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DEPARTMENT OF MINES AND ENERGY



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OTWAY BASIN

SOURCE ROCK STUDIES - DATA (Reports for the period October 1981 - July 1991)

Submitted by

various petroleum exploration companies plus SADME project officers

1991

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Analytical procedures.

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Preliminary Evaluation of the Source Rock Potential of the Eumeralla Formation in Chama-1A and Geltwood Beach-1, Otway Basin

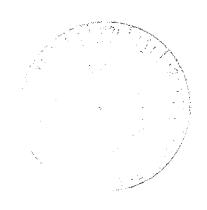
for

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1. Introduction

Organic petrology, TOC, Rock-Eval pyrolysis and preliminary biological marker data are summarised to provide an assessment of the source rock potential of the Eumeralla Formation in Chama-1A and Geltwood Beach-1.

2. Analytical Procedures

2.1 Samples

Coals and grey argillaceous sediments (shales, siltstones) were selected from cuttings of the Eumeralla Formation in Chama-1A, and from core and cuttings of the same unit in Geltwood Beach-1. Following petrological examination, TOC and Rock-Eval pyrolysis, 6 samples from Chama-1A (Table 1) and 6 from Geltwood Beach-1 (Table 2) were chosen for further geochemical analyses.

2.2 Preparation

The procedures used in this study are presented in Appendix A.

3. Source Rock Analysis

3.1 Maturity: Chama-1A

Vitrinite reflectance data (Table 1; Padley, 1991) indicate that the Eumeralla Formation from 5910 to 8530 ft depth (1801 - 2599 m) is mature (Rv = 0.53 - 0.89%). From petrological examination (Padley, 1990a) the organic-rich lithofacies were found to be predominantly coals and shaley coals. Samples A-923.070 and A-923.010 comprise the least mature coals (5910 - 7540 ft, Rv = 0.53 - 0.58%) and are resinite-rich. The top of the oil window for such coals is at Rv = 0.45% (Snowdon & Powell, 1982; Monnier et al., 1983). Hence, these coals are well within the zone of hydrocarbon generation. The deeper Eumeralla coals are typical Type III kerogen, comprising woody herbaceous components (vitrinite > inertinite = liptinite) ranging in maturity from Rv = 0.71 to 0.89%. The onset of oil generation from resinite-poor terrestrial organic matter occurs at Rv = 0.7% (Monnier et al., 1983; Powell, 1985) and significant quantities of wet gas are produced from Rv = 0.65 to 0.85%. Hence, the Eumeralla Formation at Chama-1A has the potential to generate both gaseous and liquid hydrocarbons.

TABLE 1: Samples Selected for Geochemical Analysis, Eumeralla Formation, Chama-1A

Sample N°s	Depth (ft)	Depth (m)	Sample Type	Lithology	VRmax (%)	Analysis
A-923.007/8	5910 - 5950	1801 - 1814	Cuttings	С	0.53	GC-MS
A-923.009	7300 - 7310	2225 - 2228	Cuttings	С	0.58	
A-923.010/11	7490 -7540	2286 - 2287	Cuttings	Sh-C & Silt	0.71	GC-MS
A-923.013	7850 - 7860	2392 - 2395	Cuttings	С	0.80	
A-923.014	7890 - 7940	2405 - 2420	Cuttings	C	0.83	GC-MS
A-923.016	8010 - 8020	2441 - 2444	Cuttings	С	0.86	
A-923.017/18	8160 - 8200	2487 - 2499	Cuttings	Sh-C & Silt	0.87	GC-MS
A-923.020	8240 - 8250	2512 - 2515	Cuttings	Sh-C	0.86	
A-923.021/22	8330 - 8370	2539 - 2551	Cuttings	Sh-C	-	GC-MS
A-923.025/26	8490 - 8530	2588 -2596	Cuttings	Sh-C & Silt	0.89	GC-MS

TABLE 2: Samples Selected for Geochemical Analysis, Eumeralla Formation, Geltwood Beach-1

Sample N°s	Depth (ft)	Depth (m)	Sample Type	Lithology	Analysis
A-923.060	8939	2725	Core 22	Silt	GC-MS
A-923.165/7	9610 - 9630	2929 - 2935	Cuttings	Sh & Silt	GC-MS
A-923.081	9700 - 9710	2957 - 2960	Cuttings	Sh	GC-MS
A-923.082	9760 - 9770	2975 - 2978	Cuttings	Silt & Sh	GC-MS
A-915.63	10400 - 10420	3170 - 3176	Cuttings	Sh-C	GC-MS
A-923.168	11220 - 11230	3420 - 3423	Cuttings	C	GC-MS

C - Coal

Sh-C - Shaley coal

Silt - Siltstone

Indeed, in the deeper section of the Eumeralla Formation (below 7520 ft), the secondary liptinite maceral exudatinite is common. Bitumen and free oil (observed at 8010 ft) are also evidence of hydrocarbon generation.

The Rock-Eval Tmax values for Chama-1A (Table 4) reveals a gradual, but not continuous, down hole increase in organic maturity from 5910 to 8030 ft. The exception to this is sample A-923.016 (8010 ft) where Tmax is slightly depressed and the S1 peak is high (14.64), perhaps signifying the presence of free oil.

The lower Eumeralla section (8160-8530 ft) shows an unexplained decrease in Tmax which is opposite to the measured increase in vitrinite reflectance.

3.2 Source Richness: Chama-1A

The TOC content of the Eumeralla coals and shaley coals is high, ranging from 2.98 - 54% (mean = 24%). Potential hydrocarbon yield values (S1+S2) are in excess of 12 kg hydrocarbons/tonne, suggesting very good source richness. The shales and siltstones examined are leaner (TOC = 0.17 - 1.13%) and hence have poor source richness (S1+S2 = 1.3 - 1.9 kg hydrocarbons/tonne).

3.3 Source Quality & Kerogen Type: Chama-1A

The coals examined have moderately high hydrogen indices (HI = 155 - 237 mgS2/g TOC; Fig. 1; Table 4) indicating the presence of type II/III kerogen with the potential to generate both oil and gas.

Table 3: Key to Rock-Eval Pyrolysis

Parameter	Definition	Specificity
T _{max}	Position of S2 peak in temperature program (°C)	Maturity/Kerogen type
S_1	Kg hydrocarbons (extractable) / tonne rock	Kerogen type/Maturity/Migrated oil
S ₂	Kg hydrocarbons (kerogen pyrolysate) / tonne rock	Kerogen type/Maturity
S ₃	Kg CO ₂ (organic) / tonne	Kerogen type/Maturity*
S ₁ +S ₂	Potential Yield	Organic richness/Kerogen type
PI	Production Index (S ₁ /S ₁ +S ₂)	Maturity/Migrated oil
PC	Pyrolysis Carbon (wt. percent)	Organic richness/Kerogen type/Maturity
TOC	Total Organic Carbon (wt. percent)	Organic richness
HI	Hydrogen Index (mg HC (S2)/g TOC	Kerogen type/ Maturity
OI	Oxygen Index (mg CO ₂ (S ₃)/g TOC	Kerogen type/ Maturity*

^{*}Also subject to interference by CO_2 from decomposition of carbonate minerals.

Table 4: Rock-Eval Pyrolysis Data for Eumeralla Formation, Chama-1A

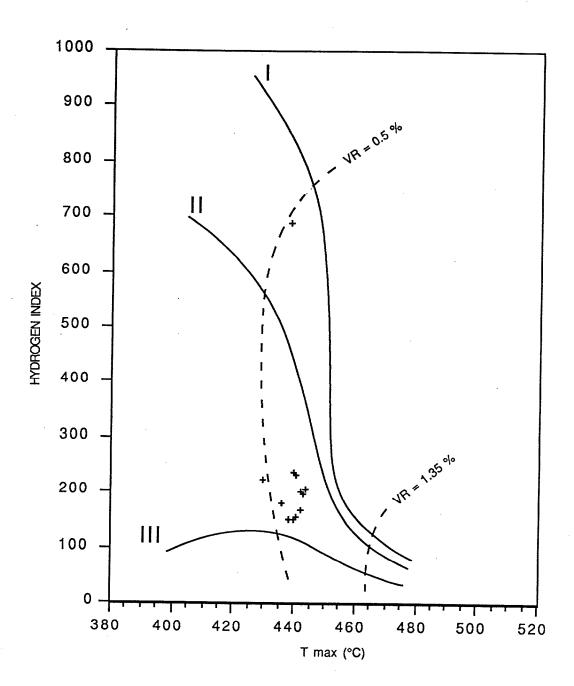
Sample	Depth (ft)	Tmax	S 1	S 2	S 3	S1+S2	PI	S2/S3	PC	TOC 1	ні	OI
A-923.007	5910 - 5950	430	3.90	85.14	7.14	89.04	0.04	11.92	7.42	38.20	222	18
A-923.183	7260 - 7280	438	0.15	1.17	0.50	1.32	0.11	2.34	0.11	0.17	688	294
A-923.010	7490 - 7540	436	0.43	5.36	0.94	5.79	0.07	5.70	0.48	2.98	179	31
A-923.012	7820 - 7830	438	0.23	1.68	0.67	1.91	0.12	2.50	0.15	1.13	149	59
A-923.014	7890 - 7940	441	5.60	64.67	2.24	70.27	0.08	28.87	5.85	28.20	229	7
A-923.015	7990 - 8100	442	5.08	50.50	1.69	55.58	0.09	29.88	4.63	25.30	199	6
A-923.016	8010 - 8020	440	14.64	128.12	2.50	142.76	0.10	51.24	11.89	54.00	237	4
A-923.182	8030 - 8040	444	8.34	63.39	1.55	71.73	0.12	40.89	5.97	31.00	204	5
A-923.017	8160 - 8200	443	3.52	38.61	1.28	42.13	0.08	30.16	3.5 1	19.60	196	6
A-923.019	8210 - 8230	442	1.67	14.83	1.02	16.50	0.10	14.53	1.37	8.83	168	12
A-923.021	8330 - 8370											
A-923.025	8490 - 8530	441	0.65	8.12	1.26	8.77	0.07	6.44	0.73	5.21	155	24

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Figure 1: Cross-Plot of HI versus Tmax, Chama-1A



3.4 Maturity: Geltwood Beach-1

The lower Eumeralla Formation from 8579 to 12241 ft depth (2615 - 3731 m) in Geltwood Beach-1 is characterised by vitrinite reflectances of 0.6 - 0.9% (Serafini, 1989) and hence lies within the upper part of the main oil generation zone.

Rock-Eval Tmax values (Table 5) and production indices of less than 0.2 are consistent with this maturity level. The coaly shale interval at 11739 - 11749 ft is exceptional in having a high production index of 0.50 to 0.81.

3.5 Source Richness: Geltwood Beach-1

Shales and siltstones are the dominant lithologies throughout the Eumeralla Formation. Only in the lower part of the sequence are coaly shales and coals developed. The shales and siltstones have very low to moderate organic richness (TOC = 0.2 - 2.6%) and their genetic potential is correspondingly poor to fair (S1+S2 = 0.2 - 4.1 kg hydrocarbons/tonne). In contrast, the coaly shales and coals have much higher TOC values (5.92 - 48.7%) and a good genetic potential (S1+S2 = 15 - 102 kg hydrocarbons/tonne) and are therefore good source rocks.

3.6 Source Quality & Kerogen Type: Geltwood Beach-1

The plot of hydrogen index versus Tmax indicates that the majority of the argillaceous sediments contain Type III/IV kerogen whereas the coals consist of Type II/III kerogen (Fig. 2). The hydrogen indices of the shales and siltstones are low (HI = 15 - 47 mgS2/g TOC; Table 5). Petrological examination (Padley, 1990b) revealed that these shales and siltstones contained low to moderate amounts of dispersed organic matter, comprising liptinite and vitrinite as the most abundant macerals with minor inertinite. However, the vitrinite phytoclasts appear reworked and hydrogen-poor liptinites (notably sporinite: cf. Powell et al., 1991) are predominant. The hydrogen-rich liptinites (telalginite and lamalginite) were only present in trace amounts. This may account for the generally low hydrogen indices, although uniformly low organic carbon contents (TOC < 1%) mean that suppression of hydrogen index by the mineral matrix effect is also likely (cf. Powell et al., 1989; Michaelsen & McKirdy, 1989). Hence, the combination of Type III/IV kerogen, low organic richness and moderate maturity would imply that the sequence is at best only gas and condensate-prone.

Table 5: Rock-Eval Pyrolysis Data for Eumeralla Formation, Geltwood Beach-1

Sample N°	Depth (ft)	Depth (m)	Tmax	S1	S 2	S3	S1+S2	PI	S2/S3	PC	тос	ні	OI
A-923.169	4090 - 4100	1247 - 1250	361	0.00	0.00	0.16	0.00	0.00	0.00	0.00	0.22	0	72
A-923.170	4521	1378	250	0.00	0.00	0.18	0.00	0.00	0.00	0.00	0.26	0	69
A-923.171	5333	1625	311	0.01	0.05	0.21	0.06	0.17	0.23	0.00	0.33	15	63
A-923.172	5680	1731	229	0.02	0.00	0.33	0.02	1.00	0.00	0.00	0.31	0	106
A-923.173	6084	1854	437	0.01	0.71	0.14	0.72	0.01	5.07	0.06	0.49	144	28
A-923.174	6519	1986	443	0.00	0.00	0.18	0.00	0.00	0.00	0.00	0.26	0	69
A-923.175	7035	2144	442	0.01	0.01	0.14	0.02	0.50	0.07	0.00	0.07	14	200
A-923.178	8476	2583	273	0.02	0.06	0.11	0.08	0.25	0.54	0.00	0.33	18	33
*	8579 - 8589	2615 - 2618	438	0.06	0.81	1.99	0.87	0.07	0.41	0.07	2,65	30	75
A-923.060	8939	2725	441	0.01	0.28	0.10	0.29	0.04	2.80	0.02	0.73	38	13
*	9281 - 9288	2829 - 2831	439	0.09	0.14	0.62	0.23	0.41	0.23	0.01	0.79	17	78
A-923.080	9520	2902	394	0.05	0.23	0.22	0.28	0.18	1.04	0.02	0.52	44	42
*	9521 - 9531	2902 - 2905	439	0.08	0.80	1.19	0.88	0.09	0.67	0.07	1.67	47	71
A-923.165*	9610 - 9630	2929 - 2932	439	0.12	4.02	0.89	4.14	0.03	4,54	0.34	1.67	240	53
A-923.081	9700 - 9710	2957 - 2960	461	0.03	0.13	0.18	0.16	0.19	0.72	0.01	0.55	23	32
*	9700 - 9710	2957 - 2960	441	0.07	0.37	1.13	0.44	0.16	0.33	0.03	1.38	26	82
A-923.082	9760 - 9770	2975 - 2978	444	0.01	0.21	0.17	0.22	0.05	1.23	0.01	0.59	35	28
*	10049 - 10066	3063 - 3068	440	4.69	61.42	21.78	66.11	0.07	2.82	5.50	33.50	183	65
A-923.168	10400 - 10420	3169 - 3176	454	0.49	14.09	2.98	14.58	0.03	4.72	1.21	20.10	70	14
*	10400 - 10410	3170 - 3173	438	8.22	66.35	20.50	74.57	0.11	3.24	6.21	33.60	197	61
*	10509 - 10525	3203 - 3208	441	9.14	93.08	24.58	102.22	0.09	3.79	8.51	40.30	230	61
*	10787 - 10801	3288 - 3292	_	0.04	0.11	0.50	0.15	0.29	0.22	0.01	0.62	17	81
*	11211 - 11220	3417 - 3420	450	10.19	30.48	30.68	90.67	0.11	2.62	7.55	48.70	165	63

 A_{cog}

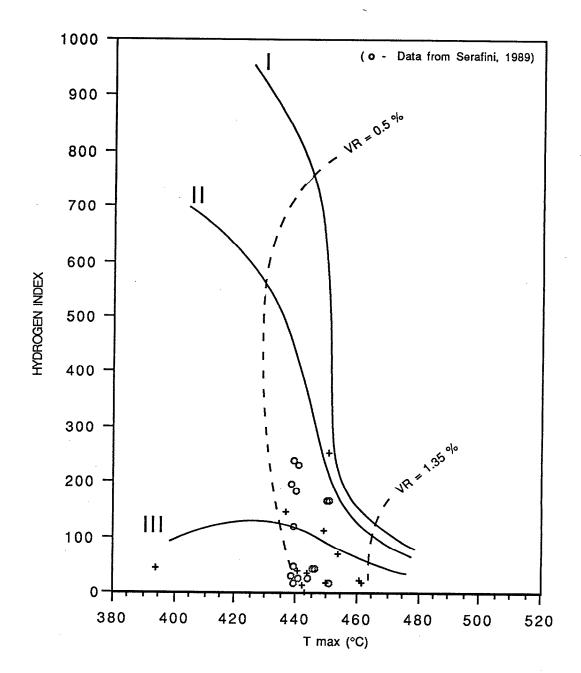
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Sample N°	Depth (ft)	Depth (m)	Tmax	S 1	S2	S3	S1+S2	ΡI	S2/S3	PC	TOC	ні	OI
A-915.066	11220 - 11230	3420 - 3423	451	0.66	15.01	2.67	15.67	0.04	5.26	1.30	5.92	254	45
*	11234 - 11243	3424 - 3427	-	0.06	0.28	0.61	0.34	0.10	0.46	0.02	0.89	31	69
*	11339 - 11348	3456 - 3459	444	0.19	0.34	0.79	0.53	0.37	0.43	0.04	1.22	27	65
A-923.180	11462	3494	449	0.28	1.38	0.18	1.66	0.17	7.66	0.13	1.26	109	14
*	11463 - 11473	3494 - 3497	451	5.67	42.45	14.08	48.12	0.12	3,01	4.01	25.60	165	55
*	11565 - 11575	3525 - 3528	445	0.18	0.63	0.93	0.81	0.22	0.68	0.06	1.48	42	63
A-923.64	11740	3578	462	0.07	0.08	0.08	0.15	0.50	1.00	0.01	0.47	17	17
*	11739 - 11749	3578 - 3581	451	0.05	0.12	0.61	0.17	0.81	0.20	0.01	0.76	15	80
*	12231 - 12241	3728 - 3731	439	0.17	2.63	1.34	2.00	0.06	1.96	0.23	2.20	119	61

^{*} Taken from Serafini, 1989

Figure 2: Cross-Plot of HI versus Tmax, Geltwood Beach-1



One possible shale source rock occurs at a depth of 9610 - 9630 ft (2929 - 2932 m; TOC = 1.67%; S1+S2 = 4.41 kg hydrocarbons/tonne; HI = 240 mgS2/g TOC). This shale was discovered to contain significant quantities of telalginite. However, the source interval is very thin (<< 10 ft). The shale cuttings were mixed with organically lean siltstone, and comparable shale facies were not identified immediately above or below this depth.

The coaly shales and coals are generally good source rocks with moderately high hydrogen indices (70 - 254 mgS2/g TOC) and therefore have the potential to generate both oil and gas.

4. Geochemistry

4.1 Maturity Parameters: Chama-1A & Geltwood Beach-1

Maturity parameters based on the saturated biomarker ratios of sterane and hopane compounds are presented in Tables 6, 7 & 8.

The values of the sterane (parameters 5 & 6) and hopane (parameters 10, 11 & 12) maturity ratios obtained for the Eumeralla Formation in Chama-1A and Geltwood Beach-1 demonstrate that it is mature at both localities, although optimum hydrocarbon generation has not yet been attained.

4.2 Source Affinity: Chama-1A & Geltwood Beach-1

The source affinity of the Chama-1A extracts is clearly evident from the C27-C28-C29 sterane distributions. The corresponding parameters 1 and 2 (Table 7) show that the steranes are dominated by the C29 homologues of terrestrial higher plant origin (Huang & Meinschein, 1979). This is a characteristic feature of most Australian non-marine sediments and crude oils (Philp & Gilbert, 1986). The hopane/sterane ratios (parameter 7) are high. The hopanes are between 2 and 6 times more abundant than the steranes. High hopane/sterane ratios are a common feature of sediments which contain high abundances of terrestrial organic matter (Moldowan et al., 1985; Vincent et al., 1985; Philp & Gilbert, 1986). Hopanes are derived from bacteria, and hence their presence in significant amounts indicates that bacterial reworking occurred during deposition of the organic matter.

Table 6: Key to Biomarker Parameters

	BIOMARKER PARAMETER	SPECIFICITY	M/Z
1	C ₂₇ : C ₂₈ : C ₂₉ 5α(H)14α(H)17α(H) 20R steranes	Source	217
2	C29 $5\alpha(H)14\alpha(H)17\alpha(H)$ 20R sterane / C27 $5\alpha(H)14\alpha(H)17\alpha(H)$ 20R sterane	Source	217
3	C ₂₉ 13β(H)17α(H) 20R diasterane / C ₂₇ 13β(H)17α(H) 20R diasterane	Source	259
4	C27 $13\beta(H)17\alpha(H)$ 20S diasterane / C27 $13\beta(H)17\alpha(H)$ 20R diasterane	Maturity	259
5	C29 $5\alpha(H)14\alpha(H)17\alpha(H)$ 20S sterane / C29 $5\alpha(H)14\alpha(H)17\alpha(H)$ 20R sterane	Maturity & Biodegradation	217
6	C29 $5\alpha(H)14\beta(H)17\beta(H)$ 20R sterane / C29 $5\alpha(H)14\alpha(H)17\alpha(H)$ 20R sterane	Maturity & Migration	217
7	C30 $17\alpha(H)21\beta(H)$ hopane / C29 $5\alpha(H)$ steranes	Source	191:217
8	C ₃₁ tricyclic terpane / C ₃₀ 17α(H)21β(H) hopane	Migration & Source	191
9	C27 17α(H)-22,29,30-trisnorhopane / C27 18α(H)-22,29,30-trisnorhopane (Tm/Ts)	Maturity & Source	191
10	Ts / C_{30} 17 α (H)21 β (H) hopane	Maturity	191
11	C ₃₂ 17α(H)21β(H) 22S homohopane / C ₃₂ 17α(H)21β(H) 22R homohopane	Maturity	191
12	C ₃₀ 17 β (H)21 α (H) moretane / C ₃₀ 17 α (H)21 β (H) hopane	Maturity	191

Table 7: Biomarker Parameters for Eumeralla Formation, Chama-1A

Sample N°	Depth	Steranes								ies			
	(ft)	Source			Maturity				Source		Maturity		y
		1	2	3	4	5	6	7	8	9	10	11	12
A-923.007/8	5910 - 5950	5:13:82	17.82	4.9	1.50	0.16	0.20	5.89	0.05	30.6	0.02	0.61	0.55
A-923.010/11	7500 - 7540	13:24:63	4.89	1.00	0.97	0.74	0.40	3.62	0.06	11.25	0.06	1.41	0.44
A-923.014	7890 - 7940	4:12:84	23.75	3.00	6.00	0.52	0.44	2.03	0.08	8.77	0.07	1.48	0.31
A-923.017/18	8160 - 8200	0:18:82	-	2.75	4.63	0.64	0.50	2.24	0.08	7.5	0.06	1.60	0.16
A-923.021	8330 - 8370	0:21:79	-	-	-	0.87	0.73	2.58	0.09	7.29	0.07	1.31	0.17
A-923.025/26	8490 - 8520	0:28:72	-	4.16	1.08	0.85	0.90	2.99	0.09	6.6	0.08	1.44	0.16
A-923.027/28	8860 - 8910	20:16:64*	3.3*	3.18*	1.47*	1.11	0.74	2,49	0.09	4.29	0.12	1.29	0.20

Table 8: Biomarker Parameters for Eumeralla Formation, Geltwood Beach-1

Sample N°	Depth			Stera	nes		H/S	Triterpanes					
	(ft)	Source			Maturity				Source		Maturity		у
,		1	2	3	4	5	6	7	8	9	10	11	12
A-923.060	8939	6:11:83	12.19	1.61	1.05	0.50	0.17	4.29	0.04	15.0	0.05	1.34	0.66
A-923.165	9610 - 9630	12:14:74	5.94	1.21	0.73	0.36	0.19	0.47	0.03	16.25	0.04	1.41	0.43
A-923.081	9700 - 9710	14:18:68	4.79	1.63	1.70	0.74	0.40	7.30	0.03	10.0	0.07	1.44	0.37
A-923.082	9760 - 9770	13:21:66	5.00	2.15	0.75	0.88	0.49	6.97	0.05	16.0	0.05	1.58	0.39
A-915.63	10400 - 10420	3:14:83	27.29	6.95	0.63	0.72	0.41	4.62	0.09	10.86	0.05	1.29	0.34
A-923.168	11220 - 11230	6:16:78	12.86	2.83	0.67	0.76	0.59	1.96	0.29	2.70	0.23	1.38	0.24

The coaly samples (A-923-060, -168 and A-915.63) in Geltwood Beach-1 exhibit very similar sterane distributions to those described for Chama-1A, whereas samples A-923-165, -081 and -082 all show a significant increase in the C27 steranes. In addition to the terrigenous origin of the C29 steranes, fresh water vascular plants and some algae (Volkman, 1988) may also contribute C29 sterols to sediments. Similarly some higher plants also produce C27 steranes (Huang & Meinschein, 1976). Therefore, sterane distributions have distinct limitations for distinguishing between lacustrine and coalswamp palaeoenvironments.

The hopane/sterane ratios are also high, with the hopanes being between 2 and 7 times more abundant. The only exception to this is the algal-rich sample (A-923.165) which has the noticably low ratio of 0.47. This may be due to a lack of bacterial activity and would account for the well preserved nature of the liptinite in this sample.

5. Summary & Conclusions

- 1) The non-marine sediments of the Eumeralla Formation are thermally mature, ranging from Rv = 0.53 0.89 % over a depth interval of 5910 8530 ft in Chama-1A and Rv = 0.6 0.9 % from 8579 12241 ft depth in Geltwood Beach-1.
- 2) The maturation threshold for the onset of oil generation from Type III kerogen occurs at Rv = 0.7%. Hence, the terrestrial organic matter in the basal Eumeralla sequence of both wells is sufficiently mature to have generated and expelled hydrocarbons.
- 3) Organic rich sediments (TOC = 1.35-2%; S1+S2 = 12 kg hydrocarbons /tonne; HI = 155 237 mgS2/g TOC) were identified throughout the Eumeralla Formation in Chama-1A and generally appear to be good source rocks.
- 4) The shales and siltstones above 8476 ft in the upper Eumeralla Formation in Geltwood Beach-1 are generally organically lean (TOC < 0.33%). The shales in the lower Eumeralla Formation are somewhat richer but still poor potential source rocks (TOC = 0.5 2.6%; S1+S2 = 0.15 4.1 kg hydrocarbons/tonne; HI = 15 47 mgS2/g TOC) whereas the coaly shales and coals are good source rocks (TOC = 5.9 48.7%; S1+S2 = 14.5 102 kg hydrocarbons/tonne; HI = 70 254 mgS2/g TOC). An algal-rich horizon at 9610 9630 ft was identified by petrological examination and confirmed by Rock-Eval pyrolysis to be a possible source rock (TOC = 1.67%; S1+S2 = 4.14 kg hydrocarbons/tonne; HI = 240 mgS2/g TOC). However, the internval appears to be of

extremely limited thicknness which somewhat diminishes its significance as a source rock.

5) From the assessment of the source rock potential of the Eumeralla Formation in the two wells, Chama-1A and Geltwood Beach-1, it is apparent that the best source rocks are coaly shales and coals which are developed towards the base of the formation.

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APPENDIX A Analytical Techniques

Analytical Techniques

1.1 Sample Preparation

Optical examination of the samples was carried out prior to the chemical analyses. The most organic-rich sediments, preferentially containing vitrinite and liptinite, were selected (see Padley, 1990a, b). All samples were cleaned to remove any contaminants e.g. drilling mud and ink. Drilling mud was removed by seiving the cuttings through a 72-mesh sieve with purified water, followed by washing with methanol in an ultrasonic bath for 1 minute and finally air drying. The samples were then ground in a Tema mill to < 20 mesh.

1.2 Extraction of samples

A weighed amount of each sample was placed in a thimble, plugged with cotton wool and extracted in Soxhlet apparatus for 72 hours with an azeotropic solvent mixture of dichloromethane and methanol (93:7). Activated copper turnings were added to remove elemental sulphur.

The total extractable organic matter (EOM) was concentrated using a Büchi vacuum rotary evaporator. The extracts were transfered to pre-weighed 100ml flasks and the remaining solvent was removed under vacuum. The EOM was weighed and then stored in vials.

1.3 TOC Analysis

To determine the total organic carbon content of the whole rock, 200 mg of the powered sample was first digested by 50% HCl to remove any carbonates and air dried. This was followed by combustion in oxygen in the induction furnace of a Leco IR-12 Carbon Determinator and measurement of the resultant CO₂ by infra-red detection.

1.4 Rock-Eval Pyrolysis

A 100 mg portion of the decarbonated sample was analysed by Rock-Eval pyrolysis using a Girdel IFP-Fina Mark 2 instrument (operating mode, Cycle 1).

1.5 Liquid Chromatography

Asphaltenes were not precipitated from the EOM prior to liquid chromatography. The EOM was separated into hydrocarbons (saturates and aromatics) and polar compounds (resins) by liquid chromatography on activated silica (sample: adsorbent ratio - 1:100) Saturates were eluted with 80 ml of petroleum ether, aromatics with petroleum ether/dichloromethane (50:50) and resins with methanol/dichloromethane (65:35).

1.6 Gas Chromatography-Mass Spectrometry (GC-MS)

The saturated hydrocarbon (alkane) fractions were examined by gas chromatographymass spectrometry using the following instrumental parameters:

Gas Chromatography	
Instrument	Varian 3400
Column	30 m x 0.25 mm fused silica, DB-1 interfaced directly with the source of the mass spectrometer
Carrier Gas	Hydrogen at 15 psi head pressure
Injector temperature	Split/splitless injector operated in the split mode (ratio 20:1) at 300°C
Temperature programming	50°C for 1 minute, ramped at 8°C per minute to 120°C, then from 120°C to 300°C at 4°C per minute and held isothermal at 300°C for 20 minutes
Mass Spectrometry	
Mass Spectrometer	Varian 3400 was directly ineterfaced with a TSQ-70 mass spectrometer
Conditions	Electron energy 70eV; Emission current 200μA; Source temperature 250°C; Scan mode; 47:500 amu.

The following diagnostic mass fragmentograms were examined:

m/z	Compound Type
177	Demethylated triterpanes
191	Triterpanes (incl. hopanes, moretanes, oleananes)
205	Methyl triterpanes
217	Steranes
218	Steranes
231	4-methyl steranes
259	Diasteranes, diterpanes

Integration of the m/z 191, 217, 218, 231 and 259 mass fragmentograms allowed calculation of the biomarker ratios in Tables 7 & 8.