

**Characterisation of weathered hydrocarbon wastes at contaminated sites
by GC-simulated distillation and nitrous oxide chemical ionisation GC/MS,
with implications for bioremediation**

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Contents list entry

Weathered hydrocarbon wastes at five hydrocarbon-contaminated sites in Alberta are characterised by preparative chromatography, GC-SIMDIS and CI GC/MS. Asphaltene and polar classes predominate and recalitrant C₁₄-C₂₂ 2-4 ring alicyclics, alkylbenzenes and benzocycloparaffins dominate the saturate class composition. Implications for bioremediation are discussed.

Summary

An extended analytical characterisation of weathered hydrocarbons isolated from historically contaminated sites in Alberta is presented. The characterisation of soil extracts, chromatographically separated into component classes, by GC-simulated distillation and

nitrous oxide (N₂O) chemical ionisation (CI) GC/MS provides new insights into the composition of the residual oil at these sites, the principal partition medium for risk critical components. The combined polar and asphaltene content of representative soil extracts ranged from 40%^{w/w} to 70%^{w/w} of the oils encountered. An abundance of C₁₄-C₂₂ 2-4 ring alicyclics, alkylbenzenes and benzocycloparaffins is prevalent within the saturate fractions of site soils. Implications for the partitioning of risk critical compounds at sites with weathered hydrocarbons and the practical application of bioremediation technologies are discussed.

Introduction

The identification of soils that have become contaminated with hydrocarbons through accidental spillage and gradual inventory loss often occurs when industrial facilities are identified for decommissioning, sale or redevelopment¹⁻². The more toxic components of hydrocarbon residues drive the health and environmental risk assessments that inform decisions on site remediation^{1, 3-4}. Historic hydrocarbon contamination long in the ground will have often weathered through a range of physical, chemical and microbiological processes. The complexity of weathered mid-distillate and heavy oil residues is widely acknowledged⁵⁻⁸ and is evidenced at sites where they are encountered by (i) the heterogeneity of the oil-soil matrix; (ii) the extensive analytical cleanup procedures required for the quantification of contaminants in these matrices; and (iii) the unresolved gas and ion chromatograms that analytical chemists invariably elicit from soil extracts⁷.

Research into heavy oils at contaminated sites has been sparse. Research efforts need to extend beyond simply quantifying the concentrations of risk critical compounds (*e.g.* the polynuclear aromatic hydrocarbons (PAH)) for risk assessment purposes to a broader analysis of the oil matrix. This is because the partitioning behaviour that drives the environmental fate of individual compounds is largely determined by the affinity of the residual weathered

oil for priority compounds; a property that changes with the type of oil (the ‘source term’) and degree of weathering it experiences. In our attempts to better understand the characteristics and environmental behaviour of these weathered hydrocarbon wastes in soils, we have reported on the composition⁹ and relative toxicities¹⁰ of a range of chemical classes isolated from soil extracts at petroleum and creosote-contaminated sites, the development of tiered strategies for the analysis of weathered oils¹¹ and the performance of a suite of biomarker indices as indicators of the source and progressive biotransformation of oily wastes¹². We have demonstrated (i) that the analysis of the class composition (*e.g.* saturates, aromatics, polars, asphaltenes) of solvent extracts at hydrocarbon-contaminated sites provides valuable insights into the potential biotreatability of oily wastes⁹; (ii) that the residual oil phase is the principal partition medium for PAH in hydrocarbon-contaminated soils¹³; and (iii) that biomarker indices comprising hopane pairs (*e.g.* the ratio of 17 α (H)21 β (H)-norhopane to 17 α (H)21 β (H)-hopane) within the saturate class can be used (a) to track biotransformation; or (b) as source identification indices for weathered oils¹².

Aim of investigation

Despite these and other insights and the frequent occurrence of weathered oils at contaminated sites, our understanding of how weathered mid-distillate and heavy oils behave in the environment remains incomplete. In part, this is a consequence of standardised analytical procedures used for the analysis of priority substances in soils sampled during site investigation, which tends not to require in-depth characterisation of the oil matrix in which these substances reside. The unresolved complex mixtures (UCM) characteristic of weathered mid-distillate and heavy oils¹⁴⁻¹⁶ mask a complexity of composition rarely elucidated using conventional electron impact (EI) gas chromatography/mass spectrometry (GC/MS).

1 In this study, we apply preparative chromatography and specialist GC and GC/MS
2 techniques to an extended characterisation of weathered oily wastes (Fig. 1). We present a
3 qualitative analysis, using simulated distillation GC¹⁷ (GC-SIMDIS) and nitrous oxide (N₂O)
4 chemical ionisation GC/MS¹⁸ (CI-GC/MS) of the chemical classes that comprise
5 chromatographically separated fractions of soil extracts at five historically contaminated sites
6 in Alberta, Canada. Simulated distillation GC allows the elucidation of the effective carbon
7 number (EC_n) distribution of the component classes of contaminated soil extracts by using a
8 non-polar GC column as a surrogate distillation column from which analytes are ‘distilled’
9 using a linear temperature profile. Chemical ionisation GC/MS, in which gaseous atoms of
10 the analyte are bombarded in a mass spectrometer with ions produced from the electron
11 bombardment of a reagent gas (here, N₂O), allows construction of parent (molecular) ion
12 chromatograms and, with reference to our previous work^{9, 11}, the analysis of the ‘fine’
13 composition of individual chemical classes separated by preparative chromatography.

14 The results presented are relevant in the context of the recent interest in establishing
15 soil assessment criteria for the heavier hydrocarbons and, more broadly, in setting realistic
16 remediation objectives for hydrocarbon-contaminated sites¹⁻². Our analysis further informs
17 the source characterisation of these difficult wastes and our understanding of their candidacy
18 for bioremediation.

19 20 **Materials and experimental procedures**

21 Sample preparation and analysis were performed according to the schedule set out in Fig. 1.

22 **Soil sampling and preparative soil extract fractionation**

23 We have previously described the sampling of residual hydrocarbon contamination from soils
24 at five historically contaminated hydrocarbon waste sites in Alberta⁹. Briefly, surficial
25 (0-0.25m depth) soils (*ca.* 5kg) from the process areas of four former petroleum refineries

(site I-IV) and a former coal-tar creosote wood treatment facility (site V) were sampled using a hand held soil auger, sieved to below 2.38mm (< ASTM No. 8) and stored at 4°C prior to analysis. Air-dried (20±2°C, overnight), cone and quartered sub-samples (ca. 30g) of grab sampled soils contained between 2.4%^{w/w} (typically ±0.2%^{w/w} % RSD) and 5.4%^{w/w} dichloromethane (HPLC grade DCM; 16h) extractable organic matter as gravimetrically determined by Soxhlet extraction to exhaustion (Fig. 2).

DCM extracts were chromatographically separated (Fig. 2) into six component class fractions (saturates, mono-, di- and polyaromatics, polars and asphaltenes) using an established technique¹¹; the fractions being designated S, A_m, A_d, A_p, P and A, respectively (Fig. 2). Asphaltenes (A) were gravimetrically recovered from separate portions of DCM extracts using *n*-pentane^{9, 12} precipitation. A stainless steel chromatography column (137cm x 0.94cm) was then pre-packed in its lower length with 42g Davidson grade 12 silica (28-200 BS mesh) activated at 250°C for 20h. The top half was packed with 47g Alcoa F-20 alumina (80-200 BS mesh) activated at 400°C for 20h. Preparative class fractions were obtained and quantified using the following series of eluents;

- for saturates (S): 150mL *n*-pentane;
- for monoaromatics (A_m): 250mL 5%^{v/v} toluene in *n*-pentane;
- for diaromatics (A_d): 300 mL 30%^{v/v} toluene in *n*-pentane;
- for polyaromatics (A_p): 300mL 70%^{v/v} toluene in *n*-pentane; and
- for polars (P): 300mL 50%^{v/v} methanol in DCM.

Pre-weighed glass beakers were used to collect eluates. The solvent was then removed by air-drying in a fume hood (16h) and the gravimetric recoveries (typically ±5%RSD⁹) for each class fraction estimated (Fig. 2). A well characterized Athabasca bitumen reference material (#82-02; Alberta Research Council, Edmonton, Canada) was fractionated for comparative purposes.

1 Simulated distillation

2 Simulated distillation (GC-SIMDIS) of the S, A_m, A_d, A_p, and P class fractions of each soil
3 extract was undertaken using a modified ASTM method D2887-89¹⁹. Boiling point
4 distribution profiles for each class fraction, illustrated in Fig. 3 for soils extracts from sites I,
5 III and V, were obtained using a *n*-carbon number calibration curve extended to an
6 atmospheric equivalent boiling point of 720°C (*n*C₁₀₀). GC-SIMDIS was performed on a
7 Hewlett-Packard 5890 GC equipped with an on-column temperature-controlled injector and
8 an aluminum clad Quadrex column (8m x 0.53mm ID) coated with 5% phenyl silicone of
9 0.15µm thickness. An oven temperature programme of 55-420°C was employed at a linear
10 rate of 10°C/min.

12 Chemical ionisation GC/MS

13 S, A_m, A_d and A_p class fractions from individual soil extracts were analyzed by GC separation
14 (Carlo Erba HRGC 5160) on a DB-5 column (25m x 0.25mm ID x 0.32µm film thickness)
15 using a 2µL injection volume and linear temperature profile between 50-320°C at 10°C/min.
16 Subsequent mass spectrometric determination (*m/z* range 44-500) of the eluates employed
17 nitrous oxide (N₂O) chemical ionisation (CI) on a Finnigan Mat 4500 mass spectrometer
18 calibrated using standard reference oils (Alberta Research Council) used routinely for the
19 analysis of homologous hydrocarbon series in tar sands and heavy oils. CI spectra were
20 obtained using a Townsend discharge ionisation source (0.6 Torr N₂O at 120°C). For each
21 class fraction the identification of the following class components: open chain and alicyclic
22 saturates, alkyl benzenes, benzo-, naphtho- and phenanthro- alicyclics and the homocyclic
23 polyaromatics was performed from reconstructed ion chromatograms by analysis of the raw
24 molecular ion data and by reference to the nominal masses of these hydrocarbons²⁰ and their
25 alkyl-substituted analogues (Fig. 1). Relative ring type and carbon number distributions for

these class components were constructed for each of the S, A_m, A_d and A_p class fractions, illustrated in Figure 4 for site III class fractions and Fig. 5 for the saturate fractions of sites I, II and V. Our results are necessarily qualitative since the CI-GC/MS of the polar and asphaltene class fractions was not possible and, for the remaining fractions, the interpretations of the relative distributions of chemical components in each class assumes (i) that the total ion count for each fraction equals the sum of intensities of all identified components; and (ii) equal response factors for all individual components. The resulting distributions (Fig. 4(a)-(c) and Fig. 5(a)-(c)) provide a relative abundance of homologous classes (*e.g.* the benzocycloalkanes) reporting in each of the S, A_m, A_d and A_p class fractions for selected site soils.

Results and discussion

Distillation profiles

The boiling point distribution of class fractions can be broadly related to their relative resistance to microbial degradation. In general, high molecular weight compounds are less easily biotransformed. GC-SIMDIS profiles for extract fractions from sites I, III, and V are illustrated in Fig 3 (a)-(c) with the reference plot of boiling point versus carbon number for *n*-alkanes C₄-C₁₀₀ included for interpretive purposes. Differences in character between class fractions can be inferred by interrogating three aspects of these profiles: (1) The relative gradient of the boiling point curve indicates the boiling point range over which a class fraction is 'distilled' from the column, and by inference, the relative molecular weight distribution of that fraction. Steep curves have tight distributions; lower gradient curves have broader distributions. (2) The relative position of individual curves away from the y-axis allows a qualitative description of the distillation range of the class fraction (*e.g.* light, mid-

distillate, heavy). (3) The point above the temperature on the x axis obtained from interpolating 25% cumulative weight distilled from the profile of an individual class fraction represents the boiling point of the remaining 75%^{w/w} of the sample, and can be expressed in terms of an equivalent *n*-saturate carbon number. For example, the saturate fraction at site I (Fig. 3(a)) was >75%^{w/w} in C₂₅ equivalent compounds (402°C) and above, and the polar fraction was >75%^{w/w} in C₄₂ equivalent compounds (534°C) and above. Similar results obtained from the polars fraction of the remaining GC-SIMDIS plots for the samples enables them to be ranked in terms of their boiling distribution: site I > (heavier than) site III ≈ site II > site IV > site V. This sequence is in agreement with the decreasing percentage contributions of the polars fraction to site soil extracts obtained by preparative column fractionation (Fig. 2), inferring that the polar fraction of these extracts dominates their distillation behaviour on the GC column.

Site V (Fig. 3 (c)), the creosote soil extract, exhibited a distinct distillation profile with narrower boiling point distributions and lower atmospheric equivalent boiling points for all extract fractions except the polars and an anomalous portion of the monoaromatics fraction. This indicates the extract is composed of a lower and narrower distillate, consistent with the documented use of N^o. 2 fuel oil as a diluent for coal tar creosote at this site. This contrasts with the heavy oil residues observed at each of the petroleum sites (I, III), now confirmed by their apparent molecular weight distribution by GC-SIMDIS analysis.

Class components in individual class fractions at weathered hydrocarbon sites

The boiling point and the general resistance of heavy oils to microbial degradation is a function of their chemical structure and molecular weight distribution⁵⁻⁶. Among the saturate class components found in petroleum residues, the isoprenoid alkanes (pristane, phytane) and the polycyclic alkanes (hopanes, steranes) are recognized as refractory compounds²¹⁻²³.

Detailed analysis of the total ion data by examining the nominal molecular masses²⁰ for homologous aliphatic, alicyclic and aromatic groups provides useful information on the relative composition of each component class by equivalent carbon number. In Fig. 4 (a) to (c), the progressive representation of linear, alicyclic, alkyl benzene, benzocycloparaffin, naphthalene and phenanthrene class components is illustrated in successive class fractions of the soil extract. In each case the carbon number range suggests the extent of alkyl substitution of the parent structure. Significant 'carry over' is observed, with identical class components reporting in more than one class fraction. The class composition of individual fractions from different soils can be compared using this approach. The saturate class composition from sites I, II, and V, for example (Fig. 5 (a) to (c)), is presented as distribution of component type and carbon number. Extract fractions identifiable by CI GC-MS show hydrocarbon distributions typical of mid- to high distillate residues (C₁₃-C₂₇). In general, the saturate fractions are dominated by the 2-4 ring alicyclics, recognised as resistant to biotransformation.

A tentative assessment of the extent of weathering experienced by these samples may also be obtained from the C₁₇ open chain/pristane and C₁₈ open chain/phytane ratios²⁴; pristane and phytane being regarded generally as more resistant to microbial degradation than the equivalent *n*-alkanes, though this indicator has recognised limitations^{25, 12}. From the raw molecular ion data, these ratios were calculated, respectively as: site I (0.3,0.3); site II (0.5,0.5); site III (0.5,0.7); site IV (1.2, 1.1); and site V (6.9, 4.9). Hence, residual contamination can be ranked according to its apparent relative weathered state: site I > (more weathered than) site II ≈ site III > site IV > site V. This sequence is in close agreement with the GC-SIMDIS profile and %^w/_w polars contribution data (Fig. 2), lending additional support to these indicators as surrogates for distinguishing between the extent of weathering of the hydrocarbons at these sites. Hydrocarbon contamination at the creosote site (site V) was

markedly different in its characteristics from that encountered at petroleum sites (Fig. 3(c)) and was significantly less weathered in composition. Sites I-III were characteristic of significantly weathered oils and site IV (not shown) characteristic of a mid-distillate residue. Significant contributions come from alkyl benzenes, which report in the saturate class in cases where extensive alkyl- substitution overrides ring aromaticity as a determinant of polarity. Likewise, some of the benzocycloparaffins (also resistant to biotransformation) report in this class.

Implications for bioremediation

Many hydrocarbon-contaminated sites (former refineries, coal carbonisation plants, and integrated steelworks) contain (i) oils that are weathered because the source term has aged significantly since release; (ii) heavy fuel oil residues such as Nos. 4, 5 and 6 fuel oil used in commercial boilers or heavy diesel engines; and/or (iii) viscous tars and solid bituminous process residues that are difficult to treat biologically. In contrast to lighter gasoline (petrol), diesel and aviation fuels, the literature on heavy oil wastes is not extensive and the risks to human health are not well characterised. Understanding the physicochemical and toxicological characteristics of contaminants in these wastes is critical because these factors drive the design of analytical strategies, our understanding of exposure and the selection and operation of remediation technologies.

Our previous studies¹³ have shown that compositional changes during weathering affect the partitioning behaviour of risk critical components (*e.g.* the PAHs) prior to, during and following biological soil treatment. The data presented in this study provides additional insight into the composition of weathered hydrocarbon wastes and the residual oil that form the principal partition medium for risk critical components. We have shown that taken together, the recalcitrant polar and asphaltene content of soil extracts from these sites

1 contributes between 40%^{w/w} and 70%^{w/w} of the oils encountered (Fig. 1), with the polars
2 determining the distribution profile of the soil extracts. With respect to the class composition
3 of chromatographically-separated fractions, the abundance of C₁₄-C₂₂ 2-4 ring alicyclics (Fig.
4 5), alkylbenzenes and the benzocycloparaffins is prevalent within the saturate (S) class
5 fractions of site soil extracts. These hydrophobic, recalcitrant compounds, together with the
6 polar and extended hetero-polyaromatic asphaltene structures in these oils²⁶ comprise the
7 partitioning medium for PAHs in weathered oil wastes into which between 71-96%^{w/w} of PAH
8 in the creosote-contaminated soils is partitioned (84%^{w/w} for benzo[*a*]pyrene)¹³. Under
9 these conditions, the availability of PAHs to pose human health risk may be substantially
10 reduced.

11 Bioremediation is used internationally for sites contaminated with fossil fuel
12 hydrocarbons, organic wood-preserving wastes and high volume industrial organics (*e.g.*
13 polychlorinated biphenyls, tetrachloroethylene). Its application to hydrocarbon wastes,
14 principally as *ex-situ* engineered biopiling, has been assisted in the US by a shift to risk-
15 based, remedial objective-led remediation which has forced a scientific rigour on site
16 investigation, remedial design and technology verification. The move towards risk-based
17 corrective action has been slow in the UK and, whilst some progress has been made in
18 integrating aspects of analysis, exposure assessment and technology verification³, there tends
19 to be a lack of synthesis between these aspects of risk management. Indeed, bioremediation
20 performance tends to be evaluated by reductions in hydrocarbon load²⁷⁻²⁸ rather than
21 combined reductions in toxicity, chemical mass and risk. Studies that integrate these aspects
22 are essential if risk-based decision-making is to become the basis for the management of
23 contaminated land.

1 **Conclusions**

2 The data in this study further inform our understanding of the nature of oily wastes at
3 weathered hydrocarbon sites and the extent to which bioremediation might practically
4 progress for these wastes. Bioremediation technologies express operational limits beyond
5 which, because of the composition and recalcitrance of the partition medium for risk critical
6 compounds and the strength of association between, say PAH, and the weathered source
7 term, prolonged bioremediation is futile in terms of progressive risk reduction. A key factor
8 is to recognise the partitioning behaviour of contaminants like PAHs into recalcitrant heavy
9 oil residues. One research challenge is to now demonstrate the optimisation of
10 bioremediation (*e.g.* biopiling) processes by reference to risk reduction and to identify the
11 practical limits of this technology using knowledge gained on the composition of weathered
12 hydrocarbons.

14 **Acknowledgements**

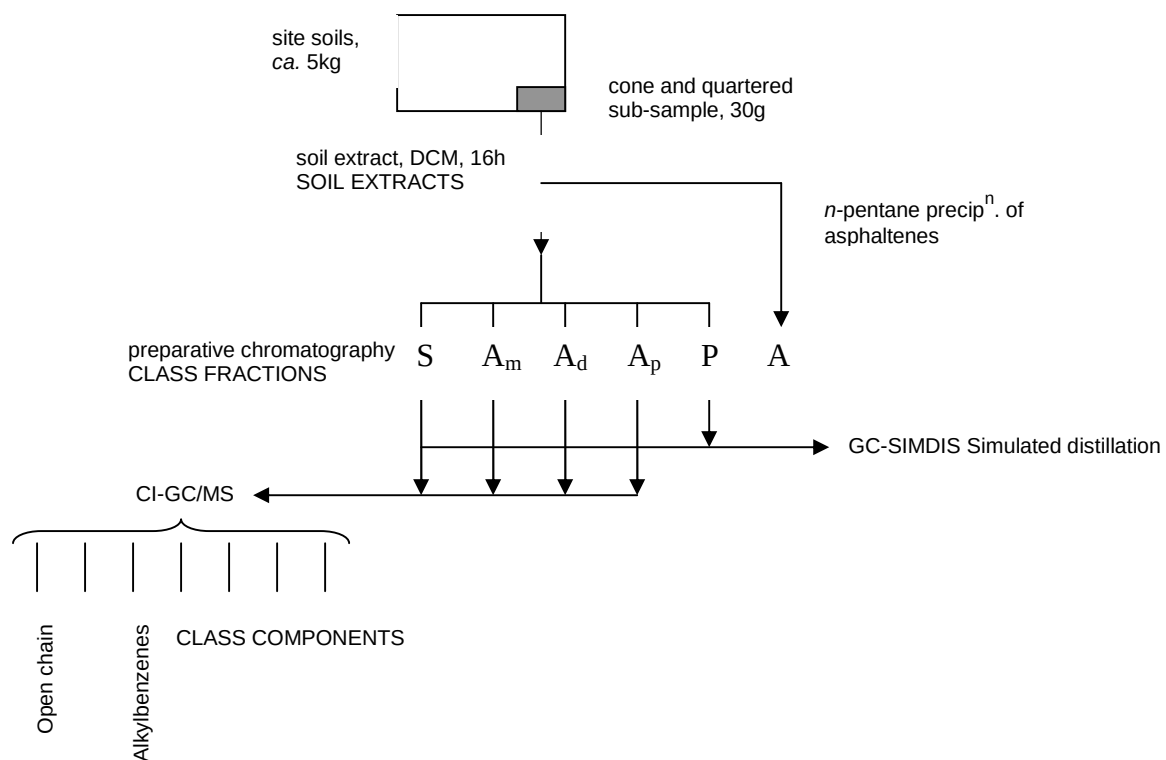
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1

2 Fig. 1 Analytical strategy and terminology employed in this study

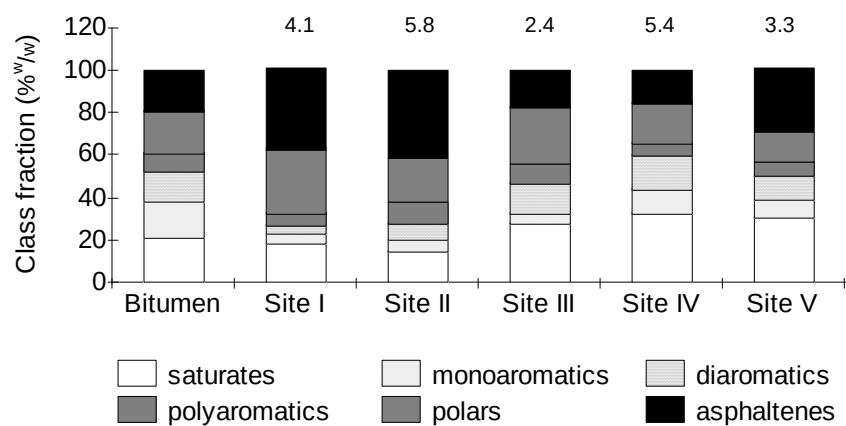


Fig. 2 Normalised composition of soil extracts from Sites I-V by class fraction (bar labels showing %^w/_w DCM-extractable matter in sample, $\pm 0.2^w/w$ % RSD).

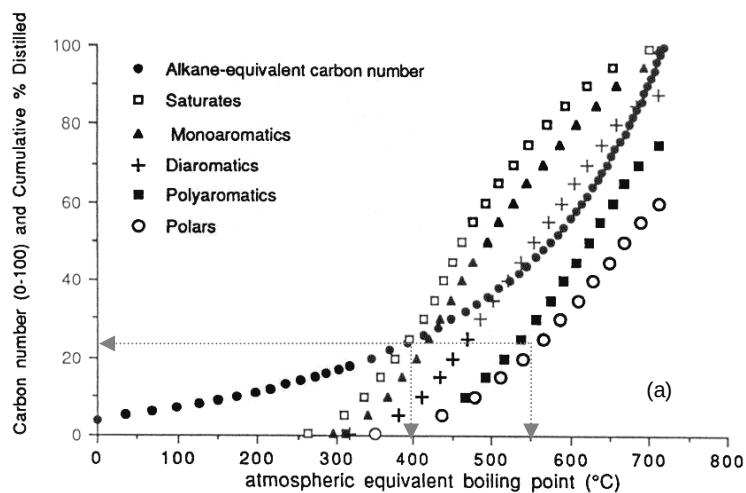
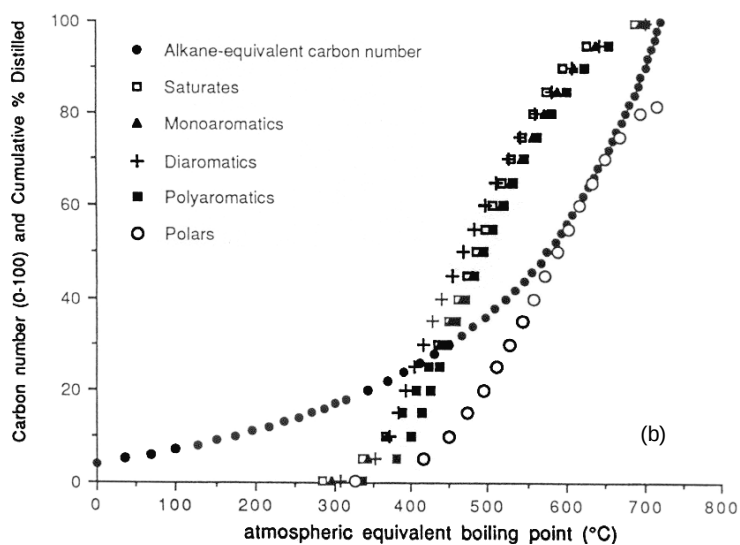
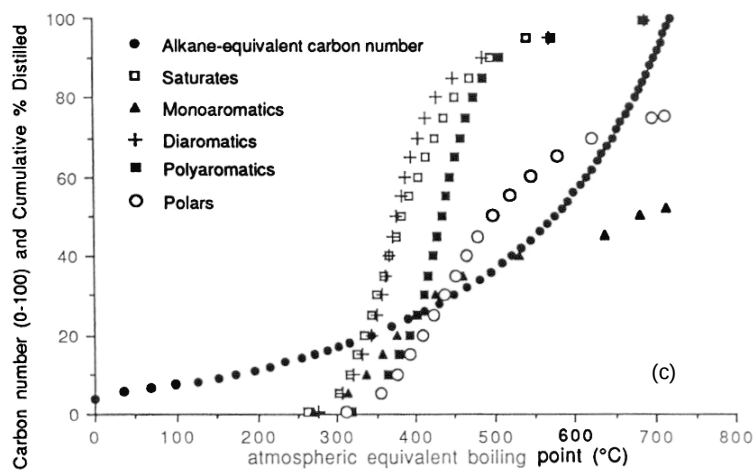


Fig. 3 SIMDIS GC profiles for DCM extracts from Sites (a) I; (b) III; and (c) V

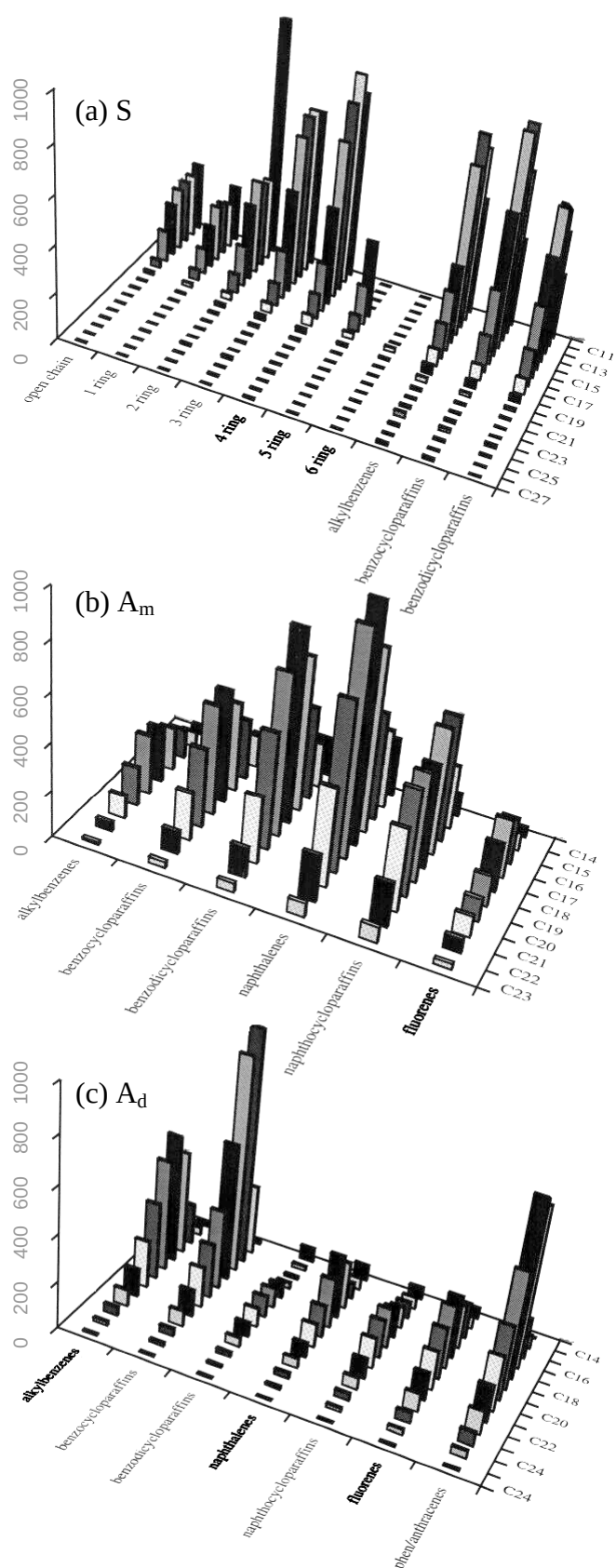
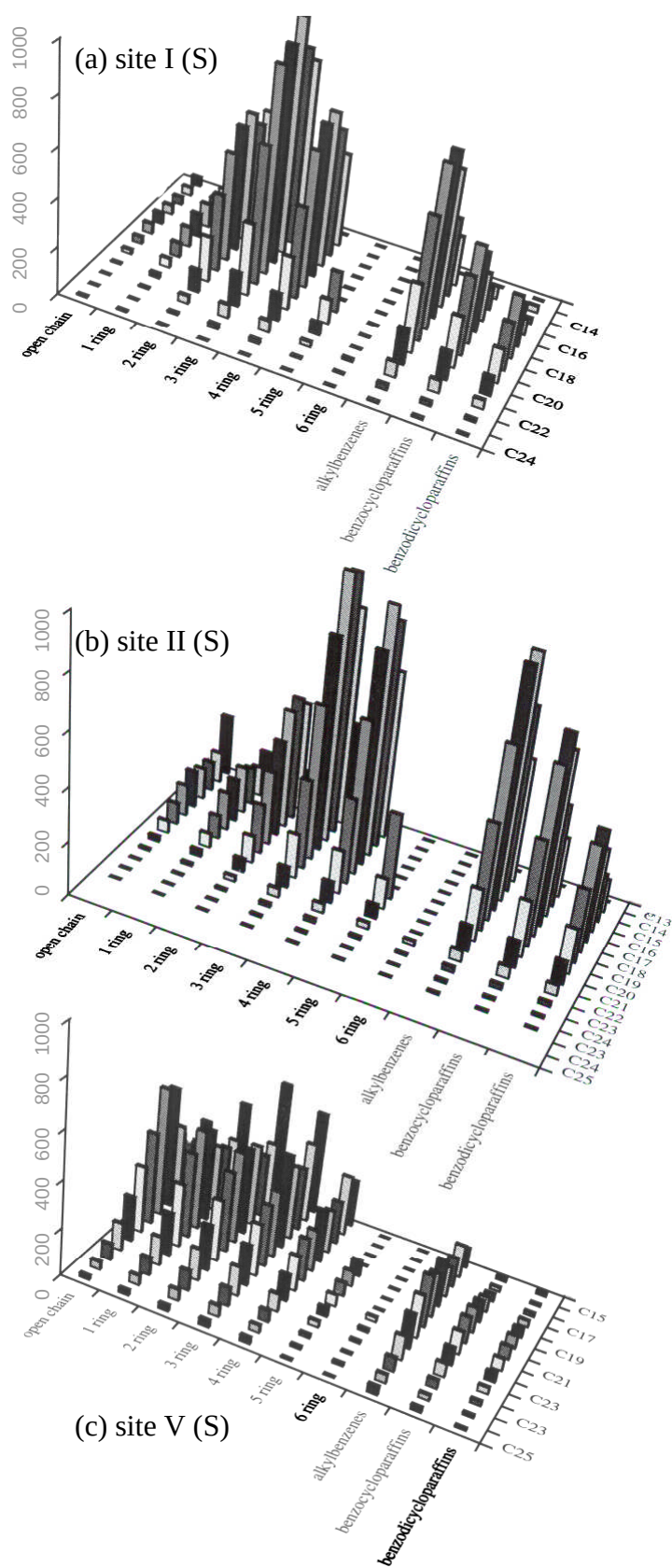


Fig. 4 Relative composition of site III soil extract fractions by class component, carbon number and relative ion count (unitless)

1



2

3 Fig. 5 Relative composition of site extract saturate (S) fractions by class component, carbon
 4 number and relative ion count (unitless)



DSPACE

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