1986

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Recommended Citation

deFur, P. O., Bender, M. E., Su, C. W., & et al. (1986) Polynuclear hydrocarbons in sediments and clams in the vicinity of a refinery outfall. Virginia Institute of Marine Science, William & Mary. https://scholarworks.wm.edu/reports/2679

Polynuclear Hydrocarbons in Sediments and Clams in the Vicinity of a Refinery Outfall

by

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September 1986

INTRODUCTION

The objectives of this study were:

1) to determine whether substances present in refinery process water are found in adjacent sediments and bivalves

2) to determine the concentrations of compounds detected

3) to identify compounds that may have adverse effects on marine animals or their human consumers.

Sediments were selected as the primary target for analysis since they often contain concentrations of polynuclear aromatic hydrocarbons (PAHs), pesticides, and metals several orders of magnitude higher than those of the overlying water (Neff, 1980 and Armstrong et al., 1977). In the case of PAHs, Neff estimated that sediments will always contain concentrations greater by a factor of 1,000 than the overlying water (Neff, 1980). He concluded that, "sediment samples have a substantial integrating effect on the temporal patterns of PAH input and offer good geographical resolution".

A survey of organic compounds detected in Chesapeake Bay sediments indicated an influx of PAHs from the major tributaries to the Bay (Bieri et al., 1981). These authors extended their surveys of sediments into two highly industrialized sub-estuaries of the Bay, the Elizabeth and Patapsco rivers. They found concentration distributions which, in some cases, could be interpreted by the movement of pollutants from their sources. Within the dredged channel of the Patapsco River, for example, the concentration of total aromatic hydrocarbons in surface sediments ranged from several hundred ppm in the Baltimore Harbor area to about 20 ppm at the mouth. In the Elizabeth River the concentration maximum of 440 ppm was in the sample furthest upstream. A general decrease in concentrations towards the mouth of the river indicated export of pollutants from the Elizabeth River.

The ability of bivalves to bioaccumulate hydrocarbons allows them to be used to: (1) monitor changes after a spill; (2) monitor levels of hydrocarbons in estuaries with chronic inputs; (3) establish baseline levels of hydrocarbons; and (4) determine areas of impact from effluents. These molluscs, which filter large volumes of water while feeding, can accumulate petroleum hydrocarbons from solution and/or suspension (Anderson, 1975; Boehm and Quinn, 1976; Neff, 1980). Depuration of accumulated hydrocarbons is dependent upon several factors including length of exposure; the existence of metabolic pathways for excretion; the physiological state of the animal (lipid content) and environmental factors, e.g., salinity and temperature. Chronically exposed animals appear to depurate much more slowly than those from short term laboratory experiments. Boehm and Quinn (1977) showed that the clam Mercenaria mercenaria lost only 30% of accumulated hydrocarbons in 120 days after transfer from Narragansett Bay to clean sea water. Laboratory exposures have shown much shorter half-lives ranging between 1 and 10 days (Lee al al., 1972 and Jackim and Wilson, 1977). Stegeman and Teal (1973) showed that the lipid content of oysters influenced hydrocarbon uptake and Fosato et al (1979) found that maxima for benzo(a)pyrene and perylene were influenced by lipid content and the spawning cycle in Mytilus edulis.

Fucik et al. (1977) showed good correlation between the rates of naphthalene uptake by the clam Rangia cuneata and naphthalene levels in the sediments. In oiled areas burrowing bivalves; e.g., Mya arenaria or Modiolus demissus, have been found with higher concentrations than epibenthic bivalves; e.g., Mytilus edulis or Crassostrea virginica (Augenfeld et al., 1982; Lee et al., 1981; Vandermeulen and Gordon, 1976).

In addition to the study of organic contaminants, the environmental conditions in the lower York River were determined through a review of the existing data on the chemical, physical and biological characteristics of the estuary. A brief description of the estuary is given below. The complete summary can be found in Bender (1986).

The York River is formed by the confluence of the Mattaponi and Pamunkey rivers at West Point, Virginia, approximately 50 km upstream from the refinery. The entire length of the York is tidal with tides extending well up into the freshwater regions of the Mattaponi and Pamunkey rivers.

Salinity stratification in the lower estuary varies with tidal phase and is extremely important in determining the chemical characteristics of the water column. Stratification of the water column in the summer months causes oxygen depletion in the deeper waters of the river and nutrients are regenerated during these periods. The low oxygen tensions in the deeper portions of the river limit fish and crab populations in these areas and may cause mortalities of benthic fauna.

A bay wide decline in submerged aquatic vegetation has been observed over the last 15 years and communities in the York have decreased in a similar manner.

Benthic animal populations are dominated by polychaete worms. The type of substrate present plays and important role in determining community structure. Benthic populations near the refinery outfall have been studied and slight, although statistically significant, depressions in diversity were found in 1976.

Oyster populations in the lower river are limited by disease and predators. Commercial harvests come mainly from private grounds located upstream where average salinities are less than 15 $^{o}/oo$.

The lower river supports a hard clam fishery of 10-20 patent tong boats. Landings reported for the river average about 200,000 pounds/year.

Fish populations of the York are composed of resident, anadromous and catadromous species. As in the Bay proper, populations of many important species have shown dramatic fluctuations in abundance. The causes of these population fluctuations are known for only a few species.

Finfish are harvested commercially in the York by pound nets, fixed and drift gill nets, fish traps and by haul seining. Major species include; bluefish, grey trout, croaker, spot, flounder, eels, striped bass and American shad. Large fluctuations in landings for individual species occur with time, with trends in harvest from the York following those observed from the state as a whole.

The estimated dockside value of commercial fishery landings from the York was 1.6 million dollars in both 1980 and 1981. Finfish landings account for about one-third of the total value, blue crabs are usually the most economically important species followed by oysters and clams.

To determine the spatial variability of organic compounds in sediments near the refinery outfall, a sampling grid measuring 4 by 1.5 kilometers was established around the outfall. Sediment samples were collected at 500 meter intervals along the grid (36 stations). In order to estimate temporal variability, samples were collected at the same stations during March and December of 1983.

The hard clam, Mercenaria mercenaria, is abundant and is harvested both commercially and recreationally in the lower York River. It is the only shellfish found in the vicinity of the refinery that is consumed by humans. Sampling sites for resident clams were determined by the results of the initial sediment sampling program. Clams were collected from 4 locations, over a range of sediment PAH concentrations.

As mentioned previously, several investigators have found that the physiological condition of bivalves, e.g., as determined by their spawning cycles, influences hydrocarbon uptake and retention. To provide preliminary data on these cycles in the York River, we determined PAH residues in clams during periods near expected maxima and minima; e.g., just prior to spawning and after spawning has occurred, respectively. To minimize the impact of individual variability due to factors such as sex, age, etc., composite samples of five individuals were utilized and four composites were analyzed from each station.

To characterize refinery process water, two 24-hour composite samples were collected several months apart.

METHODS

Sediment Collection

Sediment samples were collected on March 21, 1983 and December 2, 1983 from the VIMS vessel R/V Captain John Smith. The sampling grid is shown in Figure 1. The distance between stations on any transect is 500 meters. Navigation was done by LORAN C, with the first station established close to the Eastern end of the refinery pier in order to obtain accurate offsets for conversion of LORAN to latitude and longitude. The exact locations of the stations are shown in Table 1 along with their depths. Depth contours are shown on Figure 1. The LORAN C navigation system enabled reoccupation of stations to within approximately 100 ft. on the second sampling. Sediment samples were taken with a 0.1 m^2 stainless steel Smith-MacIntyre grab manufactured at the University of Rhode Island. Before each deployment, the grab was washed with river water pumped from an intake 1.5 meters below the surface level and then rinsed thoroughly with methanol. The grab is equipped with stainless steel doors covering the top to maintain sample integrity while it is being retrieved. Methanol rinsed stainless steel scoops were used to transfer the top 3 cm. of sediment into precleaned glass jars, which were refrigerated on board and frozen immediately on returning to the laboratory.

Clam Collection

Because of the difficulty of collecting clams in the vicinity of the refinery, a commercial clammer was employed to sample them on April 27, 1983 and December 12, 1983. The areas sampled are indicated by circles on Figure 1 because the vessel had to drift several hundred feet while sampling.

Locations of the centers of the areas are given in Table 2 and Figure 1. Clams were sealed in plastic bags and refrigerated until they were returned to the laboratory and frozen. All samples remained frozen until analysis.

Effluent Collection

Effluent water samples were collected from the refinery process stream at AMOCO site 101 which is immediately down stream from the biological settling ponds and before mixing with cooling water prior to discharge. Twenty-four hour composite samples were taken on August 8-9, 1983 and April 15-16, 1984 by collecting 500 ml of water each hour and combining the volumes in a pre-cleaned glass carboy. At the time of the second sampling, a sample of the influent water before use was taken as well as a sample of York River water from the VIMS pier. All water samples were extracted as soon as possible.

Analysis

Sediment samples from the first sampling, and all clam samples were freeze dried, ground in a mortar and pestle to crush and homogenize them and then stored in a freezer until extraction. The second set of sediment samples was dried with a 9:1 mixture of sodium sulfate and precipitated silica by mixing wet sediment with pre-extracted sodium sulfate + silica mixture and refreezing. This mixture was then extracted in the same manner as the freeze dried material. All samples were spiked with an internal standard before soxhlet extraction for 24 hours with methylene chloride.

Water samples were adjusted to pH 12 with 4N NaOH and then extracted with three separate 100 ml portions of methylene chloride to yield a base/neutral fraction. These extracts were reduced in volume and treated the same as sediment and clam extracts. The water was then adjusted to pH 2 with 4N HCl and extracted with three more 100 ml portions of methylene chloride to give an acid extractable fraction. This fraction was reduced in volume and analyzed by gas chromatography without further treatment. An aliquot of the effluent was analyzed for low molecular weight material by a purge and trap technique (Voznakova et al., 1978). Helium gas was used to strip volatile components which were trapped on a cartridge containing adsorption resin. The trapped compounds were thermally desorbed and analyzed by gas chromatography.

The high concentrations of biogenic compounds in environmental samples necessitates a "clean-up" step to remove as many interferences as possible. The extracts were reduced in volume with a rotary evaporator and "cleaned" by gel permeation chromatography on a styrene/divinyl benzene copolymer size exclusion resin using methylene chloride as the elution solvent. Most biogenic molecules, which are generally larger than simple hydrocarbons, were unretained by the resin and eluted before the molecules of interest. (Bieri et al., 1981). Thus, two fractions named G1+G2 and G3 were collected. Aromatic hydrocarbons and many polar anthropogenic substances eluted in the G3 fraction which was then separated into six subfractions (G3.1 through G3.6) of increasing polarity using high pressure liquid chromatography (HPLC). HPLC fractionation was carried out on a semi-preparative cyano-amino normal phase column. The first, non-polar, subfraction was eluted with hexane, after which methylene chloride was programmed into the solvent mixture. Twenty five percent methylene chloride in hexane was used to elute the aromatic fraction and more polar fractions were eluted with 100% methylene chloride, acetonitrile and methanol sequentially. Compound classes eluted in each fraction are given below:

G3.1	aliphatic
G3.2	polynuclear aromatic hydrocarbons (PAHs) polychlorinated biphenyls (PCBs) DDT DDD DDE mononitro-PAHs
G3.3	cyano-PAHs ketones amines indole carbazoles azaarenes with blocked nitrogen atoms
G3.4	hydroxy-PAHs
G3.5	azaarenes and aldehydes
G3.6	organic acids

Preliminary examination of several samples from the first sampling showed few compounds in the G3.3 and G3.4 fractions enabling these two fractions to be combined and analyzed as G3.3+4. Further, there were no identifiable compounds in the G3.5 fraction, and the G3.6 fraction was overwhelmed by straight chain fatty acids typical of natural biological activity (Wakeham et al., 1983). These two fractions together with the G3.1 fraction which contains no compounds of interest were not analyzed, but were archived in the event that their future study is desired.

Gas Chromatography

The two fractions of major interest, the G3.2 and G3.3+4 were analyzed by capillary column gas chromatography using flame ionization detection. A Varian 3700 gas chromatograph temperature programmed from 75° C to 300° C at 6°/min was used for all analyses. Persilated glass capillary columns coated with 0.2μ of phenylmethyl silicone stationary phase were prepared in this laboratory according to the method of Grob (Grob, 1982). Columns were approximately 25m x 0.32mm id and used Helium carrier gas at a linear flow of 27 cm/sec. Data was collected and stored on a Hewlett Packard 3354B laboratory data system. Peak identification on the G3.2 fraction was done using the aromatic retention index system of Bieri (Bieri et al., 1981). Selected marker peaks from each chromatogram were identified by visual comparison with standard runs made the same day. Using these markers, computer programs written in this laboratory used the stored data to assign each peak an aromatic retention index (ARI). The ARI is calculated by the formula:

$$ARI_x = \frac{T_x - T_{mp}}{T_{mf} - T_{mp}} \times 100 + ARI_{mp}$$

T_x = retention time of peak x

T_{mp} = retention time of the last marker preceeding peak x

T_{mf} = retention time of the next marker following peak x

ARI_{mp} = ARI defined for the last marker preceeding x (ARI of the markers are defined as 000, 100, 200, 300, 400, 500, 600)

Using the calculated ARI, computer programs then identified peaks from an ARI library generated from previously injected standards and mass spectral identifications. Quantitation of these chromatograms was carried out using the internal standard added prior to extraction. This method corrects for extraction efficiency variations and losses of material during the analytical procedure.

Selected samples were analyzed by gas chromatography-mass spectrometry using a Varian 2700 GC interfaced to a DuPont 21-492B magnetic sector mass spectrometer. Ionization was by electron impact at 70 eV energy and a scan was taken every 2.3 seconds. A reverse search computer program utilizing ARI's was used to aid in aromatic compound identification (Hein, 1981). The mass spectral data for the G3.2 fractions was used largely to confirm identifications made using ARI's, but the G3.3,4 fraction could only be accurately characterized using mass spectral identifications.

Data Analysis

In sampling from a population it sometimes happens that an extraneous factor or factors will influence the magnitude of the parameter we are attempting to measure. In the case of sediments and their corresponding chemical burdens we know that differences between samples for factors such as percent volatile solids and grain size will cause variations in many of the target compounds we are attempting to measure. If enough data are available on the effect of an extraneous factor on the concentration of the variable of interest we can 'normalize' the data to account for differences between samples caused by the external variable.

Statistically we can often account for the effects of external variables on the parameter we are most interested in by pairing samples with like characteristics, this is frequently done on the basis of sex, age, etc. In the case of sediment samples the pairing can be done on the basis of sample location, given two sampling periods or on the basis of some other factor, e.g., grain size.

In our analysis of the chemical data from this study we have made extensive use of paired t-tests. These tests have been applied to: (1) replicate sample extractions; (2) different drying techniques; and, (3) different sampling times.

In addition, to reveal locations which might be influenced by the refinery outfall, we have plotted the concentrations of total resolved aromatic compounds, pyrogenic compounds and some individual compounds against percent volatile solids. On these graphs we have identified stations which appear to be outliers, i.e. not within the normal range for a constituent at a corresponding percent volatile solids level.

Prior to statistical analysis, the distribution of the variable being analyzed was tested for normality by plotting the cumulative frequency distribution on normal probability paper. When deviations from normality occurred logarthmic transformation of the data resulted in a normal distribution.

Replicate Extractions

In comparing the results between stations or sampling times it would be ideal to be able to extract each sample more than one time. However, due to the time and expense involved when performing relatively large numbers of analysis, this is not usually possible. In order to estimate the variability in extraction and subsequent analysis of samples, five samples were extracted in duplicate. The results of these extractions were compared with "paired t tests" which tested the variability between the concentrations of the eleven most abundant compounds in each pair. Similar tests were performed on the total resolved aromatics in each pair. The result of these tests are shown in Table 3. A provision was made in the tests for 'injection error' (the error expected on replicate injections of the same sample) of approximately 5%. As can be seen from the table, the replicate extractions were quite similar with the largest deviation being 25% and the mean difference between samples being much less, 11%. None of the paired extractions were shown to be statistically different.

Freeze Drying vs Chemical Desiccation

In an attempt to reduce loss of some of the lower molecular weight compounds during freeze drying it was decided to try desiccation of the sediment samples with Na_2SO_4 and precipitated silica, a technique utilized in pesticide analysis. Before adopting this procedure for use in the second grid sampling, we compared results obtained with this technique to the freeze dried samples from the first sampling period and made comparisons using samples with higher contamination levels from the Elizabeth River. Differences between samples were tested with 'paired t tests' for the eleven most abundant compounds and on the total resolved aromatics.

Samples were desiccated with a 9:1 mixture of Na_2SO_4 and silica, the amount of desiccant mixture utilized varied depending on the moisture content of the sample, normally a 1:1 mixture was used. The desiccant was mixed with the sediment sample and the mixture was then refrozen to facilitate drying. After freezing, the sample was triturated and extracted as previously described.

Results from the two methods of drying are shown in Table 4. For the five comparisons of the York River samples, none were shown to be statistically different ($\alpha = 0.05$).

Statistical comparisons of three Elizabeth River samples, for the eleven most abundant compounds did not show significant differences between the two drying techniques. However, when the chemically desiccated samples from the Elizabeth River were compared to those freeze dried, five lower molecular weight compounds (naphthalene, methyl naphthalene, C_2+C_3 naphthalenes and biphenyl) were shown to be higher in the chemically desiccated samples. Similar comparisons with the York River samples did not show significant differences between the two drying techniques. However, concentrations of these compounds in the samples tested were quite low, making meaningful comparisons difficult.

Since no significant differences were detected in comparisons of the two drying techniques with the York samples, it was concluded that the use of the chemical desiccation technique would not bias the results of the second survey and perhaps more accurate information on the concentrations lower molecular weight compounds could be achieved. We decided therefore to utilize the chemical desiccation technique in the second sediment survey.

RESULTS

Sediments

Two characteristics (percent volatile solids and percent solids) of the sediments sampled along the grid during the two surveys are shown in Table 5. In comparing the March and December sampling periods, most stations were quite similar, however, relatively large decreases in volatile solids were observed at stations 3, 5 and 29 while increases were noted at stations 16, 17, 24 and 35. These changes between sampling periods were reflected in corresponding decreases and increases in total resolved aromatic hydrocarbons.

Correlations between percent solids and percent volatile solids are shown in Figure 2 for the March sampling period while Figure 3 shows the relationship between percent organic carbon and percent solids. Similar relationships were observed between percent solids and percent volatile solids in the December samples. Organic carbon analysis were not conducted on the December samples, since the correlation with loss on ignition (volatile solids) gave such a good estimate of total organic matter in the March samples.

Levels of total resolved aromatic hydrocarbons and the 14 most abundant pyrogenic compounds found during the two surveys are tabulated in Table 6.

Three dimensional views of two sediment parameters along the sampling grid are shown in Figures 4 and 5 for percent solids and in Figures 6 and 7 for loss on ignition. The figures show that the shallower inshore stations had high levels of total solids and correspondingly reduced levels of volatile solids. Station 31 located inshore of the refinery pier is an exception to this general trend. Deeper offshore stations had lower levels of total solids and higher levels of volatile solids.

Figures 8 and 9 give three dimensional views of total resolved aromatic hydrocarbons along the sampling grid during the two surveys. While these figures appear to show that some stations have elevated levels, they do not consider the influence of volatile solids levels on the 'contaminants' being measured.

Regressions of total resolved aromatic hydrocarbons in the sediments samples against percent volatile solids are shown in Figures 10 and 11 for the March and December surveys. As can be seen from the figures, as percent volatile solids increase so do the concentrations of aromatic hydrocarbons, although considerable scatter is evident. On these figures we have noted stations which appear to deviate significantly from the general trend line. Identification of these outliers is somewhat subjective but more rigorous statistical treatment is precluded by the fact that a potential source is known. If we were sampling from a completely homogenous population, we could calculate a regression line by least squares utilizing all the data and identify the outliers with confidence intervals. However, when we suspect that some factor, e.g., an outfall or shipping activity, may be contributing to the variability, it is not statistically correct to include all the data points in a regression calculation.

Figures 12 and 13 show the location of the stations identified as outliers in the regressions of percent volatile solids and total aromatic hydrocarbons. On both sampling dates all stations identified as exceeding the expected levels of total aromatic hydrocarbons were located relatively near the refinery pier and outfall. Station 1 located near the downstream end of pier was much higher than the other stations in both surveys. Averaging the data from the two surveys produces the plot shown in Figure 14. Stations 1 and 31 deviate most significantly from the expected levels, with all other 'outlying' stations being located relatively near the pier in deeper water with sediments high in volatile solids.

We believe the method outlined above allows us to distinguish areas with unusual concentrations; however, the actual magnitude of the elevations observed appear to be quite small. Although few samples are available from the York to make comparisons, those collected by Voudrias (1981) in tributary streams where marinas were present had total aromatic hydrocarbons levels between 23 and 9.5 ppm. His control site, a tributary approximately 17 KM upstream from the refinery, had a concentration of 2.5 ppm. Sediments collected from the mouth of the York during the spring and fall of 1979 had levels of approximately 1 ppm (Bieri et al., 1981). Unkulvasapaul (1984) analyzed sediments from two stations in the upper York

River near West Point, where she found total aromatic hydrocarbon concentration of 3.6 and 2.7 ppm.

During March of 1984 we collected 2 samples in the middle reach of the York River and analyzed them for total aromatic hydrocarbons. The concentrations found were almost extactly those predicted by the regression line for the December refinery survey.

Temporal changes in sediment levels for most compounds were observed between the two surveys, concentrations decreasing at nearly all stations from March to December. Statistical comparisons between the sampling periods were made with paired 't' tests and some of these are shown in Table 7. Although decreases were observed for all compounds tested, statistically significant differences were shown for only the total resolved aromatic hydrocarbons.

The concentration of the 20 most abundant aromatic compounds with their ARIs and tentative identifications for the two sediment surveys can be found in Appendix II. Qualitatively the aromatic fractions were quite similar in composition between stations and sampling periods. Fluoranthene, benzofluoranthenes, pyrene and chrysene were usually ranked at the top, followed by perylene, benzo(a&b)fluorene, benzo(a&e)pyrene, phenanthrene, and benzo(ghi)perylene and C-2 (phenanthrene/anthracene).

Recently, Sporstol et al. (1983), proposed a method which may be used to distinguish between PAHs from petroleum and combustion sources in sediments. Selected series of aromatics (unsubstituted compounds and their C_1-C_3 alkyl homologs) are quantified and the ratios of their abundance calculated. Since petroleum contains a greater proportion of alkyl homologues for given series of aromatic compounds than combustion sources do, the finding of high ratios of the alkyl substituted compounds in sediments may be used to indicate a possible source of these compounds.

We calculated these ratios for the phenanthrene/anthracene series at the various stations along the sampling grid, where they were identified. At most stations the unsubstituted compounds ranked one, as would be expected if combustion processes were the major source. However, at 7 of 17 stations compared the C_1-alkyl homologue was almost as abundant, indicating at least some contribution from petroleum. These calculations suggest that the aromatic hydrocarbons identified in the sediments during the study were of mixed origin.

Clams

The levels of total resolved aromatic hydrocarbons found in clams during the two surveys are shown in Table 8. Concentrations at all stations were much lower during the December survey. In the April survey it appeared that station to station differences existed with the station located nearest the outfall, station 1, exhibiting the highest average concentration. However, an analysis of variance performed on the April survey data failed to reveal significant differences between stations. Even though composite samples were analyzed, considerable variation in the results within a station is evident from the data and this variability undoubtedly made detection of differences, if they exist, difficult. Increasing the number of analyses within stations to at least 10 would be required at this level of variation to detect between station differences.

An analysis of variance on the total data set to detect differences either between sampling periods and/or stations is shown in Table 9. As expected, no differences between stations were shown by this test, but a highly significant difference between sampling periods was shown.

We hypothosized in the proposal that the clams spawning cycle with its corresponding lipid, build up and release, might cause variation in aromatic hydrocarbon residues. The data collected certainly seem to indicate that season plays an important role in determining the levels of aromatic residues in clams from the lower York River. Lowered metabolic rates of clams during the winter could also contribute to the lower residues observed by slowing the rate of uptake from the water and/or suspended solids.

As discussed in the next section, the compounds found in clams at highest concentrations were of relatively low molecular weight (see Appendix III and mass spectra section). Although these compounds were also found in the sediment samples, their relative abundance in clams was much higher than in the sediments. The lower molecular weight aromatic compounds are more water soluble than those of higher molecular weight (MacKay and Shiu, 1977; May and Wasik, 1972); however, bioconcentration by animals from solution usually increases linearly with decreasing water solubility (Chiou et al., 1977 and Yang & Sun, 1977). The differential partitioning of compounds between sediment and water may account for these observations. If the higher molecular weight compounds desorb less from sediments than those of lower molecular weight they might not be available for uptake if the uptake is primarily from solution. In addition, a relatively constant source of the lower molecular weight aromatics, e.,g., from the effluent, might account for the uptake patterns observed. Although only two composite samples of the effluent were analyzed, the first sample showed an abundance of the lower molecular weight compounds.

MASS SPECTRA ANALYSIS

Sediment

Compounds identified by mass spectral analysis of the G3.2 fractions are shown in Table 10. The identifications were made by comparison to previously published spectra, or comparison to spectra of authentic standards run in this laboratory as well as by ARI data. Correlations of these spectra with ARI data generated in this laboratory allow a great many of those compounds to be identified by the ARI computer program used with the GC/FID data. Many compounds identified in these extracts have been reported in aromatic fractions of sediment extracts from the Chesapeake Bay and adjoining waters (Bieri et al., 1981; Bieri et al., 1982; Smith et al., 1979). Moreover, these compounds are ubiquitous to many widely separated aquatic sediments (Wakeham et al., 1980a; Laflamme and Hites 1978).

Sources for these hydrocarbons are difficult to assess since most aromatic hydrocarbons have several potential sources. In general, the major sources are combustion and petroleum input. Many compounds (substituted and unsubstituted) found in these sediments have been reported in both crude oils and in combustion products from a variety of fuels (Ramdahl 1983a; Grimmer et al., 1983; Yu and Hites 1981). Therefore, the presence of the compounds identified by mass spectral analysis does not give a positive indication of their origin. There are also low levels of some compounds, notably retene and the tetramethyloctahydrochrysenes, that are considered to be of natural origin (Wakeham et al., 1980b). The tetramethyloctahydrochrysenes have been found in sediment cores at depths preceeding anthropogenic inputs and are believed to derive from the amyrin family of plant products. Retene is believed to be a degradation product of abietic acid, a predominant component of pine resin (Simoneit, 1977).

A typical chromatogram of the moderately polar G3.3+4 fraction is shown in Figure 15. The large group of peaks eluting between 30 and 40 minutes was determined to consist of sterols and sterones, biogenic compounds commonly found in the environment (Gagosian, et al., 1982). Other than these natural products, concentrations of compounds eluting in this fraction were quite low. Since the mass spectrometer system has a detection limit five times greater than the FID used for these chromatograms, identifications could only be made on peaks which were relatively concentrated. Identifications are given in Table 11 along with approximate concentrations. Concentrations are approximate because FID response factors for hetero-atom containing hydrocarbons may be significantly different from that of the binaphthyl used for quantitation. Of the compounds listed, 5,6,7,7-tetrahydro-4,4,7a-trimethyl-2(4H)benzofuranone is believed to be a natural product, although its origin is unknown. Seven of the compounds identified are organic ketones or diketones, with anthroquinone being in the most samples and in the highest concentration. These ketones have been identified from a variety of combustion related sources and also in air samples (Yu and Hites, 1981; Konig et al., 1983; Ramdahl, 1983b). They can be emitted directly from combustion or formed by partial oxidation of the parent PAH in the vapor phase, adsorbed on particles or in solution (Nikolaou et al., 1984). Some of these ketones have also been found in other sediments from the Chesapeake Bay area (Bieri et al., 1981; Bieri et al., 1982; unpublished work in this laboratory) indicating that they are not unique to sediments near the refinery. Since the concentrations of aromatic ketones from pyrogenic sources are on the same order as those of the parent PAHs emitted, it is unclear why the concentrations are so low in sediments, but it is likely that the oxygen functionality gives them a greater reactivity and results in faster degradation after deposition. More research in this area is needed.

Also present in these sediments are carbazole and several of its derivatives . Like the aromatic ketones, they have been found in other areas of the Chesapeake Bay (Bieri et al., 1982 and unpublished work in this laboratory). Carbazoles and other nitrogen heterocycles have been found in petroleums (Albert, 1978), coals (unpublished work in this laboratory) and coal tars (Burchill et al., 1983). A complex mixture of nitrogen containing heterocycles has been isolated from urban air (Dong and Locke, 1977), automobile exhaust and street dust (Wakeham, 1979). While these studies did not analyze for carbazoles specifically, it may be assumed that carbazole and its derivatives are also widespread in the environment with origins from many sources.

Clams

There were far fewer compounds identified in the aromatic fractions of the clam extracts than in those of the sediments. Results are presented in Table 12. One noticable point in these data is that primarily low molecular weight compounds are present. Most of the compounds identified were also identified in sediment extracts. Because most of the higher molecular weight substances found in the sediments were not found in the clams, it is possible that uptake by the clams occurs primarily from dissolved components and not from particulates or sediments. Hydrocarbons are relatively insoluble in water with the solubility decreasing rapidly with increasing molecular weight (Mackay and Shiu, 1977; May and Wasik, 1978). Thus, the lower molecular weight species with the higher solubilities would be expected to be more available in the dissolved state, producing the observed trend. One clam sample, 4B, contained many isomers of alkylated benzenes from C_3-benzene to C_{12}-benzene. These alkylbenzenes have been detected in other marine systems (Eganhouse et al., 1983) and are considered to be trace contaminants in alkyl sulfonate surfactants and detergents used domestically and industrially. The clams analyzed during the second sampling period had concentrations too low for mass spectral analysis.

The G3.3 + 4 fraction of the clams reflected the low number of compounds found in the G3.2 fractions. Compounds identified are listed in Table 13. Except for carbazole in samples 4B and 6A, there were no compounds found that were also in the sediment samples. There are two possible explanations for this observation. One is that the lack of ketones and higher carbazoles reflects their relatively low sedimentary concentrations. This is unlikely because the most abundant compound in the G3.3+4 sediment fractions, anthraquinone, was not detected in the clams while carbazole was. If the clams, indeed, do accumulate hydrocarbons primarily from a dissolved phase, the absence of the ketones may reflect a combination of low abundance and low solubility. Unfortunately, solubility data on those ketones is lacking. It is possible that the ketones found in the sediment are metabolized more rapidly than PAHs by the clams and thus do not accumulate in the tissue. Further study is needed to clarify this point.

Effluent

Qualitatively the two effluent samples collected were quite dissimilar. While the acid fraction of both consisted of relatively few resolved peaks and a large unresolved complex mixture (UCM), the base neutral fraction of the first sampling had a large number of low molecular weight resolved peaks and the second sampling had fewer resolved peaks and a larger unresolved concentration. Mass spectral identifications of the base/neutral (B/N) aromatic fractions are included in Table 10 along with the compounds identified from the sediment. No identifications could be made on the acid fraction. The B/N aromatic fraction of the first sampling is shown in Figure 16. The range of compounds is primarily low molecular weight with many isomers of substituted benzenes and naphthalenes identified by mass spectrometry. The volatile fraction can only be interpreted by coinjection of a standard of known composition. The sample and the sample plus standards are shown in Figures 17 and 18 with compounds identified. The range of compounds identified is similar to those reported for water soluble fractions of both crude and refined petroleums (Dimock et al., 1980 and McAuliffe, 1977). Solvent extraction of the second effluent sampling showed a different pattern for the same fraction (Figure 19). Besides the far lower number of resolved peaks and the higher UCM, the range of molecular weights is shifted higher. Because of this shift, volatile analysis was not undertaken on this sample. There are far fewer total compounds identified, and fewer substituted isomers. The ratio of substituted aromatics to the unsubstituted parent compound has been used to assess sources of aromatic compounds (Youngblood & Blumer, 1975; Sporstol et al., 1983). For the first sampling, the total of substituted naphthalenes are greater than naphthalene by a factor of 3.8 for monosubstituted, 6.8 for disubstituted and 4.4 for trisubstituted isomers, giving a clear indication of petroleum input. The low levels of individual peaks in the second sampling makes this measurement unreliable.

The G3.3, 4 fractions of the two sampling reflected these same trends. Compounds identified in these fractions are listed in Table 14. Besides the many substituted phenols in the first sampling, there are carbazole and many substituted carbazoles. Phenols are produced in the refining process (Jenkins et al., 1979) and were detected in the sediments (Table 11). The large number of substituted carbazoles is similar to that in petroleums (Albert, 1978). In contrast, carbazole and its derivatives are not detected in the second sampling, and there were few compounds detected in this fraction. The presence of ketones suggested oxidation, either combustion or chemical modification of parent compounds. The origin of the family of sulfones is unknown.

A summary of the total concentrations found in both samplings is given in table 15. The total concentration measured decreased by a factor of 3.5 in addition to the molecular weight range shift noted above. The proportion of the total organics contained in the unresolved mixture increased from 48% in the first sampling to 89% in the second sampling. Because the presence of an unresolved mixture is considered to be evidence of biodegradation of petroleum (Jones et al., 1983; Atlas et al., 1981), this may be taken as evidence that the treatment ponds were operating more effectively at the time of the second sampling. The changes observed in the effluent may help explain the differences in the clams between the first and second sampling. If the amount of low molecular weight dissolved hydrocarbons available to the clams decreased as it did in the effluent, their body burden would also decline. A continuous monitoring program of clams and effluent would be needed to adequately address this point.

Two possible sources of input water to the refinery, York River and Newport News City water, were examined for organic compounds. Total extractable organics were 0.002 ppm for the Newport News City water and 0.010 ppm for the York River. All of the compounds in these samples appeared to be of biological origin. Aromatic concentrations in the B/N fraction of both effluent samplings are comparable to those found in some municipal sewage effluent. Barrick (1982) reported aromatic totals up to 3 ppm while Eganhouse and Kaplan (1982) found total aromatics up to 1.2 ppm.

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Table 1

Sediment Sampling Locations

Station Number	Loran Coordinates	Latitude Longitude	Depth (feet)
1	27314.3 41438.0	37° 13.67' 76° 26.17'	35
2	27313.4 41438.6	37° 13.67' 76° 25.83'	25
3	27311.7 41439.1	37° 13.67' 76° 25.49'	18
4	27310.3 41439.7	37° 13.67' 76° 25.15'	6
5	27308.9 41439.9	37° 13.67' 76° 24.81'	10
6	27309.5 41443.4	37° 13.94' 76° 24.81'	35
7	27311.0 41442.6	37° 13.94' 76° 25.15'	38
8	27311.9 41447.4	37° 13.94 76° 25.49'	40
9	27313.5 41447.0	37° 13.94 76° 25.83'	42
10	27315.3 41441.4	37° 13.94' 76° 26.17'	44
11	27315.7 41444.7	37° 14.21' 76° 26.17'	52

Table 1 (continued)

Station Number	Loran Coordinates	Latitude Longitude	Depth (feet)
12	27314.0 41445.3	37° 14.21' 76° 25.83'	76
13	27312.9 41445.6	37° 19.21' 76° 25.49'	53
14	27311.5 41446.2	37° 14.21' 76° 25.15'	42
15	27309.8 41446.6	37° 14.21' 76° 29.81'	42
16	27317.0 41443.9	37° 14.21 76° 26.51'	44
17	27318.1 41443.4	37° 14.21' 76° 26.85'	38
18	27319.5 41442.8	37° 14.21' 76° 27.19'	38
19	27321.1 41442.4	37° 14.21' 76° 27.53'	36
20	27320.5 41439.2	37° 13.94' 76° 27.53'	56
21	27318.9 41439.6	37° 13.94' 76° 27.19'	60
22	27317.8 41440.3	37° 13.94' 76° 26.85'	60
23	27316.5 41440.9	37° 13.94' 76° 26.51'	58

Table 1 (continued)

Station Number	Loran Coordinates	Latitude Longitude	Depth (feet)
24	27315.6 41437.3	37° 13.67' 76° 26.51'	44
25	27316.8 41437.0	37° 13.67' 76° 26.85'	35
26	27318.7 41436.8	37° 13.67' 76° 27.19'	38
27	27319.6 41436.1	37° 13.67' 76° 27.53'	40
28	27319.2 41432.7	37° 13.40' 76° 27.53'	12
29	27317.7 41433.2	37° 13.40' 76° 27.19'	16
30	27317.3 41433.7	37° 13.40' 76° 26.85'	12
31	27315.0 41434.1	37° 13.40' 76° 26.51'	16
32	27313.8 41435.0	37° 13.40' 76° 26.17'	4
33	27312.3 41435.4	37° 13.40' 76° 25.83'	5
34	27311.2 41435.9	37° 13.40' 76° 25.49'	4
35	27309.7 41436.5	37° 13.40' 76° 25.15'	4

Table 1 (continued)

Station Number	Loran Coordinates	Latitude Longitude	Depth (feet)
36	27308.4 41437.8	37° 13.40' 76° 24.81'	3

Sediment samples were collected at the above locations. This information is displayed in Fig. 1.

Table 2

Clam Sampling Locations

Station Number	Loran Coordinates	Latitude Longitude	Depth (ft)
1	27313.8 41436.4	37° 13.55' 76° 26.01'	20
4	27313.9 41441.3	37° 13.90' 76° 25.80'	39
6	27318.5 41433.7	37° 13.45' 76° 27.36'	15
7	27318.5 41436.3	37° 13.74' 76° 27.47'	40

Clams were sampled at the above locations. This information is displyed in Fig. 1.

Table 3

Statistical treatment of replicate sediment extractions

			Station Number		
	21	22	25	27	2
Extraction 1 Mean in ppb	38	40	108	90	105
Extraction 2 Mean in ppb	35	47	80	89	100
Std. Deviation of Difference	8	8	35	11	12
Std. Error of Difference	2.5	2.4	11	3.6	4
Mean Difference	-2.6	7.3	-28	-1.2	-5
Difference ± 5% of mean	2	3	5	5	5
Paired 't'	-0.2	1.80	-2.09	1.10	1.20
DF - Degrees of freedom	10	10	10	10	10

Paired 't' tests were used to compare replicate extractions of sediment samples at five stations in order to determine the reproducibility of the analytical method.

Table 4

Statistical Comparison of Freeze Dried and Chemically Desiccated Sediments

	Station Number				
	2	16	19	25	30
Mean of Freeze Dried	352	76	189	205	45
Mean of Na_2SO_4+Q	368	66	169	230	51
Std. Deviation of Difference	108	36	63	59	15
Std. Error of Difference	31	11	19	18	4
Mean Difference	−16	10	−20	25	−6
Difference ± 5% of Mean	17	3	9	11	3
Paired 't'	−0.03	0.6	−0.6	0.8	−0.8
Degrees of Freedom	11	11	11	11	11

Paired 't' tests were used to compare the two drying techniques for sediments.

Table 5

Station Number	Sediment % Volatile Solids 3/83	12/83	Sediment % Solids 3/83	12/83
1	9.1	9.2	28.4	26.5
2	8.2	7.7	31.9	29.4
3	2.5	0.9	61.4	76.1
4	0.9	1.0	71.9	72.1
5	2.6	0.7	57.8	73.4
6	8.3	7.6	31.6	30.3
7	7.9	7.3	32.8	28.1
8	7.5	7.2	33.3	32.8
9	7.7	7.6	30.4	32.8
10	7.9	4.6	31.9	31.4
11	7.9	7.7	32.1	30.7
12	8.7	8.2	29.1	29.6
13	7.5	7.3	33.9	29.8
14	6.7	6.8	37.4	35.5
15	7.8	6.2	30.6	36.6
16	3.5	6.4	53.0	36.4
17	4.5	7.5	51.4	32.6
18	8.7	7.3	25.7	32.6
19	8.8	7.7	29.6	29.9
20	8.7	8.1	29.5	28.6
21	8.1	8.0	31.4	29.3
22	8.1	8.5	30.3	30.7
23	7.6	8.1	32.3	30.9
24	5.7	7.8	45.4	32.1
25	7.7	6.9	32.1	34.3
26	9.1	7.6	29.2	33.5
27	10.1	7.4	27.1	34.7
28	1.3	0.8	71.3	73.8
29	2.8	0.9	57.6	71.9
30	2.0	1.1	62.4	72.0
31	6.0	7.0	37.9	35.1
32	0.6	0.7	74.7	72.4
33	0.9	0.8	71.3	72.8
34	0.5	0.7	74.3	73.2
35	0.4	2.4	77.6	59.4
36	0.6	0.6	73.6	74.8

Percent solids is the weight percent of total sediment remaining after drying at 100°C. Percent volatile solids is the percent of dry sediment lost after heating it to 600°C.

Table 6
Sediments

Total and Pyrogenic PAH's (ppb dry wt)

Station Number	Total Resolved* 3-83	Total Resolved* 12-83	Total Pyrogenic* 3-83	Total Pyrogenic* 12-83
01	9547	5715	5240	3205
02	3235	1492	1074	959
03	888	106	404	65
04	43	73	24	42
05	368	19	177	12
06	2481	1020	707	436
07	2580	880	823	441
08	2565	782	802	443
09	1910	452	717	374
10	2063	1602	853	1031
11	2170	953	563	493
12	2112	455	574	383
13	2233	929	886	414
14	1561	813	566	371
15	1970	663	647	398
16	623	748	331	341
17	1824	3038	505	1515
18	1834	970	749	433
19	1582	764	659	521
20	2022	702	707	427
21	714	640	356	423
22	2406	852	655	403
23	1292	1580	500	462
24	272	1753	126	835
25	1602	2592	867	1191
26	1620	2270	941	1092
27	1910	2095	742	1003
28	192	63	91	37
29	1440	12	543	5
30	328	156	229	90
31	3690	3101	1600	1353
32	20	51	13	16
33	34	41	32	23
34	15	4	4	2
35	28	344	10	155
36	20	35	8	0

* Total resolved is the sum of all aromatic compounds resolved by the GC methods used.

** Total pyrogenic is the sum of selected compounds typically generated by combustion processes.

Table 7

Paired t Tests

Statistical Comparison of March and December Sediment Samples

	Total[1] Res. Arom.	Pyrogenics	B(a)A	B(a)pyrene	Fla
Mean of March 1983 all Stations	6.65	659	43	38	129
Mean of December 1983 all Stations	6.18	538	34	33	98
Std. Deviation of Difference Between Sampling Periods	1.10	447	36	28	131
Std. Error of Difference Between Sampling Periods	0.19	75	6	5	22
Mean Difference	-0.47	-121	-9	-5	-30
Mean Difference ± 5% of the Mean	0.05	30	2	2	5
Paired 't'	-2.20*	-1.21	-1.2	-0.6	-1.3
Degrees of Freedom	35	35	35	35	35

Paired 't' tests

[1] log transformation
* sign. different at = 0.05

Table 8

Concentrations of Total Aromatic Hydrocarbons in Clams (ppb/dry weight)

		Station			
		1	4	6	7
April		415	585	230	165
		1,140	1,485	295	235
		1,950	550	170	1,255
		2,300	360	395	1,555
	$\bar{x}$	1,450	745	275	805
December		75	230	130	360
		185	310	170	20
		95	405	65	25
		110	70	115	250
	$\bar{x}$	115	255	120	165

Total resolved aromatic compounds for each of four composite samples (five individuals per composite) at each station.

Table 9

Statistical Analysis of Total Aromatic Concentrations in Clams

	Sum of* Squares	df	Mean Square	F
Sampling Time	34.35	1	34.35	17.98**
Station	13.84	3	4.61	2.41
Interaction	14.64	3	4.88	2.55
Sub Total	62.83	7		
Within Groups	45.78	24	1.91	
Total	108.61	31		

*times 10^5
**sign at $F_{.99}(1,24)=4.72$

ANOVA for data listed in Table 8.

TABLE 10

Compounds Identified by Mass Spectrometry
Sediment - G3.2 and Effluent - G3.2
Numbers are ARIs.

		Sediment March 1983								Sediment December 1983				
Compound	Molecular Weight	1A	3A	12A	13A	21A	24A	31A	Effluent #1	1C	6C	23C	25C	Effluent #2
C_2-Benzene	106	-	-	-	-	-	-	-	+	-	-	-	-	-
C_2-Benzene	106	-	-	-	-	-	-	-	+	-	-	-	-	-
C_3-Benzene	120	-	-	-	-	-	-	-	+	-	-	-	-	-
C_3-Benzene	120	-	-	-	-	-	-	-	+	-	-	-	-	-
C_9H_{10}	118	-	-	-	-	-	-	-	+	-	-	-	-	-
C_4-Benzene	134	-	-	-	-	-	-	-	+	-	-	-	-	-
Methylthiobenzene	124	-	-	-	-	-	-	-	+	-	-	-	-	-
C_4-Benzene	134	-	-	-	-	-	-	-	+	-	-	-	-	-
C_4-Benzene	134	-	-	-	-	-	-	-	+	-	-	-	-	-
Me-C_9H_{10}	132	-	-	-	-	-	-	-	+	-	-	-	-	-
Me-C_9H_{10}	132	-	-	-	-	-	-	-	+	-	-	-	-	-
C_4-Benzene	134	-	-	-	-	-	-	-	+	-	-	-	-	-
Naphthalene	128	000	-	000	000	000	-	000	000	000	000	000	000	000
Benzothiophene	134	-	-	-	-	-	-	-	4.9	-	-	-	-	-
C_2-C_9H_{10}	146	-	-	-	-	-	-	-	33.7	-	-	-	-	-
C_2-C_9H_{10}	146	-	-	-	-	-	-	-	37.2	-	-	-	-	-
Me-Benzothiophene	148	-	-	-	-	-	-	-	53.1	-	-	-	-	-
2-Me-Naphthalene	142	54.9	-	51.8	53.9	54.8	-	55.6	58.1	55.2	54.4	53.6	54.7	56.5
Me-Benzothiophene	148	-	-	-	-	-	-	-	60.3	-	-	-		-
1-Me-Naphthalene	142	62.7	-	60.7	62.4	62.3	-	62.9	-	63.4	63.1	63.7	62.9	-
Biphenyl	154	100	-	100	100	100	-	100	100	100	100	100	100	100
Et-Naphthalene	156	103.2	-	103.9	103.7	-	-	103.3	103.5	-	-	-	-	-

Table 10 (continued)

Compound	Molecular Weight	Sediment March 1983 1A	3A	12A	13A	21A	24A	31A	Effluent #1	Sediment December 1983 1C	6C	23C	25C	Effluent #2
C_2-Naphthalene	156	106.2	-	106.1	106.3	105.9	-	106.3	-	-	106	-	106.7	106.5
C_2-Naphthalene	156	-	-	106.9	-	-	-	-	107.2	-	-	-	-	-
C_2-Benzothiophene	162	-	-	-	-	-	-	-	108.9	-	-	-	-	-
C_2-Naphthalene	156	109.9	-	109.9	110.5	109.9	-	110.8	-	-	110	110.6	-	-
C_2-Naphthalene	156	-	-	111.2	-	-	-	-	111.0	-	-	-	-	-
C_2-Naphthalene	156	-	-	-	-	-	-	-	111.9	-	-	-	-	-
C_2-Naphthalene	156	115.3	-	115.6	115.7	115.1	-	115.7	116	115.2	-	115.6	-	-
Acenaphthrylene	156	117.2	-	117.4	-	-	-	117.7	-	117.5	-	-	-	-
C_2-Naphthalene	156	119.8	-	119.3	-	-	-	119.4	120.2	-	-	-	-	119.1
Acenaphthene	154	127.5	-	127.1	127.0	126.9	-	127.5	128.1	127.2	-	-	127.5	126.9
4-Me-Biphenyl	168	129.3	-	128.2	129.0	128.7	-	129.3	-	-	-	128.5	-	129.1
3-Me-Biphenyl	168	131.0	-	130.8	130.9	-	-	131.9	131.6	-	-	-	-	-
C_3-Naphthalene	170	133.5	-	133.3	133.7	133.3	-	133.7	-	-	-	-	-	-
Me-154	168	-	-	-	-	-	-	-	133.7	-	-	-	-	-
C_3-Naphthalene	170	-	-	-	-	-	-	-	133.7	-	-	-	-	133.0
Dibenzofuran	168	135.4	-	135.2	136.0	-	-	135.8	-	136.0	-	135..7	135.6	-
C_3-Naphthalene	170	-	-	-	-	-	-	-	135.8	-	-	-	-	-
Me-154	168	-	-	-	-	-	-	-	137.2	-	-	-	-	-
C_3-Naphthalene	170	-	-	-	-	-	-	-	137.2	-	-	-	-	-
C_3-Naphthalene	170	-	-	139.0	138.8	-	-	-	139.0	-	-	-	-	138.6
C_3-Naphthalene	170	-	-	140.0	139.8	-	-	139.9	140.4	-	-	-	-	139.6
Unknown	159	-	-	-	-	-	-	140.6	-	-	-	-	-	-
C_3-Naphthalene	170	-	-	-	-	-	-	-	141.9	-	-	-	-	-
C_2-Biphenyl	182	141.4	-	141.0	-	140.7	-	140.6	-	141.2	-	-	-	-
C_3-Naphthalene	170	-	-	143.9	144.4	-	-	-	142.9	-	-	-	-	143.6

Table 10 (continued)

		Sediment March 1983								Sediment December 1983				
Compound	Molecular Weight	1A	3A	12A	13A	21A	24A	31A	Effluent #1	1C	6C	23C	25C	Effluent #2
C_2-154	182	-		143.9	-	143.1	-	-	-	-	-	-	-	-
C_3-Benzothiophene	176	-	-	-	-	-	-	-	144.8	-	-	-	-	144.9
C_3-Naphthalene	170	145.2	-	145.0	-	-	-	145.1	-	-	-	-	-	-
C_3-Naphthalene	170	-	-	-	-	-	-	-	147.1	-	-	-	-	-
C_3-Naphthalene	170	149.1	-	148.9	148.8	-	-	148.9	151.4	-	-	-	-	152.1
Fluorene	166	152.8	-	152.7	152.1	152.4	-	152.9	-	152.9	-	152.4	152.5	-
C_3-Naphthalene	170	-	-	154.6	-	-	-	-	-	-	-	-	-	-
Me-154	168	-	-	-	-	-	-	-	-	-	-	-	-	155.3
Fluorene	166	-	-	-	-	-	-	-	155.5	-	-	-	-	-
C_2-154	182	-	-	156.9	-	-	-	-	-	-	-	-	-	156.7
Me-Acenaphthene	168	157.7	-	-	-	-	-	157.9	158.3	157.6	-	-	-	-
C_2-154	182	157.7	-	-	-	-	-	-	-	-	-	-	-	-
Me-Acenaphthene	168	159.1	-	-	-	-	-	159.9	159.9	-	-	-	-	-
C_2-154	182	159.1	-	159.5	159.6	-	-	159.9	-	-	-	-	-	-
C_4-Napthtlene	184	-	-	-	-	-	-	162.3	-	-	-	-	-	-
C_2-154	182	-	-	-	-	-	-	-	-	-	-	-	-	162.5
Me-Dibenzofuran	182	163.6	-	162.7	163.1	162.3	-	163.2	-	163.3	-	-	162.9	-
Me-Dibenzofuran	182	166.3	-	166.4	166.3	166.2	-	166.8	166.4	167.1	-	165.6	165.9	-
C_4-Naphthalene	184	-	-	-	-	-	-	-	167.8	-	-	-	-	-
Me-Dibenzofuran	182	168.7	-	168.8	-	-	-	169.4	-	-	-	-	168.6	-
C_4-Naphthalene	184	-	-	-	-	-	-	170.9	169.8	-	-	-	-	169.2
C_2-154/ Me-Dibenzofuran	182	-	-	-	-	-	-	-	170.8	-	-	-	-	-
C_4-Naphthalene	184	-	-	-	-	-	-	-	172.5	-	-	-	-	-
C_4-154	210	173.4	175.0	174.0	-	173.7	-	-	-	-	-	-	-	-

Table 10 (continued)

Compound	Molecular Weight	Sediment March 1983 1A	3A	12A	13A	21A	24A	31A	Effluent #1	Sediment December 1983 1C	6C	23C	25C	Effluent #2
C_4-Naphthalene	184	-	-	175.6	-	-	-	176.4	174.9	-	-	-	-	-
9,10-Dihydroanthracene	180	176.9	-	-	-	-	-	-	-	-	-	-	-	-
C_4-Naphthalene	184	-	-	-	-	-	-	-	177.9	-	-	-	-	-
C_4-Naphthalene	184	-	-	-	-	-	-	-	179.4	-	-	-	-	-
C_5-Naphthalene	198	-	-	-	-	-	-	179.4	-	-	-	-	-	-
C_4-Naphthalene	184	-	-	-	-	179.7	-	180.4	-	-	-	-	-	-
2-Me-Fluorene	180	182.3	-	-	181.9	182.5	-	182.3	-	-	-	-	-	-
C_3-154/C_2-Dibenzofuran	196	182.3	-	-	-	-	-	-	-	-	-	-	-	-
C_4-Naphthalene	184	-	-	-	-	-	-	-	182.9	-	-	-	-	-
C_2-154	182	-	-	-	-	-	-	-	-	-	-	-	-	183.3
1-Me-Fluorene	180	183.8	-	183.6	183.8	-	-	183.3	-	-	-	-	-	-
Unknown	-	-	-	-	-	-	-	183.3	-	-	-	-	-	-
Me-Fluorene	180	-	-	-	-	-	-	-	184.5	-	-	-	-	-
C_2-154	182	-	-	-	-	-	-	-	-	-	-	-	-	184.8
Me-Fluorene	180	185.4	-	-	-	-	-	185.8	186.3	-	-	-	-	-
C_3-154/C_2-Dibenzofuran	196	185.4	-	185.4	185.6	185.3	-	185.8	186.3	-	-	-	184.9	-
Mixture	-	-	-	-	-	-	-	187.7	187.5	-	-	-	-	-
C_5-Naphthalene	198	-	-	-	-	-	-	-	188.4	-	-	-	-	-
Me-Fluorene	180	-	-	-	-	-	-	-	188.4	-	-	-	-	-
C_3-154/C_2-Dibenzofuran	196	-	-	-	-	-	-	-	188.6	-	-	-	-	-
C_3-154/C_2-Dibenzofuran	196	-	-	190.8	190.8	-	-	190.5	189.7	-	-	-	190.6	189.4
Me-Fluorene	180	-	-	-	-	-	-	190.5	-	-	-	-	-	-
C_4-Naphthalene	184	-	-	190.8	-	-	-	-	-	-	-	-	190.6	-
C_3-154/C_2-Dibenzofuran	196	191.3	-	-	-	191.5	-	-	192.0	191.6	-	-	-	192.6
C_3-154/C_2-Dibenzofuran	196	192.2	-	192.3	192.2	192.4	-	192.0	-	192.9	-	192.4	192.3	-

Table 10 (continued)

		Sediment March 1983								Sediment December 1983				
Compound	Molecular Weight	1A	3A	12A	13A	21A	24A	31A	Effluent #1	1C	6C	23C	25C	Effluent #2
C_4-Naphthalene	184	-	-	-	-	-	-	-	193.1	-	-	-	-	-
C_2-154/Me-Dibenzofuran	182	-	-	-	-	-	-	-	193.1	-	-	-	-	-
Dibenzothiophene	184	193.9	-	193.8	194.0	194.0	-	193.6	194.8	194.2	193.3	-	193.7	-
C_3-154/C_2-Dibenzofuran	196	195.3	-	-	-	195.4	-	195.8	194.8	-	-	-	195.5	195.0
C_3-154/C_2-Dibenzofuran	196	-	-	-	-	196.2	-	-	196.3	-	-	-	-	195.9
C_4-154/C_3-Dibenzofuran	210	-	-	-	-	-	-	-	196.3	-	-	-		-
C_3-154/C_2-Dibenzofuran	196	-	-	-	-	198.2	-	-	198.6	-	-	-	198.1	-
Phenanthrene	178	200	200	200	200	200	200	200	-	200	200	200	200	-
C_5-Naphthalene	198	-	-	-	-	-	-	-	203.9	-	-	-	-	-
C_3--154/C_2-Dibenzofuran	196	-	-	-	-	-	-	-	203.9	-	-	-	-	-
Anthracene	178	203.7	-	202.6	203.8	203.7	-	202.7	203.9	202.5	203.3	204.1	204.5	203.4
C_4-154/C_3-Dibenzofuran	210	-	-	-	-	-	-	206.6	-	-	-	-	-	-
C_3-154/C_2-Dibenzofuran	196	-	-	-	-	-	-	206.6	-	-	-	-	-	-
C_2-Fluorene	194	-	-	213.2	213.5	213.5	-	213.5	-	-	-	212.8	-	-
C_2-Fluorene	194	-	-	215.5	215.5	-	-	215.8	216.1	-	215.6	-	-	214.8
C_3-154/C_2-Dibenzofuran	196	-	-	-	-	-	-	-	216.1	-	-	-	-	215.8
C_2-Fluorene	194	-	-	217.5	217.9	-	-	216.9	216.9	-	-	217.4	-	216.8
C_4-154/C_3-Dibenzofuran	210	-	-	-	-	-	-	216.9	-	-	-	-	-	-
C_4-154/C_3-Dibenzofuran	210	-	-	-	-	218.0	-	-	-	-	-	-	-	-
C_2-Fluorene	194	-	-	-	-	-	-	221.2	219.7	-	-	-	-	-
C_4-154/C_3-Dibenzofuran	210	-	-	220.6	-	-	-	221.1	219.7	-	-	-	-	-
C_5-Naphthalene	198	-	-	-	-	-	-	-	221.4	-	-	-	-	-
C_3-154/C_2-Dibenzofuran	196	-	-	-	-	-	-	-	221.4	-	-	-	-	220.5

Table 10 (continued)

		Sediment March 1983								Sediment December 1983				
Compound	Molecular Weight	1A	3A	12A	13A	21A	24A	31A	Effluent #1	1C	6C	23C	25C	Effluent #2
C_2-Fluorene	194	-	-	-	-	-	-	-	221.4	-	-	-	-	-
Me-Dibenzothiophene	198	223.4	-	223.6	224.4	224.1	-	224.2	-	-	-	223.8	-	-
C_4-154/C_3-Dibenzofuran	210	-	-	226.4	227.1	226.9	-	225.9	-	-	-	-	-	-
Me-Dibenzothiophene	198	-	-	-	-	-	-	-	226.4	-	-	-	-	-
1-Phenylnaphthalene	204	227.3	-	227.7	228.4	228.3	-	228.4	-	228.7	228.1	227.3	227.9	-
C_4-154/C_3-Dibenzofuran	210	227.3	-	227.7	-	-	-	228.4	229.5	-	-	-	-	229.5
C_2-Fluorene	194	-	-	-	-	-	-	-	229.5	-	-	-	-	-
C_4-154/C_3-Dibenzofuran	210	-	-	-	-	230.5	-	230.4	-	-	-	-	-	-
Me-Dibenzothiophene	198	230.1	-	230.7	230.5	-	-	230.4	-	231.3	231.6	231.0	-	-
C_4-154/C_3-Dibenzofuran	210	-	-	-	-	-	-	-	232.0	-	-	-	-	-
Me-Dibenothiphene	198	-	-	-	-	-	-	-	232.0	-	-	-	-	-
Me-Dibenzothiophene	198	-	-	-	-	-	-	-	233.4	-	-	-	-	-
3-Me-Phenanthrene	192	236.6	237.6	236.6	236.8	237.5	237.2	237.6	-	238.1	237.5	237.9	237.5	236.9
2-Me-Phenanthrene	192	-	239.1	239.0	239.0	239.0	239.6	239.0	240.7	239.6	239.1	239.3	238.9	238.4
Me-Phenanthrene	192	-	-	-	-	-	-	-	242.0	-	-	-	-	-
4-H-Cyclopenta(def)-phenanthrene	190	241.9	243.3	243.0	-	243.3	-	242.5	-	243.8	243.8	243.4	243.0	-
Me-178	192	244.2	-	244.4	244.9	244.8	-	244.0	-	245.2	-	244.5	244.3	244.3
Me-178	192	245.8	246.5	245.9	246.4	246.2	245.6	245.4	-	246.6	246.7	-	245.9	-
C_4-Acenaphthrylene/ C_3-Fluorene	208	-	-	-	-	-	-	250.1	251.8	-	-	-	-	-
C_2-Dibenzothiophene	212	-	-	254.9	-	-	-	-	-	-	-	-	-	-
C_2-Dibenzothiophene	212	-	256.2	-	255.3	256.3	255.5	255.4	-	-	-	256.0	-	-
C_4-Acenaphthrylene/ C_3-Fluorene	208	-	-	-	-	-	-	256.7	256.7	-	-	-	-	257.8
C_2-Dibenzothiophene	212	-	-	-	260.3	-	-	-	258.2	-	-	-	-	-
C_3-Fluorene	208	-	-	-	-	-	-	-	260.8	-	-	-	-	261.6

Table 10 (continued)

Compound	Molecular Weight	Sediment March 1983 1A	3A	12A	13A	21A	24A	31A	Effluent #1	Sediment December 1983 1C	6C	23C	25C	Effluent #2
2-Phenylnaphthalene	204	261.1	262.5	262.1	262.9	262.5	261.7	261.6	-	262.5	-	261.7	261.7	-
C_2-Dibenzothiophene	212	-	262.5	262.1	-	-	-	-	-	-	-	-	-	-
C_3-Fluorene	208	-	-	-	-	-	-	-	263.1	-	-	-	-	263.0
C_2-Dibenzthiophene	212	-	-	-	-	-	-	-	265.7	-	-	-	-	-
C_2-178	206	266.0	-	-	-	267.3	-	266.6	-	-	-	262.1	-	-
C_2-Dibenzothiophene	212	-	-	-	-	-	-	269.3	-	269.7	270.0	269.8	-	268.6
Unknown	240	-	-	-	-	-	-	269.3	-	-	-	-	-	-
C_2-178	206	-	-	-	-	-	-	-	269.2	-	-	-	-	-
C_2-Dibenzothiophene	212	-	-	-	-	-	-	-	270.4	-	-	-	-	-
C_2-178	206	271.3	272.2	-	-	272.4	-	-	-	272.3	-	272.3	272.3	271.2
C_2-Dibenzothiophene	212	-	-	-	-	-	-	-	-	-	-	272.3	-	-
C_2-178	206	-	-	273.0	273.3	-	-	272.9	-	-	-	-	-	273.7
C_2-178	206	273.2	-	-	-	-	274.1	-	274.4	-	-	-	-	-
C_2-178	206	-	-	276.5	275.7	-	-	275.8	277.2	275.2	-	-	-	-
C_2-178	206	278.8	279.1	-	279.8	279.1	-	279.3	-	279.7	280.4	279.9	279.8	-
C_2-178	-	-	-	281.5	281.2	281.8	-	280.8	281.8	-	-	-	-	280.8
C_2-178	206	-	-	282.6	283.3	-	-	282.8	283.9	283.2	-	283.5	282.2	282.7
C_3-Dibenzothiophene	226	-	-	-	283.3	-	-	282.8	-	-	-	-	-	-
C_3-Dibenzothiophene	226	-	-	284.7	-	-	-	-	-	-	-	-	-	-
C_2-178	206	-	-	284.7	-	-	-	-	285.1	-	-	-	-	-
C_3-Dibenzothiophene	226	-	-	-	-	-	-	-	285.1	-	-	-	-	-
C_2-178	206	-	-	-	-	-	-	-	287.0	-	-	-	-	-
Fluoranthene	202	286.4	285.8	287.6	285.4	286.5	285.9	286.8	287.0	285.9	286.2	285.9	286.9	-

Table 10 (continued)

		Sediment March 1983								Sediment December 1983				
Compound	Molecular Weight	1A	3A	12A	13A	21A	24A	31A	Effluent #1	1C	6C	23C	25C	Effluent #2
C_3-Dibenzothiophene	226	-	-	291.1	289.8	-	290.0	290.1	291.6	-	290.4	290.3	290.2	289.7
Benzo(def)dibenzothiophene	208	293.7	294.8	296.1	-	295.0	294.7	294.8	-	295.7	295.6	295.6	295.8	-
C_2-178	206	-	-	-	-	-	-	-	295.5	-	-	-	-	-
Me-phenylnaphthalene	218	293.7	-	296.1	-	-	294.7	294.8	-	-	295.6	-	-	-
C_3-Dibenzothiophene	226	-	-	-	-	-	-	-	297.5	-	-	-	-	296.2
Pyrene	202	300	300	300	300	300	300	300	300	300	300	300	300	300
C_3-178	220	-	-	-	-	-	-	-	300	-	-	-	-	300
C_3-Dibenzothiophene	226	-	-	-	-	-	-	-	300	-	-	-	-	-
Me-Cyclopenta(def)-phenanthrene	204	302.2	-	-	-	-	-	-	303.6	-	-	-	-	-
Me-Phenylnaphthalene	218	302.2	303.8	304.1	303.7	303.9	303.4	304.0	-	303.5	304.6	304.2	304.4	-
C_3-178	220	-	-	306.3	-	-	-	-	303.6	-	-	-	-	-
C_3-Dibenzothiophene	226	-	-	-	-	-	-	-	303.6	-	-	-	-	-
Me-Phenylnaphthalene	218	306.5	-	-	-	-	-	-	-	-	-	-	-	-
Me-Phenylnaphthalene	218	-	309.1	308.6	308.1	309.3	-	309.4	-	309.8	310.6	310.4	309.6	-
C_3-Dibenzothiophene	226	-	309.1	-	-	-	-	-	309.8	-	-	-	-	308.8
Unknown	252	-	-	-	-	-	310.6	-	-	-	-	-	-	-
Me-Phenylnaphthalene	218	312.1	-	-	-	-	-	-	-	-	-	-	-	-
C_3-178	220	-	-	313.2	314.0	313.8	-	-	-	-	-	-	-	-
Me-Phenylnaphthalene	218	-	-	313.2	314.0	313.8	-	314.0	-	-	-	-	-	-
C_3-178	220	-	315.3	314.5	315.1	-	315.0	315.4	315.5	-	315.5	315.3	315.7	315.7
Me-Phenylnaphthalene	218	-	-	-	-	-	315.0	-	-	316.1	-	-	-	-
C_3-178	220	-	-	-	-	-	-	-	316.6	-	-	-	-	-
Me-Phenylnaphthalene	218	317.8	-	-	-	-	-	-	-	-	-	-	-	-
C_3-178	220	-	-	318.5	-	-	-	318.4	-	-	-	-	-	-
Me-202	216	320.2	-	-	321.0	-	-	-	-	-	-	-	-	-

Table 10 (continued)

Compound	Molecular Weight	Sediment March 1983							Effluent	Sediment December 1983				Effluent
		1A	3A	12A	13A	21A	24A	31A	#1	1C	6C	23C	25C	#2
C_3-178	220	-	-	320.2	-	319.6	-	320.8	320.4	-	-	319.4	319.5	320.4
C_3-178	220	-	-	-	-	-	-	-	321.9	-	-	-	-	322.9
Me-Phenylnaphthalene	218	-	-	320.2	-	-	-	320.8	-	321.2	-	-	-	-
Me-202	216	-	323.0	322.6	-	321.8	-	323.3	-	323.5	323.9	323.9	324.3	-
Me-208	222	-	-	-	-	-	-	324.8	-	-	-	-	-	-
C_4-Dibenzothiophene	240	-	-	-	-	-	-	324.8	-	-	-	-		
p,p'-DDE	316	-	-	326.3	-	-	-	326.8	-	-	-	-	-	-
C_3-178	220	-	-	-	-	-	-	326.8	-	-	-	-	-	-
Me-202	220	-	-	-	328.2	328.3	-	328.9	-	-	-	-	-	-
Me-202	216	329.7	331.5	330.1	330.9	-	330.2	330.7	-	-	330.9	-	-	-
C_2-Phenylnaphthalene	232	329.7	-	-	-	-	-	-	-	-	-	-	-	-
Benzo(a)fluorene/ Me-202	216	-	331.5	-	-	331.6	-	-	-	332.0	-	332.2	331.6	331.1
Retene	234	-	-	334.2	-	-	334.3	334.5	-	-	-	335.4	-	-
Benzo(b)fluorene/ Me-202	216	-	-	336.3	336.2	336.9	-	-	-	337.0	-	-	334.6	-
Me-Phenylnaphthalene	218	337.6	-	-	-	-	-	-	-	-	-	-	-	-
C_2-Phenylnaphthalene	232	337.6	-	-	-	-	-	-	-	-	-	-	-	-
Me-202	216	-	336.4	-	-	-	336.3	337.0	-	-	-	337.0	337.6	-
Me-Phenylnaphthalene	218	-	-	-	-	340.2	-	341.1	-	340.9	-	-	341.5	341.6
C_2-Phenylnaphthalene	232	-	-	-	-	340.2	-	-	-	-	-	-	-	-
Me-202	216	341.8	-	-	-	-	-	-	-	-	-	-	-	-
Me-202	216	344.1	343.5	343.6	343.2	343.6	343.2	344.0	344.6	344.8	344.5	344.7	-	344.0
C_2-Phenylnaphthalene	232	344.1	-	-	343.2	-	-	-	-	-	-	-	-	-
Me-202	216	-	345.8	345.9	345.6	345.9	-	346.3	346.8	-	-	-	345.4	346.5
C_2-Phenylnaphthalene	232	-	-	-	-	345.9	-	346.3	-	-	-	-	-	-
C_2-Phenylnaphthalene	232	349.2	-	-	-	-	-	-	-	-	-	-	-	-
C_2-Phenylnapthtalene	232	-	-	-	-	352.0	-	-	-	352.2	-	-	-	351.6

Table 10 (continued)

		Sediment March 1983								Sediment December 1983				
Compound	Molecular Weight	1A	3A	12A	13A	21A	24A	31A	Effluent #1	1C	6C	23C	25C	Effluent #2
Unknown	244	-	-	-	-	357.8	-	-	-	-	-	-	-	-
C_2-202	230	359.4	-	-	-	-	-	-	-	-	-	359.7	-	-
C_4-178/Benzo-naphthothiopene	234	-	-	360.2	-	-	-	360.0	361.7	-	-	-	360.1	360.2
C_2-202	230	-	-	-	-	-	-	362.4	-	362.0	-	362.8	-	-
C_2-202	230	364.9	-	366.6	367.7	366.0	-	-	-	367.5	367.4	-	-	-
C_2-208	236	-	-	-	-	366.0	-	-	-	-	-	-	-	367.9
C_2-202	230	369.8	-	-	-	-	-	-	-	-	-	-	-	-
C_2-202	230	-	-	372.3	-	371.5	-	372.4	-	372.8	372.3	373.0	372.6	-
C_2-202	230	376.1	-	-	-	-	-	377.7	377.7	-	-	-	-	-
Benzonaphthothiophene	234	376.1	377.8	378.7	377.4	376.9	-	377.7	-	378.9	-	379.0	378.9	-
C_2-202	230	-	-	-	-	-	-	-	-	-	-	-	-	379.2
Benzonaphthothiophene	234	-	-	-	-	-	-	380.2	-	-	-	-	-	-
Benzo(ghi)fluoranthene	226	379.3	381.1	379.8	379.9	379.3	-	380.2	-	381.9	380.5	381.2	382.0	-
Benzo(c)phenanthrene	228	379.3	381.1	379.8	381.5	380.9	-	381.8	-	-	-	-	-	-
C_2-202	230	-	381.1	379.8	-	-	-	-	-	-	-	-	-	380.4
Benzonaphthothiophene	234	383.0	-	-	-	383.1	-	-	-	-	-	-	-	-
Benzonaphthothiophene	234	-	-	385.5	-	-	-	385.8	-	-	385.7	386.8	-	-
C_2-202	230	-	-	-	-	-	-	385.8	-	-	-	-	386.7	387.5
C_2-202	230	-	-	-	-	-	-	388.3	-	-	-	-	388.9	388.7
Benzonaphthothiophene	234	387.5	-	-	-	-	-	-	-	-	-	-	-	-
Benzonaphthothiophene	234	389.9	-	-	-	390.7	-	391.7	-	-	390.4	-	-	-
Unknown	295	-	-	-	-	-	391.1	-	-	-	-	-	-	-
Benzo(a)anthracene	228	396.7	396.4	398.0	396.7	396.9	397.9	398.2	-	397.6	397.7	397.4	398.6	-
Chrysene/triphenylene	228	400	400	400	400	400	400	400	400	400	400	400	400	400
Tetramethyloctahydro-chrysene	292	-	-	-	400.8	-	-	-	-	-	-	398.7	400.7	-
Unknown (Base 178)	-	-	-	-	401.6	400.6	-	400	-	-	-	-	-	-

Table 10 (continued)

Compound	Molecular Weight	Sediment March 1983 1A	3A	12A	13A	21A	24A	31A	Effluent #1	Sediment December 1983 1C	6C	23C	25C	Effluent #2
Unknown	228	403.5	-	-	-	403.4	-	-	-	-	-	403.4	-	-
Tetramethyloctahydrochrysene	292	-	-	404.4	405.6	-	404.5	-	-	-	-	404.5	403.6	-
Tetramethyloctahydrochrysene	292	-	-	-	-	-	408.2	-	-	-	-	-	407.6	-
Unknown	258	404.4	-	-	-	-	-	-	-	-	-	-	-	-
C_3-202	244	-	-	-	-	-	-	404.5	-	-	-	-	-	405/4
Unknown	226	-	-	-	-	-	-	404.5	-	405.1	-	-	-	-
Unknown	228	-	-	-	-	-	-	404.5	-	405.1	-	-	-	-
Me-234	248	-	406.5	-	-	-	-	-	-	-	-	407	-	-
C_3-202	244	-	-	-	-	-	-	-	-	-	-	-	-	408.3
Unknown	292	-	-	409.1	410.2	-	-	409.0	-	-	-	-	-	-
C_3-202	244	-	-	-	-	-	-	-	-	-	-	-	-	410.8
Me-228	242	411.6	-	-	-	411.8	-	-	-	-	-	-	-	-
Me-234	248	411.6	-	-	-	411.8	-	411.4	-	-	-	-	-	-
C_3-202	244	-	-	-	-	-	-	-	-	-	-	-	-	412.8
Me-234	248	414.4	-	-	413.0	-	-	-	-	-	-	-	412.9	-
Me-228	242	414.1	-	-	-	-	-	-	-	413.2	-	-	-	-
C_3-202	244	-	-	-	-	-	-	-	-	-	-	-	-	417.6
Unknown	292	-	-	-	-	-	-	415.6	-	-	-	-	-	-
Me-234	248	417.1	-	-	-	-	-	-	-	-	-	-	-	-
C_3-202	244	-	-	-	-	-	-	-	-	-	-	-	-	419.0
Me-228	242	-	-	-	-	419.3	-	-	-	-	-	-	-	-
Me-228	242	-	-	-	-	419.3	-	-	-	-	-	-	-	-
Me-234	248	-	-	420.1	-	419.3	-	420.2	-	-	-	420.1	420.6	-
C_3-202	244	-	-	-	-	-	-	-	-	-	-	-	-	420.9
Unknown (Base peak 178)	-	-	-	-	-	419.3	-	-	-	-	-	-	-	-
Me-234	248	-	-	-	-	-	-	-	-	-	-	-	424.5	-
Me-234	248	-	-	-	-	427.6	-	426.3	-	-	-	-	-	-

Table 10 (continued)

Compound	Molecular Weight	Sediment March 1983							Effluent #1	Sediment December 1983				Effluent #2
		1A	3A	12A	13A	21A	24A	31A		1C	6C	23C	25C	
C_3-202	244	-	-	-	-	-	-	-	-	-	-	-	-	428.8
Me-228	242	429.1	429.7	-	-	-	-	428.2	-	-	-	428.1	-	-
Me-228	242	431.1	-	431.3	-	431.1	431.6	431.3	-	431.0	431.4	431.4	431.0	430.9
Me-228	242	-	-	-	-	433.2	-	433.5	-	-	-	433.3	433.6	-
Me-226	240	434.1	-	-	-	434.6	-	-	-	434.0	-	-	-	-
ME-228	242	-	-	-	-	-	-	-	-	-	-	-	-	435.0
Unknown	326	-	-	-	-	-	434.4	-	-	-	-	-	-	-
Me-226	240	435.5	-	-	-	436.7	-	-	-	-	-	-	-	-
Me-226	240	438.0	-	-	-	-	-	437.8	-	437.6	-	-	-	-
C_2-234	262	-	437.5	-	-	-	-	437.8	-	-	-	-	438.0	-
ME-228	242	-	-	-	-	-	-	-	-	-	-	-	-	438.5
C_2-234	262	-	-	-	-	-	-	440.1	-	-	-	-	-	-
Me-228	242	-	-	-	-	440.7	-	440.1	-	-	439.5	-	440.1	-
1-Phenylphenanthrene	254	-	-	-	-	440.7	-	-	-	-	-	-	-	-
2,2'-Binaphthyl (spike)	254	443.6	446.3	447.1	445.9	444.8	447.0	446.1	-	446.0	446.7	-	446.0	-
Unknown	420	-	-	-	-	-	-	457.1	-	-	-	-	-	-
C_2-228	256	458.7	-	-	-	-	-	459.8	-	460.9	-	459.1	459.9	460.1
C_2-228	256	-	-	-	-	-	-	463.0	-	-	-	-	-	462.9
Benzo(j,b,& k)-fluoranthenes	252	475.8	474.8	475.8	475.2	475.2	476.4	474.6	-	476.4	476.9	475.1	475.9	476.9
C_3-234	276	-	-	-	-	-	-	-	-	-	-	-		480.4
Benzo(e)acephenanthrylene	252	481.5	-	-	-	-	-	-	-	482.0	482.4	481.8	482.7	-
C_3-234	276	-	-	-	-	-	-	-	-	-	-	-	-	484.0
C_3-234	276	-	-	-	-	-	-	-	-	-	-	-	-	488.4
Benzo(e)pyrene	252	490.7	493.9	491.0	492.1	491.1	491.2	491.0	-	491.6	492.5	491.0	492.1	492.3
Benzo(a)pyrene	252	494.7	496.7	495.1	494.8	495.0	494.9	493.9	-	495.7	494.8	494.9	495.1	494.7
Unknown (Base peak 178)	-	-	-	-	494.8	495.0	-	-	-	-	-	-	-	-
Perylene	252	500	500	500	500	500	500	500	-	500	500	500	500	500
Me-252	266	506.1	-	-	-	-	-	-	-	-	-	-	-	-

Table 10 (continued)

		Sediment March 1983								Sediment December 1983				
Compound	Molecular Weight	1A	3A	12A	13A	21A	24A	31A	Effluent #1	1C	6C	23C	25C	Effluent #2
Unknown	306	-	-	-	-	507.7	-	-	-	-	-	-	-	-
Me-252	266	-	-	-	-	-	-	-	-	-	-	508.4	-	508.0
ME-252	266	-	-	-	-	-	-	-	-	-	-	-	-	510.5
Hopanoid	-	-	-	510.9	-	-	-	-	-	-	-	-	-	-
Unknown	-	-	-	-	-	511.9	-	-	-	-	-	-	-	-
Me-258	272	-	-	-	-	-	-	512.1	-	-	-	-	-	-
Me-252	266	516.0	-	-	-	-	-	-	-	-	516.2	-	517.5	515.5
Unknown	306	-	-	-	-	522.8	-	-	-	-	-	-	-	-
Me-252	266	525.7	-	-	-	-	-	524.6	-	524.6	-	524.3	525.1	525.8
Me-252	266	-	-	-	-	-	-	-	-	530.7	-	529.3	-	-
Me-252	266	534.1	-	-	-	-	-	-	-	535.1	-	-	-	-
Unknown	264	534.1	-	-	-	-	-	-	-	-	-	-	-	-
Me-252	266	537.0	-	-	-	-	-	-	-	538.2	537.7	537.0	-	538.1
C_2-252	280	-	-	-	-	-	-	-	-	-	-	-	-	549.0
Hopanoid	-	-	-	553.4	-	-	-	-	-	-	-	-	-	-
C_2-252	280	-	-	-	-	-	-	560.6	-	558.7	560.1	558.3	558.3	559.3
p-Quaterphenyl (Spike)	306	567.8	566.9	570.8	571.0	567.0	-	568.3	-	-	-	-	-	-
C_2-252	280	-	-	-	-	-	-	-	-	-	-	-	-	578.0
Hopanoid	-	-	-	578.5	-	-	-	-	-	-	-	-	-	-
C_2-252	280	-	-	-	-	-	-	-	-	-	-	-	-	580.7
Hopene	-	-	583.0	-	-	-	583.3	-	-	-	-	-	-	-
Indeno(cd)pyrene	276	583.3	-	584.4	585.6	582.9	-	584.3	-	584.9	585.0	583.9	583.5	-
Benzo(ghi)perylene	276	600	600	600	600	600	-	600	-	600	600	600	600	600
Terpenoid	-	-	-	-	-	-	600	-	-	-	-	-	-	-

Table 11

Concentrations of Polar Compounds in Sediments (G3.3+4 Fractions)

Peak # in Fig. 15	Compound	Molecular Weight	Station March 1983 1A	2A	4A	5A	12A	13A	19A	25A	31A	December 1983 21C	23C	25C	31C	33C
1	Ortho or para cresol	108	–	+	–	–	+	+	+	–	+	–	–	–	–	–
2	Benzylmethyl ketone	120	–	–	–	–	+	+	–	–	–	–	–	–	–	–
3	Meta-cresol	108	+	+	–	–	+	+	+	–	+	–	–	–	–	–
4	5,6,7,7a-tetrahydro-4,4,7a-trimethyl-2(4H)-benzofuranone	180	2.5	8.3	1.0	–	2.6	1.6	3.4	5.3	3.4	–	–	–	–	–
5	9H-fluorene-9-one	180	1.2	1.6	1.0	–	2.3	<.8	3.1	2.4	<1	–	–	6.0	–	–
6	Methyl-9H-fluorene-9-one	194	–	<1	–	–	–	–	–	–	–	–	–	–	–	–
7	Carbazole	167	<1	1.3	–	–	<.9	.8	<.8	<.7	1.7	–	–	3.7	3.1	–
	Perinaphthenone (spike)	180	–	–	–	–	+	+	+	+	+	+	+	+	+	+
8	Methyl carbazole	181	–	<1	–	–	–	–	–	–	<1	–	–	–	<1	–
9	Anthraquinone	208	12	17	0.7	4.2	6.2	.9	8.9	8.0	2.0	8.6	–	21	20	–
10	Aminonitrophenanthrene	240	–	2.2	–	–	–	–	–	–	–	–	–	–	–	–
11	Ketone from PNA mw 190	204	3.4	4.0	<.6	<1	<.9	<.8	<.8	2.1	<1	–	–	6.9	2.6	–
	1,1'Binaphthyl (standard)	254	+	+	+	+	+	+	+	+	+	+	+	+	+	+
12	Ketone from PNA mw 216	230	2.0	<1	–	1.7	<.9	–	1.3	1.0	–	–	–	3.0	3.3	–
13	Ketone from PNA mw 216	230	5.0	3.9	–	–	2.1	2.3	3.6	3.2	6.8	–	–	9.1	7.4	–
14	Benzocarbazole	217	–	–	–	<1	<.9	2.1	<.8	1.7	2.7	–	–	1.0	6.0	–
15	Ketone from PNA mw 216	230	–	–	–	–	5.2	3.1	5.4	4.4	4.4	–	–	2.5	3.5	–
16	Benzocarbazole	217	2.7	3.9	–	<1	<.9	1.0	2.2	4.6	<1	–	–	1.0	2.0	–

Numbers are approximate concentrations in ppb, + indicates that the compound was detected but not quantified.

Table 12

Aromatic Compounds Detected in Clams (G3.2 Fraction)

Compound Name	Molecular Weight	1A	1D	4B	7D
C_3-Benzene	120	-	+	-	+
C_3-Benzene	120	-	+	-	+
C_4-Benzene	134	-	+	+	+
C_4-Benzene	134	-	+	+	+
C_4-Benzene	134	-	+	+	+
C_5-Benzene	148	-	+	+	+
C_5-Benzene	148	-	-	+	-
C_4-Benzene	134	-	+	-	-
Cl_3-Benzene	180	-	+	-	-
Naphthalene	128	000	000	000	000
2-Me-Naphthalene	142	51.7	45.0	54.7	54.4
1-Me-Naphthalene	142	60.0	54.7	65.3	61.9
Biphenyl	154	100	100	100	-
C_7-Benzene	176	-	-	101.5	-
C_3-Naphthalene	170	-	106.8	-	-
C_2-Naphthalene	156	-	110.2	-	110.0
Phenylthiophene	160	-	-	112.4	-
C_2-Naphthalene	156	-	115.0	-	-
C_7-Benzene	176	-	-	117.4	-
Halogenated compound	240	119.5	119.3	-	118.9
C_7-Benzene	176	-	-	119.6	-
Unknown	208	-	122.3	-	-
C_7-Benzene	176	-	-	122.2	-
2,6-Di-t-butyl-p-quinone	220	-	124.8	-	-
Acenaphthene	154	126.7	125.9	-	127
C_8-benzene	190	-	-	126	-
Me-154	168	-	127.9	-	128.5
C_8-benzene	190	-	-	128	-
Chloro compound	-	-	127.9	-	-
Me-154	168		130.2	131.4	-
Dibenzofuran	168	134.6	134.9	138.5	-
Bibenzyl	182	137.1	-	140.4	137.6
2,6-Di-t-butyl-4-methylphenol	210	-	137.5	-	-

Table 12 (continued)

Compound Name	Molecular Weight	1A	1D	4B	7D
C_3-Naphthalene	170	-	139.5	-	-
C_3-Naphthalene	170	-	144.2	-	-
C_9-benzene	204	-	-	147.3	-
C_3-Naphthalene	170	-	148.1	-	-
Chloro compound	-	-	-	-	148.4
Fluorene	166	152.2	152.9	154.8	-
C_3-Naphthalene	170	-	154.2	-	-
C_2-154	182	-	157.5	-	157
C_9-benzene	204	-	-	158	-
C_2-154	182	-	159.4	-	160.4
Me-Dibenzofuran	182	162.6	163.6	166	-
Me-Dibenzofuran	182	165.6	-	168.1	-
C_4-Naphthalene	184	-	166.3	-	-
C_3-154/C_2-Dibenzofuran	196	167.9	167.3	-	-
C_3-154/C_2-Dibenzofuran	196	-	168.7	-	-
C_4-Naphthalene	184	-	171.2	-	-
C_9-benzene	204	-	-	173.3	-
C_3-154/C_2-Dibenzofuran	196	-	174.6	-	-
C_4-Naphthalene	184	-	177.1	-	-
C_5-Naphthalene	198	-	179.7	-	-
C_{10}-benzene	218	-	-	180.2	-
C_4-Naphthalene	184	-	181.8	-	-
C_{11}-benzene	232	-	-	181.7	-
Me-Fluorene	180	-	183.9	-	-
C_5-Naphthalene	198	-	186.3	-	-
C_2-Dibenzofuran	196	-	186.3	-	-
C_4-Naphthalene	184	-	191.3	-	-
C_2-Dibenzofuran	196	-	192.7	-	-
Dibenzothiophene	184	193.1	-	-	-
C_{10}-benzene	218	-	-	195.9	-
Unknown (Base 181)	210	196.5	-	-	-
Phenanthrene	178	200	200	200	200
Anthracene	178	-	203.6	-	-
C_5-Naphthalene	198	-	205.5	-	-
C_4-154/C_3-Dibenzofuran	210	-	205.5	-	-

Table 12 (continued)

Compound Name	Molecular Weight	1A	1D	4B	7D
C_3-152/C_2-Fluorene	194	-	208.9	-	-
Unknown (Base 253)	346	-	211.0	-	-
C_4-154/C_3-Dibenzofuran	210	-	216.6	-	-
C_3-152/C_2-Fluorene	194	-	214.6	-	-
C_{11}-benzene	232	-	216.1	-	-
C_6-Naphthalene	212	-	216.8	-	-
C_3-152/C_2-Fluorene	194	-	216.8	-	-
C_4-154/C_3-Dibenzofuran	210	-	217.3	-	-
Chloro compound	256	-	217.3	-	-
C_3-152/C_2-166	194	-	217.8	-	-
Unknown (No M-15)	210	-	217.8	-	-
Me-Dibenzothiophene	198	-	225.0	-	-
C_{11}-benzene	232	-	-	225.3	-
C_4-154/C_2-Dibenzofuran	210	-	227.7	-	-
C_4-154/C_3-Dibenzofuran	210	-	229.8	-	-
C_5-Naphthalene	198	-	229.8	-	-
C_4-154/C_3-Dibenzofuran	210	-	230.8	-	-
Me-Dibenzothiophene	198	-	230.8	-	-
C_5-Naphthalene	198	-	236.1	-	-
Me-178	192	237.6	237.1	238	-
Me-178	192	238.9	239.9	-	-
C_{12}-benzene	246	-	-	240.9	-
Cyclopentaphenanthrene	190	-	243.7	242.8	-
Me-178	192	244.9	245.5	245.5	-
C_5-154/C_4-Dibenzofuran	224	-	245.5	-	-
Me-178	192	-	247.0	-	-
C_4-152/C_3-Fluorene	208	-	250.3	-	-
C_4-152/C_3-Fluorene	208	-	253.0	-	-
C_4-152/C_3-Fluorene	208	-	254.5	-	-
C_2-Dibenzothiophene	212	256.6	256.5	-	-
Chloro compound	290	-	256.5	-	-
C_2-Dibenzothiophene	212	-	262.5	-	-
C_2-Dibenzothiophene	212	-	269.7	-	-
C_2-178	206	-	275.0	-	-
C_2-178	206	280.4	279.0	-	278.3
C_2-178	206	-	282.1	-	-

Table 12 (continued)

Compound Name	Molecular Weight	1A	1D	4B	7D
C_2-178	206	-	283.4	-	-
C_3-Dibenzothiophene	226	-	283.4	-	-
Fluoranthene	202	286.2	286.2	286.1	285.4
C_3-Dibenzothiophene	226	290.0	290.2	-	289.6
Chloro compound	-	-	-	-	289.6
C_3-Dibenzothiophene	226	-	295.5	-	-
Pyrene	202	300	300	300	300
C_3-Dibenzothiophene	226	-	300	-	-
Me-Cyclopentaphenanthrene	204	-	303.5	-	-
Cl_5-Biphenyl	326	-	-	-	306.5
C_3-178	220	312.6	-	-	-
C_3-178	220	-	316.9	-	-
p,p'-DDE	-	-	-	-	324.3
Me-202	216	336.4	-	-	-
Cl_5-biphenyl	326	-	-	-	328.0
Me-202	216	343.4	-	-	-
Cl_6-biphenyl	-	-	-	-	342
Benzo(ghi)fluoranthene	226	380.1	-	-	-
Cl_6-biphenyl	360	-	-	-	356.7
Benzo(c)phenanthrene	228	381.8	-	-	-
Benzo(a)anthracene	228	396.7	-	-	-
Cl_7-biphenyl	394	-	-	-	398.9
Chrysene	228	400	400	-	-
Tetramethyloctahydrochrysene	292	400	400	-	-
Benzo(e)pyrene	252	491.2	-	-	-
Perylene	252	500	-	-	-

Numbers are ARI's and + indicates that the compound was detected but no ARI is defined.

Table 13

Polar Compounds Detected in Clams (G3.3+4 fraction)

Compound	Molecular Weight	Sample								
		1A	1D	4B	6A	7D	1B4	4B2	6B4	7B2
Ortho or para cresol	108	+	-	-	+	-	-	+	+	-
Phenylmethyl ketone	120	-	+	-	-	+	-	+	-	-
1 phenyl-1,2-propanedione	148	6.2	10	-	-	15	15	4.2	22	8.2
Dibenzylamine	197	-	-	-	-	-	-	19. 1	-	-
Carbazole	167	-	-	22	47	-	-	-	-	-
Perinaphthenone (std)	180	+	+	+	+	+	+	+	+	+
C_2-Carbazole	181	-	-	-	4.8	-	-	-	-	-

Numbers are approximate concentrations in ppb, and + indicates that the compound was detected but not quantified.

Table 14

Polar Compounds Detected in Effluent Samples (G3.3+4 Fractions)

Compound	Molecular Weight	Sample #1	Sample #2
C_2-Aniline	121	−	+
Phenol	94	+	−
$C_6H_{10}O$	98	+	−
Ortho cresol/p-cresol	108	+	−
Me-aniline	107	+	−
m-cresol	108	+	−
C_2-phenol	122	+	−
C_2-phenol	122	+	−
C_2-phenol	122	+	−
C_2-phenol	122	+	−
C_2-phenol	122	+	−
C_2-phenol	122	+	−
C_3-phenol	136	+	−
C_3-phenol	136	+	−
C_3-phenol	136	+	−
C_3-phenol	136	+	−
C_3-phenol	136	+	−
1,3,5 Trithiane	138	+	−
C_4-phenol	150	+	−
Methylsulfonylbeneze	156		2.0
1-methyl-4-methylsulfonylbeneze	170		8.0
(methylsulfonyl)methylbeneze	170		2.4
Carbazole	167	2.2	−

Table 14 (continued)

Compound	Molecular Weight	Sample #1	Sample #2
α-phenylbenezemethanol	184	-	2.5
Me-carbazole	181	2.0	-
Me-carbazole	181	0.8	-
Me-carbazole	181	0.9	-
Me-carbazole	181	1.1	-
C_2-carbazole	195	0.5	-
C_2-carbazole	195	0.5	-
C_2-carbazole	195	0.5	-
C_2-carbazole	195	0.5	-
C_2-carbazole	195	1.1	-
C_2-carbazole	195	0.8	-
C_2-carbazole	195	0.5	-
C_2-carbazole	209	0.2	-
C_3-carbazole	209	0.5	-
C_3-carbazole	209	0.2	-
C_3-carbazole	209	0.4	-
C_3-carbazole	209	0.4	-
C_3-carbazole	209	0.4	-
C_4-carbazole	223	0.4	-
C_4-carbazole	223	0.3	-
C_4-carbazole	223	0.2	-

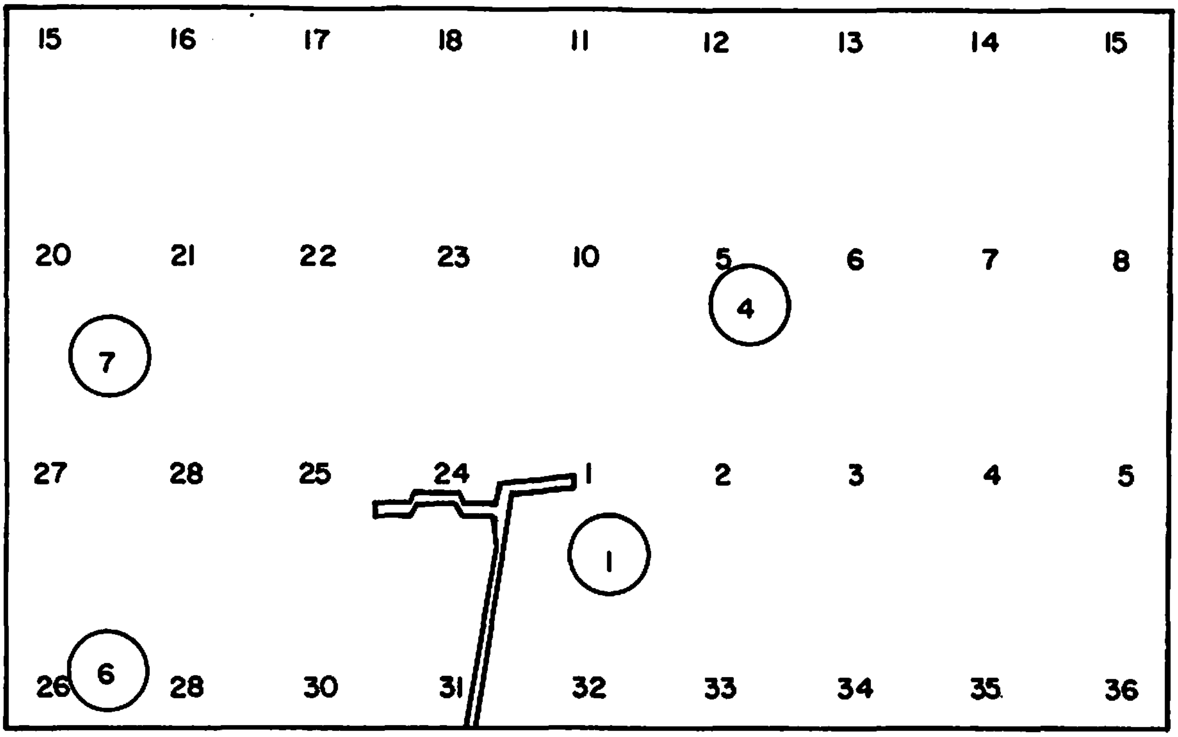
Numbers are approximate concentrations in ppb, + indicates that the compound was detected but not quantified.

Table 15

Total Aromatic Compounds Detected in Effluent Samples

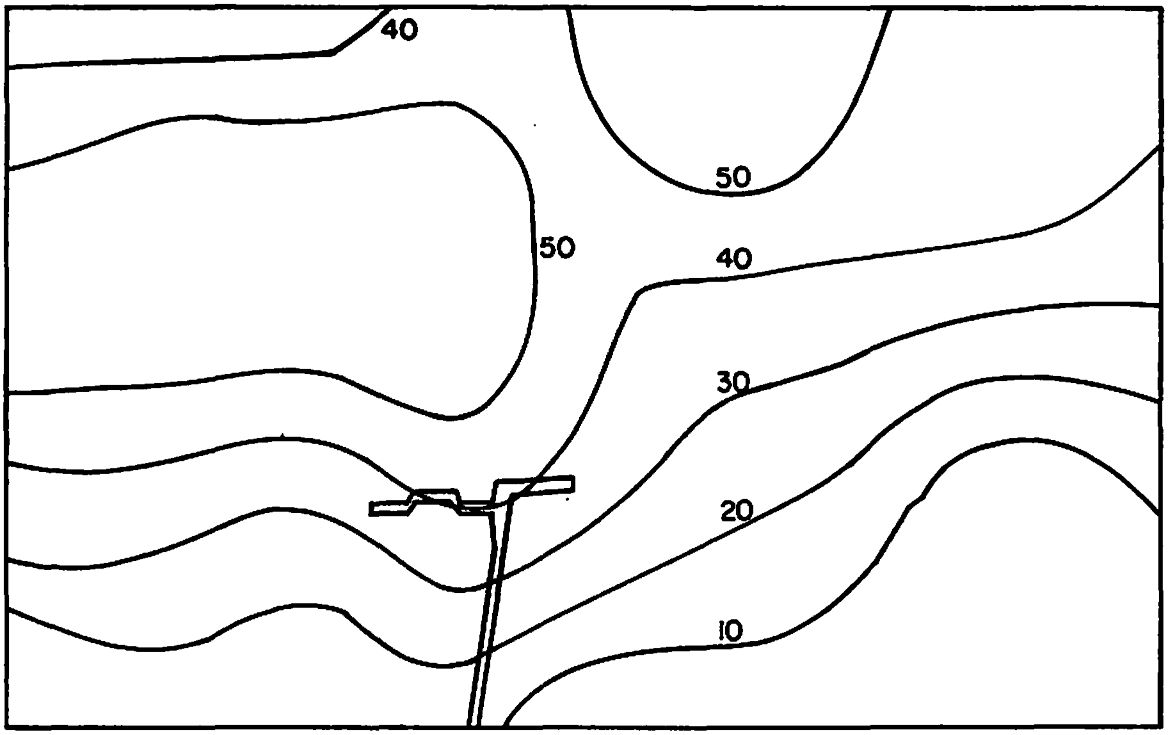
Fraction	Resolved(ppm)		Unresolved(ppm)		Resolved+UCM		Total (ppm)	
	#1	#2	#1	#2	#1	#2	#1	#2
B/N(aromatic)	0.9	0.1	1.1	0.4	2.0	0.5	6.3	1.8
Acid	0.8	0.1	1.9	1.2	2.7	1.3		
Volatile	1.6	-	0	-	1.6	-		

Figure 1



STATION LOCATIONS

Circles are clam sampling stations, other numbers are sediment stations.

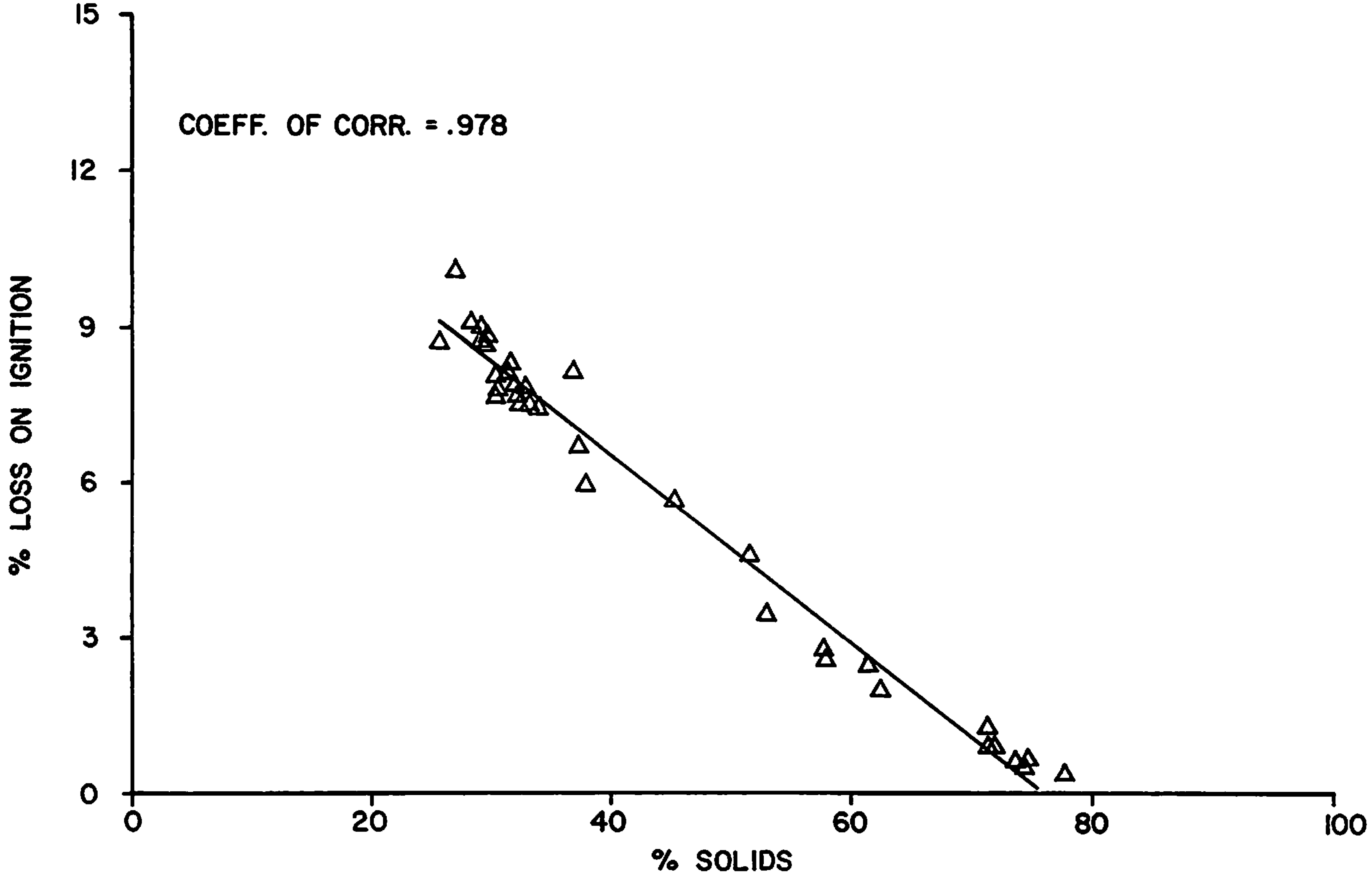


BOTTOM TOPOGRAPHY

Contours are labeled in feet.

Figure 2

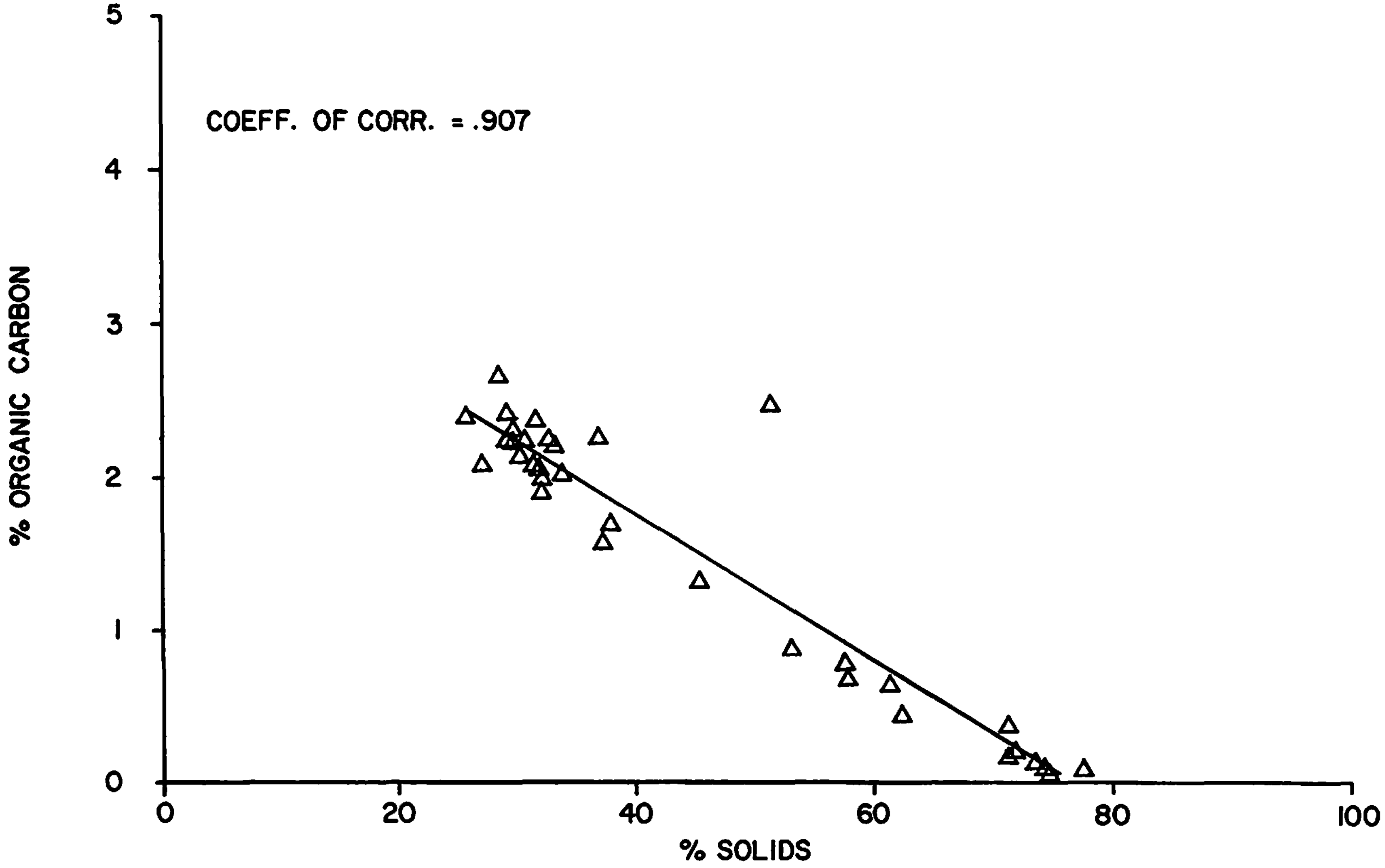
Correlation between loss on ignition and percent solids



Sediment samples collected in March, 1983.

Figure 3

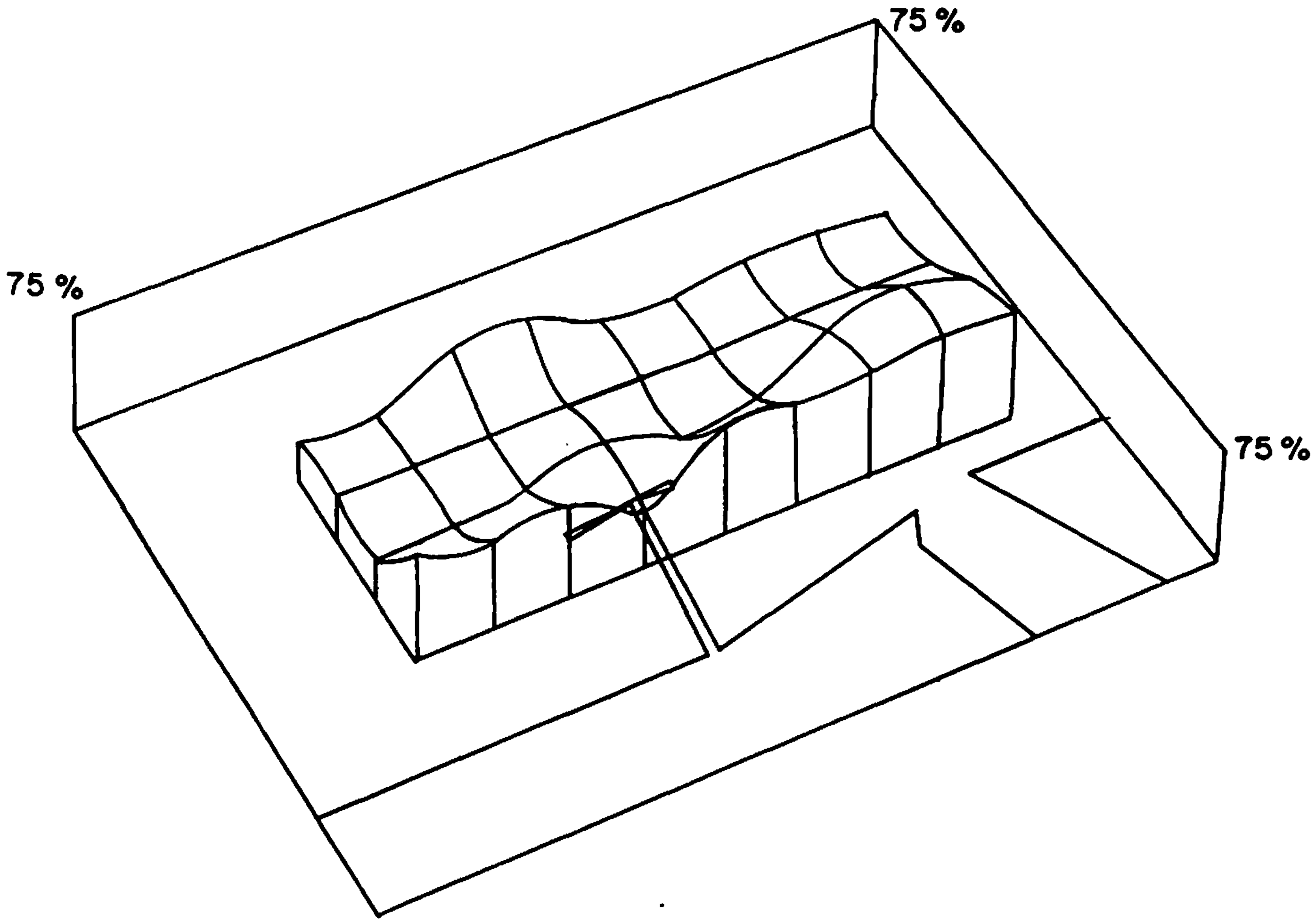
Correlation between organic carbon content and percent solids



Sediment samples collected in March, 1983.

Figure 4

Percent solids in sediments collected in March 1983

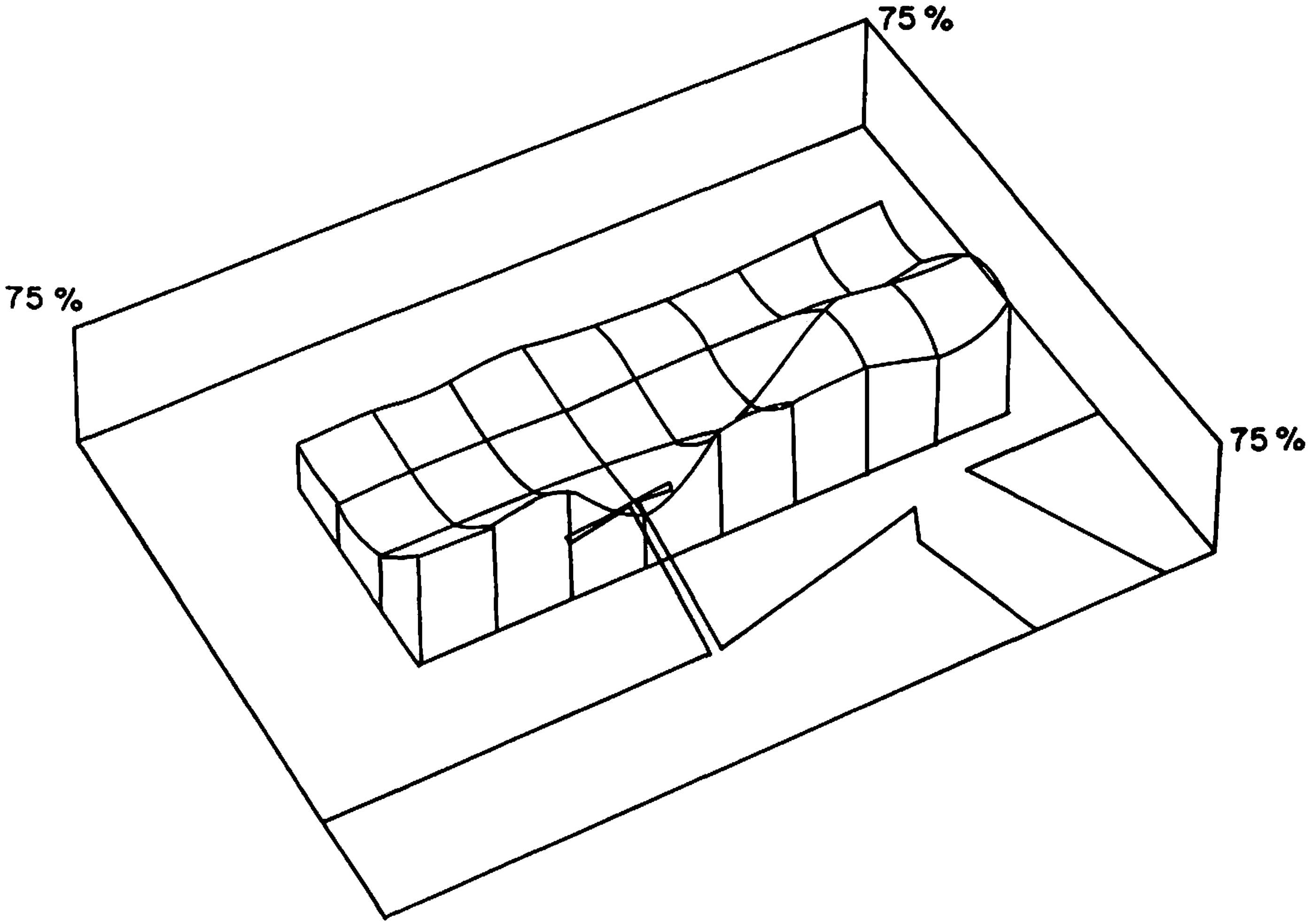


% SOLIDS

Percent solids was measured in sediments collected at the nodes of the grid shown.

Figure 5

Percent solids in sediments collected in December, 1983

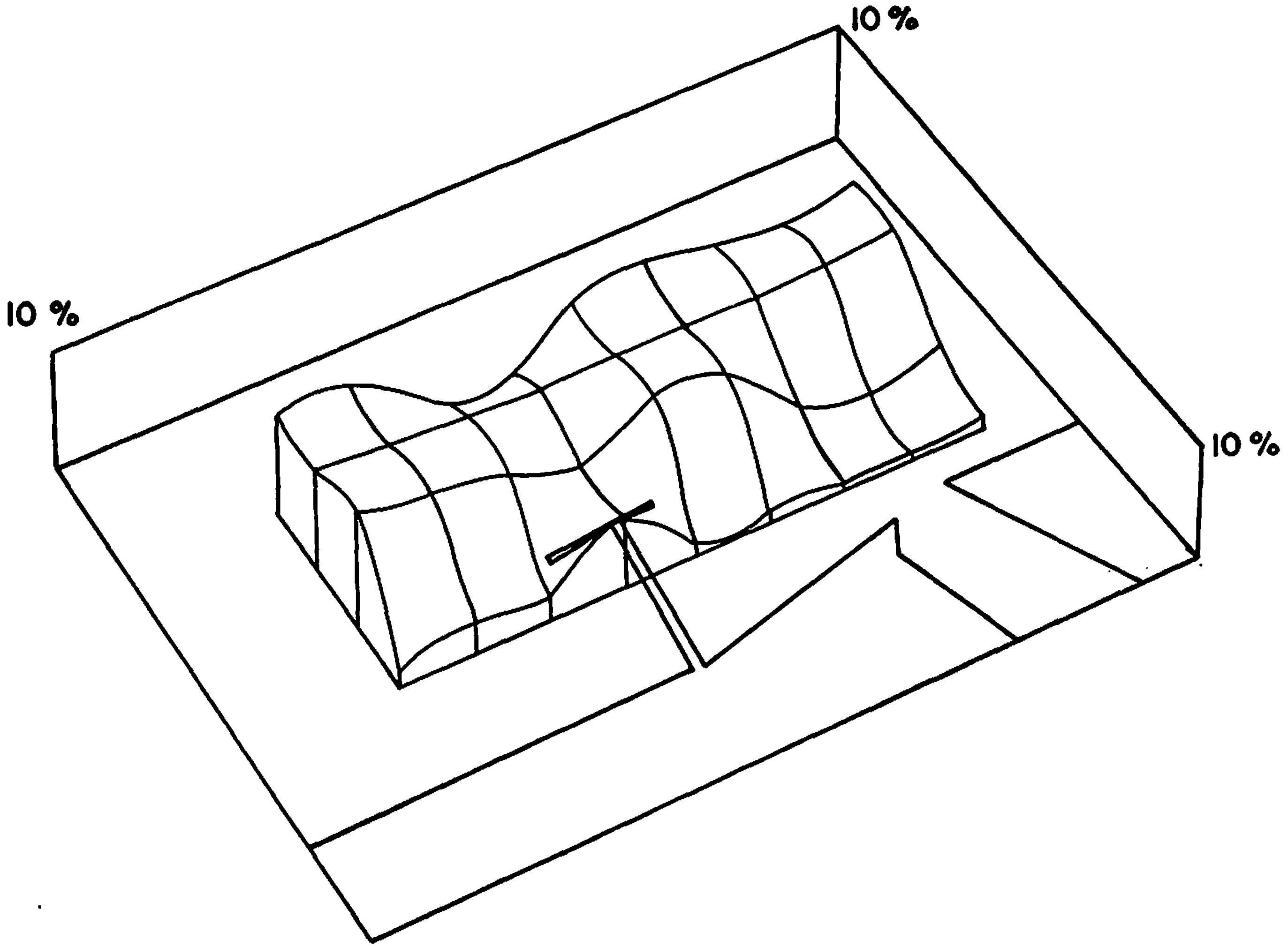


% SOLIDS

Percent solids was measured in sediments collected at the nodes of the grid shown.

Figure 6

Loss on ignition in sediments collected in March, 1983

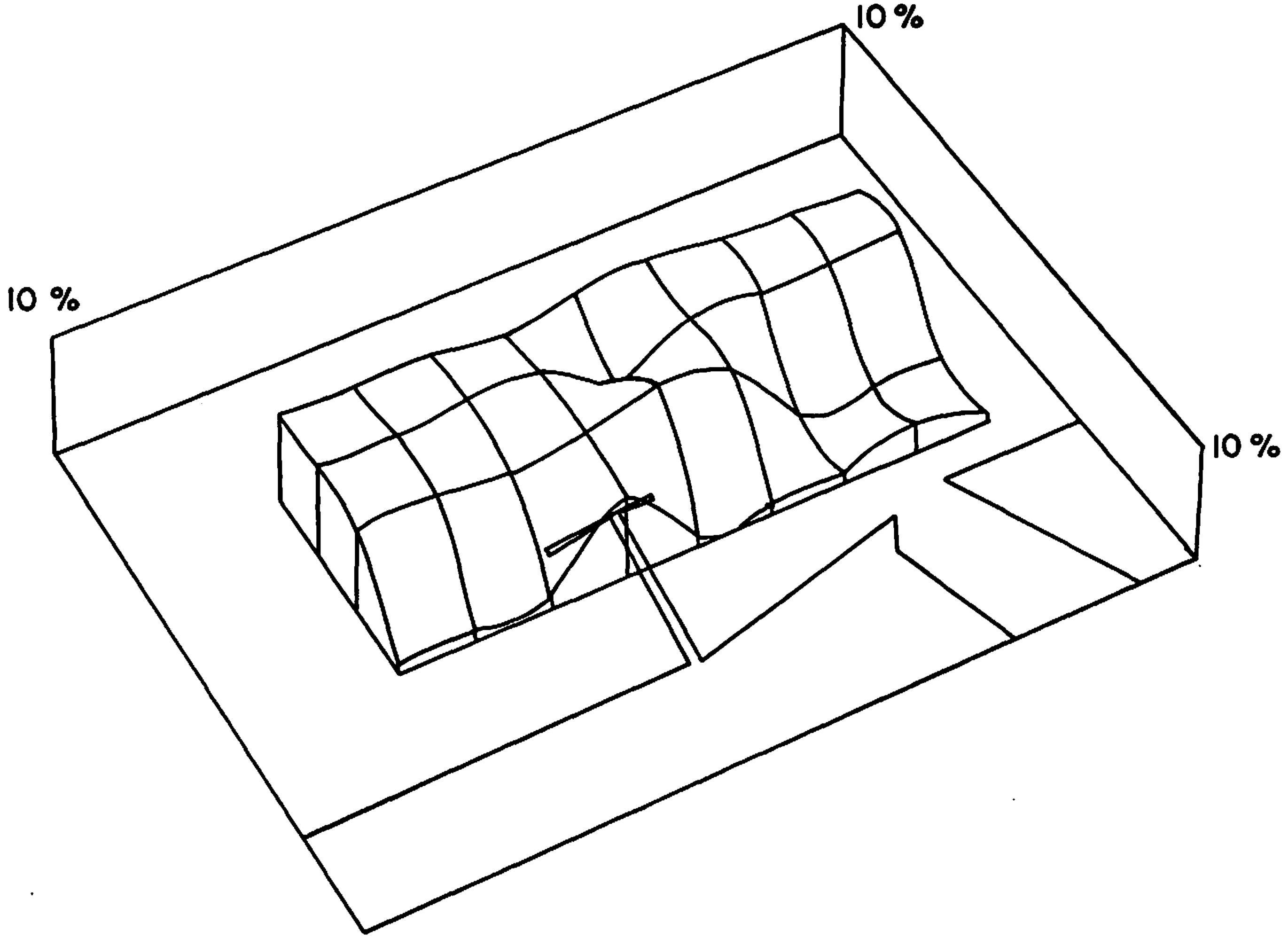


% LOSS ON IGNITION

Loss on ignition was measured in sediments collected at the nodes of the grid shown.

Figure 7

Loss on ignition in sediments collected in December 1983

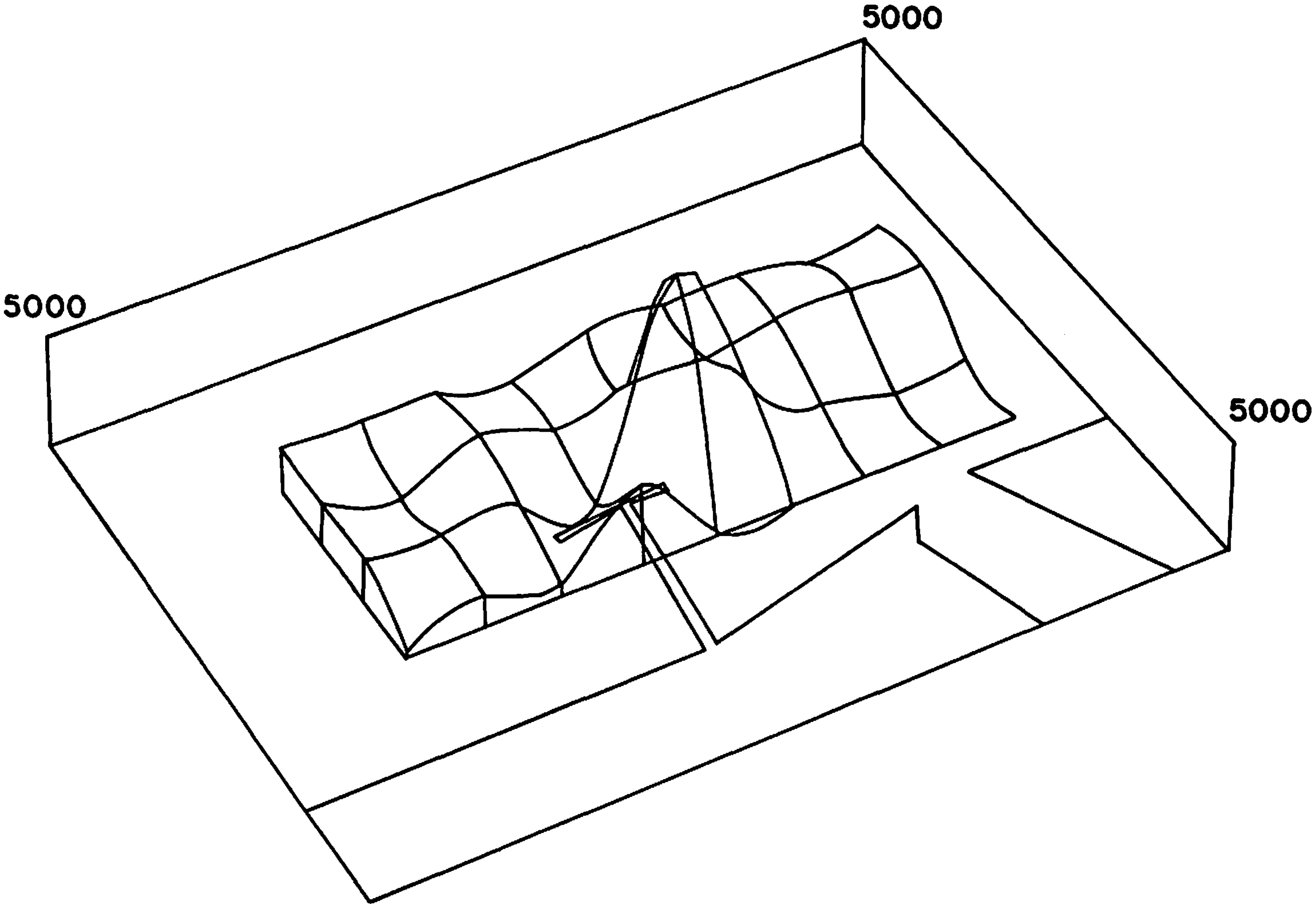


% LOSS ON IGNITION

Loss on ignition was measured in sediments collected at the nodes of the grid shown.

Figure 8

Total resolved PAH in sediments collected in March 1983

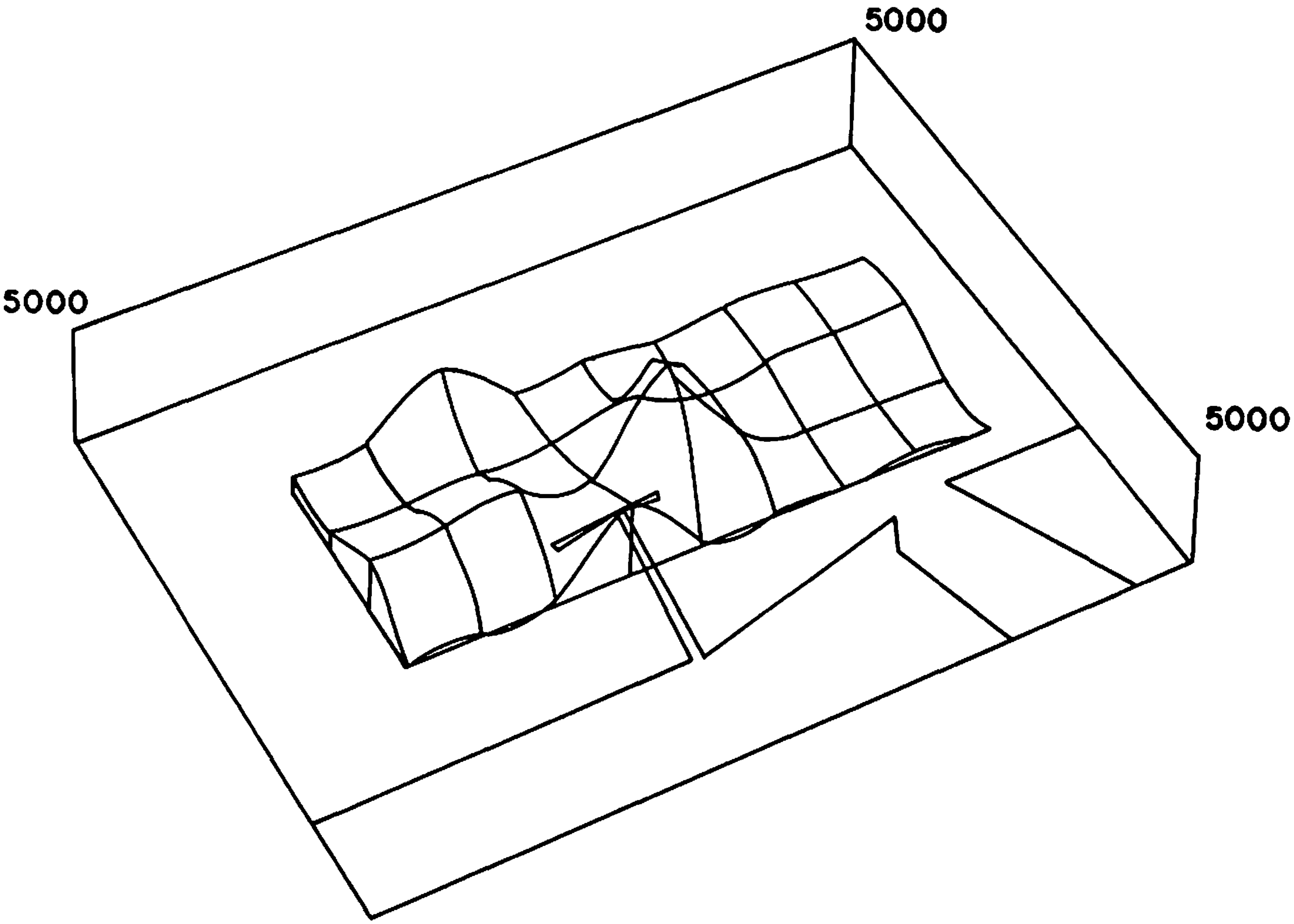


TOTAL RESOLVED PAH (PPB): 3-83

PAH concentrations were measured in sediments collected at the nodes of the grid shown.

Figure 9

Total resolved PAH in sediments collected in December, 1983

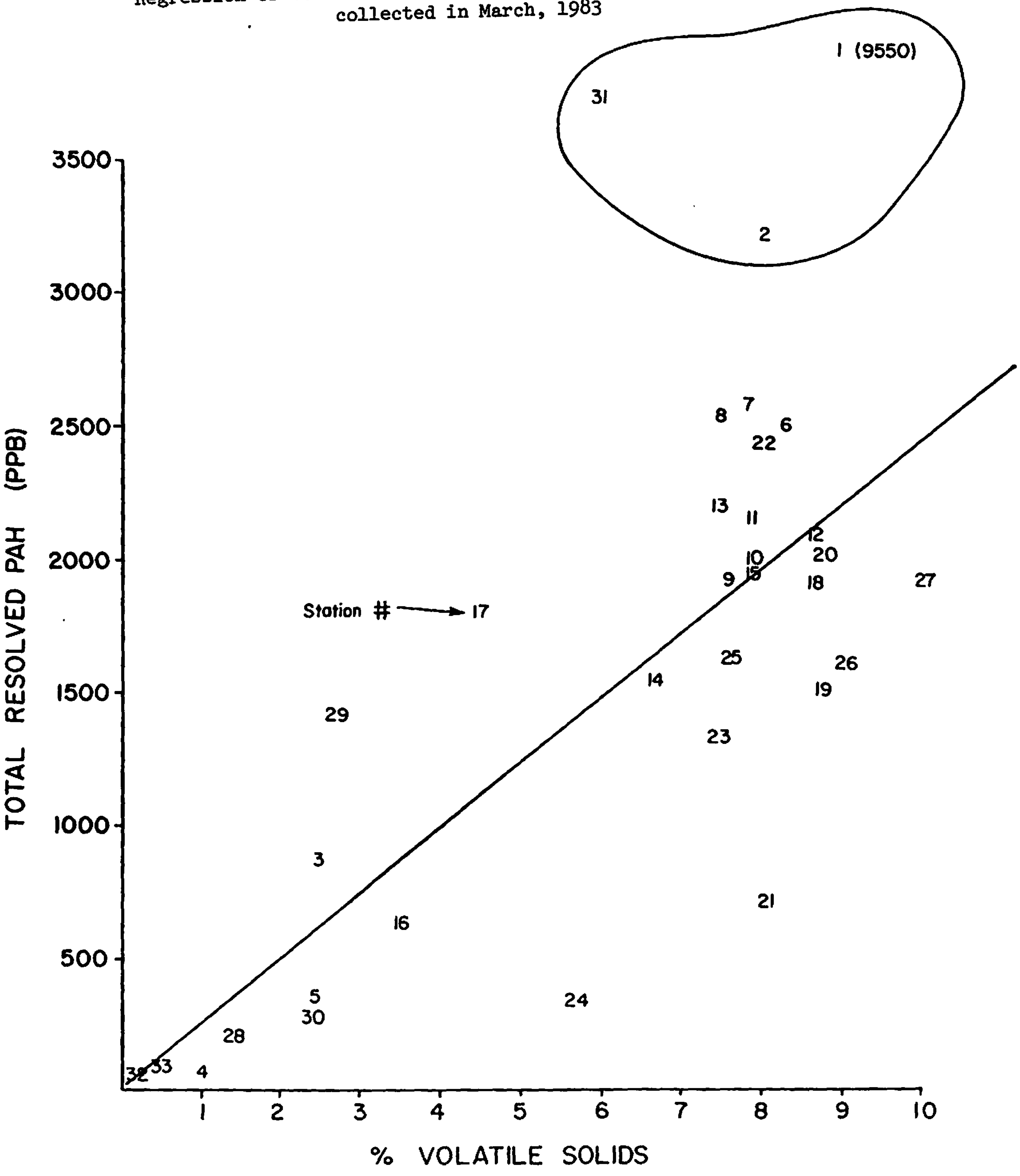


TOTAL RESOLVED PAH (PPB)

PAH concentrations were measured in sediments collected at the nodes of the grid shown.

Figure 10

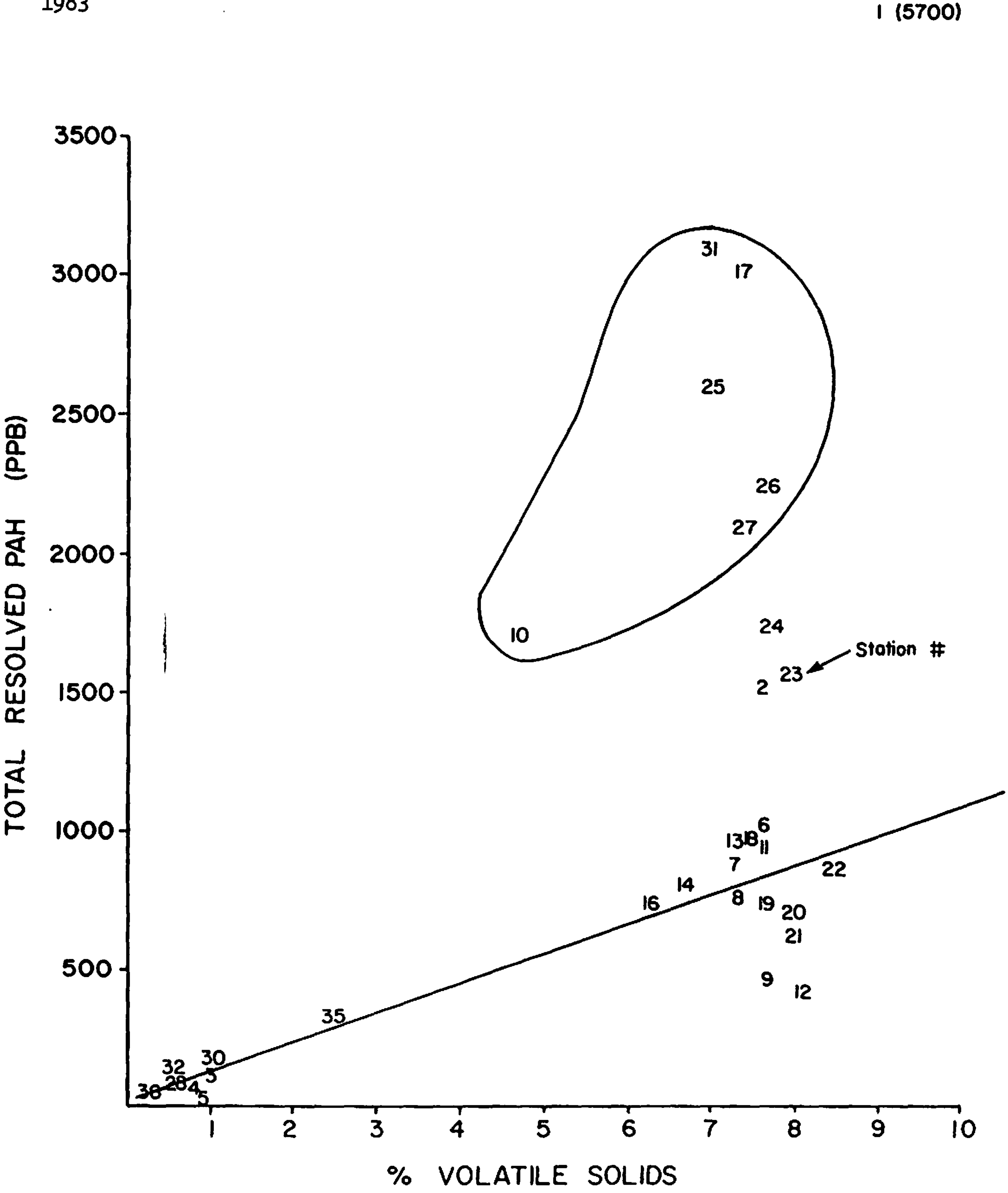
Regression of total PAH against volatile solids for sediments collected in March, 1983



Stations off the regression line are circled.

Figure 11

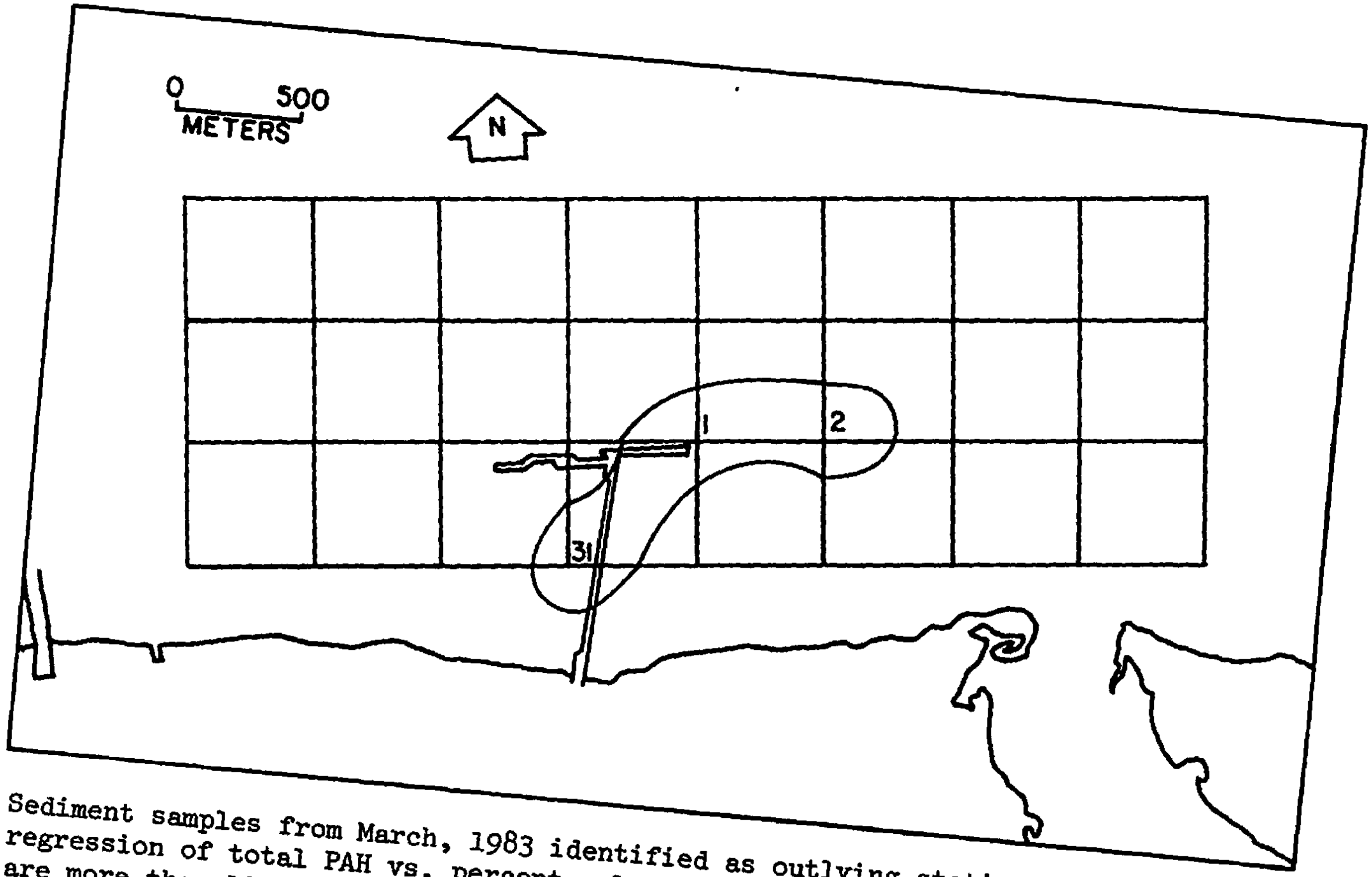
Regression of total PAH against volatile solids for sediments collected in December, 1983



Stations off the regression line are circled.

Figure 12

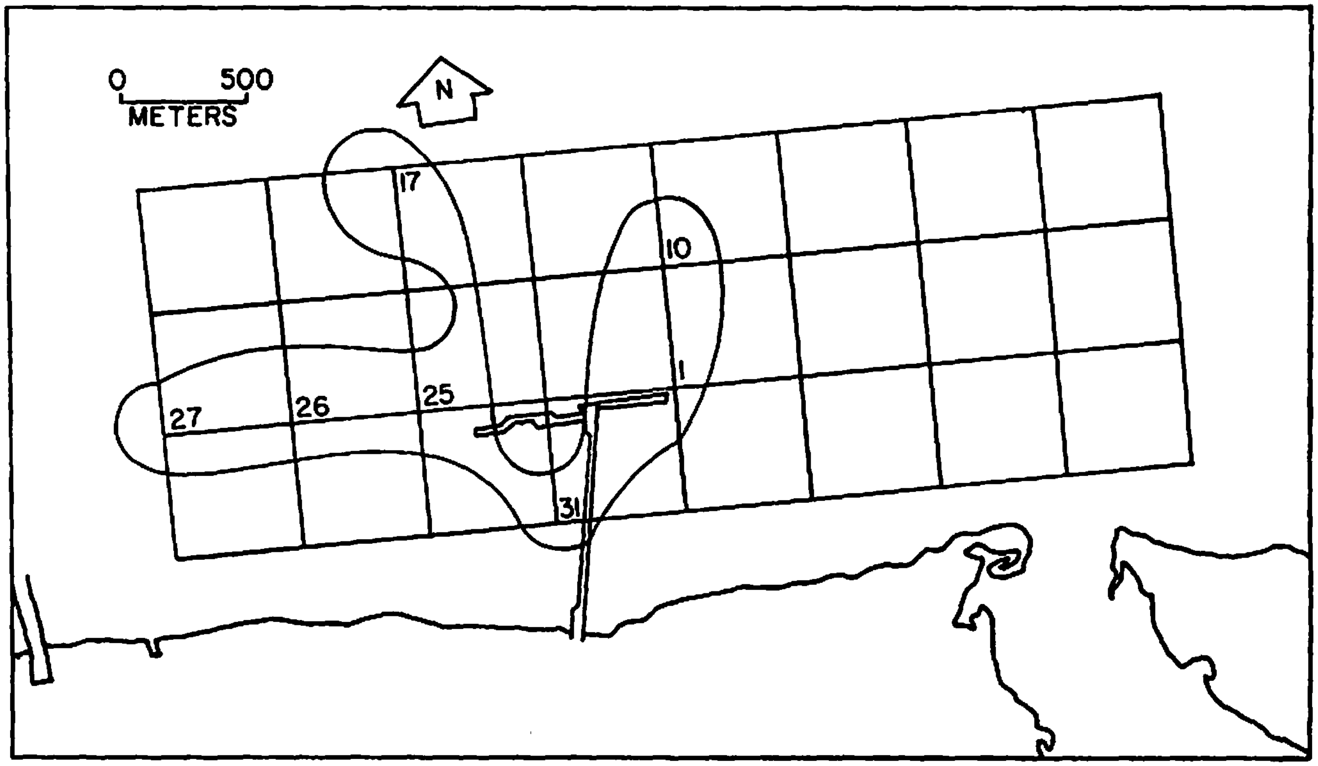
Stations off of regression line of Figure 10



Sediment samples from March, 1983 identified as outlying stations from the regression of total PAH vs. percent volatile solids. The stations circled are more than 1200 ppb above the regression line.

Figure 13

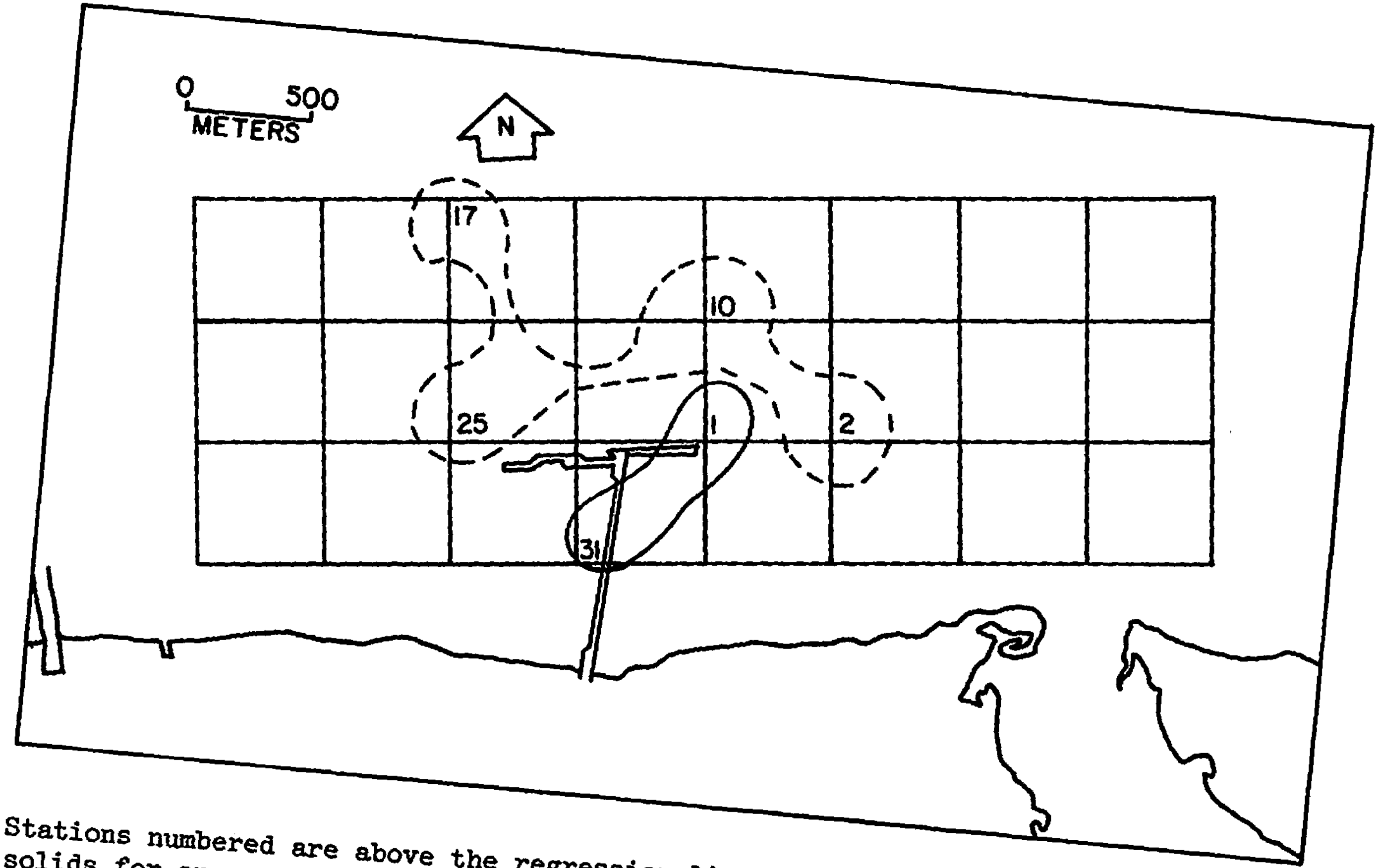
Stations off of regression line of Figure 11



Sediment samples from December 1983 identified as outlying stations from the regression of total PAH vs. percent volatile solids. The stations circled are more than 1200 ppb above the regression line.

Figure 14

Sediment station outliers for the mean of March and December



Stations numbered are above the regression line of total PAH vs. percent volatile solids for an average of the two samplings. Stations inside the dotted line are 500 ppb to 2000 ppb above the regression line and stations inside the solid line are more than 2000 ppb above the regression line.

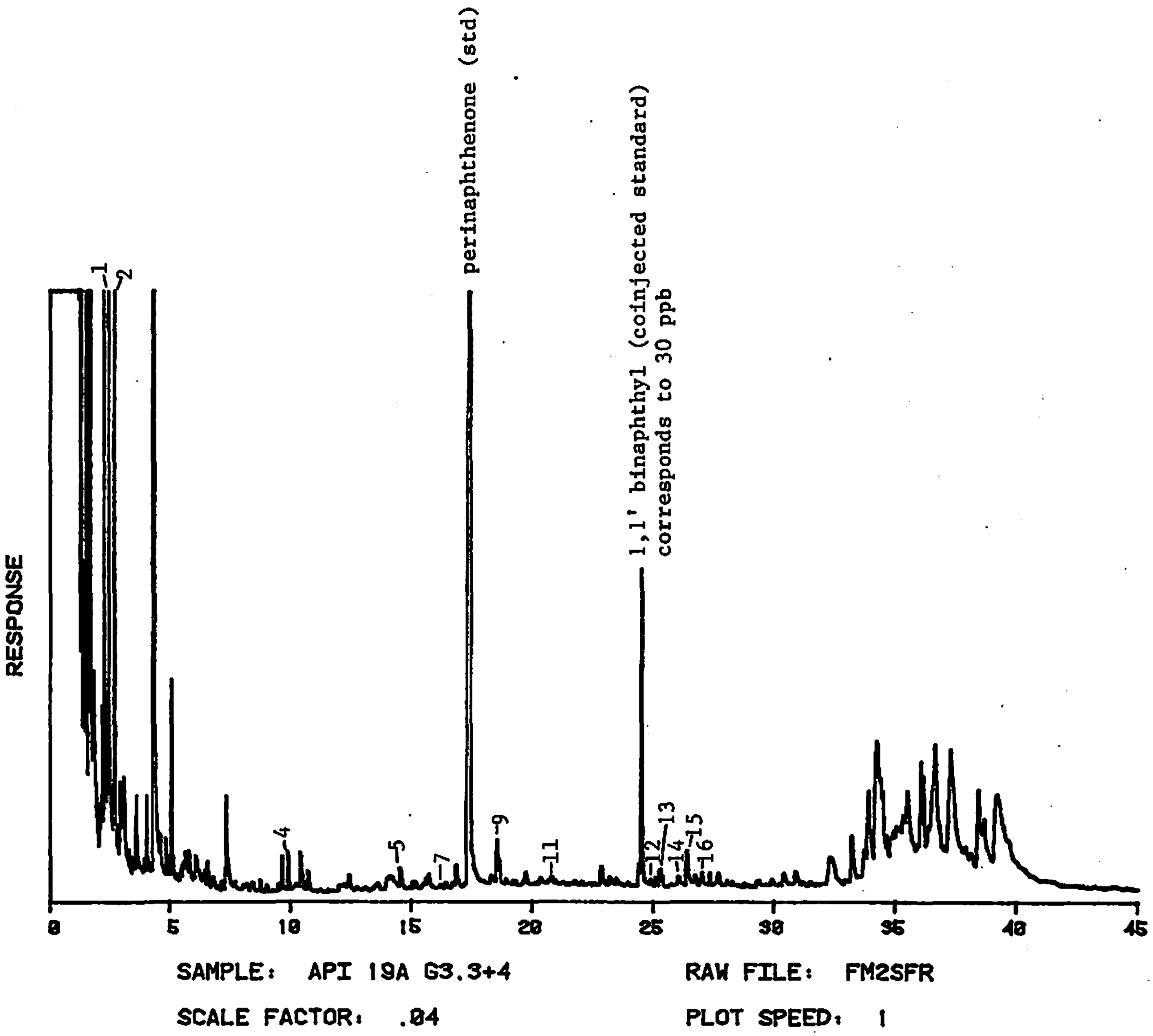


Figure 15

Representative sediment G3.3+4 fraction. Peak identifications are in Table 10, unlabeled peaks are unknowns.

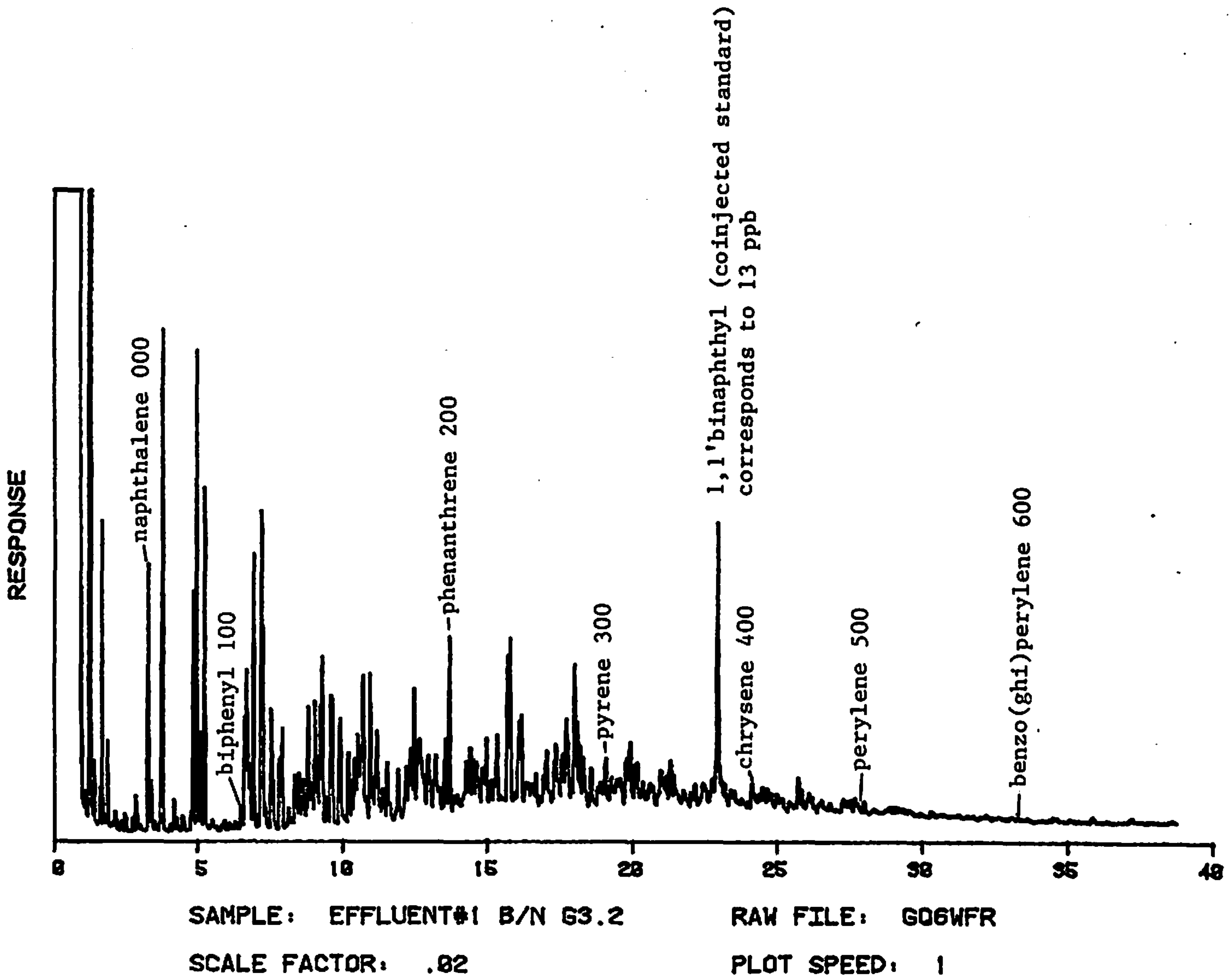


Figure 16

Base /neutral extract of effluent #1. Numbered peaks are used to calculate retention indices for identification of other peaks.

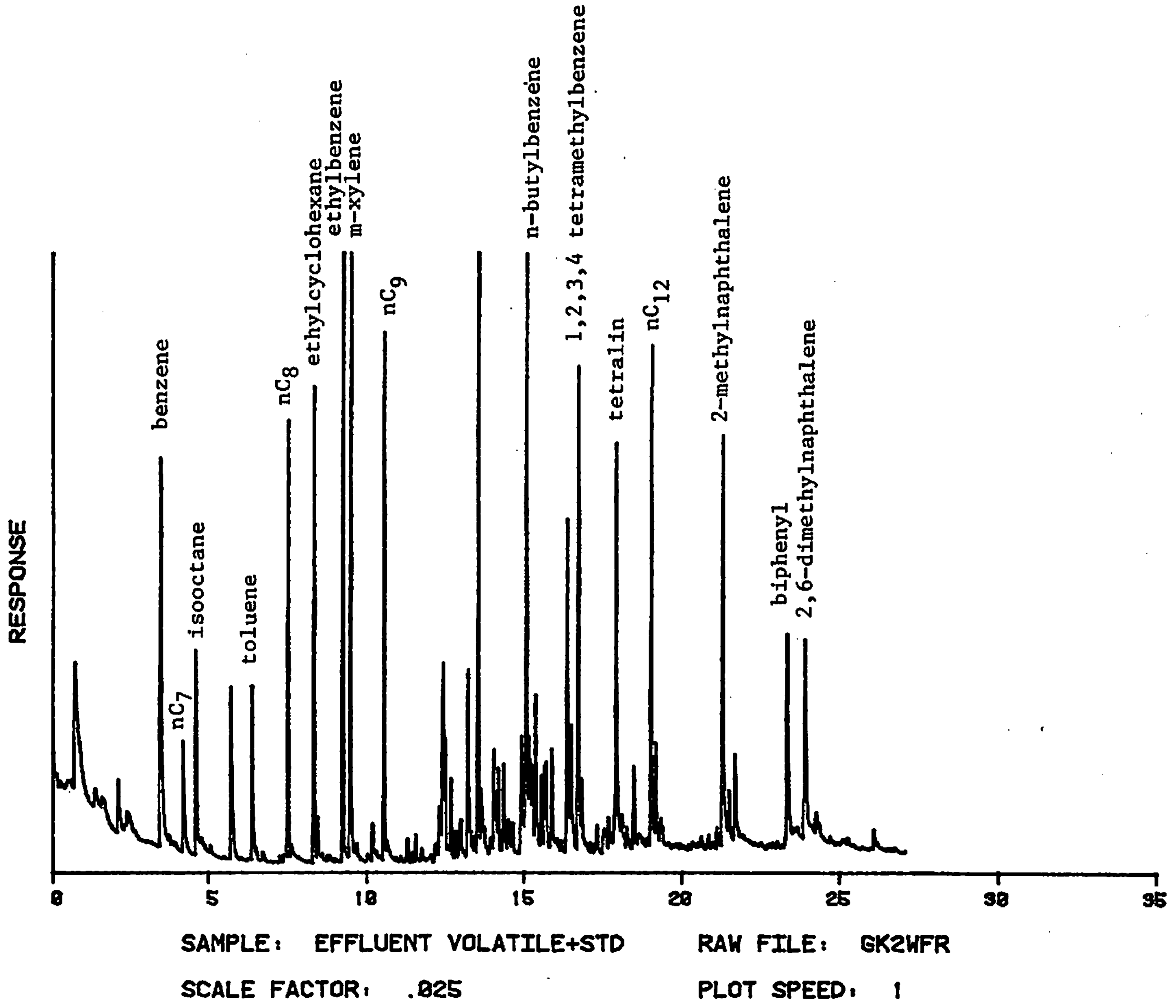


Figure 17

Volatile compounds in effluent #1. The labeled peaks are standards added for retention identifications.

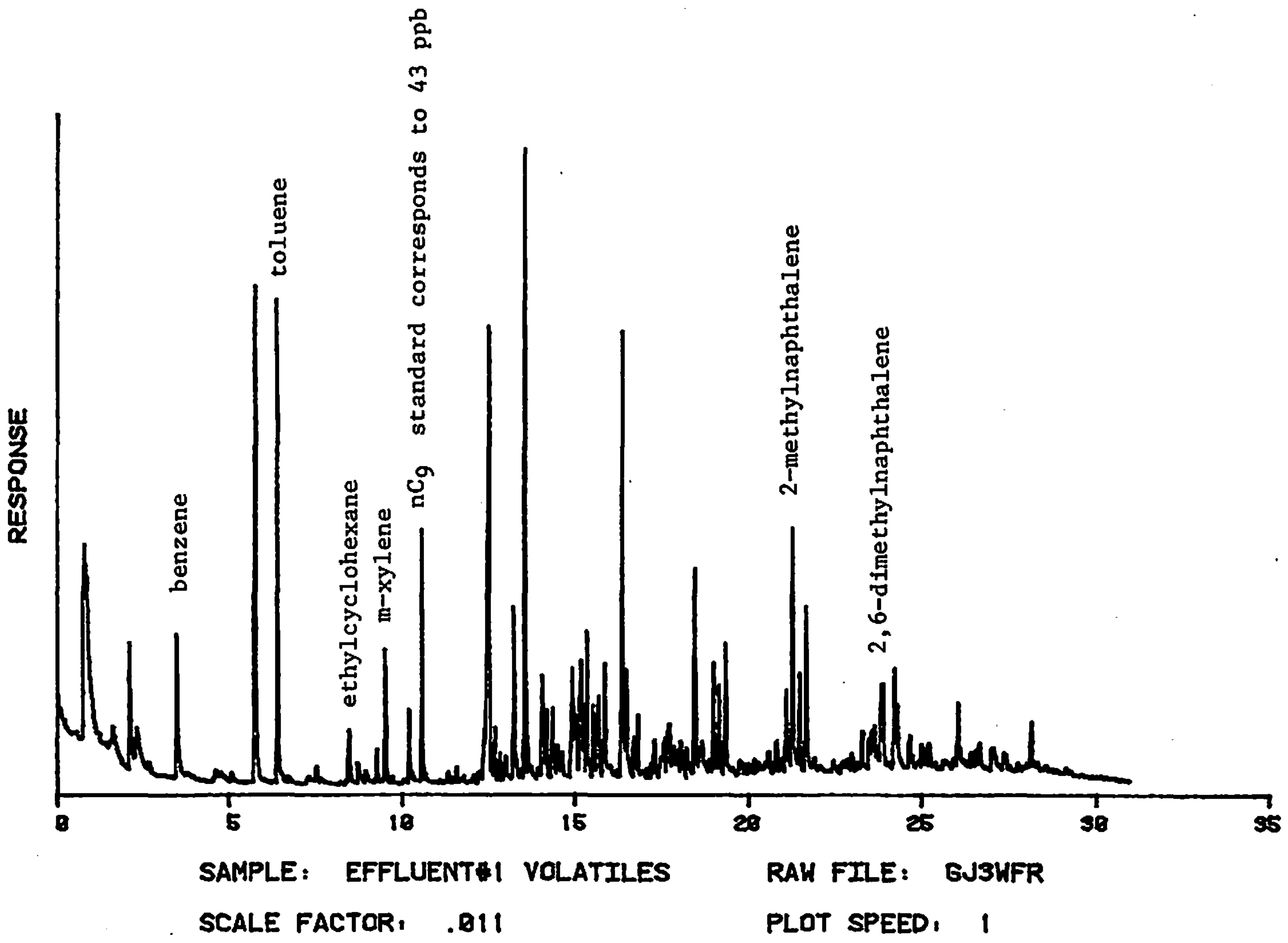


Figure 18

Volatile compounds in effluent #1. Labeled peaks have been identified by comparison with Figure 17.

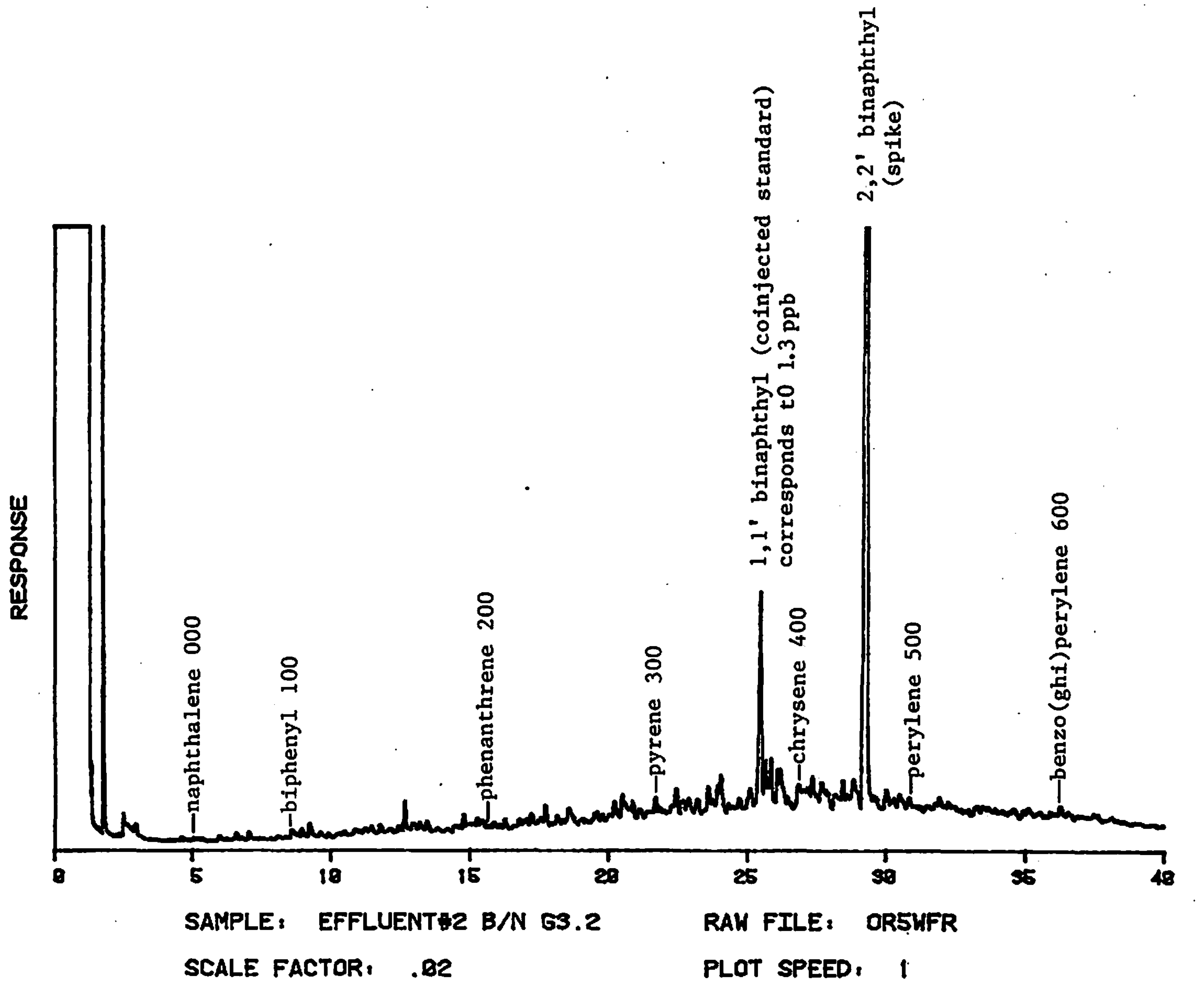


Figure 19

Base/neutral extract of effluent #2 to illustrate the difference with effluent #1. Most of the marker peaks are not present.