:        20

4000 SCANS (759 SECTORS) OF LINEAR UP CENTROID DATA

LOW MASS:     34  SCAN TIMES (SECS.) UP:   0.45      TOP:   0.00
HIGH MASS:   334                     DOWN:   0.00   BOTTOM:   0.05

THERE IS A SCAN LIST WITH     0 ENTRIES

THERE IS A QUANTITATION LIST WITH  61 ENTRIES
```

Figure 300-3 Sample MS Data Report
Page 1 - Header (Acquisition Parameters)

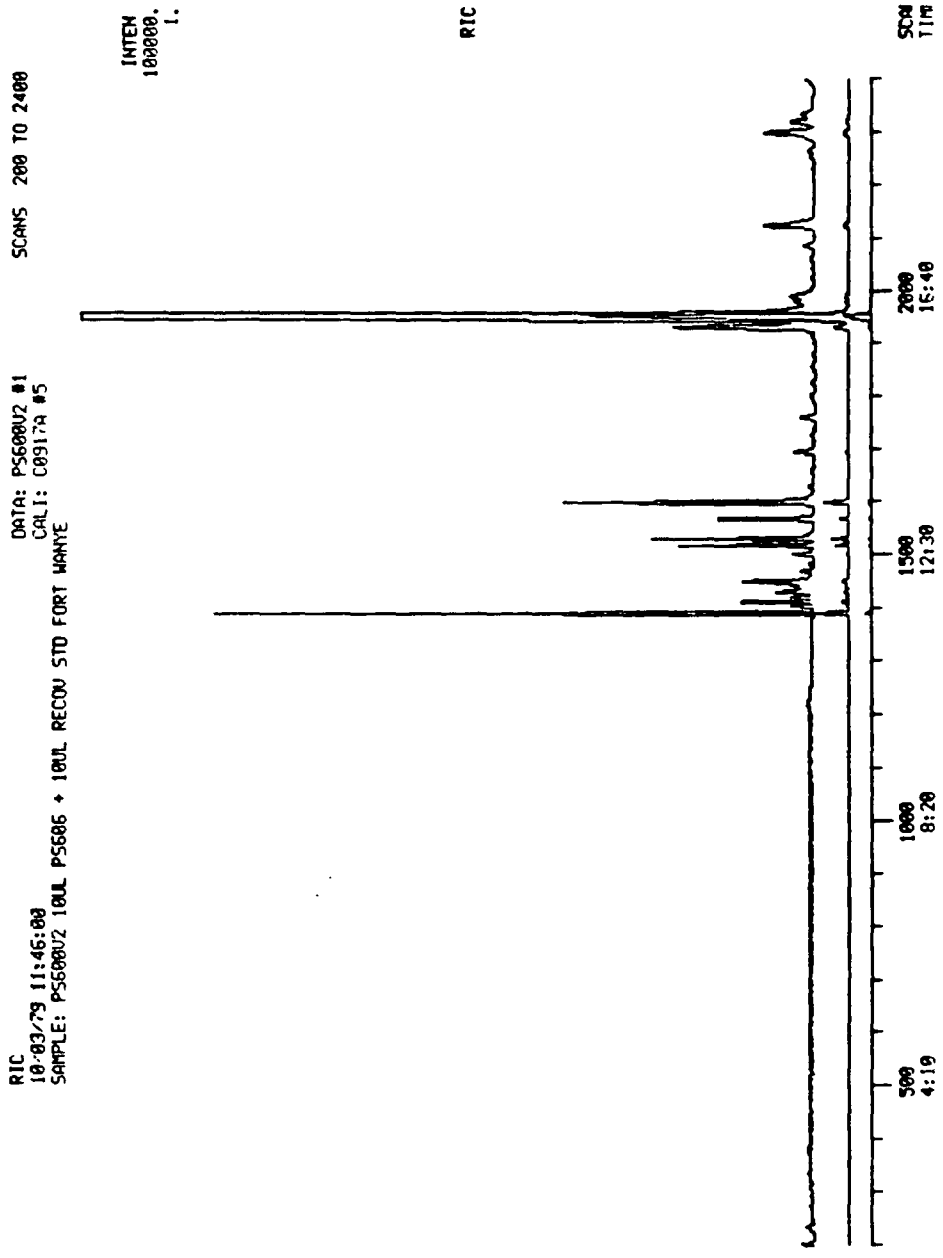


Figure 300-3 Continued

Page 2 - RGC Trace of Data

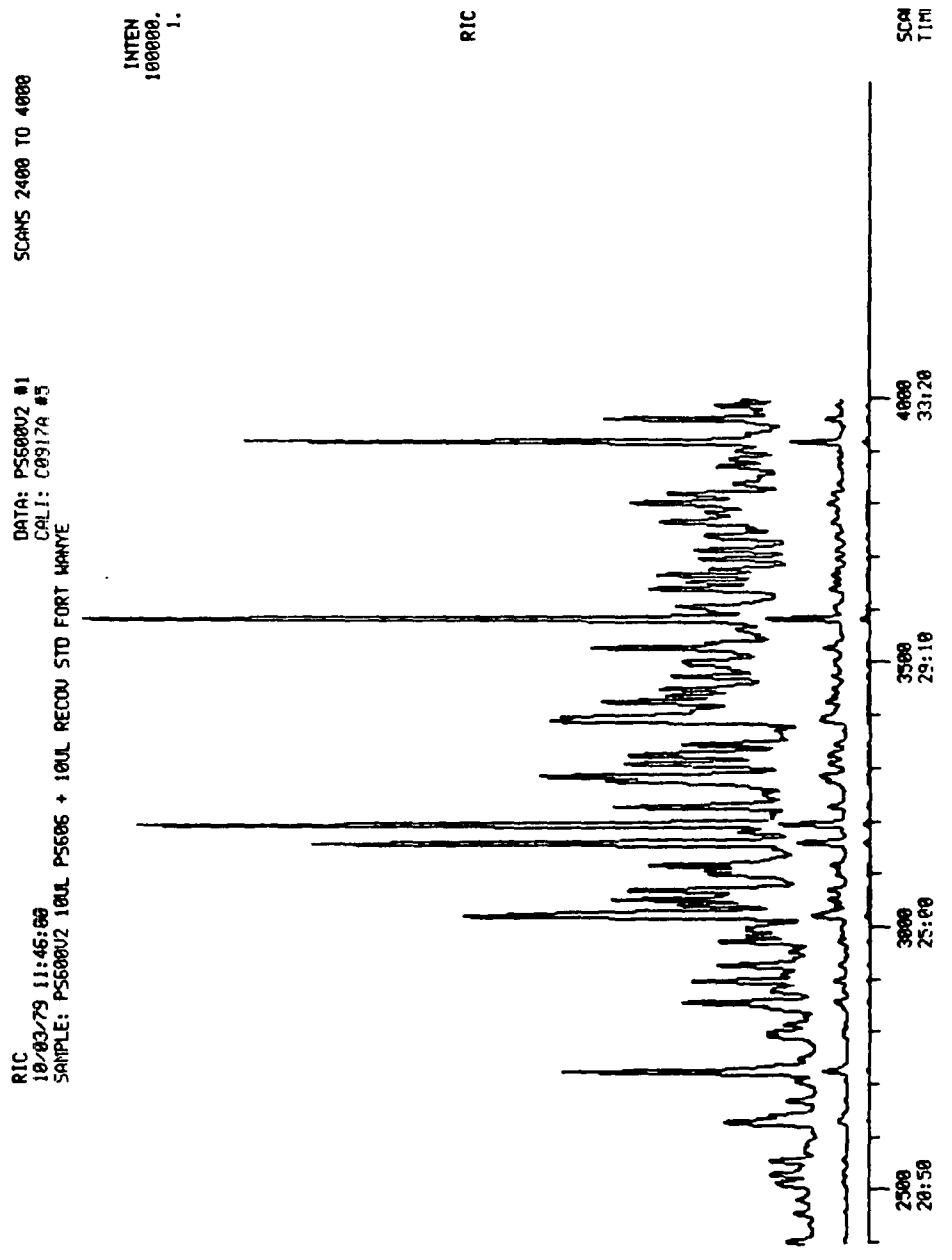


Figure 300-3 Continued
RGC Trace of Data

```
QUANTITATION REPORT          FILE: PS600V2

DATA: PS600V2.TI
10/03/79 11:46:00
SAMPLE: PS600V2 10UL PS606 + 10UL RECOV STD FORT WAYNE
CONDS.: C0926A.CT
FORMULA: WB1003A             INSTRUMENT: FINN              WEIGHT.     0.000
SUBMITTED BY: COLDTRAP       ANALYST: RD                   ACCT. NO..

AMOUNT=AREA * REF.AMNT/(REF.AREA* RESP.FACT)

 NO  NAME
  1  D6-2,2-DICHLOROPROPANE
  2  D5-CHLOROETHANE
  3  D3-ACRYLONITRILE
  4  D1-CHLOROFORM
  5  D3-1,1,1-TRICHLOROETHANE
  6  D6-BENZENE
  7  D8-TOLUENE
  8  D1-BROMOFORM
  9  D4-1,4-DICHLOROBENZENE
 10  METHANE,DICHLORODIFLUORO-
 11  ETHENE,CHLORO-
 12  METHANE,CHLORO-
 13  METHANE,BROMO-
 14  ETHANE,CHLORO-
 15  METHANE,TRICHLOROFLUORO-
 16  ETHENE,1,1-DICHLORO-
 17  2-PROPENAL
 18  2-PROPENENITRILE
 19  PROPANE,2,2-DICHLORO-
 20  METHANE,DICHLORO-
 21  ETHENE,1,2-DICHLORO-,(Z)-
 22  ETHANE,1,1-DICHLORO-
 23  METHANE,TRICHLORO-
 24  ETHENE,TRICHLORO-
 25  ETHANE,1,2-DICHLORO-
 26  METHANE,TETRACHLORO-
 27  ETHANE,1,1,1-TRICHLORO-
 28  BENZENE
 29  D8-TOLUENE
 30  D1-BROMOFORM
 31  D4-1,4-DICHLOROBENZENE
 32  ETHENE,TRICHLORO-
 33  ETHANE,1,2-DICHLORO-
 34  METHANE,TETRACHLORO-
 35  D3-1,1,1-TRICHLOROETHANE
 36  ETHANE,1,1,1-TRICHLORO-
 37  D6-BENZENE
 38  BENZENE
 39  PROPANE,1,2-DICHLORO-
 40  METHANE,BROMODICHLORO-
 41  1-PROPENE,1,3-DICHLORO-,(Z)-
 42  BENZENE,METHYL-
 43  1-PROPENE,1,3-DICHLORO-,(E)-
 44  ETHANE,1,1,2-TRICHLORO-
 45  METHANE,DIBROMOCHLORO-
 46  ETHENE,TETRACHLORO-
 47  D1-BROMOFORM
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Figure 300-3 Continued
Page 4 - List of Compounds Searched

NO	NAME
48	METHANE, DIBROMOCHLORO-
49	ETHENE, TETRACHLORO-
50	BENZENE, CHLORO-
51	BENZENE, ETHYL-
52	METHANE, TRIBROMO-
53	ETHANE, 1, 1, 2, 2-TETRACHLORO-
54	D4-1, 4-DICHLOROBENZENE
55	BENZENE, 1, 4-DICHLORO-

NO	M/E	SCAN	TIME	REF	RRT	METH	AREA	AMOUNT		%TOT
1	83	1519	12:39	1	1.000	A BB	12893.	11.120	NG	2.55
2	69	1412	11:46	1	0.930	A BB	8904.	18.652	NG	4.27
3	56	1449	12:04	1	0.954	A BB	5589.	55.051	NG	12.62
4	84	1531	12:45	1	1.008	A BB	35161.	14.988	NG	3.43
5	100	1568	13:04	1	1.032	A BB	15724.	13.503	NG	3.09
6	84	1600	13:20	1	1.053	A BB	72753.	15.116	NG	3.46
7	98	1931	16:05	1	1.271	A BB	63202.	50.799	NG	11.64
8	NOT FOUND									
9	150	3229	26:54	1	2.126	A BB	45700.	8.051	NG	1.84
10	NOT FOUND									
11	NOT FOUND									
12	NOT FOUND									
13	NOT FOUND									
14	NOT FOUND									
15	NOT FOUND									
16	NOT FOUND									
17	NOT FOUND									
18	NOT FOUND									
19	NOT FOUND									
20	49	1451	12:05	1	0.955	A BB	6728.	1.883	NG	0.43
21	NOT FOUND									
22	NOT FOUND									
23	83	1519	12:39	1	1.000	A BB	12893.	0.968	NG	0.22
24	130	1697	14:08	1	1.117	A BB	1470.	1.167	NG	0.27
25	NOT FOUND									
26	NOT FOUND									
27	NOT FOUND									
28	78	1604	13:22	1	1.056	A BB	2556.	0.576	NG	0.13
29	98	1931	16:05	29	1.000	A BB	63567.	8.670	NG	1.99
30	174	2629	21:54	29	1.361	A BB	14577.	8.036	NG	1.84
31	150	3229	26:54	29	1.672	A BB	44797.	10.070	NG	2.31
32	130	1697	14:08	29	0.879	A BB	1470.	0.618	NG	0.14
33	NOT FOUND									
34	NOT FOUND									
35	100	1568	13:04	29	0.812	A BB	15724.	7.145	NG	1.64
36	NOT FOUND									
37	84	1600	13:20	29	0.829	A BB	72753.	7.998	NG	1.83
38	78	1604	13:22	29	0.831	A BB	2556.	0.305	NG	0.07
39	NOT FOUND									
40	NOT FOUND									
41	NOT FOUND									
42	91	1954	16:17	29	1.012	A BB	1170400.	133.491	NG	30.59
43	NOT FOUND									
44	NOT FOUND									
45	NOT FOUND									
46	NOT FOUND									

Figure 300-3 Continued
Page 5 - Quantitation Report

NO	M/E	SCAN	TIME	REF	RRT	METH	AREA	AMOUNT		%TOT
47	174	2629	21:54	47	1.000	A BB	14577.	28.900	NG	6.62
48	NOT FOUND									
49	NOT FOUND									
50	NOT FOUND									
51	91	2485	20:42	47	0.945	A BB	8677.	3.388	NG	0.78
52	NOT FOUND									
53	NOT FOUND									
54	150	3229	26:54	47	1.228	A BB	44397.	35.891	NG	8.22
55	NOT FOUND									

Figure 300-3 Continued
Page 6 - Quantitation Report

UNKNOWN SAMPLE QUANTITATION REVERSE SEARCH STATUS REPORT

EXPECTED SCAN	BEST SCAN	FIT	PURITY	LIBRARY	ENTRY	# PEAKS FOUND	# PEAKS QUANT
1519	1519	943	803	V1	1	1	1
1401	1412	959	697	V1	2	1	1
1440	1449	946	381	V1	3	1	1
1533	1531	991	885	V1	4	2	1
1572	1568	894	816	V1	5	1	1
1605	1600	989	920	V1	6	1	1
1939	1931	988	884	V1	7	1	1
2649	2630	951	322	V1	8	1	0
3235	3229	958	673	V1	9	1	1
1377	0	0	0	V1	10	0	0
1388	0	0	0	V1	11	0	0
1388	0	0	0	V1	12	0	0
1398	0	0	0	V1	13	0	0
1401	0	0	0	V1	14	0	0
1415	0	0	0	V1	15	0	0
1433	0	0	0	V1	16	0	0
1747	0	0	0	V1	17	0	0
1443	0	0	0	V1	18	0	0
1519	0	0	0	V1	19	0	0
1445	1451	971	549	V1	20	2	1
1467	0	0	0	V1	21	0	0
1482	0	0	0	V1	22	0	0
1535	1519	827	219	V1	23	1	1
1706	1697	906	265	V1	24	1	1
1590	0	0	0	V1	25	0	0
1609	1568	942	119	V1	26	1	0
1577	0	0	0	V1	27	0	0
1609	1604	841	169	V1	28	1	1
1931	1931	988	884	V2	1	1	1
2633	2630	951	322	V2	2	1	1
3218	3229	958	673	V2	3	1	1
1696	1697	906	265	V2	4	1	1
1581	0	0	0	V2	5	0	0
1599	1568	942	119	V2	6	1	0
1563	1568	894	816	V2	7	1	1
1568	0	0	0	V2	8	0	0
1595	1600	989	920	V2	9	1	1
1599	1604	841	169	V2	10	1	1
1696	0	0	0	V2	11	0	0
1725	0	0	0	V2	12	0	0
1847	0	0	0	V2	13	0	0
1949	1954	989	900	V2	14	1	1
1969	0	0	0	V2	15	0	0
1996	0	0	0	V2	16	0	0
2108	0	0	0	V2	17	0	0
2162	0	0	0	V2	18	0	0
2630	2630	951	322	V3	1	1	1
2105	0	0	0	V3	2	0	0
2160	0	0	0	V3	3	0	0
[illegible]	0	0	0	V3	4	0	0
2483	2535	962	699	V3	5	2	1
2638	0	0	0	V3	6	0	0
2825	0	0	0	V3	7	0	0
3214	3229	958	673	V3	8	1	1
3214	0	0	0	V3	9	0	0

DATA PROCESSING OF PS600V2 COMPLETED ON 10/03/79 21:07:29

Figure 300-3 Continued
Page 7 - Reverse Search Status Report

QUANTITATION REPORT FILE: TEMP2

DATA: PS600V2 TI
10/03/79 11:46:00
SAMPLE: PS600V2 10UL PS606 + 10UL RECOV STD FORT WAYNE
CONDS : C0926A CT
FORMULA: WB1003A INSTRUMENT: FINN WEIGHT: 0.000
SUBMITTED BY: COLDTRAP ANALYST: RD ACCT. NO.:

AMOUNT=AREA * REF.AMNT/(REF.AREA* RESP.FACT)

NO	NAME
1	METHANE, DICHLORO-
2	D1-CHLOROFORM
3	D6-2,2-DICHLOROPROPANE
4	UNKNOWN
5	UNKNOWN
6	METHANE, DICHLORO-
7	D6-2,2-DICHLOROPROPANE
8	D1-CHLOROFORM
9	UNKNOWN

NO	M/E	SCAN	TIME	REF	RRT	METH	AREA	AMOUNT		%TOT
1	TOT	1451	12:05	8	0.948	A VB	19511.	8.629		6.06
2	TOT	1531	12:45	8	1.000	A BB	83660.	37.000	NG	25.99
3	TOT	1519	12:39	8	0.992	A BB	57765.	25.548	NG	17.95
4	NOT FOUND									
5	NOT FOUND									
6	TOT	1451	12:05	8	0.948	A VB	19511.	8.629		6.06
7	TOT	1519	12:39	8	0.992	A BB	57765.	25.548	NG	17.95
8	TOT	1531	12:45	8	1.000	A BB	83660.	37.000	NG	25.99
9	NOT FOUND									

NO	RET(L) RATIO	RRT(L)	RATIO	AMNT	AMNT(L)	R.FAC	R.FAC(L)	RATIO
1		1.000	0.95	8.63	100.00	0.086	1.000	0.09
2		1.000	1.00	37.00	37.00	1.000	1.000	1.00
3		1.000	0.99	25.55	27.80	0.919	1.000	0.92
4								
5								
6		1.000	0.95	8.63	100.00	0.086	1.000	0.09
7		1.000	0.99	25.55	27.80	0.919	1.000	0.92
8		1.000	1.00	37.00	37.00	1.000	1.000	1.00
9								

Figure 300-3 Continued
Page 8 - Forward Search Quantitation Report

SAMPLE PS600V2 AUTOGC RUN ON 10/03/79 21:07:36

SCAN	FIT	PURITY	LIBRARY	ENTRY
1451	991	956	VT	19
1531	993	985	VT	4
1519	948	923	VT	3
1604	781	750	VT	26
1604	781	750	VT	26
1451	991	956	VT	19
1519	948	923	VT	3
1531	993	985	VT	4
1604	781	750	VT	26

ANALYSIS OF TEMP2 COMPLETED ON 10/03/79 21:14:43

Figure 300-3 Continued
Page 9 - Forward Search Status Report

ANALYSIS REPORT — PURGABLE ORGANICS
Sample:

Lab:
Sample Size:
Date Sampled:

Quantitation Standard:
Quantitation Method:

COMPOUND		AMT	FIT	ΔT	AMT	FIT	ΔT
D5-CHLOROETHANE	12893						
D3-ACRYLONITRILE	8904						
D6-2,2-DICHLOROPROPANE	5569						
D1-CHLOROFORM	35161						
D3-1,1,1-TRICHLOROETHANE	15714						
D6-BENZENE	72753						
D8-TOLUENE	63224						
D1-BROMOFORM	14517						
D4-1,4-DICHLOROBENZENE	44367						
METHANE, DICHLORODIFLUORO-							
ETHENE, CHLORO-							
METHANE, CHLORO-							
METHANE, BROMO-							
ETHANE, CHLORO-							
METHANE, TRICHLOROFLUORO-							
ETHENE, 1,1-DICHLORO-							
2-PROPENAL							
2-PROPENENITRILE							
PROPANE, 2,2-DICHLORO-							
METHANE, DICHLORO-							
ETHENE, 1,2-DICHLORO-, (Z)-							
ETHANE, 1,1-DICHLORO-							
METHANE, TRICHLORO-							
ETHENE, TRICHLORO-							
ETHANE, 1,2-DICHLORO-							
METHANE, TETRACHLORO-							
ETHANE, 1,1,1-TRICHLORO-							
BENZENE							
PROPANE, 1,2-DICHLORO-							
METHANE, BROMODICHLORO-							
1-PROPENE, 1,3-DICHLORO-, (Z)-							
BENZENE, METHYL-							
1-PROPENE, 1,3-DICHLORO-, (E)-							
ETHANE, 1,1,2-TRICHLORO-							
METHANE, DIBROMOCHLORO-							
ETHENE, TETRACHLORO-							
BENZENE, CHLORO-							
BENZENE, ETHYL-							
METHANE, TRIBROMO-							
ETHANE, 1,1,2,2-TETRACHLORO-							
BENZENE, 1,4-DICHLORO-							

blank=not found; NQ=detected but not quantitated; NA=not analyzed for

Figure 300-3 Continued
Page 10 - Analysis Worksheet

QUANTITATION REPORT FILE: P5600V2

AMOUNT=AREA * REF. AMT/(REF. AREA* RESP. FACT)

NO	NAME	NO	M/E	SCAN	TIME	REF	RRT	METH	AREA	AMOUNT	EXPECTED SCAN	BEST SCAN	FIT	PURITY
1	D6-2,2-DICHLOROPROPANE	1	83	1519	12:39	1	1.000	A BB	12093	11.120 NG	1519	1519	943	803
2	D3-CHLOROETHANE	2	69	1412	11.46	1	0.930	A BB	8904	18.632 NG	1401	1412	959	697
3	D3-ACRYLONITRILE	3	56	1449	12:04	1	0.954	A BB	9589.	33.091 NG	1440	1449	946	381
4	D1-CHLOROFORM	4	84	1531	12:43	1	1.008	A BB	35161.	14.988 NG	1533	1531	971	885
5	D3-1,1,1-TRICHLOROETHANE	5	100	1568	13:04	1	1.032	A BB	15724.	13.503 NG	1572	1568	894	816
6	D6-BENZENE	6	84	1600	13:20	1	1.053	A BB	72753	15.116 NG	1605	1600	909	920
7	D8-TOLUENE	7	98	1931	16:05	1	1.271	A BB	63202	50.799 NG	1939	1931	980	884
8	D1-BROMOFORM	8	NOT FOUND								2649	2630	931	322
9	D4-1,4-DICHLOROBENZENE	9	150	3229	26:54	1	2.126	A BB	45700.	0.051 NG	3205	3229	958	673
10	METHANE, DICHLORODIFLUORO-	10	NOT FOUND								1377	0	0	0
11	ETHENE, CHLORO-	11	NOT FOUND								1388	0	0	0
12	METHANE, CHLORO-	12	NOT FOUND								1388	0	0	0
13	METHANE, BROMO-	13	NOT FOUND								1398	0	0	0
14	ETHANE, CHLORO-	14	NOT FOUND								1401	0	0	0
15	METHANE, TRICHLOROFLUORO-	15	NOT FOUND								1415	0	0	0
16	ETHENE, 1,1-DICHLORO-	16	NOT FOUND								1433	0	0	0
17	2-PROPENAL	17	NOT FOUND								1747	0	0	0
18	2-PROPENENITRILE	18	NOT FOUND								1443	0	0	0
19	PROPANE, 2,2-DICHLORO-	19	NOT FOUND								1519	0	0	0
20	METHANE, DICHLORO-	20	49	1451	12:05	1	0.955	A BB	6720.	1.885 NG	1445	1451	971	549
21	ETHENE, 1,2-DICHLORO-, (Z)-	21	NOT FOUND								1467	0	0	0
22	ETHANE, 1,1-DICHLORO-	22	NOT FOUND								1482	0	0	0
23	METHANE, TRICHLORO-	23	83	1519	12:39	1	1.000	A BB	12893.	0.968 NG	1535	1519	827	219
24	ETHENE, TRICHLORO-	24	130	1697	14:08	1	1.117	A BB	1470.	1.167 NG	1706	1697	906	265
25	ETHANE, 1,2-DICHLORO-	25	NOT FOUND								1590	0	0	0
26	METHANE, TETRACHLORO-	26	NOT FOUND								1609	1568	942	119
27	ETHANE, 1,1,1-TRICHLORO-	27	NOT FOUND								1577	0	0	0
28	BENZENE	28	78	1604	13:22	1	1.056	A BB	2556.	0.576 NG	1609	1604	841	169
29	D8-TOLUENE	29	98	1931	16:05	29	1.000	A BB	63567.	8.670 NG	1931	1931	988	884
30	D1-BROMOFORM	30	174	2629	21:54	29	1.361	A BB	14577.	8.036 NG	2633	2630	951	322
31	D4-1,4-DICHLOROBENZENE	31	150	3229	26:54	29	1.672	A BB	44797.	10.070 NG	3218	3229	958	673
32	ETHENE, TRICHLORO-	32	130	1697	14:08	29	0.879	A BB	1470.	0.618 NG	1696	1697	906	265
33	ETHANE, 1,2-DICHLORO-	33	NOT FOUND								1581	0	0	0
34	METHANE, TETRACHLORO-	34	NOT FOUND								1599	1568	942	119
35	D3-1,1,1-TRICHLOROETHANE	35	100	1568	13:04	29	0.812	A BB	15724.	7.145 NG	1563	1568	894	816
36	ETHANE, 1,1,1-TRICHLORO-	36	NOT FOUND								1568	0	0	0
37	D6-BENZENE	37	84	1600	13:20	29	0.829	A BB	72753.	7.978 NG	1595	1600	909	920
38	BENZENE	38	78	1604	13:22	29	0.831	A BB	2556.	0.305 NG	1599	1604	841	169
39	PROPANE, 1,2-DICHLORO-	39	NOT FOUND								1696	0	0	0
40	METHANE, BROMODICHLORO-	40	NOT FOUND								1725	0	0	0
41	1-PROPENE, 1,3-DICHLORO-, (Z)-	41	NOT FOUND								1847	0	0	0
42	BENZENE, METHYL-	42	91	1954	16:17	29	1.012	A BB	1170400.	133.491 NG	1949	1954	989	900
43	1-PROPENE, 1,3-DICHLORO-, (E)-	43	NOT FOUND								1969	0	0	0
44	ETHANE, 1,1,2-TRICHLORO-	44	NOT FOUND								1996	0	0	0
45	METHANE, DIBROMOCHLORO-	45	NOT FOUND								2108	0	0	0
46	ETHENE, TETRACHLORO-	46	NOT FOUND								2162	0	0	0
47	D1-BROMOFORM	47	174	2629	21:54	47	1.000	A BB	14577.	28.900 NG	2630	2630	951	322
48	METHANE, DIBROMOCHLORO-	48	NOT FOUND								2105	0	0	0
49	ETHENE, TETRACHLORO-	49	NOT FOUND								2160	0	0	0
50	BENZENE, CHLORO-	50	NOT FOUND								2386	0	0	0
51	BENZENE, ETHYL-	51	91	2485	20:42	47	0.945	A BB	8677.	3.388 NG	2483	2535	962	699
52	METHANE, TRIBROMO-	52	NOT FOUND								2638	0	0	0
53	ETHANE, 1,1,2,2-TETRACHLORO-	53	NOT FOUND								2025	0	0	0
54	D4-1,4-DICHLOROBENZENE	54	150	3229	26:54	47	1.228	A BB	44397.	35.891 NG	3214	3229	958	673
55	BENZENE, 1,4-DICHLORO-	55	NOT FOUND								3214	0	0	0

Figure 300-4 Sample Analysis Summary Sheet

Appendix II

Analytical Data From First Three POTW Sites

The following tables report actual analytical results together with ancillary information concerning the analysis. No correction for recovery or other QA/QC result is applied to these results.

ANALYSIS REPORT PURGABLE ORGANICS
Sample: Renton, Raw Sewage
PS106V1
Lab: U of W
Sample Size: 1 ml
Date Sampled:

Quantitation Standard: Indicated by (*)
Quantitation Method: GC/MS

COMPOUND	PPB	FIT	ΔT	PPB	FIT	ΔT
D5-CHLOROETHANE	29.14	965	-3			
D3-ACRYLONITRILE	23.65	656	-6			
*D6-2,2-DICHLOROPROPANE	27.80	992	0			
D1-CHLOROFORM	50.99	996	0			
D3-1,1,1-TRICHLOROETHANE	36.02	984	1			
D6-BENZENE	28.21	997	-1			
*D8-TOLUENE	21.70	993	0			
*D1-BROMOFORM	72.30	986	0			
D4-1,4-DICHLOROBENZENE	51.20	988	10			
METHANE, DICHLORODIFLUORO-						
ETHENE, CHLORO-						
METHANE, CHLORO-						
METHANE, BROMO-						
ETHANE, CHLORO-						
METHANE, TRICHLOROFLUORO-						
ETHENE, 1,1-DICHLORO-	7.74	944	0			
2-PROPENAL						
2-PROPENENITRILE						
PROPANE, 2,2-DICHLORO-						
METHANE, DICHLORO-	654.2	891	-5			
ETHENE, 1,2-DICHLORO-, (Z)-	0.34	931	-5			
ETHANE, 1,1-DICHLORO-	3.56	940	-3			
METHANE, TRICHLORO-	15.02	852	1			
ETHENE, TRICHLORO-						
ETHANE, 1,2-DICHLORO-	3.48	946	-2			
METHANE, TETRACHLORO-						
ETHANE, 1,1,1-TRICHLORO-	65.18	937	1			
BENZENE	17.70	988	0			
PROPANE, 1,2-DICHLORO-						
METHANE, BROMODICHLORO-						
1-PROPENE, 1,3-DICHLORO-, (Z)-						
BENZENE, METHYL-	21.96	992	-1			
1-PROPENE, 1,3-DICHLORO-, (E)-						
ETHANE, 1,1,2-TRICHLORO-						
METHANE, DIBROMOCHLORO-						
ETHENE, TETRACHLORO-	49.08	950	9			
BENZENE, CHLORO-						
BENZENE, ETHYL-	1.20	996	-5			
METHANE, TRIBROMO-						
ETHANE, 1,1,2,2-TETRACHLORO-						
BENZENE, 1,4-DICHLORO-						

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=probable artifact

ANALYSIS REPORT PURGABLE ORGANICS
Sample: Renton, Primary Effluent
PS95V1
Lab: U of W
Sample Size: 1 ml
Date Sampled:

Quantitation Standard: Indicated by (*)
Quantitation Method: GC/MS

COMPOUND	PPB	FIT	ΔT	PPB	FIT	ΔT
D5-CHLOROETHANE	67.52	998	-19			
D3-ACRYLONITRILE						
*D6-2,2-DICHLOROPROPANE	27.80	987	0			
D1-CHLOROFORM	37.96	995	2			
D3-1,1,1-TRICHLOROETHANE	34.61	987	5			
D6-BENZENE	18.46	990	7			
*D8-TOLUENE	21.70	997	0			
*D1-BROMOFORM	72.30	982	0			
D4-1,4-DICHLOROBENZENE	16.08	977	24			
METHANE, DICHLORODIFLUORO-						
ETHENE, CHLORO-						
METHANE, CHLORO-						
METHANE, BROMO-						
ETHANE, CHLORO-						
METHANE, TRICHLOROFLUORO-						
ETHENE, 1,1-DICHLORO-	1.04	870	-13			
2-PROPENAL						
2-PROPENENITRILE						
PROPANE, 2,2-DICHLORO-						
METHANE, DICHLORO-	606.94	<928	-6			
ETHENE, 1,2-DICHLORO-, (Z)-	0.56	848	-5			
ETHANE, 1,1-DICHLORO-	0.90	822	-3			
METHANE, TRICHLORO-	7.96	848	2			
ETHENE, TRICHLORO-						
ETHANE, 1,2-DICHLORO-	1.06	850	5			
METHANE, TETRACHLORO-						
ETHANE, 1,1,1-TRICHLORO-	22.48	987	5			
BENZENE	5.96	972	6			
PROPANE, 1,2-DICHLORO-						
METHANE, BROMODICHLORO-						
1-PROPENE, 1,3-DICHLORO-, (Z)-						
BENZENE, METHYL-	16.78	988	-2			
1-PROPENE, 1,3-DICHLORO-, (E)-						
ETHANE, 1,1,2-TRICHLORO-						
METHANE, DIBROMOCHLORO-						
ETHENE, TETRACHLORO-	39.50	980	11			
BENZENE, CHLORO-						
BENZENE, ETHYL-	0.34	994	-9			
METHANE, TRIBROMO-						
ETHANE, 1,1,2,2-TETRACHLORO-						
BENZENE, 1,4-DICHLORO-						

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=probable artifact

ANALYSIS REPORT PURGABLE ORGANICS
Sample: Renton, Secondary Effluent
PS85V2
Lab: U of W
Sample Size: 1 ml
Date Sampled:

Quantitation Standard: Indicated by (*)
Quantitation Method: GC/MS

COMPOUND	PPB	FIT	ΔT	PPB	FIT	ΔT
D5-CHLOROETHANE	5.74	923	11			
D3-ACRYLONITRILE	35.30	988	-1			
*D6-2,2-DICHLOROPROPANE	27.80	994	0			
D1-CHLOROFORM	56.15	999	1			
D3-1,1,1-TRICHLOROETHANE	31.74	988	-9			
D6-BENZENE	19.34	984	-13			
*D8-TOLUENE	21.70	998	0			
*D1-BROMOFORM	72.30	983	0			
D4-1,4-DICHLOROBENZENE						
METHANE, DICHLORODIFLUORO-						
ETHENE, CHLORO-						
METHANE, CHLORO-						
METHANE, BROMO-						
ETHANE, CHLORO-						
METHANE, TRICHLOROFLUORO-						
ETHENE, 1,1-DICHLORO-						
2-PROPENAL						
2-PROPENENITRILE						
PROPANE, 2,2-DICHLORO-						
METHANE, DICHLORO-	130.52	982	1			
ETHENE, 1,2-DICHLORO-, (Z)-						
ETHANE, 1,1-DICHLORO-						
METHANE, TRICHLORO-	5.56	839	-2			
ETHENE, TRICHLORO-						
ETHANE, 1,2-DICHLORO-	1.74	931	-7			
METHANE, TETRACHLORO-	0.16	965	-4			
ETHANE, 1,1,1-TRICHLORO-						
BENZENE	8.92	979	-5			
PROPANE, 1,2-DICHLORO-						
METHANE, BROMODICHLORO-						
1-PROPENE, 1,3-DICHLORO-, (Z)-						
BENZENE, METHYL-						
1-PROPENE, 1,3-DICHLORO-, (E)-						
ETHANE, 1,1,2-TRICHLORO-						
METHANE, DIBROMOCHLORO-						
ETHENE, TETRACHLORO-	11.68	979	1			
BENZENE, CHLORO-						
BENZENE, ETHYL-						
METHANE, TRIBROMO-						
ETHANE, 1,1,2,2-TETRACHLORO-						
BENZENE, 1,4-DICHLORO-						

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=probable artifact

ANALYSIS REPORT PURGABLE ORGANICS

Sample: Renton, **Secondary** effluent, dechlorinated
PS76V1

Lab: U of W

Sample Size: 1 ml

Date Sampled:

Quantitation Standard: Indicated by (*)

Quantitation Method: GC/MS

COMPOUND	PPB	FIT	ΔT	PPB	FIT	ΔT
D5-CHLOROETHANE	98.16	990	-2			
D3-ACRYLONITRILE						
*D6-2,2-DICHLOROPROPANE	27.80	992	0			
D1-CHLOROFORM						
D3-1,1,1-TRICHLOROETHANE	35.75	988	15			
D6-BENZENE	24.61	944	17			
*D8-TOLUENE	21.70	998	0			
*D1-BROMOFORM	72.30	988	0			
D4-1,4-DICHLOROBENZENE	51.61	985	3			
METHANE, DICHLORODIFLUORO-						
ETHENE, CHLORO-						
METHANE, CHLORO-						
METHANE, BROMO-						
ETHANE, CHLORO-						
METHANE, TRICHLOROFLUORO-	3.08	759	-2			
ETHENE, 1,1-DICHLORO-						
2-PROPENAL						
2-PROPENENITRILE						
PROPANE, 2,2-DICHLORO-						
METHANE, DICHLORO-	585.68	<921	-7			
ETHENE, 1,2-DICHLORO-, (Z)-						
ETHANE, 1,1-DICHLORO-						
METHANE, TRICHLORO-	8.38	844	5			
ETHENE, TRICHLORO-						
ETHANE, 1,2-DICHLORO-	3.00	934	15			
METHANE, TETRACHLORO-						
ETHANE, 1,1,1-TRICHLORO-	18.10	995	15			
BENZENE	17.36	990	17			
PROPANE, 1,2-DICHLORO-						
METHANE, BROMODICHLORO-						
1-PROPENE, 1,3-DICHLORO-, (Z)-						
BENZENE, METHYL-	1.70	817	-3			
1-PROPENE, 1,3-DICHLORO-, (E)-						
ETHANE, 1,1,2-TRICHLORO-						
METHANE, DIBROMOCHLORO-						
ETHENE, TETRACHLORO-	12.70	939	-4			
BENZENE, CHLORO-						
BENZENE, ETHYL-	0.36	801	4			
METHANE, TRIBROMO-						
ETHANE, 1,1,2,2-TETRACHLORO-						
BENZENE, 1,4-DICHLORO-						

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=probable artifact

ANALYSIS REPORT PURGABLE ORGANICS
Sample: Renton, Prim.+ Waste Act.Sludge
PS115V1
Lab: U of W
Sample Size: 1 ml
Date Sampled:

Quantitation Standard: Indicated by (*)
Quantitation Method: GC/MS

COMPOUND		PPB	FIT	ΔT	PPB	FIT	ΔT
D5-CHLOROETHANE		2.97	954	5			
D3-ACRYLONITRILE							
*D6-2,2-DICHLOROPROPANE		27.80	929	0			
D1-CHLOROFORM		33.09	985	-4			
D3-1,1,1-TRICHLOROETHANE		34.13	880	10			
D6-BENZENE		17.34	985	-13			
*D8-TOLUENE		21.70	998	0			
*D1-BROMOFORM		72.30	955	0			
D4-1,4-DICHLOROBENZENE	A	193.10	985	18			
METHANE, DICHLORODIFLUORO-							
ETHENE, CHLORO-							
METHANE, CHLORO-							
METHANE, BROMO-							
ETHANE, CHLORO-							
METHANE, TRICHLOROFLUORO-							
ETHENE, 1,1-DICHLORO-							
2-PROPENAL							
2-PROPENENITRILE							
PROPANE, 2,2-DICHLORO-							
METHANE, DICHLORO-							
ETHENE, 1,2-DICHLORO-, (Z)-		3.24	806	7			
ETHANE, 1,1-DICHLORO-		24.10	975	-8			
METHANE, TRICHLORO-		9.08	760	-5			
ETHENE, TRICHLORO-							
ETHANE, 1,2-DICHLORO-							
METHANE, TETRACHLORO-		0.08	762	-12			
ETHANE, 1,1,1-TRICHLORO-		24.10	975	-8			
BENZENE		10.30	855	-14			
PROPANE, 1,2-DICHLORO-							
METHANE, BROMODICHLORO-							
1-PROPENE, 1,3-DICHLORO-, (Z)-							
BENZENE, METHYL-		71.54	992	-1			
1-PROPENE, 1,3-DICHLORO-, (E)-							
ETHANE, 1,1,2-TRICHLORO-							
METHANE, DIBROMOCHLORO-							
ETHENE, TETRACHLORO-		76.34	942	6			
BENZENE, CHLORO-							
BENZENE, ETHYL-		3.02	986	-4			
METHANE, TRIBROMO-							
ETHANE, 1,1,2,2-TETRACHLORO-		4.36	815	8			
BENZENE, 1,4-DICHLORO-							

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=probable artifact

ANALYSIS REPORT PURGABLE ORGANICS
Sample: Atlanta, Primary Effluent
PS126V1
Lab: U of W
Sample Size: 1 ml
Date Sampled:

Quantitation Standard: Indicated by (*)
Quantitation Method: GC/MS

COMPOUND	PPB	FIT	ΔT	PPB	FIT	ΔT
D5-CHLOROETHANE	317.99					
D3-ACRYLONITRILE	4.53					
*D6-2,2-DICHLOROPROPANE	11.22					
D1-CHLOROFORM	15.45					
D3-1,1,1-TRICHLOROETHANE						
D6-BENZENE	9.25					
*D8-TOLUENE	8.76					
*D1-BROMOFORM	29.18					
D4-1,4-DICHLOROBENZENE						
METHANE, DICHLORODIFLUORO-	3.71					
ETHENE, CHLORO-						
METHANE, CHLORO-						
METHANE, BROMO-						
ETHANE, CHLORO-						
METHANE, TRICHLOROFLUORO-	23.72					
ETHENE, 1,1-DICHLORO-	17.42					
2-PROPENAL						
2-PROPENENITRILE						
PROPANE, 2,2-DICHLORO-						
METHANE, DICHLORO-	261.96					
ETHENE, 1,2-DICHLORO-, (Z)-	0.36					
ETHANE, 1,1-DICHLORO-	7.71					
METHANE, TRICHLORO-	6.91					
ETHENE, TRICHLORO-						
ETHANE, 1,2-DICHLORO-	3.37					
METHANE, TETRACHLORO-						
ETHANE, 1,1,1-TRICHLORO-	723.05					
BENZENE	3.11					
PROPANE, 1,2-DICHLORO-						
METHANE, BROMODICHLORO-	0.27					
1-PROPENE, 1,3-DICHLORO-, (Z)-						
BENZENE, METHYL-	28.30					
1-PROPENE, 1,3-DICHLORO-, (E)-						
ETHANE, 1,1,2-TRICHLORO-						
METHANE, DIBROMOCHLORO-						
ETHENE, TETRACHLORO-	224.42					
BENZENE, CHLORO-						
BENZENE, ETHYL-						
METHANE, TRIBROMO-						
ETHANE, 1,1,2,2-TETRACHLORO-						
BENZENE, 1,4-DICHLORO-						

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=probable artifact

ANALYSIS REPORT — PURGABLE ORGANICS

Sample: PS176 — Atlanta, Secondary Effluent before Chlorination

Lab:
Sample Size:
Date Sampled:

Quantitation Standard:
Quantitation Method:

COMPOUND		PPB	FIT	ΔT	PPB	FIT	ΔT
D5-CHLOROETHANE		19.63	953	-14			
D3-ACRYLONITRILE							
D6-2,2-DICHLOROPROPANE		11.12	939	0			
D1-CHLOROFORM		13.539	989	0			
D3-1,1,1-TRICHLOROETHANE		13.51	870	1			
D5-BENZENE		15.00	989	3			
D8-TOLUENE		8.67	976	0			
D1-BROMOFORM		28.9	945	0			
D4-1,4-DICHLOROBENZENE		3.50	958				
METHANE, DICHLORODIFLUORO-							
ETHENE, CHLORO-							
METHANE, CHLORO-							
METHANE, BROMO-							
ETHANE, CHLORO-							
METHANE, TRICHLOROFLUORO-	A	5.948	877	+20			
ETHENE, 1,1-DICHLORO-	A	1.251	<757	-2			
2-PROPENAL							
2-PROPENENITRILE							
PROPANE, 2,2-DICHLORO-							
METHANE, DICHLORO-		182.54	947	-2			
ETHENE, 1,2-DICHLORO-, (Z)-							
ETHANE, 1,1-DICHLORO-		0.56	788	-2			
METHANE, TRICHLORO-		0.38	<863	0			
ETHENE, TRICHLORO-		2.49	968	+9			
ETHANE, 1,2-DICHLORO-		1.27	851	+3			
METHANE, TETRACHLORO-							
ETHANE, 1,1,1-TRICHLORO-		36.12	986	+1			
BENZENE		0.69	871	+3			
PROPANE, 1,2-DICHLORO-							
METHANE, BROMODICHLORO-							
1-PROPENE, 1,3-DICHLORO-, (Z)-							
BENZENE, METHYL-		0.68	907	+2			
1-PROPENE, 1,3-DICHLORO-, (E)-							
ETHANE, 1,1,2-TRICHLORO-							
METHANE, DIBROMOCHLORO-							
ETHENE, TETRACHLORO-		5.32	978	+18			
BENZENE, CHLORO-							
BENZENE, ETHYL-							
METHANE, TRIBROMO-							
ETHANE, 1,1,2,2-TETRACHLORO-							
BENZENE, 1,4-DICHLORO-		3.50	<934	15			

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=probable artifact

ANALYSIS REPORT — PURGABLE ORGANICS

Sample: PS167 — Atlanta, Secondary Effluent after Chlorination

Lab:
Sample Size:
Date Sampled:

Quantitation Standard:
Quantitation Method:

COMPOUND	PPB	FIT	ΔT	PPB	FIT	ΔT
D5-CHLOROETHANE	19.26	951	14			
D3-ACRYLONITRILE						
D6-2,2-DICHLOROPROPANE	11.12	945	0			
D1-CHLOROFORM	13.73	972	-1			
D3-1,1,1-TRICHLOROETHANE	13.50	878	-3			
D6-BENZENE	14.83	997	-5			
D8-TOLUENE	50.27	981	-10			
D1-BROMOFORM	28.90	944	1			
D4-1,4-DICHLOROBENZENE	8.98	853	-5			
METHANE, DICHLORODIFLUORO-						
ETHENE, CHLORO-						
METHANE, CHLORO-						
METHANE, BROMO-						
ETHANE, CHLORO-						
METHANE, TRICHLOROFLUORO-						
ETHENE, 1,1-DICHLORO-	0.65	762	+9			
2-PROPENAL						
2-PROPENENITRILE						
PROPANE, 2,2-DICHLORO-						
METHANE, DICHLORO-	140.59	976	+7			
ETHENE, 1,2-DICHLORO-, (Z)-						
ETHANE, 1,1-DICHLORO-						
METHANE, TRICHLORO-	0.564	<865	-2			
ETHENE, TRICHLORO-	1.77	964	-8			
ETHANE, 1,2-DICHLORO-	1.13	800	-4			
METHANE, TETRACHLORO-						
ETHANE, 1,1,1-TRICHLORO-	30.82	982	-4			
BENZENE	1.08	901	-4			
PROPANE, 1,2-DICHLORO-						
METHANE, BROMODICHLORO-						
1-PROPENE, 1,3-DICHLORO-, (Z)-						
BENZENE, METHYL-	1.51	971	+1			
1-PROPENE, 1,3-DICHLORO-, (E)-						
ETHANE, 1,1,2-TRICHLORO-						
METHANE, DIBROMOCHLORO-						
ETHENE, TETRACHLORO-	4.37	955	-2			
BENZENE, CHLORO-						
BENZENE, ETHYL-	1.269	943	-1			
METHANE, TRIBROMO-						
ETHANE, 1,1,2,2-TETRACHLORO-						
BENZENE, 1,4-DICHLORO-						

blank=not found; NQ=detected but not quantitated; NA=not analyzed for

ANALYSIS REPORT PURGABLE ORGANICS
Sample: Oakland, Raw Sewage
PS205V1
Lab: U of W
Sample Size: 1 ml
Date Sampled:

Quantitation Standard: Indicated by (*)
Quantitation Method: GC/MS

COMPOUND	PPB	FIT	ΔT	PPB	FIT	ΔT
D5-CHLOROETHANE						
D3-ACRYLONITRILE	38.49	987	-7			
*D6-2,2-DICHLOROPROPANE	11.12	991	0			
D1-CHLOROFORM	24.09	984	1			
D3-1,1,1-TRICHLOROETHANE	13.68	987	3			
D6-BENZENE	11.69	990	4			
*D8-TOLUENE	8.67	994	-1			
*D1-BROMOFORM	28.90	980	0			
D4-1,4-DICHLOROBENZENE	19.45	993	-4			
METHANE, DICHLORODIFLUORO-	NQ					
ETHENE, CHLORO-						
METHANE, CHLORO-						
METHANE, BROMO-						
ETHANE, CHLORO-						
METHANE, TRICHLOROFLUORO-						
ETHENE, 1,1-DICHLORO-						
2-PROPENAL						
2-PROPENENITRILE						
PROPANE, 2,2-DICHLORO-						
METHANE, DICHLORO-	4.63	987	-6			
ETHENE, 1,2-DICHLORO-, (Z)-						
ETHANE, 1,1-DICHLORO-	1.57	951	-4			
METHANE, TRICHLORO-	19.13	830	2			
ETHENE, TRICHLORO-	21.10	982	1			
ETHANE, 1,2-DICHLORO-	2.02	909	5			
METHANE, TETRACHLORO-						
ETHANE, 1,1,1-TRICHLORO-	26.86	982	4			
BENZENE						
PROPANE, 1,2-DICHLORO-	1.20	600	4			
METHANE, BROMODICHLORO-						
1-PROPENE, 1,3-DICHLORO-, (Z)-						
BENZENE, METHYL-	0.95	990	-2			
1-PROPENE, 1,3-DICHLORO-, (E)-						
ETHANE, 1,1,2-TRICHLORO-						
METHANE, DIBROMOCHLORO-						
ETHENE, TETRACHLORO-	10.54	966	-2			
BENZENE, CHLORO-	0.17	781	5			
BENZENE, ETHYL-	0.33	847	3			
METHANE, TRIBROMO-	1.29	708	-1			
ETHANE, 1,1,2,2-TETRACHLORO-						
BENZENE, 1,4-DICHLORO-						

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=Probable artifact

ANALYSIS REPORT PURGABLE ORGANICS
Sample: Oakland, Primary Effluent
PS215V1
Lab: U of W
Sample Size: 1 ml
Date Sampled:

Quantitation Standard: Indicated by (*)
Quantitation Method: GC/MS

COMPOUND	PPB	FIT	ΔT	PPB	FIT	ΔT
D5-CHLOROETHANE						
D3-ACRYLONITRILE	34.48	971	1			
*D6-2,2-DICHLOROPROPANE	11.12	969	0			
D1-CHLOROFORM	16.64	873	1			
D3-1,1,1-TRICHLOROETHANE	12.88	975	0			
D6-BENZENE	8.67	989	1			
*D8-TOLUENE	8.67	994	1			
*D1-BROMOFORM	28.90	975	-1			
D4-1,4-DICHLOROBENZENE	15.22	993	3			
METHANE, DICHLORODIFLUORO-	3.45	619	0			
ETHENE, CHLORO-						
METHANE, CHLORO-						
METHANE, BROMO-						
ETHANE, CHLORO-						
METHANE, TRICHLOROFLUORO-						
ETHENE, 1,1-DICHLORO-						
2-PROPENAL						
2-PROPENENITRILE						
PROPANE, 2,2-DICHLORO-						
METHANE, DICHLORO-	7.34	982	3			
ETHENE, 1,2-DICHLORO-, (Z)-						
ETHANE, 1,1-DICHLORO-	1.94	891	0			
METHANE, TRICHLORO-	34.19	870	0			
ETHENE, TRICHLORO-	65.11	978	-4			
ETHANE, 1,2-DICHLORO-	5.71	934	-5			
METHANE, TETRACHLORO-						
ETHANE, 1,1,1-TRICHLORO-	34.58	971	-5			
BENZENE						
PROPANE, 1,2-DICHLORO-						
METHANE, BROMODICHLORO-	0.89	847	-5			
1-PROPENE, 1,3-DICHLORO-, (Z)-						
BENZENE, METHYL-	0.99	967	0			
1-PROPENE, 1,3-DICHLORO-, (E)-						
ETHANE, 1,1,2-TRICHLORO-						
METHANE, DIBROMOCHLORO-						
ETHENE, TETRACHLORO-	25.22	966	11			
BENZENE, CHLORO-						
BENZENE, ETHYL-	0.81	<909	1			
METHANE, TRIBROMO-						
ETHANE, 1,1,2,2-TETRACHLORO-						
BENZENE, 1,4-DICHLORO-						

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=Probable Artifact

ANALYSIS REPORT PURGABLE ORGANICS
Sample: Oakland, Secondary Effluent
PS185V1
Lab: U of W
Sample Size: 1 ml
Date Sampled:

Quantitation Standard: Indicated by (*)
Quantitation Method: GC/MS

COMPOUND	PPB	FIT	ΔT	PPB	FIT	ΔT
D5-CHLOROETHANE	8.78	984	-2			
D3-ACRYLONITRILE	10.48	964	-3			
*D6-2,2-DICHLOROPROPANE	11.12	940	0			
D1-CHLOROFORM	13.78	853	1			
D3-1,1,1-TRICHLOROETHANE	18.36	975	-10			
D6-BENZENE	6.80	991	6			
*D8-TOLUENE	8.67	992	0			
*D1-BROMOFORM	28.90	945	0			
D4-1,4-DICHLOROBENZENE	15.25	747	-14			
METHANE, DICHLORODIFLUORO-						
ETHENE, CHLORO-						
METHANE, CHLORO-						
METHANE, BROMO-						
ETHANE, CHLORO-	0.89	713	-2			
METHANE, TRICHLOROFLUORO-	34.47	906	-1			
ETHENE, 1,1-DICHLORO-	1.83	850	-1			
2-PROPENAL						
2-PROPENENITRILE						
PROPANE, 2,2-DICHLORO-						
METHANE, DICHLORO-	9.03	973	-2			
ETHENE, 1,2-DICHLORO-, (Z)-						
ETHANE, 1,1-DICHLORO-	1.74	939	-4			
METHANE, TRICHLORO-	37.28	939	1			
ETHENE, TRICHLORO-	11.22	991	13			
ETHANE, 1,2-DICHLORO-	0.46	758	6			
METHANE, TETRACHLORO-	0.59	958	6			
ETHANE, 1,1,1-TRICHLORO-	88.37	969	5			
BENZENE	10.46	991	6			
PROPANE, 1,2-DICHLORO-						
METHANE, BROMODICHLORO-	1.47	928	-2			
1-PROPENE, 1,3-DICHLORO-, (Z)-						
BENZENE, METHYL-	112.41	989	3			
1-PROPENE, 1,3-DICHLORO-, (E)-						
ETHANE, 1,1,2-TRICHLORO-						
METHANE, DIBROMOCHLORO-						
ETHENE, TETRACHLORO-	49.25	995	-4			
BENZENE, CHLORO-	117.25	992	-3			
BENZENE, ETHYL-	148.27	989	0			
METHANE, TRIBROMO-						
ETHANE, 1,1,2,2-TETRACHLORO-						
BENZENE, 1,4-DICHLORO-						

blank=not found; NQ=detected but not quantitated; NA=not analyzed for

A=probable artifact

ANALYSIS REPORT PURGABLE ORGANICS
Sample: Oakland, Secondary Effluent, dechlorinated
PS195V2
Lab: U of W
Sample Size: 1 ml
Date Sampled:

Quantitation Standard: Indicated by (*)
Quantitation Method: GC/MS

COMPOUND	PPB	FIT	ΔT	PPB	FIT	ΔT
D5-CHLOROETHANE	3.31	965	1			
D3-ACRYLONITRILE	9.29	945	-3			
*D6-2,2-DICHLOROPROPANE	11.12	987	-1			
D1-CHLOROFORM	15.71	981	0			
D3-1,1,1-TRICHLOROETHANE	12.46	963	3			
D6-BENZENE	7.59	995	6			
*D8-TOLUENE	8.67	992	0			
*D1-BROMOFORM	28.90	946	0			
D4-1,4-DICHLOROBENZENE	47.11	843	-2			
METHANE, DICHLORODIFLUORO-	NQ					
ETHENE, CHLORO-						
METHANE, CHLORO-						
METHANE, BROMO-						
ETHANE, CHLORO-						
METHANE, TRICHLOROFLUORO-						
ETHENE, 1,1-DICHLORO-						
2-PROPENAL						
2-PROPENENITRILE						
PROPANE, 2,2-DICHLORO-						
METHANE, DICHLORO-						
ETHENE, 1,2-DICHLORO-, (Z)-	1.64	995	-2			
ETHANE, 1,1-DICHLORO-						
METHANE, TRICHLORO-	6.0	813	0			
ETHENE, TRICHLORO-	6.16	980	12			
ETHANE, 1,2-DICHLORO-						
METHANE, TETRACHLORO-						
ETHANE, 1,1,1-TRICHLORO-	11.65	958	4			
BENZENE	3.05	948	6			
PROPANE, 1,2-DICHLORO-						
METHANE, BROMODICHLORO-						
1-PROPENE, 1,3-DICHLORO-, (Z)-						
BENZENE, METHYL-	13.73	981	-1			
1-PROPENE, 1,3-DICHLORO-, (E)-						
ETHANE, 1,1,2-TRICHLORO-						
METHANE, DIBROMOCHLORO-						
ETHENE, TETRACHLORO-	12.41	974	-2			
BENZENE, CHLORO-	15.09	861	-3			
BENZENE, ETHYL-	34.54	991	0			
METHANE, TRIBROMO-	3.12	643	0			
ETHANE, 1,1,2,2-TETRACHLORO-	2.56	647	-5			
BENZENE, 1,4-DICHLORO-						

blank=not found; NQ=detected but not quantitated; NA=not analyzed for

A=probable artifact

ANALYSIS REPORT PURGABLE ORGANICS
Sample: Oakland, Digested Sludge
PS220
Lab: U of W
Sample Size: 1 ml
Date Sampled:

Quantitation Standard: Indicated by (*)
Quantitation Method: GC/MS

COMPOUND	PPB	FIT	ΔT	PPB	FIT	ΔT
D5-CHLOROETHANE						
D3-ACRYLONITRILE	43.49	974	0			
*D6-2,2-DICHLOROPROPANE	11.12	980	0			
D1-CHLOROFORM	17.29	993	-1			
D3-1,1,1-TRICHLOROETHANE	14.19	973	0			
D6-BENZENE	9.23	987	2			
*D8-TOLUENE	8.67	994	0			
*D1-BROMOFORM	28.90	970	1			
D4-1,4-DICHLOROBENZENE						
METHANE, DICHLORODIFLUORO-						
ETHENE, CHLORO-						
METHANE, CHLORO-						
METHANE, BROMO-						
ETHANE, CHLORO-						
METHANE, TRICHLOROFLUORO-						
ETHENE, 1,1-DICHLORO-	1.05	706	6			
2-PROPENAL						
2-PROPENENITRILE						
PROPANE, 2,2-DICHLORO-						
METHANE, DICHLORO-	0.24	980	2			
ETHENE, 1,2-DICHLORO-, (Z)-	NQ					
ETHANE, 1,1-DICHLORO-	2.34	871	-1			
METHANE, TRICHLORO-						
ETHENE, TRICHLORO-						
ETHANE, 1,2-DICHLORO-	1.17	620	6			
METHANE, TETRACHLORO-						
ETHANE, 1,1,1-TRICHLORO-						
BENZENE	12.43	982	1			
PROPANE, 1,2-DICHLORO-						
METHANE, BROMODICHLORO-						
1-PROPENE, 1,3-DICHLORO-, (Z)-						
BENZENE, METHYL-	126.18	990	9			
1-PROPENE, 1,3-DICHLORO-, (E)-						
ETHANE, 1,1,2-TRICHLORO-						
METHANE, DIBROMOCHLORO-						
ETHENE, TETRACHLORO-						
BENZENE, CHLORO-	22.77	956	-3			
BENZENE, ETHYL-	160.64	997	1			
METHANE, TRIBROMO-	7.63	788	1			
ETHANE, 1,1,2,2-TETRACHLORO-	NQ					
BENZENE, 1,4-DICHLORO-						

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=Probable artifact

ANALYSIS REPORT — BASE + NEUTRAL EXTRACTABLES
Sample: Renton, Primary Effluent
PS90N1
Lab: U of W
Sample Size: 1 liter
Date Sampled

Quantitation Standard: D_8Naphthalene
Quantitation Method: GC/MS

COMPOUND		PPB	FIT	ΔT	PPB	FIT	ΔT
N-NITROSODIMETHYLAMINE							
BIS(2-CHLOROETHYL)ETHER		0.07	890	-6			
(1,3-)DICHLOROBENZENE		0.56	991	-5			
1,4-DICHLOROBENZENE		3.57	<995	-6			
(1,2-)DICHLOROBENZENE		4.83	997	-5			
BIS(2-CHLOROISOPROPYL)ETHER		0.32	<803	-3			
HEXACHLOROETHANE							
N-NITROSO-DI-N-PROPYL AMINE							
NITROBENZENE		0.05	<845	0			
BIS(2-CHLOROETHOXY)METHANE		0.13	816	3			
1,2,4-TRICHLOROBENZENE	A	0.21	738	4			
NAPHTHALENE		1.18	990	0			
HEXACHLOROBUTADIENE							
2-CHLORO-NAPHTHALANE							
ACENAPHTHYLENE							
DIMETHYLPHTHALATE		0.52	998	14			
2,6-DINITROTOLUENE	A	0.18	628	17			
ACENAPHTHENE		0.06	827	23			
2,4-DINITROTOLUENE							
FLUORENE							
DIETHYLPHTHALATE		7.44	996	-4			
AZOBENZENE (FROM DIPHENYLHYDRAZINE)							
N-NITROSODIPHENYLAMINE							
4-BROMODIPHENYL ETHER							
HEXACHLOROBENZENE							
PHENANTHRENE		0.14	963	2			
ANTHRACENE		0.01	<959	1			
DI-N-BUTYLPHTHALATE		3.01	998	15			
FLUORANTHENE	A	0.02	<650	14			
PYRENE							
BUTYLBENZYLPHTHALATE		2.59	985	27			
BENZO(A)ANTHRACENE							
CHRYSENE							
3,3'-DICHLOROBENZIDENE							
BIS(2-ETHYLHEXYL)PHTHALATE							
DI-N-OCTYL PHTHALATE							
BENZO(B)FLUORANTHENE							
BENZO(K)FLUORANTHENE							
BENZO(A)PYRENE							
INDENO(1,2,3-CD)							
DIBENZO(A,H)ANTHRACENE							
BENZO(GHI)PERYLENE							
CHLOROPHENYL,PHENYL ETHER		NA					
HEXACHLOROCYCLOPENTADIENE		NA					

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=probable artifact

ANALYSIS REPORT BASE + NEUTRAL EXTRACTABLES
Sample: Renton, Secondary Effluent before chlorination
PS80
Lab: U of W
Sample Size: 1 liter
Date Sampled

Quantitation Standard: D_8Naphthalene
Quantitation Method: GC/MS

COMPOUND	PPB	FIT	ΔT	PPB	FIT	ΔT
N-NITROSODIMETHYLAMINE						
BIS(2-CHLOROETHYL) ETHER						
(1,3-)DICHLOROBENZENE	0.02	791	7			
1,4-DICHLOROBENZENE	0.74	945	5			
(1,2-)DICHLOROBENZENE	2.95	994	6			
BIS(2-CHLOROISOPROPYL)ETHER	0.06	839	0			
HEXACHLOROETHANE						
N-NITROSO-DI-N-PROPYL AMINE	0.06	<812	-2			
NITROBENZENE						
BIS(2-CHLOROETHOXY)METHANE	0.02	848	6			
1,2,4-TRICHLOROBENZENE						
NAPHTHALENE	0.21	986	1			
HEXACHLOROBUTADIENE						
2-CHLORO-NAPHTHALANE						
ACENAPHTHYLENE						
DIMETHYLPHTHALATE						
2,6-DINITROTOLUENE						
ACENAPHTHENE						
2,4-DINITROTOLUENE						
FLUORENE						
DIETHYLPHTHALATE	0.16	983	0			
AZOBENZENE (FROM DIPHENYLHYDRAZINE)						
N-NITROSODIPHENYLAMINE						
4-BROMODIPHENYL ETHER						
HEXACHLOROBENZENE						
PHENANTHRENE	0.40	942	2			
ANTHRACENE	NQ					
DI-N-BUTYLPHTHALATE	1.79	992	0			
FLUORANTHENE						
PYRENE						
BUTYLBENZYLPHTHALATE						
BENZO(A)ANTHRACENE						
CHRYSENE						
3,3'-DICHLOROBENZIDENE						
BIS(2-ETHYLHEXYL)PHTHALATE	0.92	839	-4			
DI-N-OCTYL PHTHALATE						
BENZO(B)FLUORANTHENE						
BENZO(K)FLUORANTHENE						
BENZO(A)PYRENE						
INDENO(1,2,3-CD)						
DIBENZO(A,H)ANTHRACENE						
BENZO(GHI)PERYLENE						
CHLOROPHENYL,PHENYL ETHER	NA					
HEXACHLOROCYCLOPENTADIENE	NA					

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=probable artifact

ANALYSIS REPORT BASE + NEUTRAL EXTRACTABLES
Sample: Renton, Secondary Effluent after dechlorination
PS70
Lab: U of W
Sample Size: 1 liter
Date Sampled

Quantitation Standard: DgNaphthalene
Quantitation Method: GC/MS

COMPOUND	PPB	FIT	ΔT	PPB	FIT	ΔT
N-NITROSODIMETHYLAMINE						
BIS(2-CHLOROETHYL)ETHER						
(1,3-)DICHLOROBENZENE	NQ					
1,4-DICHLOROBENZENE	1.93	834	-8			
(1,2-)DICHLOROBENZENE	3.88	994	-6			
BIS(2-CHLOROISOPROPYL)ETHER						
HEXACHLOROETHANE						
N-NITROSO-DI-N-PROPYL AMINE	0.31	<841	-6			
NITROBENZENE						
BIS(2-CHLOROETHOXY)METHANE						
1,2,4-TRICHLOROBENZENE						
NAPHTHALENE						
HEXACHLOROBUTADIENE						
2-CHLORO-NAPHTHALANE						
ACENAPHTHYLENE						
DIMETHYLPHTHALATE						
2,6-DINITROTOLUENE						
ACENAPHTHENE						
2,4-DINITROTOLUENE						
FLUORENE						
DIETHYLPHTHALATE						
AZOBENZENE (FROM DIPHENYLHYDRAZINE)						
N-NITROSODIPHENYLAMINE						
4-BROMODIPHENYL ETHER						
HEXACHLOROBENZENE						
PHENANTHRENE						
ANTHRACENE						
DI-N-BUTYLPHTHALATE	5.41	971	-2			
FLUORANTHENE						
PYRENE						
BUTYLBENZYLPHTHALATE						
BENZO(A)ANTHRACENE						
CHRYSENE						
3,3'-DICHLOROBENZIDENE						
BIS(2-ETHYLHEXYL)PHTHALATE	12.75	976	6			
DI-N-OCTYL PHTHALATE						
BENZO(B)FLUORANTHENE						
BENZO(K)FLUORANTHENE						
BENZO(A)PYRENE						
INDENO(1,2,3-CD)						
DIBENZO(A,H)ANTHRACENE						
BENZO(GHI)PERYLENE						
CHLOROPHENYL,PHENYL ETHER	NA					
HEXACHLOROCYCLOPENTADIENE	NA					

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
▲=probable artifact

ANALYSIS REPORT — BASE + NEUTRAL EXTRACTABLES

Sample: Atlanta, Raw Sewage
PS150N2
Lab: U of W
Sample Size: 1 liter
Date Sampled

Quantitation Standard: D_8Naphthalene
Quantitation Method: GC/MS

COMPOUND		PPB	FIT	ΔT	PPB	FIT	ΔT
N-NITROSODIMETHYLAMINE							
BIS(2-CHLOROETHYL)ETHER							
(1,3-)DICHLOROBENZENE		3.15	<975	2			
1,4-DICHLOROBENZENE		13.94	975	2			
(1,2-)DICHLOROBENZENE		43.86	<982	2			
BIS(2-CHLOROISOPROPYL)ETHER							
HEXACHLOROETHANE							
N-NITROSO-DI-N-PROPYL AMINE	A	NQ	783	>10			
NITROBENZENE		0.12	<844	2			
BIS(2-CHLOROETHOXY)METHANE							
1,2,4-TRICHLOROBENZENE		0.61	788	0			
NAPHTHALENE		10.94	988	0			
HEXACHLOROBUTADIENE							
2-CHLORO-NAPHTHALANE							
ACENAPHTHYLENE		0.25	846	0			
DIMETHYLPHTHALATE							
2,6-DINITROTOLUENE							
ACENAPHTHENE		0.88	942	-2			
2,4-DINITROTOLUENE							
FLUORENE		1.72	979	-2			
DIETHYLPHTHALATE		1.36	950	14			
AZOBENZENE (FROM DIPHENYLHYDRAZINE)							
N-NITROSODIPHENYLAMINE	A	NQ	<862	9			
4-BROMODIPHENYL ETHER							
HEXACHLOROBENZENE							
PHENANTHRENE		3.24	962	16			
ANTHRACENE		NQ	<966	"1"			
DI-N-BUTYLPHTHALATE		12.91	963	11			
FLUORANTHENE			875	21			
PYRENE			875	22			
BUTYLBENZYLPHTHALATE		17.88	962	25			
BENZO(A)ANTHRACENE							
CHRYSENE							
3,3'-DICHLOROBENZIDENE							
BIS(2-ETHYLHEXYL)PHTHALATE							
DI-N-OCTYL PHTHALATE							
BENZO(B)FLUORANTHENE							
BENZO(K)FLUORANTHENE							
BENZO(A)PYRENE							
INDENO(1,2,3-CD)							
DIBENZO(A,H)ANTHRACENE							
BENZO(GHI)PERYLENE							
CHLOROPHENYL,PHENYL ETHER		NA					
HEXACHLOROCYCLOPENTADIENE		NA					

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=probable artifact

ANALYSIS REPORT — BASE + NEUTRAL EXTRACTABLES
Sample: Atlanta, Primary Effluent
PS120
Lab: U of W
Sample Size: 1 liter
Date Sampled

Quantitation Standard: DgNaphthalene
Quantitation Method: GC/MS

COMPOUND		PPB	FIT	ΔT	PPB	FIT	ΔT
N-NITROSODIMETHYLAMINE							
BIS(2-CHLOROETHYL)ETHER		0.36	<882	3			
(1,3-)DICHLOROBENZENE		3.87	991	1			
1,4-DICHLOROBENZENE		14.26	998	0			
(1,2-)DICHLOROBENZENE		52.87	996	1			
BIS(2-CHLOROISOPROPYL)ETHER							
HEXACHLOROETHANE							
N-NITROSO-DI-N-PROPYL AMINE		1.38	<845	-5			
NITROBENZENE							
BIS(2-CHLOROETHOXY)METHANE	A	0.78	<774	-10			
1,2,4-TRICHLOROBENZENE							
NAPHTHALENE		14.55	995	0			
HEXACHLOROBUTADIENE							
2-CHLORO-NAPHTHALANE							
ACENAPHTHYLENE	A	0.23	<792	-7			
DIMETHYLPHTHALATE		0.94	940	-1			
2,6-DINITROTOLUENE	A	0.35	<798	-6			
ACENAPHTHENE		0.63	945	-1			
2,4-DINITROTOLUENE							
FLUORENE		1.05	944	0			
DIETHYLPHTHALATE		5.50	987	13			
AZOBENZENE (FROM DIPHENYLHYDRAZINE)							
N-NITROSODIPHENYLAMINE							
4-BROMODIPHENYL ETHER							
HEXACHLOROBENZENE							
PHENANTHRENE		1.56	994	18			
ANTHRACENE		NQ					
DI-N-BUTYLPHTHALATE		22.92	997	18			
FLUORANTHENE							
PYRENE							
BUTYLBENZYLPHTHALATE		26.07	995	19			
BENZO(A)ANTHRACENE							
CHRYSENE							
3,3'-DICHLOROBENZIDENE							
BIS(2-ETHYLHEXYL)PHTHALATE		26.21	970	19			
DI-N-OCTYL PHTHALATE							
BENZO(B)FLUORANTHENE							
BENZO(K)FLUORANTHENE							
BENZO(A)PYRENE							
INDENO(1,2,3-CD)							
DIBENZO(A,H)ANTHRACENE							
BENZO(GHI)PERYLENE							
CHLOROPHENYL,PHENYL ETHER		NA					
HEXACHLOROCYCLOPENTADIENE		NA					

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=probable artifact

ANALYSIS REPORT BASE + NEUTRAL EXTRACTABLES
Sample: Atlanta, Secondary Effluent
Lab: U of W PS171
Sample Size: 1 liter
Date Sampled

Quantitation Standard: D_8Naphthalene
Quantitation Method: GC/MS

COMPOUND		PPB	FIT	ΔT	PPB	FIT	ΔT
N-NITROSODIMETHYLAMINE							
BIS(2-CHLOROETHYL)ETHER		0.09	950	3			
(1,3-)DICHLOROBENZENE		0.45	935	3			
1,4-DICHLOROBENZENE		2.04	993	1			
(1,2-)DICHLOROBENZENE		1.22	988	3			
BIS(2-CHLOROISOPROPYL)ETHER		0.34	872	-2			
HEXACHLOROETHANE							
N-NITROSO-DI-N-PROPYL AMINE							
NITROBENZENE		0.01	<801	<1			
BIS(2-CHLOROETHOXY)METHANE							
1,2,4-TRICHLOROBENZENE		0.09	883	1			
NAPHTHALENE		4.06	993	1			
HEXACHLOROBUTADIENE							
2-CHLORO-NAPHTHALANE							
ACENAPHTHYLENE		0.04	<864	3			
DIMETHYLPHTHALATE		0.12	858	0			
2,6-DINITROTOLUENE							
ACENAPHTHENE		0.05	802	1			
2,4-DINITROTOLUENE							
FLUORENE		0.12	893	0			
DIETHYLPHTHALATE		0.26	952	-1			
AZOBENZENE (FROM DIPHENYLHYDRAZINE)	A	0.06	<887	-14			
N-NITROSODIPHENYLAMINE							
4-BROMODIPHENYL ETHER							
HEXACHLOROBENZENE							
PHENANTHRENE		0.28	993	1			
ANTHRACENE		0.07	986	3			
DI-N-BUTYLPHTHALATE		4.97	995	-1			
FLUORANTHENE		0.05	899	4			
PYRENE		0.14	976	4			
BUTYLBENZYLPHTHALATE		1.63	975	-2			
BENZO(A)ANTHRACENE							
CHRYSENE							
3,3'-DICHLOROBENZIDENE							
BIS(2-ETHYLHEXYL)PHTHALATE		11.17	969	-3			
DI-N-OCTYL PHTHALATE		0.54	823	-3			
BENZO(B)FLUORANTHENE							
BENZO(K)FLUORANTHENE							
BENZO(A)PYRENE							
INDENO(1,2,3-CD)							
DIBENZO(A,H)ANTHRACENE							
BENZO(GHI)PERYLENE							
CHLOROPHENYL,PHENYL ETHER		NA					
HEXACHLOROCYCLOPENTADIENE		NA					

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=probable artifact

ANALYSIS REPORT BASE + NEUTRAL EXTRACTABLES
Sample: Atlanta discharge, Secondary Effluent
PS16ON after chlorination
Lab: U of W
Sample Size: 1 liter
Date Sampled

Quantitation Standard: DgNaphthalene
Quantitation Method: GC/MS

COMPOUND		PPB	FIT	ΔT	PPB	FIT	ΔT
N-NITROSODIMETHYLAMINE							
BIS(2-CHLOROETHYL)ETHER							
(1,3-)DICHLOROBENZENE		1.32	996	9			
1,4-DICHLOROBENZENE		5.74	<996	7			
(1,2-)DICHLOROBENZENE		10.12	997	8			
BIS(2-CHLOROISOPROPYL)ETHER		0.13	<862	2			
HEXACHLOROETHANE							
N-NITROSO-DI-N-PROPYL AMINE		0.11	840	-1			
NITROBENZENE							
BIS(2-CHLOROETHOXY)METHANE							
1,2,4-TRICHLOROBENZENE		0.34	963	1			
NAPHTHALENE		0.37	952	1			
HEXACHLOROBUTADIENE							
2-CHLORO-NAPHTHALANE							
ACENAPHTHYLENE							
DIMETHYLPHTHALATE		0.12	869	-16			
2,6-DINITROTOLUENE							
ACENAPHTHENE		0.04	835	-16			
2,4-DINITROTOLUENE							
FLUORENE							
DIETHYLPHTHALATE		0.25	941	2			
AZOBENZENE (FROM DIPHENYLHYDRAZINE)							
N-NITROSODIPHENYLAMINE		0.20	807	1			
4-BROMODIPHENYL ETHER							
HEXACHLOROBENZENE							
PHENANTHRENE		0.28	984	1			
ANTHRACENE		NQ					
DI-N-BUTYLPHTHALATE		1.82	991	-9			
FLUORANTHENE		0.06	782	-3			
PYRENE		0.18	956	-5			
BUTYLBENZYLPHTHALATE	A	3.68	992	-17			
BENZO(A)ANTHRACENE							
CHRYSENE							
3,3'-DICHLOROBENZIDENE							
BIS(2-ETHYLHEXYL)PHTHALATE							
DI-N-OCTYL PHTHALATE							
BENZO(B)FLUORANTHENE							
BENZO(K)FLUORANTHENE							
BENZO(A)PYRENE							
INDENO(1,2,3-CD)							
DIBENZO(A,H)ANTHRACENE							
BENZO(GHI)PERYLENE							
CHLOROPHENYL,PHENYL ETHER		NA					
HEXACHLOROCYCLOPENTADIENE		NA					

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=probable artifact

ANALYSIS REPORT — BASE + NEUTRAL EXTRACTABLES

Sample: Atlanta, Digested Sludge
PS141
Lab: U of W
Sample Size: 50 ml
Date Sampled

Quantitation Standard: DgNaphthalene
Quantitation Method: GC/MS

COMPOUND		PPB	FIT	ΔT	PPB	FIT	ΔT
N-NITROSODIMETHYLAMINE							
BIS(2-CHLOROETHYL)ETHER							
(1,3-)DICHLOROBENZENE		17.0	961	4			
1,4-DICHLOROBENZENE		89.4	<962	2			
(1,2-)DICHLOROBENZENE		312.4	996	2			
BIS(2-CHLOROISOPROPYL)ETHER							
HEXACHLOROETHANE							
N-NITROSO-DI-N-PROPYL AMINE							
NITROBENZENE							
BIS(2-CHLOROETHOXY)METHANE							
1,2,4-TRICHLOROBENZENE							
NAPHTHALENE		318.4	992	1			
HEXACHLOROBUTADIENE							
2-CHLORO-NAPHTHALANE							
ACENAPHTHYLENE		19.6	805	7			
DIMETHYLPHTHALATE							
2,6-DINITROTOLUENE							
ACENAPHTHENE		21.50	947	0			
2,4-DINITROTOLUENE							
FLUORENE		51.2	929	0			
DIETHYLPHTHALATE							
AZOBENZENE (FROM DIPHENYLHYDRAZINE)		3.6	<784	6			
N-NITROSODIPHENYLAMINE		122.6	<930	0			
4-BROMODIPHENYL ETHER							
HEXACHLOROBENZENE							
PHENANTHRENE		101.	993	1			
ANTHRACENE		9.6	<991	2			
DI-N-BUTYLPHTHALATE		2002.	978	-2			
FLUORANTHENE		27.2	986	3			
PYRENE		39.6	976	2			
BUTYLBENZYLPHTHALATE		3115.8	993	0			
BENZO(A)ANTHRACENE		28.8	893	6			
CHRYSENE		23.0	931	5			
3,3'-DICHLOROBENZIDENE							
BIS(2-ETHYLHEXYL)PHTHALATE		3794.8	987	1			
DI-N-OCTYL PHTHALATE		181.8	819	0			
BENZO(B)FLUORANTHENE		14.6	<811	<11			
BENZO(K)FLUORANTHENE		13.6	<832	10			
BENZO(A)PYRENE	A	8.4	759	-3			
INDENO(1,2,3-CD)							
DIBENZO(A,H)ANTHRACENE							
BENZO(GHI)PERYLENE							
CHLOROPHENYL,PHENYL ETHER		NA					
HEXACHLOROCYCLOPENTADIENE		NA					

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=probable artifact

ANALYSIS REPORT — BASE + NEUTRAL EXTRACTABLES

Sample: Oakland, Raw Sewage
PS181
Lab: U of W
Sample Size: 1 liter
Date Sampled

Quantitation Standard: D_8Naphthalene
Quantitation Method: GC/MS

COMPOUND		PPB	FIT	ΔT	PPB	FIT	ΔT
N-NITROSODIMETHYLAMINE							
BIS(2-CHLOROETHYL)ETHER		0.12	881	-4			
(1,3-)DICHLOROBENZENE		0.22	861	3			
1,4-DICHLOROBENZENE		1.52	<869	1			
(1,2-)DICHLOROBENZENE		1.78	983	3			
BIS(2-CHLOROISOPROPYL)ETHER							
HEXACHLOROETHANE							
N-NITROSO-DI-N-PROPYL AMINE		6.73	<834	6			
NITROBENZENE	A	0.29	<824	-6			
BIS(2-CHLOROETHOXY)METHANE							
1,2,4-TRICHLOROBENZENE							
NAPHTHALENE		6.85	995	0			
HEXACHLOROBUTADIENE							
2-CHLORO-NAPHTHALANE							
ACENAPHTHYLENE							
DIMETHYLPHTHALATE		0.38	913	-3			
2,6-DINITROTOLUENE	A	2.61	792	5			
ACENAPHTHENE		0.28	892	-2			
2,4-DINITROTOLUENE							
FLUORENE		0.57	933	-2			
DIETHYLPHTHALATE		9.77	989	-1			
AZOBENZENE (FROM DIPHENYLHYDRAZINE)							
N-NITROSODIPHENYLAMINE	A	1.28	<844	-2			
4-BROMODIPHENYL ETHER							
HEXACHLOROBENZENE							
PHENANTHRENE		1.21	987	1			
ANTHRACENE		0.05	983	4			
DI-N-BUTYLPHTHALATE		11.07	993	1			
FLUORANTHENE		3.10	995	2			
PYRENE		0.80	978	2			
BUTYLBENZYLPHTHALATE		126.34	996	-1			
BENZO(A)ANTHRACENE		0.68	<941	1			
CHRYSENE		0.66	948	0			
3,3'-DICHLOROBENZIDENE							
BIS(2-ETHYLHEXYL)PHTHALATE		275.03	995	-1			
DI-N-OCTYL PHTHALATE		10.60	916	-4			
BENZO(B)FLUORANTHENE							
BENZO(K)FLUORANTHENE		0.38	854	-3			
BENZO(A)PYRENE		0.08	839	5			
INDENO(1,2,3-CD) PYRENE							
DIBENZO(A,H)ANTHRACENE							
BENZO(GHI)PERYLENE							
CHLOROPHENYL,PHENYL ETHER		NA					
HEXACHLOROCYCLOPENTADIENE		NA					

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=probable artifact

ANALYSIS REPORT BASE + NEUTRAL EXTRACTABLES
Sample: Oakland, Primary Effluent
PS190
Lab: U of W
Sample Size: 1 liter
Date Sampled

Quantitation Standard: D_8Naphthalene
Quantitation Method: GC/MS

COMPOUND		PPB	FIT	ΔT	PPB	FIT	ΔT
N-NITROSODIMETHYLAMINE							
BIS(2-CHLOROETHYL)ETHER		0.70	<859	6			
(1,3-)DICHLOROBENZENE		1.29	<977	4			
1,4-DICHLOROBENZENE		6.88	<984	2			
(1,2-)DICHLOROBENZENE		5.90	980	-3			
BIS(2-CHLOROISOPROPYL)ETHER							
HEXACHLOROETHANE							
N-NITROSO-DI-N-PROPYL AMINE		NQ	<845				
NITROBENZENE	A	1.97	<955	-10			
BIS(2-CHLOROETHOXY)METHANE		NQ	<785				
1,2,4-TRICHLOROBENZENE							
NAPHTHALENE		10.70	993	1			
HEXACHLOROBUTADIENE							
2-CHLORO-NAPHTHALANE							
ACENAPHTHYLENE	A	0.27	<804	11			
DIMETHYLPHTHALATE		0.31	757	-1			
2,6-DINITROTOLUENE		1.35	<788	7			
ACENAPHTHENE							
2,4-DINITROTOLUENE							
FLUORENE		0.58	802	1			
DIETHYLPHTHALATE		5.18	825	-2			
AZOBENZENE (FROM DIPHENYLHYDRAZINE)							
N-NITROSODIPHENYLAMINE		0.70	<867	-1			
4-BROMODIPHENYL ETHER							
HEXACHLOROBENZENE							
PHENANTHRENE		1.43	961	0			
ANTHRACENE		NQ					
DI-N-BUTYLPHTHALATE		12.87	978	0			
FLUORANTHENE		3.48	992	2			
PYRENE		1.52	965	3			
BUTYLBENZYLPHTHALATE		154.72	997	-2			
BENZO(A)ANTHRACENE		1.53	<923	1			
CHRYSENE		1.42	926	-1			
3,3'-DICHLOROBENZIDENE							
BIS(2-ETHYLHEXYL)PHTHALATE		309.50	990	-2			
DI-N-OCTYL PHTHALATE		10.79	835	-6			
BENZO(B)FLUORANTHENE		NQ					
BENZO(K)FLUORANTHENE		0.77	774	-1			
BENZO(A)PYRENE		NQ	<760				
INDENO(1,2,3-CD)							
DIBENZO(A,H)ANTHRACENE							
BENZO(GHI)PERYLENE							
CHLOROPHENYL,PHENYL ETHER		NA					
HEXACHLOROCYCLOPENTADIENE		NA					

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=probable artifact

ANALYSIS REPORT — BASE + NEUTRAL EXTRACTABLES
Sample: Renton, Raw Sewage
PS100
Lab: U of W
Sample Size: 1 liter
Date Sampled

Quantitation Standard: D_8Naphthalene
Quantitation Method: GC/MS

COMPOUND		PPB	FIT	ΔT	PPB	FIT	ΔT
N-NITROSODIMETHYLAMINE							
BIS(2-CHLOROETHYL)ETHER	A	0.06	<944	-6			
(1,3-)DICHLOROBENZENE		0.71	979	4			
1,4-DICHLOROBENZENE		5.02	<997	3			
(1,2-)DICHLOROBENZENE		4.79	993	4			
BIS(2-CHLOROISOPROPYL)ETHER		0.16	<829	2			
HEXACHLOROETHANE							
N-NITROSO-DI-N-PROPYL AMINE	A	0.49	<854	-4			
NITROBENZENE							
BIS(2-CHLOROETHOXY)METHANE		0.21	839	5			
1,2,4-TRICHLOROBENZENE							
NAPHTHALENE		1.27	977	1			
HEXACHLOROBUTADIENE							
2-CHLORO-NAPHTHALANE							
ACENAPHTHYLENE		0.04	<757	1			
DIMETHYLPHTHALATE		0.72	979	-2			
2,6-DINITROTOLUENE	A	0.20	<780	7			
ACENAPHTHENE		0.09	830	-1			
2,4-DINITROTOLUENE							
FLUORENE		0.15	924	-1			
DIETHYLPHTHALATE		6.57	996	-1			
AZOBENZENE (FROM DIPHENYLHYDRAZINE)							
N-NITROSODIPHENYLAMINE	A	0.24	<827	-7			
4-BROMODIPHENYL ETHER							
HEXACHLOROBENZENE							
PHENANTHRENE		0.37	<996	1			
ANTHRACENE		NQ					
DI-N-BUTYLPHTHALATE		5.58	992	1			
FLUORANTHENE		0.16	975	3			
PYRENE		0.16	986	2			
BUTYLBENZYLPHTHALATE		6.69	991	-4			
BENZO(A)ANTHRACENE		0.06	<930	1			
CHRYSENE		0.09	930	1			
3,3'-DICHLOROBENZIDENE							
BIS(2-ETHYLHEXYL)PHTHALATE		1.33	914	-6			
DI-N-OCTYL PHTHALATE							
BENZO(B)FLUORANTHENE		0.07	811	1			
BENZO(K)FLUORANTHENE							
BENZO(A)PYRENE							
INDENO(1,2,3-CD)							
DIBENZO(A,H)ANTHRACENE							
BENZO(GHI)PERYLENE							
CHLOROPHENYL,PHENYL ETHER		NA					
HEXACHLOROCYCLOPENTADIENE		NA					

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=probable artifact

ANALYSIS REPORT — BASE + NEUTRAL EXTRACTABLES
Sample: Renton, Primary & Waste Activated Sludge
PS110
Lab: U of W
Sample Size: 50 ml
Date Sampled

Quantitation Standard: D_8Naphthalene
Quantitation Method: GC/MS

COMPOUND		PPB	FIT	ΔT	PPB	FIT	ΔT
N-NITROSODIMETHYLAMINE							
BIS(2-CHLOROETHYL)ETHER							
(1,3-)DICHLOROBENZENE							
1,4-DICHLOROBENZENE							
(1,2-)DICHLOROBENZENE							
BIS(2-CHLOROISOPROPYL)ETHER							
HEXACHLOROETHANE							
N-NITROSO-DI-N-PROPYL AMINE	A	59.8	<832	-2			
NITROBENZENE							
BIS(2-CHLOROETHOXY)METHANE							
1,2,4-TRICHLOROBENZENE							
NAPHTHALENE		11.0	851	2			
HEXACHLOROBUTADIENE							
2-CHLORO-NAPHTHALANE							
ACENAPHTHYLENE							
DIMETHYLPHTHALATE		4.2	934	-1			
2,6-DINITROTOLUENE		42.6	791	9			
ACENAPHTHENE							
2,4-DINITROTOLUENE							
FLUORENE							
DIETHYLPHTHALATE		6.0	895	-2			
AZOBENZENE (FROM DIPHENYLHYDRAZINE)							
N-NITROSODIPHENYLAMINE							
4-BROMODIPHENYL ETHER							
HEXACHLOROBENZENE							
PHENANTHRENE							
ANTHRACENE							
DI-N-BUTYLPHTHALATE		119.2	990	-1			
FLUORANTHENE							
PYRENE							
BUTYLBENZYLPHTHALATE		20.0	845	1			
BENZO(A)ANTHRACENE							
CHRYSENE							
3,3'-DICHLOROBENZIDENE	A						
BIS(2-ETHYLHEXYL)PHTHALATE		4009	988	2			
DI-N-OCTYL PHTHALATE		124.0	798	1			
BENZO(B)FLUORANTHENE							
BENZO(K)FLUORANTHENE							
BENZO(A)PYRENE							
INDENO(1,2,3-CD)							
DIBENZO(A,H)ANTHRACENE							
BENZO(GHI)PERYLENE							
CHLOROPHENYL,PHENYL ETHER		NA					
HEXACHLOROCYCLOPENTADIENE		NA					

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=probable artifact

ANALYSIS REPORT BASE + NEUTRAL EXTRACTABLES
Sample: Oakland, Secondary Effluent
PS200N
Lab: U of W
Sample Size: 1 liter
Date Sampled

Quantitation Standard: D8Naphthalene
Quantitation Method: GC/MS

COMPOUND		PPB	FIT	ΔT	PPB	FIT	ΔT
N-NITROSODIMETHYLAMINE		0.40	<832	2			
BIS(2-CHLOROETHYL)ETHER		<.01					
(1,3-)DICHLOROBENZENE		0.41	<996	8			
1,4-DICHLOROBENZENE		2.75	<995	7			
(1,2-)DICHLOROBENZENE		1.34	<996	7			
BIS(2-CHLOROISOPROPYL)ETHER	A	0.27	916	-2			
HEXACHLOROETHANE							
N-NITROSO-DI-N-PROPYL AMINE		0.10	<871	0			
NITROBENZENE							
BIS(2-CHLOROETHOXY)METHANE							
1,2,4-TRICHLOROBENZENE		0.32	958	1			
NAPHTHALENE							
HEXACHLOROBUTADIENE							
2-CHLORO-NAPHTHALANE							
ACENAPHTHYLENE							
DIMETHYLPHTHALATE			NQ				
2,6-DINITROTOLUENE							
ACENAPHTHENE							
2,4-DINITROTOLUENE							
FLUORENE							
DIETHYLPHTHALATE							
AZOBENZENE (FROM DIPHENYLHYDRAZINE)							
N-NITROSODIPHENYLAMINE							
4-BROMODIPHENYL ETHER							
HEXACHLOROBENZENE							
PHENANTHRENE							
ANTHRACENE							
DI-N-BUTYLPHTHALATE		5.99	994	-9			
FLUORANTHENE							
PYRENE							
BUTYLBENZYLPHTHALATE							
BENZO(A)ANTHRACENE							
CHRYSENE							
3,3'-DICHLOROBENZIDENE							
BIS(2-ETHYLHEXYL)PHTHALATE							
DI-N-OCTYL PHTHALATE							
BENZO(B)FLUORANTHENE							
BENZO(K)FLUORANTHENE							
BENZO(A)PYRENE							
INDENO(1,2,3-CD)							
DIBENZO(A,H)ANTHRACENE							
BENZO(GHI)PERYLENE							
CHLOROPHENYL,PHENYL ETHER		NA					
HEXACHLOROCYCLOPENTADIENE		NA					

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=probable artifact

ANALYSIS REPORT BASE + NEUTRAL EXTRACTABLES
Sample: Oakland, Secondary Effluent after Dechlorination
PS210
Lab: U of W
Sample Size: 1 liter
Date Sampled

Quantitation Standard: D8Naphthalene
Quantitation Method: GC/MS

COMPOUND		PPB	FIT	ΔT	PPB	FIT	ΔT
N-NITROSODIMETHYLAMINE							
BIS(2-CHLOROETHYL)ETHER		0.01	<883	-3			
(1,3-)DICHLOROBENZENE							
1,4-DICHLOROBENZENE		0.30	962	-1			
(1,2-)DICHLOROBENZENE		0.23	943	3			
BIS(2-CHLOROISOPROPYL)ETHER	A	0.23	<888	13			
HEXACHLOROETHANE							
N-NITROSO-DI-N-PROPYL AMINE							
NITROBENZENE							
BIS(2-CHLOROETHOXY)METHANE							
1,2,4-TRICHLOROBENZENE							
NAPHTHALENE							
HEXACHLOROBUTADIENE							
2-CHLORO-NAPHTHALANE							
ACENAPHTHYLENE							
DIMETHYLPHTHALATE		0.05	902	-1			
2,6-DINITROTOLUENE							
ACENAPHTHENE							
2,4-DINITROTOLUENE							
FLUORENE							
DIETHYLPHTHALATE		0.11	906	-1			
AZOBENZENE (FROM DIPHENYLHYDRAZINE)	A	0.02	<833	-16			
N-NITROSODIPHENYLAMINE							
4-BROMODIPHENYL ETHER							
HEXACHLOROBENZENE							
PHENANTHRENE							
ANTHRACENE							
DI-N-BUTYLPHTHALATE		4.90	992	-1			
FLUORANTHENE							
PYRENE							
BUTYLBENZYLPHTHALATE		0.38	971	-1			
BENZO(A)ANTHRACENE							
CHRYSENE							
3,3'-DICHLOROBENZIDENE							
BIS(2-ETHYLHEXYL)PHTHALATE		3.35	967	-3			
DI-N-OCTYL PHTHALATE							
BENZO(B)FLUORANTHENE							
BENZO(K)FLUORANTHENE							
BENZO(A)PYRENE							
INDENO(1,2,3-CD)							
DIBENZO(A,H)ANTHRACENE							
BENZO(GHI)PERYLENE							
CHLOROPHENYL,PHENYL ETHER		NA					
HEXACHLOROCYCLOPENTADIENE		NA					

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=probable artifact

ANALYSIS REPORT — BASE + NEUTRAL EXTRACTABLES

Sample: Oakland, Digested Sludge
PS220
Lab: U of W
Sample Size: 1 liter
Date Sampled

Quantitation Standard: D_8Naphthalene
Quantitation Method: GC/MS

COMPOUND		PPB	FIT	ΔT	PPB	FIT	ΔT
N-NITROSODIMETHYLAMINE							
BIS(2-CHLOROETHYL)ETHER							
(1,3-)DICHLOROBENZENE		5.5	942	5			
1,4-DICHLOROBENZENE		37.0	944	2			
(1,2-)DICHLOROBENZENE		15.8	981	5			
BIS(2-CHLOROISOPROPYL)ETHER							
HEXACHLOROETHANE							
N-NITROSO-DI-N-PROPYL AMINE							
NITROBENZENE							
BIS(2-CHLOROETHOXY)METHANE							
1,2,4-TRICHLOROBENZENE		13.5	982	2			
NAPHTHALENE		76.0	990	1			
HEXACHLOROBUTADIENE							
2-CHLORO-NAPHTHALANE							
ACENAPHTHYLENE							
DIMETHYLPHTHALATE	A	0.75	754	6			
2,6-DINITROTOLUENE							
ACENAPHTHENE		2.8	981	3			
2,4-DINITROTOLUENE							
FLUORENE		5.8	983	4			
DIETHYLPHTHALATE							
AZOBENZENE (FROM DIPHENYLHYDRAZINE)	A	0.01	823	-3			
N-NITROSODIPHENYLAMINE							
4-BROMODIPHENYL ETHER							
HEXACHLOROBENZENE							
PHENANTHRENE		27.3	996	1			
ANTHRACENE		NQ					
DI-N-BUTYLPHTHALATE		1.8	862	-1			
FLUORANTHENE		42.3	992	2			
PYRENE		24.8	995	3			
BUTYLBENZYLPHTHALATE							
BENZO(A)ANTHRACENE		18.8	991	6			
CHRYSENE		14.0	990	5			
3,3'-DICHLOROBENZIDENE							
BIS(2-ETHYLHEXYL)PHTHALATE		7,429	984	5			
DI-N-OCTYL PHTHALATE	A	237	870	-3			
BENZO(B)FLUORANTHENE		NQ					
BENZO(K)FLUORANTHENE		8.0	905	8			
BENZO(A)PYRENE		4.8	909	-4			
INDENO(1,2,3-CD)							
DIBENZO(A,H)ANTHRACENE							
BENZO(GHI)PERYLENE							
CHLOROPHENYL,PHENYL ETHER		NA					
HEXACHLOROCYCLOPENTADIENE		NA					

blank=not found; NQ=detected but not quantitated; NA=not analyzed for
A=probable artifact

ANALYSIS REPORT ACID-EXTRACTABLES (PHENOLS)
Sample: Oakland, Digested Sludge
: PS220
Lab: U of W
Sample Size: 40ML
Date Sampled: 7/3/79

Quantitation Standard: HMB
Quantitation Method: GC FID

COMPOUND	AMT	FIT	ΔT	AMT	FIT
PHENOL (M)					
2-CHLOROPHENOL					
PHENOL					
2-CHLOROPHENOL (M)					
2,4-DIMETHYLPHENOL					
4-CHLORO-3-METHYLPHENOL (M)					
2,4-DICHLOROPHENOL (M)					
4-CHLORO-3-METHYLPHENOL					
2,4,6-TRICHLOROPHENOL (M)					
2-NITROPHENOL (M)					
4-NITROPHENOL (M)					
4,6-DINITRO-2-METHYLPHENOL (M)					
PENTACHLOROPHENOL (M)					
2,4-DINITROPHENOL (M)					

blank=not found; NQ=detected,not quantitated; NA=not analyzed for
(M)=derivatized with diazomethane

ANALYSIS REPORT ACID-EXTRACTABLES (PHENOLS)
Sample: Oakland, Secondary Effluent, dechlorinated
PS210
Lab: U of W
Sample Size: 1000ml
Date Sampled: 7/3/79

Quantitation Standard: HMB
Quantitation Method: GC FID

COMPOUND	AMT	FIT	ΔT	AMT	FIT
PHENOL (M)					
2-CHLOROPHENOL					
PHENOL					
2-CHLOROPHENOL (M)					
2,4-DIMETHYLPHENOL					
4-CHLORO-3-METHYLPHENOL (M)					
2,4-DICHLOROPHENOL (M)					
4-CHLORO-3-METHYLPHENOL					
2,4,6-TRICHLOROPHENOL (M)					
2-NITROPHENOL (M)					
4-NITROPHENOL (M)					
4,6-DINITRO-2-METHYLPHENOL (M)					
PENTACHLOROPHENOL (M)					
2,4-DINITROPHENOL (M)					

blank=not found; NQ=detected,not quantitated; NA=not analyzed for
(M)=derivatized with diazomethane

ANALYSIS REPORT ACID-EXTRACTABLES (PHENOLS)
Sample: Oakland, Secondary Effluent
PS200
Lab: U of W
Sample Size: 1000ml
Date Sampled: 7/3/79

Quantitation Standard: HMB
Quantitation Method: GC FID

COMPOUND	AMT	FIT	ΔT	AMT	FIT
PHENOL (M)					
2-CHLOROPHENOL					
PHENOL					
2-CHLOROPHENOL (M)					
2,4-DIMETHYLPHENOL					
4-CHLORO-3-METHYLPHENOL (M)					
2,4-DICHLOROPHENOL (M)					
4-CHLORO-3-METHYLPHENOL					
2,4,6-TRICHLOROPHENOL (M)					
2-NITROPHENOL (M)					
4-NITROPHENOL (M)					
4,6-DINITRO-2-METHYLPHENOL (M)					
PENTACHLOROPHENOL (M)					
2,4-DINITROPHENOL (M)					

blank=not found; NQ=detected,not quantitated; NA=not analyzed for
(M)=derivatized with diazomethane

ANALYSIS REPORT ACID-EXTRACTABLES (PHENOLS)

Sample: Oakland, Primary Effluent
PS190
Lab: U of W
Sample Size: 1000ml
Date Sampled: 7/3/79

Quantitation Standard: HMB
Quantitation Method: GC FID

COMPOUND	AMT	FIT	ΔT	AMT	FIT
PHENOL (M)					
2-CHLOROPHENOL					
PHENOL					
2-CHLOROPHENOL (M)	479.26				
2,4-DIMETHYLPHENOL	42.57				
4-CHLORO-3-METHYLPHENOL (M)					
2,4-DICHLOROPHENOL (M)					
4-CHLORO-3-METHYLPHENOL					
2,4,6-TRICHLOROPHENOL (M)	12.12				
2-NITROPHENOL (M)					
4-NITROPHENOL (M)					
4,6-DINITRO-2-METHYLPHENOL (M)					
PENTACHLOROPHENOL (M)					
2,4-DINITROPHENOL (M)					

blank=not found; NQ=detected,not quantitated; NA=not analyzed for
(M)=derivatized with diazomethane

ANALYSIS REPORT — ACID-EXTRACTABLES (PHENOLS)

Sample: Oakland, Raw Sewage
PS181
Lab: U of W
Sample Size: 1000ml
Date Sampled: 7/3/79

Quantitation Standard: HMB
Quantitation Method: GC FID

COMPOUND	AMT	FIT	ΔT	AMT	FIT
PHENOL (M)	163.89 201.67				
2-CHLOROPHENOL					
PHENOL					
2-CHLOROPHENOL (M)					
2,4-DIMETHYLPHENOL	58.17				
4-CHLORO-3-METHYLPHENOL (M)					
2,4-DICHLOROPHENOL (M)					
4-CHLORO-3-METHYLPHENOL					
2,4,6-TRICHLOROPHENOL (M)	14.33				
2-NITROPHENOL (M)					
4-NITROPHENOL (M)					
4,6-DINITRO-2-METHYLPHENOL (M)					
PENTACHLOROPHENOL (M)					
2,4-DINITROPHENOL (M)					

blank=not found; NQ=detected,not quantitated; NA=not analyzed for
(M)=derivatized with diazomethane

ANALYSIS REPORT — ACID-EXTRACTABLES (PHENOLS)

Sample: Renton, Prim.+Ac.Sludge
PS110
Lab: U of W
Sample Size: 767ml
Date Sampled: 4/12/79

Quantitation Standard: HMB
Quantitation Method: GC FID

COMPOUND	AMT	FIT	ΔT	AMT	FIT
PHENOL (M)					
2-CHLOROPHENOL					
PHENOL					
2-CHLOROPHENOL (M)					
2,4-DIMETHYLPHENOL					
4-CHLORO-3-METHYLPHENOL (M)					
2,4-DICHLOROPHENOL (M)					
4-CHLORO-3-METHYLPHENOL					
2,4,6-TRICHLOROPHENOL (M)					
2-NITROPHENOL (M)	1.44				
4-NITROPHENOL (M)					
4,6-DINITRO-2-METHYLPHENOL (M)	7.70				
PENTACHLOROPHENOL (M)					
2,4-DINITROPHENOL (M)					

blank=not found; NQ=detected,not quantitated; NA=not analyzed for
(M)=derivatized with diazomethane

ANALYSIS REPORT ACID-EXTRACTABLES (PHENOLS)
Sample: Renton, Secondary Effluent, dechlorinated
PS70
Lab: U of W
Sample Size: 1000ml
Date Sampled: 4/12/79

Quantitation Standard: HMB
Quantitation Method: GC FID

COMPOUND	AMT	FIT	ΔT	AMT	FIT
PHENOL (M)					
2-CHLOROPHENOL					
PHENOL					
2-CHLOROPHENOL (M)					
2,4-DIMETHYLPHENOL					
4-CHLORO-3-METHYLPHENOL (M)					
2,4-DICHLOROPHENOL (M)					
4-CHLORO-3-METHYLPHENOL					
2,4,6-TRICHLOROPHENOL (M)					
2-NITROPHENOL (M)					
4-NITROPHENOL (M)					
4,6-DINITRO-2-METHYLPHENOL (M)					
PENTACHLOROPHENOL (M)	14.71				
2,4-DINITROPHENOL (M)					

blank=not found; NQ=detected,not quantitated; NA=not analyzed for
(M)=derivatized with diazomethane

ANALYSIS REPORT ACID-EXTRACTABLES (PHENOLS)
Sample: PS90S
Renton, Primary Effluent
Lab: U of W
Sample Size: 1000ml
Date Sampled: 4/12/79

Quantitation Standard: HMB
Quantitation Method: GC FID

COMPOUND	AMT	FIT	ΔT	AMT	FI
PHENOL (M)					
2-CHLOROPHENOL					
PHENOL					
2-CHLOROPHENOL (M)					
2,4-DIMETHYLPHENOL					
4-CHLORO-3-METHYLPHENOL (M)					
2,4-DICHLOROPHENOL (M)					
4-CHLORO-3-METHYLPHENOL	1.26				
2,4,6-TRICHLOROPHENOL (M)					
2-NITROPHENOL (M)	23.02				
4-NITROPHENOL (M)					
4,6-DINITRO-2-METHYLPHENOL (M)	1.62				
PENTACHLOROPHENOL (M)	4.68				
2,4-DINITROPHENOL (M)					

blank=not found; NQ=detected,not quantitated; NA=not analyzed for
(M)=derivatized with diazomethane

ANALYSIS REPORT — ACID-EXTRACTABLES (PHENOLS)

Sample: Renton, Raw Sewage
PS100
Lab: U of W
Sample Size: 1000ml
Date Sampled: 4/12/79

Quantitation Standard: HMB
Quantitation Method: GC FID

COMPOUND	AMT	FIT	ΔT	AMT	FIT
PHENOL (M)					
2-CHLOROPHENOL					
PHENOL					
2-CHLOROPHENOL (M)					
2,4-DIMETHYLPHENOL					
4-CHLORO-3-METHYLPHENOL (M)					
2,4-DICHLOROPHENOL (M)					
4-CHLORO-3-METHYLPHENOL					
2,4,6-TRICHLOROPHENOL (M)					
2-NITROPHENOL (M)					
4-NITROPHENOL (M)					
4,6-DINITRO-2-METHYLPHENOL (M)					
PENTACHLOROPHENOL (M)					
2,4-DINITROPHENOL (M)					

blank=not found; NQ=detected,not quantitated; NA=not analyzed for
(M)=derivatized with diazomethane

ANALYSIS REPORT ACID-EXTRACTABLES (PHENOLS)
Sample: PS80S
Renton, Secondary Effluent
Lab: U of W
Sample Size: 1000ml
Date Sampled: 4/12/79

Quantitation Standard: HMB
Quantitation Method: GC FID

COMPOUND	AMT	FIT	ΔT	AMT	FIT
PHENOL (M)					
2-CHLOROPHENOL					
PHENOL					
2-CHLOROPHENOL (M)					
2,4-DIMETHYLPHENOL					
4-CHLORO-3-METHYLPHENOL (M)					
2,4-DICHLOROPHENOL (M)					
4-CHLORO-3-METHYLPHENOL					
2,4,6-TRICHLOROPHENOL (M)					
2-NITROPHENOL (M)					
4-NITROPHENOL (M)					
4,6-DINITRO-2-METHYLPHENOL (M)	5.35				
PENTACHLOROPHENOL (M)	16.80				
2,4-DINITROPHENOL (M)					

blank=not found; NQ=detected,not quantitated; NA=not analyzed for
(M)=derivatized with diazomethane

ANALYSIS REPORT — ACID-EXTRACTABLES (PHENOLS)

Sample: Atlanta, Sludge
PS141
Lab: U of W
Sample Size: 20ml
Date Sampled: 6/14/79

Quantitation Standard: HMB
Quantitation Method: GC FID

COMPOUND	AMT	FIT	ΔT	AMT	FIT
PHENOL (M)					
2-CHLOROPHENOL					
PHENOL					
2-CHLOROPHENOL (M)					
2,4-DIMETHYLPHENOL	11.42				
4-CHLORO-3-METHYLPHENOL (M)					
2,4-DICHLOROPHENOL (M)	9.41				
4-CHLORO-3-METHYLPHENOL					
2,4,6-TRICHLOROPHENOL (M)					
2-NITROPHENOL (M)					
4-NITROPHENOL (M)					
4,6-DINITRO-2-METHYLPHENOL (M)	0.14				
PENTACHLOROPHENOL (M)					
2,4-DINITROPHENOL (M)					

blank=not found; NQ=detected,not quantitated; NA=not analyzed for
(M)=derivatized with diazomethane

ANALYSIS REPORT ACID-EXTRACTABLES (PHENOLS)
Sample: Atlanta, Secondary Effluent, chlorinated
PS160
Lab: U of W
Sample Size: 1000ml
Date Sampled: 6/14/79

Quantitation Standard: HMB
Quantitation Method: GC FID

COMPOUND	AMT	FIT	ΔT	AMT	FIT
PHENOL (M)					
2-CHLOROPHENOL					
PHENOL					
2-CHLOROPHENOL (M)					
2,4-DIMETHYLPHENOL					
4-CHLORO-3-METHYLPHENOL (M)					
2,4-DICHLOROPHENOL (M)					
4-CHLORO-3-METHYLPHENOL					
2,4,6-TRICHLOROPHENOL (M)					
2-NITROPHENOL (M)					
4-NITROPHENOL (M)					
4,6-DINITRO-2-METHYLPHENOL (M)					
PENTACHLOROPHENOL (M)	13.47				
2,4-DINITROPHENOL (M)					

blank=not found; NQ=detected,not quantitated; NA=not analyzed for
(M)=derivatized with diazomethane

ANALYSIS REPORT ACID-EXTRACTABLES (PHENOLS)

Sample: Atlanta, Secondary Effluent
PS171

Lab: U of W
Sample Size: 1000ml
Date Sampled: 6/14/79

Quantitation Standard: HMB
Quantitation Method: GC FID

COMPOUND	AMT	FIT	ΔT	AMT	FIT
PHENOL (M)					
2-CHLOROPHENOL					
PHENOL					
2-CHLOROPHENOL (M)					
2,4-DIMETHYLPHENOL					
4-CHLORO-3-METHYLPHENOL (M)					
2,4-DICHLOROPHENOL (M)					
4-CHLORO-3-METHYLPHENOL					
2,4,6-TRICHLOROPHENOL (M)					
2-NITROPHENOL (M)					
4-NITROPHENOL (M)					
4,6-DINITRO-2-METHYLPHENOL (M)					
PENTACHLOROPHENOL (M)					
2,4-DINITROPHENOL (M)					

blank=not found; NQ=detected,not quantitated; NA=not analyzed for
(M)=derivatized with diazomethane

ANALYSIS REPORT ACID-EXTRACTABLES (PHENOLS)
Sample: Atlanta, Primary Effluent
PS120
Lab: U of W
Sample Size: 1000ml
Date Sampled: 6/14/79

Quantitation Standard: HMB
Quantitation Method: GC FID

COMPOUND	AMT	FIT	ΔT	AMT	FIT
PHENOL (M)					
2-CHLOROPHENOL					
PHENOL					
2-CHLOROPHENOL (M)					
2,4-DIMETHYLPHENOL					
4-CHLORO-3-METHYLPHENOL (M)					
2,4-DICHLOROPHENOL (M)					
4-CHLORO-3-METHYLPHENOL					
2,4,6-TRICHLOROPHENOL (M)					
2-NITROPHENOL (M)					
4-NITROPHENOL (M)					
4,6-DINITRO-2-METHYLPHENOL (M)					
PENTACHLOROPHENOL (M)	13.31				
2,4-DINITROPHENOL (M)					

blank=not found; NQ=detected,not quantitated; NA=not analyzed for
(M)=derivatized with diazomethane

ANALYSIS REPORT — ACID-EXTRACTABLES (PHENOLS)

Sample: Atlanta, Raw Sewage
PS150
Lab: U of W
Sample Size: 1000ml
Date Sampled: 6/14/79

Quantitation Standard: HMB
Quantitation Method: GC FID

COMPOUND	AMT	FIT	ΔT	AMT	FIT
PHENOL (M)					
2-CHLOROPHENOL					
PHENOL					
2-CHLOROPHENOL (M)					
2,4-DIMETHYLPHENOL					
4-CHLORO-3-METHYLPHENOL (M)					
2,4-DICHLOROPHENOL (M)					
4-CHLORO-3-METHYLPHENOL					
2,4,6-TRICHLOROPHENOL (M)					
2-NITROPHENOL (M)					
4-NITROPHENOL (M)					
4,6-DINITRO-2-METHYLPHENOL (M)					
PENTACHLOROPHENOL (M)	29.56				
2,4-DINITROPHENOL (M)					

blank=not found; NQ=detected,not quantitated; NA=not analyzed for
(M)=derivatized with diazomethane

ANALYSIS REPORT — PESTICIDES + PCB's

Sample: PS 100 — Renton Raw Sewage

Lab:
Sample Size:
Date Sampled

Quantitation Standard:
Quantitation Method:

COMPOUND	PPB	ΔT
α-BHC	0.130	MI
β-BHC	0.579	MI
γ-BHC (LINDANE	0.226	MI
δ-BHC		0
P,P'-DDD	0.181	
P,P'-DDE	0.183	I
P,P'-DDT	0.525	MI
DIELDRIN	0.022	
ENDRIN	0.191	
ENDRINALDEHYDE		
α-ENDOSULFAN	0.101	0.00
β-ENDOSULFAN		
ENDOSULFANE SULFATE		0
HEPTACHLOR	37.019	OMI
HEPTACHLOR EPOXIDE	0.154	
PCB 1242		
PCB 1254		
PCB 1221		
PCB 1232		
PCB 1248		
PCB 1260		
PCB 1016		
ALDRIN		
METHOXYCHLOR		
MIREX	0.019	
CHORDENE		0
OXYCHLOR		
γ-CHLORDANE	0.214	0.6
α-CHLORDANE	0.207	2.4

blank=not found; NQ=detected but not quantitated; NA=not analyzed for; M=merged peak; D=detected but not integrated; O=area obscured; I=improper integration.

ANALYSIS REPORT — PESTICIDES + PCB's

Sample: PS90 neutral — Renton primary effluent

Lab:
Sample Size:
Date Sampled

Quantitation Standard:
Quantitation Method:

COMPOUND	PPB		ΔT
α-BHC	0.125		+0.6
β-BHC			
γ-BHC (LINDANE			
δ-BHC			
P,P'-DDD			
P,P'-DDE			
P,P'-DDT			
DIELDRIN	NQ		+0.6
ENDRIN			
ENDRINALDEHYDE			
α-ENDOSULFAN		D	
β-ENDOSULFAN			
ENDOSULFANE SULFATE		O	
HEPTACHLOR			
HEPTACHLOR EPOXIDE	0.1757		+0.6
PCB 1242			
PCB 1254			
PCB 1221			
PCB 1232			
PCB 1248			
PCB 1260			
PCB 1016			
ALDRIN	NQ		+0.6
METHOXYCHLOR			
MIREX			
CHORDENE	0.224	MIO	-1.2
OXYCHLOR			
γ-CHLORDANE	0.185		1.2
α-CHLORDANE	0.126		3.0

blank=not found; NQ=detected but not quantitated; NA=not analyzed for; M=merged peak; D=detected but not integrated; O=area obscured; I=improper integration.

ANALYSIS REPORT — PESTICIDES + PCB's

Sample: PS80 neutral — Renton secondary effluent

Lab:
Sample Size:
Date Sampled

Quantitation Standard:
Quantitation Method:

COMPOUND	PPB		ΔT
α-BHC		D	+1.2
β-BHC			
γ-BHC (LINDANE			
δ-BHC	0.895	IM	+1.2
P,P'-DDD			
P,P'-DDE			
P,P'-DDT			
DIELDRIN			0
ENDRIN			
ENDRINALDEHYDE			
α-ENDOSULFAN	0.009		1.8
β-ENDOSULFAN			
ENDOSULFANE SULFATE			
HEPTACHLOR			
HEPTACHLOR EPOXIDE	0.084		+0.6
PCB 1242			
PCB 1254			
PCB 1221			
PCB 1232			
PCB 1248			
PCB 1260			
PCB 1016			
ALDRIN			+0.6
METHOXYCHLOR			
MIREX			
CHORDENE	0.097		2.4
OXYCHLOR			
γ-CHLORDANE	0.198		3.0
α-CHLORDANE	0.078		1.2

blank=not found; NQ=detected but not quantitated; NA=not analyzed for; M=merged peak; D=detected but not integrated; O=area obscured; I=improper integration.

ANALYSIS REPORT — PESTICIDES + PCB's

Sample: PS70 neutral — Renton secondary effluent dechlorinated

Lab:
Sample Size:
Date Sampled

Quantitation Standard:
Quantitation Method:

COMPOUND	PPB	ΔT
α-BHC		
β-BHC		
γ-BHC (LINDANE		
δ-BHC	0.889	-2.4
P,P'-DDD		
P,P'-DDE		
P,P'-DDT		
DIELDRIN	0.001	+1.8
ENDRIN	0.188	+1.8
ENDRINALDEHYDE		
α-ENDOSULFAN		
β-ENDOSULFAN		
ENDOSULFANE SULFATE		
HEPTACHLOR	0.012	+1.2
HEPTACHLOR EPOXIDE	0.0849	+2.4
PCB 1242		
PCB 1254		
PCB 1221		
PCB 1232		
PCB 1248		
PCB 1260		
PCB 1016		
ALDRIN		
METHOXYCHLOR		
MIREX		+1.2
CHORDENE		
OXYCHLOR		
γ-CHLORDANE	0.066	2.4
α-CHLORDANE		

blank=not found; NQ=detected but not quantitated; NA=not analyzed for; M=merged peak; D=detected but not integrated; O=area obscured; I=improper integration.

ANALYSIS REPORT PESTICIDES + PCB's

Sample: PS151 neutral Atlanta Raw Sewage

Lab:
Sample Size:
Date Sampled

Quantitation Standard:
Quantitation Method:

COMPOUND	PPB		ΔT
α-BHC	2.934	M	+06
β-BHC			
γ-BHC (LINDANE	11.157	M	+0.6
δ-BHC	5.139		+0.6
P,P'-DDD	6.799		+0.6
P,P'-DDE	3.147	M	+0.6
P,P'-DDT	5.392		+0.6
DIELDRIN	4.379		+1.2
ENDRIN	7.764		1.2
ENDRINALDEHYDE			
α-ENDOSULFAN	13.313	M	0.6
β-ENDOSULFAN	8.845		-2.4
ENDOSULFANE SULFATE			
HEPTACHLOR	4.066		+0.6
HEPTACHLOR EPOXIDE	8.2575		+1.2
PCB 1242			
PCB 1254			
PCB 1221			
PCB 1232			
PCB 1248			
PCB 1260			
PCB 1016			
ALDRIN	1.973		+0.6
METHOXYCHLOR			
MIREX	4.260		+.1
CHORDENE	3.246		1.8
OXYCHLOR			
γ-CHLORDANE	3.815	MI	0.6
α-CHLORDANE	3.548		1.2

blank=not found; NQ=detected but not quantitated; NA=not analyzed for; M=merged peak; D=detected but not integrated; O=area obscured; I=improper integration.

ANALYSIS REPORT PESTICIDES + PCB's

Sample: PS110 neutral Renton Sludge

Lab:
Sample Size:
Date Sampled

Quantitation Standard:
Quantitation Method:

COMPOUND	PPB		ΔT
α-BHC	0.159		+0.6
β-BHC			
γ-BHC (LINDANE	0.323	I	+0.6
δ-BHC			
P,P'-DDD			
P,P'-DDE			
P,P'-DDT			
DIELDRIN	0.017	I	-0.6
ENDRIN			
ENDRINALDEHYDE			
α-ENDOSULFAN	0.042		0.00
β-ENDOSULFAN			
ENDOSULFANE SULFATE		O	
HEPTACHLOR			
HEPTACHLOR EPOXIDE			
PCB 1242			
PCB 1254			
PCB 1221			
PCB 1232			
PCB 1248			
PCB 1260			
PCB 1016			
ALDRIN			
METHOXYCHLOR	0.468		
MIREX			
CHORDENE	0.535	MI	-1.2
OXYCHLOR			
γ-CHLORDANE		D	
α-CHLORDANE	0.174		-1.8

blank=not found; NQ=detected but not quantitated; NA=not analyzed for; M=merged peak; D=detected but not integrated; O=area obscured; I=improper integration.

ANALYSIS REPORT PESTICIDES + PCB's

Sample: PS150 neutral Atlanta Raw Sewage

Lab:
Sample Size:
Date Sampled

Quantitation Standard:
Quantitation Method:

COMPOUND	PPB		ΔT
α-BHC			
β-BHC			
γ-BHC (LINDANE	0.140	M	+1.2
δ-BHC			
P,P'-DDD	0.173		+0.6
P,P'-DDE			
P,P'-DDT			
DIELDRIN			
ENDRIN			
ENDRINALDEHYDE			
α-ENDOSULFAN			
β-ENDOSULFAN			
ENDOSULFANE SULFATE			
HEPTACHLOR			
HEPTACHLOR EPOXIDE			
PCB 1242			
PCB 1254			
PCB 1221			
PCB 1232			
PCB 1248			
PCB 1260			
PCB 1016			
ALDRIN			
METHOXYCHLOR			
MIREX			
CHORDENE			
OXYCHLOR			
γ-CHLORDANE	0.327		0.6
α-CHLORDANE	0.100	MI	1.2

blank=not found; NQ=detected but not quantitated; NA=not analyzed for; M=merged peak; D=detected but not integrated; O=area obscured; I=improper integration.

ANALYSIS REPORT — PESTICIDES + PCB's

Sample: PS120 Neutral — Atlanta primary effluent

Lab:
Sample Size:
Date Sampled

Quantitation Standard:
Quantitation Method:

COMPOUND	PPB		ΔT
α-BHC	0.123	MI	-0.6
β-BHC			
γ-BHC (LINDANE	0.171		+1.2
δ-BHC			
P,P'-DDD	0.180		0
P,P'-DDE			
P,P'-DDT	0.569		-0.6
DIELDRIN	0.016	0	
ENDRIN			
ENDRINALDEHYDE			
α-ENDOSULFAN	0.038		0.00
β-ENDOSULFAN	0.050		-3.0
ENDOSULFANE SULFATE			
HEPTACHLOR	0.012		-0.6
HEPTACHLOR EPOXIDE			
PCB 1242			
PCB 1254			
PCB 1221			
PCB 1232			
PCB 1248			
PCB 1260			
PCB 1016			
ALDRIN		0	
METHOXYCHLOR	0.484		
MIREX			
CHORDENE	0.078	I	1.8
OXYCHLOR			
γ-CHLORDANE	0.349		0.00
α-CHLORDANE	0.324		0.6

blank=not found; NQ=detected but not quantitated; NA=not analyzed for; M=merged peak; D=detected but not integrated; 0=area obscured; I=improper integration.

ANALYSIS REPORT PESTICIDES + PCB's

Sample: PS171 neutral Atlanta Secondary effluent

Lab:
Sample Size:
Date Sampled

Quantitation Standard:
Quantitation Method:

COMPOUND	PPB		ΔT
α-BHC	0.126	MT	-0.6
β-BHC			
γ-BHC (LINDANE	0.147		0
δ-BHC			
P,P'-DDD	0.143		+1.8
P,P'-DDE	0.048	M	-0.6
P,P'-DDT	0.401		-1.8
DIELDRIN	0.048	M	-0.6
ENDRIN			
ENDRINALDEHYDE			
α-ENDOSULFAN	0.009		0.00
β-ENDOSULFAN	0.128		-5.4
ENDOSULFANE SULFATE			
HEPTACHLOR			
HEPTACHLOR EPOXIDE			
PCB 1242			
PCB 1254			
PCB 1221			
PCB 1232			
PCB 1248			
PCB 1260			
PCB 1016			
ALDRIN			
METHOXYCHLOR			
MIREX			+0.6
CHORDENE	0.197	MI	0.61
OXYCHLOR			
γ-CHLORDANE	0.249		0.00
α-CHLORDANE	0.325		0.6

blank=not found; NQ=detected but not quantitated; NA=not analyzed for; M=merged peak; D=detected but not integrated; O=area obscured; I=improper integration.

ANALYSIS REPORT — PESTICIDES + PCB's

Sample: PS160 neutral — Atlanta secondary effluent chlorinated

Lab:
Sample Size:
Date Sampled

Quantitation Standard:
Quantitation Method:

COMPOUND	PPB		ΔT
α-BHC			
β-BHC	0.437		
γ-BHC (LINDANE	0.165		
δ-BHC			
P,P'-DDD	0.169		+1.8
P,P'-DDE	0.187		-1.8
P,P'-DDT	0.456		-1.8
DIELDRIN	0.036		0
ENDRIN	0.391		-1.8
ENDRINALDEHYDE			
α-ENDOSULFAN			
β-ENDOSULFAN	0.045		-2.4
ENDOSULFANE SULFATE	4.837		0.6
HEPTACHLOR			
HEPTACHLOR EPOXIDE	0.3608		+1.8
PCB 1242			
PCB 1254			
PCB 1221			
PCB 1232			
PCB 1248			
PCB 1260			
PCB 1016			
ALDRIN			
METHOXYCHLOR	0.501		
MIREX			
CHORDENE	0.106	I	0.6
OXYCHLOR			
γ-CHLORDANE	0.823	MI	-0.6
α-CHLORDANE	0.609	M	3.0

blank=not found; NQ=detected but not quantitated; NA=not analyzed for; M=merged peak; D=detected but not integrated; O=area obscured; I=improper integration.

ANALYSIS REPORT PESTICIDES + PCB's

Sample: PS141 neutral Atlanta Sludge

Lab:
Sample Size:
Date Sampled

Quantitation Standard:
Quantitation Method:

COMPOUND	PPB	ΔT
α-BHC		
β-BHC		
γ-BHC (LINDANE	0.130	+0.2
δ-BHC		
P,P'-DDD	0.203	+1.2
P,P'-DDE		
P,P'-DDT		
DIELDRIN	0.060	+1.2
ENDRIN		
ENDRINALDEHYDE		
α-ENDOSULFAN	0.219	1.2
β-ENDOSULFAN	0.049	-1.8
ENDOSULFANE SULFATE		
HEPTACHLOR		
HEPTACHLOR EPOXIDE	0.2632	+0.6
PCB 1242		
PCB 1254		
PCB 1221		
PCB 1232		
PCB 1248		
PCB 1260		
PCB 1016		
ALDRIN		
METHOXYCHLOR		
MIREX		
CHORDENE		
OXYCHLOR		
γ-CHLORDANE	0.710	1.2
α-CHLORDANE	0.470	1.2

blank=not found; NQ=detected but not quantitated; NA=not analyzed for; M=merged peak; D=detected but not integrated; O=area obscured; I=improper integration.

ANALYSIS REPORT — PESTICIDES + PCB's

Sample: PS181 neutral — Oakland Raw Sewage

Lab:
Sample Size:
Date Sampled

Quantitation Standard:
Quantitation Method:

COMPOUND	PPB		ΔT
α-BHC			
β-BHC			
γ-BHC (LINDANE	0.414	0	+0.6
δ-BHC			
P,P'-DDD	0.290		+0.6
P,P'-DDE			
P,P'-DDT			
DIELDRIN	0.050		+0.6
ENDRIN			
ENDRINALDEHYDE			
α-ENDOSULFAN	0.277		1.8
β-ENDOSULFAN	0.228		-3.6
ENDOSULFANE SULFATE		0	
HEPTACHLOR			
HEPTACHLOR EPOXIDE	4.974		-1.2
PCB 1242			
PCB 1254			
PCB 1221			
PCB 1232			
PCB 1248			
PCB 1260			
PCB 1016			
ALDRIN			
METHOXYCHLOR	0.632		
MIREX			
CHORDENE	1.141	MO	-1.2
OXYCHLOR			
γ-CHLORDANE	0.510		1.2
α-CHLORDANE	0.444		1.8

blank=not found; NQ=detected but not quantitated; NA=not analyzed for; M=merged peak; D=detected but not integrated; O=area obscured; I=improper integration.

ANALYSIS REPORT PESTICIDES + PCB's

Sample: PS190 neutral Oakland Primary effluent

Lab:
Sample Size:
Date Sampled

Quantitation Standard:
Quantitation Method:

COMPOUND	PPB		ΔT
α-BHC			
β-BHC			
γ-BHC (LINDANE	0.572	0	-0.6
δ-BHC			
P,P'-DDD	0.201		0
P,P'-DDE			
P,P'-DDT			
DIELDRIN	0.123		-13.2
ENDRIN			
ENDRINALDEHYDE			
α-ENDOSULFAN	0.515		0.6
β-ENDOSULFAN			
ENDOSULFANE SULFATE			
HEPTACHLOR			
HEPTACHLOR EPOXIDE	0.6451		0
PCB 1242			
PCB 1254			
PCB 1221			
PCB 1232			
PCB 1248			
PCB 1260			
PCB 1016			
ALDRIN			
METHOXYCHLOR	0.550		
MIREX			
CHORDENE	1.636	MQ	-1.6
OXYCHLOR			
γ-CHLORDANE	1.152	I	0.00
α-CHLORDANE	0.692		1.2

blank=not found; NQ=detected but not quantitated; NA=not analyzed for; M=merged peak; D=detected but not integrated; O=area obscured; I=improper integration.

ANALYSIS REPORT — PESTICIDES + PCB's

Sample: PS200 neutral — Oakland secondary Effluent

Lab:
Sample Size:
Date Sampled

Quantitation Standard:
Quantitation Method:

COMPOUND	PPB		ΔT
α-BHC			
β-BHC			
γ-BHC (LINDANE	0.155		+1.2
δ-BHC			
P,P'-DDD	0.151		+1.2
P,P'-DDE			
P,P'-DDT			
DIELDRIN			
ENDRIN			
ENDRINALDEHYDE			
α-ENDOSULFAN	0.039		1.2
β-ENDOSULFAN	0.141		2.4
ENDOSULFANE SULFATE			
HEPTACHLOR			
HEPTACHLOR EPOXIDE	0.4049		+1.8
PCB 1242			
PCB 1254			
PCB 1221			
PCB 1232			
PCB 1248			
PCB 1260			
PCB 1016			
ALDRIN	0		
METHOXYCHLOR			
MIREX			
CHORDENE	0.060	I	3.0
OXYCHLOR			
γ-CHLORDANE	0.221		0.6
α-CHLORDANE			

blank=not found; NQ=detected but not quantitated; NA=not analyzed for; M=merged peak; D=detected but not integrated; O=area obscured; I=improper integration.

ANALYSIS REPORT — PESTICIDES + PCB's

Sample: PS210 neutral — Oakland secondary effluent dechlorinated

Lab:
Sample Size:
Date Sampled

Quantitation Standard:
Quantitation Method:

COMPOUND	PPB		ΔT
α-BHC			
β-BHC			
γ-BHC (LINDANE	0.139		+0.6
δ-BHC	0.867		+0.6
P,P'-DDD			
P,P'-DDE			
P,P'-DDT			
DIELDRIN			
ENDRIN			
ENDRINALDEHYDE			
α-ENDOSULFAN			
β-ENDOSULFAN			
ENDOSULFANE SULFATE			
HEPTACHLOR			
HEPTACHLOR EPOXIDE			
PCB 1242			
PCB 1254			
PCB 1221			
PCB 1232			
PCB 1248			
PCB 1260			
PCB 1016			
ALDRIN			+0.6
METHOXYCHLOR			
MIREX			
CHORDENE	0.071	I	1.2
OXYCHLOR			
γ-CHLORDANE		D	-2.4
α-CHLORDANE			

blank=not found; NQ=detected but not quantitated; NA=not analyzed for; M=merged peak; D=detected but not integrated; O=area obscured; I=improper integration.

ANALYSIS REPORT PESTICIDES + PCB's

Sample: PS220 neutral Oakland Sludge

Lab:
Sample Size:
Date Sampled

Quantitation Standard:
Quantitation Method:

COMPOUND	PPB		ΔT
α-BHC			
β-BHC			
γ-BHC (LINDANE	0.122		+1.2
δ-BHC			
P,P'-DDD	0.273		-0.6
P,P'-DDE			
P,P'-DDT	0.550		-3.6
DIELDRIN	0.149		0
ENDRIN			
ENDRINALDEHYDE			
α-ENDOSULFAN	0.386		0.6
β-ENDOSULFAN	0.403	MI	-4.2
ENDOSULFANE SULFATE	1.327		2.4
HEPTACHLOR			
HEPTACHLOR EPOXIDE			
PCB 1242			
PCB 1254			
PCB 1221			
PCB 1232			
PCB 1248			
PCB 1260			
PCB 1016			
ALDRIN			
METHOXYCHLOR			
MIREX	0.021		+.06
CHORDENE	0.101		1.8
OXYCHLOR			
γ-CHLORDANE	0.72		0.00
α-CHLORDANE			

blank=not found; NQ=detected but not quantitated; NA=not analyzed for; M=merged peak; D=detected but not integrated; O=area obscured; I=improper integration.

ANALYSIS REPORT — PESTICIDES + PCB's

Sample: 2-119 neutral — Oakland Blank

Lab:
Sample Size:
Date Sampled

Quantitation Standard:
Quantitation Method:

COMPOUND	PPB		TΔ
α-BHC			
β-BHC			
γ-BHC (LINDANE	0.127	I	+0.6
δ-BHC			
P,P'-DDD	0.159		+1.8
P,P'-DDE			
P,P'-DDT	0.404		0
DIELDRIN		M	-1.2
ENDRIN	0.83		0
ENDRINALDEHYDE			
α-ENDOSULFAN	0.012		-0.6
β-ENDOSULFAN	0.043		-3.0
ENDOSULFANE SULFATE			
HEPTACHLOR			
HEPTACHLOR EPOXIDE			0
PCB 1242			
PCB 1254			
PCB 1221			
PCB 1232			
PCB 1248			
PCB 1260			
PCB 1016			
ALDRIN			
METHOXYCHLOR			
MIREX			-1.2
CHORDENE			
OXYCHLOR			
γ-CHLORDANE			
α-CHLORDANE			

blank=not found; NQ=detected but not quantitated; NA=not analyzed for; M=merged peak; D=detected but not integrated; O=area obscured; I=improper integration.