

## **Dissolved and Suspended Polycyclic Aromatic Hydrocarbons (PAH) in the North Aegean Sea**

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### **Abstract**

*The distribution and sources of polycyclic aromatic hydrocarbons (PAH) were investigated in the seawater of the North Aegean Sea. The measured PAH concentrations in SPM are generally considered as elevated for open sea waters and were evenly distributed in the area. Their levels in the dissolved phase (1.6-33.0 ng/l) were much higher than those encountered in the corresponding particulate phases (0.04-10.2 ng/l). The PAH patterns in both phases were dominated by the three ring aromatics and their alkylated derivatives, reflecting a predominant contribution of fossil hydrocarbons probably related to ship traffic, whereas no significant inputs from the rivers outfalling in the area were detected. In bottom waters PAH values were generally lower, whereas a higher depletion of the petroleum PAH in comparison with the pyrolytic ones according to depth was observed.*

**Keywords:** PAH, Hydrocarbons, Seawater, Aegean sea.

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### **Introduction**

Polycyclic aromatic compounds (PAH) are widespread organic contaminants, deriving primarily from the partial combustion of coal, wood and various fossil fuels, while low molecular weight PAHs are also major constituents of petroleum. PAHs are considered to be hazardous to the environment. Some of the higher molecular weight compounds have strong carcinogenic properties, whereas two to three aromatic ring PAHs may be acutely toxic to aquatic organisms (FERNANDES *et al.*, 1997, WOODHEAD *et al.*, 1999). Their

main input sources into the marine environment include riverine inflows, ship traffic, accidental oil spills and sewage disposal. In addition, atmospheric long-range transport and deposition is considered an important introduction pathway in the open sea (BURNS & SALIOT, 1986). PAHs have been found to be ubiquitous constituents in both coastal and open sea marine sediments (BOULOUBASSI & SALIOT, 1993, WOODHEAD *et al.*, 1999, GOGOU *et al.*, 2000, MAGI *et al.*, 2002, READMAN *et al.*, 2002) but the complex biogeochemical processes influencing their distribution in the seawater column are still

poorly understood (DACHS *et al.*, 1997). Since PAHs are nonpolar and slightly soluble in water their biogeochemical cycling is controlled by their high affinity to aquatic particles, especially to the organic carbon or lipid content (KARICKHOFF *et al.*, 1979). Their partitioning between particulate and dissolved phases, which can be described by a partition coefficient  $K_p$  (ZHOU & ROWLAND, 1997), is primarily controlled by the physicochemical characteristics of the PAH compounds and particles, including the solubility, hydrophobicity, and molecular weight of the PAHs and the chemical composition and surface area of the particles. Dissolved PAH are transported by molecular and turbulent diffusion and advection, while particle bound compounds are transported by turbulent diffusion and sediment transport, including settling and resuspension (KO & BAKER, 1995). Bioaccumulation by marine organisms and biological or photochemical degradation and oxidation could also play an important role in controlling the environmental fate of PAHs in marine systems (LATIMER *et al.*, 1999).

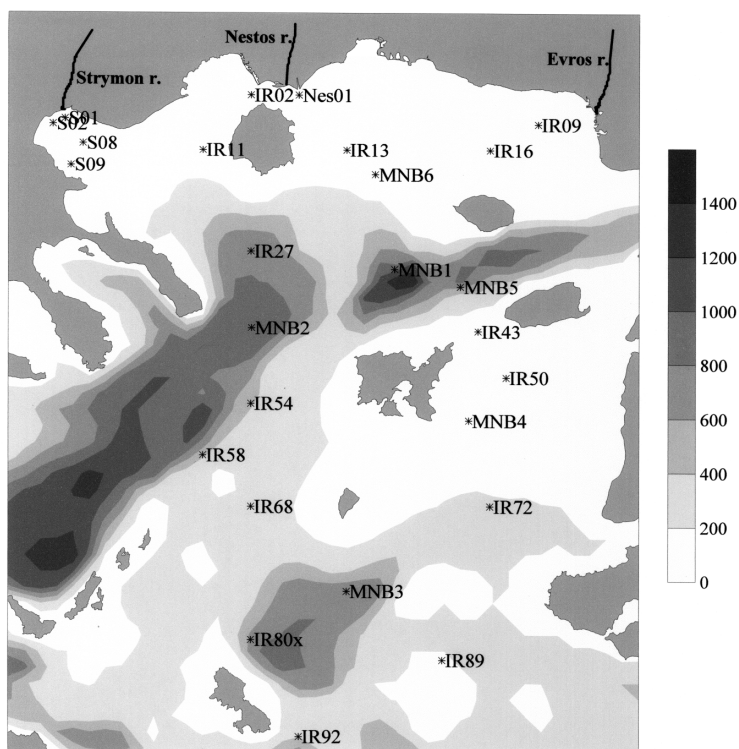
Although many studies regarding PAH sources, fate and distribution in the marine environment have been carried out in the western part of the Mediterranean Sea (BOULOUBASSI & SALIOT, 1991, 1993, DACHS *et al.*, 1997, TOLOSA *et al.*, 1996, BENLAHCEN *et al.*, 1997), much less attention has been given to the eastern part of this region (GOGOU *et al.*, 2000). The North Aegean Sea, a dynamically active area in the eastern Mediterranean with unique physiographic and hydrodynamic characteristics, is generally characterized by well oxygenated waters and low primary productivity. Its surface layers are directly influenced by the brackish and low temperature Black Sea water entering the Aegean Sea through the Dardanelles Straits, whereas the water exchange between North and South Aegean is limited to only above 400 m as the bathymetry of the region restricts the movement of the waters below that level (GEORGOPOULOS *et al.*, 1998).

Furthermore the North Aegean Sea is the recipient of riverine discharges (Evros, Nestos, Strymon), while deposition of aeolian particles may also contribute to the pollution burden of the area.

The aims of this work, undertaken in the framework of the INTERREG program, were (i) to study the distribution of PAHs in the North Aegean seawater in both the dissolved phase (DP) and the suspended particulate matter (SPM) and (ii) to recognize their sources and to investigate the transformations affecting PAHs in the water column.

## Materials and Methods

Surface water samples (10l volume) were collected from 26 stations (Fig. 1) in May 1997, February 1998, June 1998, September 1998 and February 1999 by means of a water sampling device consisting of a weighted bottle holder with a clean amber-glass bottle (UNEP, 1984). Water from the maximum depth was also collected in some stations. In order to have a representative picture of the whole area, our sampling strategy included stations believed to be directly influenced from coastal activities and/or river discharges (STR01, STR02, STR09, STR08, IR02, NES01, IR09) and deep open sea stations (IR27, MNB1, MNB2, MNB3, IR68, IR89, IR80x). The samples were filtered immediately after collection through precleaned glass fibre filters (Whatman GF/F, 0.7  $\mu\text{m}$ ). After the addition of internal standards (phenanthrene-d10, pyrene-d10, chrysene-d12, perylene-d12), the filtrates were extracted on board, within a few hours after filtration, with n-hexane. The n-hexane extracts were dried over sodium sulphate, reduced to 2ml in a rotary evaporator and stored in the refrigerator until analysis. The filters after the addition of internal standards were extracted with dichloromethane in a sonicator bath for 30 min. In the laboratory the extracts were further reduced to a final volume of 50  $\mu\text{l}$  with the aid of a stream of pure nitrogen and PAH



**Fig. 1:** Sampling stations in the North Aegean Sea.

analyses were conducted by gas chromatography – mass spectrometry (Hewlett Packard 6890 GC-MS) in full scan mode. A CP-Sil 8 MS analytical column (30 m X 0.25 mm i.d. X 0.25  $\mu$ m film thickness) was used and the oven temperature was programmed from 60° C (1 min) to 290° C (10 min) at 15° C/min. For the calculations the molecular ion extracted chromatograms for each compound were used and the PAH quantitation was based on the internal standards added before the extraction. The recoveries were higher than 70 %. Procedural blanks were processed in the same manner as real samples and they were below 20 % of the abundance of analytes. The detection limit for each PAH was 0.01 ng/l.

## Results and Discussion

The PAHs determined in this study include the following compounds with 2-6 aromatic

rings: Acenaphthylene, acenaphthene, fluorene, phenanthrene, anthracene, dibenzothiophene, methyl-dibenzothiophenes, methyl- and dimethyl- phenanthrenes, fluoranthene, pyrene, benzo( $\alpha$ )anthracene, chrysene, benzo( $\alpha$ )fluoranthenes, benzo( $\alpha$ )pyrene, benzo(e)pyrene, perylene, dibenzo( $\alpha$ ,h)anthracene, indeno-(1,2,3-cd)pyrene and benzo(g,h,i)perylene. Their total concentrations (the sum of all the above PAH,  $\Sigma$ PAH) for all the stations and sampling periods are given in Table 1.  $\Sigma$ PAH values range from 1.6 to 33.0 ng/l (mean value 9.6 ng/l) in the dissolved phase and from 0.04 to 10.2 ng/l (mean value 1.7 ng/l) in the particulate phase. Although comparative data on PAH concentrations in seawater are few in the literature and intercomparison studies of PAH analysis relatively poorly developed, the values measured in this study can be considered as rather elevated for open sea waters. In Table 2, PAH concentrations found in the North

**Table 1**  
**Concentrations (ng/l) of dissolved and suspended total polycyclic aromatic hydrocarbons ( $\Sigma$ PAH) in the North Aegean Sea during the five sampling periods (S: surface, B: bottom).**

Station	Sampling Depth	Cruise 5/97		Cruise 2/98		Cruise 6/98		Cruise 9/98		Cruise 2/99	
		Diss.	Susp.	Diss.	Susp.	Diss.	Susp.	Diss.	Susp.	Diss.	Susp.
IR02	S	13.7	2.9	4.6	5.4	10.4	3.3	9.2	0.23	17.1	1.7
IR09	S	10.2	1.9	6.6	0.93	8.6	2.6	8.8	0.33	15.6	1.1
IR11	S	4.3	1.8			7.6	3.4	5.5	0.75	8.9	0.68
IR13	S			3.6	1.3						
IR16	S	6.7	3.4	6.3	1.1	4.0	3.7	33.0	0.21	12.5	0.96
	B (50m)									14.4	0.08
IR27	S									31.8	0.84
IR43	S	9.0	3.0	26.5	1.7	7.0	1.1	32.8	0.66	13.7	1.2
IR50	S	9.4	1.6		0.14	5.9	1.0	8.3	2.5	23.1	1.1
IR54	S			3.9	2.0						
IR58	S			10.9	4.2						
	B (480m)			3.8	4.0						
IR68	S	5.7	2.0			17.7	2.0	26.2	0.27	8.9	0.32
	B (350m)	4.8	1.7			12.3	0.73	8.4	0.44		
IR72	S	6.1	1.8			3.1	3.1	9.0	0.35		
IR80x	S					31.4	2.1				
IR89	S							5.9	1.6	8.8	0.24
IR92	S	10.0	1.8					7.3	0.44	14.2	1.1
MNB1	S	12.7	2.2	5.0	0.72	5.2	10.2	6.5	0.64	14.4	5.4
	B (1297m)			1.5	0.15					3.7	5.6
MNB2	S	3.4	1.2	7.1	1.0	9.7	2.9	5.2	0.19	5.1	2.2
	B (976m)	3.6	1.4	3.0	1.2	7.4	1.4	7.2	0.23		
MNB3	S									26.7	0.44
MNB4	S	7.4	2.4		2.6	8.5	4.1	4.5	0.12	26.0	0.83
	B (94m)	4.3	2.5	2.3	0.04	6.9	1.5	2.3	2.8		
MNB5	S	7.4	2.3	9.2	0.56	4.4	6.6	5.8	0.33	17.2	1.7
	B (520m)	5.0	1.6	1.9	0.45	4.0	3.6	5.0	0.10		
MNB6	S			6.1	1.5						
NES01	S			5.7	0.71	4.7	1.2	7.9	1.5		
STR01	S					8.9	1.2				
STR02	S	9.1	2.3					18.1	0.13		
STR08	S					8.1	2.4				
STR09	S	15.1	1.9					14.5	0.16		
<b>Mean</b>	<b>S</b>	<b>8.7</b>	<b>2.2</b>	<b>8.0</b>	<b>1.7</b>	<b>9.1</b>	<b>3.2</b>	<b>12.3</b>	<b>0.62</b>	<b>16.3</b>	<b>1.3</b>
<b>Mean</b>	<b>B</b>	<b>4.4</b>	<b>1.8</b>	<b>2.5</b>	<b>1.2</b>	<b>7.7</b>	<b>1.8</b>	<b>5.7</b>	<b>0.90</b>	<b>9.0</b>	<b>2.8</b>

Aegean are compared with measurements from other regions of the world. For comparison reasons the PAH concentrations given in Table 2 include only the parent PAH and not all the compounds determined. As can be seen, the values found in the North Aegean Sea are higher than those measured in the open western Mediterranean (DACHS *et al.*, 1997), in the Black Sea (MALDONADO *et al.*, 1999)

and the Cretan Sea (GOGOU & STEPHANOPOULOU, 1997). Similar values to ours have been reported in the North Sea and the Baltic Sea (LAW *et al.*, 1997, WITT & MATTHAUS, 2001) and in southeastern Mediterranean (EHRHARDT & PETRICK, 1993). However our values are significantly lower than those found in the Tyrrhenian Sea (CINCINELLI *et al.*, 2001) and in polluted estuarine systems

**Table 2**  
**Reported values of total parent PAH concentrations in seawater (ng/l).**

Area	Dissolved	Suspended	Total	Reference
North Aegean	2.5-19.5	0.02-2.9	3.9-20.2	THIS STUDY
Cretan sea	0.4-1.4	0.3-1.4		GOGOU & STEPHANOU, 1997
Open Western Mediterranean	0.5-2.2	0.2-1.1		DACHS <i>et al.</i> , 1997
Northwestern Black Sea	0.8	1.0		MALDONADO <i>et al.</i> , 1999
Southeastern Mediterranean	50			EHRHARDT & PETRICK, 1993
North Sea			0-15	LAW <i>et al.</i> , 1997
Baltic Sea			3.1-9.2	WITT & MATTHUS, 2001
Rhone Delta	31-120	2.9-19		BOULOUBASSI & SALIOT, 1991
Tyrrhenian Sea	63-3050	581-7540		CINCINELLI <i>et al.</i> , 2001
Chesapeake Bay estuaries	3200-43000	1800-23000		GUSTAFSON & DICKHUT, 1997

such as the Rhone Delta in the western Mediterranean (BOULOUBASSI & SALIOT, 1991) and the Chesapeake Bay estuaries in the USA (GUSTAFSON & DICKHUT, 1997).

#### *PAH in the dissolved phase*

PAH are considered to occur almost exclusively associated with particles due to their high hydrophobic properties (ZHOU & ROWLAND, 1997). Because of this, data on the occurrence of PAHs in the dissolved phase are scarce in the literature. However, the dissolved phase, usually defined by filtration on a 0.7 µm filter, contains not only the truly dissolved compounds but also most colloidal material and very fine particles, which are not retained in the glass fiber filters. In the North Aegean Sea PAH levels in the dissolved phase were much higher than those encountered in the corresponding particulate phases. Dissolved PAHs were measured in percentages varying between 33.8 % and 99.4 % (mean value 83.2 %) of the total (sum of dissolved and particulate PAH). These percentages are higher than those usually reported in the literature (DACHS *et al.*, 1997, MALDONADO *et al.*, 1999) and are probably related to the low SPM content of the oligotrophic Aegean Sea waters (KARAGEORGIS *et al.*,

2001). These findings suggest that in the Aegean Sea the dissolved phase may play a significant role in the transport and flux of PAHs, as well as in their bioavailability and ultimate fate.

The spatial distribution of the dissolved PAH concentrations in surface waters is presented in Figure 2 and seems to follow a rather uncommon pattern. The highest values were not detected in stations located close to the north coasts of the Aegean Sea or close to the river mouths as could be expected. On the contrary, high values, above 25 ng/l, were occasionally recorded in open sea stations (IR27, IR68, IR80x, MNB3). This fact suggests that the PAH measured probably correlate with the ship traffic in the area, while the rivers flowing into the North Aegean Sea and the various coastal activities cannot be considered as major PAH suppliers. This is in accordance with studies on PAH levels in sediments, performed in coastal areas of northern Greece (HATZIANESTIS *et al.*, 2001, SKLIVAGOU *et al.*, 2001, HATZIANESTIS & SKLIVAGOU, 2000) which have shown that no significant PAH pollution occurred in the sediments.

No systematic seasonal variation was observed. The highest values in both surface and bottom waters were measured in February 1999 (mean value 16.3 ng/l in surface and 9.0

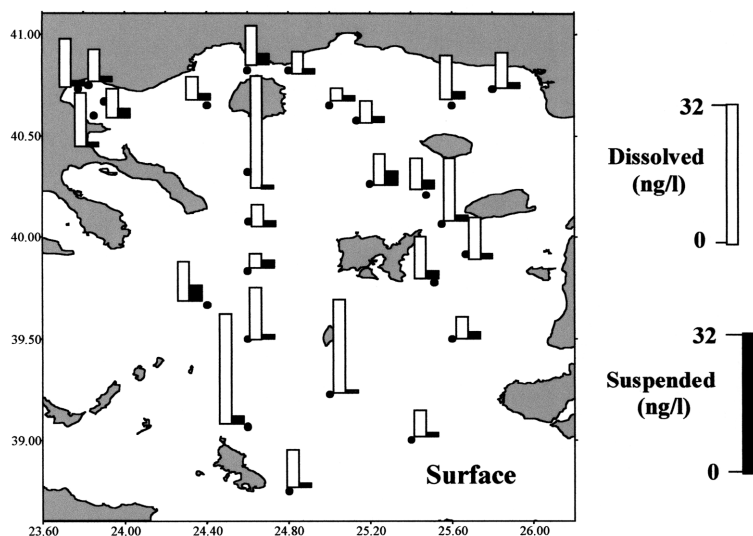


Fig. 2: Mean PAH concentrations in seawater of the North Aegean Sea.

ng/l in bottom) in contrast with February 1998 when the lowest values had been recorded (mean value 8.0 ng/l in surface and 2.5 ng/l in bottom). In September 1998, when river discharges were negligible and SPM content was also minimal (KARAGEORGIS *et al.*, 2001), dissolved PAH concentrations remained high, even in near-shore samples (stations STR02, STR09, NES01) and this is an additional indication that the main PAH sources could not be directly related to anthropogenic activities on the land.

In Figure 3 the mean surface PAH concentrations are compared with those in bottom waters. It should be noted that the mean surface PAH values presented in Figure 3 correspond only to the stations where both surface and bottom waters were collected. As can be seen a surface-enrichment depth depletion distribution was found, in agreement with the introduction of organic contaminants into the surface waters. However, PAH values in bottom waters were still high and this is attributed to their high residence time in the water column, whereas bottom currents or resuspension events could also contribute to an enrichment of the PAH content close to the bottom.

#### PAH in the particulate phase

PAH concentrations in the particulate material were very low compared to those in the dissolved phase and in many cases high MW compounds presented values very close or even below the detection limit of the method. These low values are attributed to the very low SPM content in Aegean Sea waters which in most cases was well below 0.8 mg/l (KARAGEORGIS *et al.*, 2001). PAH spatial distribution in SPM was more homogenous than in the dissolved phase (Fig. 2) suggesting again the absence of point sources of pollution.

The seasonal variation and the vertical distribution of PAH concentrations in SPM was different from those in the dissolved phase. The lowest values in both surface and bottom waters were recorded in September 1998 in accordance with the low riverine runoff and the low SMP concentrations recorded this season (KARAGEORGIS *et al.*, 2001). In February 1999, when the highest dissolved PAH concentrations were measured, particulate PAH presented relatively low values. Close to the bottom, PAHs in SPM presented similar, and in some cases even greater, values



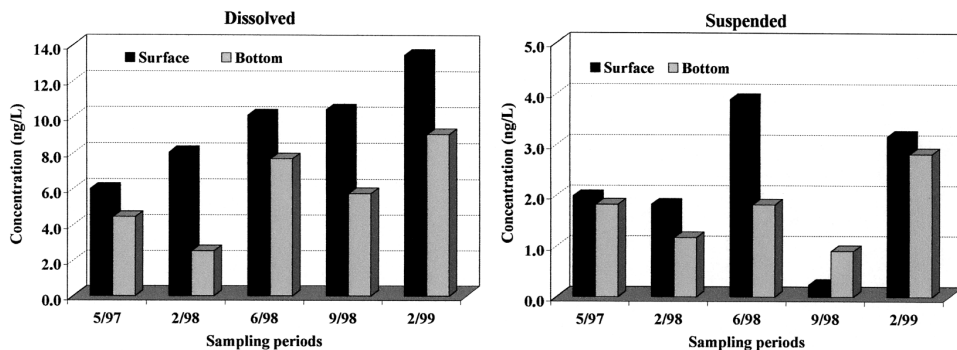


Fig. 3: Mean PAH values in surface and bottom waters during the five sampling periods.

than those in surface waters (Fig. 3). This is consistent with the pronounced benthic nepheloid layer found in all sampling periods (KARAGEORGIS *et al.*, 2001) and could be attributed to sediment resuspension caused by near bed currents that may lift fine-grained material from the seabed even at very low velocities.

It is interesting to examine the relationship between the PAH values in the dissolved phase and SPM. As it can be seen in Figure 4, where these two values are plotted, there is no positive correlation between them. On the contrary, when the highest values in the dissolved phase were recorded, the PAH concentrations in SPM were low. A possible explanation for this lack of correlation could be the high contribution of PAHs in colloidal form or in very fine particles, which are not retained in the filters as SPM, and a differentiation of PAH association according to particle size.

#### *Composition of PAHs –*

##### *Sources and transformation processes*

The study of the composition of PAH mixtures can provide useful information regarding the origin of these compounds and the transformation processes occurring in the water column. In the samples examined in this study no significant compositional differences among the stations were found and this is an additional confirmation that no obvious point

PAH sources existed in the area. For this reason the discussion will be focused only on the mean compositional patterns of PAH mixtures. These patterns in the dissolved phase and in SPM are illustrated in Fig. 5.

In the dissolved phase the distribution pattern of PAHs is dominated by phenanthrene (0.6-7.4 ng/l, mean 2.0 ng/l) followed by its mono- (0.1-7.8 ng/l, mean 1.7 ng/l) and di-alkylated derivatives (0.1-5.6 ng/l, mean 0.9 ng/l) and dibenzothiophenes, as well as by the lower MW and more water soluble compounds (Acenaphthylene, acenaphthene, fluorene). This predominance of the low MW PAH, common to all stations and sampling periods in both surface and bottom waters, is characteristic of uncombusted fossil fuel residues (SAEED & AL-MUTAIRI, 2000). The decreased values of the alkylated homologues of phenanthrene relative to the parent compound, which are inconsistent with a petroleum origin, are attributed either to a selective photolytic or biological degradation of the more labile alkylated PAHs (EHRHARDT & PETRICK, 1993). Similar composition of the dissolved aromatic material was also found in western and south-eastern Mediterranean Sea water (EHRHARDT & PETRICK, 1993, DACHS *et al.*, 1997). The concentrations of PAHs with four or more aromatic rings which are considered of clear pyrolytic origin, were in all cases one order of magnitude lower than the fossil fuel-derived PAHs (0.01-1.4 ng/l),

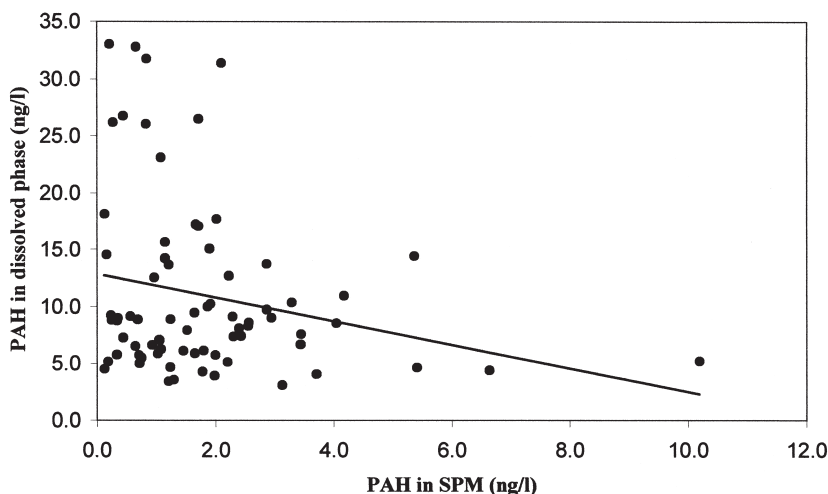


Fig. 4: Correlation between PAH concentration in the dissolved phase and in SPM.

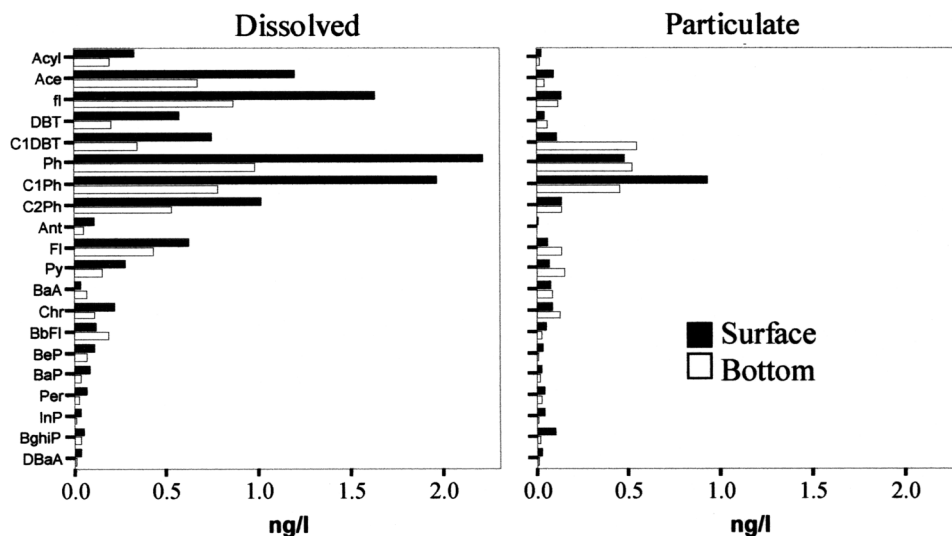


Fig. 5: Distribution patterns of individual PAHs (mean values) in dissolved and particulate phases.

whereas the most abundant compounds were fluoranthene, chrysene and benzofluoranthenes. PAHs of terrestrial – biogenic origin (retene, perylene) were detected in very small quantities, accounting in all cases for less than 1.5 % of the total PAHs.

The PAH distribution found in the SPM was slightly different from that in dissolved phase. Mono-alkylated phenanthrenes were the dominant compounds (0.01-7.8 ng/l, mean

0.8 ng/l) followed by phenanthrene (0.02-2.7 ng/l, mean 0.5 ng/l) and its dialkylated derivatives (0.01-0.5 ng/l, mean 0.1 ng/l). Pyrolytic PAHs presented very low concentrations and in many cases were undetectable. This composition pattern clearly indicates uncombusted fossil fuel origin for the PAHs, but it contrasts with that found in surface sediments in this area, where the pyrolytic higher MW PAHs are predominant



(HATZIANESTIS *et al.*, 1998). This contradiction is explained by the higher resistance of pyrolytic PAH towards bio- or photo- degradation during their downward transport through the water column and also by the solubilization processes of the low MW PAHs occurring during sedimentation, resulting in preferential accumulation of pyrolytic PAH in the sediments. The degradation of three-ring aromatics in the water column to a greater extent than pyrolytic PAHs is further indicated from the ratio fossil/pyrolytic PAHs (F/P) which shows a decrease with depth in all the stations (F/P mean values: 5.3 in surface and 3.5 in bottom waters for the dissolved phase and 6.7 in surface and 4.7 in bottom for SPM). These results emphasize the differential fate of PAHs according to their source.

## Conclusion

PAH concentrations in northern Aegean Sea water were slightly elevated and higher than those expected for open sea waters. Their spatial distribution was rather homogeneous and no significant seasonal variation was observed. PAH percentages greater than 80 % were found in the dissolved phase, which seems to play an important role in PAH transport. The lower MW PAHs predominated in both dissolved and particulate phases and this demonstrates a mainly fossil origin for the PAHs. The ship traffic in the area seems to be an important source of PAHs, whereas river runoff and coastal activities cannot be considered as major PAH suppliers.

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