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DIAGENESIS OF MARINE SEDIMENTARY HYDROCARBONS AND THE SOURCE ROCK POTENTIAL OF SEDIMENTS IN THE ASHMORE REEF NO. 1, IVIRI NO. 1, NAUTILUS NO. 1A AND OROKOLO NO. 1 WELLS

by

T.G. Powell and D.M. McKirdy

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SUMMARY

The effects of diagenesis on organic material of marine origin are compared to the effects on terrestrial organic material in four Australian exploration wells, namely Ashmore Reef No. 1, Iviri No. 1, Nautilus No. 1A, and Orokolo No. 1. N-alkanes derived from marine organic material have a distribution ranging from C_{16} to C_{25} with no odd predominance, whereas n-alkanes derived from a terrestrial source range up to C_{33}, and have a pronounced odd predominance towards the higher molecular weight range. With increasing diagenesis the hydrocarbons of marine origin show little change in distribution whereas the n-alkanes of terrestrial origin show a decrease in odd predominance and a change in distribution maximum to lower carbon number. Variations in the n-alkane distributions of the different samples are related to the type of source material, degree of diagenesis, and effects of clay catalysts. The source rock potential of sediments in the four exploration wells is assessed in these terms.

Sediments from the Nautilus and Orokolo wells contained n-alkanes with a pronounced odd-even predominance, indicating a low source-rock potential. Shales towards the base of the Ashmore Reef well showed evidence for the generation and migration of hydrocarbons. The Cretaceous Tubu shales in the Iviri well show evidence of good hydrocarbon potential.

INTRODUCTION

Modern theories of petroleum genesis require that petroleum hydrocarbons be generated in sediments at depth, under the influence of an increase in temperature, from the lipid components of organisms incorporated in the sediment during deposition (Cooper & Bray, 1963; Martin et al., 1963; Philippi, 1965; Louis & Tissot, 1967; Erdman, 1967; Albrecht & Ourisson, 1969). This process is reflected in a number of changes which the organic material undergoes with increasing depth of burial. These include an increase in the gross hydrocarbon content, an increase in the ratio of saturated hydrocarbons to total organic carbon and a diminution in the ratio of odd numbered carbon n-alkanes to even numbered carbon n-alkanes (Philippi, 1965; Louis & Tissot, 1967; Albrecht & Ourisson, 1969).

A feature of previous work is that these diagenetic studies have usually been made on sediments which had a predominance of odd-numbered carbon n-alkanes in the range C_{25} to C_{35}. Studies on the hydrocarbon distribution of modern organisms have shown that this odd predominance is characteristic of higher plants of terrestrial origin (Mazliak, 1969). Bacteria and algae generally have a smooth n-alkane distribution with a maximum in the range C_{17} to C_{20} and no odd predominance (Stransky et al., 1967; Oro et al., 1967; Clark & Blumer, 1967; Han et al., 1968; Winters et al., 1969), although certain algae do contain long chain odd-numbered carbon alkenes (Gelpi et al., 1970). Apparently, most diagenetic studies have been made on sediments which have contained some organic material of terrestrial origin. A possible exception is the work of Albrecht & Ourisson (1969). The unaltered sediments, at the top of their sedimentary sequence, had an even distribution of n-alkanes with a maximum at n-C_{17}. With increasing depth, longer chain alkanes appeared in the extracts to give a bimodal distribution with maxima at n-C_{17} and n-C_{27}. On chemical and microscopic evidence Douglas et al. (1971) have shown that where longer chain n-alkanes (C_{25}-C_{31}) represent a considerable proportion of the n-alkanes it is likely that they are derived from terrestrial plant material.

Although there is evidence that organic material of terrestrial origin may play a significant role in the genesis of petroleum, lower organisms are still thought to constitute the source material for many petroleums. There exists, therefore, an anomaly in that organic material derived from lower organisms is still widely regarded as the source of many petroleums, yet the effects of diagenesis on this material have not been clearly established. This study is aimed at assessing the diagenetic changes which may occur with burial of marine organic material, to compare them with the changes which have already been established for land plant material, and to assess the source rock prospects in the boreholes selected for study.

Marine detrital sediments frequently contain organic material derived from the land. Limestones were chosen for this study, since they usually contain little terrestrial organic material. Tertiary sequences only were considered in an attempt to ensure that the geological history of the sediment was as simple as possible. The Ashmore Reef No. 1 well, which was drilled in the Timor Sea, was selected since there was an adequate supply of cores through an almost completely limestone section. Two boreholes were selected, in which the section comprises both limestones and shales, to enable a comparison between the two possible source materials. These two wells were the Nautilus No. 1A well, in Victorian waters, and the Iviri No. 1 well, on land in Papua. A borehole was also selected in which shales formed the dominant lithology, namely the Orokolo No. 1 well in the Gulf of Papua.

EXPERIMENTAL

All solvents used in the analytical procedure were distilled through a Podbielniak fractionating column. The general procedures followed to avoid contamination during the course of the analytical procedures were essentially those outlined by Eglinton et al. (1966).

The samples were extracted for three days with benzene and methanol (1:1) in Soxhlet apparatus, and evaporation of the solvents yielded the crude extract as a brown gum. The crude extract was dissolved in a minimum amount of chloroform, and the asphaltenes were precipitated by the addition of a large excess of petroleum ether. The material, soluble in petroleum ether, was chromatographed on alumina (Grade 1), which was then eluted with successive volumes of petroleum ether, benzene, and methanol. Infra-red spectroscopy showed that the petroleum ether eluate comprised saturated hydrocarbons which on treatment with 5A molecular sieve (Union Carbide) yielded n-alkanes and branched and cyclic alkanes.

Gas chromatography of the n-alkanes was carried out using 20-foot x 0.040 inch I.D. stainless steel columns packed with 3% OV-1 on 100-120 mesh Gas Chrom Q (Applied Science Laboratories). The columns routinely had efficiencies of 10,000 theoretical plates. N-alkanes were identified by co-injection of authentic n-alkanes (Polyscience).

The ratio of odd to even-numbered carbon n-alkanes was determined for each n-alkane by the method of Scalan & Smith (1970). This ratio is termed the OEP value, and is determined by the following formula:-

$$OEP = \left(\frac{C_i + 6C_{i+2} + C_{i+4}}{4C_{i+1} + 4C_{i+3}} \right)^{(-1)^{i+1}}$$

C_i is the relative weight percent of a n-alkane containing i carbon atoms per molecule. The ratio incorporates the weight percent of five consecutive alkanes centred about the alkane containing i + 2 carbon atoms. These ratios are plotted against carbon number to give the OEP profile for each sample examined.

RESULTS

Ashmore Reef No. 1 Well

The Ashmore Reef No. 1 well was drilled on a seismically defined structure on the outer portion of the Sahul Shelf, immediately east of the Timor Trough (Lat. 12°10'49.7" S; Long. 123°05'10.6" E). The well penetrated a sequence ranging from Recent to Triassic in age to a depth of 12,843 feet (Fig. 1).

The Tertiary and Upper Mesozoic rocks comprise foraminiferal calcarenites and calcilutites interspersed with marls. The Lower Mesozoic sequence, for the greater part, comprises volcanic and clastic sediments. Palaeontological evidence suggests that the pelagic oozes of the Upper Cretaceous were deposited in a considerable body of water (Burmah Oil Co., 1968; Mollan et al., 1970). Lutitic sediments built up in Palaeocene-Eocene times, culminating in the deposition of extensive foraminiferal arenites and the initiation of a reef complex in the Ashmore area during upper Eocene time. These conditions persisted into the lower middle Miocene, when there was a break in sedimentation. During Quaternary time the reef developed its present form. Sixteen cores, comprising twelve limestones and four shales, were selected from the well. The depth, lithology, and stratigraphic position are given in Figure 1.

In Figures 2 and 3 the analytical results are summarized. The quantity of soluble organic material in the limestones is quite low, ranging from 20 to 140 ppm. There is a slight increase in total extract to a depth of 8,000 feet and a slight increase in the proportion of saturated hydrocarbons. The shales below 8,000 feet, however, show a decrease in both total extract and in the proportion of saturated hydrocarbons with depth. The n-alkane distributions of the limestones are very similar, showing a distribution maximum in the range n-C_{18} to n-C_{20} and only small amounts of higher n-alkanes. The OEP profiles show that there is little odd predominance and no significant variation with depth. A feature of some of the limestone samples is the slight predominance of the n-C_{22} alkane, which is shown as a minimum at n-C_{22} in the OEP profiles. The n-alkanes occurring in the shales below 8,000 feet show a wide distribution ranging up to n-C_{29}. The distribution is smooth and, in the sample at 9,741 feet reaches a maximum at n-C_{24}, whereas in the sample at 11,024 feet the distribution maximum is shifted to a lower carbon number.

Iviri No. 1 Well

The Iviri No. 1 well was drilled on a seismically defined structure in the Central Delta region of Papua (Lat. 07°35'54" S; Long. 144°46'30" E). The well penetrated a sequence ranging in age from Recent to Cretaceous to a depth of 12,015 feet (Fig. 4). The upper part of the sequence comprises freshwater muds and sands with some lignites and grades down into slightly calcareous marine shales and limestones of Lower Miocene and Eocene age forming the base of the Tertiary sequence. Lower Cretaceous sediments comprising black shales and sandstones of marine origin underlie the Tertiary sequence.

Throughout the Mesozoic and Tertiary times the Delta Region of Papua, in which Iviri is situated, accumulated sediments in a period of quiescent subsidence. The Lower Cretaceous shales encountered in this well were massive, black homogeneous shales of marine origin (Australasian Petroleum Co., 1965). There is a hiatus between the topmost Cretaceous and the Tertiary, but there is no structural discordance. Limestone sedimentation, commencing in the Eocene, persisted through to the lower Miocene, but there is no Oligocene present in the Iviri well. The limestone phase was superseded by marl and mudstone sediments of middle and upper Eocene Age. The Pliocene comprised a thick estuarine series made up of sandstone, mudstone and coal and is conformable with the marine shales of Miocene Age. Nineteen cores comprising twelve shales and seven limestones were examined. Their depth, stratigraphic position, and lithology are given in Figure 4.

The results of the geochemical work are summarized in figures 5 and 6. The plot of total organic extract with depth shows a division into two categories; the shales containing 300-3,000 ppm, and the limestones containing 20-200 ppm. There is a slight increase in total extract from the shales down to the top of the limestone sequence and a marked increase in the total extract obtained from the Tubu Shales at the bottom of the hole. The plot of the total extract obtained from the limestones parallels the trend shown by the shales in showing an increase in total extract with depth. The proportion of saturated hydrocarbons to total extract also increases with depth, though there appears to be a difference in the rate of this increase between the shales and the limestones.

The n-alkanes of the samples taken from above the limestone sequence show a marked bimodal distribution. There are two maxima; one in the region C_{15}-C_{20} and the other in the region C_{25}-C_{31} (Fig. 6). The OEP profiles show that the lower carbon number n-alkanes have no odd predominance, but with increasing carbon number the OEP values increase, demonstrating an odd predominance in the higher molecular weight range. Some of the OEP profiles have a minimum at C_{22}, indicating a slight predominance of the n-C_{22} alkane.

The n-alkane distributions of the limestones are similar to those obtained for the Ashmore Reef samples in having a maximum at C_{18}-C_{20} and diminishing concentration with increasing carbon number. The OEP profiles show no odd predominance.

The n-alkanes from the samples of Cretaceous age show an even distribution ranging from C_{15}-C_{32} with a single maximum between C_{20} and C_{25}. At a depth of 10,437 feet there is a slight odd predominance at the distribution maximum, but this disappears in the two deeper samples as the distribution maximum shifts to lower carbon number.

The n-C_{29} OEP values of samples in the upper part of the sequence are higher than those at the base of the hole. The values for n-C_{25} are lower than those for n-C_{29}, but again diminish with depth. There is no change in the n-C_{21} OEP values with depth.

Nautilus No. 1A Well

The Nautilus No. 1A well was drilled in Victorian waters 35 miles off Warrnambool, Victoria and 28 miles from the nearest land (Lat. 38°58'40.97" S; Long. 142°32'45.74" E). The well penetrated rocks of Miocene, Oligocene and Upper Cretaceous age to a depth of 6,597 feet (Fig. 7). The uppermost 1,000 feet of the sequence comprises calcarenite which passes down into an interbedded sequence of siltstone, marl, limestone, and shale for the greater part of the Tertiary succession. A sand occurs at the base of the Tertiary, and overlies black shale of upper Cretaceous age.

The Upper Cretaceous succession in the Otway Basin was deposited during a major marine transgression. The only representative of the Cretaceous in the Nautilus No. 1 well is the carbonaceous sediments of the Belfast Formation (Esso Exploration, 1968).

The Palaeocene and upper Eocene sediments found elsewhere in the Otway Basin are considered to be absent from the Nautilus location (Esso Exploration 1968). From upper Eocene to Pliocene a marine transgressive phase deposited a thick marl and limestone which represent the bulk of the sediments penetrated in this well. Ten cores were examined from the sequence, and the depth, stratigraphic position, and lithology of each are given in Figure 7.

The results obtained for this borehole are given in Figures 8 and 9. The total organic extracts range from 20 to 400 ppm with the exception of one reaching 1,661 ppm. The ratio of saturated hydrocarbons to total extract is essentially constant throughout the well.

The n-alkane distributions of the three samples taken from above 4,200 feet are smooth, with a maximum between C_{18} and C_{22}. The OEP profiles show a slight odd predominance in the higher molecular weight range for one of these samples, and two of these samples show a slight predominance of the n-C_{22} alkane.

In the lower part of the borehole the n-alkane distributions show a maximum in the higher molecular weight range, with a pronounced odd predominance. The sample at a depth of 6,102 feet shows an unusual n-alkane distribution in having a maximum at n-C_{24}. The change in n-alkane distribution with depth is reflected in an increase in OEP value of n-C_{25} and n-C_{29}. Again, the n-C_{21} value shows little variation.

Orokolo No. 1 Well

The Orokolo No. 1 well is 150 miles northwest of Port Moresby and 9 miles offshore in the Gulf of Papua (Lat. 8°04'24.8" S; Long. 145°23.5'56.9" E). The well penetrated Pleistocene and Pliocene clastic sediments to a depth of 11,999 feet (Fig. 10).

The Orokolo well is situated in the western portion of the Aure Trough in the central part of the Mesozoic/Tertiary Papuan Basin (Phillips Australian Oil Co., 1968). The trough was initiated in Oligocene times and continued to receive sediment throughout Miocene and Pliocene times to a thickness of 25,000 feet. Eight cores were selected for study (Fig. 10).

Figure 11 shows that the total extracts vary from 100 to 600 ppm with no regular depth variation. The ratio of saturated hydrocarbons to total organic extract shows a slight increase with depth. The *n*-alkane distributions all show a maximum in the range C_{25} to C_{30} with a pronounced odd predominance (Fig. 12). The *n*-C_{21} OEP value remains constant at about 1 throughout the well, but the *n*-C_{25} and *n*-C_{29} OEP values show no regular variation.

DISCUSSION

The distribution of *n*-alkanes obtained from the limestones in the Ashmore Reef No. 1 well are virtually the same, showing no variation with depth (Fig. 3). The relatively low proportion of higher molecular weight *n*-alkanes would seem to indicate that there was no derivation from the waxes of terrestrial plants, and that the organic material was primarily derived from microorganisms. The other possibility is that the organic material derived from these limestones has reached a mature state in resembling the distribution found in crude oils; the absence of n-alkanes below C_{16} can be explained by loss, due to evaporation during storage and isolation procedures. Limestones occurring towards the top of the sequence in the Nautilus well (Fig. 9) have similar distributions to those from the Ashmore Reef well. They overlie shales in which the *n*-alkanes have a pronounced odd predominance, indicative of an immature state for this organic material. Clearly, the limestones overlying these shales could not have undergone any extensive thermal alteration; hence, it would appear that the type of distribution displayed by the limestones is indicative of material derived from microorganisms.

Albrecht & Ourisson (1969) have suggested that longer chain *n*-alkanes are generated from the parent kerogen during the diagenesis of marine organic material. In the Iviri well there is clear evidence that diagenetic changes are taking place, since there is a marked decrease in the odd to even predominance in the *n*-alkane distributions of the shales (Fig. 6) and an increase in the proportion of saturated hydrocarbons in the total extract with depth (Fig. 5). The limestone sequence in the Iviri well falls in the region where marked diagenetic changes appear to be taking place in the shales (Figs. 5 and 6), yet there is no evidence for the production of longer chain *n*-alkanes.

Furthermore, in the Ashmore Reef well a limestone at the base of the borehole shows no evidence for the generation of longer chain *n*-alkanes (Fig. 3). Fats of microorganisms, in general, have a limited chain length of sixteen to twenty carbon atoms; longer chain lengths are produced by certain microorganisms, but are only minor constituents of the total lipid fraction (Kates, 1964; Miller, 1962). Hence, if long chain alkanes are to be produced in the diagenesis of material derived from microorganisms significant chain lengthening processes must occur in the sediment. Experimental studies on the decarboxylation of fatty acids (Jurg & Eisma, 1967) have shown that alkanes with longer chains than the original acid can be produced, but their contribution to the total hydrocarbon product is quite low.

The likely diagenetic pathway for marine organic material in regard to n-alkane formation would seem to be the generation of n-alkanes in the range C_{16} to C_{25} with no significant change in distribution. With increasing diagenesis there would presumably be a shift to lower carbon number as indicated in other studies (Albrecht & Ourisson, 1969).

The rate of hydrocarbon generation and breakdown appears to be greater in the shales than in the limestones. In the Iviri well the plot of the ratio of saturated hydrocarbons to total extract (Fig. 5) shows that the rate of production of saturated hydrocarbons is less in the limestones than in the shales. This is presumably due to the relative lack of clay catalysts in the limestones. In the Ashmore Reef well the shale samples towards the base of the borehole show a diminution in both total extract and in the proportion of saturated hydrocarbons with depth (Fig. 2). This is accompanied by a shift in the n-alkane distribution maximum to lower carbon numbers. Albrecht & Ourisson (1969) have attributed similar changes at depth in a sedimentary sequence in the Cameroons to catalytic cracking and migration of the hydrocarbons. However, a limestone at the base of the Ashmore Reef borehole shows no evidence of cracking and migration of hydrocarbons (Figs. 2 and 3), and has essentially the same n-alkane distribution as limestones higher in the borehole. Hunt (1967) has suggested that shales may have a distinct advantage over carbonates in generating hydrocarbons by catalytic processes, and our results support this point of view.

It is apparent from the results from the Nautilus well that environmental or source effects may significantly change n-alkane distributions and may confuse diagenetic effects. The increase in odd predominance (Fig. 9) with depth, which is the complete reverse of the normal trend, is attributed to a change in environment from open-sea conditions in the upper part of the sequence to more near-shore conditions in the lower part of the sequence. Corresponding to this environmental change there is an increase in the proportion of terrestrial organic material in the sediments with increasing depth, which in the absence of any marked geothermal gradient results in an increase in the odd predominance. Conversely, the addition of material derived from microorganisms to terrestrial organic material will decrease the odd predominance of land-derived alkanes where the distributions overlap. This appears to have happened in the upper part of the Iviri well, where the n-alkanes contain both lower n-alkanes with a smooth distribution and higher n-alkanes with a pronounced odd predominance.

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A slight predominance of the n-C_{22} alkane occurs in many of the samples, and has been reported previously by Schenk (1968). No rational explanation has been advanced for this observation.

SOURCE ROCK POTENTIAL

The source rock potential of a sedimentary sequence can be assessed in terms of two criteria: quantitative assessment of potential source rocks and state of maturation of organic material.

In the Ashmore Reef No. 1 well the soluble organic content of the sediments is extremely low, ranging from 20 to 200 ppm. There is a slight increase in the proportion of saturated hydrocarbons in the limestones with depth, but significant amounts of hydrocarbons have not been generated. Although the hydrocarbon content of the shales in the lower part of the sequence is low, maturation has proceeded to the extent that cracking and possible migration have taken place. Thus, although the source rock potential is low in the quantitative sense, the maturation requirement for the generation of hydrocarbons is established.

The Iviri well presents considerable evidence of good source potential. The extractable organic matter is up to 3,000 ppm, and the ratio of saturated hydrocarbons to total organic extract shows a considerable increase in the Cretaceous Tubu shales, indicating conditions favourable for hydrocarbon generation. The OEP values of the n-alkanes of longer chain length diminish with depth, and there is a shift in the distribution maximum to lower carbon numbers at total depth. These indications show that hydrocarbons of suitable type are generated at depth in this sequence, and point to a good source potential for the Cretaceous sediments at this locality.

The Nautilus well, however, shows very little petroleum potential. Although the sediments are relatively rich in organic material, it is immature. The argillaceous limestones sampled at the top of the well have n-alkane distributions typical of marine organic material. At greater depths, the n-alkanes display a pronounced odd predominance. This is indicative of very little diagenetic transformation; in fact, the bottom-hole temperature is less than 60°C. Brooks (1970) has suggested that the Otway Basin is not very prospective for petroleum compared with the Gippsland Basin, because of the lower geothermal gradient in the Otway Basin. The results from this well tend to confirm this point of view.

Although the bottom-hole temperature in the Orokolo No. 1 well is quite high, at 140°C, and compares favourably with the Iviri well, the high odd predominance of the n-alkanes (Fig. 12) is maintained throughout the sequence, indicating that little hydrocarbon generation has occurred.

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CONCLUSIONS

1. Although the evidence is incomplete, diagenesis of organic material derived from microorganisms results in the production of n-alkanes, largely in the range C_{16}-C_{22}. With increasing diagenesis a shift in hydrocarbon distribution to lower carbon numbers occurs in the manner shown by other workers.

2. Organic material in limestones generates hydrocarbons at a lower rate than that in shales, presumably because of the reduced catalytic activity in the absence of clay minerals.

3. Environmental or source variations may have a profound effect on the n-alkane distributions of many sediments.

4. Although rich source beds are lacking in the Ashmore Reef No. 1 well the organic material in the shales in the depth interval 10,000 to 12,000 feet has reached an advanced stage of hydrocarbon generation.

5. In the Iviri No. 1 well the Cretaceous Tubu Shale must be regarded as prospective source sediments.

6. The geothermal gradient in the Nautilus No. 1A well is too low to allow the generation of oil at the depth drilled.

7. In spite of the relatively high geothermal gradient in the Orokolo No. 1 well the n-alkane distributions indicate that petroleum generation is not taking place in the sedimentary sequence at this locality, presumably because the sediments have not been maintained at an elevated temperature for a sufficient length of time.

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FIG I ASMORE REEF NO I WELL - STRATIGRAPHY AND SAMPLES

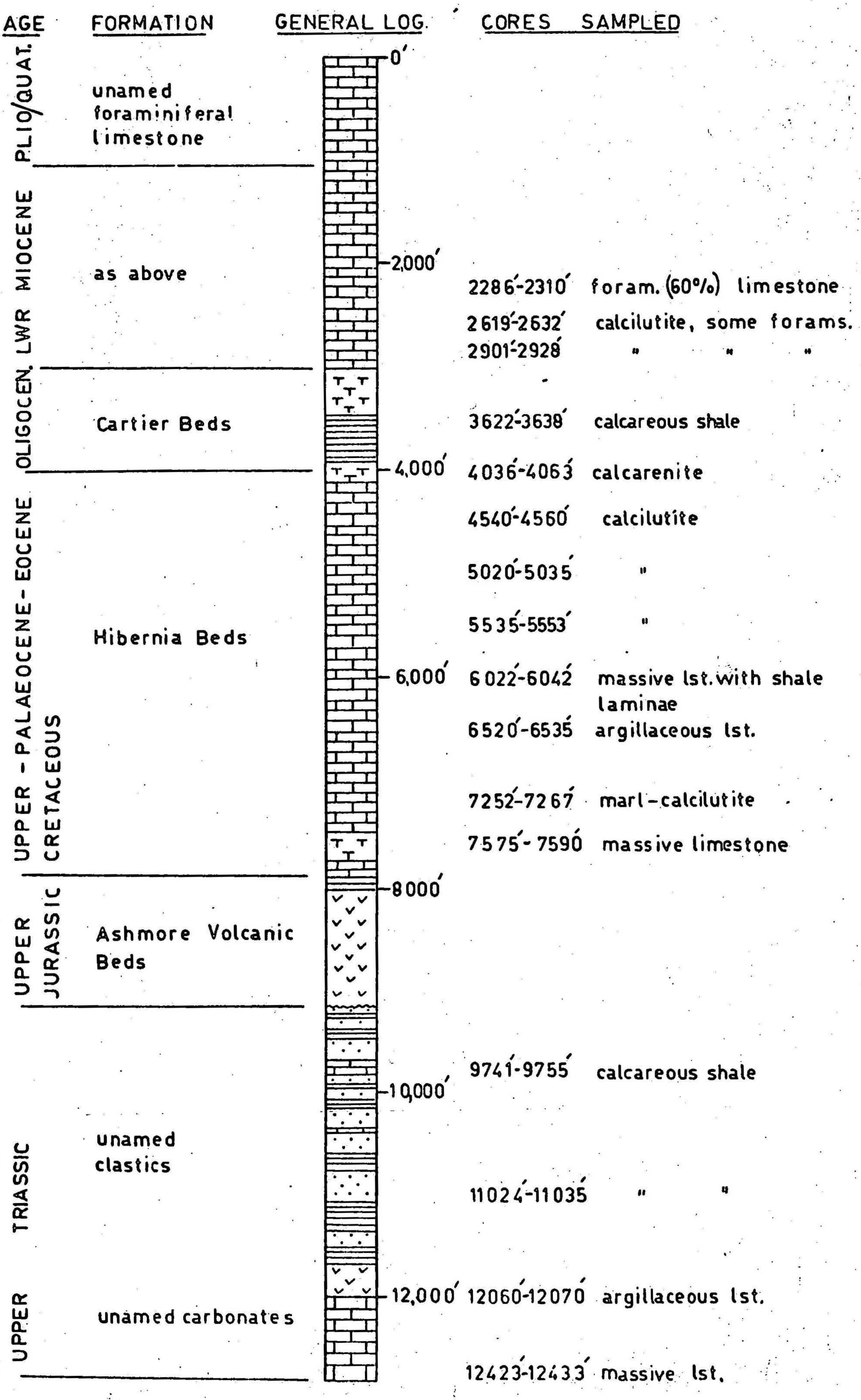


FIG 2 ASHMORE REEF NO I WELL TOTAL EXTRACT AND PROPORTION OF SATURATED HYDROCARBON

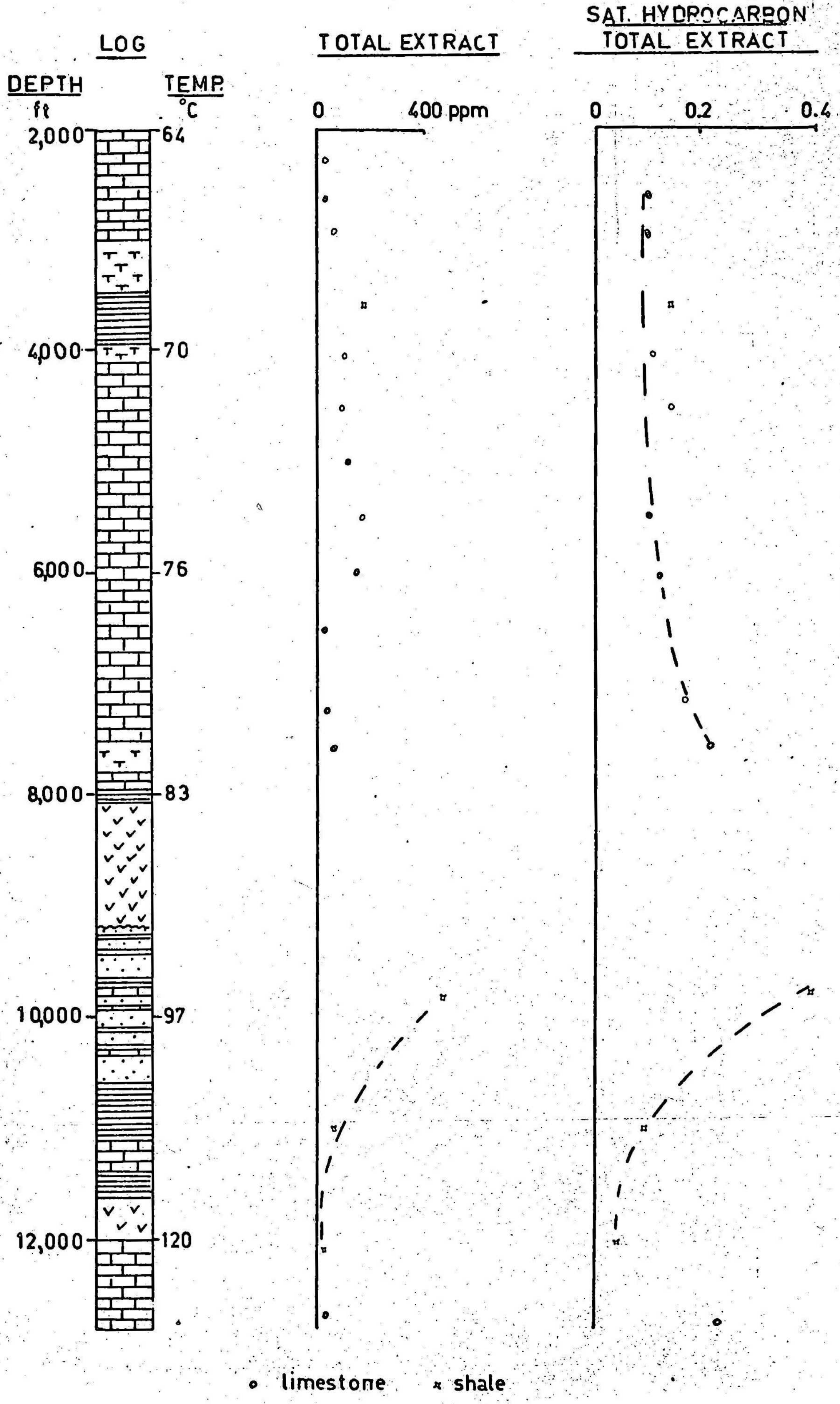


FIG 3 ASHMORE REEF NO I WELL – N ALKANES

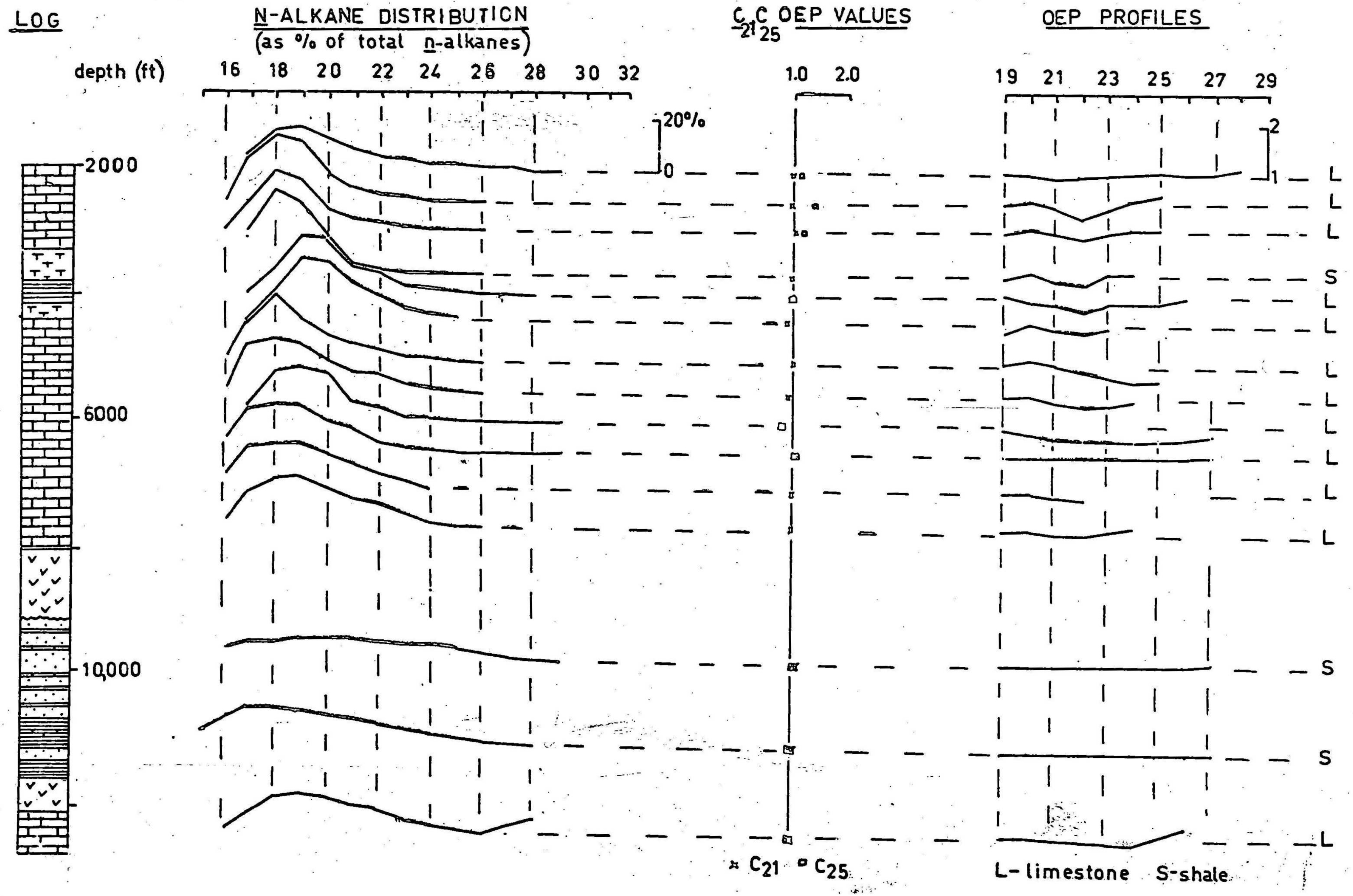


FIG 4 IVIRI NO1 WELL STRATIGRAPHY AND SAMPLES

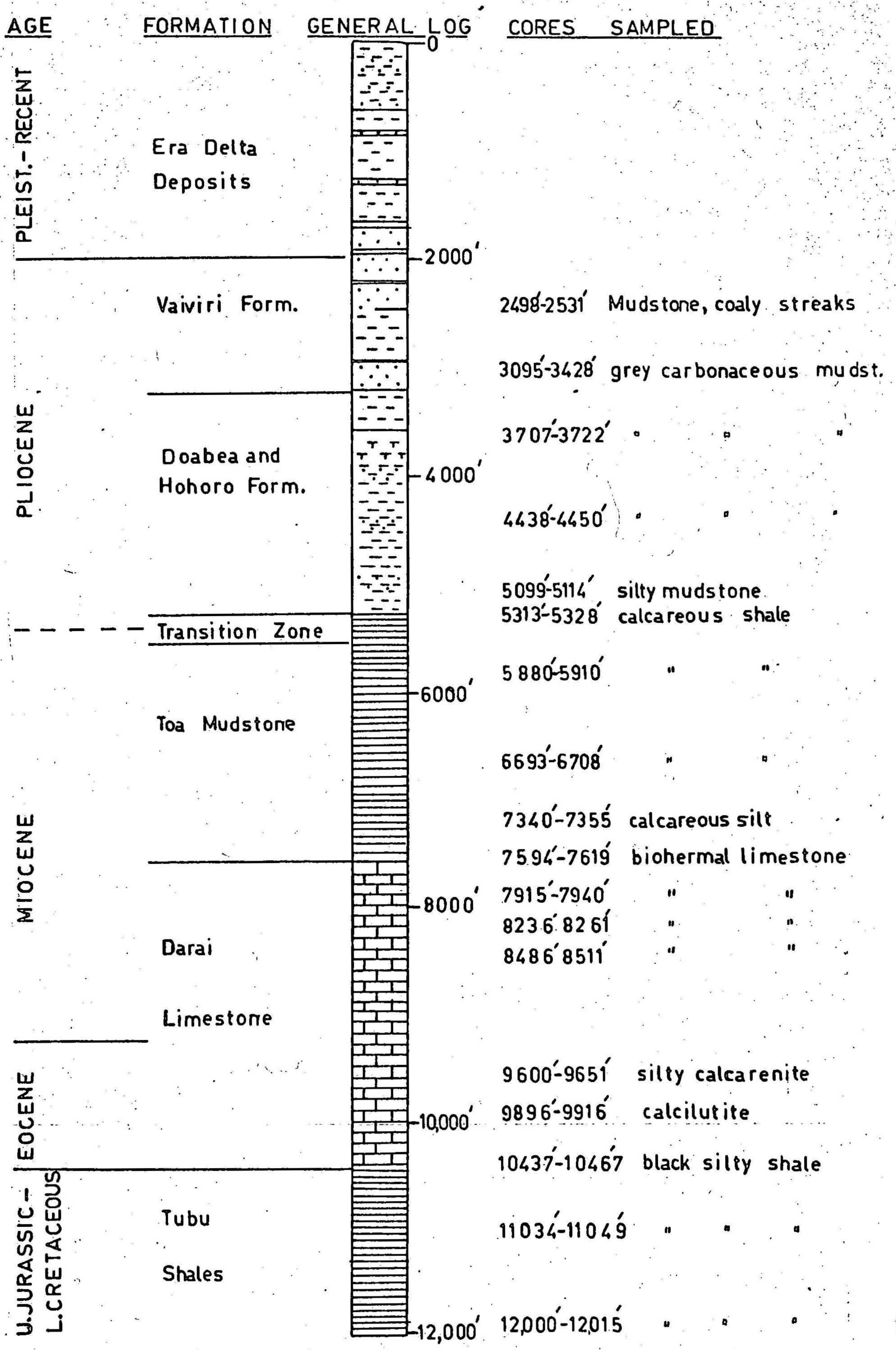


FIG 5 IVIRI NO I WELL TOTAL EXTRACT AND PROPORTION OF SATURATED HYDROCARBON

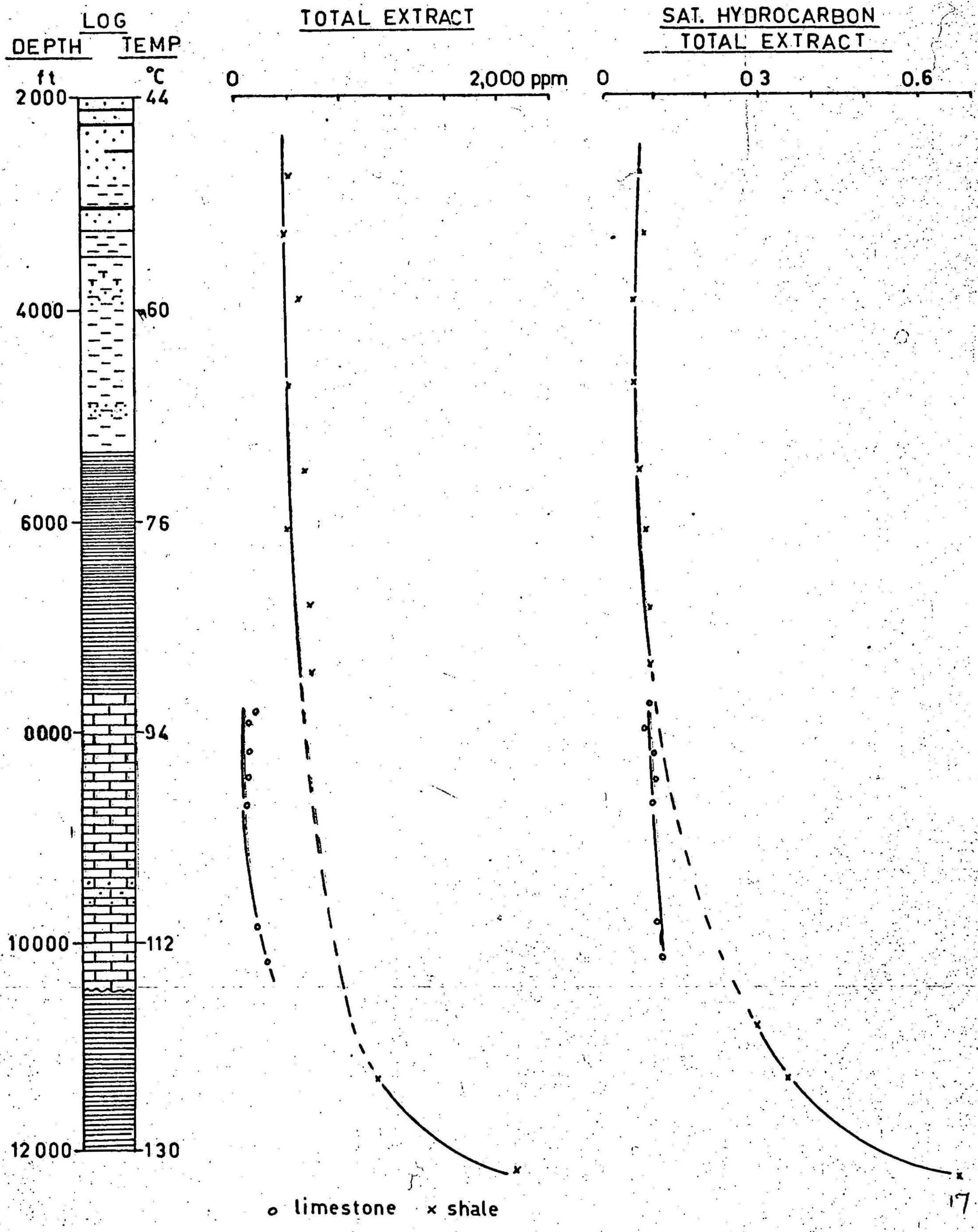


FIG 6 IVIRI NO I WELL. — N-ALKANES

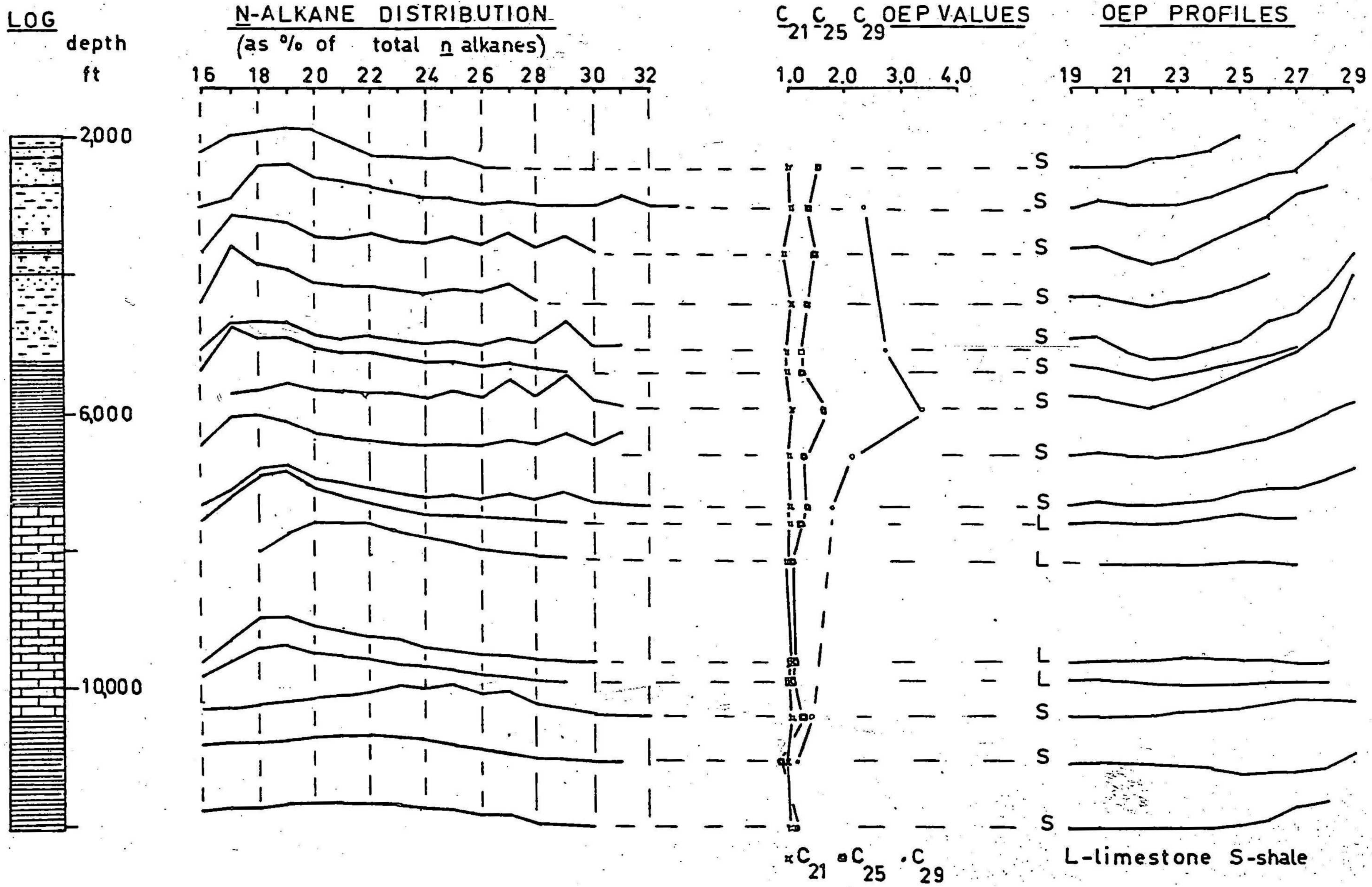
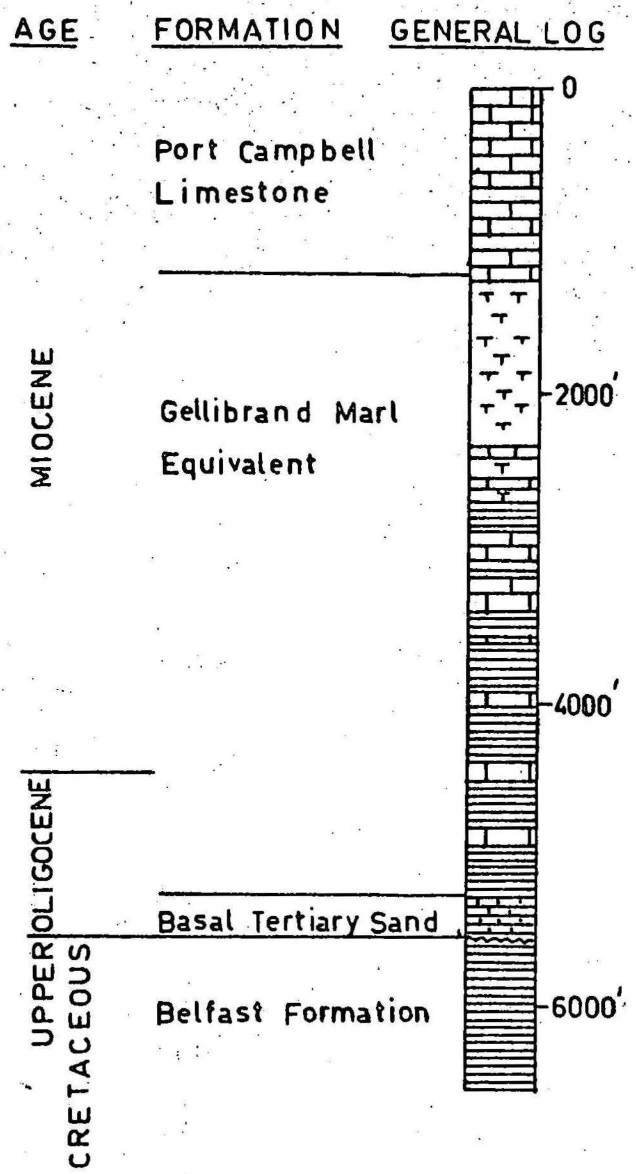


FIG 7 NAUTILUS NO A 1 WELL STRATIGRAPHY AND SAMPLES



CORES SAMPLED	
2772-2773'	argillaceous limestone
3122-3128'	calcareous shale
3656-3672'	argillaceous limestone
4133-4149'	" "
4640-4670'	" "
5175-5205'	calcareous shale
5674-5678'	shaley siltstone
6102 6117'	silty shale
6572 6597'	shale

FIG.8 NAUTILUS A 1 WELL TOTAL EXTRACT AND PROPORTION OF SATURATED HYDROCARBONS

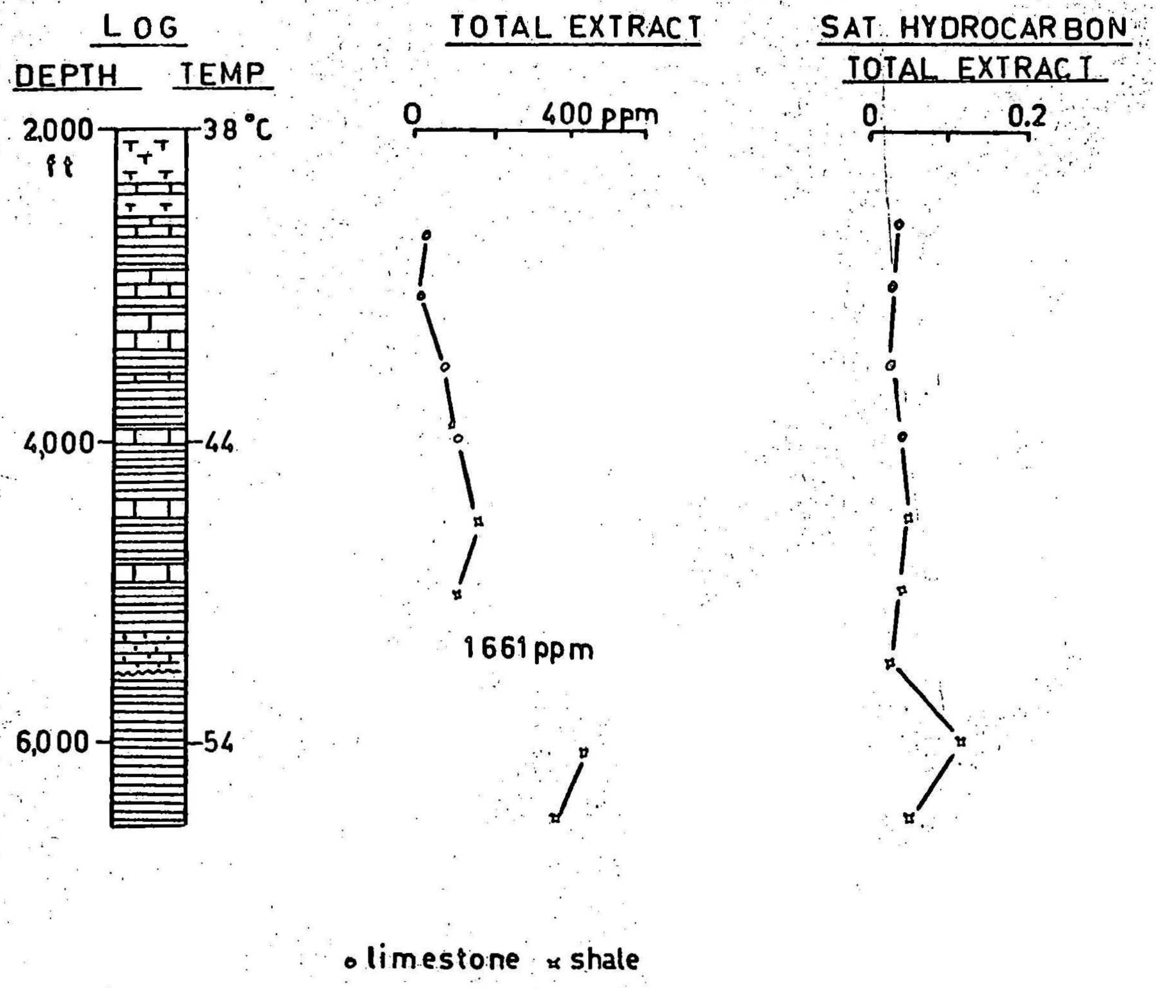


FIG 9 NAUTILUS NO 1 WELL — N-ALKANES

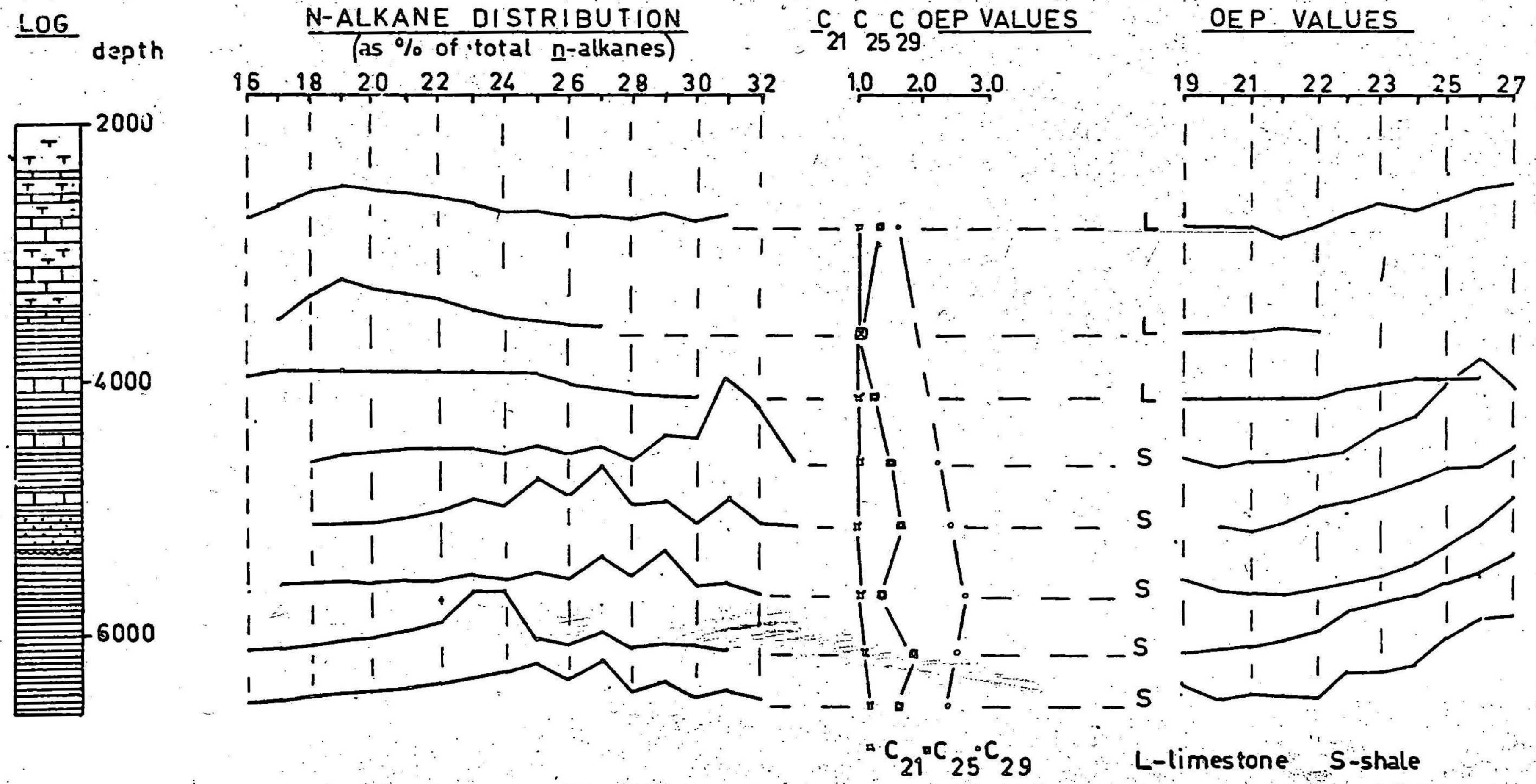


FIG10 OROKOLO NO I WELL STRATIGRAPHY AND SAMPLES

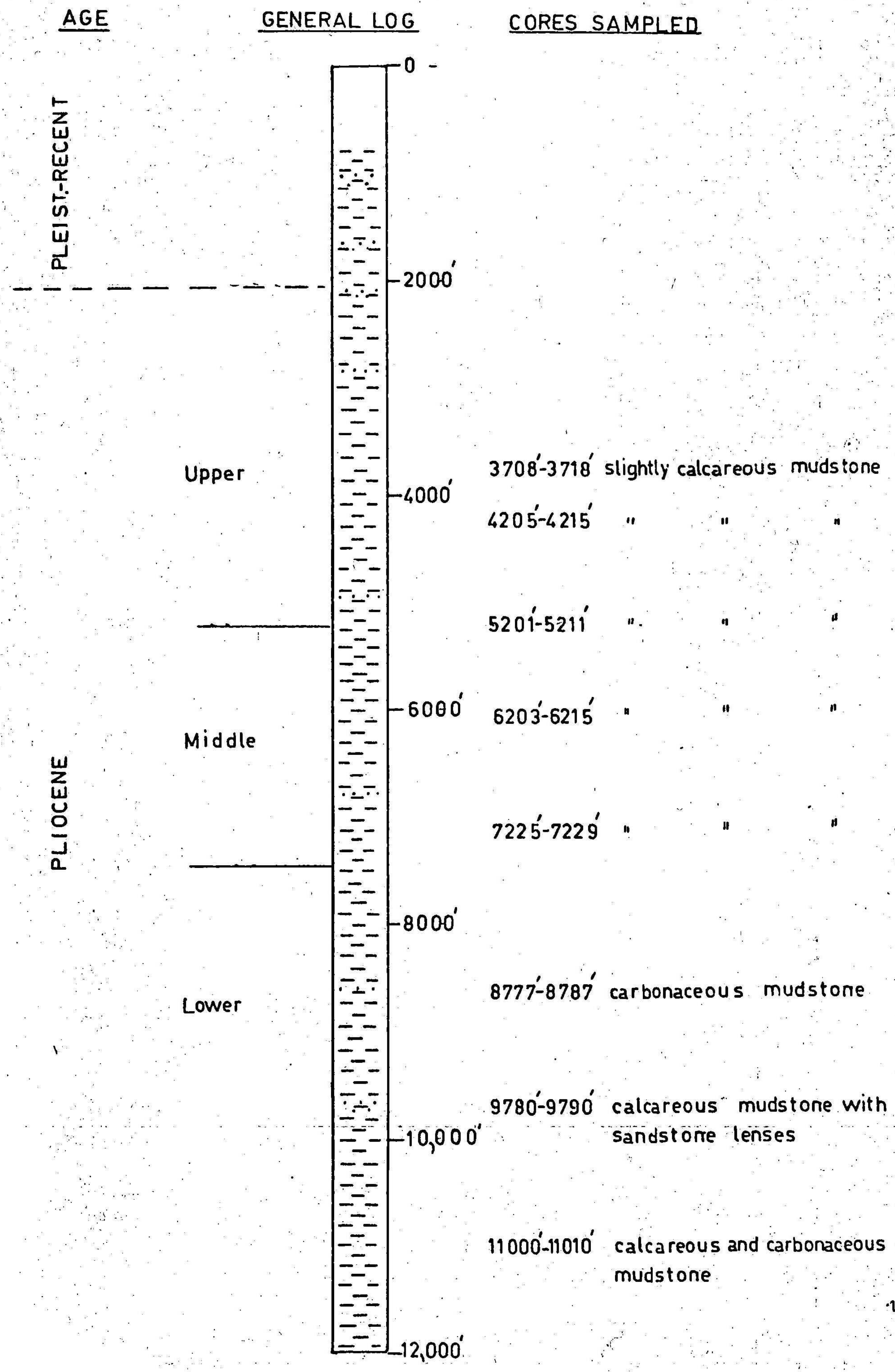


FIG 11 OROKOLO NO 1 WELL TOTAL EXTRACT AND PROPORTION OF SATURATED HYDROCARBONS

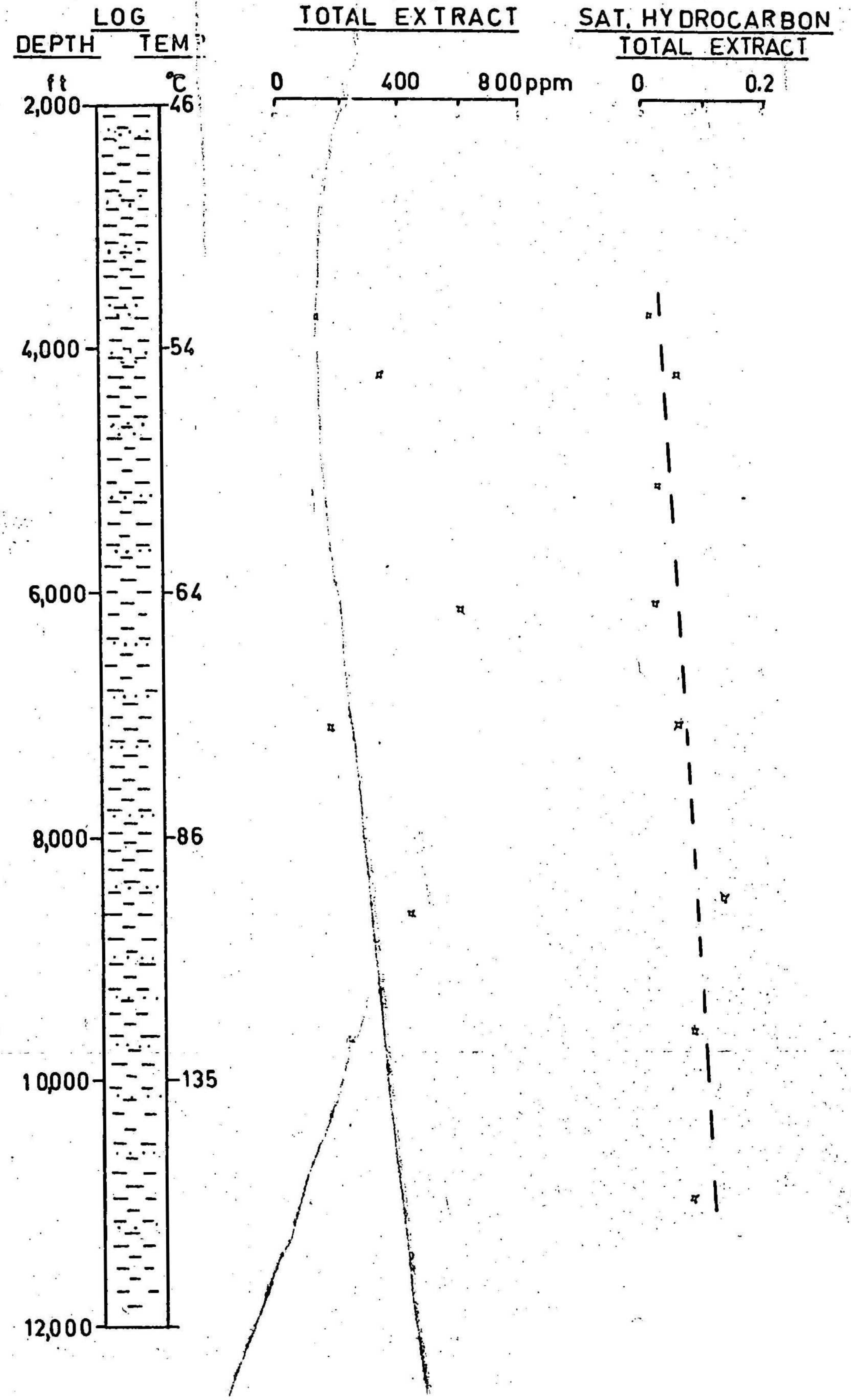


FIG 12 OROKOLO NO I WELL — N-ALKANES