VARIATIONS IN SOURCE POTENTIAL AND MATURATION OF NEW ZEALAND COALS, BASED ON RELATIONSHIPS BETWEEN CONVENTIONAL COAL CHEMISTRY, ROCK-EVAL PYROLYSIS, AND GCMS BIOMARKERS

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Abstract

Rock-Eval pyrolysis data are presented for 6 late Cretaceous and 28 middle Eocene New Zealand coals, previously analysed by gas chromatography–mass spectrometry. Most samples have more than 90% vitrinite and all have more than 80%, hence variability in properties relates primarily to vitrinite chemistry. Relationships between S1+S2 and volatile matter, and between Tmax and rank(S), demonstrate that these Rock-Eval parameters have relevance to the bulk chemistry of coals. S3 and TOC values, and any ratio based on these, are demon-strated to be unreliable in this suite of analyses.

Values for S1 and S2 yield suggest that the coals have not generated significant hydrocarbons before high volatile A bituminous rank, and that significant generative potential may remain even at medium volatile bituminous rank. Relative yields for isorank serial samples indicate that perhydrous coals generate more hydrocarbons than coals of average type. Rock-Eval data support earlier suggestions that anomalous behaviour by certain GCMS biomarkers between high volatile A and medium volatile bituminous rank may result from an expulsion event of particular significance for perhydrous coals. Methane generation by coals accelerates at these ranks and expulsion of liquid hydrocarbons may be assisted by gaseous solution.

Introduction

This paper is a sequel to work presented at the 1991 Oil Exploration Conference by Newman, Johnston and Lake (1992), *Effects of isorank variation in vitrinite chemistry on vitrinite reflectance and GCMS maturation indicators*. Rock-Eval data are now presented for the same sample suite discussed in the previous paper, and interpretations of generative potential and maturation history are extended based on integration of all available information. The principal introductory material presented by Newman et al. (1992) will not be repeated in detail here. In essence, evidence indicating a terrestrial source for Taranaki and Westland oils was reviewed, including information suggesting some diversity in source character (Czochanska, 1987,1988). A primary objective was to assess whether different kinds of coal mature at different rates, and perhaps generate different total volumes of hydrocarbons. Both postulates were tentatively answered in the affirmative, and unexpected high rank trends in GCMS biomarkers were hypothesised to record an expulsion event. The principal findings of Newman et al. (1992) are endorsed by work presented in the current paper.

The coals analysed are strongly vitrinite-dominated, particularly Eocene coals from Buller Coalfield, which typically have 93%+ vitrinite. Despite this uniformity of coal type in the sense of maceral group proportions, suites of isorank serial samples exhibit considerable variability in chemical and physical properties, as a result of isorank variation in vitrinite chemistry. Hence the usual meaning of coal "type" is extended to include inherent vitrinite chemistry (Newman et al., 1992). Although repeat analysis of individual samples was not undertaken, two serial samples, 35/765 & 35/766, have very similar bulk properties in terms of traditional coal analyses (e.g. proximate analysis and vitrinite reflectance). The degree to which these samples cluster on plots relating GCMS or Rock-Eval data to volatile matter or vitrinite reflectance provides an indication of the repeatability of the method in question, and the extent to which it representatively characterises the whole coal substance. Consequently, this sample pair is encircled, where appropriate, on figures in this paper. Another sample pair, 35/315 and 35/316, also has very similar bulk properties and proved useful for evaluation of GCMS data (Newman et al. 1992). However, the Rock-Eval analysis for 35/315 appears particularly anomalous (see later section), hence this sample pair is not always encircled.

Sampling and Analysis

Exploration of coalfields on the West Coast of the South Island has provided a large number of well characterised coal samples representing a wide range of depositional and burial histories. A suite of 34 Cretaceous and Eocene coals has previously been petrologically and chemically analysed, including gas chromatography–mass spectrometry (GCMS), in an attempt to elucidate the effect of isorank variation in vitrinite chemistry on petroleum source potential (Newman et al., 1992). This same suite of samples has also been analysed by Rock-Eval.

Coalfield samples offer a greater diversity of characteristics, over a wider range of ranks, than is generally available from petroleum wells. West Coast seams include examples which exhibit systematic variation in chemistry from roof to floor. Such sites provide serial plies with a common burial history, and these sample suites allow depositional influence on analytical properties to be evaluated in isolation from the complicating effects of rank variation. West Coast coals also encompass higher ranks than have so far been represented by drilling of correlative sequences in the Taranaki Basin, allowing fully mature samples to be investigated.

Table 1 provides stratigraphic and locality information for the samples discussed in this paper. More information is provided in the *1991 New Zealand Oil Exploration Proceedings* (Newman et al., 1992). Proximate and ultimate analyses, specific energy and Rock-Eval data appear in table 2. Rock-Eval analysis was undertaken at the United States Geological Survey in Denver, using a "Rock-Eval II plus TOC" manufactured by Delsi Inc. Other chemical analyses were undertaken by Coal Research Association of New Zealand.

Rank and Maturity

The sample suite encompasses ranks from high volatile B bituminous to medium volatile bituminous (ASTM). As shown by vitrinite reflectance and volatile matter (figure 1), type variation causes considerable overlap in the properties of coals with significantly different rank. Suggate rank (Suggate 1959, Suggate et al., 1982, 1993) is the only system which accommodates this type variation and sensitively differentiates the sample suites in terms of their relative rank. Vitrinite reflectance and Rank(S) are illustrated together in figure 2, in which the vertical lines are serial sample tie lines, and the horizontal marks represent individual samples.

Table 1. Geographic and stratigraphic information for the West Coast sample suite.

Sample	Drillhole	Seam	Ply	Thickness (m)	Location	Reference
35/763	1281	1	8–10	1.72	U. Waimangaroa	Barry (1986)
35/764	"	1	11–13	1.78	(Buller)	+
35/765	"	1	14–23	6.02	"	Newman (1987)
35/766	"	1	24	0.63	"	"
39/315	1385	1	1	0.80	"	"
39/316	"	1	2	0.40	"	"
39/317	"	1	3–5	4.42	"	"
39/318	"	1	6–8	4.75	"	"
35/767	1334	1	1–3	1.80	"	"
35/768	"	2	4–5	1.05	"	"
35/769	"	2	6–7	0.70	"	"
31/083	1222	1	1–5	3.70	Webb (Buller)	Newman (1985)
31/084	"	1	6–16	7.30	"	"
31/085	"	1	17–20	2.51	"	"
31/110	1241	1	1–5	2.35	"	"
31/111	"	1	6–13	5.00	"	"
31/112	"	1	14–18	2.85	"	"
31/044	1215	1	3–7	2.48	"	"
31/048	"	1	19–24	2.73	"	"
30/926	PRDH2	1	1	0.66	Pike River	"
30/927	"	1	2	1.74	"	"
30/928	"	1	3	2.50	"	"
30/971	PRDH6	1	1	1.32	"	"
30/985	"	1	2	3.18	"	"
33/045	"	1	3	6.75	"	"
30/150	6450N	1	3	1.00	"	"
30/151	outcrop	1	4	2.00	"	"
30/153	"	1	6	2.00	"	"
27/528	950N	Paparoa	M4	1.35	"	"
27/531	outcrop	"	"	1.86	"	"
27/522	2650N	"	"	M3	4.60	"
44/080	Liverpool	Kimbell	0.20		Greymouth	-
44/081	Mine #3	seam	0.20	"	-	
44/082	"	"	0.20	"		

Table 2. Analytical data for the West Coast sample suite. Ultimate analyses are not available for the outcrop samples.

Sample #	S1 KgHC/t	S2 KgHC/t	S3 KgHC/t	TOC %	Tmax °C	Vitrinite reflect. % Ro rand	Carbon %dmmSf	Hydrogen %dmmSf	Oxygen %dmmSf	Sulphur a.d.%	Moisture a.d.%	Ash a.d.%	Volatile matter a.d.%	Specific energy a.d.%	Volatile matter %dmmSf	Specific energy %dmmSf
35/763	7.80	232.00	16.00	63.54	426	.56				4.47	3.1	1.5	47.2	32.81	49.7	35.79
35/764	5.17	198.57	16.42	63.64	432	.66	82.9	5.7	10.2	3.03	4.8	0.7	40.4	32.22	42.6	34.97
35/765	4.02	188.96	16.89	64.01	436	.67				.80	5.1	0.4	38.5	32.45	40.7	34.58
35/766	4.37	188.33	17.50	65.28	435	.66	83.0	5.8	10.0	.57	5.0	4.0	37.5	31.54	41.0	34.93
39/315	3.96	161.50	16.60	72.18	420	.55				4.51	5.2	7.3	40.7		47.4	
39/316	3.33	200.00	15.55	77.67	433	.59	82.2	6.0	10.4	1.84	6.1	2.2	42.5	31.14	46.5	34.70
39/317	2.64	152.45	19.24	77.92	434	.64	81.7	5.4	11.5	.45	8.0	0.2	36.3	31.16	39.5	34.05
39/318	3.10	172.41	18.27	70.30	434	.64	82.1	5.7	10.9	.47	6.8	3.1	37.8	30.72	41.7	34.36
35/767	3.96	196.22	16.60	71.42	434	.62	81.9	5.8	11.1	2.25	4.9	2.6	40.2	31.91	43.5	35.37
35/768	5.28	229.43	13.96	63.89	429	.52	83.3	6.2	9.6	5.00	2.3	6.6	46.2	31.54	49.9	36.27
35/769	4.52	205.28	13.58	61.77	426	.58	84.0	6.3	8.7	4.90	2.9	11.6	40.8	29.08	46.5	36.19
31/083	5.09	150.58	23.13	66.97	460	1.02	88.3	5.6	4.9	2.81	1.0	4.6	31.5	34.11	32.6	37.23
31/084	4.71	158.49	21.50	67.43	463	1.10	88.4	5.3	5.1	1.14	1.1	.3	29.5	35.88	29.7	36.71
31/085	3.46	139.23	21.92	55.47	463	1.17	88.8	5.3	4.9	3.78	.8	4.8	27.7	33.72	28.1	37.08
31/110	4.50	140.39	24.70	66.69	455	.94	86.7	5.3	6.9	3.55	2.6	1.6	32.4	33.08	33.3	35.67
31/111	3.80	150.40	22.00	74.81	460	1.08	88.6	5.3	4.8	1.14	1.1	.4	30.3	36.08	30.5	36.97
31/112	3.20	105.60	18.80	55.41	466	1.19	89.8	5.1	4.0	3.83	.9	2.8	27.2	34.33	27.5	37.03
31/044	5.68	202.35	19.20	56.48	444	.82	87.1	5.7	6.1	4.15	.9	.9	38.4	34.95	38.6	36.82
31/048	6.73	167.69	21.53	67.24	451	.98	87.3	5.4	6.2	2.60	1.3	1.1	32.9	34.84	33.2	36.46
30/926	6.45	145.80	12.58	53.76	442	.78				8.12	1.3	34.5	32.0	21.42	46.6	
30/927	14.03	263.07	14.61	68.01	443	.62	86.6	6.5	5.7	2.79	1.2	4.2	46.6	33.84	48.7	36.64
30/928	7.54	223.39	158.40	76.61	444	.73	84.3	6.1	8.6	.84	1.1	3.1	43.7	33.87	44.8	35.61
30/971	9.00	243.20	19.20	61.71	439	.66	87.8	6.4	4.9	6.32	.8	7.0	44.5	32.56	47.5	37.62
30/985	5.66	261.13	14.71	67.42	445	.69	86.8	6.2	6.0	2.34	.9	3.1	44.4	34.65	45.6	36.71
33/045	3.33	244.70	19.60	71.31	444	.70	85.2	6.2	7.7	.64	1.2	5.1	41.7	33.97	44.0	36.45
30/150	10.00	251.66	13.75	60.94	438	.53				7.28	1.1	6.3	49.4	32.20	53.7	37.21
30/151	5.10	229.78	16.59	67.53	436	.63				3.70	1.4	3.3	45.4	33.00	47.8	35.79
30/153	2.00	208.00	16.80	72.57	440	.72				0.72	1.1	1.6	42.8	33.37	43.9	34.55
44/080	4.71	184.15	18.11	62.92	441					0.33	2.0	16.3	31.8	28.52	37.6	35.72
44/081	2.35	153.72	21.56	78.48	442	.81				0.24	2.4	2.0	34.7	33.63	36.2	35.32
44/082	2.07	156.98	22.64	78.73	442	.75				0.27	2.4	2.3	34.3	33.64	35.8	35.48
27/528	.98	158.43	18.03	71.83	443	.99				1.26	1.4	10.7	31.9	31.13	34.8	36.26
27/531	4.20	222.40	19.20	72.92	447	.83				0.36	0.8	6.9	37.0	33.99	39.4	37.22
27/522	2.74	188.23	20.39	72.31	437	.73				.44	1.8	6.3	38.3	32.25	40.5	35.44

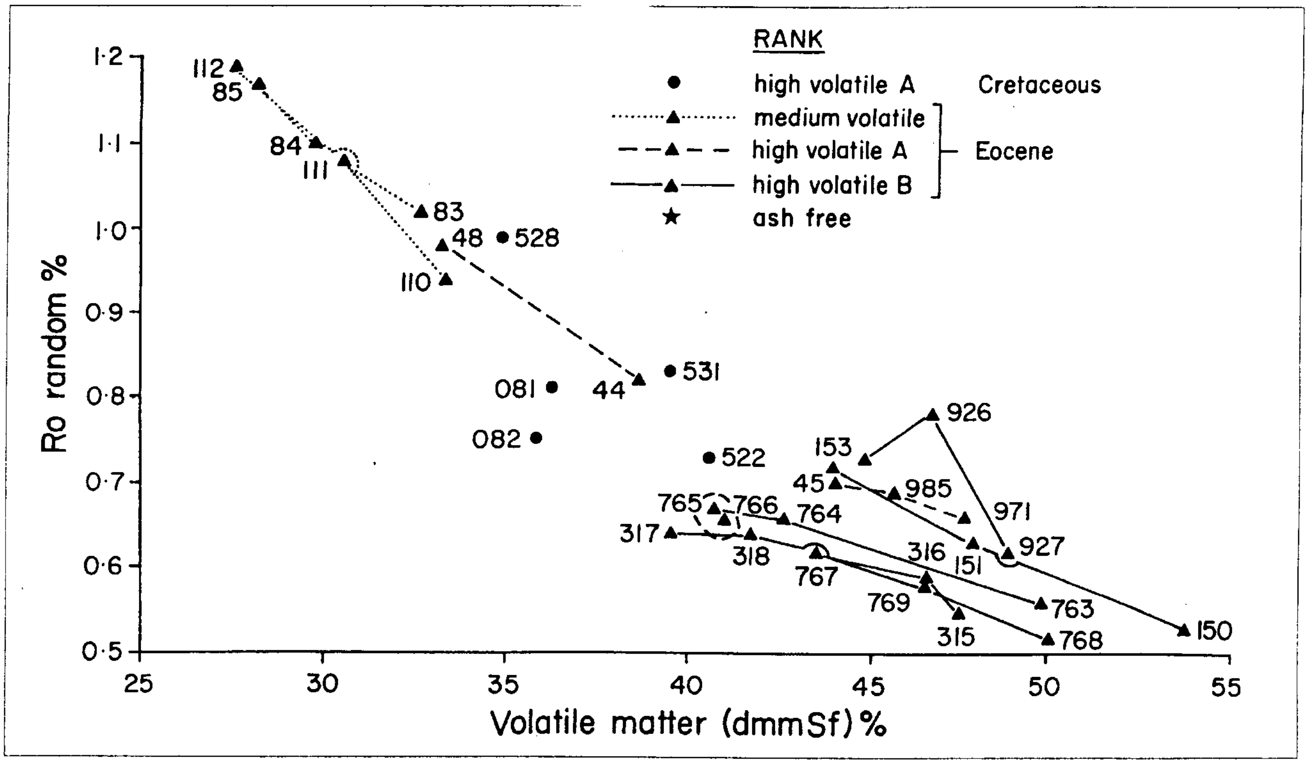

Fig. 1. Vitrinite reflectance plotted against volatile matter, all samples. Tie lines link the members of isorank serial sample suites. The significance of sample pair 35/765 and 35/766 is discussed in the Introduction. Correction of volatile matter to dry mineral matter and sulphur free basis is explained in Newman et al. (1992).

Because ultimate analyses are not available for all samples, Rank (S) has been determined on the basis of volatile matter and specific energy (Suggate, 1959). However, where possible Rank (S) should be determined on the basis of elemental analyses, using the revised rank system provided in Suggate et al. (1993). For the purposes of the present paper, the indication of relative ranks provided by the original system is adequate. The ASTM rank boundaries shown on figure 2 are based on estimated moist mineral matter free calorific values for coal of "average type" (Newman, et al. 1992), and are therefore very approximate.

In all figures the members of each serial sample suite are linked by tie lines. These lines depict the range over which coal properties can vary despite the absence of any rank variation. Figures in this paper and in Newman et al. (1992) demonstrate that properties commonly regarded as maturity parameters, in the sense of coal rank and thermal history, are strongly influenced by inherent variability in vitrinite chemistry. This variability affects vitrinite reflectance and fluorescence, GCMS biomarkers, Rock-Eval parameters and many other properties, and is attributed to depositional and early burial controls on peat composition. Such isorank variability may well result in differences in maturity, in the sense of hydrocarbon generation. Rank and maturity are therefore not synonymous qualities in the case of coals with diverse starting chemistries (Suggate 1990). This distinction has a precedent in the case of Type 1 and II organic matter, which have for some time been acknowledged to "mature" at different temperatures (Tissot & Welte, 1984). Coal "rank" therefore relates specifically to thermal history, whereas "maturity" refers to the chemical state achieved in response to both depositional and burial history. The possible detection and timing of hydrocarbon generation from West Coast coals is discussed later.

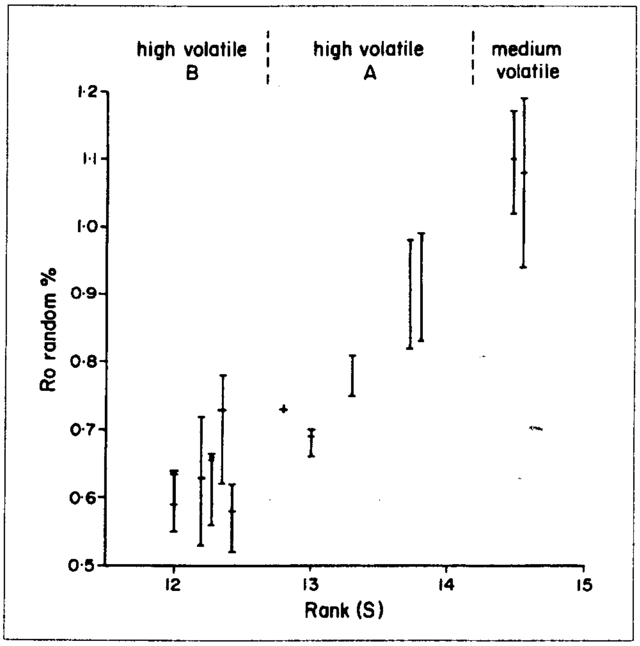

Fig. 2. Relationship between vitrinite reflectance and rank(S), all samples. Vertical lines are serial sample tie lines, on which individual samples are plotted as short horizontal marks. Rank classes shown at top of figure are based on the ASTM rank classification.

Sulphur

Serial ply suites of West Coast Eocene coals (Brunner Coal Measures) frequently exhibit a direct relationship between volatile matter and sulphur. The relationship persists even after the volatile matter value has been appropriately corrected to eliminate contributions from decomposition of organically bound and sulphide sulphur during volatile matter determination (achieved at around 900°C in the absence of air). Interpretation of these trends is complicated. Many Eocene seams have a depositional history of progressively rising water table, resulting in perhydrous characteristics (high volatile matter) upwards in the seam. Due to subsequent marine transgression, a sulphur gradient is often superimposed on this volatile matter gradient. Highest sulphur values occur towards the roof, and sometimes the floor, resulting from saline groundwater which accessed the peat after initial burial, sometimes after compaction beneath sediment thicknesses of 50 m+. Consequently, in a number of cases an increase in sulphur upwards in the seam parallels increasing perhydrousness. However, the sulphur has not necessarily caused the perhydrousness, as can be shown by examples where sulphur also accessed the floor, resulting in high sulphur values in the lower part of the seam in coal which is relatively subhydrous (e.g. drillholes 1222 and 1241, table 1). Such cases help to differentiate the effects of a syndepositionally high water table from postburial sulphur access. However, in many instances sulphur has accessed only the roof, hence elucidation of controls can be difficult.

The role of sulphur in determining coal properties appears to depend on the timing of sulphur access to the peat (Newman, 1991). In the case of Pike River Brunner coals (table 1), marine water accessed the peats prior to burial, and in this and other related examples sulphur appears to have fundamentally influenced organic chemistry and related properties (Quick, 1992). In such cases, correlations between sulphur and Rock-Eval or GCMS parameters are considered to have definite causative significance, and high sulphur coals could be expected to generate hydrocarbons earlier than low sulphur coals of equivalent rank. However, the highest rank trends discussed in the present paper relate only to Buller coals, hence to perhydrous characteristics which result not from sulphur, but from a high water table and restricted oxygen access during peat accumulation. Unfortunately, similarly high rank coals exhibiting pre-burial sulphur enrichment are not currently available for study.

Rock-Eval Data

Rock-Eval pyrolysis was developed in the 1970s as a rapid and relatively cheap method for estimating the source character and maturity of whole rock samples. Sample size can be very small (100 mg) and grinding is the only preparation needed. Various workers have discussed limitations of the method (e.g., Peters, 1986; Langford and Blanc-Valleron, 1990), and the reproducibility of analyses between different laboratories is sometimes poor (Cook, R.A. 1987; van der Meulen, 1989). Therefore, although useful as a rapid method for screening large numbers of samples, Rock-Eval is ideally interpreted with reference to the results of other characterisation techniques.

Rock-Eval pyrolysis can be applied to coal samples (Teichmuller and Durand, 1983; Verheyen, et al., 1984; Suggate and Boudou, 1993), but dilution with an inert material and cautious interpretation of results, are recommended (Peters, 1986). Typically, Rock-Eval of coals has been undertaken in an attempt to define their hydrocarbon source potential and the timing of oil generation. In addition, coal provides an opportunity to evaluate correlations between Rock-Eval data and a variety of other chemical characteristics, without the necessity to prepare kerogen concentrates. The study reported in this paper utilises isorank suites of geologically and chemically characterised New Zealand coals to evaluate the sensitivity of Rock-Eval parameters to coal type variation, and the ability of the method to define the relative hydrocarbon source potential and maturation rates of different coal types. Analyses appear in table 2.

Tmax

Tmax is the temperature at which the maximum amount of hydrocarbons is generated by pyrolytic degradation (S2 peak) during Rock-Eval pyrolysis. Tmax is known to be influenced by both coal type and rank, and the relative influence of these controls for the West Coast samples (figure 3) corresponds well with the relationship shown by Suggate et al. (1993). As in figure 2, the vertical lines link the members of the isorank serial sample suites. For the West Coast coals, Tmax appears to define coal rank about as well as vitrinite reflectance (figure 2). Consequently the two parameters exhibit a positive correlation (figure 4). A distinctive anomaly is sample 39/315, which has low Tmax and also low S2. Properties of this sample usually correspond very closely with those for 39/316 (Newman et al., 1992), and there is no clear reason why the Rock-Eval response is different. An experimental problem appears likely, because Tmax and S2 are dependant parameters, and all other sample characteristics are as expected.

It is apparent from figure 4 that at the lowest rank investigated — high volatile bituminous B (solid tie lines) — any relationship between coal type and Tmax is erratic. At higher ranks (broken tie lines) Tmax is clearly lowest in the relatively perhydrous coals, which have the highest volatile matter and lowest reflectance in each isorank suite. Low Tmax may suggest easier thermal degradation of molecular structures in perhydrous coals. This trend cannot easily be attributed to sulphur in this case, because organic sulphur is high at both high and low extremes of Tmax in the two medium volatile bituminous sample suites analysed. As discussed in an earlier section, the introduction of sulphur-containing marine water to peat during very early burial may subsequently encourage early hydrocarbon generation, but the high rank Buller coals do not fall into this category.

Whereas low Tmax in perhydrous samples is consistent with retardation of other maturity indicators, including vitrinite

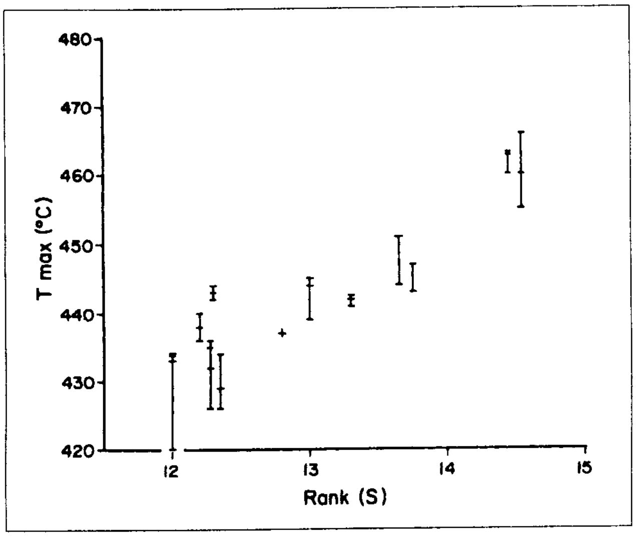

Fig. 3. Rock-Eval Tmax related to rank(S), all samples. Vertical lines are serial sample tie lines, on which individual samples are plotted as short horizontal marks.

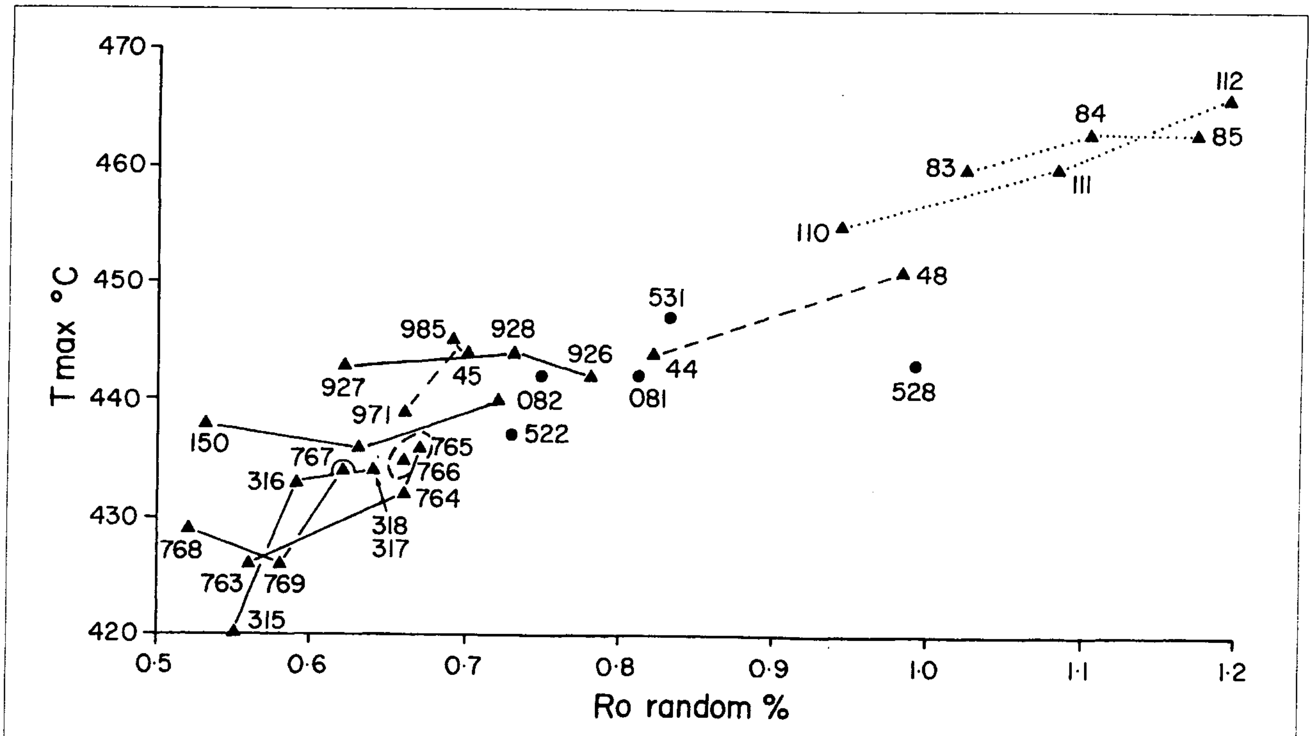

Fig. 4. Relationship between Rock-Eval Tmax and vitrinite reflectance, all samples. Tie lines link isorank serial samples, and a key to symbols appears in figure 1. The significance of sample pair 35/765 and 35/766 is discussed in the Introduction.

reflectance, this evidence may be misleading. GCMS biomarkers indicate that perhydrous coals actually have enhanced maturity (Newman et al., 1992), hence the relatively early thermal cracking indicated by low Tmax may in fact be manifest as early hydrocarbon generation.

S1, S2 and S3

S1 is the first Rock-Eval peak, and represents the hydrocarbons that can be thermally distilled from the sample at c.300°C. The second peak, S2, represents hydrocarbons generated at temperatures up to 550°C by pyrolytic degradation. S3 is the third Rock-Eval peak, and represents the amount of carbon dioxide generated at temperatures up to 390°C, analysed by thermal conductivity detection. S3 is discussed further in the following section. Although most of the West Coast coals analysed comprise well over 90% organic material, only 10% to 30% by weight of each sample reported as S1, S2 and S3 at these temperatures. This is about half the amount that reports as volatile matter during proximate analysis at standard analysis temperatures of c.900°C. The S1+S2 total is shown in relation to volatile matter in figure 5. Volatile matter is expressed dry mineral matter and sulphur free (dmmSf), for consistency with usage elsewhere. S1 and S2 are shown on their original whole coal basis, except that values for samples 30/926 and 39/315 are also shown ash free, to allow for suppression of S1 and S2 yield by dilution resulting from their relatively high ash content. Where proximate analyses are available, in the case of coals, normalising S1 and S2 to a mineral free basis is an alternative to calculating the Rock-Eval hydrogen index and oxygen index. These ratios, which are the more conventional approach to expressing organic matter composition on a mineral free basis, can be misleading as discussed below. In the case of coals with widely varying rank it may also be useful to correct S1 and S2 to dry basis, in order to meaningfully compare source characteristics. This correction has not been made for the West Coast coals reported here, because moisture variation is small.

S1 and S2 are shown in another form, and related to rank(S), in figure 6. Each serial ply suite is shown with highest volatile (most perhydrous) samples on the left, and lowest on the right. Values for the lowest rank samples (drillhole 1385, tables 1 & 2) suggest a trend to lower S1+S2 yield in ranks below high volatile A bituminous, reported by Teichmuller et al. (1983) and implicated in hydrogen index trends reported by Suggate et al. (1983). As Teichmuller et al. (1982) also noted, S1 does not increase with increasing rank; the implications of this are discussed later.

Below rank(S) 14, the highest volatile samples in each suite almost invariably have highest S1+S2, as also shown by figure 5. Usually both S1 and S2 individually conform to this trend, which is attributed to type variation. S1+S2 exhibits no clear trend in relation to coalification until after a rank of high volatile A bituminous (c. rank(S) 13) is attained. Even then, the values observed for medium volatile bituminous coals (greater than c. rank(S) 14) are not dramatically lowered. In fact, the most compelling evidence that increasing rank eventually results in hydrocarbon generation comes less from absolute S1+S2 values than from changes in the relative proportions of values for serial plies of contrasting high and low volatile type. That is, above high volatile A bituminous rank, the S1+S2 yield for perhydrous coals falls *below* that of adjacent plies of intermediate type, whereas at lower ranks high volatile coals have the *highest yield*. Both high rank suites (drillholes 1222 and 1241) exhibit this trend, which is consistent with earlier suggestions, based on hopane biomarker ratios, that perhydrous coals may mature and commence generation and expulsion of hydrocarbons before relatively subhydrous coals of equivalent rank (Newman et al., 1992). The S1+S2 values for all coal types indicate that medium volatile bituminous coals may still have significant source potential despite their relatively high rank.

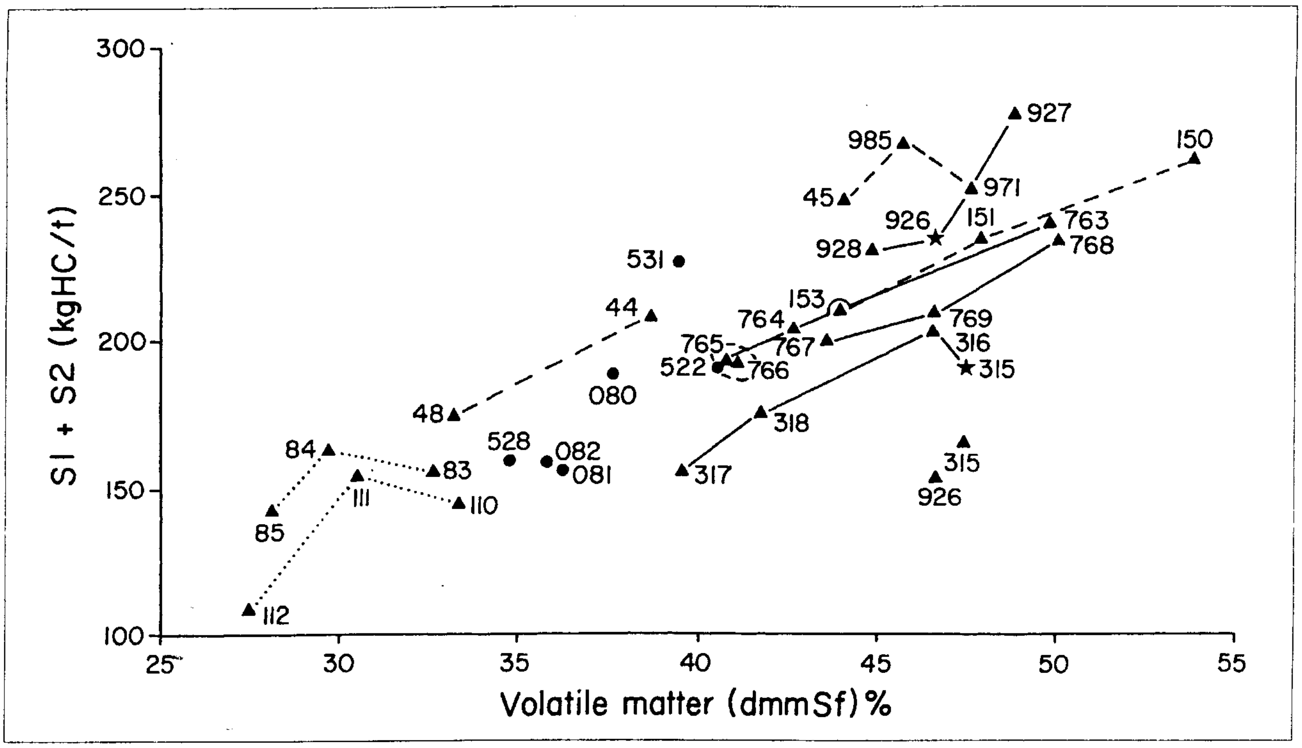

Fig. 5. Rock-Eval S1+S2 related to volatile matter, all samples. Tie lines link isorank serial samples, and a key to symbols appears in Figure 1. The significance of sample pair 35/765 and 35/766 is discussed in the Introduction.

TOC, HI and OI

Total organic carbon (TOC) can be determined by a variety of methods, and is a measure of the relative proportions of organic and inorganic material in the sample. The Delsi Rock-Eval II, used for analysis of the West Coast samples, provides TOC by summing the carbon in the pyrolyzate with that obtained by oxidising the residual organic matter at 390°C. As noted by Peters (1986), mature samples yield poor TOC values because this temperature is insufficient for complete combustion. Figure 7 relates TOC values to carbon determined by ultimate analysis (whole sample basis) for some of the the West Coast coals. TOC is significantly lower

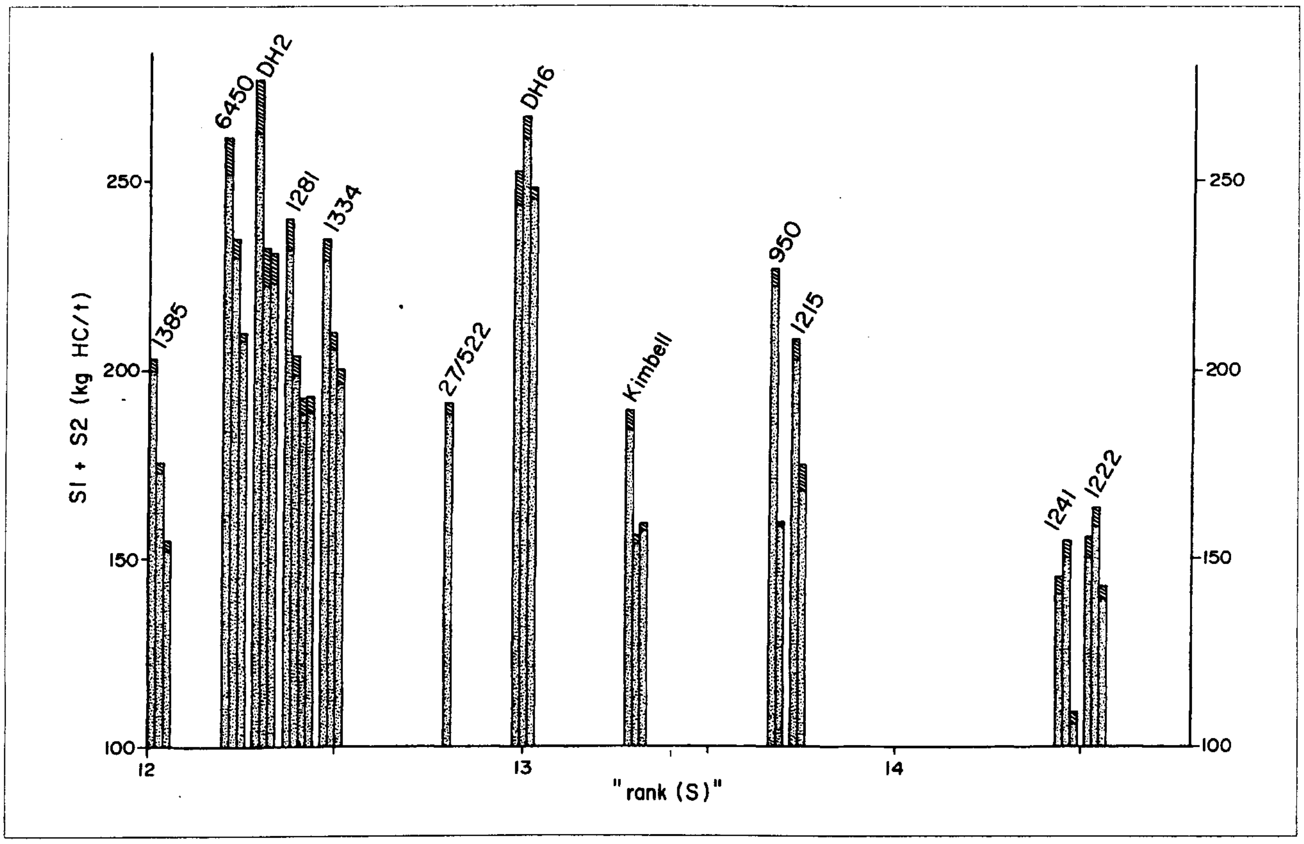

Fig. 6. Rock-Eval S1 (hatched) and S2 (stippled) yield for all serial sample suites, in order of increasing rank (S). Within each suite, highest volatile samples are positioned on the left, lowest on the right. Sample suite numbers refer to Table 1.

than ultimate carbon, particularly for the highest rank coals. Consequently, the hydrogen index (HI) and oxygen index (OI) values, which are the ratios S2/TOC and S3/TOC respectively, can be expected to be too high. For this reason, a modified HI and OI have been calculated, using ultimate carbon (whole coal basis) in place of TOC in each case. HI and OI are commonly plotted as a modified form of the van Krevelen diagram (figure 8), but it is apparent that the highest rank West Coast coals plot in the lowest rank position, due to OI values which are too high. In contrast, the true relative ranks of the coals are accurately portrayed on an H:C/O:C diagram, using ultimate analysis data (figure 9). It therefore appears that values for S3, on which OI is based, are too high for the highest rank coals. The reason for this anomaly is not clear. Peters (1986) suggests that high S3 values result from weathering, but apart from 31/110 the Buller samples appear unweathered. Certainly, other evidence of weathering, such as high Tmax and low S2 (Peters, 1986), is absent. Most other possible causes of high S3 cited by Peters appear unlikely, although he states that the peak is susceptible to instrumentation problems.

Discussion

Rock-Eval analysis was not designed for coal characterisation and has not been subject to rigorous evaluation of repeatability and reproducibility. It is therefore not surprising that conventional methods of coal analysis, which are constrained by international standards, are better suited to definition of coal properties. However, the correspondence between S1+S2 and volatile matter yield, and between Tmax and vitrinite reflectance, is better for West Coast coals than was originally anticipated. On the other hand, high S3 and low TOC are evident, and it seems clear that TOC should not be determined by the Rock-Eval II method (c.f. Bal, this volume). Ideally, coals of interest in the context of petroleum exploration should be routinely quantified by conventional proximate, ultimate and specific energy analyses, where sufficient material is available (20 to 50 grams), to aid interpretation of Rock-Eval and other data.

S1, S2 and Tmax correlate with both coal type and rank, hence exhibit significant variation among samples which have the same thermal history but differing initial composition. It seems probable that the differences in hydrocarbon yield during Rock-Eval analysis are indicative of differences in source potential during natural coalification. In the case of West Coast coals reported in this paper, such differences relate primarily to vitrinite chemistry, rather than overall maceral group proportions. Tmax of mature coals is lowest in perhydrous samples. Perhydrous coals also exhibit a marked decline in S1+S2 yield with increasing rank, relative to less perhydrous coals with the same burial history. Maturation rate assessed on this basis does not correlate with sulphur variation in the case of the highest rank coals included in this study. However, the character of certain lower rank coals suggests that sulphur is a potentially significant influence on vitrinite chemistry and source potential when peat is accessed by sulphur before, or at the time of, initial burial by sediment.

Gas Chromatography–Mass Spectrometry

The above evidence from Rock-Eval suggests that differential generation and expulsion of hydrocarbons affects the relative properties of serial plies somewhere between high volatile A and medium volatile bituminous rank. This timing corresponds well with GCMS data reported earlier (Newman et al., 1992). For the present discussion, only Eocene members of the West Coast sample suite are shown on GCMS biomarker plots, because the Cretaceous coals provided conflicting results. Further research is currently underway to resolve this problem.

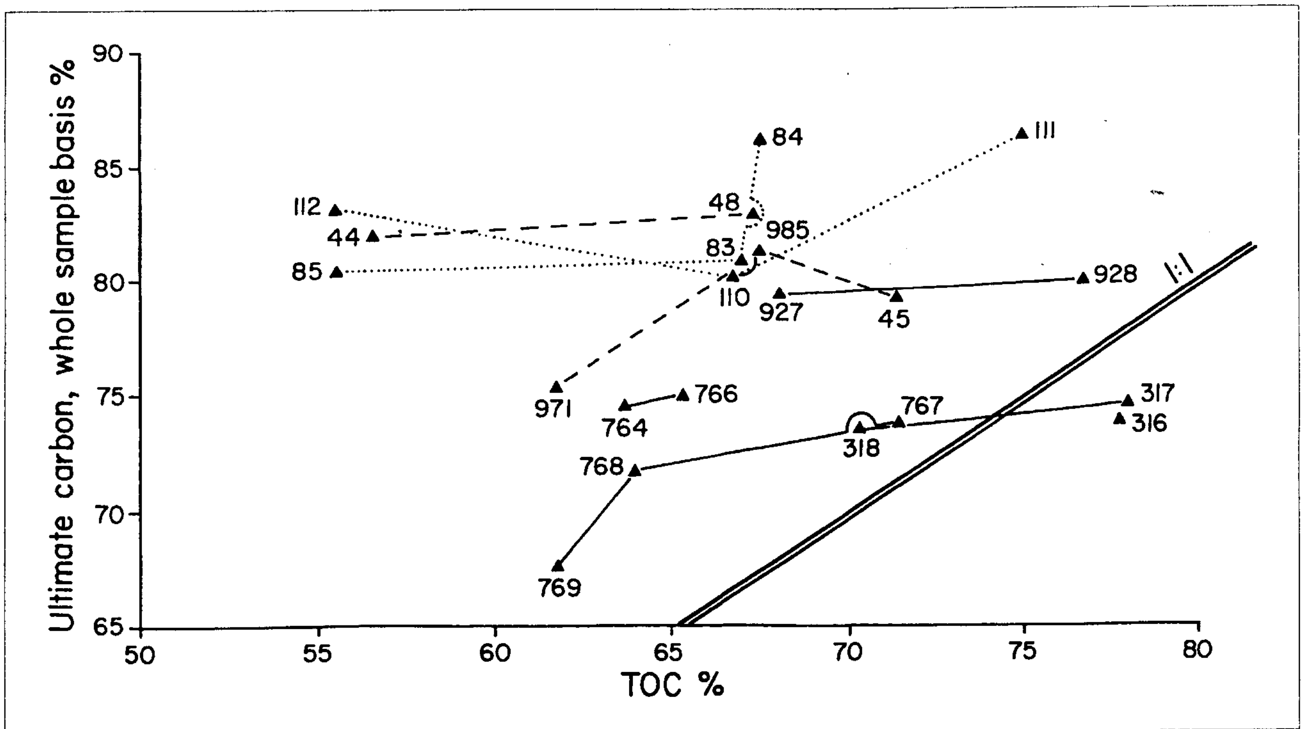

Fig. 7. Relationship between total carbon on a whole sample basis, derived from ultimate analysis, plotted against Total Organic Carbon determined by Rock-Eval analysis using the Delsi-II method. Tie lines link isorank serial sample suites. A key to symbols appears in figure 1. Only samples for which ultimate analyses are available are shown.

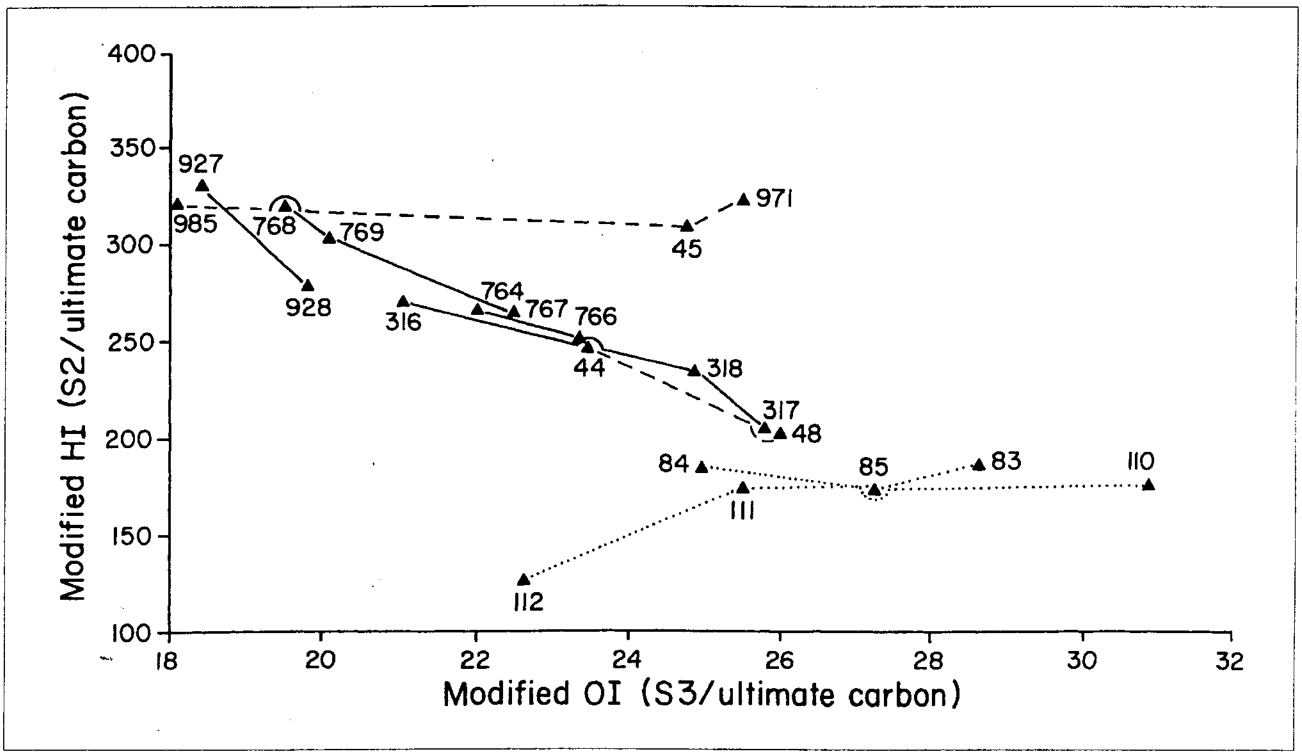

Fig. 8. Plot of Rock-Eval hydrogen index against Rock-Eval oxygen index, modified by using total organic carbon determined from ultimate analysis, instead of Rock-Eval TOC. Tie lines link isorank serial sample suites. Dotted lines define suites known to have highest coal rank, although on this plot they occupy a position which would normally be interpreted as low rank relative to the other sample suites. Only samples for which ultimate analyses are available are shown.

In the case of C29 and C30 moretane/hopane and C29/C30 hopane ratios, perhydrous samples in serial ply suites tend to have more mature values than adjacent samples up to high volatile A bituminous rank. At higher ranks, ratio values reverse towards relatively low "maturity", lead by the most perhydrous samples. For example, figure 10 shows the C30 moretane/hopane biomarker ratio for West Coast coals and some Taranaki coals, oils and condensates. Coals achieve minimum values, corresponding to maximum maturity, at ranks of high volatile B to A bituminous. Oils and

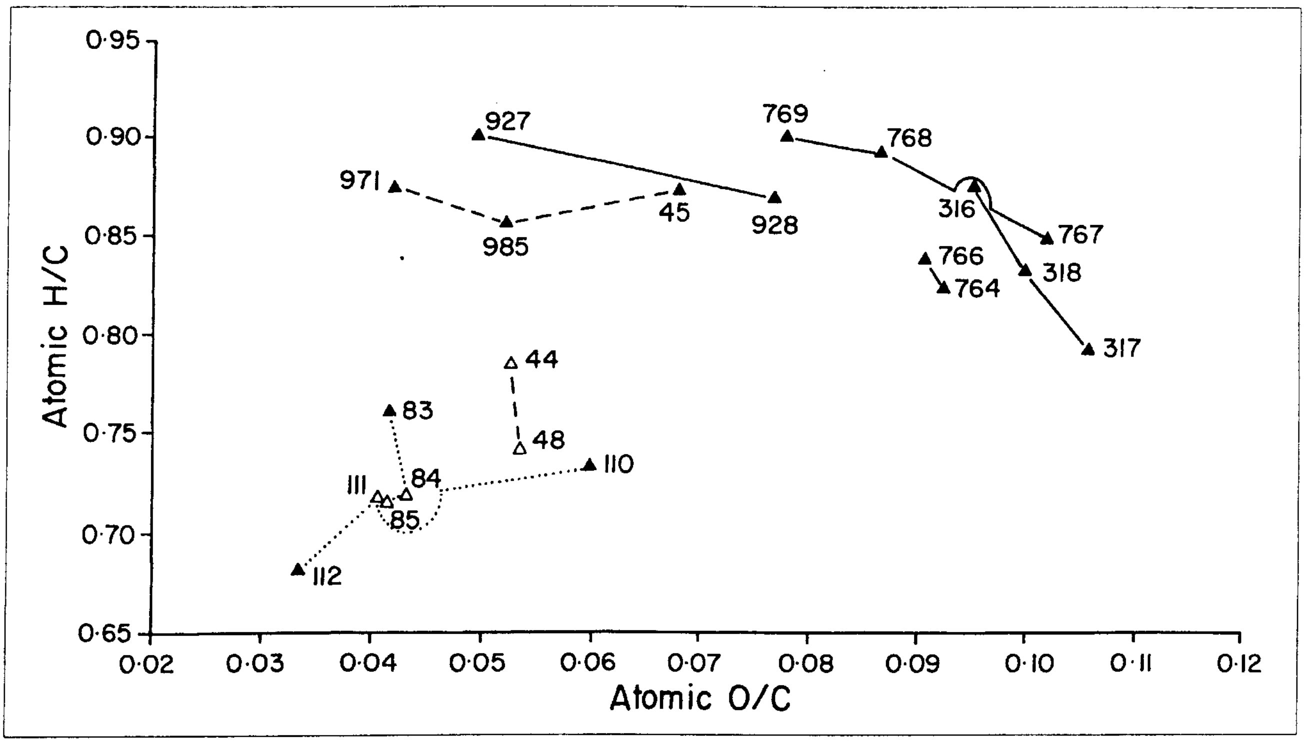

Fig. 9. Atomic H/C:O/C plot (van Krevelen diagram) for the same samples illustrated in figure 8. Tie lines link isorank serial sample suites. Solid lines represent lowest rank, dotted lines highest rank (for key see figure 1). In contrast to figure 8, samples plot in their correct relative positions on figure 9, in terms of rank relationships. Only samples for which ultimate analyses are available are shown.

condensates, which are by definition mature, have the same ratio values as these mature coals. However, for one of the medium volatile bituminous sample suites, ratio values reverse away from "maturity". This reversal at high rank occurs decisively in the case of C29 moretane/hopane (figure 11) and C29/C30 hopane (figure 12) ratios. Newman et al. (1992) postulated that these three biomarker ratios respond to petroleum hydrocarbons in the coal, and to the chemical fraction in coal which is the precursor to petroleum. This suggestion is supported by the biomarker response to type variation in immature coals, reported at the beginning of the section, whereby perhydrous samples typically have the most mature (lowest) ratio values. Developing this hypothesis, Newman et al. (1992) suggest that the highest rank coals may report declining biomarker maturity because petroleum has been expelled during increasing coalification. The relatively rapid decline in Rock-Eval S1+S2 for perhydrous coals above high volatile A bituminous rank (figure 6) also indicates loss of hydrocarbons at this stage of coalification. Figure 13 illustrates the relationship between S1+S2 and C29 moretane/hopane values for West Coast Eocene coals. As coalification advances from high volatile B to A bituminous, biomarker maturity increases and S1+S2 also increases. During further coalification from high volatile A to medium volatile bituminous, S1+S2 and biomarker maturity both decline.

Divergence of biomarker ratio values for coal versus oil would require differentiation of C29 and C30 hopane, and of the respective moretane isomers, between the petroleum and the residual coal. The relative elution times of these molecules in a gas chromatography (GC) column provide some support for this suggestion. C29 hopane elutes first, followed by C29 moretane, C30 hopane, and C30 moretane, listed in order of their appearance (P. Philp per comm.). If GC transport also relates to movement of the molecules through the coal matrix, these relative rates of migration could explain why petroleum has lower moretane/hopane ratios, in the case of C29 and C30, than the coal remaining behind. However, by this reasoning the C29/C30 hopane ratio value should be larger for the oil than the remaining coal, when in fact the opposite is the case.

Furthermore, it is interesting that the C29/C30 hopane ratio actually suggests more complete differentiation of oil and coal (figure 12), for both perhydrous and subhydrous samples, than the moretane/hopane ratios (figures 10 & 11). Tissot & Welte (1984) state that the predominance at low maturity of biologically preferred odd carbon numbers is gradually balanced by generation of new alkanes without an odd/even preference. Where such n-alkanes are not generated for some reason, the odd predominance may persist to relatively high temperatures. With respect to the high rank West Coast

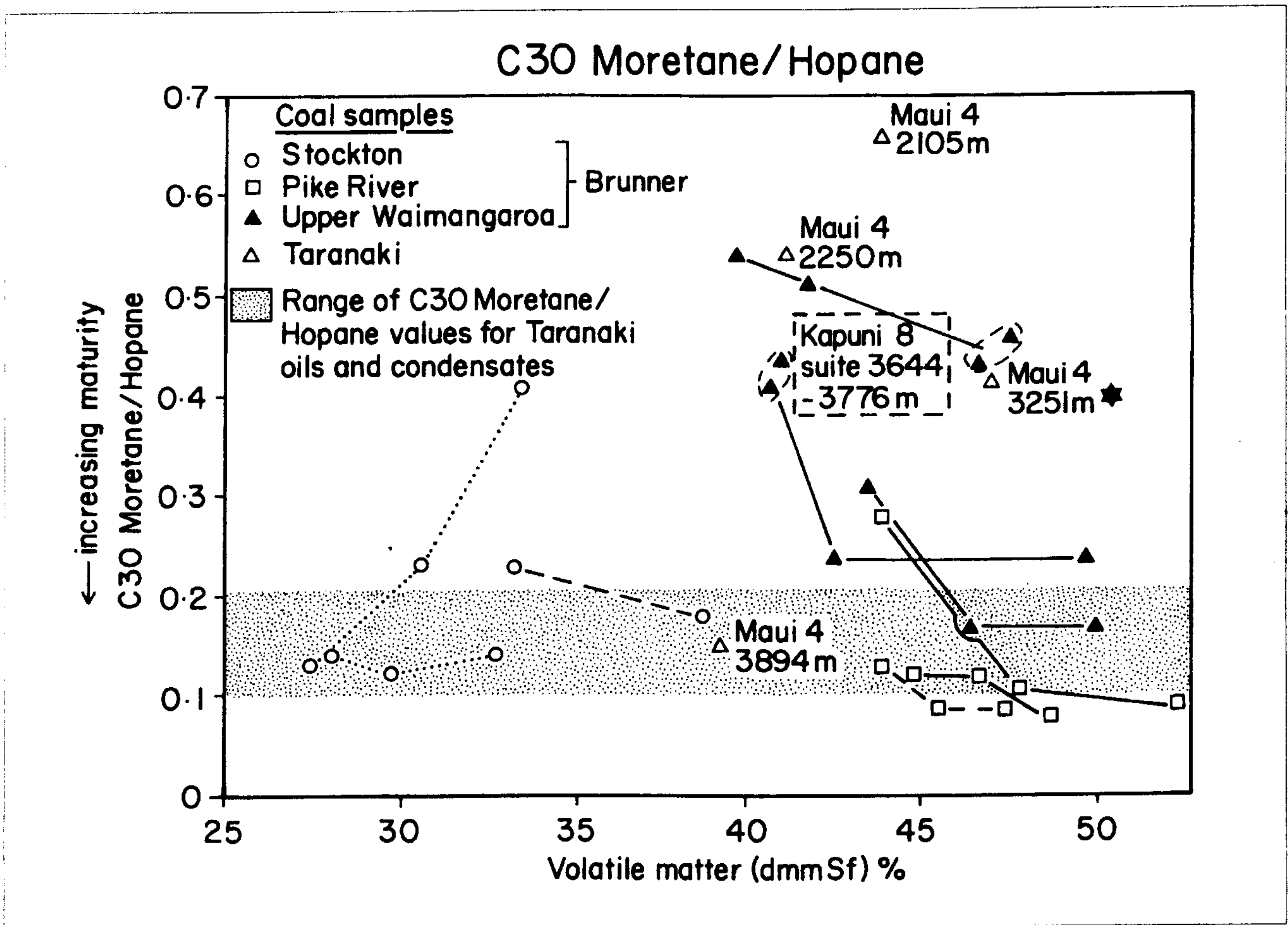

Fig. 10. C30 Moretane/Hopane ratio values plotted against volatile matter for the West Coast sample suite and some Taranaki Basin coals and oils (Johnston et al., 1988; Lowery 1988). For simplicity, only Eocene samples of the West Coast suite are shown. Tie lines link isorank serial samples, and a key to symbols appears in figure 1. The encircled sample pairs are 35/765 + 35/766, and 39/315 + 39/316, as discussed in the Introduction. Biomarker values for Taranaki oils and condensates are identified by the shaded area; no volatile matter value is implied. The high volatile character of the Maui 4 coal marked with a star is interpreted to represent a relatively high liptinite content rather than perhydrous vitrinite, based on the absence of reflectance suppression for the sample.

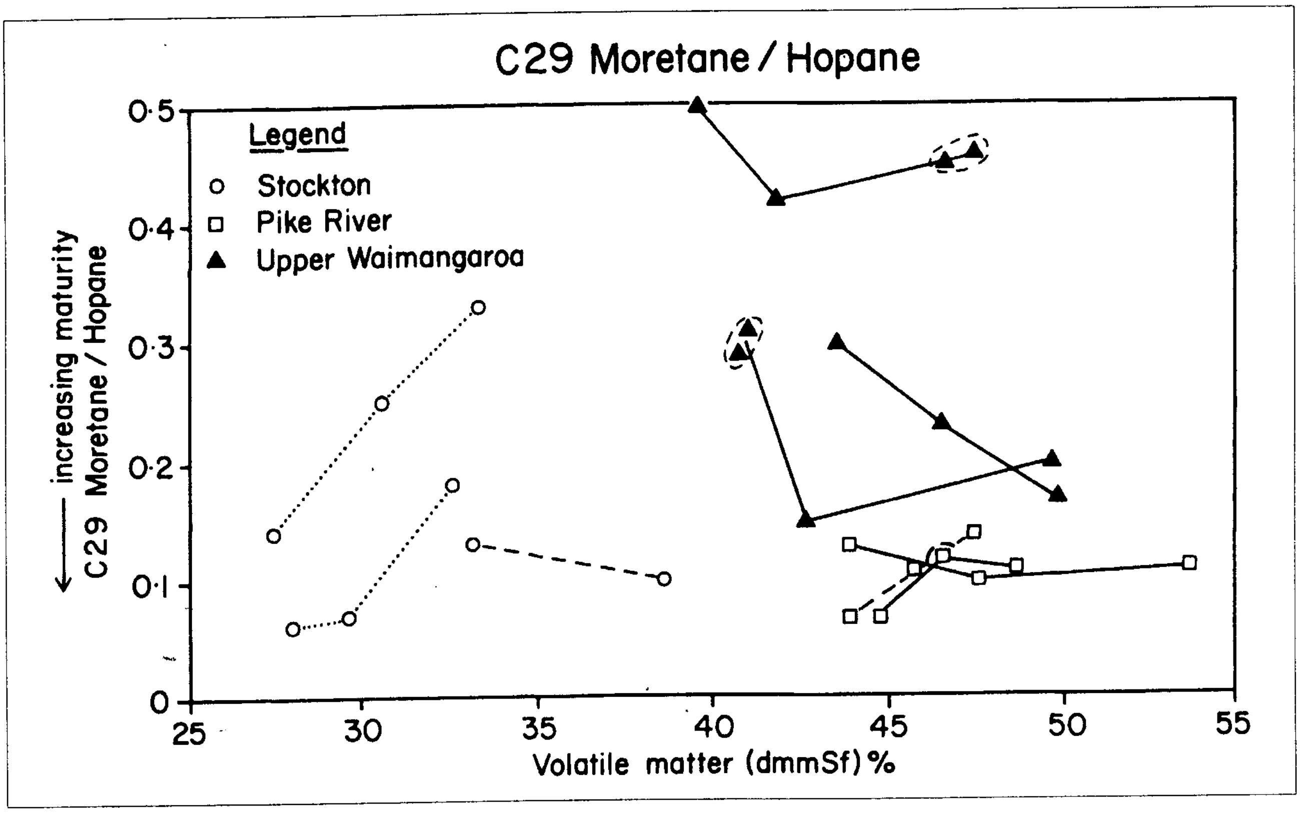

Fig. 11. C29 Moretane/Hopane ratio values plotted against volatile matter for Eocene members of the West Coast sample suite. Tie lines link isorank serial samples, and a key to line symbols appears in figure 1. The encircled sample pairs are 35/765 + 35/766, and 39/315 + 39/316, as discussed in the Introduction.

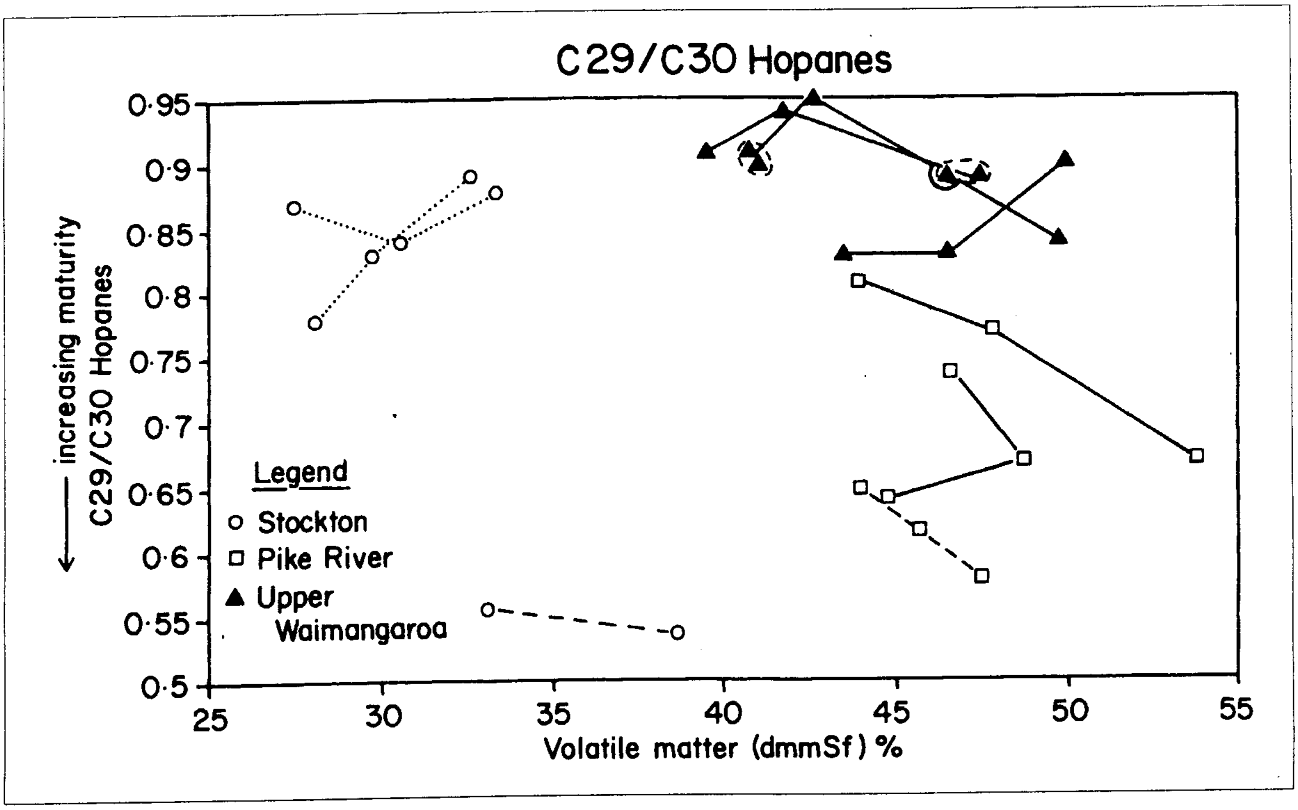

Fig. 12. C29/C30 Hopane ratio values plotted against volatile matter for Eocene members of the West Coast sample suite. Tie lines link isorank serial samples, and a key to line symbols appears in figure 1. The encircled sample pairs are 35/765 + 35/766, and 39/315 + 39/316, as discussed in the Introduction.

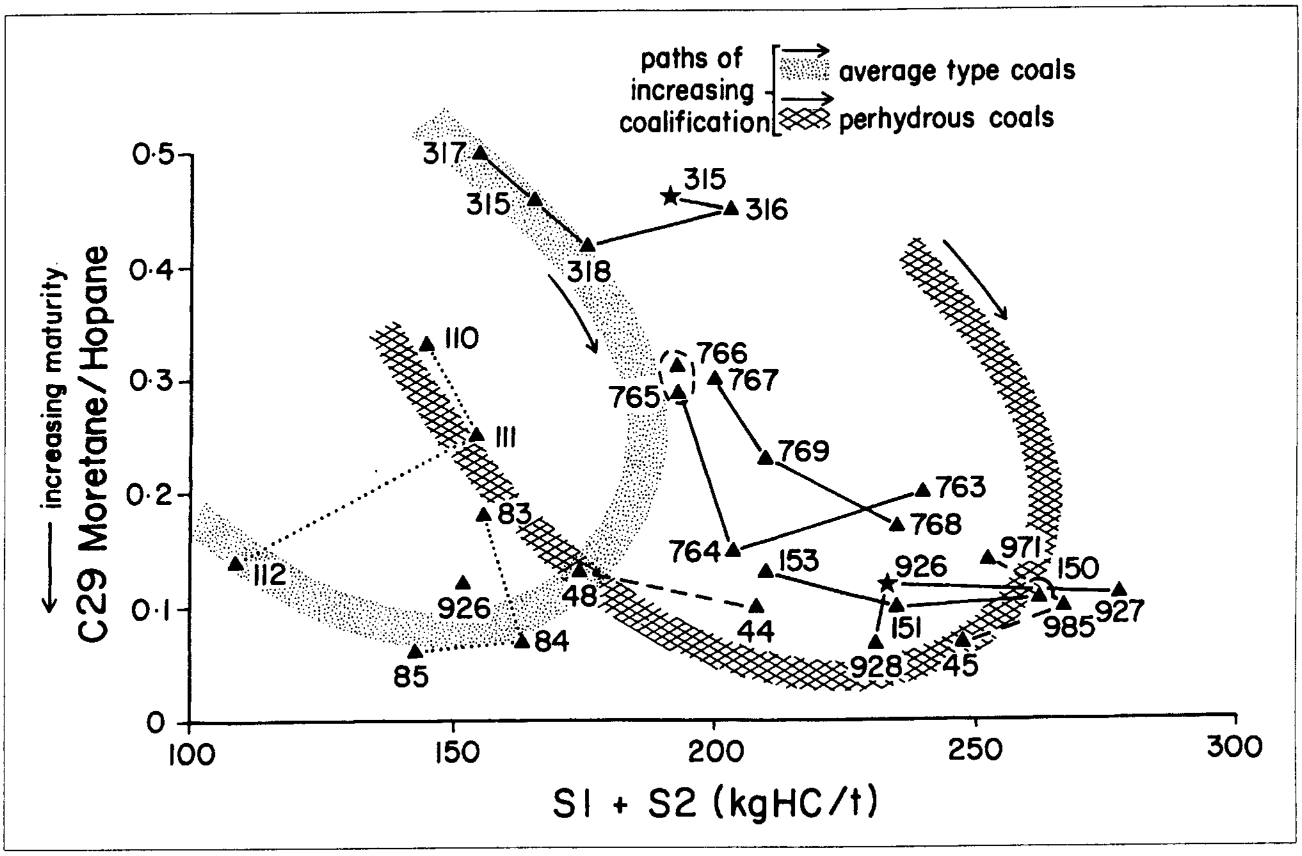

Fig. 13. Plot of C29 Moretane/Hopane ratio values plotted against Rock-Eval S1+S2 yield for Eocene members of the West Coast sample suite. Coalification paths for perhydrous and average type coals are identified by hatched and stippled zones respectively. Tie lines link isorank serial sample suites, and a key to line symbols appears in figure 1. With increasing rank, Rock-Eval yield and biomarker maturity increase up to the commencement of high volatile A bituminous rank. At higher ranks both these parameters progressively decline, most strikingly in the case of perhydrous samples. The significance of sample pair 35/765 and 35/766 is discussed in the Introduction.

coals, perhaps loss of accumulated n-alkanes during petroleum expulsion causes the observed reversal towards relatively high C29/C30 (odd/even) values with increasing rank.

Other Evidence Relevant to Source Potential and Maturation of Coal

The question of petroleum generation from coals has been approached in a number of ways by a variety of workers, and there are still no definitive answers as to how much petroleum a particular coal may generate, and when or if that product is expelled as an oil, potential condensate, or gas. In this context the observation of Teichmuller and Durand (1983), that the products of Rock-Eval pyrolysis of coals are predominantly liquid, may be pertinent. There is a widespread opinion that any oil which is generated will remain trapped in the coal structure until thermally cracked and released as gas. However, oil seeps and traces associated with New Zealand coal measures and coal seams appear to suggest generation from coal (Gage & Wellman 1944) and, as mentioned earlier, GCMS biomarkers have been used to define correlations between Taranaki oils and high volatile A bituminous coals intersected by petroleum wells (Cook, 1987; Johnston et al., 1990).

Parallels have been drawn between coal maturation with respect to petroleum and changes in coking potential. Fluidity (a measurement of coal viscosity at coking temperatures) peaks in New Zealand coals at about high volatile A bituminous rank. Vitrinite fluorescence, measured quantitatively in a nitrogen atmosphere by Quick (1992), correlates well with vitrinite reflectance, coal volatile matter and fluidity until the end of high volatile A bituminous rank. Fluorescence values then decline and become erratic at medium volatile bituminous rank. Furthermore, this decline in vitrinite fluorescence occurs first in the relatively perhydrous plies of serial sample suites. Normally well defined linear relationships between volatile matter and specific energy for isorank serial plies (volatile matter α specific energy) are also anomalous at medium volatile bituminous rank. Seam intersections at Stockton (Buller Coalfield) have a well defined upward increase in degree of perhydrousness, related to gradual drowning of the mire during peat accumulation (see "Sulphur"). Even after the postulated hydrocarbon expulsion at high volatile A to medium volatile bituminous rank, volatile matter remains highest in the upper part of the seam. However, specific energy for both the upper and middle parts of the seam is depressed in comparison with the lower interval, hence the volatile matter/specific energy relationship becomes strongly non linear. These aberrations, also noted by Suggate (pers comm.), may relate to differential expulsion of hydrocarbons, with greatest losses in the upper, relatively perhydrous parts of the seam. Why volatile matter remains relatively high in perhydrous coals after the postulated loss of hydrocarbons is not clear.

Carbon isotope research undertaken by J. Rogers of the New Zealand Geological Survey, and published in proceedings of the Eighth Commonwealth Mining and Metallurgical Congress (1965), appears to have been overlooked in some recent publications on the source of New Zealand oils. Rogers discussed C^{13}/C^{12} ratios, and pointed out that marine organic material has heavier isotopic composition than freshwater material. These trends are accentuated in oils and condensates derived from marine and non-marine sources. Rogers suggested, with evidence from experiments with coals heated under pressure, that the isotopically light character of Taranaki oils and condensates indicate a non-marine source, with subsequent fractionation. Coals are strongly implicated, because some Taranaki condensates have exceptionally light compositions, which Rogers tentatively attributed to interaction with high C^{12} methane during condensation.

Discussion

It is clear that many coal properties approach maximum values at high volatile A bituminous rank (fluidity, vitrinite fluorescence, maturity as indicated by C29/C30 hopane and C29 and C30 moretane/hopane) or, in the case of volatile matter, undergo particularly rapid change commencing at this rank. Teichmuller & Durand (1983) refer to this accelerated change in coal properties as the "second coalification jump", one manifestation of which is an increase in generation of methane. The role of methane during petroleum migration is discussed by Price et al. (1983), although with reference to sediments, not to coal. Previous workers had considered that methane could not significantly aid primary migration of petroleum through sediments because it was unable to dissolve either sufficient quantities of oil, or the higher molecular weight components (tars and asphaltenes). Price et al. determined that gaseous solution of oil by methane was much greater with water present than in the case of dry systems. That is, the presence of water lowered the temperature and pressure requirements for effective solution. These findings, and those of Rogers (1965) discussed above, have particular relevance to the derivation of oils and condensates from coals, and are consistent with suggestions that the major period of petroleum expulsion from New Zealand coals does not commence before high volatile bituminous A rank. If methane solution is important, burial pressure as well as temperature may have a positive influence on hydrocarbon expulsion.

As pointed out by Suggate et al. (1982, 1993), the reflectance corresponding to the start of high volatile A bituminous rank may range between c.0.70% and 0.90%, depending on temperature/pressure conditions during coalification. Reflectance variation will also result from coal type, which can cause reflectance suppression. Also, as suggested in this and previous papers (Newman et al., 1982, 1992), coal type may influence the relative temperature at which coals achieve maturity. It is therefore not possible to define a specific reflectance value above which expulsion is expected to have occurred, particularly as ranks near the high volatile A to medium volatile bituminous boundary may be required.

Conclusions

GCMS, Rock-Eval and traditional methods of coal analysis together provide consistent evidence that West Coast coals of Eocene age have generated significant quantities of hydrocarbons by medium volatile bituminous rank.

The tendency at high volatile bituminous B rank, for perhydrous samples to exhibit more mature GCMS biomarker values than do normal coals of the same rank, *may* indicate that perhydrous coals generate hydrocarbons relatively early. As concluded by Bal (this volume), low Tmax values for perhydrous samples in serial sample suites suggest easier thermal degradation of molecular structures, hence may imply relatively early hydrocarbon generation.

Rock-Eval S1+S2 trends for serial ply suites provide evidence that by medium volatile bituminous rank perhydrous coals have generated and expelled more hydrocarbons than coals with normal vitrinite chemistry (this does not necessarily indicate earlier generation). The reversal of trends exhibited by some GCMS biomarker ratios (C29 and C30 moretane/hopane and C29/C30 hopane) at medium volatile bituminous rank may represent an important period of hydrocarbon expulsion, particularly for perhydrous vitrinites.

However, Rock-Eval provides no clear evidence for accumulation of petroleum in the coal structure substantially prior to expulsion. S1 is approximately as abundant in low rank as in high rank coals analysed during the present study. Furthermore, peats and lignites reported by Suggate & Boudou (1983) have much higher S1 than the bituminous coals they also analysed. These trends suggest that the S1 of coals represents volatile macerals, possibly liptinites, and not petroleum at all. Nevertheless, progressive changes in vitrinite fluorescence, fluidity and GCMS biomarkers towards high volatile bituminous A rank do suggest an increase in maturity. Perhaps the precursors of petroleum are bound by the coal in such a way that they cannot report as S1. The fact that S2 exhibits no consistent decline until at least high volatile bituminous A rank certainly indicates that little free petroleum is created until then.

In the above context, it is relevant that S1+S2 values for the highest rank coals analysed appear to suggest that coals of medium volatile bituminous rank may still be capable of generating hydrocarbons with continued rank increase. It is difficult to generalise about the depth of burial required to attain medium volatile bituminous rank in the Taranaki Basin, however Sykes et al. (1992, Fig. 10) indicate that coals of rank(S) 15 could be expected about 1 km below their Kapuni Deep-1 and Kapuni-1 composite line. Vitrinite reflectance/depth relationships are generally non-linear from high volatile A bituminous rank, hence an increase from 1.0% to 1.2% reflectance could be readily achieved within 1km.

The GCMS biomarker ratios used as maturity indicators by Johnston et al. (1988, 1991) and Newman et al. (1992) either reach an equilibrium value by the time maturity is attained (C31/C30 hopane, %ββ sterane, ααS/S+R sterane) or achieve an equilibrium value for oils and condensates, while the residual coal exhibits a reversal in values (C29 and C30 moretane/hopane, C29/C30 hopane). In both circumstances GCMS provides no empirical evidence that petroleum generated above high volatile A bituminous rank will have a distinct biomarker signature. Consequently, continuation of petroleum generation into medium volatile bituminous ranks is not contradicted by available data, and it is even possible, therefore, that the principal phase of petroleum expulsion for some coals occurs at medium volatile bituminous rank, coincident with peak rates of methane genesis.

Future Work

The majority of the above conclusions have been reached primarily by investigation of Eocene coals. Preliminary evidence is that the behaviour of other coals is analogous, particularly as Bal (this volume) has identified exceptionally perhydrous Cretaceous seams within paralic Pakawau Group sediments (Wanganui Inlet, Northwest Nelson). Work on Cretaceous and Paleocene coals, to determine their relative maturation rates and generative potential, is continuing.

Discussions above cast doubt on the link between the S1 yield of coal and the presence of petroleum. Nevertheless, possible loss of the S1 fraction at outcrop and during drilling might be evaluated by analysis of appropriate gas samples, in which case the necessary information may already be available from demethanation studies. However, it should be noted that gas from Carboniferous and Permian deposits may not be pertinent to New Zealand's Cretaceous and Tertiary coals. Also, data for gas generated by coals of subbituminous and high volatile bituminous C to B rank will not be relevant if petroleum expulsion occurs primarily at higher ranks. Finally, the amount of hydrocarbon evolved in methane from coal seams which are core sampled after uplift may grossly underestimate the amounts which would evolve during coalification at depth. That is, much of the relevant outgassing can be expected to have occurred at the time of maximum burial and coalification, and further outgassing would occur during uplift. Most importantly, the solubility of hydrocarbons in methane would be vastly reduced at the low pressures and temperatures pertaining during typical gas sampling procedures.

It is still desirable to determine, if possible, (i) the relative proportions of liquid and gaseous hydrocarbons generated by coals, (ii) whether these proportions are influenced by vitrinite type, (iii) how the proportions vary with increasing rank, and (iv) the upper rank limit for generation of liquid hydrocarbons, either directly or — more probably — as condensate. A quantitative estimate of actual amounts of hydrocarbons that may have been generated per unit of coal would also be valuable. As part of progress towards this end, a collaborative study is planned involving hydrous pyrolysis and vitrinite fluorescence measurement of a suite of coals encompassing a range of coal types and ranks, including representatives of samples analysed during the present work. Further evaluation of Rock-Eval techniques, particularly with reference to GCMS and bulk chemical analyses, is underway.

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