



Weathering of an Arctic oil spill over 20 years: the BIOS experiment revisited

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Abstract

A small experimental oil spill was conducted on the northern tip of Baffin Island Nunavut, Canada (72°31' N, 79°50' W) in August 1981, and the natural weathering of the oil has been followed by periodic visits. This paper reports on the chemical composition of oil collected in August 2001. The vast majority of the initial oil has gone, but small patches remain. Some samples remain essentially unaltered despite their 20 years of exposure to the elements, while others show that biodegradation and photooxidation can play important roles in removing the majority of the components of the oil. Using 17 α (H)21 β (H)hopane as a conserved marker within the oil, we show that the most biodegraded sample has lost more than 87% of the hydrocarbons initially present, while another has lost a substantial proportion of its initial chrysene and alkylated congeners. Potential explanations for the different weathering patterns seen in samples collected from this small site are briefly discussed.

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1. Introduction

The Baffin Island Oil Spill (BIOS) Project (Sergy and Blackall, 1987) offers an opportunity to follow the natural attenuation of an Arctic marine oil spill in the absence of any attempts at remediation. Approximately 15 m³ of lightly weathered (8% weight loss) Lago Medio crude oil was discharged onto the water adjacent to a shoreline on Cape Hatt, northern Baffin Island, Nunavut, Canada (72°31' N, 79°50' W) in August 1981. About 45% of the oil stranded on the previously pristine adjacent beach (Cretney et al., 1987), and this subsequently weathered by natural attenuation without any efforts at cleanup. By 1989 there had been an approximately 80% decrease in the total oiled area (Owens et al., 1994), and this has continued to decrease until in the survey discussed here we estimate that coverage had decreased to <5% of the initial area.

The beach is exposed to waves with a fetch of <10 km, and is subject to low wave-energy levels with wave

heights typically <10 cm during the open-water season (average 63 days per year, Owens et al., 1994). Gross physical loss of oil by heavy wave action is thus an unlikely mechanism to explain the gradual loss of oil. One explanation is “oil–mineral fines interaction”, wherein oil interacts with mineral particulates to form neutrally buoyant micron sized aggregates that wash away (Owens et al., 1994). Obviously any oil that is lost by this mechanism is not readily available for analysis, although it might potentially be trapped in sediment traps. In contrast, other likely mechanisms for oil loss, such as evaporation (Fingas, 1995), dissolution, biodegradation and photooxidation, leave characteristic evidence of their occurrence in the chemical composition of the residual oil. For example, aromatic hydrocarbons are more soluble than aliphatic ones of similar molecular weight (Yaws et al., 1993), and tend to wash out preferentially. The biodegradation of oil has been studied extensively, at least under aerobic conditions, and the preferential degradation of linear before branched alkanes, and smaller before larger aromatic compounds, is well established (Prince, 2002). In contrast, photooxidation seems to only affect the aromatic compounds in the oil (Garrett et al., 1998). Thus if one has a

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Table 1
Samples collected from Bay 11, the open-water spill site. Last two columns from Wang et al. (1995)

Sample number	Description	G methylene chloride extractable material/kg sediment	% Depleted, based on hopane in GC-detectable oil	1993 sample number	% Depleted based on hopane in extracted oil
1	Coated surface granules	0.7	88	2	21
2	Soft pavement	6.1	57	10	12
3	5 cm below soft pavement surface	3.0	8	10	12
4	Black shiny oiled sediments	2.7	68	11	23
5	Floating oil from small pit	–	38	–	–
6	Floating oil from small pit	–	15	8	15

sample of the initially spilled oil, it should be possible to determine the contributions of these different weathering processes in samples collected after the spill, and project estimates of the likely fate of residual hydrocarbon into the future.

One important analytical method is gas chromatography coupled with mass spectrometry, particularly using selected ion monitoring for species of particular interest (Douglas et al., 1992; Wang et al., 1995). This technique identifies and quantifies individual hydrocarbons in the residual oil. Furthermore, rather than relying on absolute quantitation of species of interest, the relative concentration of these species with respect to a conserved internal marker within the oil leads to tighter analytical precision for understanding losses (Prince et al., 1997). We have shown that $17\alpha(H)21\beta(H)$ hopane is a useful conserved internal marker for following the biodegradation (Prince et al., 1994) and photooxidation (Garrett et al., 1998) of crude oils, and we use it here.

Another valuable tool is thin layer chromatography (Ackman et al., 1990), since this identifies the major fractions of the oil: the saturates, the aromatics, the resins and the polars. Together these two techniques allow a view of both the molecular and bulk constitution of the oil.

We find that both photooxidation and biodegradation have played a substantial role in the weathering processes of the 20 years since the spill. This is an extreme environment, where the daily annual mean air temperature is $-14.7\text{ }^{\circ}\text{C}$, and even during the open-water months of August and September it is $<5\text{ }^{\circ}\text{C}$ (Meeres, 1987). In fact, the Bay 11 intertidal zone is encapsulated by a solid and static ice foot for approximately 10 months each year and so is exposed to atmospheric and marine processes for only approximately 60 days each open-water season (Dickins, 1987). Over the period 19 August 1981 to 23 August 2002 this totals a period of approximately 40 months when weathering processes were active on the stranded oil. In terms of sunshine, this location experiences approximately 200 h of sunshine during the period when the beach is ice-free and the oil is exposed to the atmosphere. This is equivalent to a total of 11 months of days with 12 h of

continuous sunshine since the spill. Nevertheless, the maximal total extent of weathering, principally biodegradation, has been at least an 87% loss of hydrocarbon compared to the initially spilled oil. Another sample, more exposed to sunlight, has lost approximately half of its chrysene (and mono- and di-methyl forms) by photooxidation.

2. Materials and methods

Four sediment and two pit-shoen samples were collected from still oiled parts of the intertidal zone of the oiled beach (Beach 11) in August 2001 (Table 1). Most were collected near to where samples were collected in August 1993 (Wang et al., 1995). Four additional samples were collected from backshore control plots in Z lagoon (see Sergy and Blackall, 1987, Table 2). On one of these (7) the oil had been applied as a 1 cm thick layer of weathered crude oil in 1980 (T1, see Sergy and Blackall, 1987), and on another (8) the oil had been applied in 1980 as a 2 cm thick layer of this oil emulsified with an equal volume of water (T2). Samples were collected from 2–5 cm beneath the surface layer. These plots have not been subject to tidal flooding since application of the oil. Two others came from the surface (9) and subsurface (10) of a small berm where oil had been applied in August 1982 to the top part of the intertidal zone and mixed as part of a treatment experiment (IMC, see Sergy and Blackall, 1987).

Table 2
Samples collected from Z Lagoon, the protected spill site

Sample number	Description	G methylene chloride extractable material/kg sediment	% Depleted, based on hopane in GC-detectable oil
7	2–5 cm	4.5	12
8	2–5 cm	5.8	4
9	Soft pavement surface	8.4	49
10	1 cm below 9	3.6	26

Sediment samples were mixed with anhydrous sodium sulfate to a free-flowing state, extracted three times with methylene chloride, dried with anhydrous sodium sulfate, and concentrated to approximately 10 mg extractable material/ml methylene chloride. Sheen samples were collected onto absorbent paper towels, which were subsequently extracted with methylene chloride as the sediment samples. A sample of the initially spilled oil (Wang et al., 1995) and a more recent sample of Lago Medio crude oil were a generous gift from Pat Lambert at Environment Canada, Ottawa.

Gas chromatography and mass spectrometry in total scan and selected ion monitoring modes were carried out essentially as described by Douglas et al. (1992), using a Hewlett Packard HP 5890 gas chromatograph fitted with a 30 m × 0.25 mm fused silica capillary column with 5% crosslinked phenyl methyl silicone as the stationary phase. Helium was used as the carrier gas at a flow rate of 1 ml/min. Mass spectral data were obtained with a Hewlett Packard 5972 mass selective detector at an electron energy of 70 eV. Spectral tuning with Perfluorotributylamine followed USEPA method 8270C.

Thin layer chromatography used 0.9 mm Chromarod quartz rods sintered with silica gel. Samples were applied in methylene chloride, and chromatographed in *n*-hexane for 35 min, toluene for 15 min, and 95% methylene chloride, 5% methanol for 2 min. Chromatographs were analyzed on an Iatroscan MK-5 flame ionization detector (Iatron Laboratories, Tokyo, Japan).

3. Results

The sediment samples contained significant amounts of oil, as shown in Tables 1 and 2. Even cursory inspection of the total ion gas chromatograms (Fig. 1) reveals that some samples, such as 1, 2 and 5, are extensively degraded, while others, such as 3 and 8 still seem remarkably “fresh”. Crude oils contain biomarkers (Peters and Moldowan, 1993) that are the molecular fossils of the original biomass that gave rise to the oil. Figs. 2 and 3 show chromatograms of the terpanes (Peters and Moldowan, 1993) and steranes (Peters and Moldowan, 1993) found in Lago Medio oil. As expected, the most degraded samples (such as sample 1 in this study) show evidence that some of the terpanes and steranes are biodegraded (Peters and Moldowan, 1993; Wang et al., 1995), but two other important facts are also revealed. The first is that the C₂₉ and C₃₀ hopanes show no evidence of biodegradation, and thus can serve as conserved internal markers in the oil, and the second is that there is no evidence for any additional source of petroleum in these samples beyond the initially spilled oil. This latter finding is important, because the samples were collected more than 20 years after the initial spill,

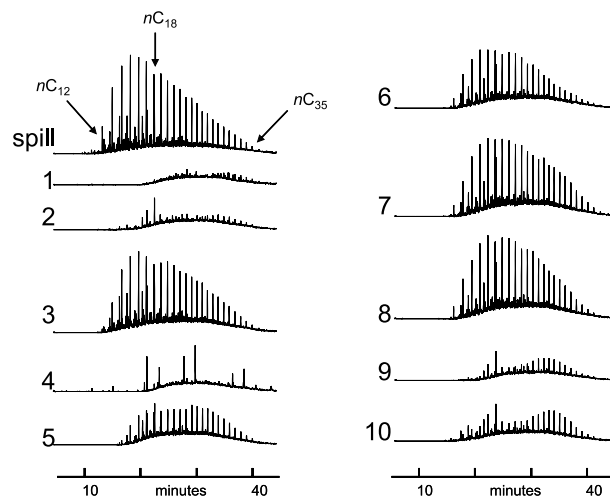


Fig. 1. Gas chromatograms (total ion mass detection) of the samples collected on Baffin Island in September 2001 (Table 1). The prominent peaks in the spill sample are principally the *n*-alkanes, and three are identified. They are the dominant features in most of the samples, except that the peaks in sample 2 are principally petroleum isoprenoids, while those in sample 4 are biogenic waxes.

and it was certainly possible that the site might have received some other source of petroleum in this time.

Since 17 α (H)21 β (H)hopane showed no evidence of biodegradation, and in laboratory experiments we have shown that it is not generated during oil biodegradation (Prince et al., 1994), we have used it as a conserved internal marker within the oil. We can calculate the percent depletion of other analytes within the oil using the equation:

$$\%Loss = [((A_0/H_0) - (A_s/H_s))/(A_0/H_0)] \times 100$$

where A_s and H_s are the concentrations of the target analyte and hopane in the oil sample, respectively, and A_0 and H_0 are the concentrations in the initially spilled oil. We have used the sample of initial oil provided by Environment Canada, and it is possible that this sample from 1981 has undergone additional evaporation since it was initially artificially weathered, in which case our estimates of the percent loss of volatile components will be underestimates. Indeed our calculations suggest that the stored sample is now some 22% depleted in total detectable hydrocarbons and total alkanes with respect to the more recent sample of whole oil, instead of the reported 8% (Sergy and Blackall, 1987). Nevertheless, the ratios of alkanes larger than *n*C₁₅, and the three and four ring polycyclic aromatic hydrocarbons discussed here, with respect to hopane are identical in the two samples. Since these are the major focus of our work we are confident that if anything we have erred on the conservative side in calculating the percent depletion of hydrocarbons within the residual oil. We note that if some 17 α (H)21 β (H)hopane had been degraded in the

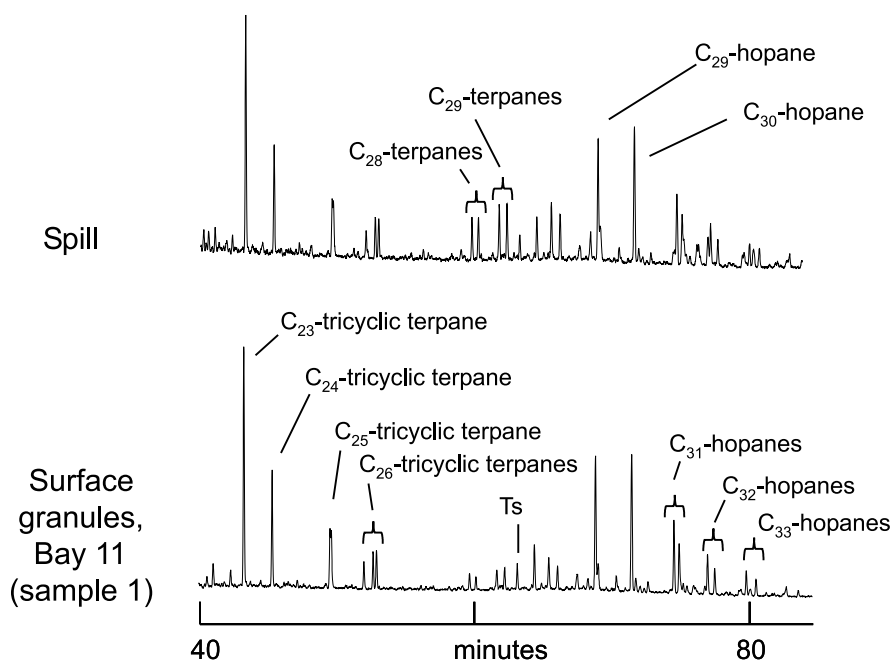


Fig. 2. Terpane biomarkers in the initially spilled oil, and in the most biodegraded sample. Scales normalized to equal concentrations of $17\alpha(H)21\beta(H)$ hopane. A full discussion of the origins of these complex molecules can be found in Peters and Moldowan (1993). Here it suffices to say that there is no evidence that the C_{29} and C_{30} hopanes are degraded, although the C_{28} and C_{29} terpanes are clearly much reduced in the biodegraded field sample. $m/z = 191$ chromatograms.

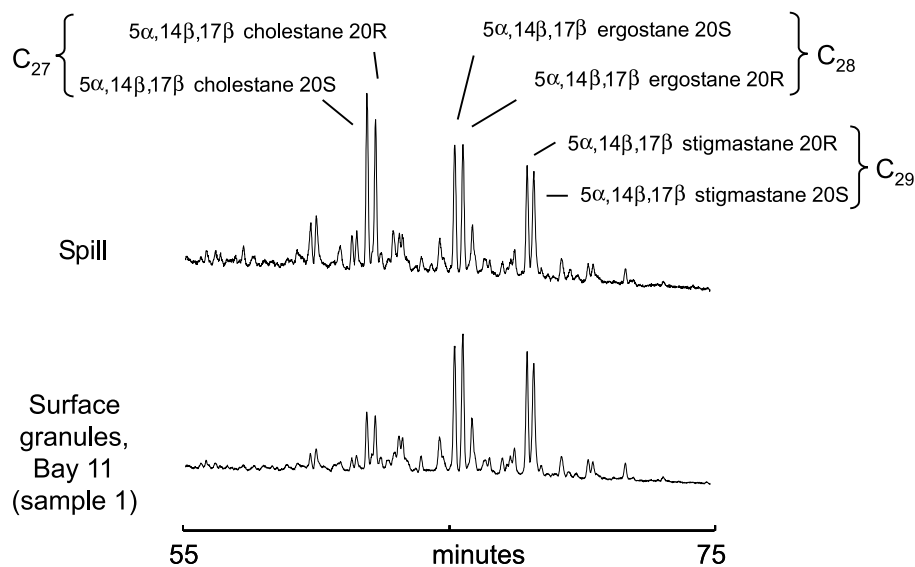


Fig. 3. $\alpha\beta$ -sterane biomarkers in the initially spilled oil, and in the most biodegraded sample. Scales normalized to equal concentrations of $17\alpha(H)21\beta(H)$ hopane. A full discussion of the origins of these complex molecules can be found in Peters and Moldowan (1993). Here it is sufficient to say that there is no evidence that the C_{28} and C_{29} forms are degraded, although the C_{27} forms are clearly much reduced in the biodegraded field sample. $m/z = 218$ chromatograms.

samples collected from the site, our estimates of the extents of loss would be underestimates.

Before addressing individual hydrocarbons, however, we first address the major classes of compounds in the spilled oil. Crude oils are complex mixtures of thousands of hydrocarbons, together with resins and polar molecules that contain heteroatoms (Tissot and Welte, 1984).

Thin layer chromatography of the initially spilled oil indicates that 47% is aliphatic hydrocarbons and 25% is aromatic hydrocarbons, with the remainder a mixture of resins and polars (Table 3). In concert with the gas chromatograms, some samples seem to have an essentially unaltered composition (e.g. 8), while others, such as 1, are very different. It is important to note that these

Table 3

Composition of the extracted oils, determined by thin layer chromatography

Sample number	Saturates	Aromatics	Resins	Polars
Initial	47	25	20	8
1	12	23	44	22
2	36	17	30	16
3	43	36	14	7
4	23	14	46	17
5	37	37	17	9
6	40	39	14	6
7	42	33	19	7
8	43	30	20	7
9	37	16	32	15
10	39	38	14	8

data represent the composition of the current oil samples. Our understanding of biodegradation (Prince, 2002) leads us to expect that the saturates and aromatics will be biodegraded most rapidly. Their contribution will therefore decrease as oil biodegradation proceeds, and so the resins and polars will increase in relative concentration. This pattern seems particularly clear in sample 1. The resins and polars may also increase in absolute amount due to the generation of metabolic intermediates, or the accumulation of biomass.

Photooxidation of crude oils has a different characteristic effect on the thin layer chromatographic properties of an oil; the saturates are unaffected, but the aromatics are converted to resins and polars (Garrett et al., 1998). The data suggest that this has occurred in samples 2, 4 and 9.

Gas chromatography coupled with mass spectrometry allows a molecular analysis of the oil samples. We have focussed on the linear and branched alkanes because these are the most abundant species in the oil (making up some 6.6% of the sample of the initial oil sample), and the three- and four-ring polycyclic aromatic hydrocarbons and their alkylated congeners, because these are on the USEPA list of priority pollutants (Keith and Telliard, 1979).

Fig. 4 shows the percentage loss of octadecane (nC_{18}), phytane (2,6,10,14-tetramethylhexadecane) and triacontane (nC_{30}) as representative alkanes. All three are biodegradable, but the n -alkanes are typically degraded preferentially to the branched alkanes, and shorter alkanes are typically degraded preferentially to the longer ones (Prince, 2002). This pattern is seen here in all the samples except 7 and 8, where there has been very little depletion of any of these compounds. Samples 1, 2, 4 and 9 show the most depletion. The one unexpected result is that sample 6, a sheen sample collected from a small pit dug in the beach has lost a greater fraction of its triacontane than of its octadecane.

Fig. 5 shows the percentage loss of phenanthrene, a three-ring aromatic compound, and its alkylated congeners. The identification of four methyl groups, for ex-

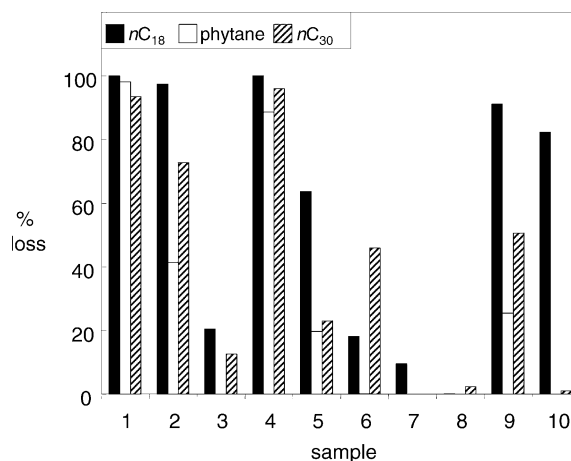


Fig. 4. The loss of representative alkanes in the different samples, using hopane as a conserved internal marker.

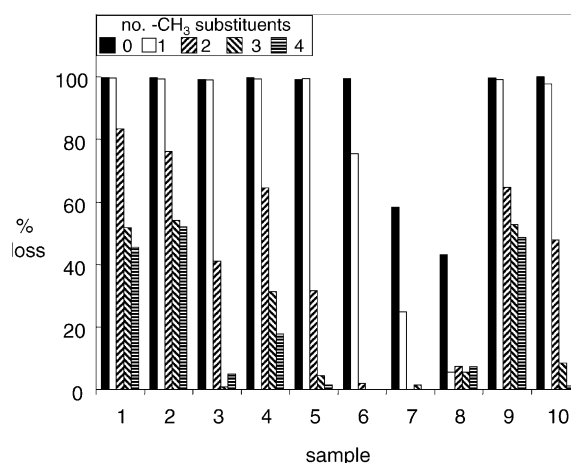


Fig. 5. The loss of phenanthrene and its alkylated forms in the different samples, using hopane as a conserved internal marker.

ample, includes tetramethyl-, ethyl-dimethyl-, propyl-, methyl- and butyl- forms. The unsubstituted parent is typically biodegraded most readily, and increasing alkylation slows biodegradation. This pattern is generally seen in all the samples, but it is also likely that the smaller molecules may have washed out of the oil, or even have evaporated. We believe this is the likely explanation for samples 3, 6, 7 and 8, because the loss of the rather less soluble alkanes (Yaws et al., 1993) is so much less in these samples. This contrasts with the extensive loss of both phenanthenes and alkanes in samples 1, 2, 4 and 9, which are consistent with extensive biodegradation, perhaps with some photooxidation. Very similar depletion patterns were seen for dibenzothiophene and its alkylated forms. This is consistent with findings in other marine environments (Douglas et al., 1996) that the biodegradation of these two families of compounds is so similar that ratios of, for example, trimethylphenanthenes to trimethyldibenzothiophenes

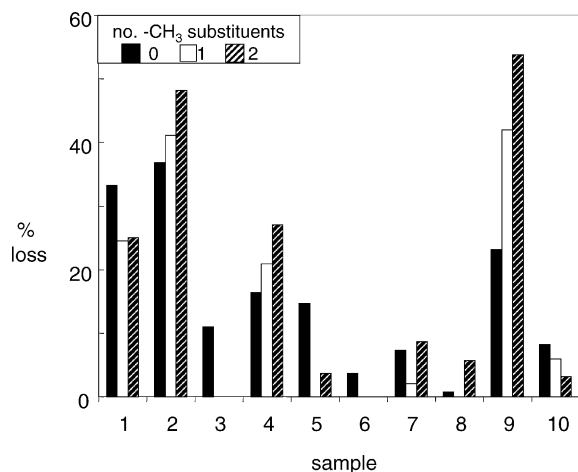


Fig. 6. The loss of chrysene and its alkylated forms in the different samples, using hopane as a conserved internal marker.

remain diagnostic fingerprints even in heavily degraded samples.

Fig. 6 shows the percentage loss of chrysene, a four-ring aromatic compound, and its alkylated congeners. Losses are much less than seen for the phenanthrenes, and samples 3, 6, 7 and 8 have lost very little if any of these compounds, consistent with the lower solubility and volatility of these compounds compared to the phenanthrenes. There are two clear patterns of loss in the other samples. The pattern exhibited by sample 1 is akin to that seen for the phenanthrenes, with the parent being the most depleted. This is the pattern expected for biodegradation (Prince, 2002). In contrast, samples 2, 4 and 9 show a reverse pattern, with the most loss of the more alkylated forms. This is the pattern seen with photooxidation (Garrett).

4. Discussion

After 20 years of “exposure to the elements”, some of the samples collected from the BIOS site are extensively biodegraded. Sample 1, from surface granules from the intertidal zone of Beach 11 is the most degraded sample we collected; it has lost 87% of the total hydrocarbons present in the sample of the initially spilled oil, and 92% of the saturates (measured as the m/z ion = 57). It has lost (Fig. 4) essentially all of its initial octadecane, 98% of its phytane and 93% of its initial tricontane ($nC_{30}H_{62}$). It has also lost (Fig. 5) essentially all of its initial phenanthrene and dibenzothiophene and even trimethyl forms are approximately 50% depleted. Perhaps surprisingly, there has been no apparent isomer-preference in the degradation of the alkylated forms; within a given mass range, all isomers are depleted to very similar extents. As shown in Fig. 6, there has also been substantial loss of chrysene and its alkylated forms up to at least dimethyl. Because the parent is more de-

pleted than the alkylated forms, we ascribe the loss to biodegradation (Prince, 2002) rather than photooxidation. As noted by Wang et al. (1995) in some of the samples collected in 1993, there has also been extensive loss of some of the terpane and sterane biomarkers (P&M) in some samples collected from the field. This is exemplified in Figs. 2 and 3, where it is apparent that more than 60% of the C_{28} and C_{29} terpanes and the C_{27} steranes have been lost from the most biodegraded sample.

Samples 2 and 4 were also collected from the surface of the intertidal zone, and they are also extensively depleted of individual hydrocarbons. The pattern of greater loss of n -alkanes than of phytane (Fig. 4) is the pattern expected of biodegradation (Prince, 2002), and the loss of phenanthrenes (Fig. 5) is also that expected for biodegradation. Nevertheless, the loss of chrysenes suggests that these samples have also undergone photooxidative loss of polycyclic aromatic hydrocarbons. Both these samples had a higher concentration of methylene chloride extractable material than sample 1, and the different extents of biodegradation can perhaps be attributed to the fact that the nutrient levels (especially biologically available nitrogen and phosphorus) of Arctic coastal waters (Rysgaard et al., 1999) are likely to limit biodegradation. This effect will be more apparent in more oiled sediments, and indeed the degradation is apparently inversely proportional to oil content (Table 1). Sample 4 was “black and shiny” possibly because of active “plucking” by clay particles that keep the surface “fresh” in appearance, as has been observed elsewhere (Owens et al., 1994).

We note that while the thin layer chromatography (Table 3) of all three of these samples suggests that they have lost saturate and aromatic components, it is clear that the changes are smaller than might be expected based on the losses measured by gas chromatography. For example sample 1 has lost approximately 90% of its saturated molecules based on gas chromatography, and the use of hopane as a conserved internal marker, yet the proportion of saturates in the total oil has only decreased by 75%. This implies that some resins and polars have also been lost, either by biodegradation or by dissolution. We cannot distinguish between these possibilities with our current data.

Despite the extensive biodegradation seen in some samples, others contain essentially unaltered oil. Samples 7 and 8, which came from a plot above the high tide mark are the least degraded, together with sample 3, which came from 5 cm below the surface of a soft pavement in the intertidal zone. The loss of phenanthrenes from these samples may perhaps be attributed to the solubility and/or volatility of these compounds (sample 3 was much more frequently washed than samples 7 and 8), and none of these samples show evidence of photooxidation. We attribute this preservation

to the lack of nutrients for microbial growth; samples 7 and 8 were rarely inundated, and sample 3 received water that had perhaps already been depleted of nutrients by microbes in the oiled surface zone.

Samples 5 and 6 were collected as floating sheens on small pits dug in obviously oiled parts of the intertidal zone. They show evidence of limited biodegradation, and also seem to be depleted of resin and polars (Table 3), suggesting that these components may have been left behind as the oil was dislodged from the subsurface to float on the interstitial water.

Samples 9 and 10 come from a small sandy berm in a very sheltered lagoon; they both show evidence of biodegradation in the patterns of loss of the alkanes and phenanthrenes (Figs. 4 and 5), and the surface sample (9) shows evidence of extensive photooxidation. We attribute the greater biodegradation in these samples to the fact that they received more frequent tidal inundation than samples 7 and 8.

In conclusion, it is clear that biodegradation can be a major fate of spilled crude oil on an Arctic beach, even though biological processes are probably restricted to an average of only 63 ice-free days per year. Recent work suggests that low temperatures (0–7 °C) can still facilitate oil biodegradation by indigenous organisms (Gibb et al., 2001; Eriksson et al., 2001), and a more important limitation may instead be the availability of the necessary trace elements for microbial growth. This in turn suggests that a bioremediation strategy would stimulate oil biodegradation, as has been shown on a shoreline in Spitsbergen (Prince et al., 1999), and in soils (Eriksson et al., 2001; Mohn et al., 2001). It seems likely that the fate of the residual oil that does not leave the shorelines by physical transport will eventually be biodegradation, but that this will continue to be severely limited by the availability of trace nutrients required for microbial growth. There is still scope for further photooxidative loss of polycyclic aromatic hydrocarbons in samples exposed to sunlight.

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