

Hydrocarbon Biogeochemical Setting of the Baffin Island Oil Spill Experimental Sites. II. Water

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ABSTRACT. A baseline for petroleum residues in the waters of the Cape Hatt region of Baffin Island in arctic Canada was obtained in anticipation of controlled oil releases of the Baffin Island Oil Spill (BIOS) Project. Total aliphatic and aromatic residues reflecting multiple sources were found to be in the low to sub $\mu\text{g}\cdot\text{l}^{-1}$ concentrations range. Alkylated naphthalenes, phenanthrenes and dibenzothiophenes were present in the sub $\text{ng}\cdot\text{l}^{-1}$ concentrations range. The hydrocarbon baseline for the waters of the BIOS study area was found to be as low as might be found anywhere on earth and therefore ideally suited to the BIOS study.

Key words: BIOS, arctic marine Canada, hydrocarbon baseline, water

RÉSUMÉ. On a obtenu un niveau de référence pour les résidus de pétrole dans les eaux de la région du cap Hatt de l'île Baffin dans l'Arctique canadien, en prévision des déversements de pétrole faits sous contrôle pour le projet BIOS (projet de déversement de pétrole à l'île Baffin). Les concentrations des résidus aliphatiques et aromatiques venant de sources multiples étaient de l'ordre d'une fraction de $\mu\text{g}\cdot\text{l}^{-1}$ à quelques $\mu\text{g}\cdot\text{l}^{-1}$. Les naphthalènes alkylisés, les phénanthrènes et les dibenzothiophènes étaient présents dans des concentrations de l'ordre d'une fraction de $\text{ng}\cdot\text{l}^{-1}$. On a trouvé que le niveau de référence des hydrocarbures dans les eaux de la zone d'étude du projet BIOS présentait des concentrations qui sont parmi les plus faibles qu'on puisse trouver dans le monde, et convenait donc parfaitement à l'étude du projet BIOS.

Mots clés: BIOS, Arctique marin canadien, niveau de référence d'hydrocarbures, eau

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INTRODUCTION

This paper is part II in a group of three that appear together in *Arctic* and describe aspects of the hydrocarbon biogeochemistry of the Cape Hatt marine area of Baffin Island. It is concerned with the hydrocarbons borne by the sea water. The group of three papers forms part of a series in a special issue of *Arctic* that describes the Baffin Island Oil Spill (BIOS) Project.

The main goal of the studies described in this paper was to quantify the background levels of hydrocarbons in the Cape Hatt waters as a baseline for the subsequent experimental oil releases of the BIOS Project. A further goal was to investigate the composition of the hydrocarbons with a view to inferring the probable sources of the water-borne hydrocarbons. Of particular concern in this respect was the possible presence of hydrocarbons from seep oil such as that found only a short distance to the east in Buchan Gulf and Scott Inlet off the northeast coast of Baffin Island (Maclean *et al.*, 1981; Levy and Ehrhardt, 1981).

METHODS

Field Methods

Water Sampling: (Refer to Part I [Cretney *et al.*, 1987] for maps showing all sample locations.) In June 1980 samples were collected for all water chemistry work through holes in the ice at the north and south microbiology stations in each of the Bays 9 (H_5 and H_6), 10 (H_3 and H_4) and 13 (H_{1A} and H_{2A}). The ice varied from 1.5 to 2 m in thickness, and the samples were

collected at three depths (1, 5 and 10 m) measured from the bottom of the ice sheet. Water (4 l) for hydrocarbon analyses was obtained using an NBS sampler (Gump *et al.*, 1975) that had been modified by using a silicone rubber stopper wrapped with Teflon tape to provide a more positive seal. Freon 113 (75 ml) was added as a preservative and extractant. The lid of the sample bottle was screwed down tightly over a sheet of pre-extracted FEP Teflon (51 μm) giving an airtight, inert seal for storage.

In August and September 1980, samples were collected at the north and south microbiology stations in Bays 9 (H_5 and H_6), 10 (H_3 and H_4) and 11 (H_1 and H_2). The collection was done from an inflatable vessel at three depths (1, 5 and 10 m) from the surface using the modified NBS sampler as before. In addition, samples were collected in Bays 102 and 103, in the central part of Z-Lagoon and off "crude oil point". Two *in situ* water extractions were also carried out using a water extractor system based closely on the design of de Lappe *et al.* (1979). Water was drawn from 1 m depth in Bay 10 and 8 m depth in Bay 11, using a peristaltic pump situated on a raft at the water surface, through a series of pre-extracted polyurethane foam plugs in a glass cylinder held in a Teflon sleeve. In Bays 10 and 11, 210 l and 130 l respectively of water was measured at the effluent of the peristaltic pump. The extractions were considered to be semi-quantitative, however, when it was discovered that the connection of the extractor to the pump tubing tended to develop a leak on repeated flexion. Following water sampling, the glass fibre filters were folded, placed in an aluminum foil envelope and

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frozen. The glass cylinders holding the foam plugs were sealed at each end with a cleaned Teflon sheet and placed in a freezer.

On 21 August 1981 water samples (4 l) were collected using a modified NBS sampler from three depths (1, 5 and 10 m) at the north microbiology stations (H_3) in Bay 10. This collection was made prior to the dispersed oil release but after the surface oil release in Bay 11. In addition, using the same sampler a water sample was collected prior to the surface oil release on 19 August 1981 along the oil containment boom where it crossed the north biological transect. The water samples were preserved and extracted as before with Freon 113.

In 1981 *in situ* water extractions were carried out in Bay 9 (13 August), Bay 10 (14, 18, 23 August) and Bay 11 (12 August) using the same system as before except that water was forced through the filter and foam with a submersible pump (Teel model 1P681A or Little Giant model 3E 12WDVR). Depending on the time available, between 30 and 200 l of water were extracted.

Laboratory Methods

Extraction of Hydrocarbons from Sea Water Samples: Water samples (4 l) were extracted without filtration with Freon 113 (3×75 ml) in the field laboratory. The extraction was done in the sampling bottle with the FEP teflon sheet held firmly in place by the lid to provide a non-contaminating seal. Each increment of Freon 113 was thoroughly mixed with the water during 3 min agitation provided by a paint shaker. The solvent was drawn off using a stopcock arrangement that allowed the Freon to be drained off while admitting air via a glass tube to the space between the liquid and bottom of the bottle. The extracts were combined, reduced in volume to about 10 ml using a rotary evaporator and transferred to a threaded test tube. The lid was screwed down tightly over a sheet (51 μ m) of solvent extracted FEP Teflon, which provided a seal for storage and transportation. Arbitrarily selected sampling bottles were extracted as described above to provide blanks. The blank extractions were interspersed with the sample extractions.

Extractions of Glass Fibre Filters and Polyurethane Foam Plugs: Each *in situ* large-volume water extract sample consisted of a glass fibre filter and several polyurethane foam plugs. The glass fibre filters were cut into small pieces and extracted with dichloromethane:methanol (9:1) by the method described for surface floc in part I (Cretney *et al.*, 1987).

The polyurethane foam plugs were extracted in a soxhlet extractor for 24 h, first with methanol and then with dichloromethane:methanol (9:1). The solvent extracts were combined in a separatory funnel (1 l) and the dichloromethane layer was drawn off. The remaining aqueous solvent mixture was extracted with three 75 ml portions of dichloromethane. After the dichloromethane extracts had been combined and dried over anhydrous sodium sulfate, the solvent was reduced in volume to less than 1 ml on a rotary evaporator and displaced with hexane. Aliquots were evaporated and the residues weighed as before.

IR Analysis of Hydrocarbon Extracts from Sea Water: The extracts of water samples were rotoevaporated to near dryness and made up to a known volume with Freon 113. The intensity of the peak from the $-\text{CH}_2$ stretching frequency at 2930 cm^{-1} was measured using a grating infrared spectrophotometer (Perkin Elmer model 457). Peak intensities were quantified by comparison with a standard curve prepared from a solution of Lagomedio crude oil in Freon 113.

Liquid Chromatography of Hydrocarbon Extracts from Sea Water: Most of the freon extracts of water samples obtained in 1980 using the modified NBS samplers were analyzed by UV/F without chromatographic separation. Selected extracts, however, were fractionated by liquid chromatography prior to GC/FID or GC/MS analyses. The water samples obtained in 1981 using the same samplers were fractionated using liquid chromatography only if the total extractables were high. Extracts of the filters and polyurethane foam plugs from the *in situ* water extractor were always chromatographed prior to GC/FID or GC/MS/DS analyses.

The extracts of the water collected in the modified NBS samplers selected for GC/FID or GC/MS analyses were prepared for liquid chromatography in the following manner. The extracts were dried over anhydrous sodium sulfate and reduced in volume (to ca. 1 ml). The Freon 113 was then displaced with hexane, and androstane and o-terphenyl (3 μ g each) were added. The extractable organics concentration was determined as previously described by weighing the residues from aliquots.

Chromatographic separation of the water extracts was accomplished by silica gel/alumina chromatography (Cretney *et al.*, 1987).

UV/F Analysis of Water Extracts: The method used is the same as that for sediment extracts described in part I (Cretney *et al.*, 1987). In the case of the 1980 water extracts, however, API no. 2 fuel oil, rather than Lagomedio crude oil, was used as the calibration standard. Thus, the concentrations were calculated from the fluorescent responses of the sea water extracts as if the extracts consisted of API no. 2 fuel oil. The phrase "in API no. 2 fuel oil equivalents" is used to denote that this calculation was used to arrive at the concentrations.

GC/FID and GC/MS Analysis of Water Extracts: The method used is described in Cretney *et al.* (1987).

RESULTS

IR Analysis

Of the 35 water samples collected in 1980 and analyzed, only 7 (Table 1) yielded extracts with detectable carbon-hydrogen stretching absorbances in their infrared spectra. The remainder of the samples had concentrations equivalent to Lagomedio crude oil concentrations of $<13 \mu\text{g} \cdot \text{l}^{-1}$, the detection limit of the method.

UV/F Analysis

Four of 28 water samples collected in August and September 1980 and analyzed provided extracts having a fluorescence response exceeding or equal to the detection limit of $3 \mu\text{g} \cdot \text{l}^{-1}$ (in API no. 2 fuel oil equivalents). These four samples were collected on 26 August in Bay 10 at 10 m, on 18 August in Bay 102 at 1 m, on 17 August in Bay 103 at 10 m and on 18 August in Bay 103 at 7 m. The concentrations were 86, 3, 13 and $67 \mu\text{g} \cdot \text{l}^{-1}$ respectively.

None of the water samples that contained measurable quantities of material by the IR method also contained measurable quantities by the UV/F method. In the case of the extracts containing measurable organics by the IR method, the absence of a fluorescence response indicated that the organics were either biogenic in origin or, if petrogenic, represented a highly paraffinic, lowly aromatic residue. Uniformly low field blanks for the IR method indicated that random contamination of samples was unlikely, especially by fresh petroleum, which

TABLE 1. Concentration of extractable organics exceeding the IR detection limit in water samples collected in 1980

Date (1980)	Location (Bay)	Depth (m)	Concentration ($\mu\text{g}\cdot\text{l}^{-1}$) ^a
14 June	10	10	126
26 Aug	10	5	26
19 Sept	10	10	80
18 Sept	11	5	72
18 Sept	11	10	1138 ^b
18 Aug	102	4	62
20 Sept	103	1	150 ^c

^aIn Lagomedio crude oil equivalents.

^bMainly non-polar material (by liquid chromatography).

^cCollected following beach plot oiling.

would be expected to show a strong UV/F response. Sporadic contamination of the UV/F samples was also unlikely in view of the field blank results for that method. However, because the water samples were not filtered prior to analysis, those with fluorescent extractables, especially the three collected in deeper water, may have contained resuspended sediments.

Gravimetric and Capillary Gas Chromatographic Analysis

The three water samples collected in June 1980 in Bay 9 at 1, 5 and 10 m contained concentrations of extractable organics below the gravimetric detection limit ($5\ \mu\text{g}\cdot\text{l}^{-1}$) for the method of sampling and analysis used at that time. Qualitative GC/FID analysis indicated that the extracts were composed primarily of compounds likely of a marine biological (planktonic) origin. With an improvement in detection limit to $0.3\ \mu\text{g}\cdot\text{l}^{-1}$, mainly by increasing the extracted volume from 4 l to 20 l, the extracts from the samples collected in September could be measured gravimetrically (Table 2). The extracts, however, on chromatographic separation yielded f_1 and f_2 fractions, in which the organic constituents were then below the detection limit. The unfractionated extracts were found again to consist mainly of putative biogenic lipids. The most abundant compounds seemed to be wax esters and fatty acid methyl esters based on their mass spectral characteristics (e.g., base peak at m/z 74). The fractionated extracts generally yielded gas chromatograms indicative of hydrocarbon material of biological origin (i.e., olefines in f_2 as indicated by mass spectral characteristics as opposed to petroleum aromatics). In two samples, those from 1 m depth in Bays 10 and 11, the f_1 fraction yielded chromatograms dominated by a suite of *n*-alkanes in which neither those with an even number of carbon atoms nor those with an odd number were predominant. The chromatograms were similar to those obtained from tar balls or specks (Butler and Harris, 1975; Wong *et al.*, 1976).

TABLE 2. Concentrations of extractable organics exceeding the gravimetric detection limit in water samples collected in 1980

Depth (m)	Concentration ($\mu\text{g}\cdot\text{l}^{-1}$)		
	Bay 10 ^a	Bay 11 ^b	Z-Lagoon ^c
1	2.8	2.7	3.2
5	1.2, 0.5	3.4	2.7

^aCollection on 19 Sept 1980.

^bCollection on 17 Sept 1980.

^cCollection on 20 Sept 1980.

The results of the *in situ* large-volume water extractions in 1980 corroborated and extended those from the discrete water samples collected in 1980. Sufficient material for gravimetric analysis (detection limit $0.03\ \mu\text{g}\cdot\text{l}^{-1}$) of f_1 and f_2 fractions was obtained (Table 3). The filterable (dissolved/dispersed) polyurethane foam-sorbed portions of the samples and the filter-

TABLE 3. Concentrations of hydrocarbon-containing fractions f_1 and f_2 extracted from water *in situ* in 1980

Phase	Concentration ($\mu\text{g}\cdot\text{l}^{-1}$) ^a			
	Bay 10 ^b		Bay 11 ^c	
	f_1	f_2	f_1	f_2
Particulate ^d	0.6	0.7	0.7	0.6
Filterable ^e	1.4	2.0	2.0	1.1

^aGravimetrically determined; [$f_1(\text{gr})$], [$f_2(\text{gr})$].

^bCollection on 7 Sept 1980 at 1 m depth.

^cCollection on 11 Sept 1980 at 8 m depth.

^dRetained by Whatman GF/C filters.

^eExtracted from filtrate *in situ* with polyurethane foam.

retained (particulate) portions yielded distinctly different chromatograms. For example, the Bay 10 particulates yielded a chromatogram (Fig. 1) for the f_1 fraction that showed such features as an unresolved complex mixture (UCM), a predominance of *n*-alkanes with an odd number of carbon atoms over those with an even number and relatively similar amounts of the

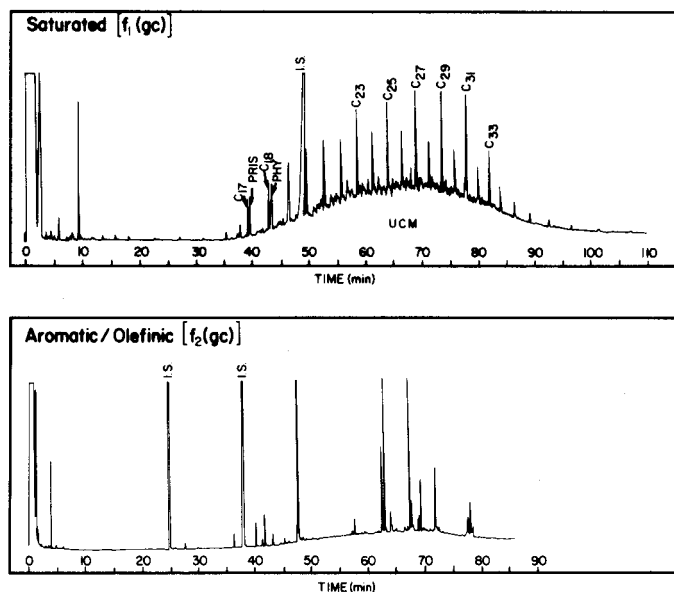


FIG. 1. Capillary gas chromatograms of liquid chromatography fractions f_1 and f_2 for particulate (filter-retained) material from a large-volume water extractor deployed in Bay 10. I.S. — internal standard.

isoprenoids pristane and phytane compared to the *n*-alkanes *n*-heptadecane and *n*-octadecane respectively. The chromatogram (Fig. 2) for the f_1 fractions of the filterable material was comparatively featureless at the detection limit of the method. In contrast, the f_2 fraction of the filter-retained material was virtually featureless, while the f_2 fraction of the filterable material showed the presence of a wealth of alkylated aromatic hydrocarbons.

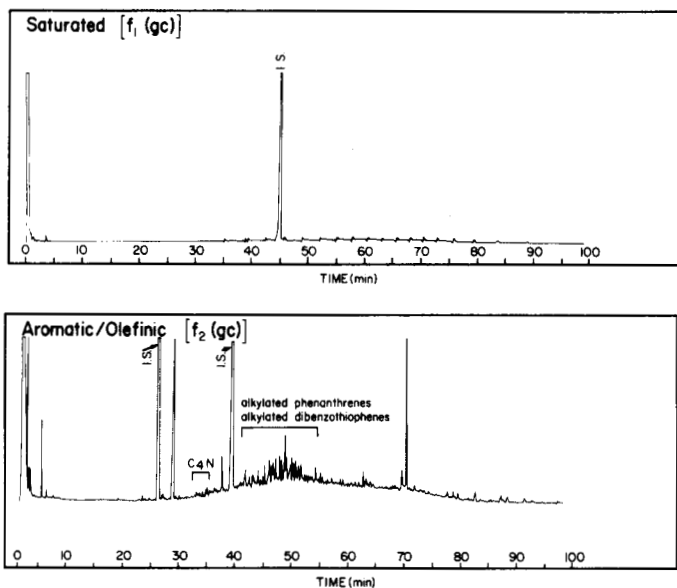


FIG. 2. Capillary gas chromatograms of liquid chromatography fraction f_1 and f_2 for filterable, foam-retained material from the large-volume water extractors deployed in Bay 10.

The gravimetric concentrations (Table 4) of the unfractionated extracts from 4 l water samples collected in 1981 were very similar to those (Table 2) found for the 1980 samples. The concentrations (sum of $[f_1(\text{gr})]$ and $[f_2(\text{gr})]$) (Table 5) of the filter-retained and filterable hydrocarbons for water samples extracted *in situ* also were similar to, though consistently lower

TABLE 4. Concentration of extractable organics in water samples prior to oil release in 1981

Depth (m)	Concentration ($\mu\text{g}\cdot\text{l}^{-1}$)	
	Bay 10 ^a	Bay 11 ^b
0-2	n.s.	0.5
2-4	1.3	n.s.
6-8	1.7	n.s.
9-10	2.6	n.s.

^aCollection at north micro station on 21 Aug 1981 at 2300 h.

^bCollection at north boom site on 19 Aug 1981 at 1500 h.

n.s. = no sample.

TABLE 5. Concentrations of hydrocarbon-containing fractions extracted from water *in situ* in 1981

Phase	Concentration ^a ($\mu\text{g}\cdot\text{l}^{-1}$)				
	Bay: 9	10	11	10	11
Particulate ^b	0.2	0.2	0.2	0.2	0.5
Filterable ^c	0.2	0.1	0.05	0.05	0.2

^aSum of fractions f_1 and f_2 , $[f_1(\text{gc})] + [f_2(\text{gc})]$.

^bRetained by Whatman GF/C filter.

^cExtracted from filtrate *in situ* with polyurethane foam.

than, those determined in 1980 (Table 3). The observed difference in the samples between 1980 and 1981 may in part be attributable to a difference in method. In 1981 quantification was by gas chromatography rather than by weighing.

The chromatograms of the f_1 and f_2 fractions of the filter-retained material collected in 1981 all showed a predominance of biogenic hydrocarbons with little indication of petroleum hydrocarbons. The chromatograms of the two lowest concentration, filterable (foam-retained) samples were similarly devoid of oil-like hydrocarbons, but those of the other three samples showed traces of what appeared to be heavily weathered oil hydrocarbons.

The GC/MS analyses of the filter-retained and filterable f_2 fractions for the 1980 Bays 10 and 11 samples confirmed the findings of the GC/FID analyses. For these samples, the filter-retained f_2 fractions were devoid of identifiable aromatic hydrocarbons at the detection limit of $0.05 \text{ ng}\cdot\text{l}^{-1}$. The filterable f_2 fractions contained a number of quantifiable aromatic compounds (Table 6).

TABLE 6. Concentrations of some aromatic compounds in the f_2 fraction from filterable, foam-retained organics extracted *in situ* in 1980

Compounds ^a	Concentration($\text{ng}\cdot\text{l}^{-1}$)	
	Bay 10	Bay 11
Tetramethylnaphthalenes	0.2	n.d. ^b
Methylphenanthrenes	0.3	0.05
Dimethylphenanthrenes	0.5	0.1
Trimethylphenanthrenes	0.15	n.d. ^b
Methyldibenzothiophenes	0.25	0.25
Dimethyldibenzothiophenes	0.4	0.1
Trimethyldibenzothiophenes	0.4	0.1

^aNamed group of compounds is principal contributor to signal, but other isomeric compounds are included.

^bn.d. = not detected; detection limit = $0.05 \text{ ng}\cdot\text{l}^{-1}$.

DISCUSSION

Although there may be some chronic inputs of petroleum residues in the study area from natural oil seeps discovered at Buchan Gulf or Scott Inlet, which are about 200 km and 400 km away by air respectively, or from nearer undiscovered ones, the present study does not provide evidence of such sources. An excellent baseline for petroleum residues in northern Baffin Bay (Levy, 1979; MacLean *et al.*, 1981) has been established by a UV/F method that differs from the one used in the present study, particularly in sensitivity. The differences unfortunately preclude a direct quantitative comparison of the Cape Hatt and Baffin Bay baselines. On the basis of all the quantitative and semiquantitative evidence from the water analyses, however, the BIOS study area waters in their natural state are as contamination free as most other marine sites that are remote from anthropogenic inputs (Clarke and MacLeod, 1977).

There is some qualitative evidence for petroleum residue input to the study area, but its source is uncertain. For example, the two discrete water samples from 1 m depth in Bays 10 and 11 whose f_1 chromatograms exhibited tar ball or tar speck characteristics may have originated from local seeps or from some distant tanker.

The presence of low levels of petroleum hydrocarbons in the waters of the area is also indicated by the results of the *in situ* filtration and extraction of large volumes of sea water. In the two 1980 samples the non-filterable (particulate) hydrocarbons seem to have at least two sources. The presence of a UCM, the near unity of the pristane:phytane ratio and the low abundance of hydrocarbons having 10-20 carbon atoms in the f_1 chromatograms all can be considered as indications of highly weathered petroleum residues (Fig. 1). The greater amount of *n*-alkanes with a chain consisting of an odd number of carbon atoms relative to those with an even number of the 20-30+ carbon range indicates a terrestrial biological source for a portion of the hydrocarbons. Taken together, the characteristics of the non-filterable hydrocarbons are reminiscent of those of eolian dusts in which there are contributions from terrestrial plant waxes and industrial petroleum spillage and combustion (Simoneit, 1977; Simoneit *et al.*, 1977; Marty and Salot, 1982). Of course, a combination of waterborne tar and local terrestrial plant waxes could account for the appearance of the chromatograms. Because of the sparseness of terrestrial plant life in the study area, the local input of plant waxes would likely be far smaller than that which occurs in temperate forested regions. Slower decomposition of the waxes in the arctic climate than in the temperate might compensate for the reduced input. Nevertheless, eolian transport of these plant waxes, along with urban pollutants, from temperate regions to the south must be considered as a real possibility.

The filterable hydrocarbons seem to point exclusively to a petroleum source. The moderately weathered appearance, the presence of a UCM and the relatively substantial portions of alkylated phenanthrene and dibenzothiophenes (Fig. 2) would be expected in chromatograms of the aromatic fraction of crude oil that had been at sea for a time. PAHs as combustion products are present, if at all, in only minor quantities compared to the petroleum aromatics. Hence there is no indication in the filterable hydrocarbon of eolian transport of combustion hydrocarbons into the waters based on the two large-volume water samples processed in 1980. Source decoupling of the non-filterable and filterable hydrocarbons, which seems to be required to explain the above results, has been observed before (Boehm, 1980).

Source decoupling seems also to explain the results from the samples collected in 1981, but with a difference. The non-filterable hydrocarbons are dominated by biogenic components to such an extent that there is no evidence of petroleum. Nevertheless, the filterable hydrocarbons of three out of the five samples display in their gas chromatograms the unmistakable characteristics of a heavily weathered oil. The major difference between the two years' results lies in the hydrocarbon composition of the non-filterable component. Month-to-month as well as year-to-year differences must be considered in searching for an explanation, because the samples were collected in early September in 1980 and in early August in 1981. Because the 1981 samples were collected soon after the major seasonal phytoplankton bloom, the non-filterable hydrocarbons might be expected to contain the dominant quantities of biogenic marine hydrocarbons observed.

CONCLUSIONS

On the basis of the hydrocarbon concentrations in sea water measured in 1980 and in 1981 before the deliberate oil releases, the burden of hydrocarbons was considered to be sufficiently low that the BIOS experiment could proceed without concern of compromising high background levels of petroleum residues. In particular, there was no quantitative evidence of large chronic inputs, such as undiscovered oil seeps, in the study area.

With respect to the sources of hydrocarbons, there was qualitative evidence of several. Of particular relevance to the BIOS Project was the finding of petroleum or petroleum-like hydrocarbon residues, the origin of which remains unclear. Nevertheless, these residues, as noted above, were present in such extremely low concentrations that it was felt they could not compromise the goals of the BIOS Project.

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