Environmental Protection Technology Series

Environmental Applications of Advanced Instrumental Analyses: Assistance Projects, FY 72

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U.S. Environmental Protection Agency
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ENVIRONMENTAL APPLICATIONS OF ADVANCED INSTRUMENTAL ANALYSES: ASSISTANCE PROJECTS, FY 72

By

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ABSTRACT

Identification and measurement of aquatic pollutants are discussed under 13 project categories. In most cases these analyses helped to solve, or at least to understand more clearly, the related pollution incident and in some cases provided evidence for enforcement of regulatory legislation.

CONTENTS

		Page
Abstract		ii
List of Figures		v
List of Tables		vii
Acknowledgments		viii
Sections		
I	Conclusions	1
II	Recommendations	2
III	Introduction	3
IV	Projects	4
	1. Excess Color in Municipal Wastes	4
	2. Organic Pollutants Associated with Power Plant Cooling Water	7
	3. Western Louisiana Industrial Waste Survey	18
	4. Polychlorinated Biphenyls in Industrial Effluent	22
	5. Organic Pollutants in Industrialized Shipping Channel	25
	6. Taste and Odor Problems in Municipal Water Supply	27
	7. Organic Components of Laboratory Waste Water	31
	8. Polychlorinated Biphenyls in Poultry Fat	32

Sections		Page
9.	Titanium in Paint Manufacturing Plant Effluent	33
10.	Zinc in Metal Finishing Plant Effluent	33
11.	Transition Elements in Clay Minerals	34
12.	Cadmium and Nickel in Battery Manufacturing Plant Effluent	35
13.	Arsenic in Gold Mine Tailings and Waste Water	37
V	References	44
VI	Glossary of Abbreviations	46

FIGURES

No.		Page
1	Mass Spectrum of Suspect Blue Pigment, Identified as Copper Phthalocyanine	8
2	Map of Texas Power Generating Plant Sampling Area	9
3	Computer Reconstructed Gas Chromatograms of Neutral Fractions of Samples #8, #17, and Blank	12
4	Computer Reconstructed Gas Chromatograms of Acidic Fractions of Samples #8, #17, and Blank	13
5	Computer Reconstructed Gas Chromatograms of Basic Fractions of Samples #8, #17, and Blank	14
6	Gas Chromatograms (FID) of Neutral Fractions of Samples #6, #7, and #8.	16
7	Gas Chromatograms (FID) of Neutral Fractions of Samples #1, #18, and #23	17
8	Chemically Characterized Gas Chromatogram of Petrochemical Company Effluent	19
9	Computer Reconstructed Gas Chromatogram of the Chloroform Extract of a Petrochemical Company Effluent	20
10	Computer Reconstructed Gas Chromatogram of a Manufacturing Plant Effluent	23
11	Computer Reconstructed Gas Chromatogram of the Hexachlorobiphenyl Molecular Ion Region of a Manufacturing Plant Effluent	24
12	Computer Reconstructed Gas Chromatogram of Houston Channel Water Sample	26

FIGURES (cont.)

No.		Page
13	Gas Chromatogram of New Orleans Drinking Water Extract	30
14	Map of Sampling Area for Arsenic from Gold Mine Tailings and Waste Waters	39

TABLES

No.		Page
1	Analyzed Samples from Texas Power Generating Plant Area	10
2	Compounds Identified in Petrochemical Company Effluent	21
3	GC and GC-MS Analyses of Terpenes	29
4	Cadmium Content of Foundry Cove Water and Sediment Samples	36
5	Nickel Content of Foundry Cove Sediment Samples	37
6	Arsenic Content of Water Samples	40
7	Arsenic Content of Sediment Samples	43

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SECTION I

CONCLUSIONS

The identification and quantification of specific chemicals responsible for a wide variety of pollution incidents helped to solve, or at least to understand more clearly, the problems associated with the incidents. In some cases evidence was provided for enforcement of regulatory legislation. These projects illustrate the need for many different analytical techniques to identify pollution sources. Continued development of new methods and improvement of existing techniques are required.

SECTION II

RECOMMENDATIONS

Analytical data about specific past and present pollutant sources should be used to establish and enforce effluent water quality standards for the industrial permit program and to update these standards as information is obtained. More laboratories concerned with environmental quality should be equipped to perform analyses such as those described in this report. Existing analytical techniques should be continually improved, and new techniques should be investigated for applicability to pollutant analyses. Information about specific pollution incidents should be widely disseminated to help solve and perhaps prevent future environmental problems.

SECTION III

INTRODUCTION

The National Water Contaminants Characterization Research Program (NWCCRP) at the Southeast Environmental Research Laboratory (SERL) develops techniques for identifying and quantifying chemical pollutants and identifies specific compounds associated with various pollution sources. The NWCCRP has analyzed many samples related to a variety of specific pollution problems. Analytical results were reported only to the persons who requested the analyses and therefore had limited distribution. The problems studied by the NWCCRP are briefly summarized in annual reports to acquaint other researchers and administrators with the type of information that can be obtained and to inform environmental chemists of technique applications and developments. This report summarizes fiscal year 1972 projects.

SECTION IV

PROJECTS

EXCESS COLOR IN MUNICIPAL WASTES

A variety of analytical techniques were used to identify colored components of municipal waste treatment plant influent. In July 1971, the city of Springfield, Missouri, requested assistance with a recurring problem of excess red color in their sewage treatment plant influent. On several occasions from spring through fall during a ten-year period, the treatment plant influent contained a red material that traveled apparently unchanged through the primary clarifiers and the activated sludge secondary treatment. This red material seemed to lower the effluent's dissolved oxygen content and interfered with settling of the sludge solids, some of which passed through the treatment system along with the absorbed red color. Treatment efficiency recovered rapidly when the colored slug passed.

Periodic sampling of area industrial wastes showed none containing reddish materials. Then, quite by accident, treatment plant personnel allowed a sample from a large cheese manufacturing plant to stand in sunlight for several hours. The color changed from the original milkish white to a red similar to that observed at the treatment plant. Cheese company officials had no idea what had caused the coloration. Treatment plant officials suspected the color might be caused by a disinfectant or cleaner used in the cheese plant.

A turbid, reddish sample of treatment plant influent was sent to the SERL for analysis in early August. The influent chloroform extract was separated into three colored components (two orange and one green) by thin-layer chromatography (TLC). The two orange compounds were suspected to be coloring materials used in the cheese plant. Their chemical and physical behavior, including visible absorption spectra obtained after elution from the TLC adsorbent, indicated they were carotenoids but probably not alpha and beta-carotene.

In mid-August a different red influent appeared at the treatment plant; it was bright pink and not very turbid. The pink sample was extracted with chloroform and chromatographed on a thin layer plate. The single intense pink spot matched a Rhodamine B standard spot in both retention time and the brilliant orange fluorescence observed under ultraviolet light. After elution from the TLC adsorbent, the pink material was examined by ultraviolet (UV) and infrared (IR) spectroscopy; these spectra were identical with standard spectra. The influent sample contained approximately 1 mg Rhodamine B/ℓ, calculated from UV measurements.

Rhodamine B is a common dye for a variety of materials: textiles, paper, leather, China clay, crayons, feathers, inks, lacquers, drugs, cosmetics, and various foodstuffs. Three possible sources in the Springfield area were a pharmaceutical firm, a paint and lacquer firm, and a paper cup manufacturer. The paper cup manufacturing plant was the suspected source because the company began using a particular red ink about the time the treatment plant began experiencing occasional difficulties.

When a third red color incident occurred in April 1972 treatment plant officials obtained another influent sample and a red ink sample from the paper cup manufacturing plant. The ink trade name was not available, but manufacturing plant officials stated that it did not contain Rhodamine B.

Methanol solutions were prepared for the red material filtered from influent sample (SR-1) and for the red paper cup ink (PCI). After concentration to a low volume, each methanol solution was examined with TLC. The PCI chromatogram produced five spots after development with ethanol. Sample SR-1 also produced five spots, but most of them were different from the PCI spots. Sample SR-1's major component, an orange-brown spot with no fluorescence, matched in retention time, color, and lack of fluorescence a medium intensity spot in Sample PCI. Some orange-brown material from each sample was separated with preparative TLC, dissolved in methanol, and analyzed with visible spectrophotometry. The visible spectra matched perfectly, both absorbing between 450 and 510 nm with

a broad maximum from 480 to 410 nm. Another SR-1 component, a visible peach-colored spot (brilliant greenish yellow fluorescence in ultraviolet light) matched in color, fluorescence, and TLC retention time a less intense spot in PCI. This evidence indicated that two of the main components in the red sewage sample, SR-1, could have originated from the paper cup ink, PCI. Sample differences shown by TLC could have been caused by biological or chemical degradation of PCI to SR-1.

Apparently there was no connection between any of the three occurrences of red treatment plant influent (cheese coloring, Rhodamine B, and paper cup ink). The source of Rhodamine B was never determined. Paper cup manufacturing plant officials were given the evidence that the paper cup ink had some of the same properties as the last red material in the treatment plant influent. Since then no operational problems have been caused by red materials in the Springfield treatment plant influent.

Later in April 1972, another color problem occurred at the Springfield municipal treatment plant; this time the influent was blue. The color was non-extractable with organic solvents and appeared to be due to flocculent material that gradually settled out and deposited on the sides of the plastic sample container. The colored solid was removed from the sides of the container with concentrated sulfuric acid, the only liquid found to dissolve the solid. These properties suggested that the material might be a phthalocyanine pigment, which is often used as the blue pigment in paints. After receiving this information, treatment plant officials obtained a sample from a paint manufacturer that was suspected to be the source of the blue colored material.

Both suspect and sewage pigments dissolved only in concentrated sulfuric acid, produced an olive color in solution, and precipitated upon dilution with water. According to the Color Index[1], copper phthalocyanine has all of these properties. Spark source mass spectrometry showed that the influent pigment contained copper. Analysis of the suspect blue pigment by low resolution electron impact mass spectrometry (MS) produced a mass

spectrum (Figure 1) corresponding to that expected for copper phthalocyanine; its unique feature was the parent ion cluster with molecular ions at m/e 575 and 577 in the ratio of 2:1, the natural abundance of ^{63}Cu to ^{65}Cu. The mass spectrum of the suspect blue pigment matched that of the blue pigment from the sewage plant influent.

A sample of copper phthalocyanine was synthesized in the laboratory by reacting phthalonitrile with copper. The mass spectrum of the synthetic sample matched those of the suspect and influent pigments.

Treatment plant officials presented this information to the paint manufacturing company officials, who admitted their use of copper phthalocyanine. No more blue material has appeared in treatment plant influent.

ORGANIC POLLUTANTS ASSOCIATED WITH POWER PLANT COOLING WATER

The Houston Ship Channel, which receives many industrial discharges, is the source of cooling water for a large power generating plant. Cooling water flows through a canal to the oil-fired power generating plant and is discharged into a nearby bay adjacent to important shrimp-producing estuaries (Figure 2). Present discharges may be detrimental to receiving water quality, and even larger volumes of cooling water will be required for planned power plant expansion. The Environmental Protection Agency (EPA) needed data to evaluate biologic and hydrologic studies prepared by power plant officials. The NWCCRP was requested by the EPA's Office of Standards and Enforcement, Washington, D.C. to identify specific organic contaminants by combined gas chromatography-mass spectrometry (GC-MS) and to qualitatively compare samples by gas chromatographic (GC) fingerprinting to establish relative degrees of pollution.

Samples were collected from nine sites (Table 1) during a four day period, stored in 5-gal. plastic containers on ice until received at a mobile laboratory, and kept frozen until extracted. A blank sample (10 ℓ distilled water) was also stored frozen in a 5-gal. plastic container for six days and extracted and analyzed by the same procedure used for the samples. Each sample

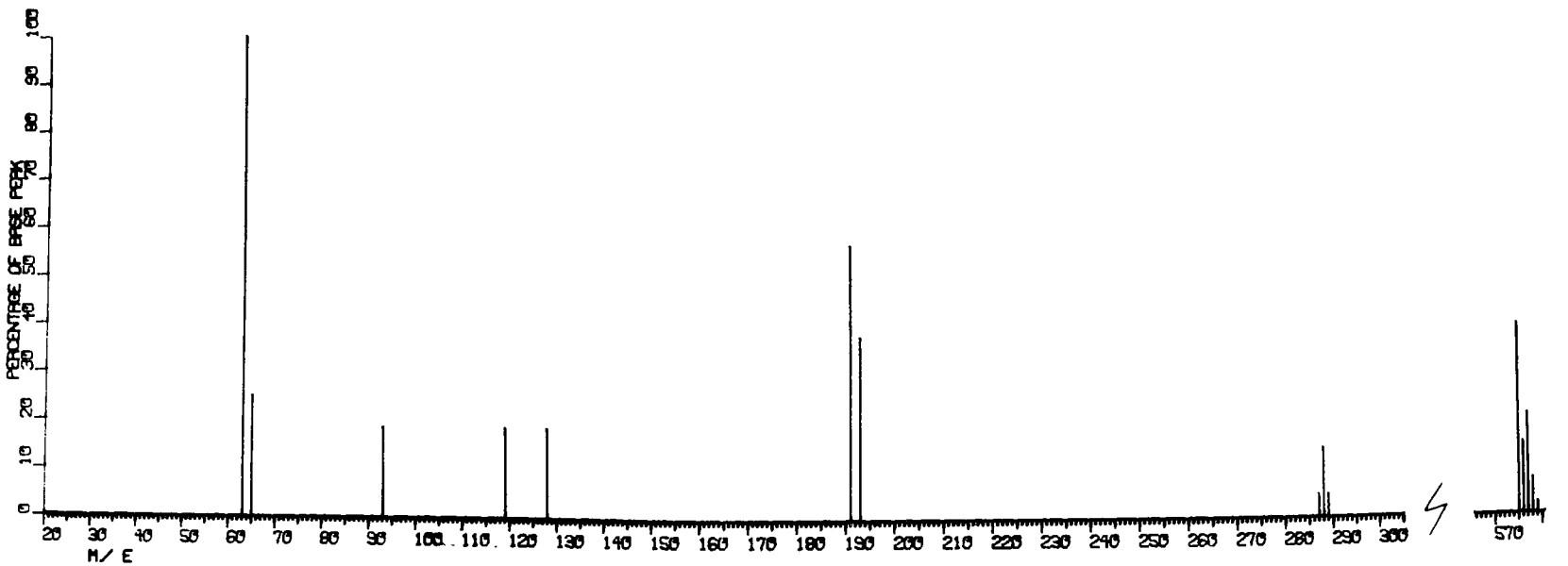

Figure 1. Mass Spectrum of Suspect Blue Pigment, Identified as Copper Phthalocyanine

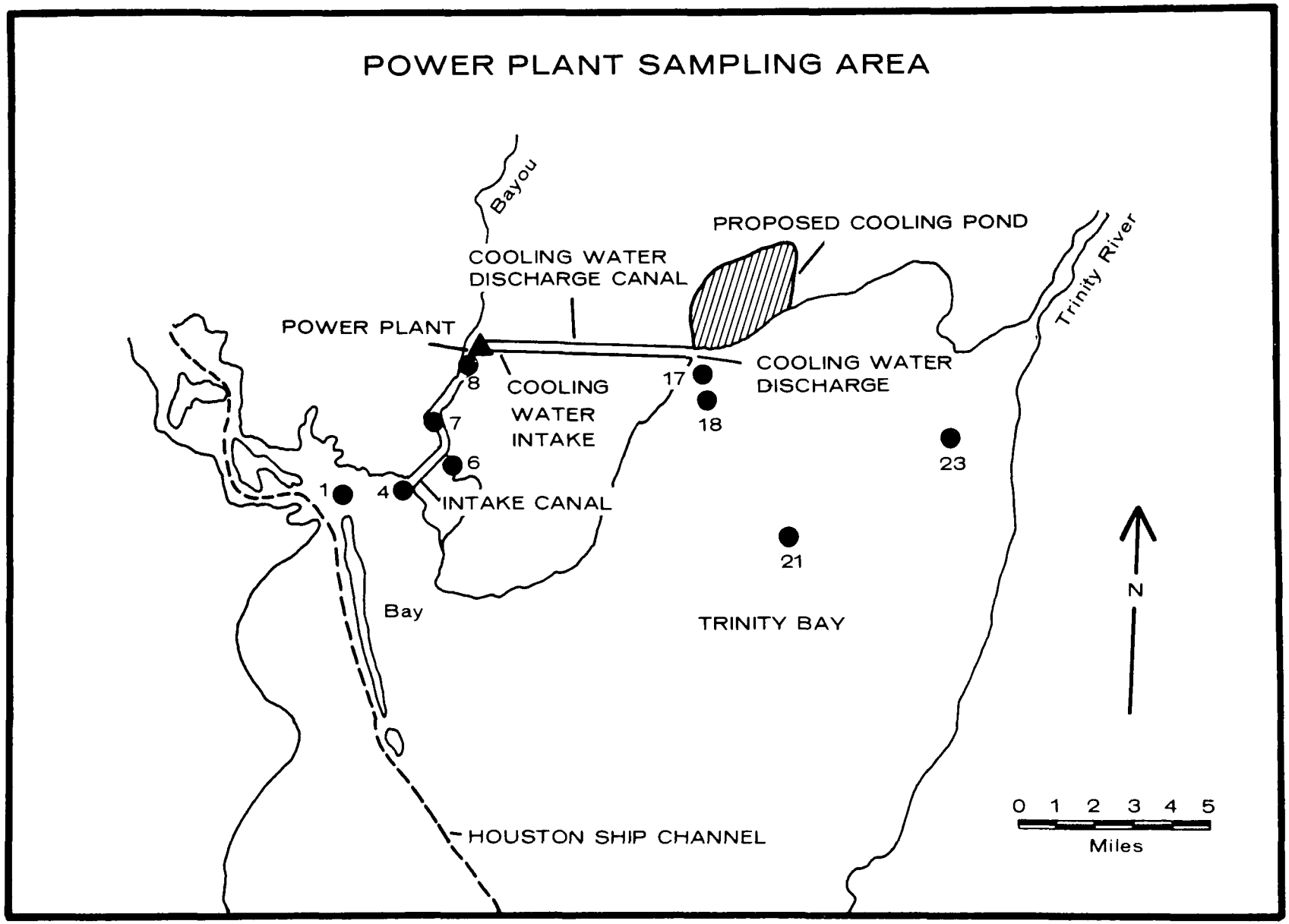

Figure 2. Map of Texas Power Generating Plant Sampling Area

Table 1

Collection Sites for Samples Analyzed at the SERL

Sample #	Collection Site
1	Dredged shipping channel water collected at center of channel near dredged intake canal
4	Water collected at mouth of dredged cooling system intake canal
6	Bayou water collected 50 ft downstream (south) from intersection of bayou and cooling system intake canal
7	Bayou water collected 50 ft upstream (north) from intersection of bayou and cooling system intake canal
8	Bayou water collected 50 ft downstream of power plant cooling system intake
17	Bay water collected 0.5 mi due south of cooling water canal discharge point
18	Bay water collected 1 mi due south of cooling water canal discharge point
21	Bay water collected from center of bay approximately 5 mi southeast of cooling water canal discharge point
23	Bay water collected near river mouth approximately 5 mi northeast of site 21

was made acidic and extracted with chloroform to obtain neutral and acidic materials; the water layer from this extraction was made basic and extracted again with chloroform to obtain basic materials. Each extract (3200 ml total) was concentrated to about 3 ml, stored in a vial, and shipped on dry ice to the SERL. A second extraction procedure[2] was used to separate acidic and neutral organics. To allow GC analyses, acidic materials were converted to methyl esters or ethers with diazomethane.[3] The volume of each extract was reduced to 50 μℓ, and a 2-μℓ aliquot was injected into the GC to be separated on a 50-ft Carbowax 20M-TPA SCOT column. For GC-MS analyses sample volumes were reduced as necessary for sensitivity requirements.

Few organic compounds were specifically identified. Computer reconstructed gas chromatograms (RGC) of neutral, acidic, and basic fractions of cooling system influent (sample #8), bay water collected 0.5 mi south of cooling water discharge point (sample #17), and the blank are shown in Figures 3-5. The blank fractions contained several organic compounds; peaks corresponding to blank compounds are shaded in each sample RGC. Some of these organics were identified from their mass spectra. In the blank fractions most of the wide peaks with longer retention times are plasticizers (phthalate and adipate esters) that were probably introduced during laboratory procedures. Other peaks are due to solvent impurities and artifacts produced during methylation of the acid fractions.

Comparison of mass spectra and GC retention times showed that several compounds were present in the samples that were not present in the blank. Mass spectra of compounds unique to sample fractions were compared with mass spectra in data collections that contained spectra of approximately 17,000 compounds.

In the neutral fraction of the cooling system influent (sample #8, Figure 3) triethyl phosphate and benzothiazole were identified; they were not found in neutral fractions of either the blank or the bay sample (#17) collected 0.5 mi from the discharge point. Comparison of sample and standard GC peak heights indicated the concentration of each was approximately 1 μg/ℓ. Azulene and 1- and 2-methylnaphthalene were

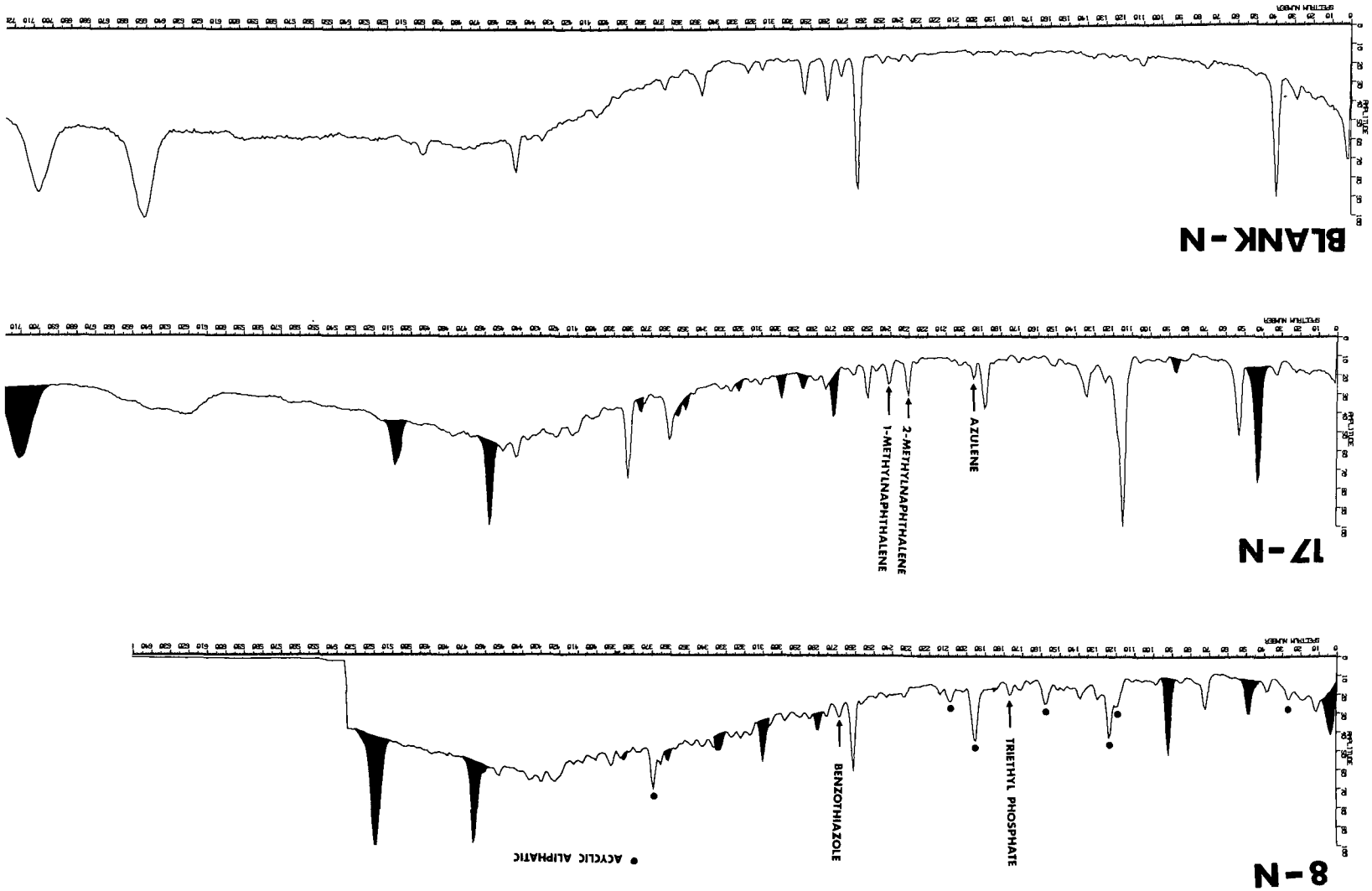

Figure 3. Computer Reconstructed Gas Chromatograms of Neutral Fractions of Samples #8, #17, and Blank. (Shaded Peaks Were in Blank.)

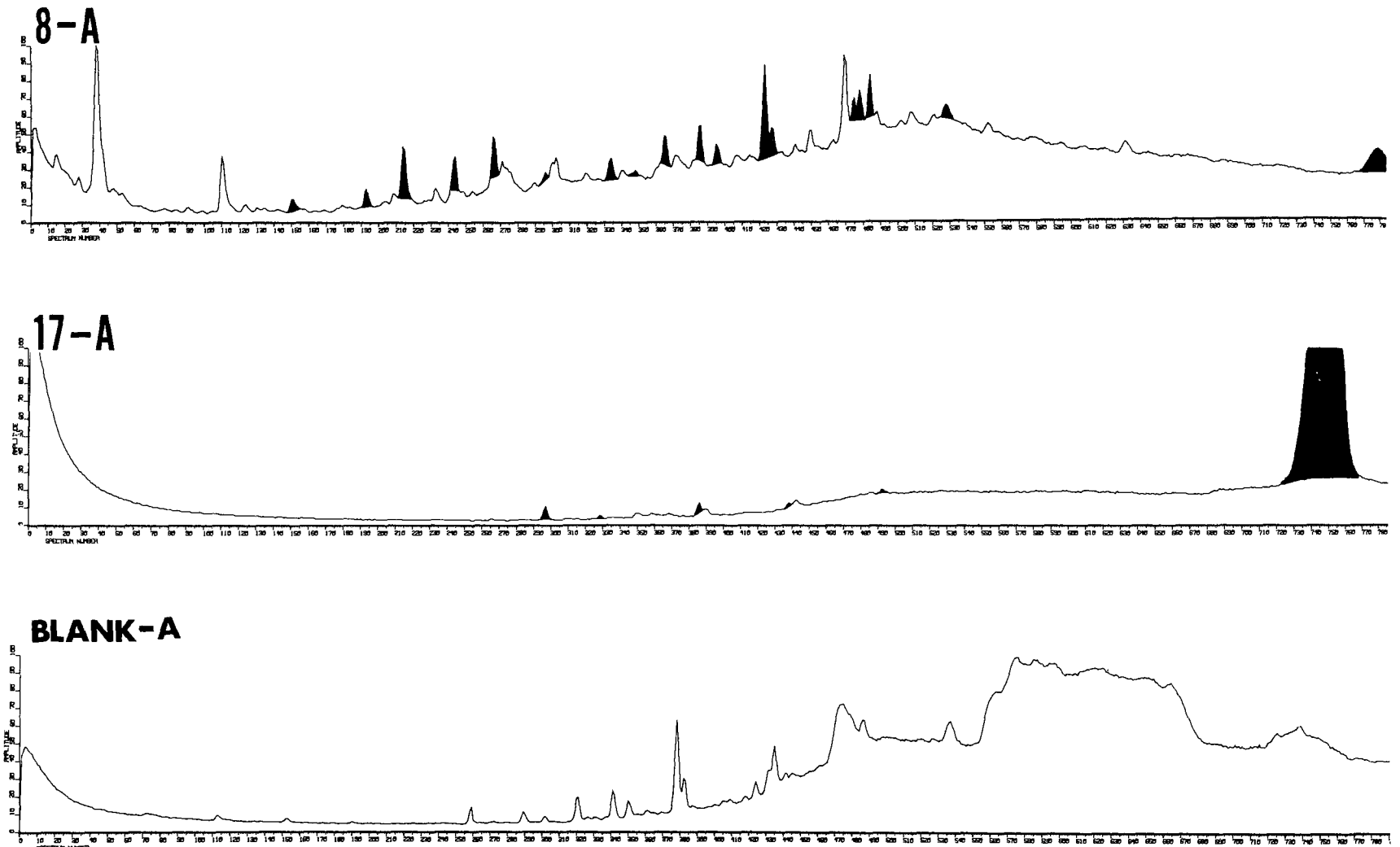

Figure 4. Computer Reconstructed Gas Chromatograms of Acidic Fractions of Samples #8, #17, and Blank. (Shaded Peaks Are in Blank.)

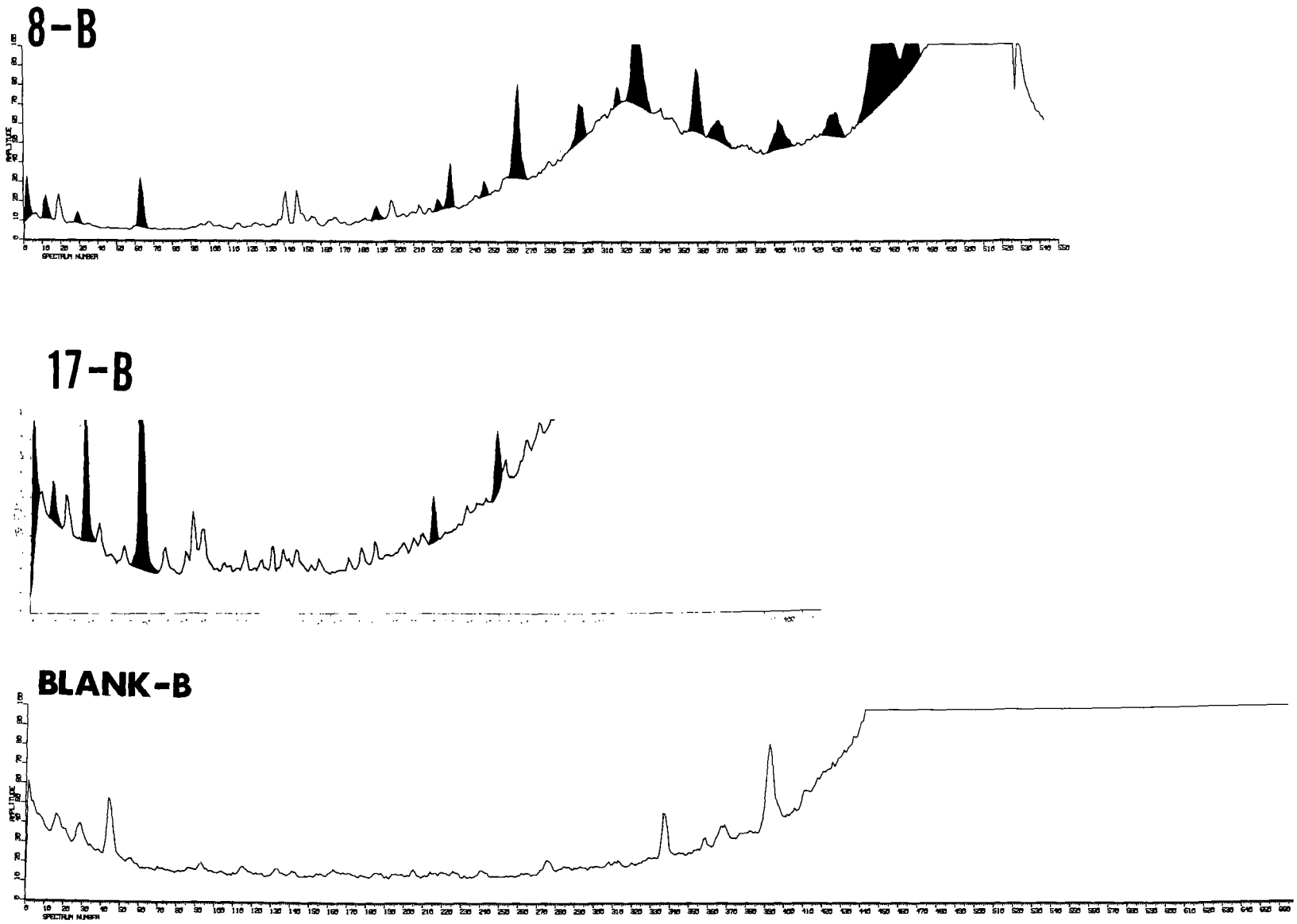

Figure 5. Computer Reconstructed Gas Chromatograms of Basic Fractions of Samples #8, #17, and Blank. (Shaded Peaks Were in Blank.)

identified in the bay sample neutral fraction (Figure 3). No spectral matches were found for the other neutral water pollutants represented by unshaded peaks. Several acyclic aliphatics (peaks indicated in Figure 6) were probably alkanes or oxygenated alkanes but were not specifically identified. The neutral fraction of a bay water sample collected 1 mi south of cooling water canal discharge point (#18) contained triethyl phosphate (a compound also identified in the influent sample), two dichloro biphenyl isomers, and two ethyl esters of long chain saturated acids. Exact structures of the latter four compounds were not determined.

Although water pollutants in acid fractions of the influent and bay water sample #17 (Figure 4) were not specifically identified, the chromatograms show that the bay sample (#17) contained fewer organics in lower concentrations than the cooling system influent (#8).

The basic fraction of the influent (sample #8) contained few organic compounds not present in the blank (Figure 5); bay sample #17 contained more organic compounds than the influent sample. The basic pollutants were not identified.

Comparison of flame ionization gas chromatograms of neutral fractions of samples #1, #6, #7, #8, #18, and #23 indicated the relative organic content of waters from different areas (collection sites in Figure 2). Peaks also found in the blank are shaded in the sample chromatograms (Figures 6 and 7).

This organic pollutant information was reported to the EPA Region VI Enforcement Division for inclusion with other analytical data (such as dissolved oxygen, total organic carbon, metals, total residue, total and Kjeldahl nitrogen, sulfates, salinity, pH, temperature, chlorides, chlorinated hydrocarbons, and various biological parameters), which were obtained from other EPA laboratories.

A report was prepared to support litigation. However, in January 1973 the issue was settled out of court when power plant officials agreed to monitor chemical and biological parameters of discharged cooling water and Trinity Bay water.

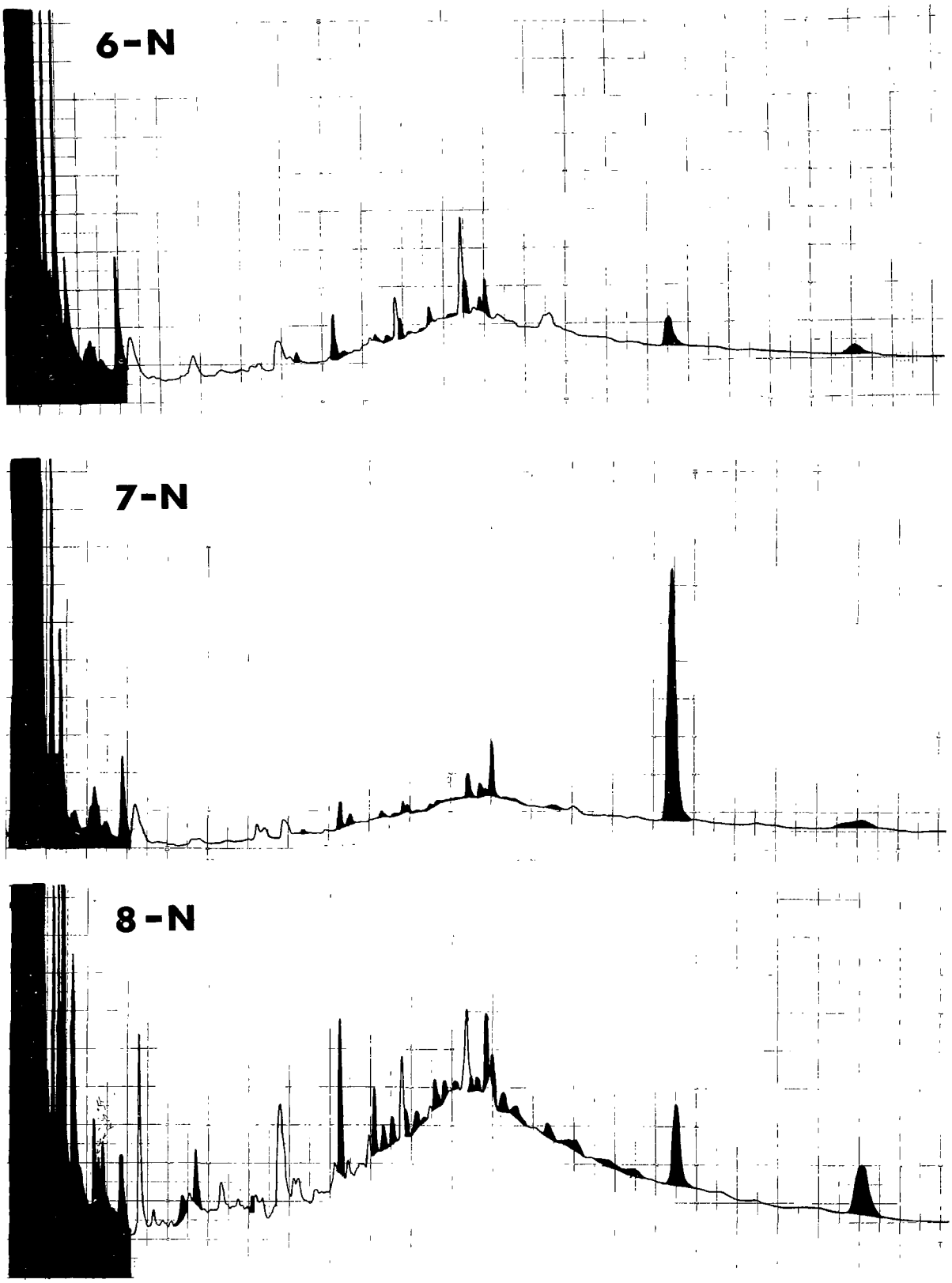

Figure 6. Gas Chromatograms (FID) of Neutral Fractions of Samples #6, #7, and #8. Shaded Peaks Were Also in Blank Chromatogram.

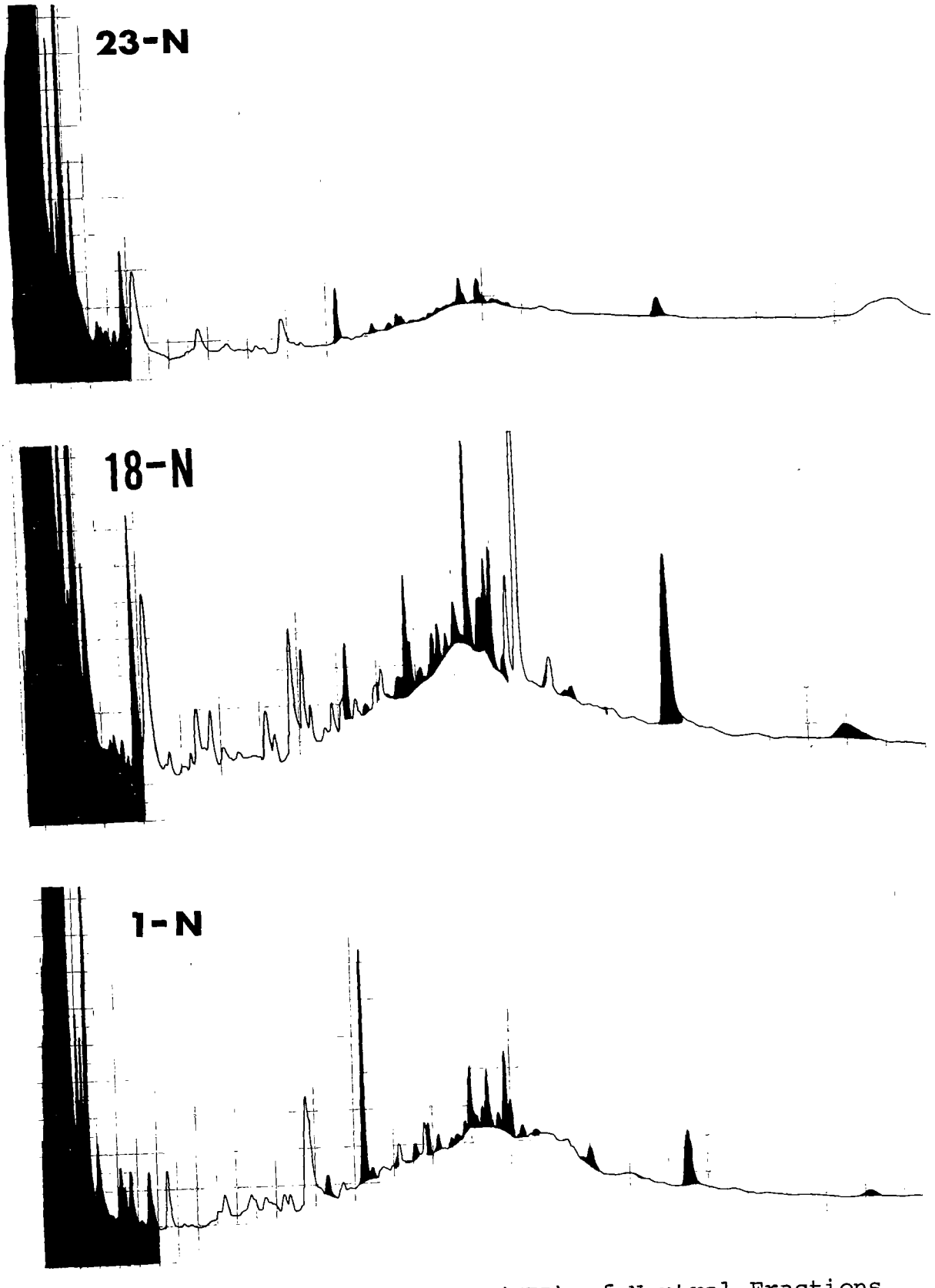

Figure 7. Gas Chromatograms (FID) of Neutral Fractions of Samples #1, #18, and #23. (Shaded Peaks Were Also in Blank.)

WESTERN LOUISIANA INDUSTRIAL WASTE SURVEY

In April 1971, the EPA Division of Field Investigations, Denver, Colorado, requested the NWCCRP to characterize chemically a number of industrial effluents from an industrialized area in western Louisiana. One liter of each effluent sample was extracted with chloroform, and an aliquot of each concentrated extract was injected into a gas chromatograph to establish optimum GC conditions. Analyses with the Hitachi-Perkin-Elmer RMU-7 GC-MS, a non-computer-controlled system, provided 50 identifications for compounds in seven different industrial effluents.[4] Some compounds occurred in more than one effluent.

The most complex mixture of organic compounds was found in the effluent from a petrochemical company that produces olefins and oxygenated hydrocarbons. Plant effluent, estimated to be 4,000,000 gal/day, flows through three treatment ponds connected in series and is discharged into a bayou. Samples were collected where the treated effluent entered the bayou. Initial analysis of this effluent with the non-computer-controlled GC-MS system provided identifications of 14 compounds (Figure 8).

After the NWCCRP obtained a Finnigan 1015 GC-MS-computer system, the sample was analyzed again in February 1972. As GC peaks eluted, mass spectra were collected continuously and stored in the computer for later output in tabular or plotted form. Appropriate spectra from the RGC (Figure 9) provided fragmentation patterns of compounds not previously identified. Computer matching of these unknown spectra with standard spectra in a central data bank located in a large computer at the Battelle Research Institute, Columbus, Ohio,[5] provided identifications for 15 additional components. The improved data handling capability of the computer-controlled system allowed the chemist to identify another eight components. A total of 37 different compounds were identified in the treated effluent of this one point (Table 2). All major components and more than 70% of all peaks and shoulders in the chromatogram were specifically identified. The impact of each of these compounds on water quality has not yet been determined, but polycyclic aromatics such

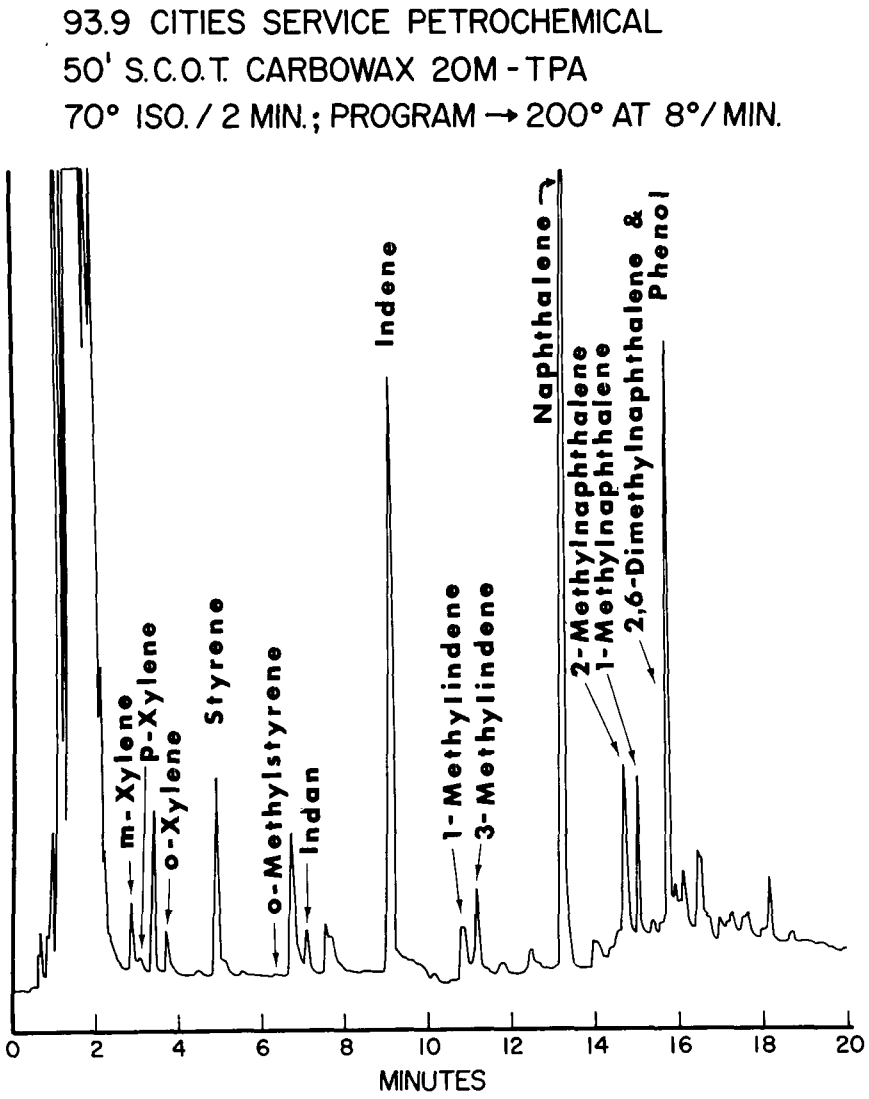

Figure 8. Chemically Characterized Gas Chromatogram of Petrochemical Company Effluent

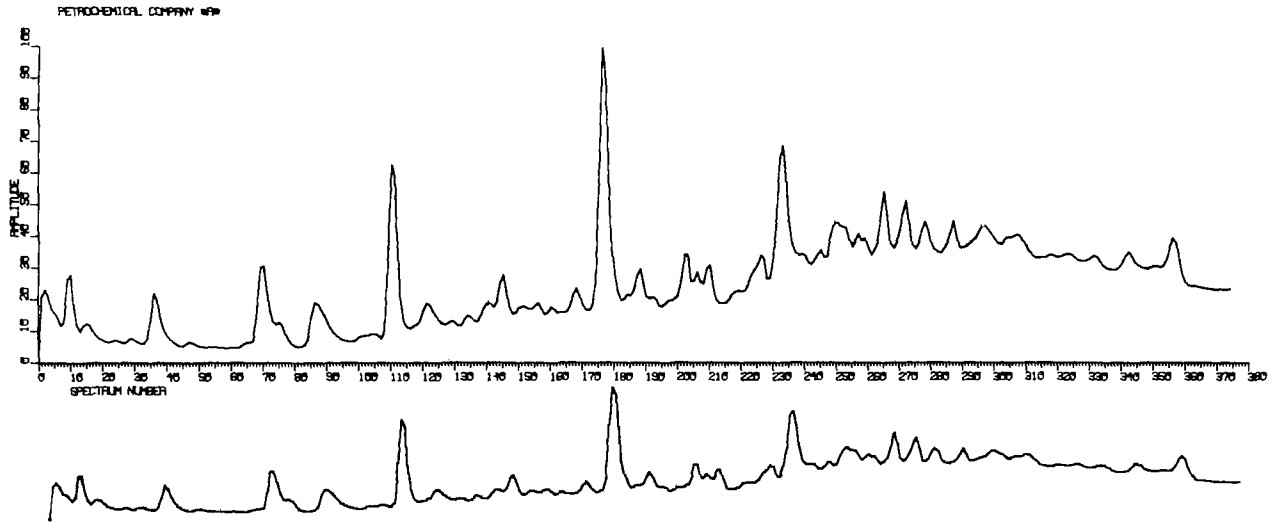

Figure 9. Computer Reconstructed Gas Chromatogram of the Chloroform Extract of Petrochemical Company Effluent

Table 2

Compounds Identified in Petrochemical Company Effluent

RGC Spectrum #[a]	Compound Name
2	m-xylene*[b]
4	p-xylene*[b]
10	1,5-cyclooctadiene
16	o-xylene*[b]
29	isopropylbenzene (cumene)
36	styrene*[b]
47	o-ethyltoluene
65	o-methylstyrene*[b]
70	diacetone alcohol
75	indan*[b]
86	2-butoxyethanol
89	β-methylstyrene
109	indene*[b]
121	dimethylfuran isomer
129	n-pentadecane
140	1-methylindene*[b]
145	3-methylindene
156	acetophenone
160	n-hexadecane
168	α-tepineol
177	naphthalene*[b]
193	α-methylbenzyl alcohol
202	2-methylnaphthalene*[b]
206	benzyl alcohol
210	1-methylnaphthalene*[b]
221	ethylnaphthalene isomer
233	2,6-dimethylnaphthalene*[b]
233	phenol*[b]
244	methylethylnaphthalene isomer
249	cresol isomer
256	acenaphthene
265	acenaphthalene
278	methylbiphenyl isomer
287	fluorene
292	phthalate diester
296	3,3-diphenylpropanol
356	phthalate diester

[a]From Figure 9

[b]Compounds identified with mass spectra from non-computer-controlled GC-MS system.

as indenes and naphthalenes are known to be odor sources[6] and could cause odor problems in drinking water.

At an EPA water quality enforcement conference these identifications provided specific information about the effectiveness of the petrochemical company's waste water treatment.

POLYCHLORINATED BIPHENYLS IN INDUSTRIAL EFFLUENT

An Alabama plant that manufactures organophosphorus pesticides, PCB's, and chlorinated hydrocarbons used as intermediates in the chemical industry was suspected to be a source of polychlorinated biphenyl (PCB) contamination. Effluent samples were collected and analyzed by electron capture gas chromatography; retention times of the observed GC peaks indicated the presence of PCB's. To confirm this identification, the EPA Region IV Surveillance and Analysis Division requested the NWCCRP to analyze a sample by combined GC-MS.

An initial run of the computer-controlled GC-MS system gave the RGC shown in Figure 10. Major peaks eluting before the peak represented by spectrum #15 were readily identified as chlorobenzenes and monochlorobiphenyls. Mass spectra of peaks with longer retention times suggested the presence of chlorinated biphenyls. However, ions from column bleed and from non-PCB sample components prevented the acquisition of an RGC comparable to the electron capture chromatogram.

To circumvent this interference, blank scans were made at highest instrument sensitivity to determine any significant column bleed and instrument background ions in the hexachlorobiphenyl molecular ion region. In a second data acquisition run, the computer was instructed to monitor only ions in the hexachlorobiphenyl molecular ion region but to ignore all significant masses noted in the blank scans. The RGC obtained from the second data run (Figure 11) was comparable to the electron capture chromatogram. This comparison and the chromatographic data indicated the effluent PCB content was approximately 0.5 $\mu g/\ell$.

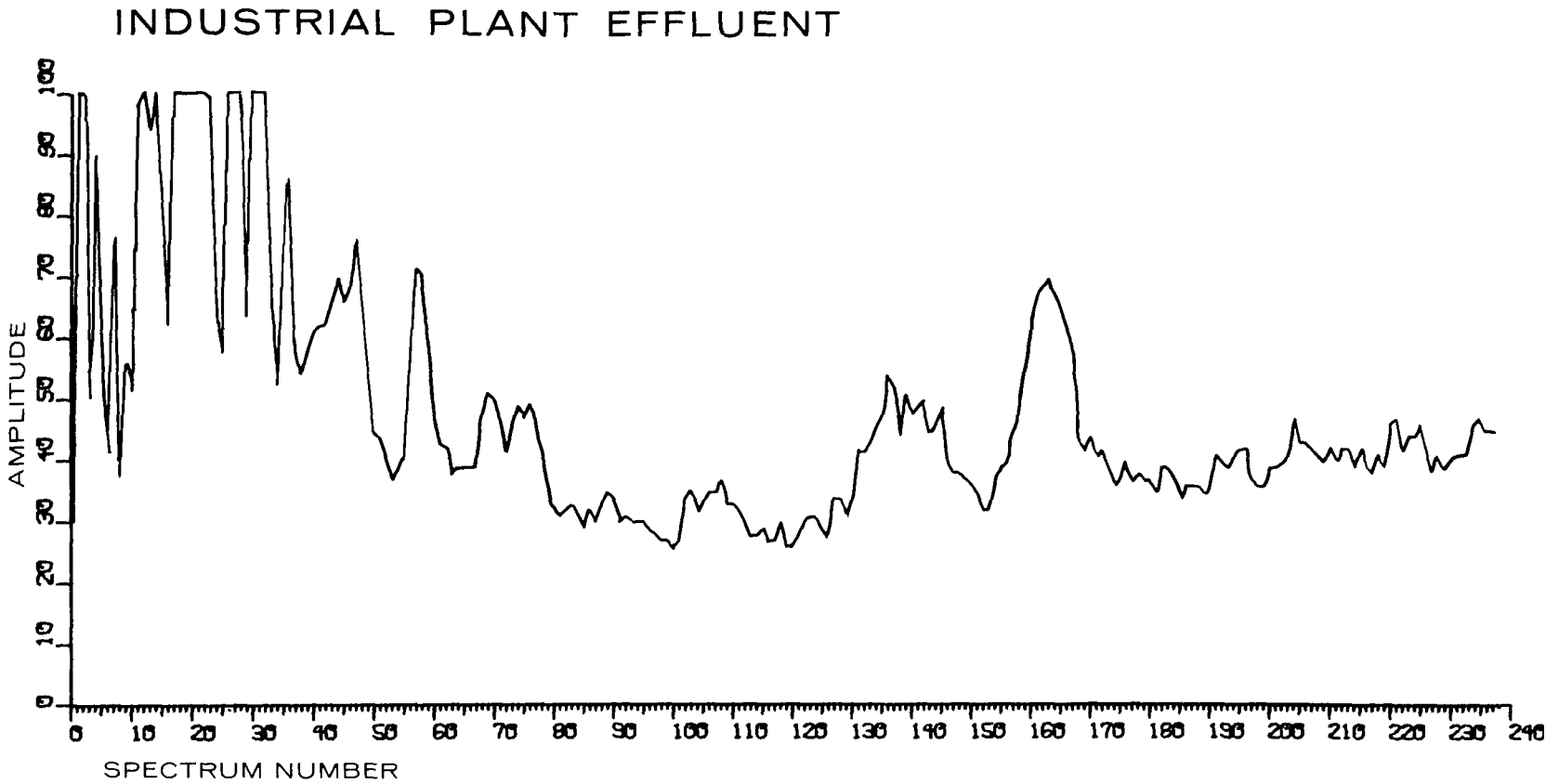

Figure 10. Computer Reconstructed Gas Chromatogram of a Manufacturing Plant Effluent

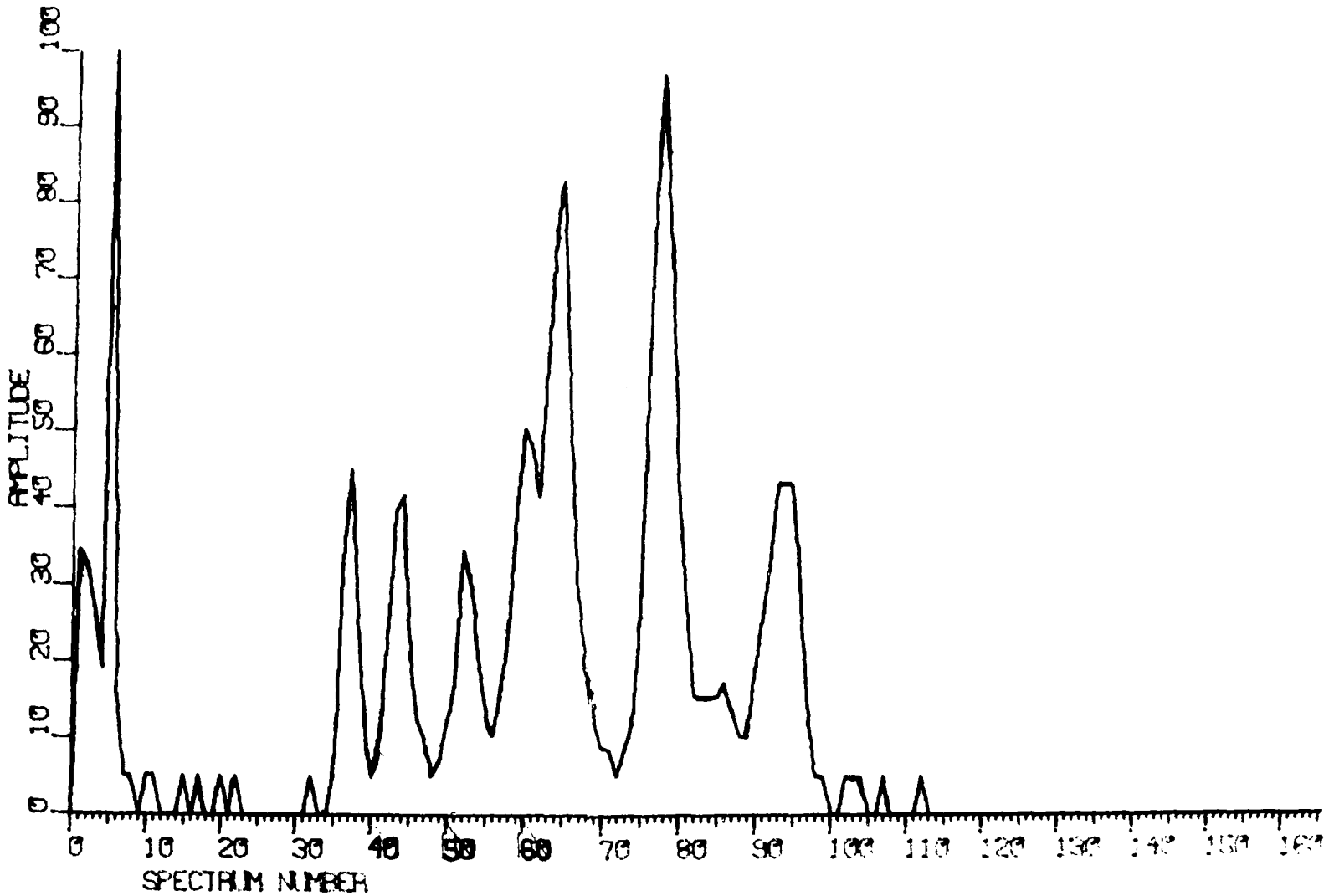

Figure 11. Computer Reconstructed Gas Chromatogram of the Hexachlorobiphenyl Molecular Ion Region of a Manufacturing Plant Effluent

During toxicity studies conducted by the Region IV Surveillance and Analysis Division, caged catfish that were placed in the plant effluent died within 24 hours. The plant effluent flowed into Snow Creek and on into Choccolocco Creek; catfish survived in Choccolocco Creek but tissue analyses showed that PCB's had been absorbed from the water.

The manufacturer has voluntarily limited production and use of PCB's.

ORGANIC POLLUTANTS IN INDUSTRIALIZED SHIPPING CHANNEL

The EPA's National Field Investigations Center, Denver, Colorado, requested identifications of major organic pollutants in the Houston Ship Channel for an enforcement conference concerning pollution of the Channel and Galveston Bay. The Houston Ship Channel probably contains the largest concentration of industrial effluents of any small channel in the United States; 250 major industries dump their effluents there. Channel waters flow into Galveston Bay, an important shrimp nursery and oyster bed along the Gulf of Mexico. The Channel was suspected to be the source of several polyaromatic compounds that had been isolated and identified in Galveston Bay oysters by workers at the Woods Hole Institute.

A 0.5-gal. water sample was collected at each of four sites along the Channel, frozen, and shipped to SERL. A 1-ℓ aliquot of each sample was extracted with a total of 600 ml chloroform, and the extract was concentrated in a Kuderna-Danish apparatus to about 0.1 ml. Gas chromatographic analysis showed that all four samples contained essentially the same compounds, but their concentrations increased as distance upstream from the Channel mouth increased.

The most concentrated sample (#97.4), which was taken 18 miles upstream from the Channel mouth, was analyzed by computer-controlled GC-MS. The RGC (Figure 12) indicates the large number of organic compounds contained in this sample. A limited mass RGC that enhanced peaks containing aromatic compounds showed that only mass spectra 111, 177, 195, 219, 295, and 340 contained mass fragments characteristic of phenyl

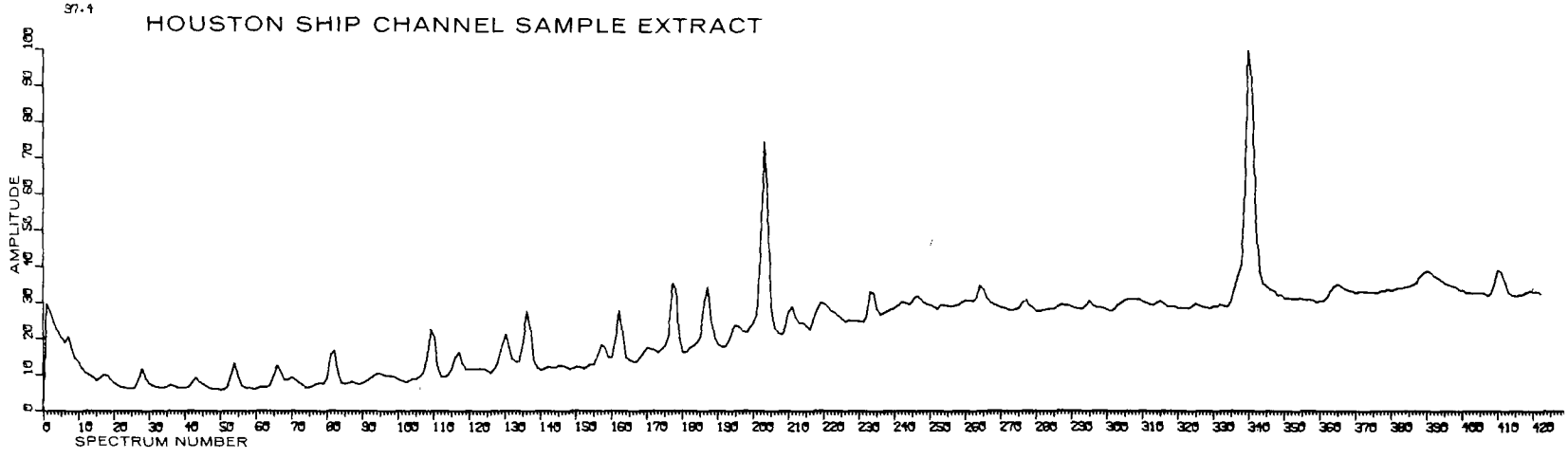

Figure 12. Computer Reconstructed Gas Chromatogram of Houston Ship Channel Water Sample

compounds (m/e 77 and 78). The very minor sample component represented by spectrum 111 was acetophenone, the only aromatic compound identified. Spectra 177, 195, and 219 represented small amounts of mixtures of aliphatic and aromatic compounds. None of the polyaromatic compounds identified in Galveston Bay oysters was found in the Channel sample.

Most of the organic compounds extracted from Sample #97.4 were aliphatic hydrocarbons (spectra 18, 28, 43, 54, 66, 82, 108, 117, 130, 136, 157, 162, and 170). Their GC retention times did not correspond to those of n-alkane standards, and no attempt was made to specifically identify them as branched isomers. Spectra 187 and 211 were due to unresolved alkane mixtures. Spectrum 411 was possibly due to caffeine, and spectrum 204 was due to an unsaturated hydrocarbon that was not specifically identified.

In another attempt to find aromatic compounds, liquid chromatographic analysis of the most concentrated sample (#97.4) showed four peaks whose retention times did not match any of the polyaromatic compounds previously identified in Galveston Bay oysters.

Although no link was found between the oyster contaminants and Channel pollutants, this characterization information was used as background material for an Enforcement Conference held in November 1971. The conference led to a clean-up schedule and allocation of industrial waste loads to be dumped into the Houston Ship Channel.

TASTE AND ODOR PROBLEMS IN MUNICIPAL WATER SUPPLY

The EPA's Lower Mississippi River Field Facility (LMRFF) requested assistance with correlation of drinking water contaminants and industrial effluent components. Taste and problems have long been associated with the drinking water of New Orleans and other municipalities that use the Lower Mississippi River as their water source. Several manufacturing plants along the river were suspected to be sources of organic compounds causing these problems. The NWCCRP's GC-MS system had proven to be a powerful tool for

studying this type problem, but such a system was not available at the LMRFF laboratory.

GC-MS analysis of the carbon-chloroform extract of a New Orleans finished water sample produced identifications of five compounds: 2-benzothiazole, 2-ethylhexanol, veratrole, camphor, and isoborneol (Figure 13). 2-Benzothiazole is often used in the synthetic rubber industry and has a strong odor very similar to that of rubber cement used to patch holes in rubber tires. This compound could cause a significant taste and odor problem. 2-Ethylhexanol probably does not cause taste and odor problems but was one of the extract's major components. Veratrole, camphor, and isoborneol are terpenes, common components of paper mill waste effluent, and could have contributed to the taste and odor problem.

Ether extracts of effluents from two upriver paper mills were also examined by GC and GC-MS to learn if chemicals from these mills were contributing to the taste and odor problems in the New Orleans drinking water. Veratrole was identified and confirmed in both paper mill effluents. Camphor was found in one paper mill effluent; it was believed to be present in the other effluent but was present in such small quantity that it could not be identified with certainty. Although an isoborneol standard had the same GC retention time as a small peak in one paper mill effluent, the mass spectra did not match. Isoborneol was not found in either plant effluent.

This project emphasized that GC identifications of compounds from complex mixtures should always be confirmed by MS or some other physical/chemical method. GC evidence indicated that the finished water contained three terpenes that had been identified by GC-MS in one or both paper mill effluents.

- GC retention times of these three terpenes (guaiacol, α-terpineol, and borneol) matched retention times of three peaks in the paper mill effluents (Table 3).

- Drinking water extracts spiked with small amounts of these three terpene standards

Table 3. GC AND GC-MS ANALYSES OF TERPENES

Chemical	Presence Indicated by GC[a]		Presence Confirmed by GC-MS
	Carbowax 20-M[b]	SE-30[b]	
Carbon Chloroform Extract of Finished Water			
Camphor	+	+	+[c]
Veratrole	+	+	+
Guaiacol	-	+	-
α-Terpineol	+	+	-
Borneol	+	+	-
Isoborneol	+	+	+[c]
Paper Mill "A" Effluent			
Camphor	+	+	+
Veratrole	+	+	+
Guaiacol	+	+	+
α-Terpineol	+	+	+
Borneol	+	+	+
Isoborneol	+	+	-
Paper Mill "B" Effluent			
Camphor	+	+	+[c]
Veratrole	+	-	+
Guaiacol	+	+	+
α-Terpineol	+	+	-
Borneol	-	-	-
Isoborneol	-	-	-

[a]Retention times were not relied on alone--sample was spiked with small amounts of standards to produce superimposed peaks.
[b]50' Support coated open tubular column
[c]Sample peak very small--major fragmentations of standard were present along with extraneous fragment ions from an unknown contaminant. Background was subtracted from the spectrum.

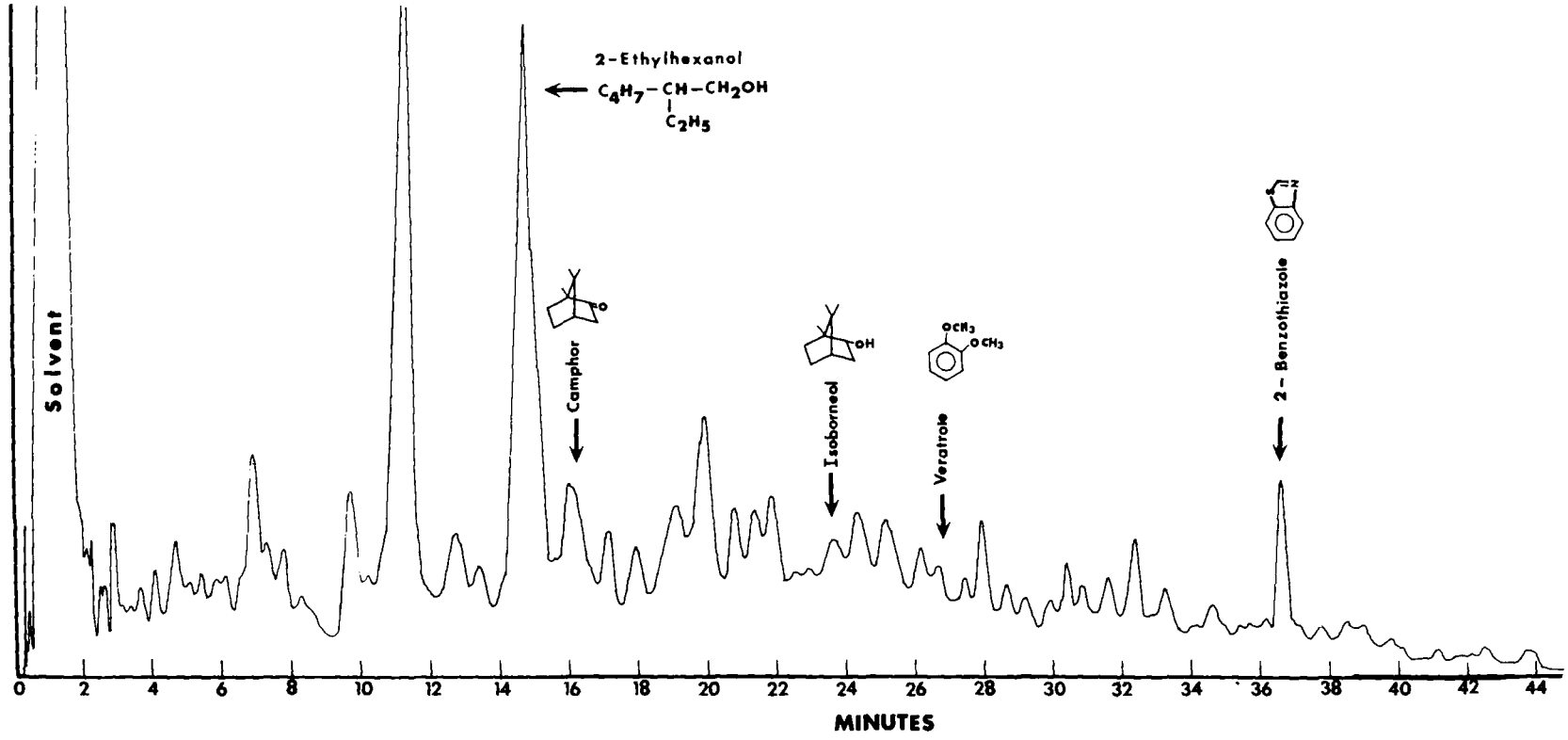

Figure 13. Gas Chromatogram of New Orleans Drinking Water Extract

produced chromatograms containing superimposed peaks.

- On two different 50-ft. SCOT columns (Carbowax 20M and SE-30) retention times of two of these terpenes matched retention times of extract peaks.

However, comparison of mass spectra showed that none of these three terpenes was present in the finished water extract.

ORGANIC COMPONENTS OF LABORATORY WASTE WATER

As part of an investigation of the content of the SERL's waste effluent, the NWCCRP analyzed laboratory sewage samples for organic content. The chloroform extract of a 1500-ml sample of filtered raw sewage was concentrated and analyzed by GC-MS. One laboratory sewage sample contained 2-ethyl-1-hexanol as a major component and phenol as a minor component. A sample taken on a different day contained isopentyl alcohol and a trace of 2-ethyl-1-hexanol. Mass spectra also indicated the presence of several long-chain hydrocarbons, but specific compounds were not identified. About a dozen other peaks were observed in each sample but were not identified either by the computerized mass spectral library or by manual inspection.

In addition to the sewage samples and a tap water blank, a sample of tap water spiked with several common laboratory solvents and chemicals (1 mg/ℓ) was analyzed. Solvents, such as carbon tetrachloride, hexane, benzene, acetone and isooctane, that are known to be discharged into the laboratory waste lines were not detected in the spiked water. Less volatile materials, such as chlorobenzene, *para*-dichlorobenzene (a common toilet bowl odor masking agent), 2-chloroaniline and *meta*-cresol, were easily detected at this level.

Organic contaminant data were reported to the Region IV Surveillance and Analysis Division for inclusion in a summary of the SERL's waste effluent content.

POLYCHLORINATED BIPHENYLS IN POULTRY FAT

The U. S. Department of Agriculture requested analyses of poultry fat for contamination by polychlorinated biphenyls (PCB's). Fish meal that is used for poultry feed was contaminated when PCB's used as heat exchange material leaked from a corroded coil in the fish meal processing plant. The leakage occurred over a relatively long period of time and was not noticed until a poultry grower noted a reduction from 90% to 2% in the hatch rate of his eggs after his breeder fowl were fed contaminated fish meal. The Department of Agriculture estimated that 60 million animals (fowl and pigs) had been exposed to contaminated feed and obtained 1400 samples from poultry growers and processing plants in the southeastern United States. The NWCCRP was requested to assist with analyses to determine if a 5 μg/g permissible limit of PCB content in the fat had been exceeded.

Using a gas chromatographic method,[7] SERL personnel analyzed 85 samples in two weeks in August and September 1971. Results were reported directly to the U. S. Department of Agriculture for compilation with analyses from other laboratories.

To assure comparable results, all participating labs used the same method. Each sample was dissolved in petroleum ether and extracted with acetonitrile. Each acetonitrile extract was diluted with water to partition PCB's into petroleum ether. After each extract was purified by Florisil column chromatography, gas chromatography was used to quantitatively determine the extract's PCB content.

In September 1972, the U. S. Department of Agriculture issued a press release stating results of the testing in the Southeast. High levels of PCB's reported by SERL and other cooperating laboratories caused condemnation and destruction of 165,000 hens and broilers, 250,000 lb of turkey product, and 75,000 lb of chicken product. Other poultry was judged acceptable only for salvage; it could be used only after fat and bone were removed. However, in many cases processing costs made the salvaged product so expensive that no buyers could be found, and little of the

salvaged poultry was actually used. No figures were available to show the total poultry loss due to PCB contamination.

TITANIUM IN PAINT MANUFACTURING PLANT EFFLUENT

Titanium in a Florida paint manufacturing plant effluent was quantitatively measured by polarography. This analysis was requested by the EPA's Region IV Surveillance and Analysis Division and was part of a survey of 36 industrial wastes. Since titanium salts, primarily the oxides, are used as paint pigments, titanium was suspected to be discharged in the plant effluent.

Each day the paint plant discharged 630 gallons of effluent into a septic tank from which pollutants could enter the ground water via a drainfield. The murky, liquid sample that was taken from the septic tank contained a large proportion of dense, white sediment. Specimens of the well-mixed sample were ashed, fused with potassium bisulfate, and leached with 0.5 M sulfuric acid. Titanium was determined by DC polarography at the dropping mercury electrode in 1 M phosphoric acid supporting electrolyte. The total sample contained 1439 mg/ℓ titanium calculated as titanium oxide (2350 mg/ℓ). With a water discharge rate of 630 gal/day, the plant was discharging titanium at a rate of 7.5 lb/day. The plant effluent also contained significant amounts of mercury (3.9 mg/ℓ, determined by another EPA laboratory).

These analyses were included in the "Industrial Waste Survey of Dade County, Florida,"[8] and in information presented at an enforcement conference concerning the pollution of navigable waters of Dade County, Florida, in November 1971. After being informed of the results of the survey, plant officials agreed to recycle waste waters to recover the metal salts to eliminate the use of mercury as a fungicide in their paint.

ZINC IN METAL FINISHING PLANT EFFLUENT

Polarographic determination of the zinc content of a metal finishing plant effluent helped resolve

analytical discrepancies between the plant's laboratory methods and Tennessee State Pollution Control methods. The plant's polarographic analyses indicated that little or no zinc was discharged in the effluent. Tennessee state officials requested the EPA's Region IV Surveillance and Analysis Division to determine the effluent's zinc content. Analysis by atomic absorption spectrophotometry showed zinc content of 0.6 mg/ℓ. To check this analysis, the NWCCRP was requested to determine zinc content by polarography, since this method was used by the plant's laboratory.

In six samples, polarographic data showed that zinc concentration ranged from 0.002 to 0.2 mg/ℓ, significantly lower than atomic absorption data. However, after the samples were wet-ashed with HNO_3-$HClO_4$, polarographic analysis showed zinc content of 0.54 mg/ℓ (standard deviation of 0.03 mg/ℓ), a value comparable to that obtained with atomic absorption. The EPA chemists concluded that zinc in the effluent existed as organic complexes, which could not be determined polarographically without special analytical procedures. The plant revised its laboratory methods to produce accurate values for evaluation of its water treatment system.

TRANSITION ELEMENTS IN CLAY MINERALS

Transition element qualitative analysis aided the SERL's Agricultural and Industrial Pollution Control Research Program scientists to interpret electronic absorption spectra of clay minerals. These spectra were being used to elucidate structural, physical, and chemical properties of clay minerals.

In the ultraviolet-visible range (195-800 nm), clay mineral spectra contain absorption bands that are due to the presence of transition metal cations. Observed adsorption bands had been assigned to iron-associated electronic transitions. Since other transition metals interfere with iron-induced bands, the NWCCRP was requested to analyze samples of two clay minerals, Wyoming bentonite and nontronite, to determine if transition elements other than iron were present. Spark source mass spectrometric analysis showed impurities of nickel, chromium, titanium and zinc.

CADMIUM AND NICKEL IN BATTERY MANUFACTURING PLANT EFFLUENT

For 12 years a battery manufacturing plant had dumped its untreated effluent into New York's Foundry Cove, whose waters flow into the Hudson River. After receiving the EPA's notification to discontinue discharge, the company began treating its waste water but resisted the EPA's demands to dredge Foundry Cove to remove sediments containing cadmium and nickel. In a legal hearing the company argued that any inorganic materials left in sediments were highly insoluble in water and would not affect biota. Although no obvious damage had been done to flora and fauna, the EPA contended that many cadmium and nickel salts are soluble in water and that even insoluble compounds could be resuspended by water agitation or could degrade and enter the aquatic system. The judge ruled that the EPA had insufficient evidence to force dredging.

The EPA Region II Enforcement Division sought proof that cadmium and nickel were present in dangerous quantities in the water and sediment. Analyses performed by the EPA's Edison Water Quality Laboratory, Edison, New Jersey, showed the presence of cadmium residues in fish tissue and aquatic plants from the area. Atomic absorption analyses showed that water and sediment samples contained unacceptable amounts of cadmium. To have irrefutable evidence for the appeal hearing, the Edison laboratory requested confirmatory analyses by the NWCCRP.

The cadmium content of two water samples was determined by four unrelated analytical techniques: neutron activation, spark source mass spectrometry, polarography, and atomic absorption spectrophotometry (Table 4). Atomic absorption data agreed well with those reported by the Edison laboratory. Neutron activation and spark source mass spectrometry confirmed atomic absorption analyses for sample #20039 but produced different values for sample #20046. These differences and the polarographic data suggested that interfering substances might be present in sample #20046 or that sample preparation techniques might require revision.

Table 4. CADMIUM CONTENT OF FOUNDRY COVE WATER AND SEDIMENT SAMPLES

	Water Samples		Sediment Samples	
Sample No.	20039	20046	15004	15043
Atomic Absorption				
Edison Lab.	230 μg/ℓ	23 μg/ℓ	0.8 mg/kg	17000 mg/kg
NWCCRP	180 μg/ℓ	20 μg/ℓ	--	--
Polarography				
acid-digested	22 μg/ℓ	1 μg/ℓ	--	--
fine-filtered	15 μg/ℓ	not detect.*	--	--
Neutron Activation	180 μg/ℓ	67 μg/ℓ	<10 mg/kg	2000 mg/kg
Spark Source Mass Spectrometry	180 μg/ℓ	5 μg/ℓ	0.06 mg/kg	1000 mg/kg

*detection limit: 1 μg/ℓ

Two sediment samples that had been analyzed by atomic absorption spectrophotometry at the Edison laboratory were also analyzed for nickel and cadmium content by the NWCCRP with spark source mass spectrometry. Atomic absorption nickel were confirmed (Table 5), but lower cadmium concentrations (Table 4) were found with spark source than with atomic absorption.

Table 5

Nickel Content of Foundry Cove Sediment Samples

Sample	15004	15043
Atomic Absorption (Edison Lab.)	100 mg/kg	8800 mg/kg
Spark Source Mass Spectrometry	140 mg/kg	4000 mg/kg

Even the lower values indicated that cadmium and nickel concentrations in sediment and water samples were unacceptable. In humans, heavy metals concentrate in the liver, kidneys, and pancreas and are not normally excreted after ingestion. Quantitative data on cadmium toxicity indicate that the lethal concentration for fish varies from 0.01 to 10 mg/ℓ, depending on fish species, water type, temperature, and exposure time.[9] Much less toxicity data exists for nickel than for cadmium, but nickel toxicity to man is believed to be very low.[10]

The data were presented at an appeal hearing, which produced a court order to force the manufacturer to dredge certain areas of Foundry Cove. Dredged sediment was piled on the ground, and cadmium and nickel were extracted. Dredging was completed in September 1972.

ARSENIC IN GOLD MINE TAILINGS AND WASTE WATER

Since the early 1900's, mine tailings and waste waters from North America's largest gold mine, located in South Dakota, were discharged into Whitewood Creek. After flowing through the Belle Fourche and Cheyenne

River, water from Whitewood Creek eventually reaches the Oahe Reservoir about 150 miles away (Figure 14).

Because of the type of ore being processed, the EPA's Denver National Field Investigation Center suspected that large amounts of arsenic were being released into these waters. However, they did not have adequate facilities for arsenic analyses and requested neutron activation analyses by the NWCCRP. One sample of pulverized mine ore and four well water samples were included with 21 water samples and 13 sediment samples collected from various locations on Whitewood Creek, Belle Fourche River, Cheyenne River, Oahe Reservoir, and various small tributaries.

Water samples that contained suspended matter were filtered to allow analyses of water and residue separately. Each water sample and a standard arsenic solution were irradiated in a 1-Mw nuclear reactor with a thermal neutron flux of about 1×10^{13} neutrons/sec/cm^2 for 30 min. When ^{24}Na background radiation had decreased to tolerable levels (usually 1-3 days), the level of 559-keV gamma photon radiation from each sample and its corresponding standard was measured with a Ge(Li) detector. From these measurements arsenic concentrations were calculated (Table 6). The arsenic content of six water samples was confirmed by spark source mass spectrometry (Table 6).

Solids filtered from water samples were left at room temperature on the filter papers until dry. Aliquots of sediment samples with uniform texture were analyzed without treatment. Two sediment samples with varying particle size (dust to rocks) were sieved, and only particles with diameter less than 0.026 in. were irradiated.

Subsequent analytical procedures were identical for water residues and sediment samples. Each weighed solid sample and its corresponding standard solution were irradiated and counted under the same conditions as used for water samples. Each of the solid samples and liquid standards was placed at the detector center to minimize counting errors caused by sample geometry.

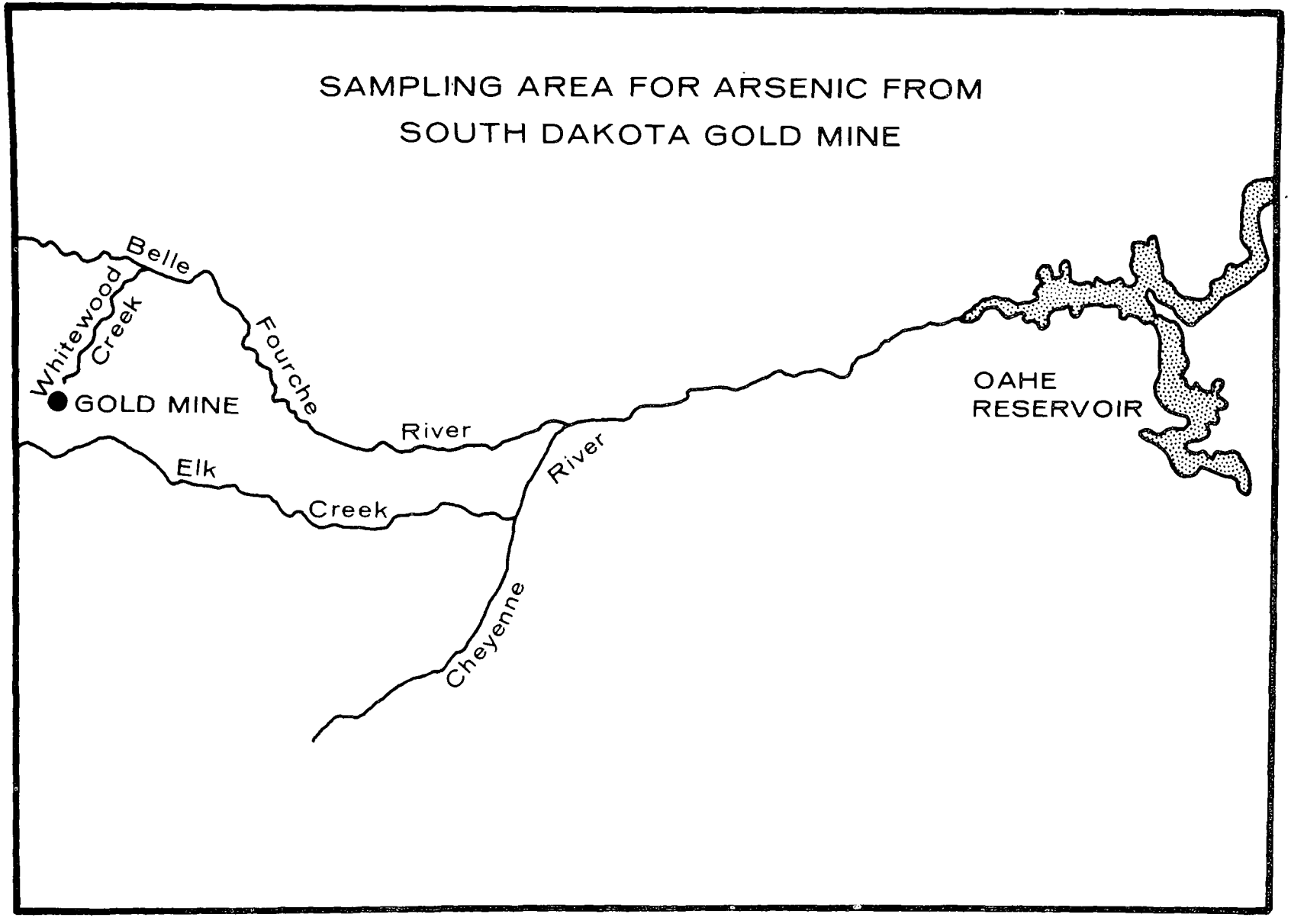

Figure 14. Map of Sampling Area for Arsenic from Gold Mine Tailings and Waste Waters

Table 6. ARSENIC CONTENT OF WATER SAMPLES

Sample Number	Sampling Site	Liquid Portion μg As/ℓ[a]				Solid Portion mg As/g		
			NAA		SSMS	NAA		SSMS
1425	Gold Run below Sand Dam	138	143			no residue		
1435	Gold Run below Sand Dam	420	340	321		2.72	2.44	
1424	Slime Plant	27	ND	28		2.93	2.88	
1431	Slime Plant	910	952			--	--	
1419	Whitewood Creek	300	230	230		no residue		
1433	Whitewood Creek	1510	1420			2.38	2.52	
1436	Whitewood Creek	1900	1270		900	2.49	2.45	3.00
1437	Whitewood Creek	970	880			3.67	3.90	
1438	Whitewood Creek	13	12			no residue		
1422	Elk Creek	ND	ND		ND	0.01	0.01	0.01
1429	Box Elder Creek	7	ND	ND		--	--	
1423	Horse Creek	ND	ND			0.01	0.01	
1421	Bear Beetle Creek	ND	ND			--	--	

Table 6 (continued). ARSENIC CONTENT OF WATER SAMPLES

Sample Number	Sampling Site	Liquid Portion µg As/ℓ[a]				Solid Portion mg As/g		
		NAA			SSMS	NAA		SSMS
1418	Spearfish Creek	5	6		ND	no residue		
1420	Deadwood Creek	8	12			no residue		
1432	Deadwood Creek	ND	ND		ND	no residue		
1427	Belle Fourche River	500	425	450		0.14	0.12	
1428	Belle Fourche River	ND	ND		0.1	0.01	0.01	0.08
1434	Belle Fourche River	560	571			0.13	0.12	
1426	Cheyenne River	210	190		100.0	0.07	0.06	0.02
1430	Cheyenne River	ND	ND			0.12	0.10	

[a]ND - not detected

Results of arsenic analyses of the filtered solids are shown in Table 6, and sediment arsenic analyses in Table 7.

The four well water samples collected in the vicinity of Whitewood Creek were also analyzed for arsenic by neutron activation, but arsenic was not present above the detection limit of 1 mg/ℓ.

Data from these arsenic analyses and the previous mercury analyses were presented at a legal hearing in late 1971, at which the mining company was ordered to improve waste treatment. The mine is building a waste water treatment plant.

Table 7. ARSENIC CONTENT OF SEDIMENT SAMPLES BY NEUTRON ACTIVATION

Sample Number	Sampling Site	mg As/g sediment		
1602	Homestake Mill Feed (pulverized ore)	3.65	3.32	4.01
1610[a]	Whitewood Creek, Gold Run	0.82	0.69	0.83
1617	Whitewood Creek at Rodeo Car	2.20	2.07	2.11
1611	Whitewood Creek near Bell Fourche River	2.93	3.27	4.26
1608	Whitewood Creek at mouth of Belle Fourche River	4.35	4.02	3.73
1621	Whitewood Creek at mouth of Belle Fourche River	1.19	1.23	1.29
1609[a]	Deadwood Creek at mouth of Whitewood Creek	0.62	0.79	0.67
1613	Elk Creek	0.02	0.02	0.02
1618	Box Elder Creek	0.02	0.02	0.02
1619	Bear Beetle	0.02	0.02	0.02
1614	Belle Fourche River	3.99	3.60	3.98
1616	Belle Fourche River	2.36	2.23	2.88
1612	Cheyenne River	0.01	0.01	0.01
1615	Cheyenne River	0.70	0.71	0.73

[a]Sample particle size varied from dust to rocks. Only particles with diameter of less than 0.026 in. were irradiated.

SECTION V

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SECTION VI

GLOSSARY OF ABBREVIATIONS

EPA - Environmental Protection Agency

FID - flame ionization detector for gas chromatograph

GC - gas chromatography

GC-MS - combined gas chromatography and mass spectrometry

IR - infrared spectroscopy

LMRFF - Lower Mississippi River Field Facility of the Environmental Protection Agency

MS - mass spectrometry

ND - not detected

NWCCRP - National Water Contaminants Characterization Research Program at the Southeast Environmental Research Laboratory

PCB - polychlorinated biphenyls

RGC - computer reconstructed gas chromatogram

SCOT - support coated open tubular columns for gas chromatography

SERL - Southeast Environmental Research Laboratory

TLC - thin-layer chromatography

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16. Abstract

Identification and measurement of aquatic pollutants are discussed under 13 project categories. In most cases these analyses helped to solve, or at least to understand more clearly, the related pollution incident and in some cases provided evidence for enforcement of regulatory legislation.

17a. Descriptors *Water pollution sources, *pollutant identification, *analytical techniques, industrial wastes, mine wastes, gas chromatography, mass spectrometry, polarographic analysis, neutron activation analysis, metals, polychlorinated biphenyls

17b. Identifiers atomic absorption spectrophotometry, infrared spectroscopy, ultraviolet spectroscopy, organoleptic compounds

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