AN ABSTRACT OF THE THESIS OF

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Title: An Evaluation of Two Methods for Determinations of

Polynuclear Aromatic Hydrocarbons in Water

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Research has been completed that evaluates two methods for the extraction of polynuclear aromatic hydrocarbons (PNAH) from water. The two methods compared were EPA Method 610, a liquid-liquid extraction method, and a method involving PNAH adsorption on commercially available short C_{18} bonded phase columns, Sep-Paks, in series with glass microfiber filters. Eleven PNAH were found to be present in water samples from Yaquina Bay, Oregon at four sites. Concentrations of the eleven PNAH ranged from 0.1 pg/mL to 16.8 pg/mL depending on the method of extraction. EPA Method 610, the approved method for analysis of effluent waters, will underestimate the concentration of environmental levels of PNAH in natural waters. This can be explained by the tendency for PNAH to be primarily associated with organic particulates in the water sample. The extraction efficiency for Method 610 has been reported to be close to 100% for PNAH in solution. However, this method is not as

efficient for particulate-bound PNAH. Adsorption on Sep-Pak minicolumns will also underestimate environmental levels of PNAH due to column overloading and irreversible adsorption. High octanol/water partition coefficients of PNAH suggest that they will be primarily associated with organic particulates, not present in the soluble form. Therefore, removal of the particulates by filtration with subsequent extraction of the filters by the Soxhlet method may provide the best estimate of PNAH concentrations in natural waters. Such a method is relatively fast, simple and inexpensive method for routine environmental monitoring.

An Evaluation of Two Methods for Determinations of Polynuclear Aromatic Hydrocarbons in Water

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TABLE OF CONTENTS

AN EVALUATION OF TWO METHODS FOR DETERMINATIONS OF POLYNUCLEAR AROMATIC HYDROCARBONS IN WATER

Introduction 1

Experimental 8

Preliminary Studies 8

Method 610 Sample Preparation 10

Sep-Pak Method Sample Preparation 11

HPLC Analysis 13

Results and discussion 15

Literature Cited 27

BIBLIOGRAPHY 30

APPENDIX A 33

LIST OF FIGURES

Figure		Page
1	Yaquina Bay, Oregon showing four sampling sites.	9
2	Sep-Pak method sampling apparatus.	12
3	Sample chromatograms for UV detector.	16
4	Sample chromatograms for fluorescence detector.	17

LIST OF TABLES

Table		Page
I	IUPAC name, structure, physical properties, biological activity, and legal status of 11 polynuclear aromatic hydrocarbons found in water samples from Yaquina Bay, Oregon.	2
II	HPLC specifications and conditions.	14
III	PNAH concentrations in water at four sites, Yaquina Bay, Oregon, determined by EPA Method 610, 6/16/82.	18
IV	PNAH concentrations in water at four sites, Yaquina Bay, Oregon determined by EPA Method 610, 6/17/82	19
V	PNAH concentration (ng/L) at four sites, Yaquina Bay, Oregon determined by two methods	20
VI	Sep-Pak recovery determination-known amounts of PNAH standards added to one liter of artificial seawater.	24

AN EVALUATION OF TWO METHODS FOR DETERMINATIONS OF POLYNUCLEAR AROMATIC HYDROCARBONS IN WATER

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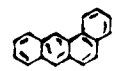
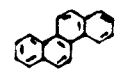
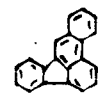
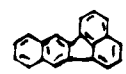
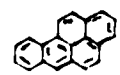
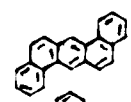
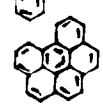
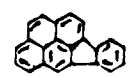
INTRODUCTION

Polynuclear aromatic hydrocarbons (PNAH)(see Appendix A for a list of abbreviations used in this report) are ubiquitous environmental contaminants (1). Their presence in the environment is cause for concern because of their demonstrated carcinogenic or mutagenic properties (2,3). In 1971 the World Health Organization set an upper limit of 200 ng/L for the total concentration of fluoranthene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(a)pyrene, benzo(ghi)perylene, and indeno(1,2,3-cd)pyrene in drinking water (4).

In 1976 the U.S. Environmental Protection Agency (EPA) was sued by the National Resources Defence Council for failing to implement portions of the Federal Water Pollution Control Act (P.L. 92-500) that pertained to the publication of a list of toxic pollutants for which an effluent standard was to be established. As a result of that suit the EPA published a list of pollutants that were to be monitored and limited in effluent waters. This "priority pollutant" list contained 16 PNAH (5,6). Table I reviews structures, relative carcinogenicity, water solubility, and the

Table I. IUPAC name, structure, physical properties, biological activity, and legal status of 11 polynuclear aromatic hydrocarbons found in water samples from Yaquina Bay Oregon.

Name	Structure	K_{ow} [1]	S (mg/l) [2]	Carc. [3]	Priority [4]
Fluoranthene			0.26	-	*
Pyrene		1.2×10^5	0.135	-	*
Benzo(a)anthracene		3.9×10^5	0.01	+	*
Chrysene			0.002	+	*
Benzo(b)fluoranthene				++	*
Benzo(k)fluoranthene				-	*
Benzo(a)pyrene		1.1×10^6	0.0038	+++	*
Dibenz(ah)anthracene		3.2×10^6	0.0002	+++	*
Benzo(ghi)perylene			0.0026	-	*
Indeno(1,2,3-cd)pyrene				+	*
Coronene			0.00014		

1 K_{ow} - Partition coefficient octanol/water ref. 8

2 S - Water solubility ref. 8

3 Carc. - Carcinogenicity ref. 2

4 Priority - EPA priority pollutant status ref. 6

classification of selected PNAH.

The concentrations of PNAH in water are usually so low that a preconcentration step, prior to determination, is required. Two preconcentration methods that were used in the present study to extract PNAH from water are liquid-liquid extraction and adsorption on short, C_{18} bonded phase columns. Both methods have certain advantages and disadvantages.

Liquid-Liquid Extraction

The efficiency of liquid-liquid extraction is dependent upon the distribution of PNAH between the organic extracting solvent and the water. Unsubstituted PNAH all have high partition coefficients (K) for organic solvents such as octanol, dichloromethane (DCM), and hexane. For example, the K value for phenanthrene for octanol/water (Kow) is 2.8×10^4 (7) and the Kow for coronene is estimated to be 5.7×10^8 (8). The theoretical extraction efficiency is based on the K value and can be computed by this relationship:

$$\% \text{ extraction} = \frac{100 \text{ K Vo}}{\text{Vw} + \text{K Vo}}$$

where Vo and Vw are the volumes of the organic and the water phases respectively, and K is the empirically derived partitiion coefficient.

Liquid-liquid extraction has been used by several investigators for extracting PNAH from water (9,10,11). EPA Method 610, developed for analyzing PNAH in water samples, calls for

liquid-liquid extraction with dichloromethane (DCM) (5). In 1979, Wilkinson et al. (12), determined extraction efficiencies for the 16 PNAH classified as priority pollutants. They determined that DCM was more efficient for extracting PNAH from water than a mixture of DCM and hexane (3:17 v/v). The average recovery for the 16 PNAH was 100 +/- 1% at pH 7 (12). This study led to the adoption of DCM as the extracting solvent for EPA Method 610.

Liquid-liquid extraction is a reproducible and highly efficient method for extracting unbound (soluble, micelle or crystalline) PNAH from water (13). However, the method has limitations when many sites or remote sites are to be sampled or when the sample contains organic particulates. Usually one or more liters of water need to be extracted to obtain the levels necessary for determination. Thus, large amounts of water would require transportation back to a lab, which may not be practical. Another problem associated with the transport of water samples occurs if the PNAH adsorb onto the glass sample bottle. While most of the adsorbed PNAH are easily removed by rinsing the bottle with DCM, this step, if omitted, could lead to large errors in measuring PNAH concentrations. For example, Basu and Saxena (14) found a loss of 77.4% of benzo(ghi)perylene attributable to adsorption within the glass sample bottle. Other PNAH also had significant adsorptive losses: benzo(a)pyrene, 63.6%; benzo(j)fluoranthene, 51.7%; indeno(1,2,3-cd)pyrene, 74.5%; and, fluoranthene, 44.3% (14). Unfortunately, those authors did not state the length of time that

the water samples were held in the sample container.

Adsorptive losses can be minimized by adding an organic solvent to the sample immediately after collection; however, information on the amounts of PNAH adsorbed to particulate matter in the water cannot then be obtained. Primarily because of difficulties associated with the transport of large volumes of water, other extraction methods have been preferred.

Adsorption on Short C_{18} Bonded Phase Columns

The efficient use of short C_{18} bonded phase columns for extracting PNAH from water depends, in theory, on the K values of the individual PNAH on to the bonded C_{18} material. Adsorption of individual PNAH on bonded C_{18} increases as an inverse function of their water solubility (15). The higher the K value of the PNAH the greater its affinity for the column packing material. In theory, all the organic compounds in the water sample, with high K values, should be concentrated at the head of the C_{18} column. That should lead to excellent extraction efficiencies, and would also constitute a cleanup step, if the process occurred as predicted theoretically.

In 1974, Kirkland (16) proposed the use of short C_{18} bonded phase precolumns to preconcentrate organics from water. Euston and Baker, (17) subsequently developed a two step process. First the water sample was forced through a short pellicular C_{18} column using a high performance liquid chromatograph (HPLC) pump to concentrate the PNAH. The flow was then reversed through the column

and the PNAH backflushed onto an analytical column for analysis (17).

Waters Associates Inc. began marketing a short C_{18} bonded phase column under the the trade name "Sep-Pak" in 1979. The column contained 0.35 g of 70um C_{18} material, radially compressed in a convenient syringe-adaptable, polyethylene column (18). Since Sep-Paks were introduced, there have been two reports on their use for extracting PNAH from water (19,20). Also, two other studies employed Sep-Paks for extracting more water soluble organic compounds (15,21). Wolkoff, a Waters Assoc. Inc. employee, described the use of Sep-Paks for PNAH extraction (20). However, he used the Sep-Paks under very favorable conditions, including: (1) high concentration of individual PNAH applied to the column; (2) an uncomplicated sample matrix of distilled water; and, (3) a low, 50 mL, sample volume. However, even under such favorable conditions, the PNAH with greatest loss due to breakthrough was benzo(a)pyrene, an event not predicted by theory (20). Cavelier (19) conducted a study using conditions more commonly encountered in PNAH analysis. Again the sample volume was low (200 mL) and the sample matrix was distilled water, but more realistic concentrations of PNAH were applied to the column (10-50 ng/L). He obtained extraction efficiencies of 60-100% for the six PNAH for which the World Health Organization has set limits. The greatest efficiency was for the most water soluble PNAH, fluoranthene, and the lowest efficiencies were for the much less soluble PNAH, indeno(1,2,3-cd)pyrene and

benzo(ghi)perylene (19). Again, those results are not in agreement with theoretical predictions.

Additional problems with employing Sep-Paks for extracting organics from water have also been described. Saner et al. (15) noted that extraction efficiencies decreased with increases in sample volumes. Both Saner et al. (15) and Puyear et al. (21) found lot-to-lot variations in the extraction efficiencies of Sep-Paks, a problem that would limit the routine use of Sep-Paks since calibrating each lot for extraction efficiency would be required. Puyear et al. (21) also found that Sep-Paks were most efficient for uncomplicated sample matrices, i.e., the extraction efficiency was good for one organic in water but decreased when additional organics were added.

The present study evaluates both EPA Method 610 and the Waters Sep-Pak method for routine field use. Both methods were modified to minimize their known disadvantages. All extractions by EPA Method 610 were done in the field to eliminate water transport and adsorption problems. Two Sep-Paks were used in series to increase capacity, slow flow rates were employed (15 mL/min) to decrease breakthrough losses, and only one lot of cartridges was used.

EXPERIMENTAL

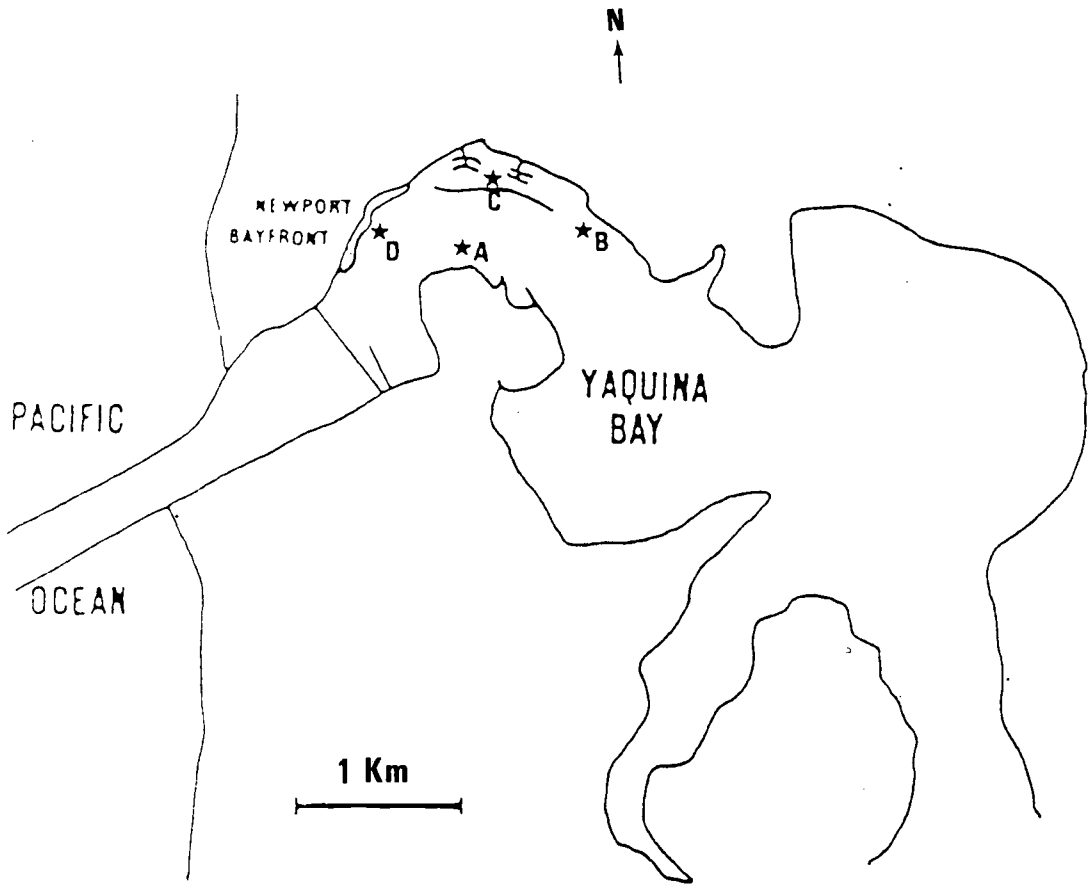
Preliminary Studies

Four sites in Yaquina Bay Oregon (Figure 1) were chosen to evaluate the two methods. Past work (22,23,24) had indicated that indigenous organisms at these sites had significantly different body burdens of several PNAH. Three sites (B,C,D) were located along the bay front and near a marina where a number of potential PNAH sources are concentrated. Slow tidal flushing combined with a number of possible sources including road runoff, creosoted piling leachate, outboard motor exhaust and spills of used oil could significantly contribute to the PNAH available to the biota. Site A was located on the less developed side of the bay and subsequently was not subjected to the combination of potential sources described above. All four sites could be subjected to other potential sources of PNAH such as atmospheric fallout and resuspended PNAH contaminated sediments.

Mussels (Mytilus edulis) located at site A had significantly greater (one or more orders of magnitude) body burdens of several PNAH compared to those from site D (23,24). Since mussels are sessile filter feeders they can only be exposed to PNAH by two routes i.e., absorption of soluble PNAH across the gill and mantle membranes and through ingestion and digestion of PNAH-contaminated particulates.

Preliminary laboratory studies were done to determine if modifications for the two water preconcentration methods were

Figure 1. Yaquina Bay, Oregon showing four sampling sites.



required to assure reliable data. Method 610 was only modified to the extent that extraction was done in the field to eliminate water transport problems. Cleanup steps (described below) for the procedure were also changed. For the Sep-Pak method numerous lab studies were undertaken to determine: (1) optimum flow rate through the cartridge, (2) cartridge capacity, (3) optimum filter combination, (4) adsorptive losses to the system, and (5) optimum Sep-Pak elution volume. These studies were done using both distilled and artificial sea water to which a known amount of PNAH standards were added. The results of those lab tests led to the Sep-Pak procedure described below.

Method 610 Sample Preparation

Water samples were collected from the four sites in the lower part of Yaquina Bay using a 14 foot outboard motor boat. Four two-liter glass separatory funnels with teflon stopcocks were mounted in a rack on the boat. Reagent grade DCM was premeasured into clean 100 mL glass sample bottles with teflon-lined caps. Subsurface water samples were collected in a 3 L stainless steel beaker. The beaker was inverted and plunged through the surface to a depth of about 40 cm,turned right side up, and rapidly returned to the surface. The top portion of the water (500 mL) was then spilled off to minimize surface contamination. Attempts were made to collect only subsurface water so as to minimize contamination due to surface slicks. One L of the unfiltered seawater was then transferred immediately to a separatory funnel and sequentially

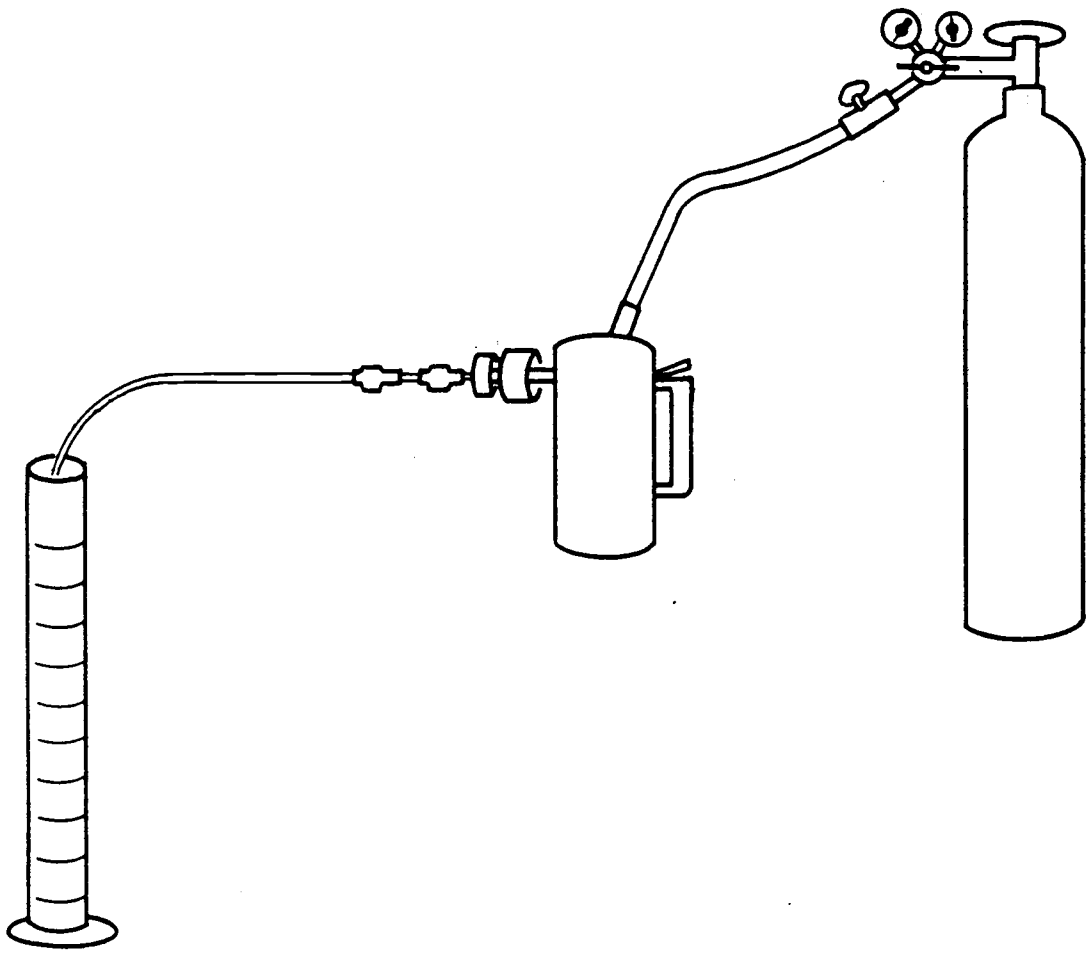
extracted with three 33 mL portions of DCM. The combined organic phases, along with a small emulsion layer, were returned to the sample bottle and brought back to Corvallis for analysis. The organic phase was extracted with reagent grade water (3 x 50 mL) and then dried by passing it through anhydrous sodium sulfate. The solvent was concentrated and exchanged by adding toluene to the DCM and then reducing the volume of the mixture by rotary evaporation. The extract was then further purified by passing it through a Sephadex LH-20 column (Pharmacia Inc.) (25,26). The eluate was reduced in volume by rotary evaporation followed by a stream of nitrogen to 100 uL. Determination of the PNAH was by HPLC.

Sep-Pak Method Sample Preparation

A one L portion of the water collected for Method 610 was transferred to a 1 L pressure vessel (Gelman Sciences Inc. Ann Arbor, MI, Cat.#7074). Two filter holders with #25 glass microfiber filters (Schleicher and Schull, Keene, NH) were attached in series followed by two prewetted Sep-Paks (4 mL acetonitrile followed by 5 mL reagent grade water) in series. A portable nitrogen tank (20 cubic foot) was used to pressurize the reservoir and control the flow rate through the cartridges. The effluent sample water was retained in a plastic graduate cylinder to determine sample volume (Figure 2).

The cartridges and filters were brought back to Corvallis for analysis. Each Sep-Pak was flushed with 5 mL of reagant grade water prior to PNAH elution to remove salts; then 5 mL of air was forced

Figure 2. Sep-Pak method sampling apparatus.



through the cartridge to remove most of the water. PNAH were then eluted with 2.5 mL of unpreserved tetrahydrofuran (THF). Both THF portions were combined and reduced in volume under a stream of nitrogen to 100 uL for HPLC analysis. The glass microfiber filters were blotted dry and extracted by the Soxhlet method for 24 hours with toluene. The resulting extract was dried with sodium sulfate and further purified by passing it through a Sephadex LH-20 column as for Method 610 extracts. The eluate was reduced in volume by rotary evaporation to about 1 mL and then further reduced under a stream of nitrogen to 100 uL for HPLC analysis.

HPLC Analysis

A Spectra-Physics model 8000 liquid chromatograph with a Schoeffel model 770 UV detector in series with a Schoeffel model 970 fluorescence detector was used. Table II lists chromatographic conditions.

Table II. HPLC specifications and conditions

LIQUID CHROMATOGRAPH

Spectra Physics 8000 with data system
Valco injector model CV-6-UHPa-N60 50 uL loop

COLUMNS

Analytical: Perkin-Elmer HC-ODS part # 258-0082 0.26 x 30 cm

Guard: Alltech high efficiency slurry packed with Vydac 201TP 0.32 x 10 cm

MOBILE PHASE

Acetonitrile/water gradient at constant flow 0.8 mL/min at 20°C

Time	%MeCN	$\%H_2O$
0	60	40
4	60	40
22	100	0
45	100	0
55	20	80
65	20	80
70	60	40
90	60	40

DETECTOR 1

Schoeffel Model 770 variable wavelength UV detector 296, nm range 0.01

DETECTOR 2

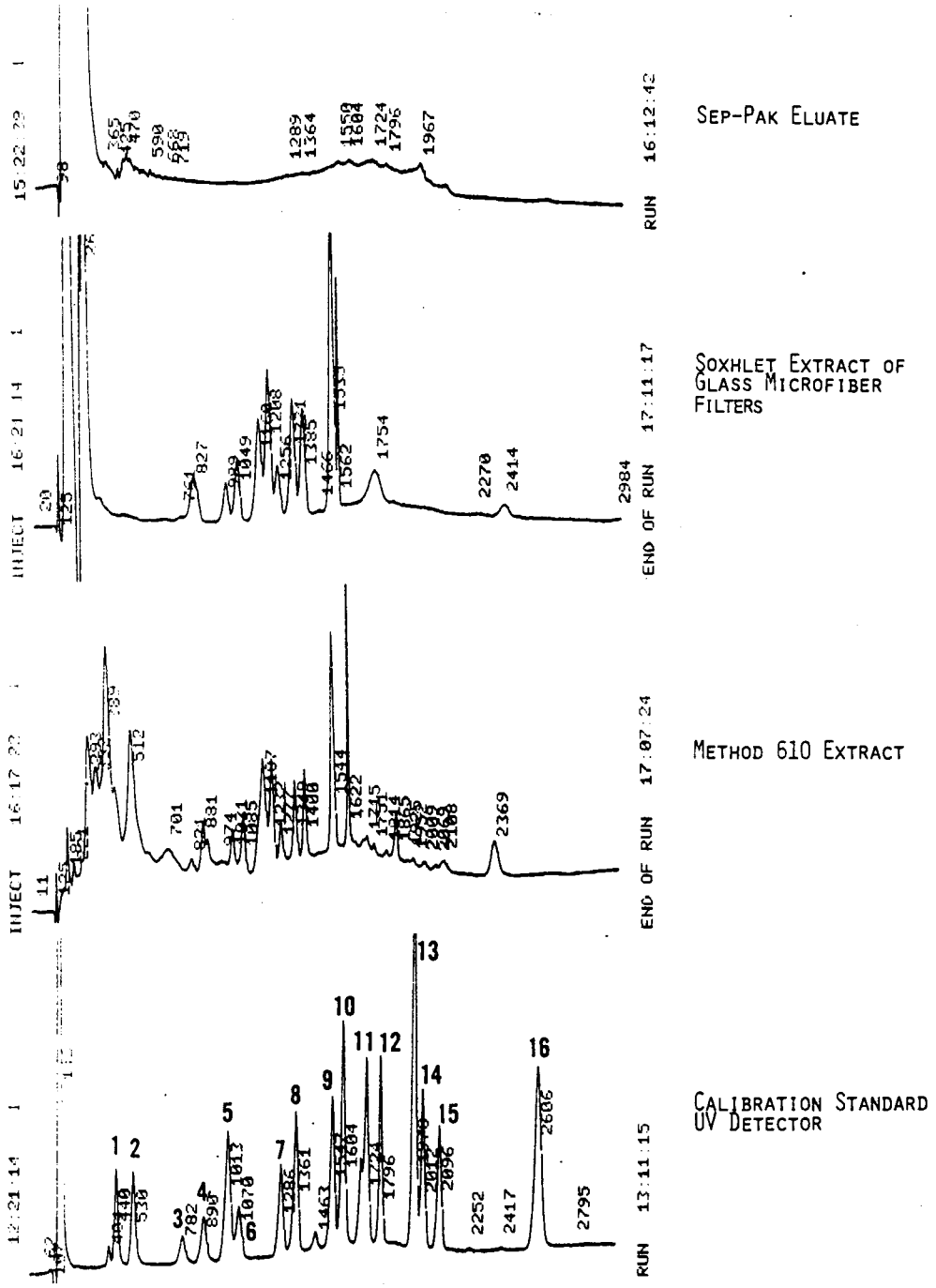
Schoeffel Model 970 variable wavelength fluorescent detector 326 nm excitation, >412 nm emission, range 0.1

RESULTS AND DISCUSSION

Typical chromatograms are shown in Figures 3 and 4 for UV and fluorescence detectors, respectively. Tables III and IV list data obtained from hourly sample collections by EPA Method 610 for the two collection dates at the four sites. Table V compares the two methods for subsamples of water. The Sep-Pak method data are split into an aqueous portion and a portion sorbed onto particulates.

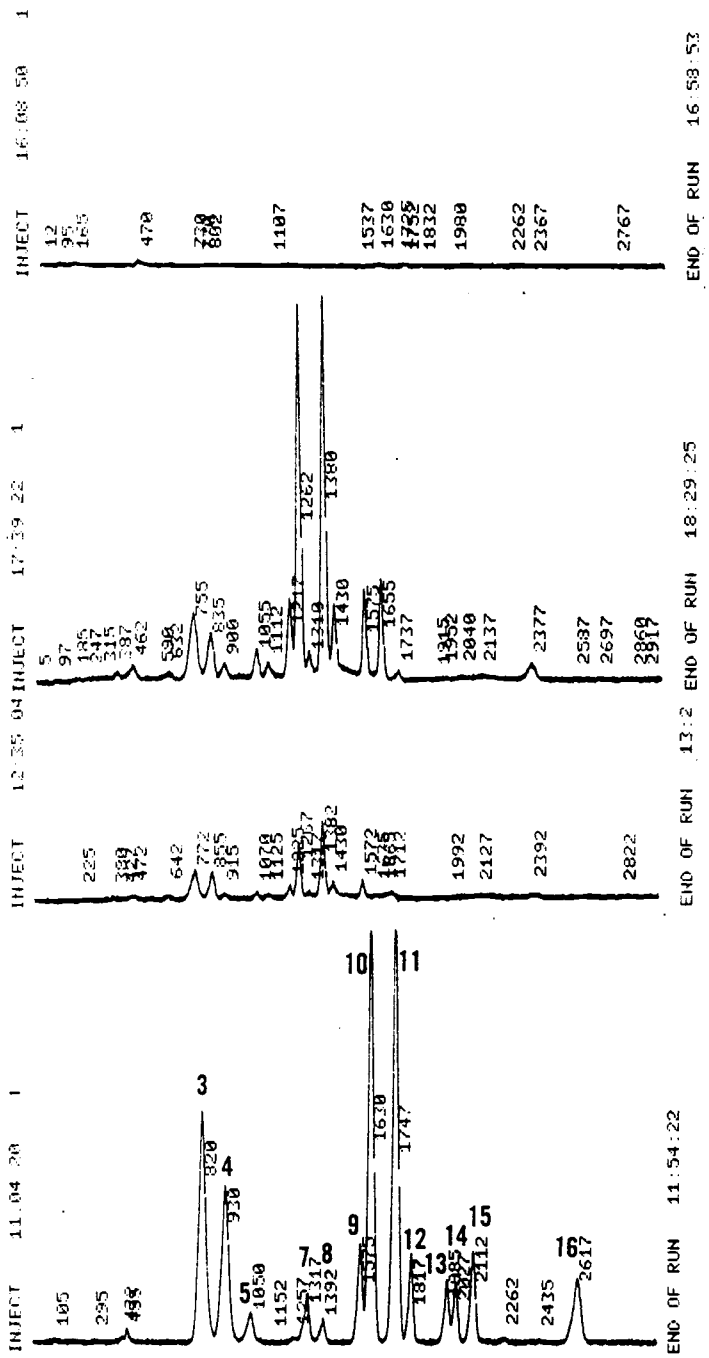
The data indicate that the majority of the PNAH present in the water column in Yaquina Bay are partitioned onto the particulates. This view is supported by the data in Table V which show that, in nearly all cases, the concentrations of PNAH for the particulate fraction is higher than the concentration determined by Method 610. However, some caution is necessary in interpreting the data since Method 610 may be inadequate for determining PNAH concentrations precisely in seawater. It appears that EPA Method 610 tends to underestimate PNAH concentrations, perhaps due to only partial extraction of PNAH bound to particulates. Based on this data, EPA Method 610 has an efficiency range of 3% to 100% compared to an assumed 100% extraction efficiency for Soxhlet extracted particulates. The efficiency depends upon the partition coefficient between the particulate fraction and the DCM for each PNAH. Since the nature of the estuarine water particulates changes as the tide changes (27), the extraction efficiencies could also change. The data in Tables III and IV also indicate that the tide has no distinct effect upon the measured concentrations of PNAH at each

Figure 3. Sample chromatograms for UV detector.



1 - Fluorene
2 - Phenanthrene
3 - Fluoranthene
4 - Pyrene
5 - Benzo(c)phenanthrene
6 - Triphenylene
7 - Benzo(a)anthracene
8 - Chrysene
9 - Benzo(j)fluoranthene/Benzo(e)pyrene
10 - Benzo(b)fluoranthene
11 - Dibenz(ac)anthracene/Benzo(k)fluor.
12 - Benzo(a)pyrene
13 - Dibenz(ah)anthracene
14 - Benzo(ghi)perylene
15 - Indeno(123-cd)pyrene
16 - Coronene

Figure 4. Sample chromatograms for fluorescence detector.



1 - Fluorene
2 - Phenanthrene
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4 - Pyrene
5 - Benzo(c)phenanthrene
6 - Triphenylene
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8 - Chrysene
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11 - Dibenz(ac)anthracene/Benzo(k)fluor.
12 - Benzo(a)pyrene
13 - Dibenz(ah)anthracene
14 - Benzo(ghi)perylene
15 - Indeno(123-cd)pyrene
16 - Coronene

Table III. PNAH concentration in water at four sites, Yaquina Bay Oregon, by EPA Method 610, 6/16/82.

Site	Obs[a]	PNAH concentration (ng/l)											
		Flr	Pyr	BaA	Chr	BbF	BkF	BaP	DBA	BgP	IP	Cor	Total
A	1	2.5	2.8	6.8	ND[b]	ND	ND	ND	ND	ND	ND	ND	12.1
	2	1.8	1.5	3.7	1.3	ND	ND	ND	ND	ND	ND	ND	8.3
	3	3.2	ND	4.1	2.3	ND	ND	ND	ND	ND	ND	ND	9.6
	4	2.0	1.8	2.2	ND	ND	ND	ND	ND	ND	ND	ND	6.0
	5	1.9	1.7	4.2	1.9	ND	ND	ND	ND	ND	ND	ND	9.7
	6	2.4	2.5	1.1	0.8	ND	ND	ND	ND	ND	ND	ND	6.8
	7	2.2	2.0	2.0	ND	ND	ND	ND	ND	ND	ND	ND	6.2
	8	3.0	1.3	4.4	ND	ND	ND	ND	ND	ND	ND	ND	8.7
	$\bar{x}$	2.4	1.9	3.6	1.6								8.4
B	1	4.1	3.7	2.8	1.4	1.0	ND	0.9	ND	ND	0.5	ND	14.4
	2	3.6	3.6	2.6	1.8	1.4	ND	0.7	ND	ND	0.1	ND	13.8
	3	2.8	5.5	2.9	2.9	1.6	ND	1.1	ND	ND	0.2	ND	17.0
	4	6.3	4.8	2.0	3.1	1.1	ND	1.2	ND	ND	0.4	ND	18.9
	5	7.1	4.8	3.1	3.0	1.8	ND	1.0	ND	ND	0.3	ND	21.1
	6	3.2	4.1	2.5	1.8	1.7	ND	0.8	ND	ND	0.2	ND	14.3
	7	3.2	4.9	1.9	3.0	1.5	ND	0.6	ND	ND	0.3	ND	15.4
	8	4.1	3.8	2.1	3.5	2.0	ND	0.7	ND	ND	0.1	ND	16.3
	$\bar{x}$	4.3	4.4	2.5	2.6	1.5		0.9			0.3		16.4
C	1	5.3	4.7	2.1	1.9	1.9	1.0	1.5	0.4	0.2	0.4	0.1	19.5
	2	6.8	4.8	2.8	1.8	2.3	1.0	1.8	0.6	ND	0.8	0.2	22.9
	3	7.1	5.4	3.6	1.7	2.5	0.9	0.9	0.3	ND	1.1	ND	23.5
	4	5.4	5.2	2.4	1.7	3.4	0.7	1.7	ND	0.1	0.9	ND	21.5
	5	4.1	3.9	3.4	1.8	2.8	1.3	1.8	0.2	0.2	0.6	0.3	20.4
	6	3.9	4.2	3.6	1.2	1.1	1.2	2.1	0.3	0.4	0.5	0.1	18.6
	7	4.8	3.7	2.8	1.4	3.1	1.0	0.8	0.1	ND	0.3	0.2	18.2
	8	5.1	4.1	2.0	1.1	2.9	0.8	0.7	0.1	0.5	0.2	ND	17.5
	$\bar{x}$	5.3	4.5	2.8	1.6	2.5	1.0	1.4	0.3	0.3	0.6	0.2	20.3
D	1	7.1	5.4	4.8	2.2	2.0	1.5	1.9	0.8	0.7	0.3	0.2	26.9
	2	6.8	3.2	3.4	2.5	2.0	1.1	2.3	1.1	0.5	0.4	0.1	23.4
	3	5.4	4.1	5.4	2.1	3.9	0.9	4.6	1.4	0.4	0.5	0.3	27.7
	4	3.2	4.8	3.2	2.0	4.3	1.2	3.2	2.0	0.3	0.7	0.2	25.1
	5	5.6	5.0	2.8	1.8	2.4	1.3	1.4	1.9	0.2	0.7	0.3	23.4
	6	5.5	4.9	4.6	1.5	3.0	1.0	4.8	0.8	0.8	0.8	0.1	27.8
	7	4.3	7.0	3.8	3.1	2.9	0.8	2.9	0.7	0.5	0.5	0.3	26.8
	8	2.8	5.4	5.1	2.4	4.7	1.4	3.0	1.4	0.6	0.3	0.2	27.3
	$\bar{x}$	5.1	5.0	4.1	2.2	3.2	1.0	3.0	1.3	0.5	0.5	0.2	26.1

[a]Obs--Hourly observation number

[b]ND--Not determined due to interference or levels below detection limit of LC

Table IV. PNAH concentration in water for four sites, Yaquina Bay Oregon, by EPA Method 610, 6/17/82.

Site	Obs[a]	PNAH concentration (ng/l)											
		Flr	Pyr	BaA	Chr	BbF	BkF	BaP	DBA	BgP	IP	Cor	Total
A	1	1.7	3.1	5.3	1.1	ND	ND	ND	ND	ND	ND	ND	11.2
	2	2.8	4.0	4.7	1.1	ND	ND	ND	ND	ND	ND	ND	12.6
	3	3.7	2.8	1.7	1.7	ND	ND	ND	ND	ND	ND	ND	9.9
	4	2.1	2.7	3.6	ND	ND	ND	ND	ND	ND	ND	ND	8.4
	5	2.6	2.6	2.1	ND	ND	ND	ND	ND	ND	ND	ND	7.3
	6	3.3	2.5	2.1	1.0	ND	ND	ND	ND	ND	ND	ND	7.9
	7	2.9	3.0	3.0	1.3	ND	ND	ND	ND	ND	ND	ND	10.2
	8	1.9	2.8	2.5	1.4	ND	ND	ND	ND	ND	ND	ND	8.6
	Ave	2.6	2.9	3.1	1.3								9.5
B	1	5.8	4.6	4.1	8.1	ND	ND	ND	ND	ND	0.3	ND	22.9
	2	6.1	5.3	4.0	4.3	ND	ND	ND	ND	ND	0.2	ND	19.9
	3	3.4	5.0	3.7	4.1	ND	ND	ND	ND	ND	ND	ND	16.2
	4	2.1	4.1	3.4	3.9	ND	ND	ND	ND	ND	ND	ND	13.5
	5	4.3	3.9	2.1	2.7	ND	ND	ND	ND	ND	0.1	ND	13.1
	6	4.0	2.8	2.9	2.8	ND	ND	ND	ND	ND	ND	ND	12.5
	7	3.7	3.6	3.5	4.0	ND	ND	ND	ND	ND	ND	ND	14.8
	8	3.1	2.7	2.1	1.1	ND	ND	ND	ND	ND	ND	ND	9.0
	Ave	4.1	4.0	3.2	3.9						0.2		15.2
C	1	8.7	6.8	7.7	2.5	2.4	1.5	2.8	0.9	0.1	0.1	0.1	33.6
	2	5.6	7.6	3.6	2.0	ND	1.6	ND	ND	0.1	0.2	ND	20.7
	3	5.4	8.0	2.1	2.1	2.3	ND	1.1	0.8	0.2	0.4	ND	22.4
	4	6.3	8.1	2.6	1.9	2.0	1.1	1.4	0.6	0.1	0.1	0.3	24.5
	5	4.1	7.6	2.5	1.7	1.8	0.9	2.6	0.6	ND	0.3	0.1	22.2
	6	3.9	4.3	3.1	2.3	ND	1.8	2.0	ND	0.1	0.2	ND	17.7
	7	4.7	3.7	2.8	2.1	3.0	0.8	0.9	0.5	0.2	0.1	0.1	18.9
	8	5.0	6.5	2.9	2.0	2.8	0.7	1.3	0.4	0.1	0.2	0.1	22.0
	Ave	5.5	6.6	3.4	2.1	2.4	1.2	1.7	0.6	0.1	0.2	0.1	22.8
D	1	8.6	5.8	6.8	2.8	3.4	1.5	1.4	2.0	1.8	0.8	0.2	35.1
	2	8.6	7.9	3.1	3.1	2.9	1.3	4.0	1.7	ND	0.7	0.3	33.6
	3	8.7	9.1	4.3	3.0	3.1	ND	3.9	1.7	1.7	0.6	ND	36.1
	4	8.9	9.0	2.8	2.9	3.3	1.7	2.8	1.5	1.5	0.6	0.2	35.2
	5	7.6	5.3	3.8	3.6	3.2	2.8	1.7	1.3	1.4	0.7	0.1	31.5
	6	7.9	4.6	2.7	2.1	2.1	1.4	5.1	1.2	1.4	0.5	0.4	29.4
	7	4.7	5.7	2.9	2.0	3.0	1.0	2.7	1.8	1.3	0.7	0.3	26.1
	8	5.3	6.0	3.7	2.0	3.4	1.3	3.6	1.9	1.5	0.8	0.2	29.7
	Ave	7.5	6.7	3.8	2.7	3.1	1.4	3.2	1.6	1.3	0.7	0.2	32.1

Table V. PNAH concentration (ng/l) at four sites Yaquina Bay Oregon; determined by two methods.

6/16	EPA Method 610				Sep-Pak Method				Soxhlet Method			
PNAH	liquid-liquid				water				particulates			
	A	B	C	D	A	B	C	D	A	B	C	D
Flr	2.2	3.4	5.9	3.0	ND[a]	ND	ND	ND	ND	5.2	4.4	5.8
Pyr	2.3	7.7	4.6	5.2	ND	ND	ND	ND	ND	9.1	5.3	6.0
BaA	5.5	2.6	3.2	4.2	ND	0.2	0.4	0.6	12.8	2.4	15.5	13.3
Chr	1.9	1.8	1.6	2.2	ND	ND	0.3	0.1	9.7	3.1	9.8	11.7
BbF	ND	1.6	2.8	4.5	ND	ND	ND	ND	5.4	3.7	11.2	10.8
BkF	ND	ND	1.0	1.3	ND	ND	ND	0.1	3.7	2.1	5.3	14.7
BaP	ND	0.8	0.9	3.1	ND	0.3	ND	0.4	6.1	4.8	7.6	15.1
DBA	ND	ND	0.2	1.7	ND	ND	ND	ND	5.4	7.3	4.8	12.9
BgP	ND	ND	ND	0.5	ND	ND	ND	ND	3.0	3.0	12.9	11.6
IP	ND	0.2	0.7	0.5	ND	ND	0.3	0.5	2.8	0.8	5.7	8.1
Cor	ND	ND	0.2	0.2	ND	ND	ND	ND	0.7	0.9	0.4	0.7
6/17												
Flr	2.0	5.1	4.8	6.7	ND	ND	ND	ND	5.8	14.9	17.6	8.7
Pyr	2.8	4.3	6.0	7.4	ND	ND	ND	ND	7.1	16.8	8.4	9.1
BaA	3.1	3.1	3.4	3.6	ND	ND	0.2	ND	14.6	13.1	8.0	11.3
Chr	1.4	5.4	2.2	2.5	ND	ND	ND	ND	9.7	10.0	3.2	12.1
BbF	ND	ND	ND	3.1	ND	0.3	ND	ND	6.3	5.1	2.8	9.3
BkF	ND	ND	1.7	1.0	ND	ND	ND	0.3	4.8	5.9	6.7	6.3
BaP	ND	ND	2.0	3.3	ND	ND	ND	ND	5.7	3.7	5.2	3.1
DBA	ND	ND	ND	1.8	ND	ND	ND	ND	6.1	4.0	4.1	2.8
BgP	ND	ND	0.1	1.5	ND	ND	0.1	ND	4.0	2.1	3.3	5.1
IP	ND	0.3	0.2	0.7	ND	ND	ND	ND	3.4	7.1	2.8	1.9
Cor	ND	ND	ND	0.3	ND	ND	ND	ND	0.9	2.0	1.3	1.0

[a]ND-not determined due to interference or levels below detection limit

site. The concentrations of PNAH at each site tend to vary, perhaps randomly, around the average. This would not be the case for sites above and below a point source. For example, if the marina was a point source, westward sites would tend to have increased PNAH levels on an outgoing tide and lower PNAH levels on an incoming tide. The opposite would be expected for a site east of the marina. Sites B and D are east and west of the marina, respectively and they do not seem to show any change in PNAH concentration over the sampling period which varies with tide flow.

The partition coefficients for the different PNAH of interest vary by about 3 orders of magnitude for octanol/water (8) so the extraction efficiencies of the PNAH is likewise expected to vary. Foerst et al. (28) found that EPA Method 610 gave recoveries of greater than 50% for two and three ring PNAH in a landfill leachate sample to which known amounts of PNAH standards had been added. Larger PNAH (five and six rings) recoveries were less than 50%; for example, the recovery for indeno(1,2,3-cd)pyrene was only 10% +/- 27. For reagent water samples, the recoveries for all PNAH tested were greater than 67% and averaged 84% (28). Foerst (personal communication 29) indicated that the leachate samples were unfiltered and he suspected that the low recoveries were due to adsorption onto the particulates.

Several authors have determined PNAH in estuarine, and industrial and municipal wastewaters (11,13,28,30,31,32) using liquid-liquid extraction methods similar to EPA Method 610. Those

investigators, in many cases, assumed that PNAH were primarily associated with suspended particulates but did not attempt to determine their exact distribution. Strup (13) found that PNAH were primarily associated with particulates in wastewater samples and that this affected sample homogenity. His recoveries using EPA Method 610 for wastewaters were all greater than 76% for samples to which known amounts of PNAH had been added. However, the recovery percentages varied irregularily between 76-115%. None of those authors attempted to determine the PNAH partitioned onto the particulates in either natural or wastewaters.

If EPA Method 610 was to be used in a routine monitoring program, one would have to determine the extraction efficiency for each PNAH of interest in each individual sample.That would be necessary because the composition of the particulates in a sample can change over time and so, extraction efficiencies would also change. Also if the particulates change it is reasonable to assume that since nearly all PNAH will partition onto the particulates, their concentrations in water would also vary.

Filtering water samples and then determining quantities of PNAH partitioned onto the particulates has two advantages. First, it is possible to rapidly filter large volumes of water (1-10L) and more easily detect small quantities of PNAH (1 pg/mL) and second, the extraction efficiencies are nearly constant (100%) for all samples.

Data from the Sep-Pak method indicate that the unbound PNAH

fraction is insignificant. However, this may be misleading due to possible irreversible adsorption of the PNAH on the cartridge. In preliminary laboratory studies using distilled water (1 L) to which 120 to 265 ng of PNAH standards had been added there were significant losses of PNAH that could only be accounted for by irreversible adsorption (Table VI). The results suggest that 39 to 94% of the PNAH were irreversibly bound to the Sep-Paks. Cavelier (19), however, found that losses for 200 mL water samples (10-50 ng/L) were not greater than 40%.

The data from the present study indicate that most of the PNAH present in the water column are partitioned onto particulates. Filtration of the water sample followed by Soxhlet extraction of the filters appears to be the best way to estimate the degree to which an aquatic environment is contaminated. Method 610 was developed for industrial effluent water where the degree of contamination is relatively high in comparison to natural waters. For natural, cleaner water systems (ng/L levels) the use of Method 610 underestimates PNAH concentrations. The use of Sep-Paks without prefilters would also result in an underestimation of PNAH concentrations because of both irreversible adsorption of aqueous PNAH and low extraction efficiencies of the particulates with the small volume of eluting solvent. The results herein indicate that a rapid and inexpensive method to measure contamination of natural waters by PNAH is to filter out the particulates, extract the filters by the Soxhlet method and then determine concentrations of

Table VI. Sep-Pak recovery determination with known amounts of PNAH standards added to one liter of artificial seawater.

PNAH	Amt. Added (ng)	Amt. Recov. Sep-Pak (ng)	Amt. on Filters (ng)	Amt. Resid. Water (ng)	Amt. in Pass Through Water (ng)	Amt. Adsor. Press. Vess. (ng)	% Recov. Sep-Pak	Total % Rec.
Flu	259	123	--	13.7	3.8	16.2	47	61
Pyr	265	124	--	2.5	--	8.3	47	51
BbF	120	12.7	--	6.2	--	4.1	11	19
BaP	125	14.0	5.4	2.1	--	2.0	11	19
IP	120	3.8	--	1.4	--	2.3	3	6

PNAH by HPLC methodology.

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Appendix

Appendix A

Abreviations used in this report

PNAH - polynuclear aromatic hydrocarbon

Flr - fluoranthene

Pyr - pyrene

BaA - benzo(a)anthracene

Chr - chrysene

BbF - benzo(b)fluoranthene

BkF - benzo(k)fluoranthene

BaP - benzo(a)pyrene

DBA - dibenz(a,h)anthracene

BgP - benzo(g,h,i)perylene

IP - indeno(1,2,3-cd)pyrene

Cor - coronene

HPLC - high performance liquid chromatography

DCM - dichloromethane

THF - tetrahydrofuran unpreserved

ODS - octadecyl silane

Kow - octanol/water partition coefficient

S - solubility (mg/L)

Carc. - carcinogenicity

Resid. water - residual water left in pressure vessel after a Sep-Pak run

Adsor. Press. Vess. - adsorbed on pressure vessel