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# Palaeofacies and biomarker characteristics of Paleogene to Neogene rocks in the Makassar Straits, Indonesia

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**Abstract**

Borehole K-1 is an exploratory well that was drilled in the North Makassar Basin (West Sulawesi) in 2011. Gas chromatography (GC) and gas chromatography-mass chromatography (GC-MS) analyses have been conducted on extracts from well cuttings from the Paleogene to Neogene interval in order to investigate the characteristics of biomarkers present. Although the well was drilled with oil-based mud and gas chromatographic analysis reveals that the alkane fractions are heavily contaminated, detailed investigation of biomarkers in these rock extracts and comparison with biomarkers in the oil-based mud has revealed that, while there are hopane and sterane biomarkers in the mud, there are also a discrete set of biomarkers that are indigenous to the rocks. These include oleanane, bicadinanes, taraxastane and other higher-plant-derived triterpanes. The presence of these compounds in environments that range from bathyal to marginal marine and even to lacustrine, shows the extent of reworking of terrestrial material into aquatic settings in this region during the Paleogene and Neogene and provides further evidence of a predominance of terrestrial material, even in deep-marine settings, with little '*in-situ*' material noted. These findings have important implications for the use of biomarkers as indicators of palaeoenvironment in both source rocks and oils.

**Key words:** geochemistry, palaeoenvironment, taraxastane, oleanane

## 1. Introduction

The K-1 well is an exploratory petroleum borehole drilled in the Makassar Strait in the northern part of the K Block, North Makassar Basin, about 40 kilometres west of Pasangkayu City (West Sulawesi, Indonesia; see Fig. 1) in 2011. The status of the K Block is currently terminated, having been returned to the Government. Petroleum geochemical analyses, including gas chromatography (GC) and gas chromatography-mass spectrometry (GC-MS) analyses, of solvent extracts from drill cuttings from this well have been carried out in order to determine the nature of these extracts and relate extractable organic matter to depositional environments.

The purpose of the present work was to find out how organic matter preserved in sediments is influenced by organic matter input and depositional environment. Biomarker compositions of the aliphatic fractions of solvent extracts from samples from different depositional settings identified in the well

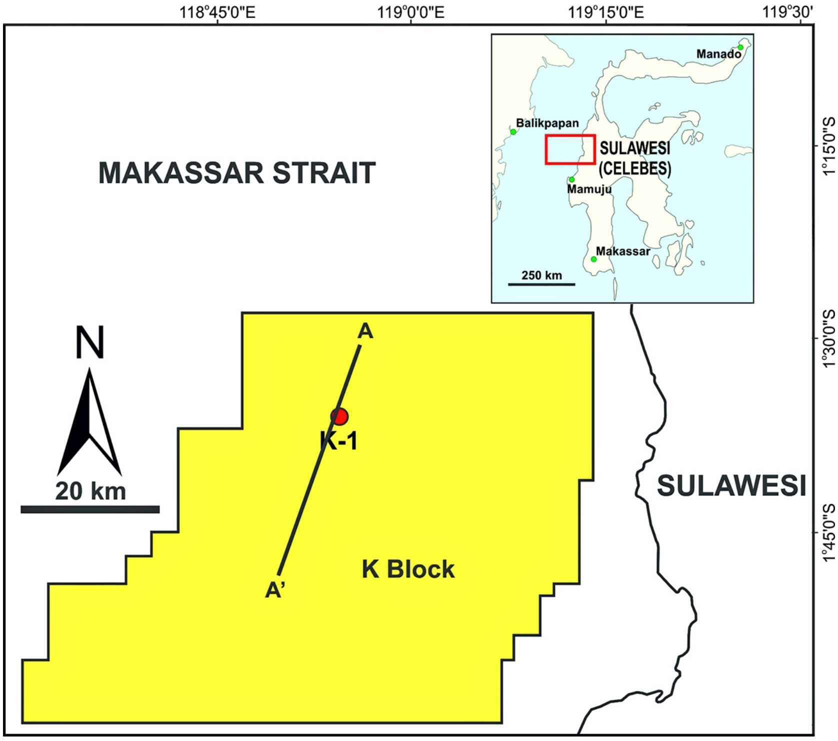

**Fig. 1**. Locality map

were used for this purpose. The wider implications for using biomarkers as indicators of depositional environment in tectonically dynamic settings has also been investigated.

## 2. Geological setting

The Makassar Strait is located between the islands of Kalimantan and Sulawesi in the central part of Indonesia. The strait extends for 700 km in a north-southerly orientation, with a width of 125–400 km and maximum water depth of nearly 2,500 m. The Makassar Strait is divided into northern and southern depressions which are often referred to as the North and South Makassar basins (Fig. 2).

Geographically, the Makassar Strait separates the western part of Indonesia from the eastern part. It was formed by rifting of the eastern part of Sundaland, resulting in separation of West Sulawesi from the main Sunda terrane. The timing and mode of the tectonic development of the Makassar Strait has been the subject of much discussion with various models proposed over the last 30 years for the nature of tectonics (rifting or sea floor spreading during the Paleogene, Neogene or Quaternary), the nature of the basement underlying the strait (continental, oceanic or transitional) and the opening speed (e.g., Katili, 1978; Hamilton, 1979; Situmorang, 1982; Hall, 1996; Cloke, 1997; Moss & Wilson, 1998; Guntoro, 1999; Calvert & Hall, 2003, 2007). This debate revolves mainly around the lack of data on the Makassar Strait, these models being based primarily on subsidence history, gravity, magnetic and tectonic information (Satyana et al., 2012).

Most authors suggest that the Makassar Strait resulted from extensional processes (Katili, 1978; Hamilton, 1979; Situmorang, 1982; Cloke, 1997; Guntoro, 1999) with early rifting during the Middle Eocene (Situmorang, 1982; Hall, 1996; Moss & Wilson, 1998; Guntoro, 1999; Calvert & Hall, 2003, 2007).

According to Hamilton (1979), there is an ocean-spreading centre that extends along the Makassar Strait, interpreted on the basis of several NW–SE trending faults. Fraser & Ichram (2000) also assumed oceanic crust to be present under the northern and southern parts of the Makassar Strait, while Cloke (1997) argued that oceanic crust was present only under the northern part of the strait. The Makassar Strait has also been interpreted as a remaining ocean basin (Malecek et al., 1993) or as a back-arc basin (Parkinson, 1998). There is another opinion that the rifting in the Makassar Strait never reached the stage of sea-floor spreading (e.g., Burollet & Salle, 1981; Situmorang, 1982). It has also been interpreted as a foreland basin that resulted from microcontinent collision in Sulawesi during the Miocene (Coffield et al., 1993; Bergman et al., 1996), in response to thrust loading on one or both sides. Satyana (2015) argued that the basement under the Makassar Strait comprised continental crust which was part of the Gondwanan Paternos-

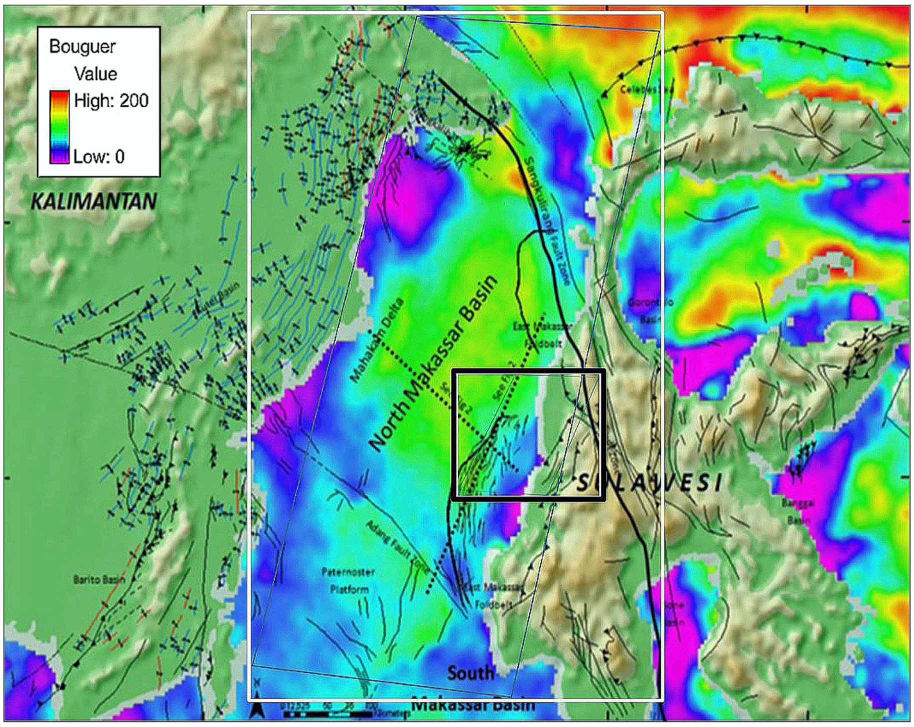

**Fig. 2.** Major Structural Elements of Trans-Eastern Sundaland, including Eastern Kalimantan, the Makassar Straits and Western Sulawesi (from Satyana et al., 2012). The area of Figure 1 is shown by a black square

ter-West Sulawesi microcontinent and thinned due to rifting during the Early/Middle Eocene to Early Miocene.

## 2.1. Exploration history

Since 1990, intensive petroleum exploration has been undertaken in the deep-water area on the western side of the Makassar Strait. This is a continuation of the success of early exploration and production achieved in the Mahakam Delta and Mahakam shelf areas, with the discovery of large oil and gas fields (Satyana et al., 2012). In contrast, the eastern part of the Makassar Straits still is at an early stage in the exploration cycle. However, during the last decade, exploration activities in this area have increased significantly, with acquisition of detailed 2D and 3D seismic grids and other geological-geophysical data collection and analysis. The exploration activity has augmented our geological knowledge of this area, although significant exploration success has not yet been achieved.

## 2.2. Tectonics

The tectonic history of the Makassar Strait should be evaluated as part of the history of Sundaland. At first, the Makassar Strait was part of eastern Sundaland. Sundaland is composed of a number of terranes from northern Gondwanaland (Metcalfe, 1996), which drifted apart and then merged to form Sundaland.

During the Middle Eocene (~50 Ma) some of the terranes of South-East Sundaland separated due to rifting and drifted to the east and southeast, leaving the rifted structures of East Sundaland currently in the Makassar Strait, East Java Sea, Gorontalo Bay and Bone Bay (Satyana, 2010).

East Kalimantan and West Sulawesi were part of one terrane during the Mesozoic, but separated during the Cenozoic. The Makassar Strait and West Sulawesi are believed to be fragments of the easternmost part of Sundaland. These early Cenozoic sequences are overlain by Cenozoic clastic sequences, carbonates and volcanics as a result of complex tectonics when the Australian-Indian, Pacific and Southeast Asian micro-plates interacted.

Figure 3 illustrates the tectonostratigraphy of the region. There is a significant difference between the stratigraphy of the North and South Makassar Basin, especially for strata of Neogene age. The differences are due to large volumes of sediment originating in East Kalimantan (from Borneo via Delta Mahakam) and West Sulawesi (volcanic foldbelt). This greatly affected the Neogene stratigraphy of the northern part of the Makassar Basin, which is different from that in the southern part.

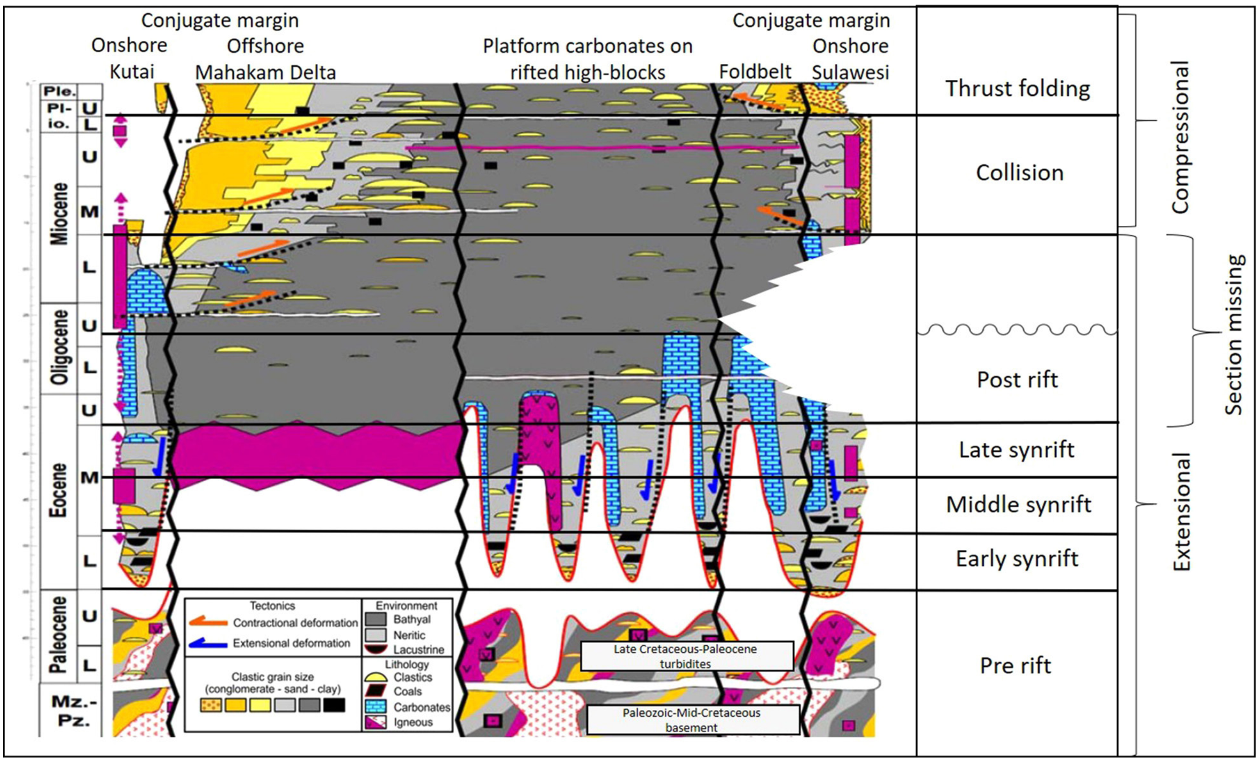

**Fig. 3**. Tectonostratigraphy of the Makassar Straits (modified from Bacheller III et al., 2011)

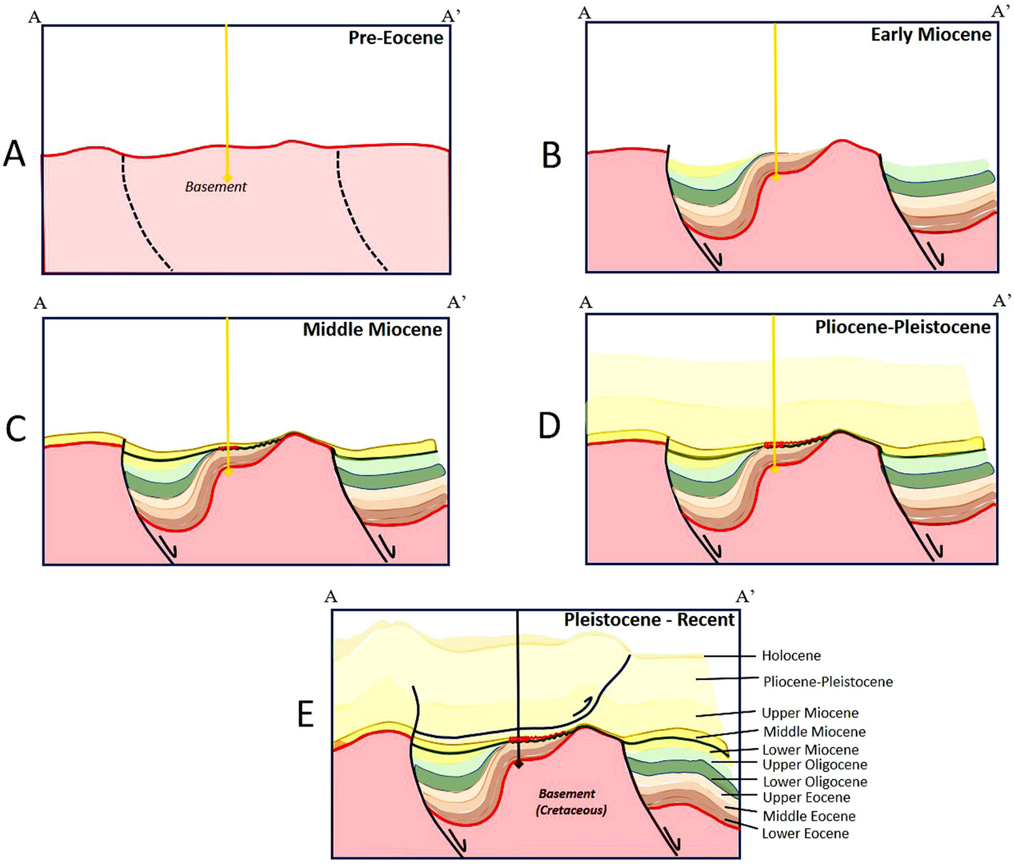

**Fig. 4**. Palinspastic reconstructions and present-day geoseismic cross section through well K-1. Location of section shown in Figure 1

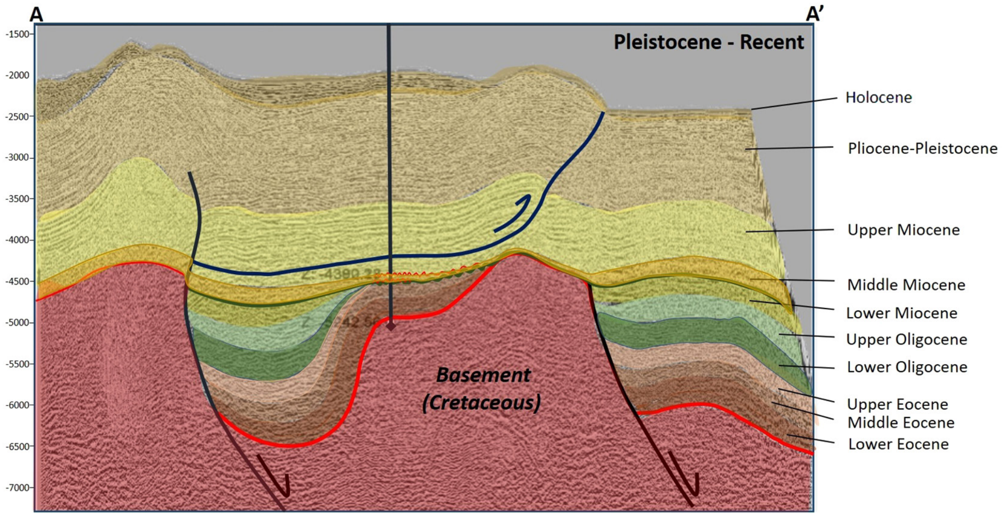

**Fig. 5**. Reinterpretation of seismic line through the K-1 well. Location of seismic line shown in Figure 1

The opening of the North Makassar Straits during the Eocene resulted in rifted structures comprising graben and horst, as indicated on seismic sections (Figs 4, 5).

The opening of the Makassar Strait due to thermal uplift during the Eocene led to suturing of the continental crust to form horst and graben structures, with these grabens becoming the site of lacustrine sedimentation. Understanding the palaeofacies of Eocene rocks in the Makassar Straits and West Sulawesi allows an estimation of the position of the initial suture.

## 3. Methods and material

Solvent extraction has been carried out on seven ditch cutting samples (YS-1–YS-7), comprising mainly claystones and siltstones, from the interval 8010′ to 16702′ in the K-1 well. The depth determination of samples was based on facies/depositional environment changes documented in existing biostratigraphical analysis. The depth of the seven samples represented each facies/depositional environment. The samples used have been prepared by combining a number of individual cutting samples collected during drilling, to obtain sufficient material for analysis. The cutting samples were crushed to powder, a known weight of this then placed in Soxhlet thimbles and extracted for 24 hours with 300 ml of a 93:7 dichloromethane/methanol mixture, using a Soxhlet extraction apparatus. The weight of the solvent extract was determined by evaporating solvent, using heat initially and then a nitrogen gas stream, after transfer to a vial. The extracts were weighed and then separated into saturate and aromatic fractions by liquid chromatography, using a chromatography column with silica inside the column. The relative proportions of saturate hydrocarbons, aromatic hydrocarbons and polar (non-hydrocarbon) compounds were recorded, using the solvent of 100% hexane, 50:50 hexane/dichloromethane mixture and 50:50 dichloromethane/methanol mixture, respectively.

The saturate fractions were submitted for gas chromatography (GC) using an AGILENT 6890N gas chromatograph with a 10 m glass capillary column with an internal diameter of 0.21 mm coated with DB-1 (J&W), and applying a temperature programme comprising an initial 35°C for 1 min, then increasing the temperature from 35°C to 315°C at 20°C/min, followed by holding the temperature at 315°C for 16 mins.

The saturate fractions were also submitted for gas chromatography-mass spectrometry (GC-MS) also using an AGILENT 6890 gas chromatograph, linked to an AGILENT 6890 Mass Selective Detector (MSD) running in multiple ion detection mode, with ion range *m/z* 191 to *m/z* 217. The GC contains a 60 m glass capillary column with an internal diameter of 0.25 mm coated with DB-5MS (J&W). The temperature programme used for this analysis comprised an initial 70°C for 1 min, then included three temperature ramps (70°C to 150°C at 10°C/min, 150–290°C at 2°C/min and 290–315°C at 5°C/min), followed by holding the temperature at 315°C for 6 mins.

Total organic carbon analysis was also run on the samples selected prior to extraction to enable calculation of extract yields relative to organic carbon content. This was carried out by a LECO Carbon Analyser (SC-114).

The results of these geochemical analyses were also integrated with existing biostratigraphical and palaeoenvironmental breakdowns for the well, as derived from microfaunal and palynological analyses of a duplicate set of cutting samples from the well and conducted by the operator as part of the post-drill well analysis (Harsanti et al., 2013). The post-drill geochemical report (Geoservices, 2012) was also available to provide back-up geochemical data such as kerogen type from visual microscopy and vitrinite reflectivity (VR).

## 4. Results and discussion

### 4.1. Evaluation of geochemical data

Table 1 lists the samples analysed, together with their organic carbon contents and extract yields. Extract yields are extremely high for these samples and high extractable percentages of organic carbon content (44.9–94.0%) are also noted. Hunt (1979)

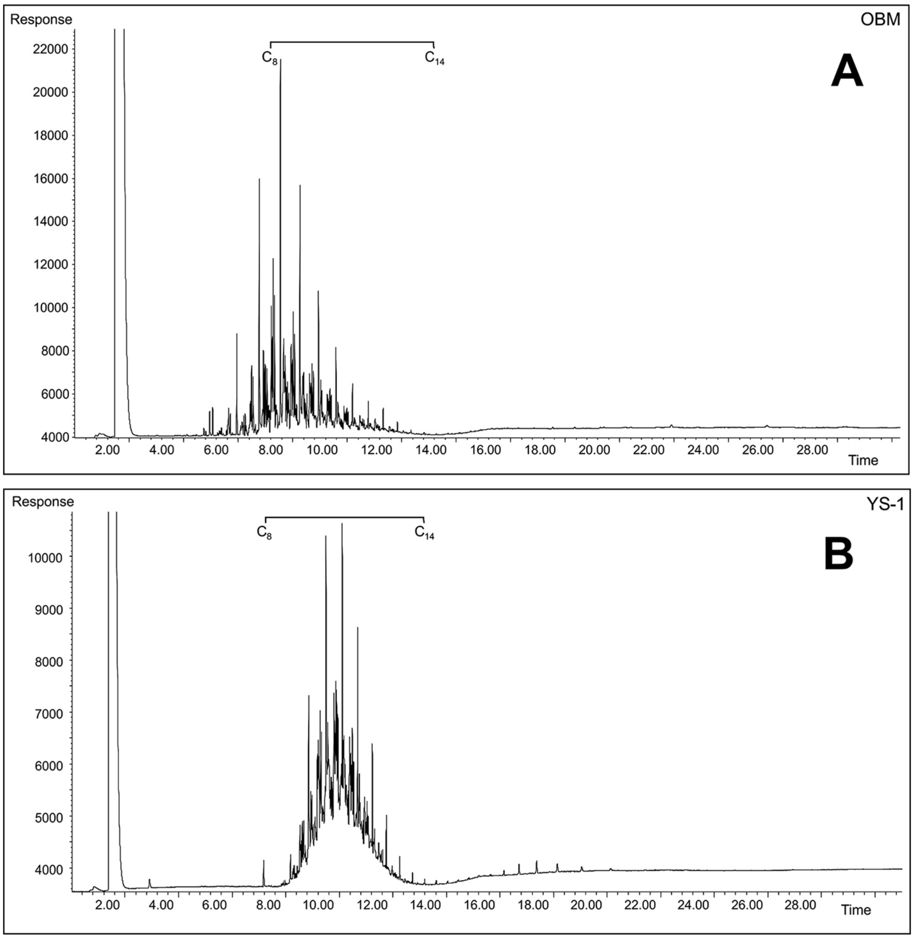

**Fig. 6**. $C_{5+}$ whole extract gas chromatograms for cuttings samples from 2441 m in K-1 well. GC profile of oil-based mud provided for comparison purposes. **A –** GC profile of cutting samples, containing Cx–Cy n-alkanes and an unresolved complex mixture (UCM) of hydrocarbons; **B –** GC profile of oil-based mud. These sediment extracs are dominated by hydrocarbon from drilling mud, which exhibits similar n-alkane profile.

**Table 1**. List of samples analyzed, together with organic carbon contents and extract yields

| Sample identification | | | | Weight (gram) | TOC (%) | Extract/ TOC (%) |
|---|---|---|---|---|---|---|
| Symbol | Depth (feet) | Depth (meter) | Lithology | | | |
| YS-1 | 8010 | 2441,5 | Claystone | 75.9 | 7.23 | 80.1 |
| YS-2 | 8910–10890 | 2715,8–3319,3 | Claystone, sandstone/siltstone interbeds | 76.7 | 4.73 | 77.5 |
| YS-3 | 14410 | 4392,2 | Siltstone | 77.8 | 2.93 | 81.7 |
| YS-4 | 15860 | 4834,1 | Claystone | 76.6 | 4.15 | 44.9 |
| YS-5 | 16250–16310 | 4953–4971,3 | Claystone, sandstone/siltstone interbeds | 76.4 | 6.15 | 77.1 |
| YS-6 | 16320–16550 | 4974,3–5044,4 | Claystone, sandstone/siltstone interbeds | 75.1 | 5.70 | 52.8 |
| YS-7 | 16702 | 5090,8 | Claystone | 75.9 | 3.48 | 94.0 |

indicated that indigenous extractable bitumens, expressed as a percentage of organic carbon, did not exceed 20% in sediments, so it is clear that these bitumens comprised either oil stain or contamination. The GC trace of the alkane fractions of each of the sediment extracts are closely similar to the example shown in Figure 6 (8010′), containing Cx – Cy *n*-alkanes and an unresolved complex mixture (UCM) of hydrocarbons, the latter component indicated by elevated GC baselines. Comparison with n-alkanes extracted from the drilling mud (Fig. 6) indicates that these sediment extracts are dominated by hydrocarbons from the drilling mud, which exhibits a similar n-alkane profile. A check of the operator's final well report confirms that the synthetic oil-based mud NAV-SOBM was added to drilling mud in the K-1 well, between 7988′ and 17360′ (ConocoPhillips, 2012).

Despite this heavy contamination of the extracts, the absence of alkanes in the $C_{25}$–$C_{35}$ region of the gas chromatogram of the alkanes in the oil-based mud suggests that any sterane and triterpane biomarkers present in the cutting extracts may be indigenous to these rocks. Consequently, GC-MS analysis was

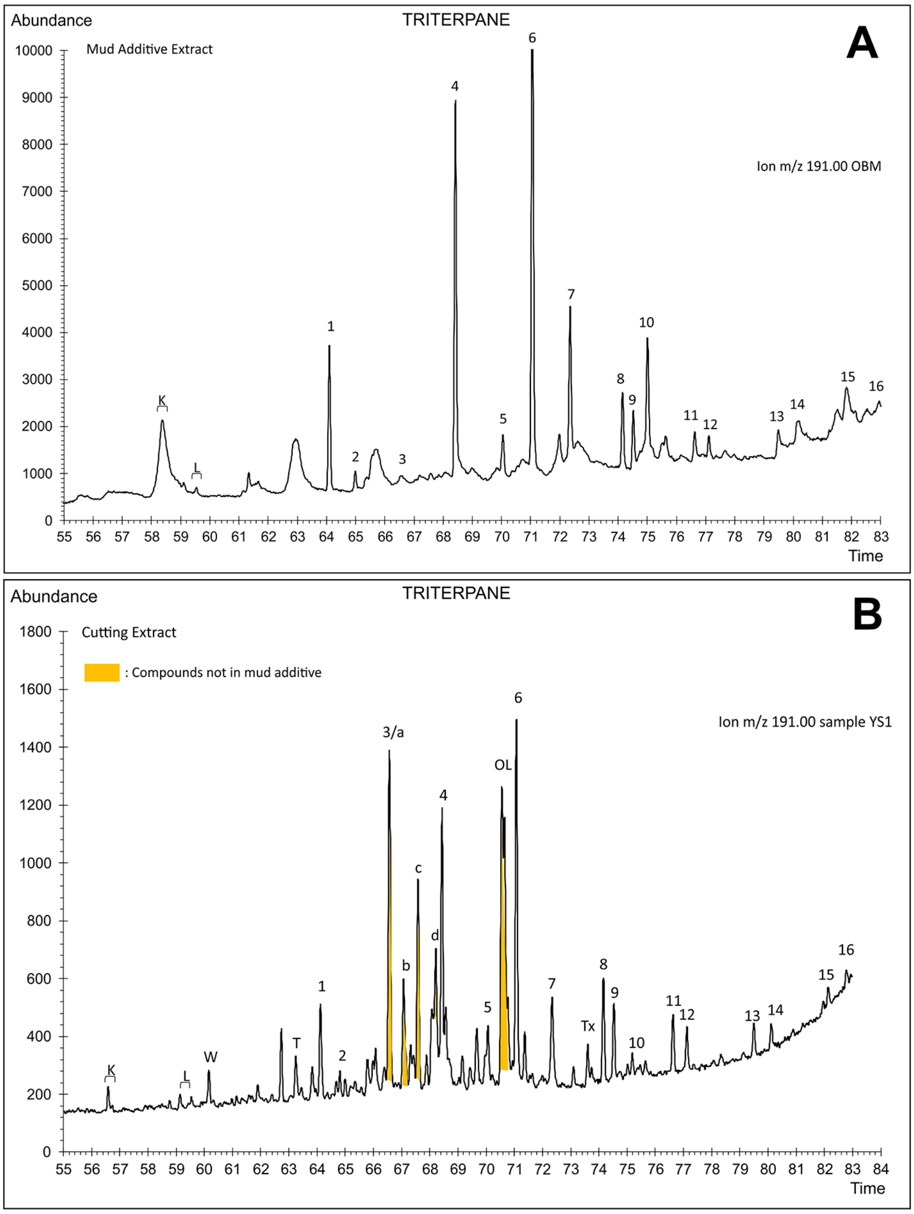

**Fig. 7**. Comparison of triterpane content of oil-based mud and rock extract (peak identifications shown in Table 2 and 3). **A –** Ion m/z 191 fragmentogram of rock extracts. There are additional triterpenoid compounds in the rock extracts that are not in the oil-based mud. These additional triterpenoids are interpreted to be indigenous to the rocks; **B –** Ion m/z 191 fragmentogram of oil-based mud extract containing a set of hopanes and steranes

performed on the aliphatic fractions of each of these extracts and an example of the *m/z* 191 (triterpane) and *m/z* 217 (sterane) fragmentograms is provided in Figures 7 and 8, together with those for an extract from a sample of the synthetic oil-based mud used in the K-1 well. Peak identifications are provided in Tables 2 and 3.

A comparison of the fragmentograms for the rock extract and the oil-based mud shows that, while the oil-based mud extract does contain a set of hopanes and steranes, there are additional triterpenoid compounds in the rock extracts that are not seen in the oil-based mud. These additional triterpenoids are interpreted to be indigenous to the rocks.

Of particular interest in this respect are a series of higher-plant derived terpanes noted on the *m/z* 191 fragmentogram which include oleanane (OL), bicadinanes (W, T & R), taraxastane (Tx) and other compounds (a, b, c, d). (Figs 7, 8). The nature and significance of these compounds are discussed in the following section.

Examination of the *m/z* 191 mass fragmentograms in Figures 7 and 8 indicates that these higher-plant derived biomarkers are abundant relative to the hopanes from 8010′ to 14410′, but show a de-

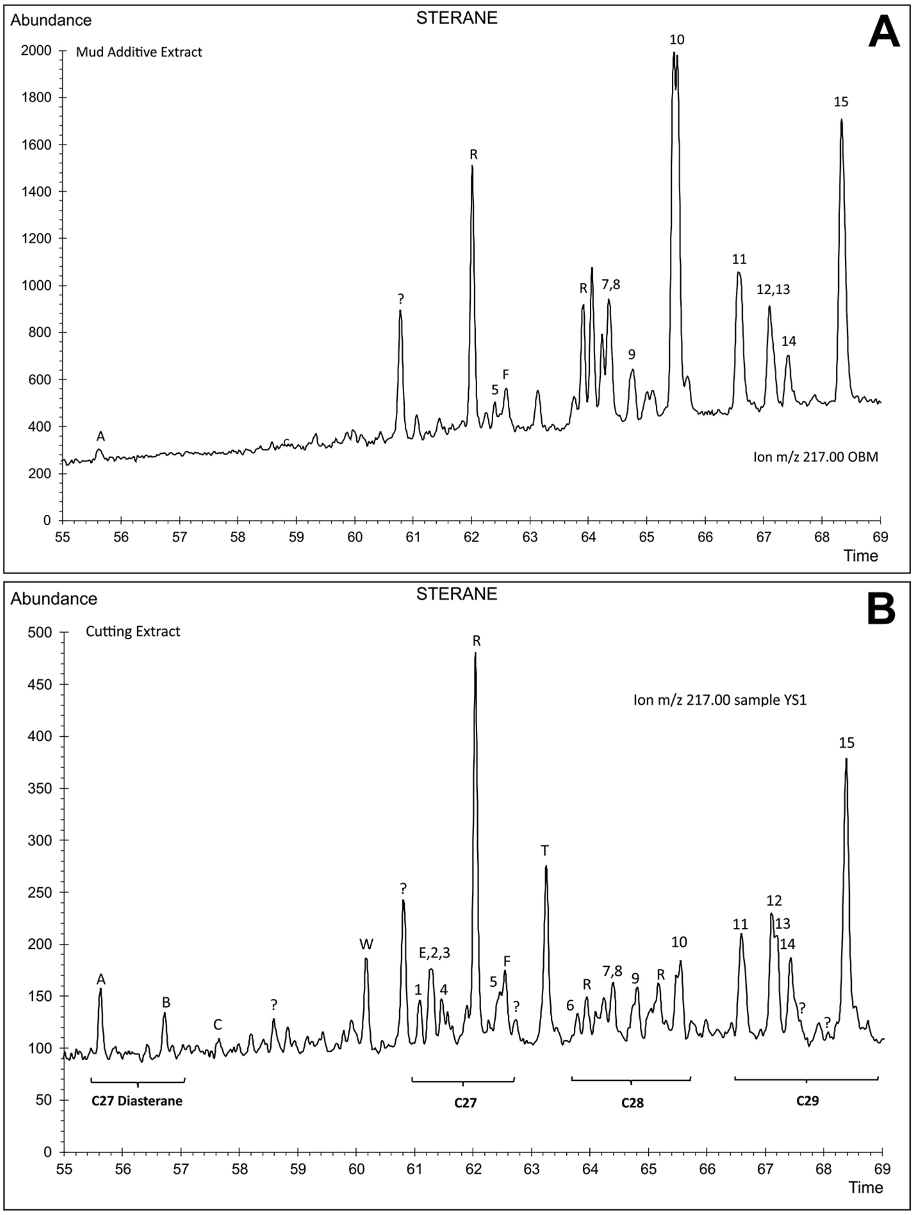

**Fig. 8**. Comparison of sterane content of oil-based mud and rock extract (peak identifications shown in Table 2 and 3). **A –** Ion m/z 217 fragmentogram of rock extracts; **B –** Ion m/z 217 fragmentogram of oil-based mud extract containing a set of hopanes and steranes

**Table 2.** Key to terpane peak identification

| Terpane | | |
|---|---|---|
| Peak ID | Carbon number | Compound name |
| K | 28 | Tricyclic diterpane |
| L | 29 | Tricyclic diterpane |
| 1 | 27 | 18α(H),21β(H)-22,29,30-trisnorhopane(Ts) |
| 2 | 27 | 17α(H),21β(H)-22,29,30-trisnorhopane(Tm) |
| 3 | 28 | 17α(H),21β(H)-28,30-bishorhopane |
| 4 | 29 | 17α(H),21β(H)-30-norhopane |
| 5 | 29 | 17β(H),21α(H)-30-normoretane |
| 6 | 30 | 17α(H),21β(H)-hopane |
| 7 | 30 | 17β(H),21α(H)-moretane |
| 8 | 31 | 17α(H),21β(H)-30-homohopane(22S) |
| 9 | 31 | 17α(H),21β(H)-30-homohopane(22R) |
| 10 | 31 | 17b(H),21α(H)-30-homomoretane |
| 11 | 32 | 17α(H),21β(H)-30,31-bishomohopane(22S) |
| 12 | 32 | 17α(H),21β(H)-30,31-bishomohopane(22R) |
| 13 | 33 | 17α(H),21β(H)-30,31,32-trishomohopane(22S) |
| 14 | 33 | 17α(H),21β(H)-30,31,32-trishomohopane(22R) |
| 15 | 34 | 17α(H),21β(H)-30,31,32,33-tetrahomohopane(22S) |
| 16 | 34 | 17α(H),21β(H)-30,31,32,33-tetrahomohopane(22R) |
| OL | 30 | 18a(H)-Oleanane |
| Gm | 30 | Gammacerane |
| Tx | 30 | Taraxastane |
| W | 30 | Cis-cis-trans-bicadinane |
| T | 30 | Trans-trans-trans-bicadinane |
| R | 30 | Trans-trans-trans-bicadinane |
| a | 30 | Higher-plant terpane |
| b | 30 | Higher-plant terpane |
| c | 30 | Higher-plant terpane |
| d | 30 | Higher-plant terpane |

**Table 3**. Key to sterane peak identification

| Sterane | | |
|---|---|---|
| Peak ID | Carbon number | Compound name |
| A | 27 | 13β(H),17α(H)-diacholestane(20S) |
| B | 27 | 13β(H),17α(H)-diachclestene(20R) |
| C | 28 | 24-Methyl-13β(H),17α(H)-diacholestane(20S) |
| E | 29 | 24-Ethyl-13β(H),17α(H)-diacholestane(20S) |
| F | 29 | 24-Ethyl-13β(H),17α(H)-diacholestane(20R) |
| 1 | 27 | 5α(H),14α(H),17α(H)-cholestane(20S) |
| 2 | 27 | 5β(H),14α(H),17α(H)-cholestane(20R) |
| 3 | 27 | 5α(H),14β(H),17β(H)-cholestane(20R) |
| 4 | 27 | 5α(H),14β(H),17β(H)-cholestane(20S) |
| 5 | 27 | 5α(H),14α(H),17α(H)-cholestane(20R) |
| 6 | 28 | 24-Methyl-5α(H),14α(H),17α(H)-cholestane(20S) |
| 7 | 28 | 24-Methyl-5β(H),14α(H),17α(H)-cholestane(20R) |
| 8 | 28 | 24-Methyl-5α(H),14β(H),17β(H)-cholestane(20R) |
| 9 | 28 | 24-Methyl-5α(H),14β(H),17β(H)-cholestane(20S) |
| 10 | 28 | 24-Methyl-5α(H),14α(H),17α(H)-cholestane(20R) |
| 11 | 29 | 24-Ethyl-5α(H),14α(H),17α(H)-cholestane(20S) |
| 12 | 29 | 24-Ethyl-5β(H),14α(H),17α(H)-cholestane(20R) |
| 13 | 29 | 24-Ethyl-5α(H),14β(H),18β(H)-cholestane(20R) |
| 14 | 29 | 24-Ethyl-5α(H),14β(H),17β(H)-cholestane(20S) |
| 15 | 29 | 24-Ethyl-5α(H),14α(H),17α(H)-cholestane(20R) |
| W | 30 | Cis-cis-trans-bicadinane |
| T | 30 | Trans-trans-trans-bicadinane |
| R | 30 | Trans-trans-trans-bicadinane |

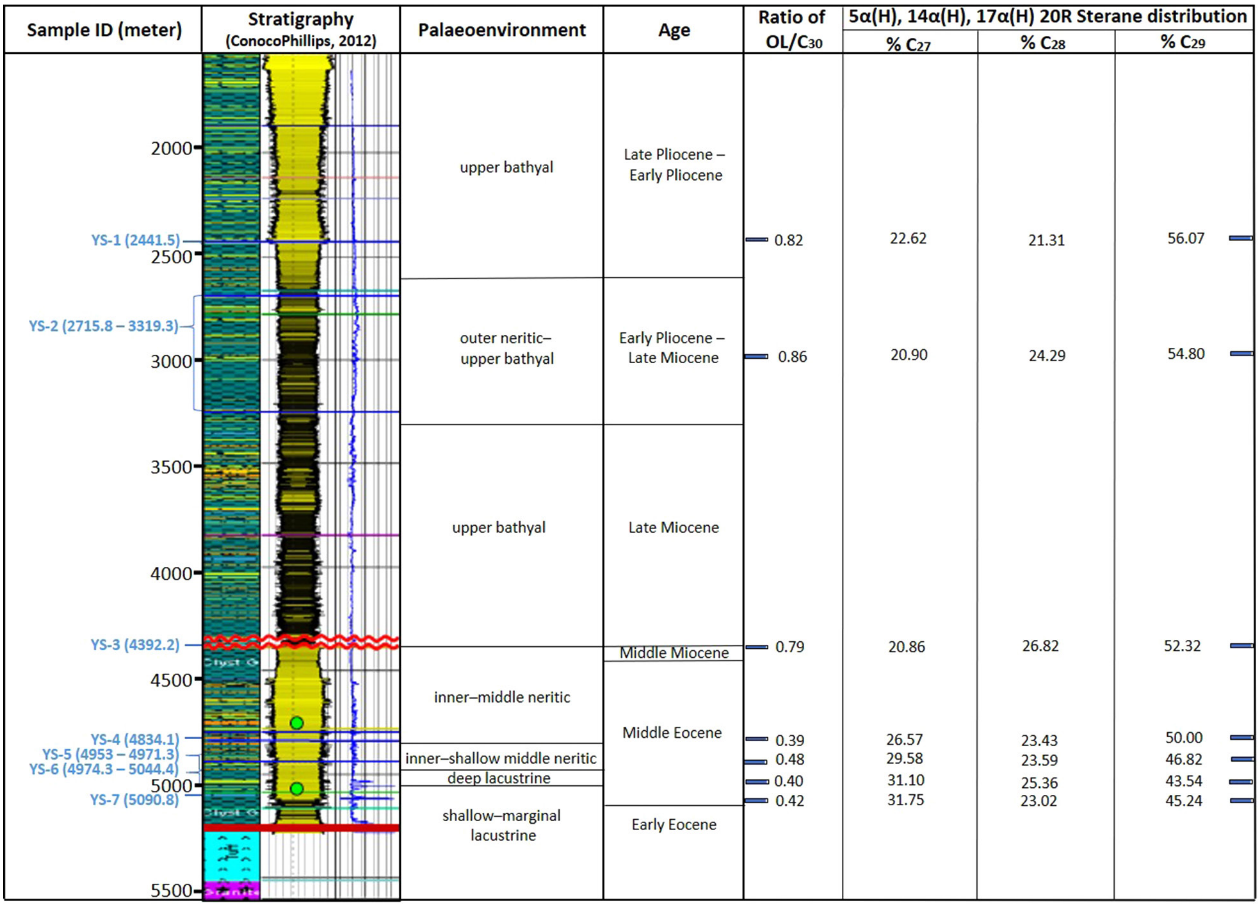

**Fig. 9**. Well depth plot illustrating changes in oleanane/hopane ratio and 5α14α17α 20R sterane carbon number distribution with depth, age and palaeoenvironment

crease in abundance below this depth. This decrease has been quantified by calculation of oleanane/$C_{30}$ hopane ratios for each of these samples. These ratios are shown in Figure 9 and demonstrate a drop from values of 0.79–0.86 at and above 14410′ to 0.39–0.48 below. Taraxastane and other higher-plant compounds show similar trends.

It was necessary to determine whether the abundance of these higher plant-derived terpanes relative to the hopanes, and the sterane carbon number distributions, of the alkanes from the rock extracts, were of geological significance or resulted from variations in the degree of contamination from the oil-based mud. Unfortunately, absolute concentrations of the biomarkers in the extracts and oil-based mud were not determined and thus could not be used to identify the degree of contamination of the sediment biomarker fraction by compounds derived from the mud.

Consequently, $C_{30}$ moretane/$C_{30}$ hopane and $C_{29}$ 5α(H)14α(H)17α(H) 20S/20R sterane depth plots, together with a vitrinite reflectivity depth plot (Fig. 10) and a 5α(H)14α(H)17α(H)20R $C_{27}$/$C_{28}$/$C_{29}$ sterane carbon number Ternary diagram (Fig. 11) were constructed to attempt to quantify the effects of contamination on the regular sterane and hopane compositions. The $C_{30}$ moretane/$C_{30}$ hopane depth plot shows a trend of decreasing values with depth below 9900′ which is consistent with the increasing maturity levels of the rocks, as indicated by the vitrinite reflectivity depth plot.

Peters et al. (2005) indicated that the moretane/hopane ratio decreased from values of ~0.8 in immature extracts to less than 0.15 in mature source rocks, although higher values may be encountered in mature Paleogene or Neogene rocks (0.1–0.3). These values are consistent with the vitrinite reflectivity profile for the well shown in Figure 10, which increases from ~0.32%Ro at 9000′ to ~0.75%Ro at 16500′. The unusually high moretane/hopane value for the shallowest sample analysed is not consistent with the other values obtained, but is not consistent with the value obtained from the oil-based mud either. The reason for this low value at this depth may be the presence of only reworked organic matter at this depth or, morelikely, the presence of mi-

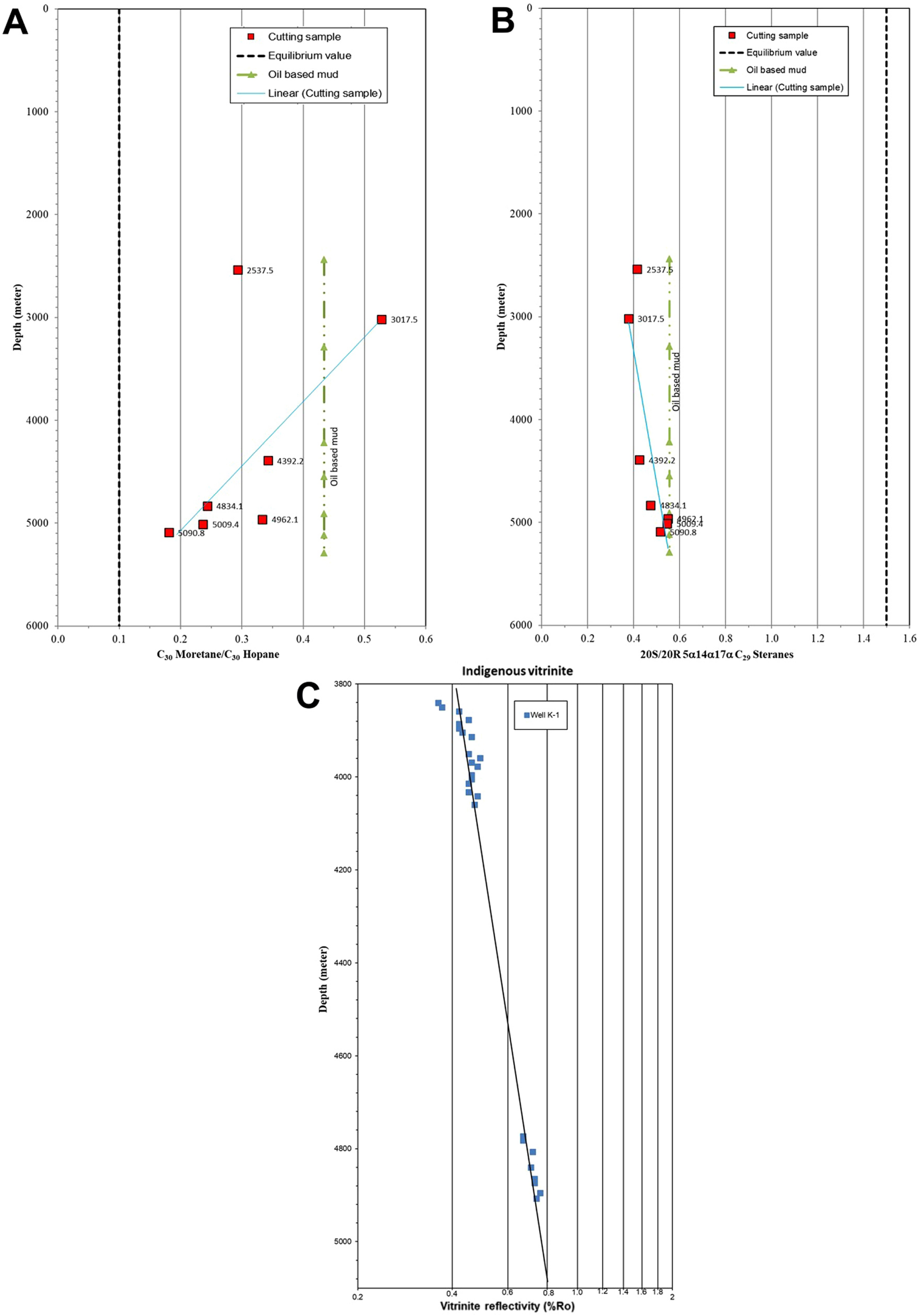

**Fig. 10**. Depth plots of: **A –** $C_{30}$ Moretane/$C_{30}$ Hopane; **B –** %20S 5α14α17α$C_{29}$ Steranes; **C –** Vitrinite reflectance (VR) value for cutting samples in K-1 well

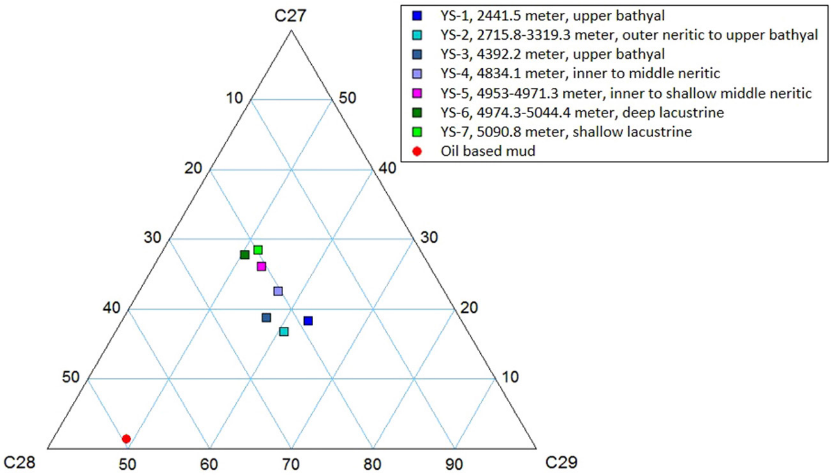

**Fig. 11.** Ternary diagram illustrating variation in 5α14α17α20R steranes for samples analysed

nor amounts of mature oil stain. The trend of decreasing moretane/hopane ratios in the remaining samples, consistent with the maturation profile for the well, suggests that the triterpane fractions of the extracts were not significantly contaminated by triterpanes from the oil-based mud.

The abundant presence of the $C_{30}$ triterpane gammacerane (peak 10 in Fig. 7) in the oil-based mud extract, but the low abundance of this compound in all sediment extracts is a further indication that the triterpane fractions of the extracts are not significantly contaminated by triterpanes from the oil-based mud.

The depth profile for the maturity dependent 20S/20R ratio for the 5α(H)14α(H)17α(H) $C_{29}$ steranes is also shown in Figure 10, together with the value obtained for the oil-based mud extract. Unfortunately, the values do not show a clear trend of increasing 20S/20R ratios with depth and, because most of the values plot close to that of the oil-based mud extract, the possibility that the sterane component was contaminated by the oil-based mud must be considered.

The Ternary diagram of the $C_{27}$, $C_{28}$ and $C_{29}$ 5α(H)14α(H)17α(H)20R sterane compositions (Fig. 11) shows a clear distinction of the cutting extracts and the oil-based mud extracts. While the latter have similar levels of $C_{29}$ steranes to the cuttings extracts it is very lean in $C_{27}$ steranes and enriched in $C_{28}$ steranes. This distinction, and the fact that the major difference between the various cutting extracts is related to changes in the $C_{29}$ and $C_{27}$ sterane content, suggests that differences observed in the sterane carbon number distribution relate to changes in organic matter type in these rocks rather than to the extent of contamination.

Although the well was drilled with oil-based mud and the cutting extracts were heavily contaminated, it is concluded that the triterpane fractions of these extracts are not significantly contaminated and the sterane carbon number distributions can also be used to investigate variations in organic matter input to the rocks.

## 4.2. Discussion of geochemical results

Triterpenoids believed to be associated with higher plants have been identified in sediments younger than Early Cretaceous and associated crude oils (e.g., Hills et al., 1970; Philp & Gilbert, 1986; Thomas, 1990). Oleananes are formed in sediments through diagenetic and catagenetic alteration from various "triterpenoids of angiosperms" (Rullkötter et al., 1994). Being diagnostic of the age of the source rock and the contribution of terrestrial plants, they are important molecular fossils in petroleum geochemistry (Peters et al., 2005; Moldowan et al., 1994).

Moldowan et al. (1994) showed the relationship between oleanane / hopane, the age of source rock and fossil evidence for the rise of angiosperms during the Cretaceous, to support the use of oleananes as age markers. The abundance of oleanane is believed to be very sensitive to changes in Eh and pH during early diagenesis and the effects on oleanane precursors. A marine influence during early diagenesis increases the abundance of oleananes in sediments and oils and reduces skeletal alteration and aromatisation (Murray et al., 1997). However, the oleanane skeleton is considered to be one of the most stable of the plant-derived triterpenoids (Rullkötter et al., 1994). For example, it has been shown that lupenes can be converted to taraxastenes and oleanenes under acidic conditions (Perkins et al., 1995).

Bicadinane is a biomarker that seems to be specific for angiosperm resin-sourced oils (Pearson & Alam, 1993). Van Aarssen et al. (1992) speculated about their distribution in relation to maturation and migration. Bicadinane forms after early diagenesis, as shown by various parameters of geochemical maturity and vitrinite reflectance, but before the onset of oil generation at temperatures within the range, 25–90°C (Pearson & Alam, 1993). Bicadinanes derived from fossil higher plant resin are present in oils and sediments also as higher oligomers and polymers (Cox et al., 1986; van Aarssen et al., 1990, 1992).

The structures of two bicadinanes, originally named compounds 'T' and 'W', were determined by Cox et al. (1986) and van Aarssen et al. (1990), respectively, and designated as trans-trans-trans- and cis-cis-trans-bicadinane. Taraxastane is often associated with 18α-oleanane, and "resin compounds" (Grantham et al., 1983). Cox et al. (1986) and van Aarssen et al. (1990, 1992) identified taraxastane as a bicadinane isomer.

The compounds labelled a, b, c and d were identified by numerous researchers (Killops et al., 1995; Murray et al., 1994; Mathur, 2014) in Cenozoic oils and rock extracts from fluviodeltaic settings, but were not structurally identified, being referred to as unidentified $C_{30}$ triterpanes and $C_{30}$ oleanoid triterpanes. Murray et al. (1994) pointed out that they were never found without oleanane in sediments and oils but often oleanane was encountered in marine and lacustrine sediments without the oleanoid triterpanes, suggesting the oleanoid triterpanes may signal *in-situ* as opposed to allochthonous deposition of terrestrial organic matter.

Based on biostratigraphical analysis of the K-1 well (Harsanti et al., 2013), summarised in Figure 9, Upper Pliocene to Middle Eocene sedimentary rocks within the interval 8010′ to 16702′ were laid down in environments that ranged from upper bathyal to shallow lacustrine. Changes in the biomarker content of these samples probably reflect organic matter input and organic matter preservation at the site of deposition and may also reflect the age of the sediments.

The ternary diagram of 5α14α17α 20R steranes (Fig. 11) shows an increase in $C_{27}$ steranes from deep- to shallow-marine and lacustrine environments. As $C_{27}$ steranes are generally associated with algal input (Huang & Meinshein, 1979) the increase in $C_{27}$ content from deep- to shallow-marine environments may be due to increased settling distances from the photic zones for phytoplankton remains and the consequent concentration of transported terrestrial material in deep-water settings. In lacustrine settings water chemistry and proximity to terrestrial input is believed to be the controlling factor.

Triterpenoids identified in the present study include oleanane, bicadinane, taraxastane and the compounds labelled a, b, c and d. The high levels of higher-plant triterpanes in the biomarker fractions of each these samples are significant, suggesting significant higher-plant contribution despite the range of environments observed. The oleanane/hopane ratio is high (0.79–0.86) in the Neogene samples from deep-marine (outer neritic to upper bathyal) and significantly lower (0.39–0.48) in the Middle Eocene samples from shallow-marine and lacustrine settings (Fig. 9). While Murray et al. (1994) noted that oleanane was often abundant in marine settings, Peters et al. (2005) also indicated that it was likely to be more abundant in Neogene than in Paleogene sedimentary rocks, due to the increasing abundance of angiosperms (flowering plants) during the Neogene.

The pattern of bicadinane (W, T and R) abundance is less clearly defined, although lowest abundances are noted for the lacustrine samples. These compounds are believed to be derived from the mainly tropical lowland rainforest family Dipterocarpaceae which are rich in resins (van Aarssen et al., 1992). Their presence in the deep-water samples again suggests reworking of organic material from coastal plain settings.

Taraxastane (Tx) abundance in the extracts mirrors oleanane abundance, suggesting that they may both have the same precursor (taraxer-14-ene) (Rullkötter et al., 1994) and consequently also reflect angiosperm input to both the marine and, to a lesser extent, lacustrine environments that are identified in the K-1 well.

The still unidentified compounds (a, b, c and d), here referred to as higher-plant terpanes, but termed oleanoid or unknown $C_{30}$ triterpanes (e.g., Murray et al., 1994; Rullkötter et al., 1994; Mathur, 2014) are most abundant in the deep-marine settings and less abundant in shallow-marine and lacustrine settings. Murray et al. (1994) suggested that these compounds represented *in-situ*, rather than allochthonous, deposition of terrestrial organic matter, although their presence in these deep-water sediments suggests that this may not be the case (Fig. 12).

Results of integrated biostratigraphical analysis of the K-1 well, summarised in Figure 9, indicates that the top of the Lower Eocene is at 16910′. During the Early to Middle Eocene, deposition occurred in environments which were initially lacustrine but with initial transgression, associated with the opening/widening of the Makassar Strait, which resulted in the deposition of shallow-marine sedimentary rocks until the end of the Middle Eocene.

Two erosional events occurred in this area, the first was at the end of the Middle Miocene, marked

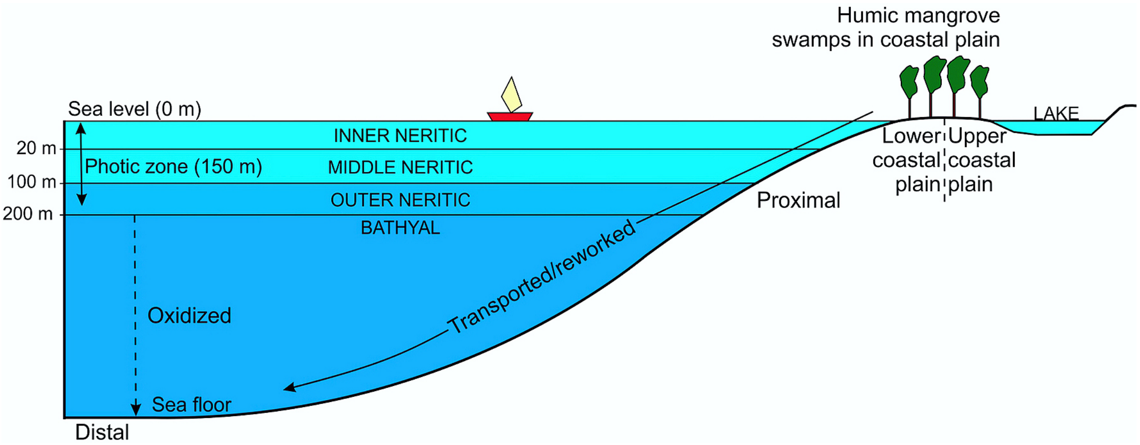

**Fig. 12**. Sketch illustrating range of depositional environments encountered in the Makassar Straits during the Paleogene and Neogene (based on A. Livsey, pers. comm., 2017)

by an unconformity at 14430′, and resulting in the absence of Upper Eocene, Lower–Upper Oligocene and Lower Miocene deposits (Figs 4, 5, 9). The second event occurred during the Late Miocene, is marked by an unconformity at 14390′ and resulted in the partial absence of Middle to Upper Miocene sedimentary rocks (Figs 4, 5, 9).

Based on the operator's final well report (ConocoPhillips, 2012), there is a detachment zone above the tuff at a depth of 14248′ (Late Miocene), which can be seen in Figure 9, and from a reinterpretation of the seismic line that intersects the K-1 well (Fig. 5). The markers in the final well report by the existing operator did not change; author did a reinterpretation so as to obtain a geological setting that could explain the loss of some sequences as a result of erosion. The methodology to do the reinterpretation of seismic line was using the well-tie as well marker based on the features of the existing seismic reflector.

Biomarker distributions in Neogene samples confirm extensive reworking of angiosperm plant material from coastal plain and shallow-marine settings into deep-water environments, an observation that is supported by the presence of mangrove pollen within the latter. The Makassar Strait was an open-marine system throughout the Neogene (Fig. 12). However, the present study indicates that the biomarker content of the sedimentary rocks formed in these deep-water settings reflects a strong contribution from land-derived organic matter. Similar conclusions were drawn for deep-water settings elsewhere in the Makassar Straits (Saller et al., 2006), where terrestrial material reworked during major lowstands is believed to have been the source of major oil accumulations. These oils also contain the same higher-plant biomarkers as here observed in deep-water deposits in the K-1 well. The absence of *in-situ* marine organic matter in these sediments is here proposed to have resulted from oxidation of marine phytoplankton remains during settling from the photic zone to the sea floor through an extensive, oxygen-rich water column (Fig. 12) (A. Livsey, pers. comm., 2017).

## 5. Conclusions

The organic materialcontained in deep-ocean environments (bathyal settings) reflected by Neogene rocks of the Makassar Strait are derived primarily from coastal plain and nearshore settings that were transported into these deep-water settings.

Biomarker distributions for these deep-water samples confirm the presence of higher-plant material related both to angiosperms (flowering plants) and tropical lowland forest trees, rich in plant resins such as Dipterocarpaceae.

Variations in the concentrations of higher-plant derived biomarkers from the Paleogene to the Neogene reflect a change from lacustrine and shallow-marine to deep-water settings, and also the greater abundance of flowering plants (angiosperms) during the Neogene. A greater contribution from marine phytoplankton in shallow-marine settings in comparison to the deep-water is believed to be due to the shorter settling distance from the photic zone to the sea floor and hence less oxidation.

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## References

Bacheller III, J., Buck, S.P., Cahyono, A.B., Polis, S.R., Helsing, C.E., Zulfitriadi, De Man, E.M., Hillock, P.M., Ruf, A.S. & Toxey, J.K., 2011. Early deepwater drilling results from a new exploration play, offshore West Sulawesi, Indonesia. *Proceedings of Indonesian Petroleum Association,* 35th Annual Convention and Exhibition Paper IPA11-G-243.

Bergman, S.C., Coffield, D.Q., Talbot, J.P. & Garrard, R.J., 1996. Tertiary tectonic and magmatic evolution of Western Sulawesi and the Makassar Strait, Indonesia: Evidence for a Miocene continent–continent collision [In:] Hall, R. & Blundell, D.J. (Eds): *Tectonic evolution of SE Asia*. Geological Society, London, Special Publications 106, 391–430.

Burollet, P.F. & Salle, C., 1981. Seismic reflection profiles in the Makassar Strait [In:] Barber, A.J. & Wiryosujono, S. (Eds): *The geology and tectonics of Eastern Indonesia*. Geological Research and Development Centre, Bandung, Special Publication 2, 273–276.

Calvert, S.J. & Hall, R., 2003. Cenozoic evolution of the Lariang and Karama regions, North Makassar Basin, Western Sulawesi, Indonesia. *Proceedings Indonesian Petroleum Association*, 29th Annual Convention, 501–517.

Calvert, S.J. & Hall, R., 2007. Cenozoic evolution of the Lariang and Karama regions, North Makassar Basin, Western Sulawesi, Indonesia. *Petroleum Geoscience* 13, 353–368.

Cloke, I.R., 1997. *Structural controls on the basin evolution of the Kutai Basin and the Makassar Straits*. Ph.D. Thesis, University of London, 374.

Coffield, D.Q., Bergman, S.C., Garrard, R.A., Guritno, N., Robinson, N.M. & Talbot, J., 1993. Tectonic and stratigraphic evolution of the Kalosi Psc area and Associated Development of a Tertiary Petroleum System, South Sulawesi, Indonesia. *Proceedings Indonesian Petroleum Association*, 22nd Annual Convention, 679–706.

ConocoPhillips, 2012. *Geology Final Well Report*. An internal report compiled by ConocoPhillips Operations Geoscience (unpublished).

Cox, H.C., de Leeuw, J.W., Schenck, P.A., van Koningsveld, H., Jansen, J.C., van de Graaf, B., van Geerestein, B., Kanters, J.A., Kruk, C. & Jans, A.W.R., 1986. Bicadinane, a C30 pentacyclic isoprenoid hydrocarbon found in crude oil. *Nature* 319, 316–318.

Fraser, T.H. & Ichram, L.A., 2000. Significance of the Celebes Sea spreading center to the Paleogene petroleum systems of the SE Sunda margin, Central Indonesia. *Proceedings Indonesian Petroleum Association*, 27th Annual Convention, 431–441.

Geoservices, 2012. *Geochemical Maturity & Kerogen Compositional Analysis of Ditch Cutting Samples from Neogen & Paleogene Intervals of Well Kaluku-1*. An internal report for ConocoPhillips Indonesia prepared by PT Geoservices (unpublished).

Grantham, P.L., Posthuma, J. & Baak, A., 1983. Triterpanes in a number of Far-Eastern crude oils [In:] Bjoray, M. (Ed.): *Advances in organic geochemistry 1981*. Wiley, Chichester, 675–683.

Guntoro, A., 1999. The formation of the Makassar Straits and the separation between SE Kalimantan and SW Borneo. *Journal of Asian Earth Sciences* 17, 79–98.

Hall, R., 1996. Reconstructing Cenozoic SE Asia. [In:] Hall, R. & Blundell, D.J. (Eds): *Tectonic evolution of SE Asia*. Geological Society, London, Special Publications 106, 153–184.

Hamilton, W., 1979. Tectonics of the Indonesian Region. *U.S. Geological Survey Professional Paper* 1078, 345.

Harsanti, S., Muhamad, S., Yuliana, P., Steve, N., Patrice, B., Muhamad, T. & Jim, C., 2013.

*Biostratigraphy and paleoenvironments of the interval 8010′ to 17360′ of Kaluku-1 Well, Sulawesi Sea, Indonesia*. An internal report for ConocoPhillips Indonesia prepared by PT Corelab Indonesia (unpublished).

Hills, I.R., Smith, G.W. & Whitehead, E.V., 1970. Hydrocarbons from fossil fuels and their relationship with living organisms. *Journal of the Institute of Petroleum* 56, 127–137.

Huang, W.Y. & Meinshein, W.G., 1979. Sterols as ecological indicators. *Geochimica et Cosmochimica Acta* 43, 739–745.

Hunt, J.M., 1979. *Petroleum Geochemistry and Geology*. W.H. Freeman and Company, 617 pp.

Katili, J.A., 1978. Past and present geotectonic position of Sulawesi, Indonesia. *Tectonophysics* 45, 289–322.

Killops, S.D., Raine, J.I., Woolhouse, A.D. & Weston, R.J., 1995. Chemostratigraphic evidence of higher plant evolution in the Taranaki Basin, New Zealand. *Organic Geochemistry* 23, 429–445.

Malecek, S.J., Reaves, C.M. & Atmadja, W.S., 1993. Seismic stratigraphy of Miocene and Pliocene age outer shelf and slope sedimentation in the Makassar PSC, offshore Kutei Basin. *Proceedings Indonesian Petroleum Association*, 22nd Annual Convention, 345–371.

Mathur, N., 2014. Tertiary oils from Upper Assam Basin, India a geochemical study using terrigenous biomarkers. *Organic Geochemistry* 76, 9–25.

Metcalfe, I., 1996. Pre-Cretaceous evolution of SE Asian terranes. [In:] Hall, R. & Blundell, D. (Eds): *Tectonic evolution of Southeast Asia*. Geological Society Special Publication 106, 97–122.

Moldowan, J.M., Dahl, J. & Huizinga, B.J., 1994. The molecular fossil record of oleanane and its relation to angiosperms. *Science* 265, 768–771.

Moss, S.J. & Wilson, M.E.J., 1998. Biogeographic implications of the Tertiary paleogeographic evolution of Sulawesi and Borneo. [In:] Hall, R. & Holloway, J.D. (Eds): *Biogeography and geological evolution of Southeast Asia*, Backhuys Publishers, Leiden, 133–163.

Murray, A.P., Summons, R.E., Boreham, C.J. & Dowling, L.M., 1994. Biomarker and n-alkane isotope profiles for Tertiary oils: relationship to source rock depositional setting. *Organic Geochemistry* 22, 521–542.

Murray, A.P., Sosrowidjojo, I.B., Alexander, R., Kagi, R.I., Norgate, C.M. & Summons, R.E., 1997. Oleananes in oils and sediments: evidence of marine influence during early diagenesis. *Geochimica et Cosmochimica Acta* 61, 1261 – 1276.

Parkinson, C., 1998. Emplacement of the East Sulawesi ophiolite: evidence from Subophiolite Metamorphic Rocks. *Journal of Asian Earth Sciences* 16, 13–28.

Pearson, M.J. & Alam, M., l993. Bicadinanes and other terrestrial terpenoids in immature Oligocene sedimentary rocks and a related oil from the Surma Basin, Bangladesh. *Organic Geochemistry* 20, 539–554.

Perkins, G.M., Bull, I.D., ten Haven, H.L., Rullkötter, J., Smith, Z.E.F. & Peakman, T.M., 1995. First positive identification of triterpenes of the taraxastane family in petroleums and oil shales: 19α(H)-Taraxastane and 24-nor-19α(H)-Taraxastane. Evidence for a previously unrecognised diagenetic alteration pathway of lup-20(29)-ene derivatives. [In:] Grimalt, J.O. & Dorronsoro, C. (Eds): *Organic geochemistry: developments and applications to energy, climate, environment, and human history*. Selected Papers 17th International Meeting on Organic Geochemistry, Donostia-San Sebastian, 247–249.

Peters, K.E., Walters, C.C. & Moldowan, J.M., 2005. *The biomarker guide*, Vol. II. Cambridge University Press, Cambridge, 487–492, 614–615.

Philp, R.P. & Gilbert, T.D., 1986. Biomarker distribution in Australian oils predominantly derived from terrigenous source material. *Organic Geochemistry* 10, 73–84.

Rullkötter, J., Peakman, T.M. & Haven, H.L.T., 1994. Early diagenesis of terrigenous triterpenoids and its implications for petroleum geochemistry. *Organic Geochemistry* 21, 215–233.

Saller, A., Lin, R. & Dunham, J., 2006. Leaves in turbidite sands: the main source of oil and gas in the deep-water Kutei Basin, Indonesia. *AAPG Bulletin* 90, 1585–1608.

Satyana, A.H., 2010. Crustal structures of the eastern Sundaland's rifts, Central Indonesia: geophysical constraints and petroleum implications. *Proceedings of the Bali 2010 International Geosciences Conference and Exposition*, Bali, Indonesia, 2–10.

Satyana, A.H., 2015. Rifting history of the makassar straits: new consideration from well penetrating the basement and oils discovered in Eocene section – implications for further exploration of West Sulawesi offshore. *Proceedings of Indonesian Petroleum Association*, 39th Annual Convention, Jakarta, paper IPA15-G-104.

Satyana, A.H., Damayanti, S. & Armandita, C., 2012. Tectonics, stratigraphy and geochemistry of the Makassar Straits: recent updates from exploring West Sulawesi offshore, opportunities and risks. *Proceedings. Indonesia Petroleum Association*, Annual Convention and Exhibition, paper IPA12-G-156.

Situmorang, B., 1982. The formation of the Makassar Basin as determined from subsidence curves. *Proceedings Indonesian Petroleum Association*, 11th Annual Convention, 83–107.

Thomas J., 1990. *Biological markers in sediments*. Ph.D. Thesis, University of Bristol.

Van Aarssen, B.G.K., Kruk, C., Hessels, J.K.C. & de Leeuw, J.W., 1990. Cis-cis-trans-bicadinane, a novel member of an uncommon triterpane family isolated from crude oils. *Tetrahedron Letters* 31, 4645–4648.

Van Aarssen, B.G.K., Hessels, J.K.C., Abbingk, O.A. & de Leeuw, J.W., 1992. The occurrence of polycyclic sesqui-, tri- and oligoterpenoids derived from a resinous polymeric cadinene in crude oils from Southeast Asia. *Geochimica et Cosmochimica Acta* 56, 1231–1246.

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