

# BIOMARKER GEOCHEMISTRY OF SEEP OILS AND POTENTIAL HYDROCARBON SOURCE ROCKS FROM THE NORTHERN EAST COAST BASIN, NEW ZEALAND

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## Abstract

Biomarker geochemistry has been used extensively, along with visual kerogen, Total Organic Carbon (TOC), Rock-Eval and some carbon isotope analyses, to study a range of oils and potential source rocks from the northern East Coast Basin, New Zealand. Sediments in the basin range in age from Cretaceous to Recent.

Although oil seeps occur throughout the northern East Coast Basin, the three main seep areas are located at Rotokautuku, Waitangi and Totangi. However seeps and oil impregnations are also found in Whangai, Waipawa and Miocene outcrops. In general, all seeps and impregnations studied are moderately mature with a marine influence.

The biomarker geochemistry of each formation suggests that in outcrop, the Eocene, Oligocene and Miocene formations are immature. It is also unlikely that the Waipawa Black Shale can be considered a potential source for the three main oil seeps as it contains distinct C<sub>30</sub> steranes in relatively high quantities. Such C<sub>30</sub> steranes only appear in minor quantities in the three main seep oils and other potential source rocks. However, oil stains from the southern East Coast Basin have been analysed and some were found to contain high C<sub>30</sub> steranes abundances. It is likely that the stains with high C<sub>30</sub> steranes are sourced from the Waipawa Black Shale. The Whangai Formation is marginally mature in outcrop, but exhibits extensive oil staining in the western margin of the basin. These oil stains are all similar in maturity and biomarker characteristics to the main seep oils.

Underlying Cretaceous formations all exhibit mature regions with good correlations to the seep oils. Oil impregnations are less common here, and while TOCs are lower than the Waipawa Black Shale (traditionally the more popular potential source rock), the thickness of the formations (up to 2.6 km for the Karekare Formation) and the depth of burial satisfy the requirements of a potential source rock. The similarity of the biomarker geochemistry of these underlying formations, and the similarity of the main oil seeps and associated stains in the northern East Coast Basin, suggests a common regional source for these oils.

## Introduction

The East Coast Basin has been recognised as a petroleum province from as early as 1865. Some 45 wells have been drilled to date with no commercial discoveries. The main seeps are at Waitangi, Totangi and Rotokautuku (figure 1). Previous workers on these seeps and potential source formations (Moore, 1988; Jackson, 1982; Fry, 1982) have noted the likely source potential of the Waipawa Black Shale. However Johnston et al. (1991), on the basis of a preliminary biomarker study suggest that the Waipawa Black Shale is not a likely source for the main oil seeps. To date the main source for the seeps has not been identified.

The East Coast Basin covers an area of some 70 000 km<sup>2</sup> with approximately half onshore. The basin has been in existence since Early to Mid Cretaceous and is thought to be up to 10 000 m deep. Strata studied range in age from Early Cretaceous (Urewera basement, Koranga and Waitahaia formations) through to the Late Cretaceous (Karekare and Whangai formations), and the notable Paleocene (Waipawa Black Shale). The study has also included Eocene (Wanstead

Formation) and some Oligocene and Miocene mudstones (figure 2).

In the present study, a suite of geochemical methods have been used for oil-oil and oil-source rock correlations to determine the formation(s) which are generating the seep oils seen at Waitangi, Totangi and Rotokautuku. This paper will deal mainly with correlations of various formations to these seeps. However, the study has also found seeps in the southern part of the basin which are not related to the main seeps in the north, and these will also be discussed.

This paper focuses on two oil seeps (Totangi and Waitangi), two oil-impregnated Whangai shale samples and representative outcrop samples from the Waipawa Black Shale, Whangai shale, Karekare Formation and basement, to geochemically characterise the formations. A comparison between the Waipawa Black Shale and an oil-stained greensand from the southern East Coast Basin is also presented.

Minor oil seepages and impregnations are abundant throughout the East Coast Basin, and occur in many

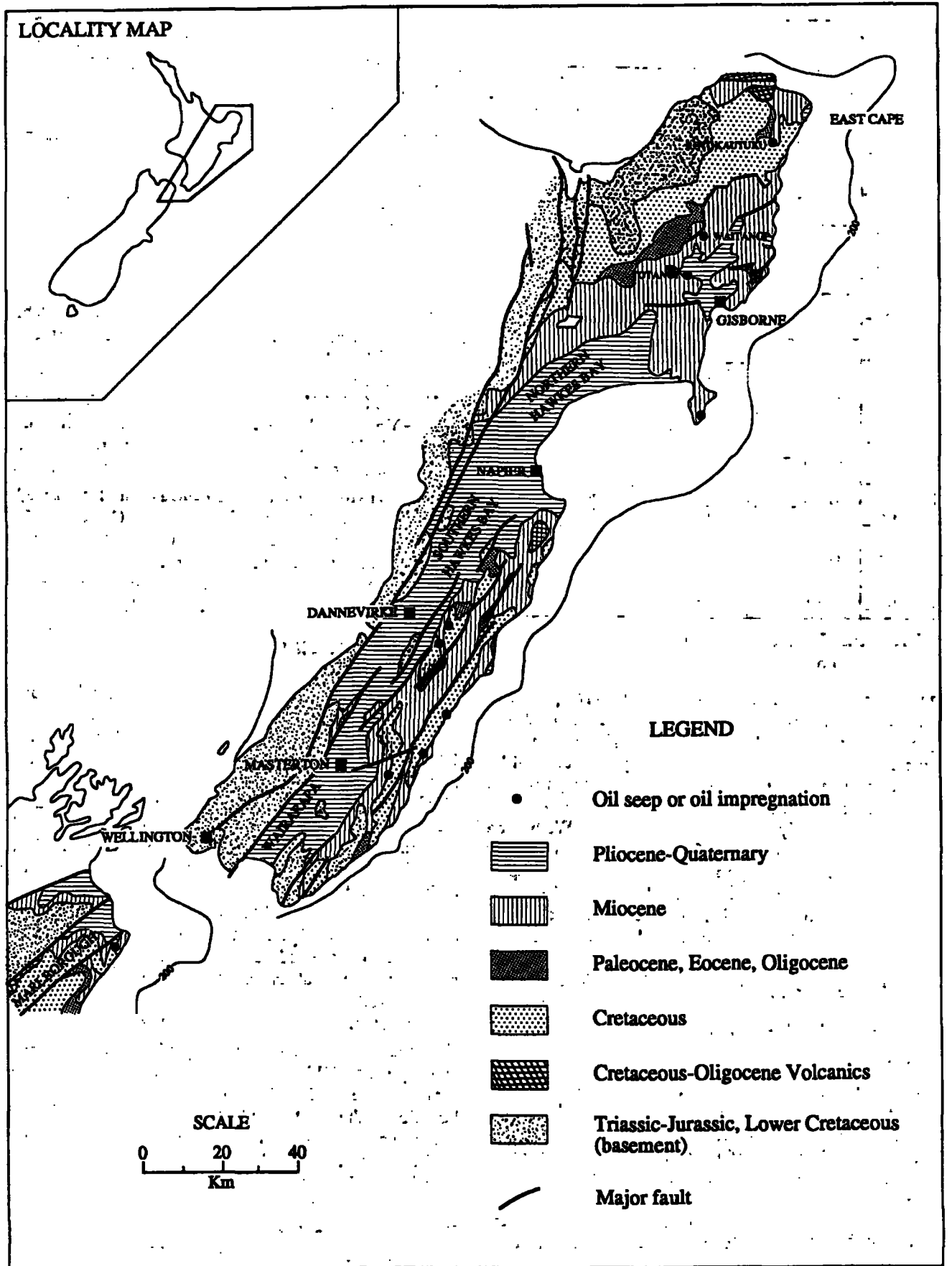


Fig. 1. Geology of the East Coast Basin (after Francis, 1992).

Million Years	Epoch	Series	Formation	Maximum Thickness (metres)	Environment of Deposition
0	Pliocene	Howa		2800	Inner Shelf
		Wanganui			
10	Miocene	Taranaki	Waitere	3000	Middle-Outer Shelf
		Southland	Narrows	800	
20		Pareora	Whakamarino	500	
30	Oligocene	Landon	Weber	1500	
40					
50	Eocene	Arnold			Middle-Outer Shelf
		Wanstead		400	
60	Paleocene	Dannevirke	Waipawa	50	Inner Shelf
70			Whangai		600
80	Late Cretaceous	Mata	Tahora	500	Nearshore-Middle Shelf
90					
100	Early Cretaceous	Rautumera	Karekare	3000	Outer Shelf
		Clarence			
			Té Wera		250
110		Taitai	Koranga	250	Marginal Marine
120					
130		Urewera Group Basement			

Fig. 2. Generalised stratigraphy of the Northern East Coast Basin (from J Simpson, 1991).

formations ranging from Late Cretaceous to Neogene age. This is due in part to fracturing in the basin, which has generated many migration pathways. These seeps and impregnations have been geochemically characterised by Gas Chromatography (GC), Gas Chromatography–Mass Spectrometry (GC–MS) and Carbon Isotope analyses as part of an ongoing study at Victoria University of Wellington into potential source formations for East Coast oils. Biomarker maturation parameters have been outlined in previous papers (Seifert and Moldowan, 1978; 1981; Philp and Gilbert, 1987; Johnston et al., 1991). The main biomarker characterisation was by steranes ( $m/z$  217) and hopanes (191  $m/z$ ) (table 1). Other characterisation techniques include Rock-Eval pyrolysis, hydrous pyrolysis, Total Organic Carbon, GC–MSMS, visual kerogen, trace and major elements, and Gas Chromatography.

## Samples

Representative samples were collected from Totangi, Rotokautuku and Waitangi oil seeps. Geochemical biomarker data for the Rotokautuku seep has been previously described (Johnston et al., 1991) and is not covered here. It is noted however, that these seeps are similar in every respect. In excess of 400 outcrop samples have been studied, ranging in age from basement through to recent sediments. Where these outcrop samples are impregnated with oil, the extracts have been treated similarly to the seep oils.

## Methods

Outcrop samples were cleaned, crushed and ground to 200  $\mu\text{m}$  by a TEMA mill. Between 30–50g of ground rock were then refluxed for 72 hours in a soxhlet extraction apparatus using dichloromethane to extract the organic fractions. The oil-impregnated shales were crushed and simply subjected

to quick extraction with warm dichloromethane to remove only the migrated fractions and not the host organic matter. Seep oils and extracted oil from the impregnated shales were then treated in the same fashion as the extracted matter from the outcrop samples.

Excess dichloromethane was removed by rotary evaporation to concentrate the extracted organic material. Asphaltenes were removed by precipitation with *n*-pentane. The saturate fraction was separated from the aromatic fraction by column chromatography. A column of 5g alumina and 3g silica was eluted with 100 ml of *n*-pentane to extract the saturates. These were split into two samples, one of which was analysed by Gas Chromatography to determine the *n*-alkane abundances. The other fraction was refluxed with iso-octane and Union Carbide SP115 molecular sieve to remove the *n*-alkanes and enable the branched and cyclic biomarker fractions to be characterised. For this, the sterane ( $m/z$  217) and hopane ( $m/z$  191) ion chromatograms were determined by GC–MS using a Hewlett-Packard HP5995C system with a 30m J&W DB1 column. The biomarkers were identified from retention times of previous work (Johnston et al., 1991).

Rock-Eval pyrolysis data were obtained from a Rock Eval 1 using 100mg of ground rock. Total Organic Carbon (TOC) was obtained from a LECO multiphase Carbon Determinator. Samples of approximately 100mg were analysed for organic carbon and carbonate. Visual kerogen analyses and carbon isotope determinations have also been made.

## Results and Discussion

### East Coast oil seeps

Particular geochemical biomarker components, their relative abundances and appropriate ratios commonly used to assess maturation levels by GC–MS (Seifert and Moldowan, 1978; 1981; Philp and Gilbert, 1987; Johnston et al., 1991), show these main seep oils and impregnated Whangai shales (figure 3) of the northern East Coast Basin to be similar and of moderate maturity. This initially suggested that the main oil seeps in the north have a common regional source rather than several localised ones. This could be one particular source rock or several formations deposited under similar conditions. The oils are isotopically light ( $\delta^{13}\text{C}_{\text{saturates}} = -29$  per mil), and relatively low in sulphur. The dominant  $\text{C}_{27}$  steranes and diasteranes (figure 3) indicate a predominantly marine source. However, traces of oleanane (a terrestrial biomarker) found in the seep oils suggest either a minor terrestrial influence, or contamination during migration through other sediments.

Tricyclic terpanes are relatively abundant in the seep oils and impregnated Whangai shale, supporting a marine origin for the source (de Grande et al., 1993), (figure 5). The  $\text{C}_{23}$  homolog is the most abundant tricyclic terpane. The  $\text{C}_{24}$  tetracyclic terpane occurs in both marine and non-marine environments. Additional tetracyclic compounds found in the Waipawa Black Shale (see below) are not present in the oil seeps or in impregnations in the Whangai shale.

### Post-Paleocene sediments

The younger Eocene, Oligocene and Miocene formations have been investigated; however, they are considered an unlikely potential source because of consistently low TOCs (<0.2%) (unpublished) and unfavourable Rock-Eval results. ( $\text{S}_2 < 0.1$  mg HC/g rock) (unpub).

### Paleocene, Waipawa Black Shale

Previous studies (Gibbons, 1980; Moore, 1989; Leckie et al., 1992) have identified the Waipawa Black Shale as the most

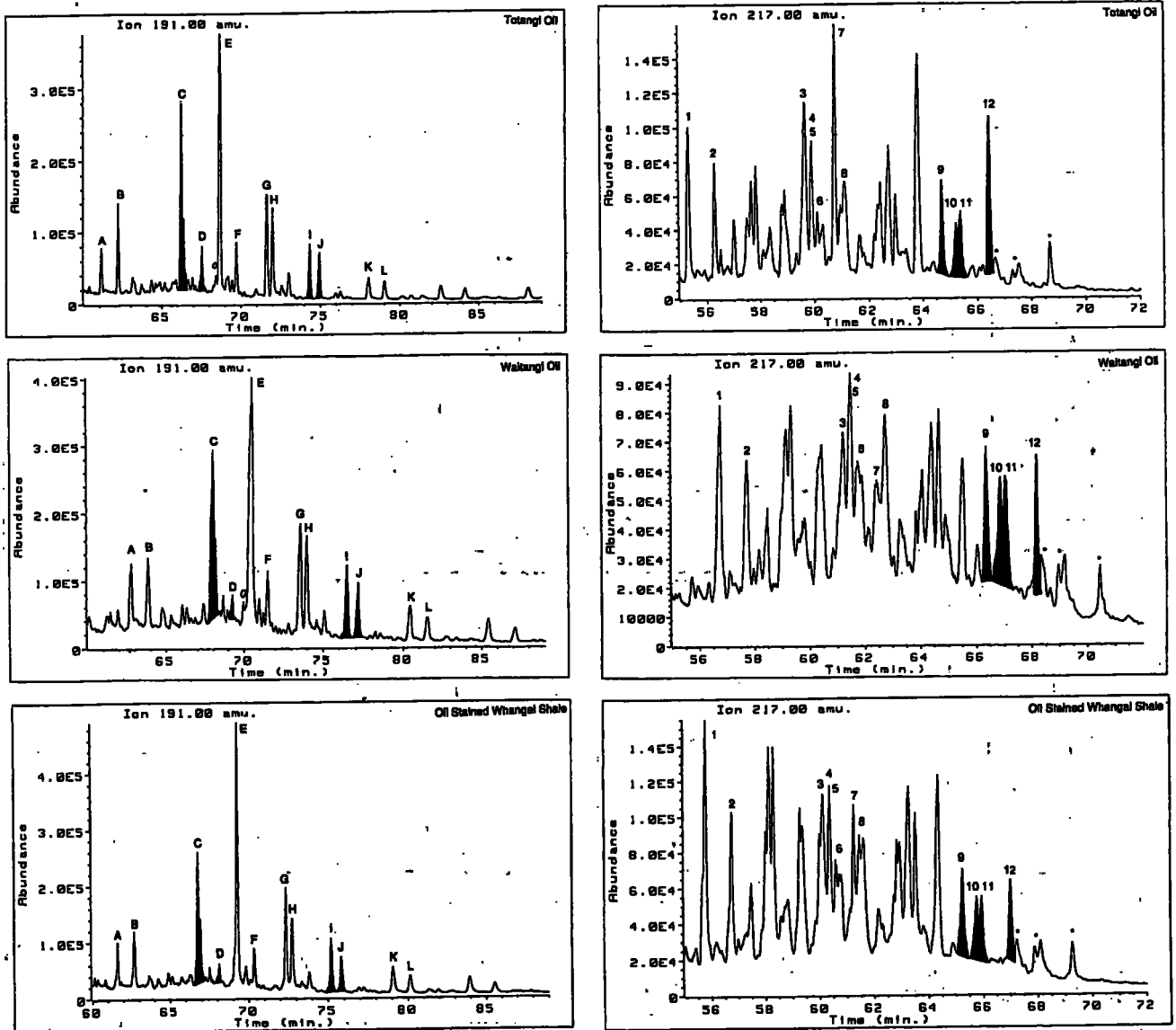
**Table 1. Sterane and Hopane identification.**

**Steranes**

- 1 C<sub>27</sub> (20S) 13β(H) 17α(H) diacholestane
- 2 C<sub>27</sub> (20R) 13β(H) 17α(H) diacholestane
- 3 C<sub>27</sub> (20S) 5α(H) 14α(H) 17α(H) cholestane
- 4 C<sub>29</sub> (20S) 24-ethyl 13β(H) 17α(H) diacholestane (co-eluting)
- 5 C<sub>27</sub> (20R) 5α(H) 14β(H) 17β(H) cholestane
- 6 C<sub>27</sub> (20S) 5α(H) 14β(H) 17β(H) cholestane
- 7 C<sub>27</sub> (20R) 5α(H) 14α(H) 17α(H) cholestane
- 8 C<sub>29</sub> (20R) 24-ethyl 13β(H) 17α(H) diacholestane (co-eluting)
- 9 C<sub>29</sub> (20S) 24-ethyl 5α(H) 14α(H) 17α(H) cholestane
- 10 C<sub>29</sub> (20R) 24-ethyl 5α(H) 14β(H) 17β(H) cholestane
- 11 C<sub>29</sub> (20S) 24-ethyl 5α(H) 14β(H) 17β(H) cholestane
- 12 C<sub>29</sub> (20R) 24-ethyl 5α(H) 14α(H) 17α(H) cholestane
- \* C<sub>30</sub> Steranes (24-n-propyl)

**Hopanes**

- A 18α(H)-22,29,30 trisnorhopane (Ts)
- B 17α(H)-22,29,30 trisnorhopane (Tm)
- C C<sub>29</sub> 17α(H) 21β(H) norhopane + C<sub>29</sub> Ts neonorhopane
- D C<sub>29</sub> 17β(H) 21α(H) normoretane
- E C<sub>30</sub> 17α(H) 21β(H) hopane
- F C<sub>30</sub> 17β(H) 21α(H) moretane
- G,H C<sub>31</sub> 22S,22R 17α(H) 21β(H) homohopane
- I,J C<sub>32</sub> 22S,22R 17α(H) 21β(H) bishomohopane
- K,L C<sub>33</sub> 22S,22R 17α(H) 21β(H) trishomohopane
- M C<sub>28,30</sub> 17α(H) 21β(H) bisnorhopane
- X tricyclic terpanes
- Y tetracyclic terpanes
- O Oleanane



**Fig. 3.** Hopane (191m/z) and sterane (217m/z) chromatograms for East Coast oil seeps and extracted oil from an impregnated Whangai shale. Peaks are highlighted for correlative purposes.

likely source rock on the basis of its dark coloration, high Total Organic Carbon content and a high S2 index from Rock-Eval analyses. Although TOCs have been reported up to 12% (Moore, 1988), measurements from samples of Waipawa Black Shale carried out in this study yielded a

maximum value of 6%, with an average of 4% (unpublished).

The Waipawa Black Shale is a thin (10–50 m) formation and all outcrop samples studied here are immature or only marginally mature. The hopane biomarkers (191 m/z) exhibit

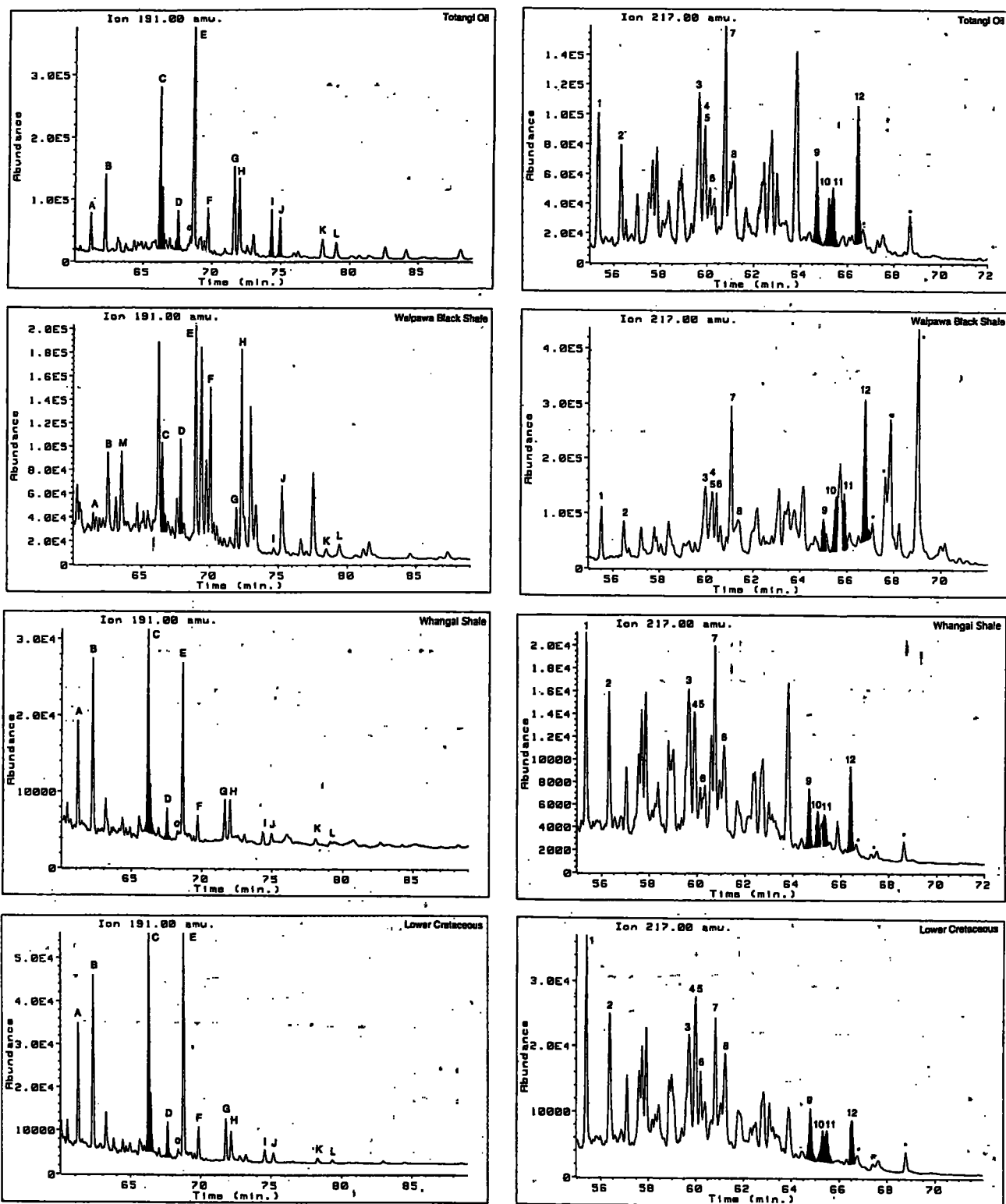


Fig. 4. Hopane (191m/z) and sterane (217m/z) chromatograms for Waipawa Black Shale, Whangai Shale and a generalised Lower Cretaceous sample. Peaks are highlighted for correlative purposes.

low  $C_{31}$  &  $C_{32}$  S:R ratios and high moretane/hopane ratios, indicating low maturity (figure 4). Unusually high levels of  $C_{28}$  bisnorhopane found in the Waipawa Black Shale also indicate an anoxic environment of deposition and low maturity (Waples, 1991).

The steranes, although reflecting the predominant marine nature of this shale by the  $C_{27}$  steranes present; are dominated by high  $C_{30}$  steranes. Gas Chromatography/Mass Spectrometry/Mass Spectrometry (GC-MSMS) study has identified the  $C_{30}$  steranes as 24-n-propyl-cholesterols (4-

desmethyl steranes) (unpublished). These  $C_{30}$ -steranes indicate a paleoenvironment with high marine algal input (Moldowan et al., 1990; Peters and Moldowan, 1993).

The  $C_{29}$  steranes are often more abundant relative to the  $C_{27}$  steranes. This suggests a more terrestrial influence for this formation. Occurrences of oleanane, a marker associated with angiosperms (Moldowan, 1991; Reed, 1991), have been seen in most hopane chromatograms (m/z 191, figure 4), and extra tetracyclic compounds are present within the tricyclic terpane chromatogram (m/z 191, figure 5),

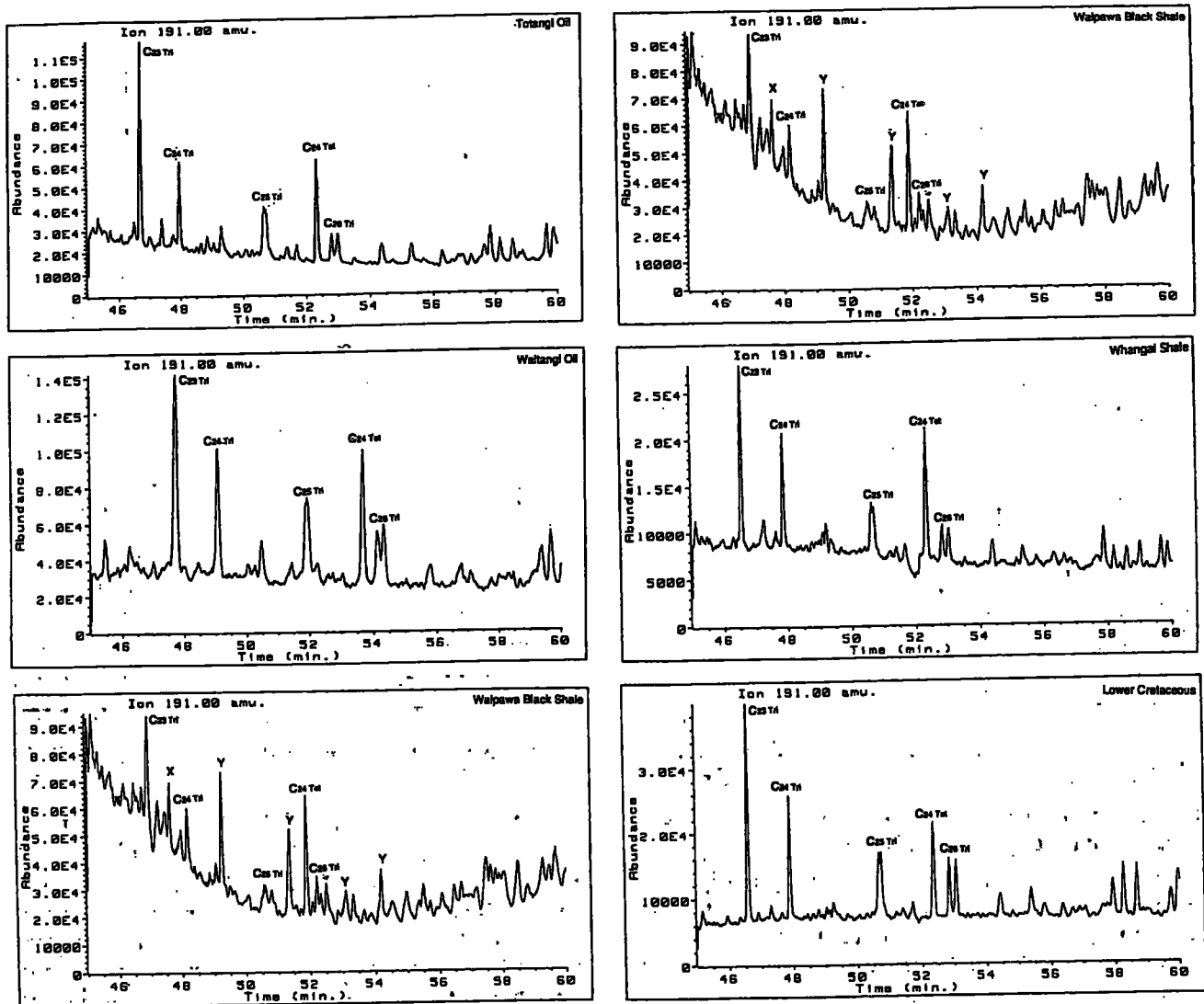


Fig. 5. Tricyclic and tetracyclic terpane chromatograms (191m/z) for East Coast oils and potential formations.

supporting a terrestrial influence. The low  $C_{27}$  diasteranes can be attributed to low maturity. Low diasterane/sterane abundances have also been noted in similar sulphur rich bitumens in source rocks which are typically clay-poor (Peters and Moldowan, 1993).

Visual kerogen analysis has identified dominantly amorphous material typical of chrysophyte algae. Algal material breaks down more readily to generate amorphous material than marine or terrestrial organic matter. Other kerogen components found in the Waipawa Black Shale include structured terrestrial material, such as leaf and plant tissue, and biodegraded terrestrial material. Both of these components are consistent with the higher abundance of  $C_{29}$  steranes seen in the Waipawa Black shale, as well as the added contribution from reworked material to the amorphous content.

Rock-Eval measurements and artificial maturation studies using hydrous pyrolysis to simulate petroleum generation have suggested that the Waipawa Black Shale is indeed capable of generating oil (Rogers et al., 1993, unpublished). This is probably due to its high algal content and the unusually high sulphur levels, which at early level of maturity aid hydrocarbon generation (Baskin and Peters, 1992).

The potential for generation of hydrocarbons by the Waipawa Black Shale is shown by significant intensity of the S2 peaks

(average = 10 mg HC/g rock) in the Rock-Eval analyses. With the high sulphur (1-3%) and algal contents of the Waipawa Black Shale, it is not surprising that the Waipawa Black Shale is capable of generating hydrocarbons with only minor burial and thermal alteration (Baskin and Peters, 1992). However, biomarker data from the Waipawa Black Shale do not correlate with those of the three main oil seeps in the north. This incompatibility is also supported by the isotopic data of the Waipawa Black Shale ( $\delta^{13}C_{\text{saturation}} = -25.0$  per mil). These results suggest, therefore, that the three main oil seeps are not sourced from the Waipawa Black Shale.

Some oil stains from the southern part of the basin have a much higher level of  $C_{30}$  steranes than the oil seeps in the north (figure 6). Biomarker characteristics of these southern oils are similar to the pyrolysate produced from hydrous pyrolysis (artificial maturation) of the immature Waipawa Black Shale (unpublished). Though immature in outcrop, it is inferred that the Waipawa Black Shale is more deeply buried elsewhere in the basin, where oil generation may be possible as discussed above.

Other evidence supporting generation from the Waipawa Black Shale is the higher level of maturity in the oil-stained greensand shown by the  $C_{30}$  steranes relative to the Waipawa Black Shale. If an oil from another source had migrated through the Waipawa Black Shale, and was contaminated with signatures of the  $C_{30}$  steranes from the Waipawa Black

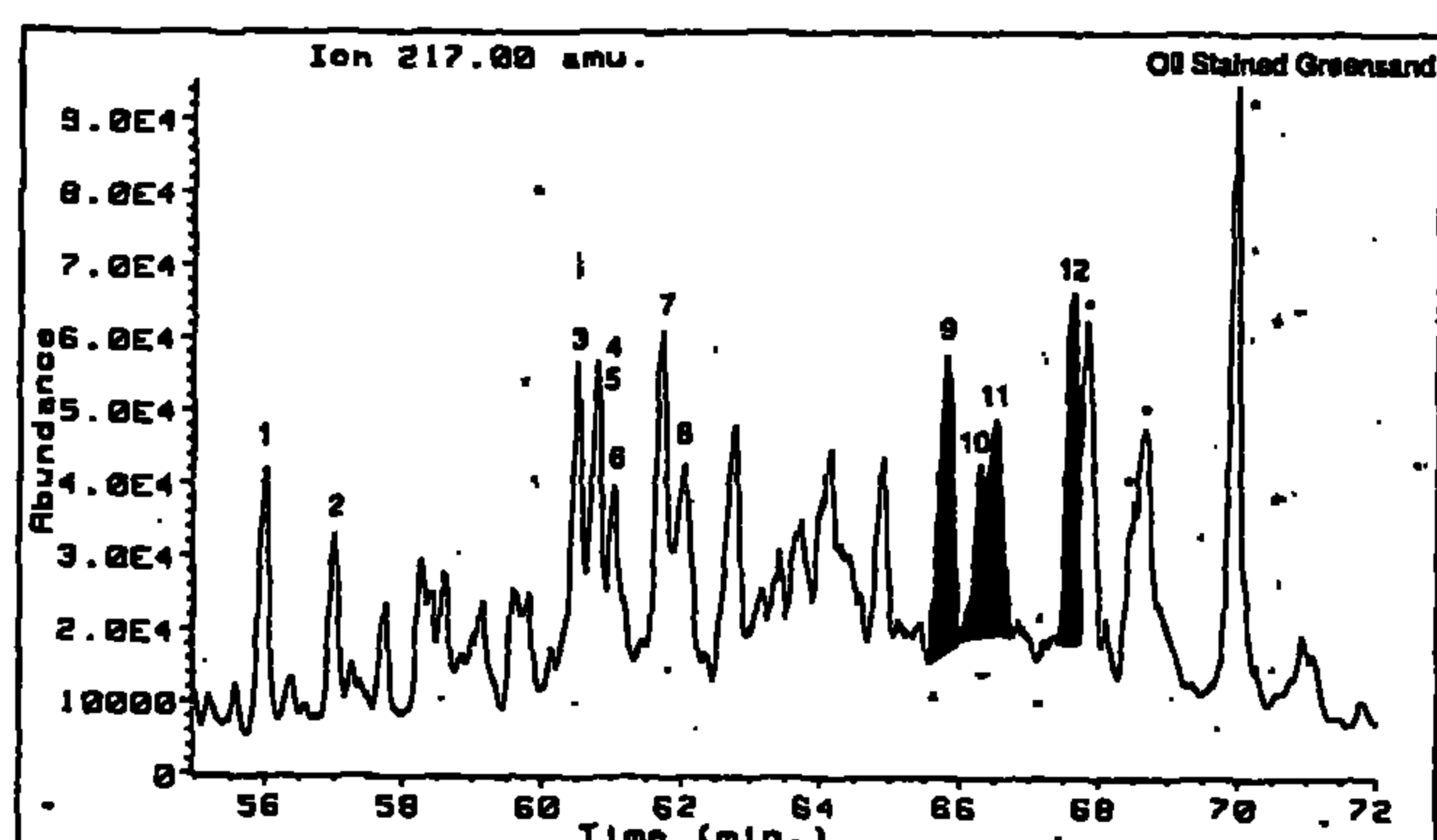
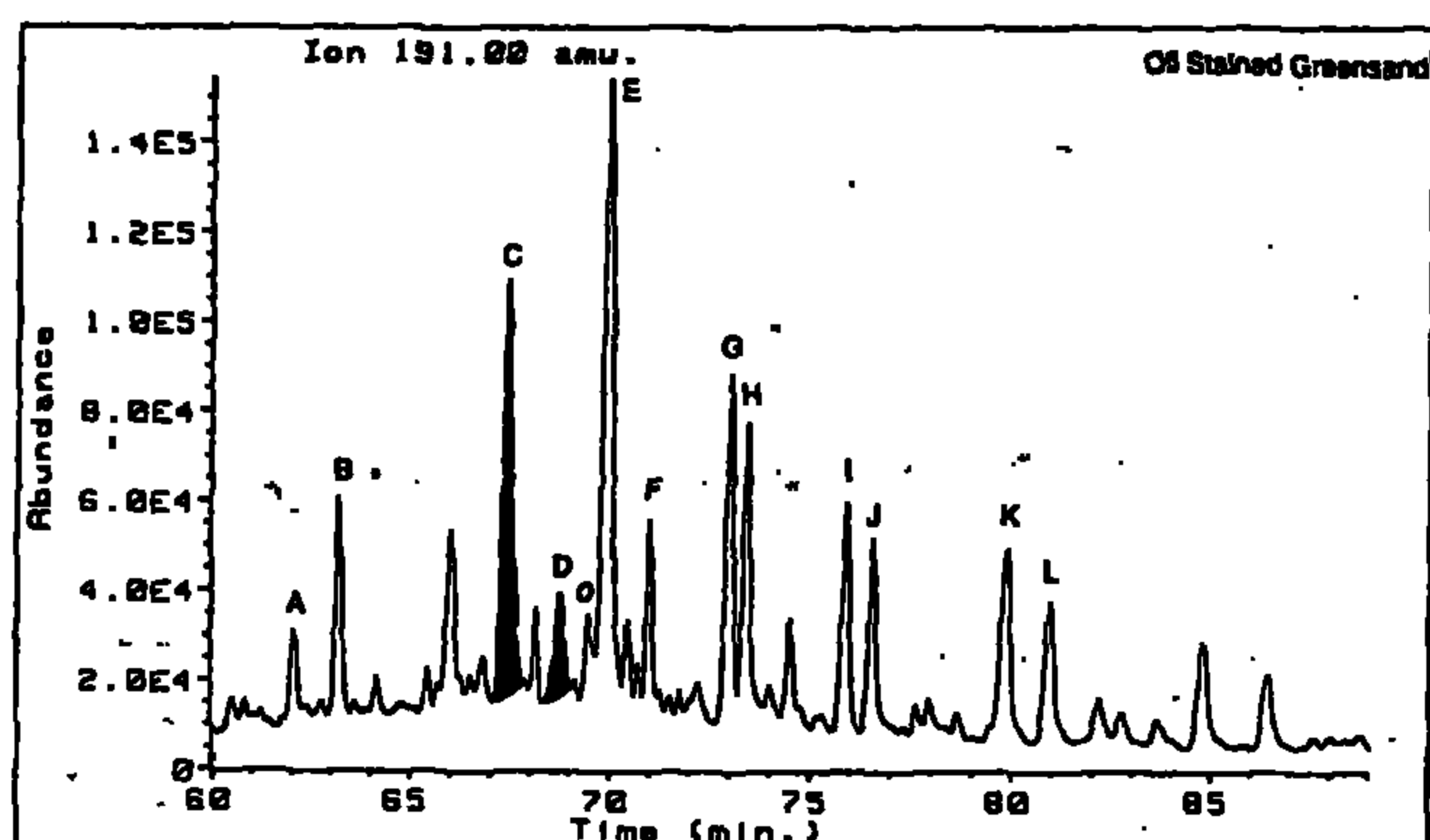
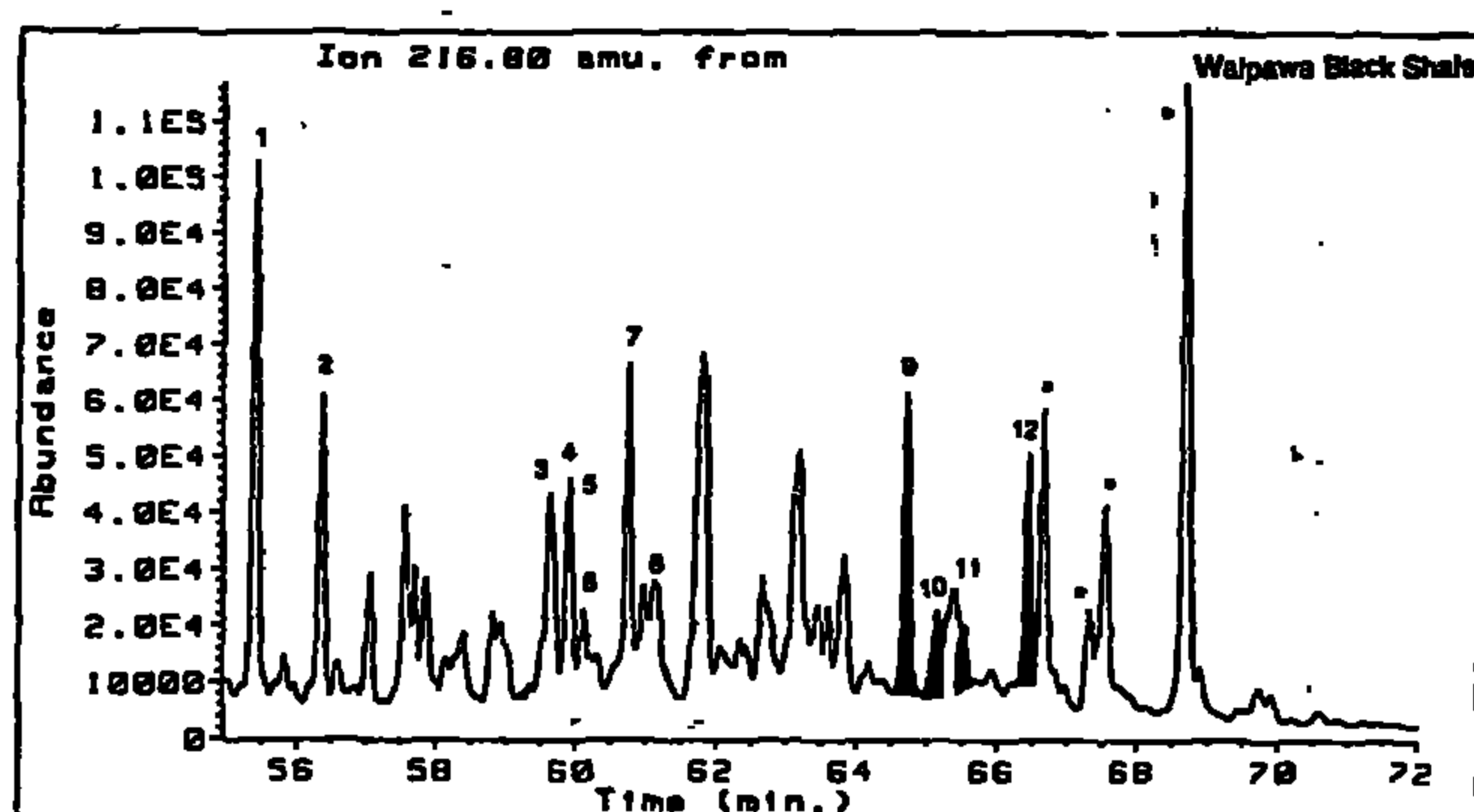
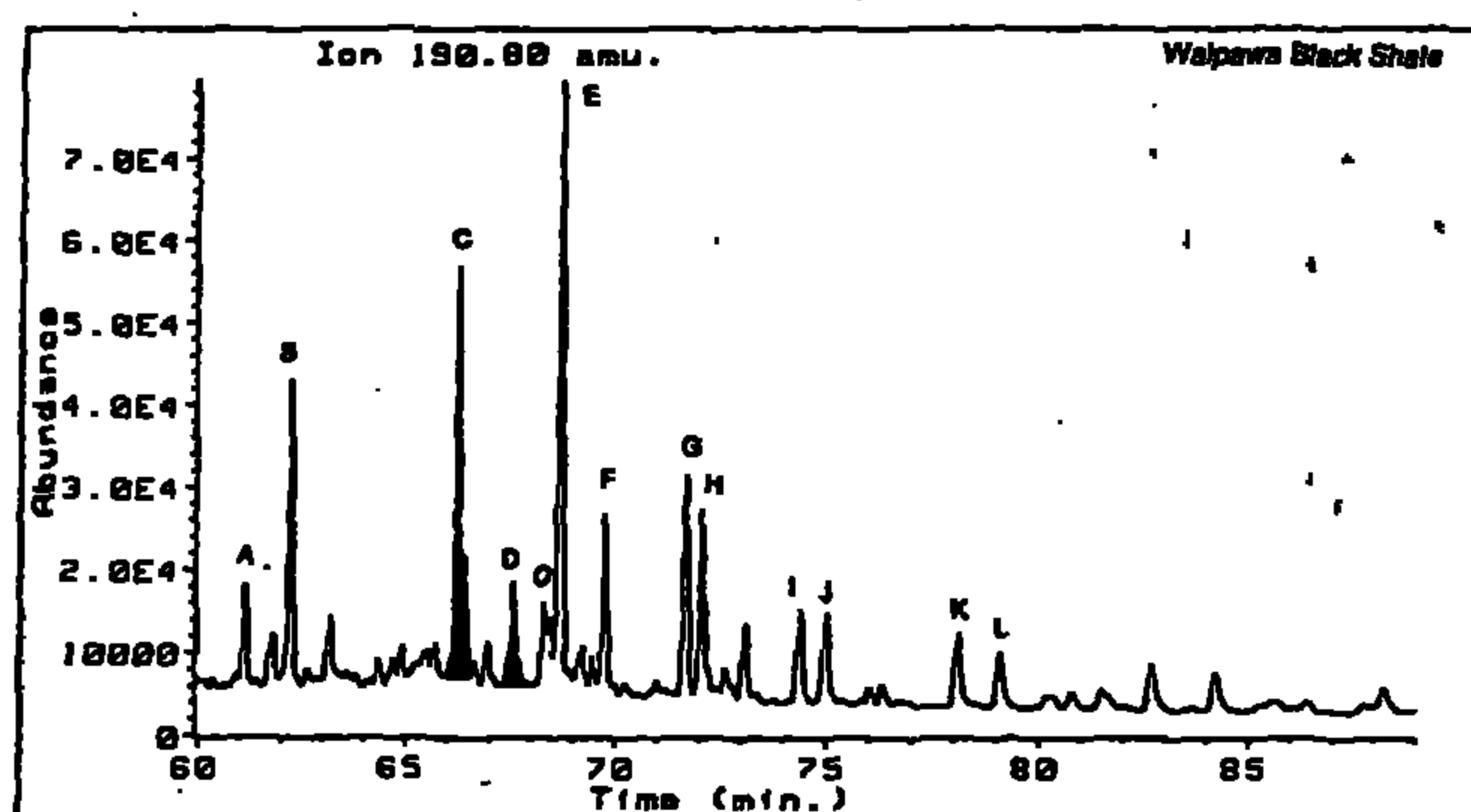


Fig. 6. Comparison of hopane (191m/z) and sterane (217m/z) chromatograms artificially matured of Waipawa Black Shale to oil impregnated greensand (note high  $C_{30}$  content of each).

Shale, it would be reflected by a similar low maturity signature of the  $C_{30}$  steranes in the oil.

Also noted is the similarity of the oil likely to be sourced from the Waipawa Black Shale and the Kora oil from the northern Taranaki Basin (Reed, 1991). The two source facies are probably similar as both are marine oils with strong  $C_{30}$  sterane signatures.

#### Upper Cretaceous, Whangai Formation

The Upper Cretaceous siliceous Whangai shale lies conformably below the Waipawa Black Shale. It is a thicker formation reaching up to 500 m in places and has reported TOC levels of up to 2% (average = 0.8%) (unpublished). The Whangai shale is widespread throughout the East Coast Basin and is extensively stained with an oil which is very similar in characteristics to the three main seep oils in the north of the basin.

Biomarkers suggest that this shale is predominantly marine. The higher hopane/moretane ratio and  $C_{31}$  &  $C_{32}$  S:R ratios in the m/z191 ion chromatogram (figure 4) show the organic matter to be moderately mature. Trace amounts of oleanane also occur in the Whangai shale, indicating minor terrestrial influence or contamination. Tricyclic terpanes show the dominantly marine nature of the Whangai shale with the absence of tetracyclic compounds other than  $C_{24}$  (figure 5) (de Grande et al., 1993).

In general the significant abundance of  $C_{30}$  sterane observed in the Waipawa Black Shale is absent from the Whangai shale. The small amounts of  $C_{30}$  steranes found in the Whangai shale are likely to be dinosteranes, an isomeric form of the 4-methyl steranes, rather than the n-propyl cholestanes previously seen in the Waipawa Black Shale. The kerogen of the Whangai Shale has a lack of biodegraded and amorphous material and an abundance of dinoflagellates determined by visual kerogen. In most cases,  $C_{27}$  steranes are

more abundant than the  $C_{28}$  and  $C_{29}$  steranes, which is more consistent with the seep oils than the Waipawa Black Shale.

Sulphur content is lower (average = 0.8%) than that of the Waipawa Black Shale, and Rock-Eval analysis suggests a lower S2 peak (average = 1.0 mg HC/g rock). Isotopic data for the Whangai shale ( $\delta^{13}C_{\text{sat}} = -29.0$  per mil) gives good correlation of this potential source rock with the main seep oils. Extensive oil stains are found within Whangai shale, especially in the northern East Coast Basin. Artificial maturation studies of Whangai shale have produced a pyrolysate which is similar to these stains (Rogers, 1993, unpublished). The similarity of geochemical biomarkers from the three main oil seeps to the stains found in this marine shale indicates that the Whangai shale is the most likely potential source for the oil seeps found to date.

#### Lower Cretaceous, Karekare Formation

The Karekare Formation (Lower Cretaceous) is mainly a blue-grey marine siltstone. The sequence is up to 2-3km thick and widespread throughout the East Coast Basin. The TOC contents of up to 2% (average = 0.5%) (unpublished) suggest that on a carbon basis the source potential of this formation is reasonable. The sulphur content is low (<0.2%). Although Rock-Eval analyses indicate smaller S2 peaks (0.4 mg HC/g rock), it is still possible for the Karekare Formation to be a potential source. Carbon isotopes for the Karekare Formation ( $\delta^{13}C_{\text{sat}} = -27.9$  per mil) are still within the accepted correlation range for the oils.

The Karekare Formation is dominated by marine biomarkers, which also indicate that the maturity level of the formation is only slightly less than that of the seep oils. Tricyclic terpanes,  $C_{27}$  diasteranes and  $C_{27}$  steranes are all abundant (figure 4), indicating a marine-dominated character. The  $C_{30}$  steranes are present only in trace quantities and are likely to be similar in nature to those of the Whangai Formation. The maturity of the Karekare Formation is consistent with that

required for hydrocarbon generation, and there is a good correlation of the biomarker characteristics with those of the Whangai Formation. This suggests that the Karekare Formation is also a potential source rock for the oil seeps.

#### **Early Cretaceous Formations, Urewera Greywacke**

Below the Karekare Formation lies the Waitahaia, Te Wera, and Koranga formations and the basal Urewera greywacke. These Early Cretaceous sediments are typically marine siltstones and mudstones with some sandstones present. The overall thickness of the Urewera Group sediments is up to 3 km (Grindley, 1960). Source potential of these formations is relatively low with TOCs up to 0.5% and Rock-Eval S2 index = 0.2 mg HC/g rock. Carbon isotopes for these lower formations are slightly lighter than the Karekare Formation ( $\delta^{13}\text{C}_{\text{saturation}} = -27.3$  per mil) suggesting only a tenuous correlation with the oil seeps.

#### **Conclusions**

From this study it is apparent that the three main seep oils in the northern area of the basin are of a similar nature. They are predominantly marine oils of moderate maturity, and exhibit similar biomarker characteristics. Previous studies have suggested that a possible source for these oils could be the younger Miocene sediments or the Waipawa Black Shale, particularly as both occasionally contain areas which are impregnated with oil.

This study has also found that sediments younger than Cretaceous age are, in general, immature for hydrocarbon generation and do not correlate to the northern main seep oils at Waitangi, Rotokautuku and Totangi. This includes the Waipawa Black Shale.

The results obtained from the present detailed study suggest that the Waipawa Black Shale is likely to be generating oil due to its high organic content and deposition in an anoxic environment, including high algal and sulphur content. However, this oil has significantly different biomarker characteristics and isotopic signatures to those of the seep oils of the northern East Coast Basin.

Potential Cretaceous source formations are still under consideration. It is likely that due to the geochemical and maturation similarities of the seep oils, there is either a common Cretaceous source rock for the seeps, or several Cretaceous formations deposited under similar conditions contributing to the hydrocarbons generated by the main seep areas in the northern part of the East Coast basin.

The most likely Cretaceous source rock is the upper Cretaceous Whangai shale which has sufficient total organic carbon content, a suitably high S2 index as determined by Rock-Eval and exhibit a good correlation on the basis of both geochemical biomarkers and carbon isotope data, to these oil seeps. In addition there is extensive oil-staining within the Whangai shale and there is an excellent similarity between the biomarker and isotope characteristics of the stains with those of the main oil seeps. It is possible that the Whangai Formation is generating (similar to the Waipawa Black Shale) with oil migrating through the shale along fractured pathways.

An alternative source is the mid-Cretaceous Karekare Formation and equivalent facies. This formation, although lower in total organic carbon and Rock-Eval S2 index, may contain localised areas, as yet unidentified, which are richer in organic carbon thus offering better source potential. A

suitable correlation, based on biomarker and isotope data, can be made between the Karekare Formation and the main seep oils.

The present and ongoing detailed study has therefore identified at least two different possible source rocks for the East Coast oils. These are a Cretaceous source (possibly upper Cretaceous) generating the main oil seeps in the northern part of the basin, and the Paleocene Waipawa Black Shale generating the seeps and impregnations in the southern part. However, there is some lesser evidence for a Cretaceous source for some seeps and impregnations in the south and evidence for a Waipawa Black Shale source for some impregnations in the north.

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