

Report 197

AN INTEGRATED SCHEME FOR THE LABORATORY ANALYSIS
OF OIL, NATURAL GAS, AND PETROLEUM SOURCE-ROCKS.

by

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SUMMARY

In recent years the geochemical study of crude oils, natural gases, and organic matter dispersed in sedimentary rocks has emerged as an important tool in petroleum exploration. The BMR Petroleum Technology Laboratory has developed an integrated scheme for the geochemical evaluation of reservoir hydrocarbons (oil and gas), and potential source-rocks encountered during exploratory drilling in Australia.

The analytical procedures involved are relatively simple in order to permit rapid processing of the samples:

- Oil - measurement of API gravity;
 determination of sulphur content;
 fractional distillation and calculation of USBM
 correlation index for each fraction;
 precipitation of asphaltenes from $>250^{\circ}\text{C}$ fractions;
 liquid chromatography of asphaltene-free material
 on alumina to obtain saturated hydrocarbons
 (alkanes), aromatic hydrocarbons, and compounds
 containing O, N, and S;
 capillary gas chromatography of C_{15+} alkanes.
- Gas - gas chromatography to determine identity and
 concentration of individual components.
- Source-rock - determination of organic carbon content;
 Soxhlet extraction of soluble organic matter with
 benzene/methanol;
 liquid chromatography of extract on alumina (as
 for oil);
 capillary gas chromatography of alkanes;
 isolation of insoluble organic matter (kerogen) by
 digestion of rock in HCl/HF ;
 X-ray diffraction analysis of kerogen;
 elemental (C, H, N, S) and ash micro-analysis of
 kerogen.

Specific examples of oil, gas, and source-rock analyses are presented to illustrate how such geochemical data can be used to indicate:

1. the gross composition and quality of the oil;
2. any alteration (e.g. thermal maturation, biodegradation, water-washing) of the oil after its accumulation in the reservoir;

3. correlation between oils of the same family;
4. the marine or non-marine nature of the precursor organic matter in the source sediments;
5. distinction between oil-prone and gas-prone source-rocks.

The value of petroleum and source-rock geochemistry lies in its ability to predict at an early stage in the exploration of a basin both the type of hydrocarbons present (whether oil, gas-condensate, or dry gas) and their likely distribution within the basin.

INTRODUCTION

Until quite recently petroleum exploration was essentially a matter of delineating a promising subsurface structure by geological mapping and geophysical (seismic) techniques, and then drilling it to ascertain whether or not it contained hydrocarbons. The emphasis was on the trap or 'container' rather than on its 'contents' (Welte, 1972). However, no matter how large or porous the trap, it will not contain oil or gas unless there are appropriate source-rocks (i.e. organic-rich shale, siltstone, or carbonate) in the vicinity.

During the 1960s and early 1970s major advances were made in understanding the processes by which oil and gas form from biogenic organic matter buried in sediments, under the influence of increased temperature and pressure at depth (e.g. Cooper & Bray, 1963; Martin et al., 1963; Philippi, 1965; Louis & Tissot, 1967; Jurg & Eisma, 1967; Erdman, 1967; Albrecht & Ourisson, 1969; Staplin, 1969; Brooks, 1970; Powell, 1970; Tissot et al., 1971; Powell & McKirdy, 1973a).

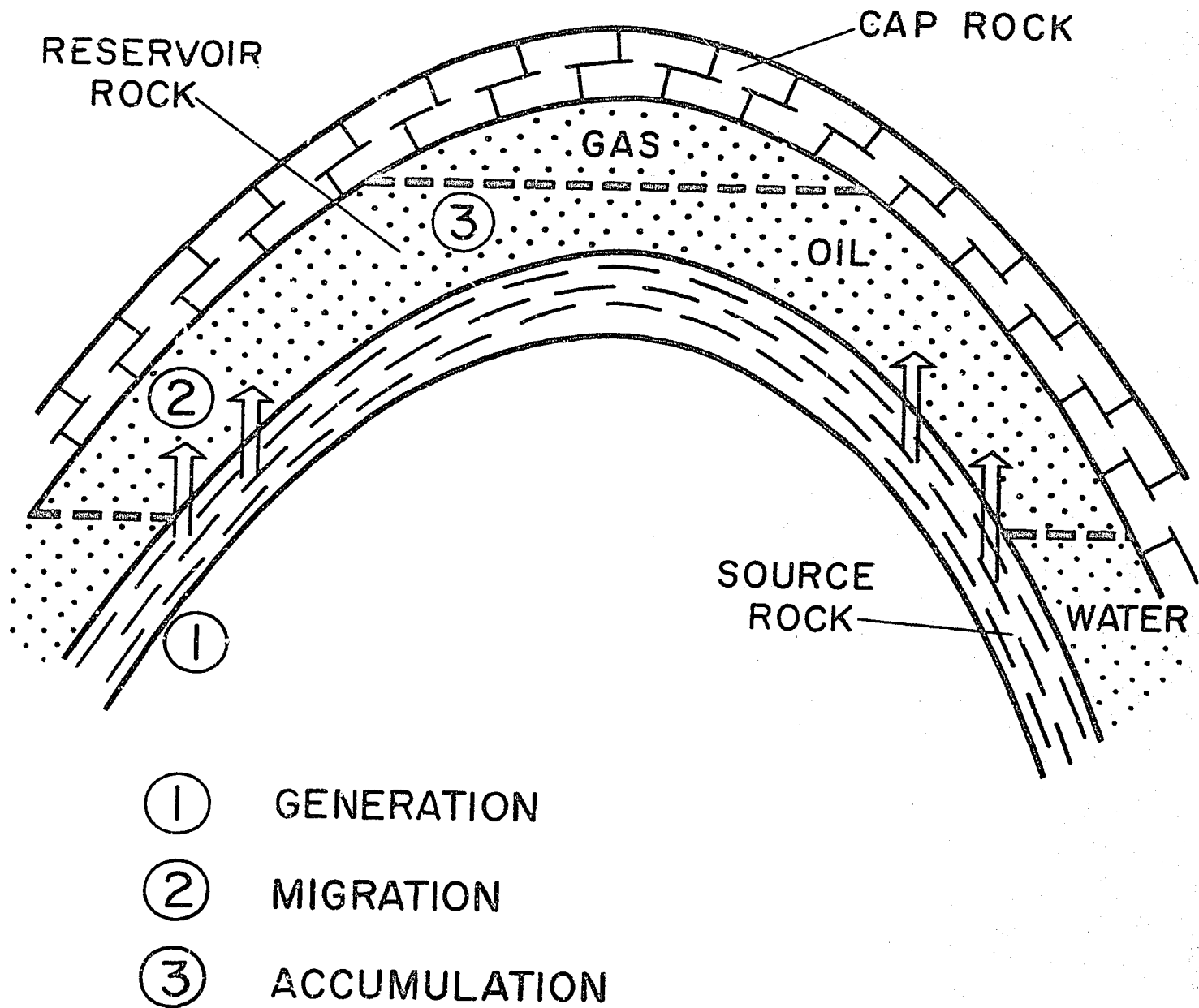
There are three important stages in the development of a commercial petroleum deposit (shown diagrammatically in Fig.1)

- (1) generation of petroleum in a source-rock;
- (2) migration of petroleum from the source-rock; and
- (3) accumulation of petroleum in a reservoir or trap

Geochemical data on oil or gas shows and organic-rich sediments encountered early in the exploration of a new area can be of significant help in assessing the probability that petroleum hydrocarbons have been generated and are available for accumulation in traps (Bailey et al., 1974; Powell, 1975).

Since 1970 a small research group in the BMR Petroleum Technology Laboratory has been engaged in the geochemical study of Australian oil and gas occurrences, and their associated source-rocks. This work has involved the analysis of more than 200 natural gases; 90 crude oils, bitumens, and condensates; and nearly 400 rock samples (Powell and McKirdy, 1971, 1972a, b, 1973a, b, c, d, 1975; Powell et al., 1974; Kurylowicz et al., 1975; McKirdy & Horvath, 1976; Powell, 1975; Powell et al., 1975).

One of the results of these investigations has been the development of an integrated scheme for the rapid analysis and evaluation of reservoir hydrocarbons and potential source-rocks found during exploratory drilling. The aim of this paper is to outline the scheme and to demonstrate how the geochemical data it provides may be used to assist the search for more oil and gas.



M (PII)20

Fig1. Diagrammatic representation of stages in the formation of a petroleum deposit.

OIL ANALYSIS

Crude oil is a complex mixture of hydrocarbons and other compounds. Although its chemistry is still inadequately understood, it is now possible to interpret the gross composition of a crude oil in terms of the geological processes which control its generation in a source-rock, its migration, and its accumulation in a reservoir (Evans et al., 1971; Powell & McKirdy, 1975).

Figure 2 illustrates the major factors that may influence the composition of a crude oil:

- (1) source-rock (in particular, the type of organic matter it contains, its mineralogy, and its environment of deposition)
- (2) maturation (i.e. thermal alteration)
- (3) action of natural gases (e.g. deasphalting)
- (4) action of meteoric waters (e.g. water washing, biodegradation)

Geochemical analysis is capable of assessing the relative importance of each factor in the genesis of a given oil.

The BMR procedure followed in the analysis of oils, condensates (i.e. light oils), and bitumens is outlined in Figure 3. The API gravity of the oil is measured with a special hydrometer according to method IP 160/68. Its pour point is measured by the standard method IP 15/67. The sulphur content of the oil is determined by the CSIRO Australian Microanalytical Service. The oil is distilled (method IP 24/55) and fractions are collected at intervals of 50°C. The distillation is stopped at 340°C, or when 90 percent of the oil has distilled. The specific gravity and the average boiling point of each fraction are used to read its so-called 'correlation index' from a set of standard curves prepared by the U.S. Bureau of Mines (Smith, 1964). Distillation fractions boiling above 250°C are combined for further analysis.

An aliquot of the combined high-boiling fractions is refluxed in petroleum ether, cooled, and filtered to remove the insoluble asphaltene (method IP 143/57).

Liquid chromatography of the asphaltene-free material on a column of Grade I neutral alumina, by eluting with petroleum ether, benzene, and methanol, effectively separates

- (a) saturated hydrocarbons (alkanes);
- (b) aromatic hydrocarbons; and
- (c) compounds containing O, N, and S.

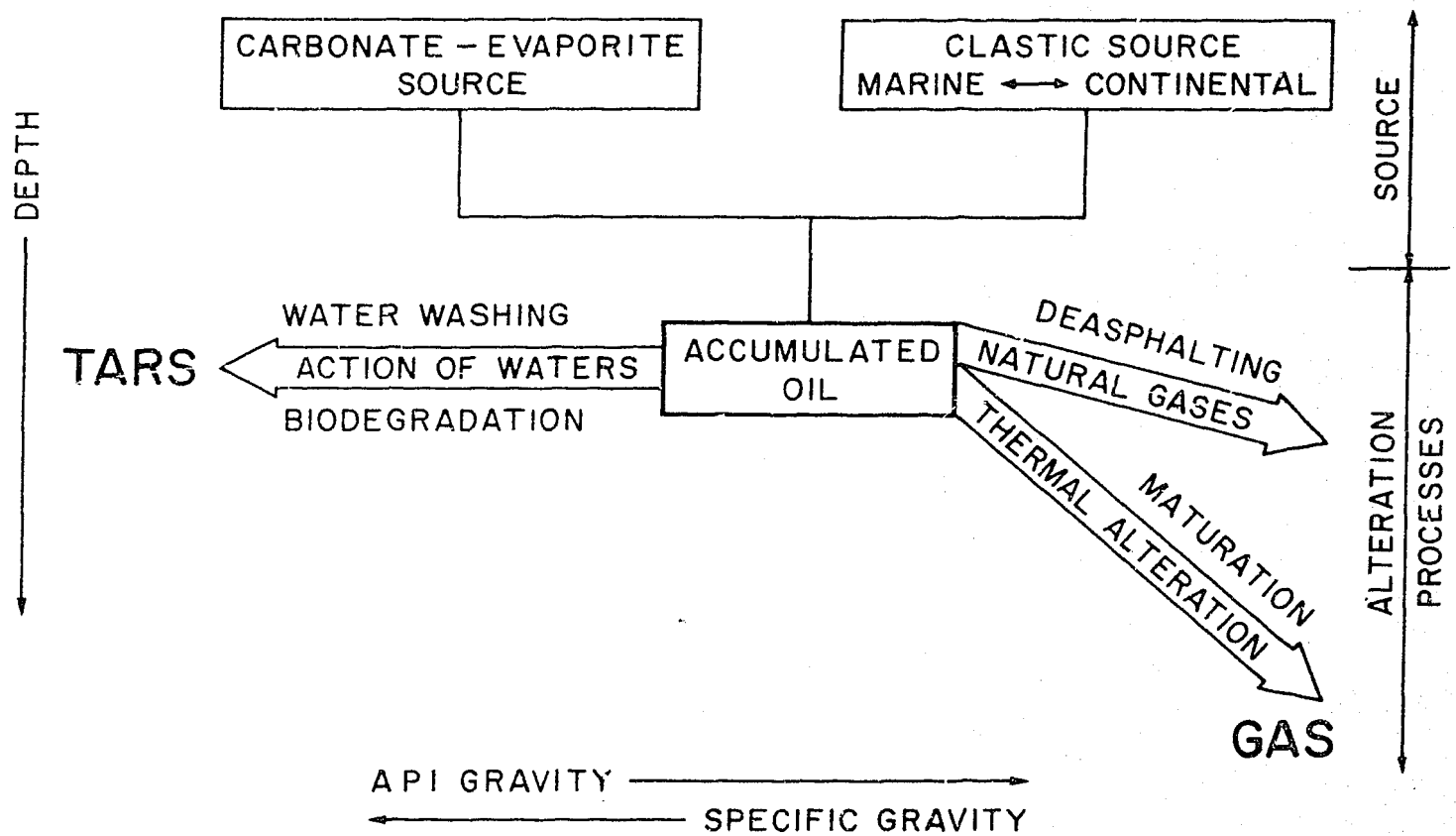
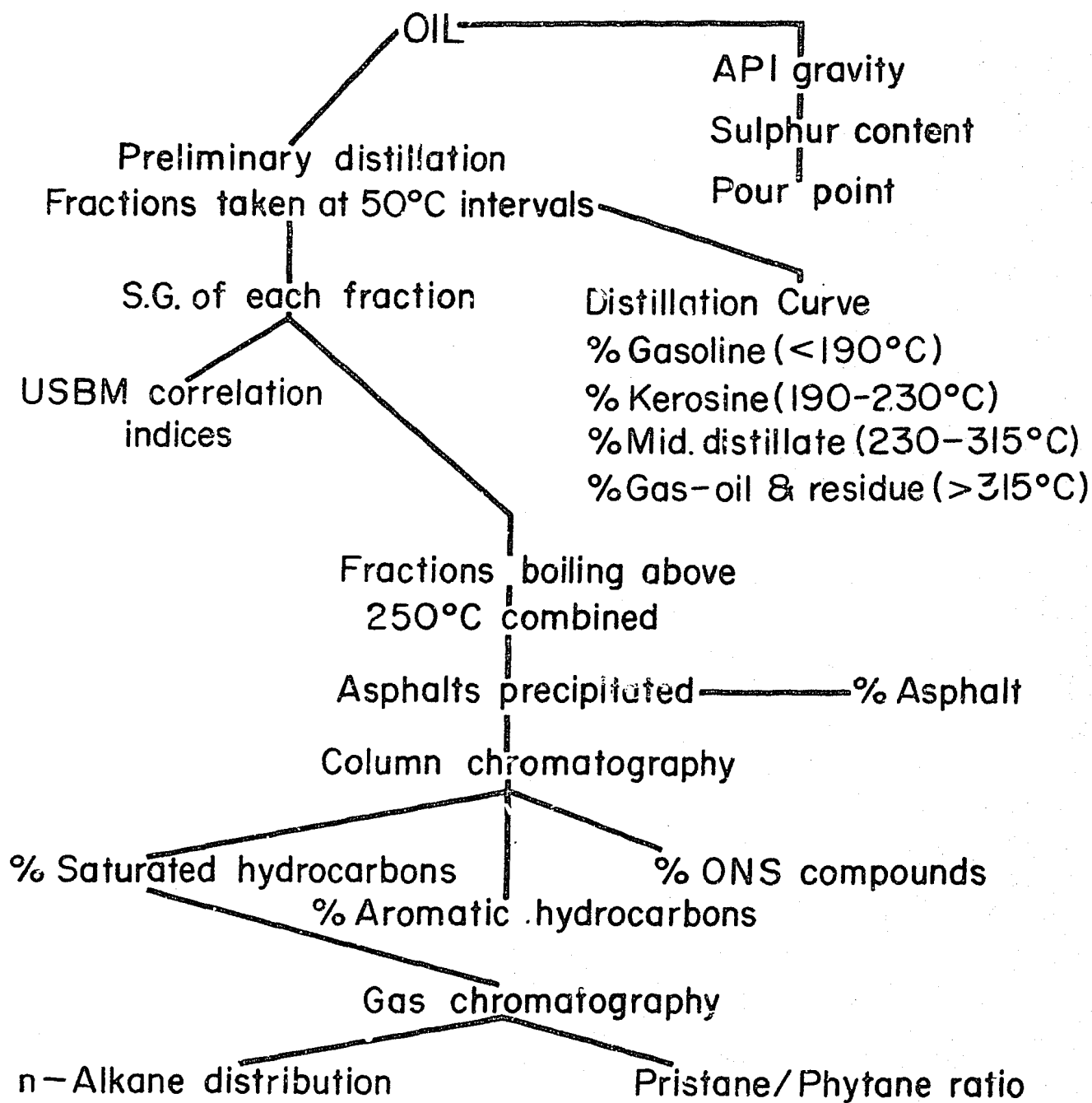


Fig 2. Geological factors affecting petroleum composition (after Bailey et al, 1974)

M1P12



M(PII)22

Fig3. Analytical scheme for crude oil and condensate

Finally, the saturated hydrocarbons are analysed by gas chromatography, using a Varian Gas Chromatograph fitted with a 50 m x 0.25 mm ID capillary column coated with Apiezon L, and nitrogen as the carrier gas. The normal alkanes, and the two isoprenoid alkanes, pristane and phytane, are identified by co-injection of standard compounds and the comparison of retention times with published values.

With the data obtained from these analyses the oil can be characterized in terms of its gross composition and its geological history, as shown in Figure 4. Reference to specific oils from various sedimentary basins across Australia shows how this scheme works.

Figure 5 is the gas chromatogram of the C₁₅₊ alkanes from crude oil from the Halibut field in Bass Strait.

- it has an API gravity of 42.4⁰, which is light by world standards;
- it is paraffinic to naphthenic in composition and low in asphaltenes (0.2%) and S (0.1%);
- it has a high wax content (indicated by a pour point >50 F);
- it contains a high proportion of C₂₀₊ n-alkanes (which give it its waxy character);
- it also has a high ratio of pristane to phytane (8.7 to 1).

The composition of the Halibut oil indicates that it has been derived largely from the spores, pollen, and leaf cuticles of land plants which were deposited in a lacustrine to near-shore marine sedimentary environment.

Figure 6 shows the alkane distribution of a similar oil (from the Conloi field in Queensland) that has undergone both water-washing and biodegradation:

- the lack of normal alkanes indicates that the oil has been altered by bacterial activity;
- its low gasoline content (6.5%, compared with 36% for the Halibut oil) is the result of flushing of the reservoir by groundwater;
- the low API gravity of the oil (28.7⁰) is a direct consequence of these two processes.

The Mereenie oil from central Australia is one of the few Australian oils derived solely from the lipids of marine organisms. Its alkane distribution (Fig. 7) differs from that of the non-marine Halibut oil in that:

<u>ITEM</u>	<u>MEASURE(S)</u>	
1. OVERALL OIL QUALITY	A.P.I. GRAVITY & % SULPHUR	
2. GROSS COMPOSITION (>250°C FRACTION)	% ASPHALTENES SATURATED HYDROCARBONS AROMATIC HYDROCARBONS N,S,O COMPOUNDS	} LIQUID CHROMATOGRAPHY
3. MATURATION & BIODEGRADATION	PARAFFINS IN SATURATE FRACTION (gas-liquid chromatogram)	
4. WATER-WASHING	GASOLINE FRACTION AS % OF TOTAL CRUDE	
5. ACTION OF NATURAL GASES	ANALYSIS OF CAP OR SOLUTION GAS	
6. THERMAL HISTORY OF RESERVOIR	CARBON CONTENT (d.a.f.) OF COAL, KEROGEN IN SURROUNDING SEDIMENTS	
7. OIL CORRELATION, TEST FOR OIL FAMILIES	U S B M CORRELATION CURVES	
8. SOURCE	POUR POINT PRISTANE TO PHYTANE RATIO n-PARAFFIN DISTRIBUTION	

M(PH)23

Fig4. BMR scheme for the geochemical characterization of crude oil
(adapted from Rogers et al, 1971)

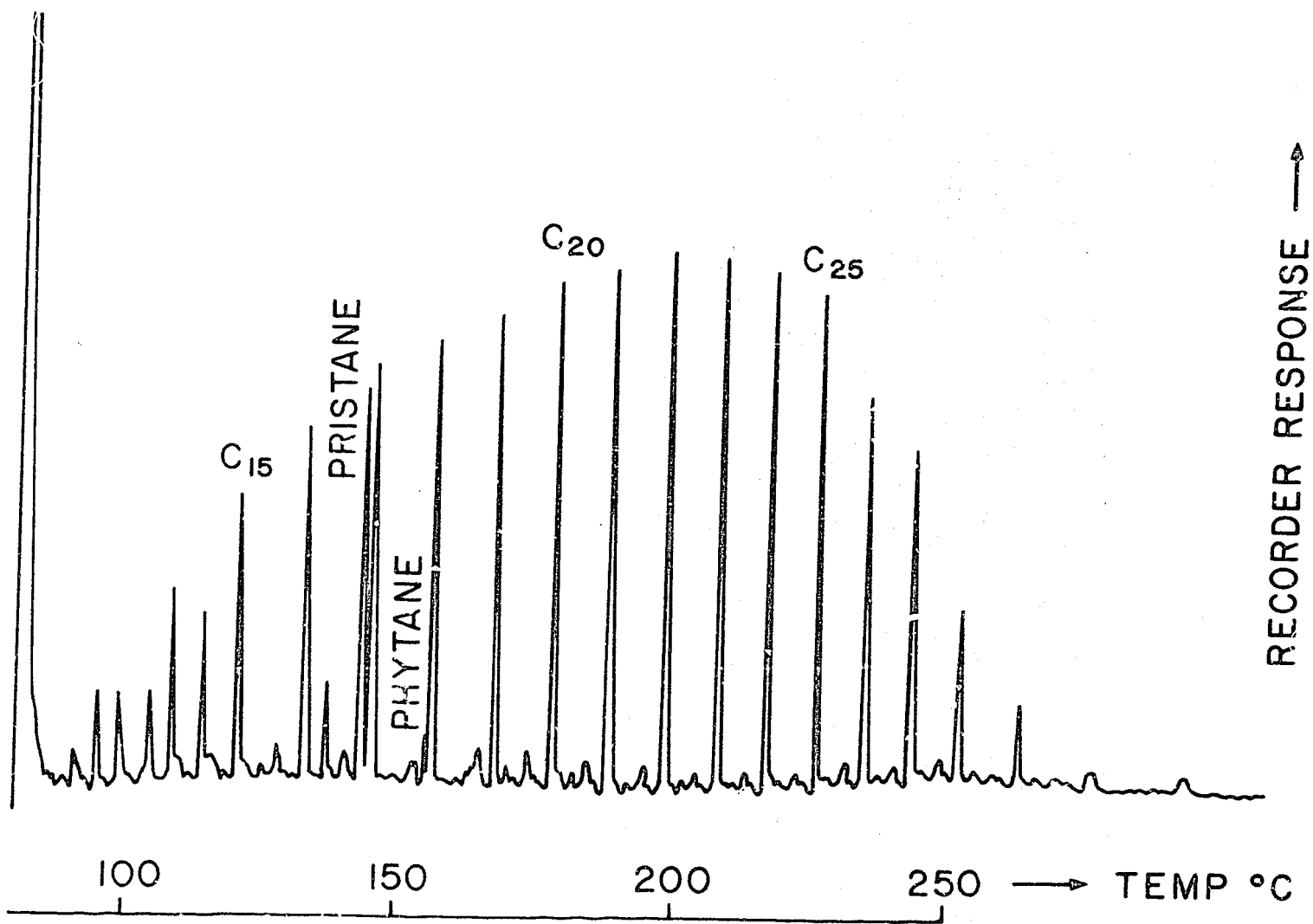


Fig 5. Gas chromatogram of the C_{15+} alkanes in Halibut oil

M(P11)24

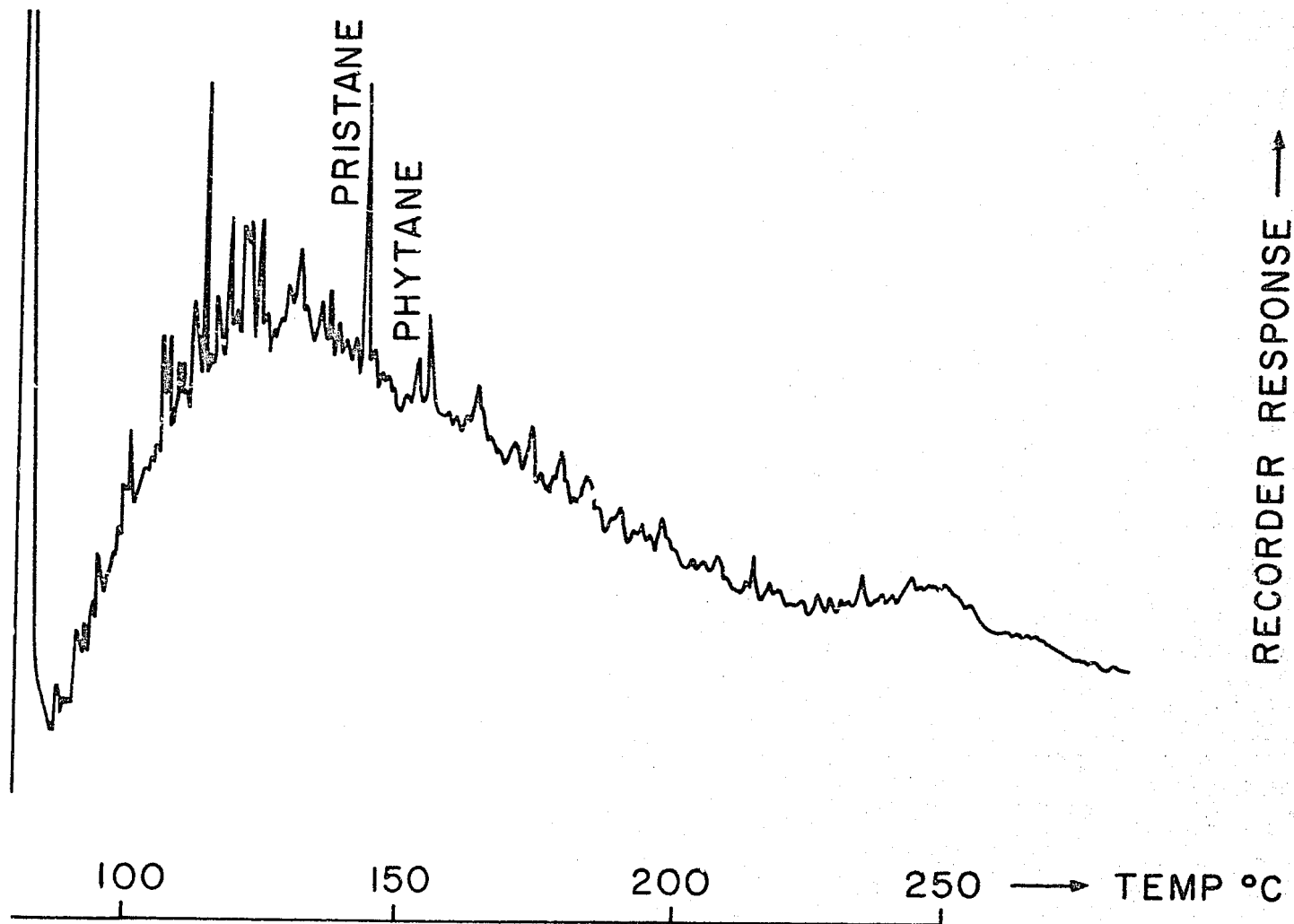
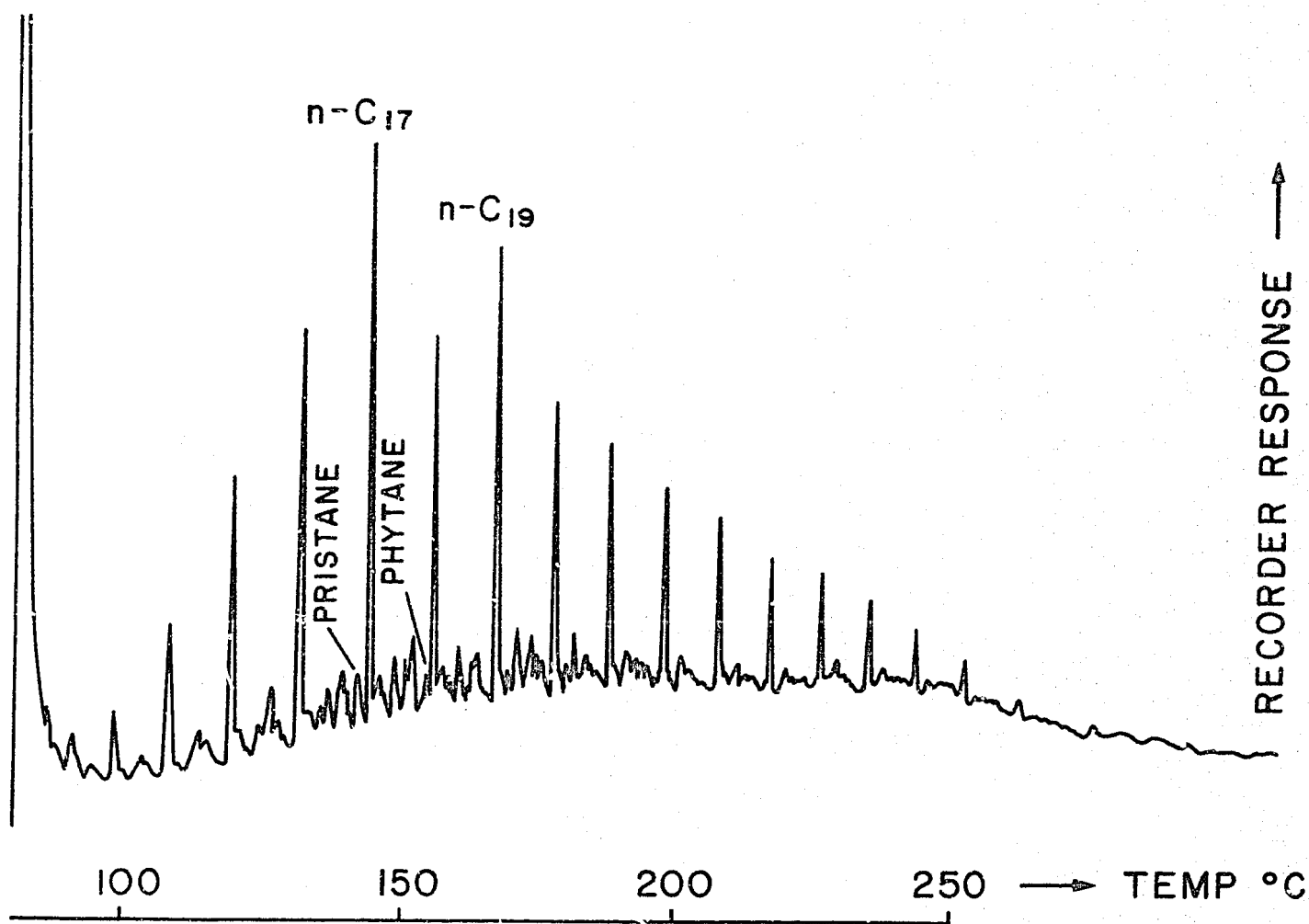


Fig6. Gas chromatogram of the $C_{15}+$ alkanes in Conloi oil



M(911)26

Fig7. Gas chromatogram of the C₁₅+ alkanes in East Mereenie oil.

- most of the n-alkanes have less than 22 carbon atoms in their chain;
- the pristane to phytane ratio (1.5 to 1) is very low.

The action of natural gas on petroleum in a reservoir is illustrated by oils from the Dongara, Mondarra, and Yardarino fields in the Perth Basin, Western Australia:

- these 3 oils contain only 4-6 percent gasoline and have obviously been stripped of their light ends;
- cap gas in the reservoirs is 'dry' (97 percent methane) and appears to have originated in coal measures down-dip from the present oil pools;
- migration of the gas through the oil reservoirs has removed the light fractions in solution.

Prolonged heating of oils, owing to their burial at depths as great as 3500 m in the Earth's crust, causes the thermal cracking of high molecular weight hydrocarbons and produces lighter oils or condensates. At elevated temperatures ($150^{\circ} + 25^{\circ}\text{C}$) petroleum liquids are ultimately destroyed leaving only dry gas (methane). The maximum temperature to which an oil in a reservoir has been exposed is indicated by the rank of coal or kerogen (i.e. dispersed insoluble organic matter) in the surrounding sediments. Figure 8 compares the degree of thermal maturation of oils in the Cooper Basin, South Australia, with that of an oil from a reservoir of similar age in the adjacent Galilee Basin, Queensland:

- the oil from the Galilee Basin has had a less severe thermal history and so its hydrocarbons have been less affected by cracking reactions.

The use of correlation indices to distinguish oils of different genetic types is illustrated in Figure 9. The Gingin and Walyering fields are 22 km apart in the Perth Basin, and gas and condensate are produced from 2 levels within each field. The correlation curves show that

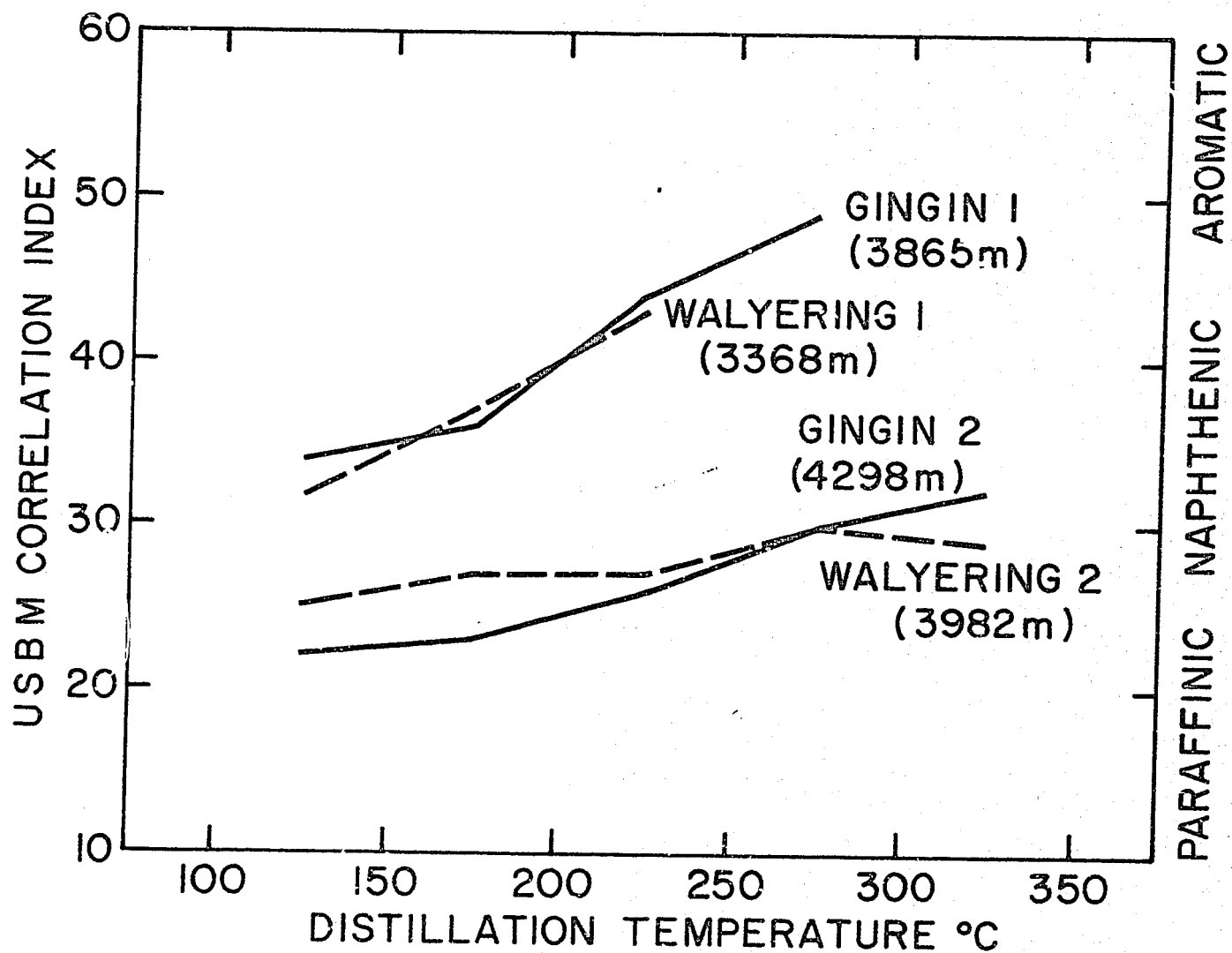
- condensates from upper and lower levels in the same field are markedly different;
- condensates from equivalent horizons in each field are markedly similar
viz. upper - naphthenic to aromatic in composition
lower - paraffinic to naphthenic in composition

Thus, there are two families of oils in this part of the Perth Basin.

	Rank of associated coals (% C daf)	Percent < 200°C fraction in oil
COOPER BASIN, S.A.	84-90	> 50
GALILEE BASIN, QLD	80	< 20

M(PH)27

Fig 8. Effect of thermal alteration on the gasoline content of crude oil.



M (PII) 28

Fig 9. Use of correlation curves to identify 'oil families'

GAS ANALYSIS

Analysis of natural gas is carried out by gas chromatography, using a Varian Aerograph Model 90 - P Gas Chromatograph, in conjunction with a Hewlett-Packard 3370 - B Digital Integrator.

The chromatograph has been modified by the inclusion of 2 columns connected to a sample loop and a calcium chloride drying tube through a special valve block which permits

- 1) switching from one column to the other, and
- 2) back flushing of the HMPA column to elute the hexanes and higher hydrocarbons.

A 20 ft x $\frac{1}{4}$ in. column packed HMPA - coated Chromosorb P (at room temperature) separates methane, ethane, CO₂, propane, isobutane, n-butane, iso-pentane, n-pentane and hexanes plus higher hydrocarbons (Fig. 10). A 5 ft x $\frac{1}{4}$ in. column packed with 5 A molecular sieve (at 35°C) separates H₂, O₂, N₂, and methane. The carrier gas is helium and the detector is of the thermal conductivity type.

Because natural gas is generated by the thermal cracking of organic matter (both liquid hydrocarbons and solid kerogen) dispersed in sediments, its composition, particularly the relative concentrations of the C₁ to C₄ hydrocarbons, will vary according to the maturity (or degree of thermal alteration) of that organic matter.

One convenient measure of this maturity is the abundance of wet gas components (i.e. C₂ - C₄) in natural gas (C₁ - C₄) recovered from gas shows in exploration wells. The Cooper Basin, South Australia provides a good illustration of this point (Fig. 11), and indicates that the 'wetter' the gas found, the more likely the area is to contain oil.

SOURCE-ROCK ANALYSIS

Source-rocks are organic-rich shale, siltstone, and limestone in which petroleum hydrocarbons have been generated at depth under the influence of heat, and from which they have subsequently been expelled.

In order to determine whether or not a particular sedimentary rock unit or sequence has served as a source of oil, it must be ascertained:

- 1) if the rock contains sufficient organic matter to have generated a significant amount of oil;

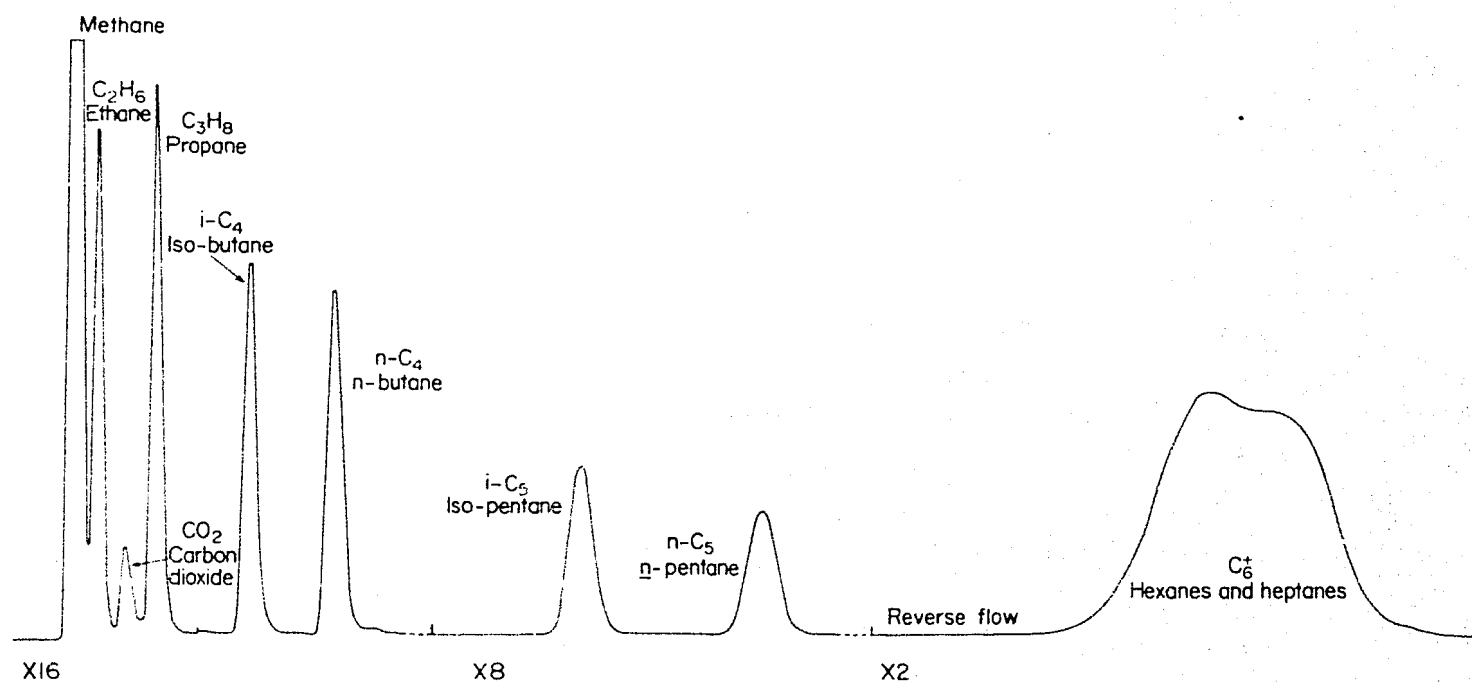


Fig 10. Gas chromatogram of a typical natural gas

1 Tirrawarra — Fly Lake — Moorari Fields

67% wet gas OIL/CONDENSATE AREA

2 Gidgealpa Field

26% wet gas GAS/CONDENSATE AREA

3 Moomba Field

6% wet gas DRY GAS AREA

M (PII) 30

FigII. Use of wet gas as a maturation index in the Cooper Basin, S.A.

- 2) whether the rock has been subjected to temperatures (and pressures) high enough to convert the organic matter to oil;
- 3) if enough soluble organic matter has been produced; and
- 4) whether the soluble (i.e. extractable) organic matter contains petroleum-like hydrocarbons.

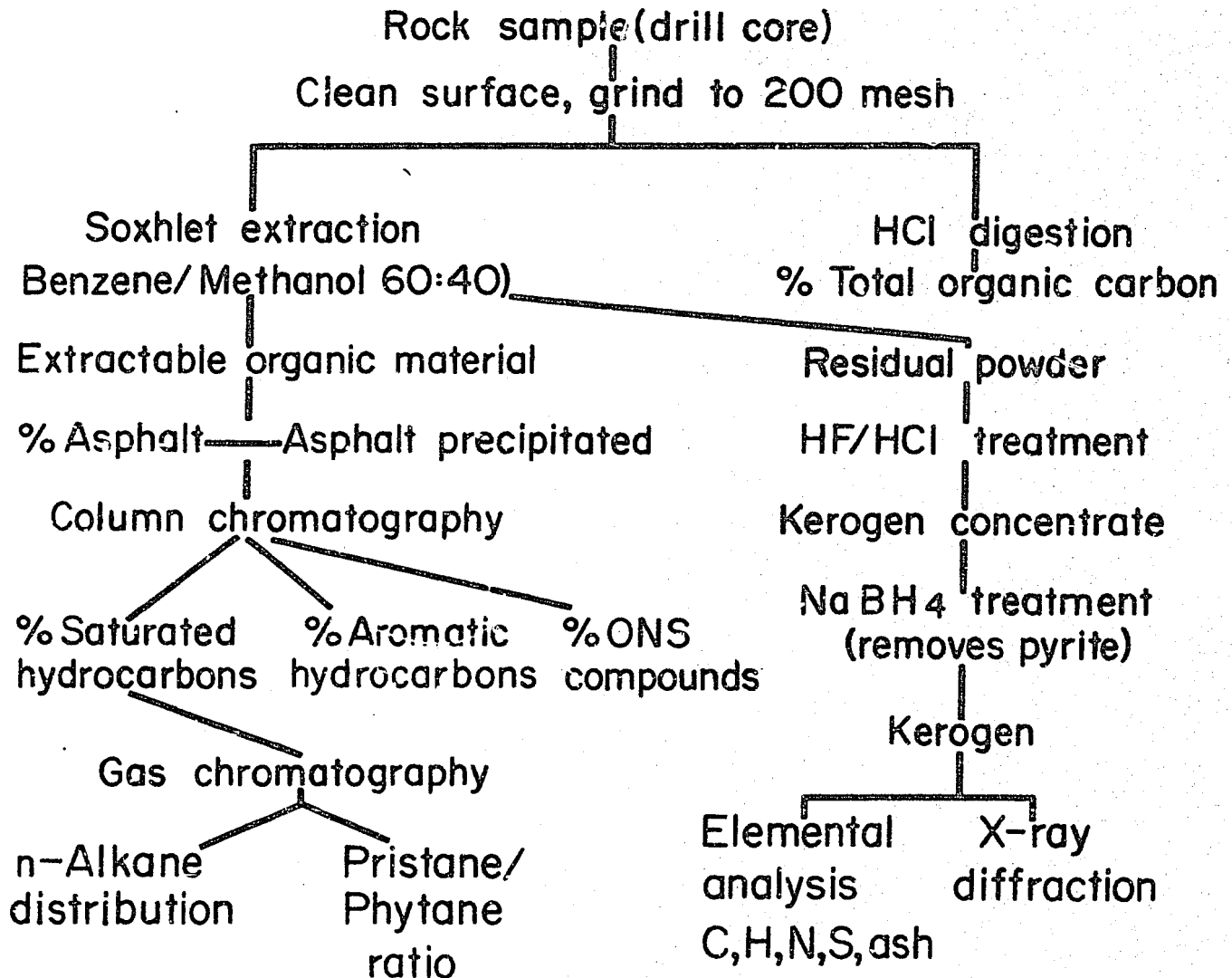
Figure 12 shows the analytical scheme that provides the geochemical data necessary to determine whether these conditions have been met.

The key geochemical parameters used to identify that part of the sedimentary sequence with the best source potential are:

- 1) total organic carbon (TOC)
 - in general, a shale needs to contain at least 0.5 percent TOC to have generated commercially significant quantities of petroleum;
 - on the other hand, a limestone may contain as little as 0.2 percent TOC and still qualify as a source-rock.
- 2) extractable organic matter (EOM)
 - the concentration of EOM is expressed as milligrams of extract per gram of total organic carbon;
 - the minimum value for a good source-rock is about 150 mg EOM/g TOC.
- 3) composition of EOM
 - the relative proportions of the 4 following fractions:
 - . saturated hydrocarbons;
 - . aromatic hydrocarbons;
 - . ONS - bearing compounds; and
 - . asphaltenes

largely depend on the degree of thermal alteration (or maturation) of the organic matter in the sediment.

- with increasing maturation saturated hydrocarbons increase at the expense of asphaltenes and ONS compounds
- in a good source-rock saturated hydrocarbons comprise at least 40 percent of the EOM.



M(Pit) 31

Fig 12. Analytical scheme for source rocks

4) GC analysis of saturated hydrocarbons

- the carbon number distribution of saturated hydrocarbons as determined by capillary gas chromatography provides information on:
 - (a) the type of precursor organic matter
i.e. whether the hydrocarbons originated from algae, or higher plants, or both.
 - (b) the maturity of the organic matter
i.e. the extent to which it has been transformed into petroleum-like hydrocarbons.
- immature organic matter, especially that derived from land plants, contains saturated hydrocarbons in which n-alkanes with an odd number of carbon atoms in their chain are present in greater concentration than those with an even number of carbon atoms (Fig. 13, 2836 m).
- with increasing maturation, owing to burial at greater depth, this odd-even predominance diminishes until, in petroleum-like hydrocarbons, it disappears (Fig. 13, 4138 m).

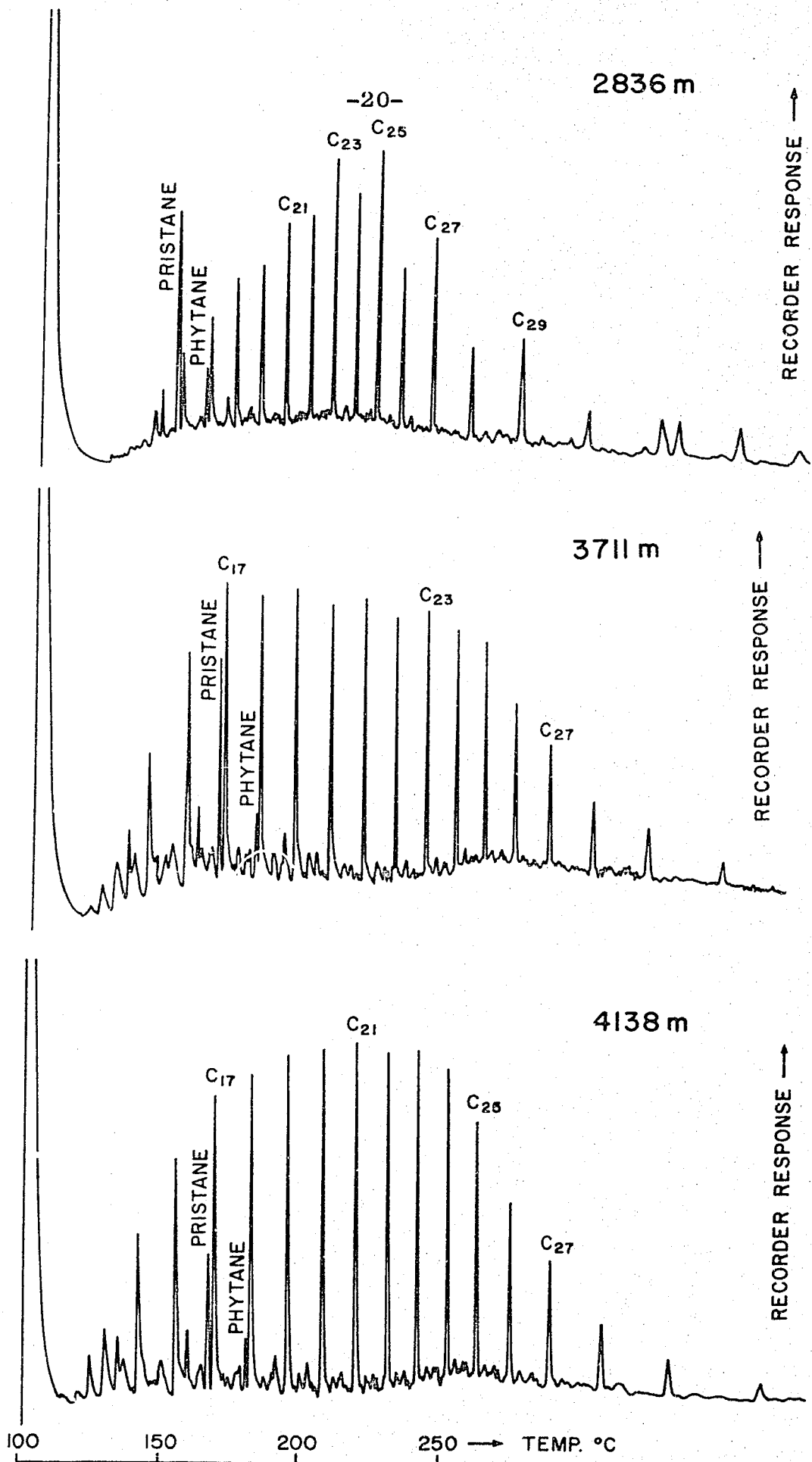
Figure 14 summarizes the source-rock data obtained on drill core samples from an exploration well in Western Australia. The trends which reflect a gradual increase in thermal alteration of the organic matter down the hole are:

- 1) an increase in the concentration of EOM;
- 2) an increase in the concentration of saturated hydrocarbons;
- 3) a shift in the maximum of the n-alkane peak profile to lower carbon number (corresponding to cracking of long-chain n-alkanes); and
- 4) a loss of the odd-even predominance, as exemplified by the n-C₂₇ alkane.

The data plotted in this figure show that the zone of maximum hydrocarbon generation lies below the total depth of the well.

Finally, the elemental composition and structure of the insoluble organic matter (i.e. kerogen) in a source-rock provide an independent check on

- 1) the dominant type of organic matter present
 - it may be algal sapropel, waxy spore, pollen and leaf coatings, or woody humic material
- 2) the type of hydrocarbons it has generated
 - either oil, condensate or gas



M(PII)32

Fig 13. Change in C₁₅+alkane patterns with depth in an exploration well.

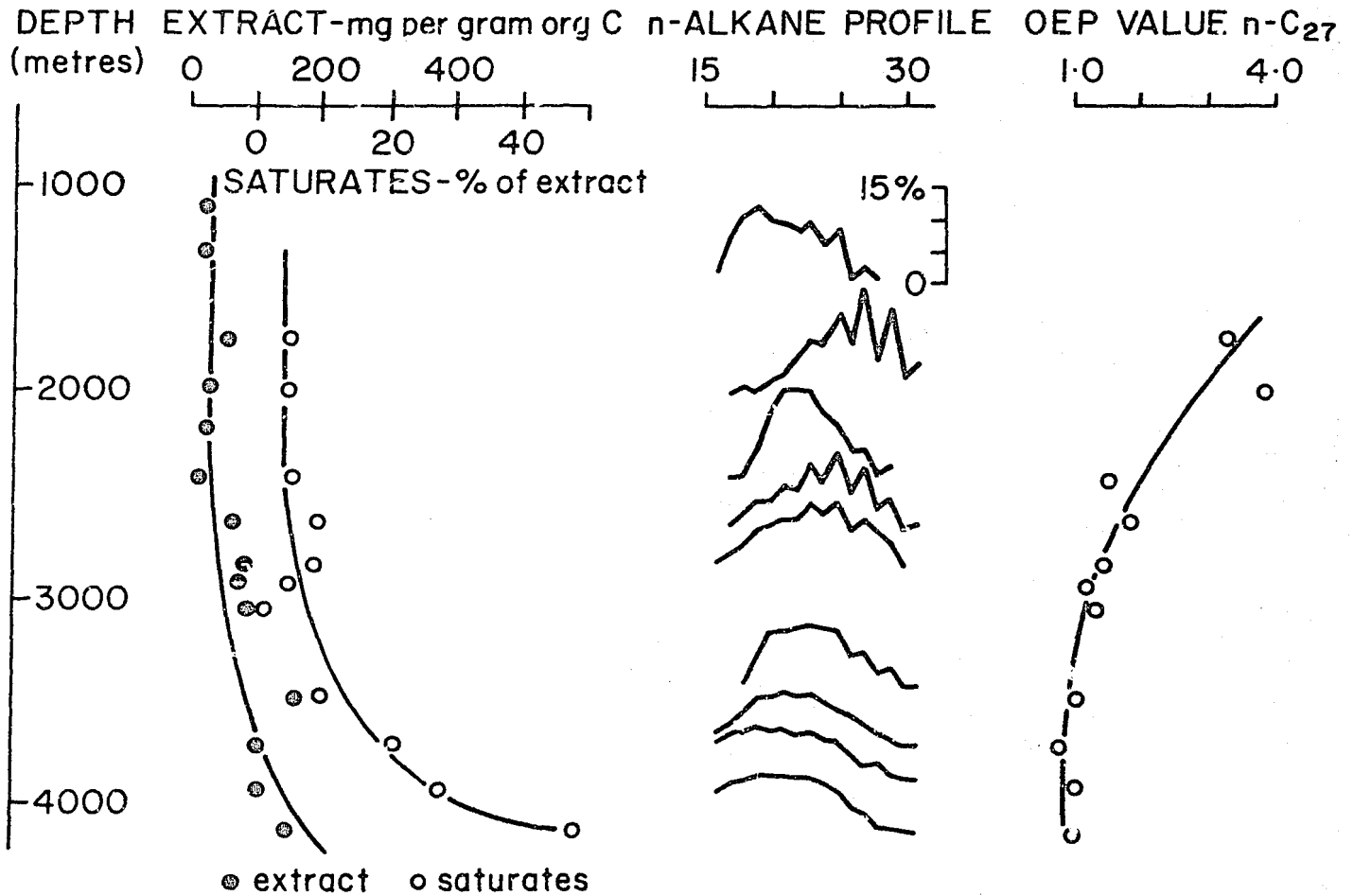


Fig14. Summary of source-rock data from an exploration well

- 3) the maturation level of the organic matter.

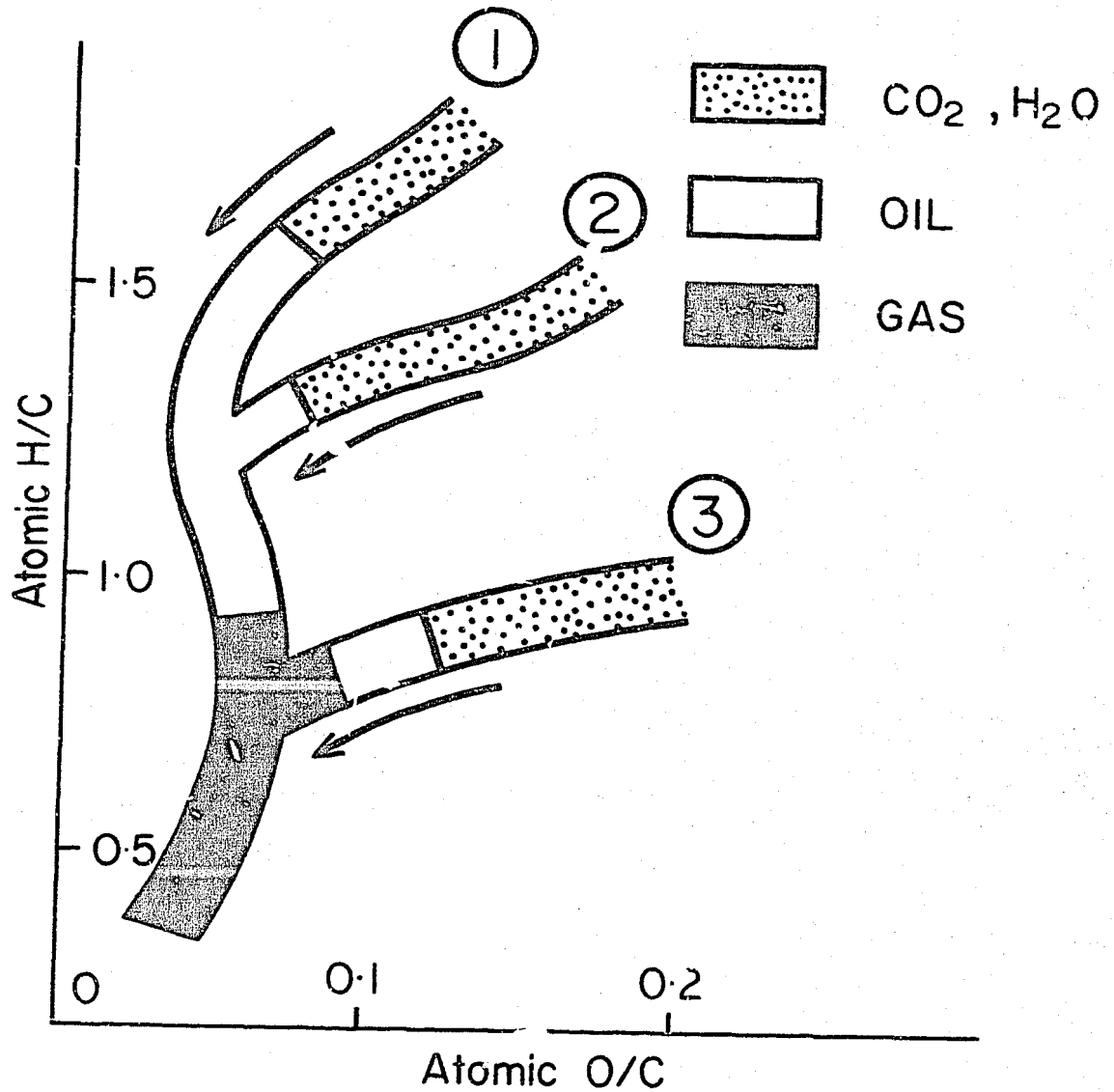
Figure 15 is a plot of kerogen atomic H/C ratio versus O/C ratio. It shows the 3 major types of kerogen:

- 1) hydrogen-rich algal kerogens with a predominantly aliphatic structure;
- 2) intermediate waxy kerogens;
- 3) hydrogen-poor aromatic kerogens.

The arrows show how the composition of each kerogen type evolves along a distinct maturation path, with increasing age and/or depth of burial. Maturation paths (1) and (2) correspond to kerogens able to generate abundant oil. Maturation path (3) represents kerogen capable of producing mainly gas.

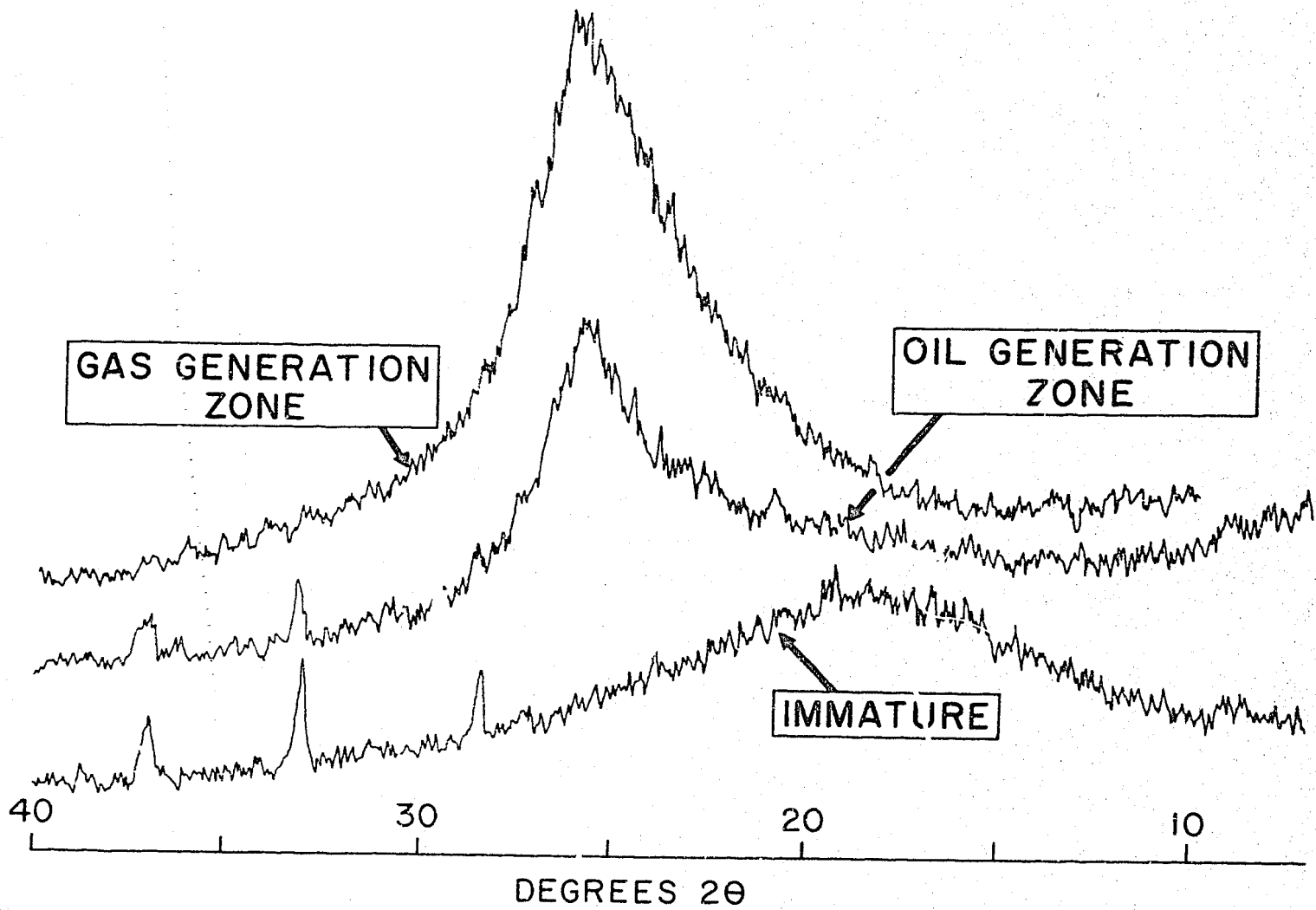
The X-ray diffraction patterns of 3 algal kerogens at different stages of thermal maturation are shown in Figure 16:

- the structure of the top gas-prone kerogen approaches that of graphite;
- graphite and methane are the end-products of the thermal maturation of sedimentary organic matter.



M(PII)34

Fig15. Maturation paths and hydrocarbon generation potential of different kerogen types (after Tissot et al., 1974)



M(PH)35

Fig 16. X-ray diffractograms of algal kerogens at different stages of maturation

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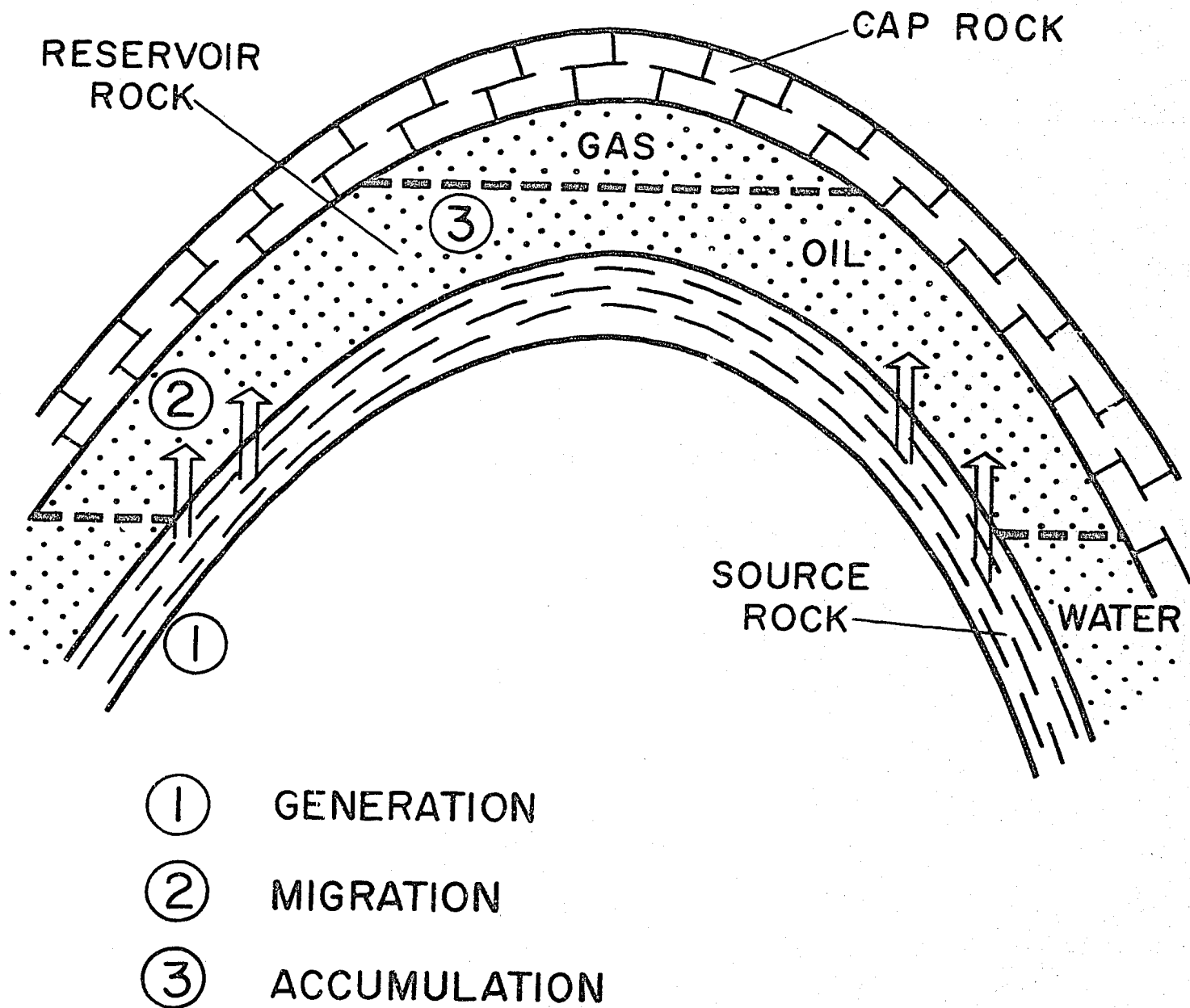
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FIGURES

1. Stages in the formation of a petroleum deposit
2. Geological factors affecting petroleum composition
3. Analytical scheme for crude oil and condensate
4. Geochemical characterization of crude oil
5. Gas chromatogram of C₁₅₊ alkanes in Halibut oil
6. Gas chromatogram of C₁₅₊ alkanes in Conloi oil
7. Gas chromatogram of C₁₅₊ alkanes in East Mereenie oil
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10. Gas chromatogram of a typical natural gas
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12. Analytical scheme for source rocks
13. Change in C₁₅₊ alkanes with depth
14. Summary of source-rock data
15. Kerogen maturation paths and hydrocarbon-generation potential
16. X-ray diffractograms of algal kerogens



M (Pit) 20

Fig1. Diagrammatic representation of stages in the formation of a petroleum deposit.

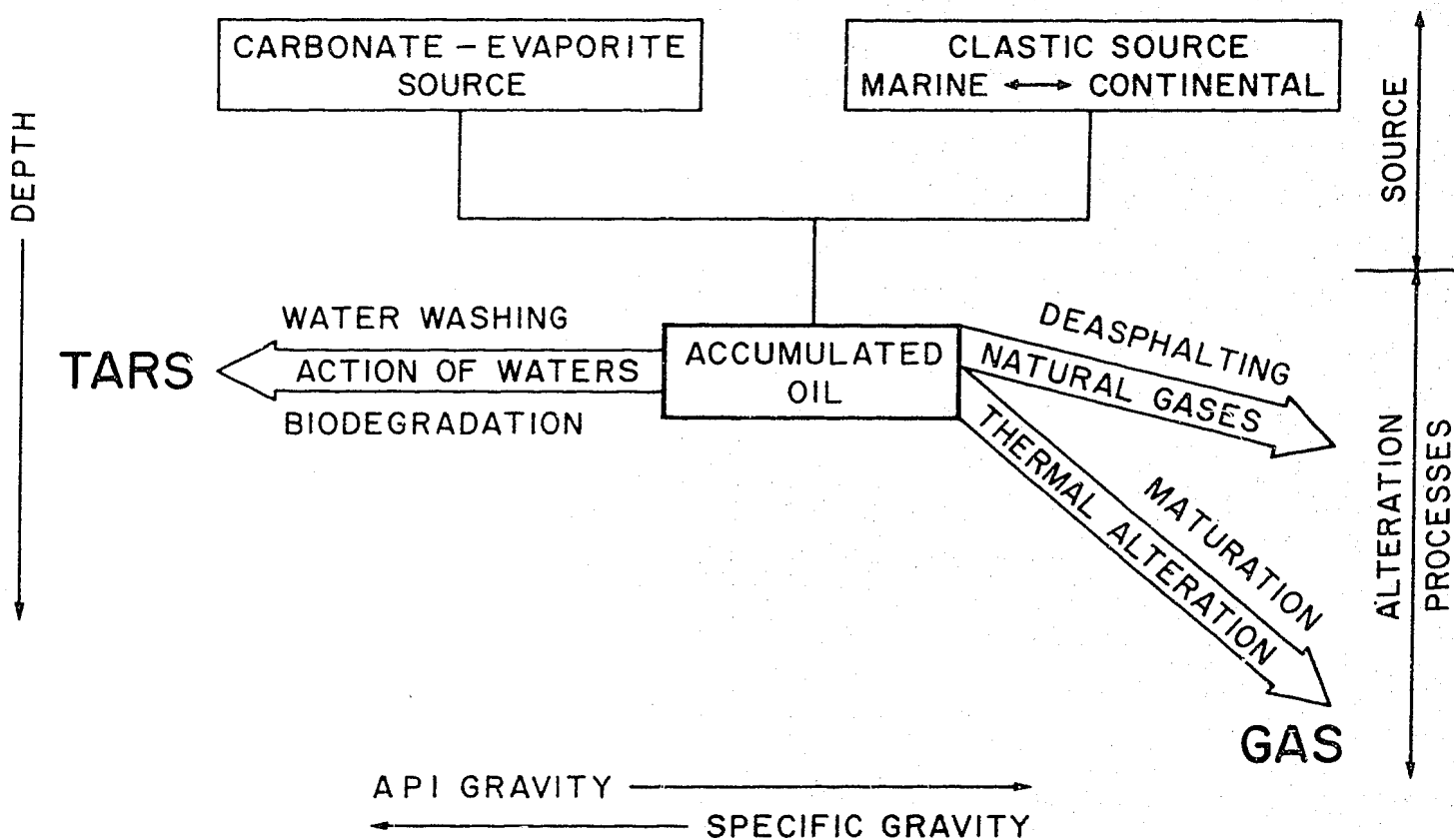
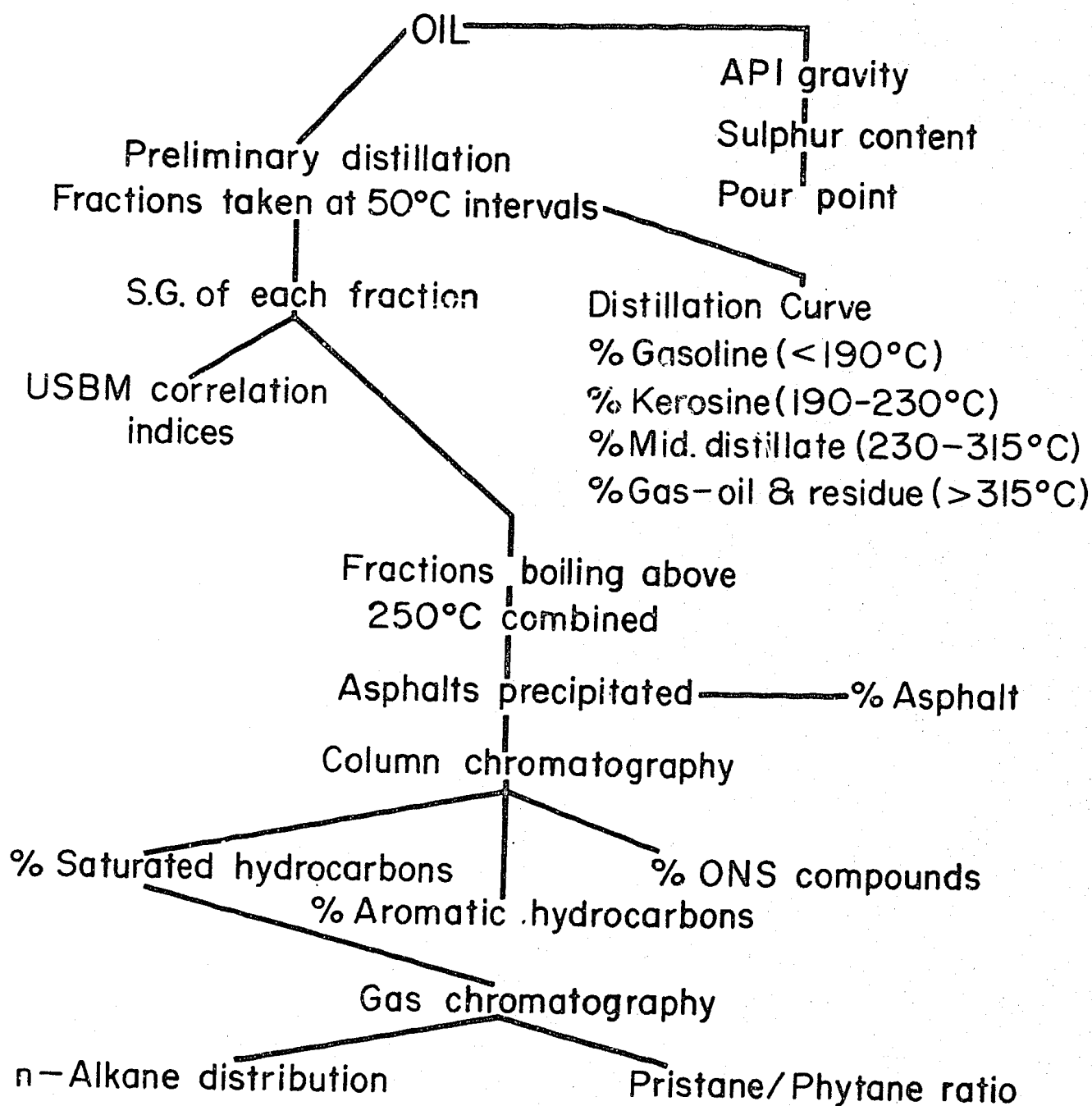


Fig2. Geological factors affecting petroleum composition (after Bailey et al., 1974)



M(Plt)22

Fig3. Analytical scheme for crude oil and condensate

ITEM

MEASURE(S)

1. OVERALL OIL QUALITY	A.P.I. GRAVITY & % SULPHUR	} LIQUID CHROMATOGRAPHY
2. GROSS COMPOSITION (>250°C FRACTION)	% ASPHALTENES SATURATED HYDROCARBONS AROMATIC HYDROCARBONS N,S,O COMPOUNDS	
3. MATURATION & BIODEGRADATION	PARAFFINS IN SATURATE FRACTION (gas-liquid chromatogram)	
4. WATER-WASHING	GASOLINE FRACTION AS % OF TOTAL CRUDE	
5. ACTION OF NATURAL GASES	ANALYSIS OF CAP OR SOLUTION GAS	
6. THERMAL HISTORY OF RESERVOIR	CARBON CONTENT (d.a.f.) OF COAL, KEROGEN IN SURROUNDING SEDIMENTS	
7. OIL CORRELATION, TEST FOR OIL FAMILIES	U S B M CORRELATION CURVES	
8. SOURCE	POUR POINT PRISTANE TO PHYTANE RATIO n-PARAFFIN DISTRIBUTION	

Fig4. BMR scheme for the geochemical characterization of crude oil
(adapted from Rogers et al., 1971)

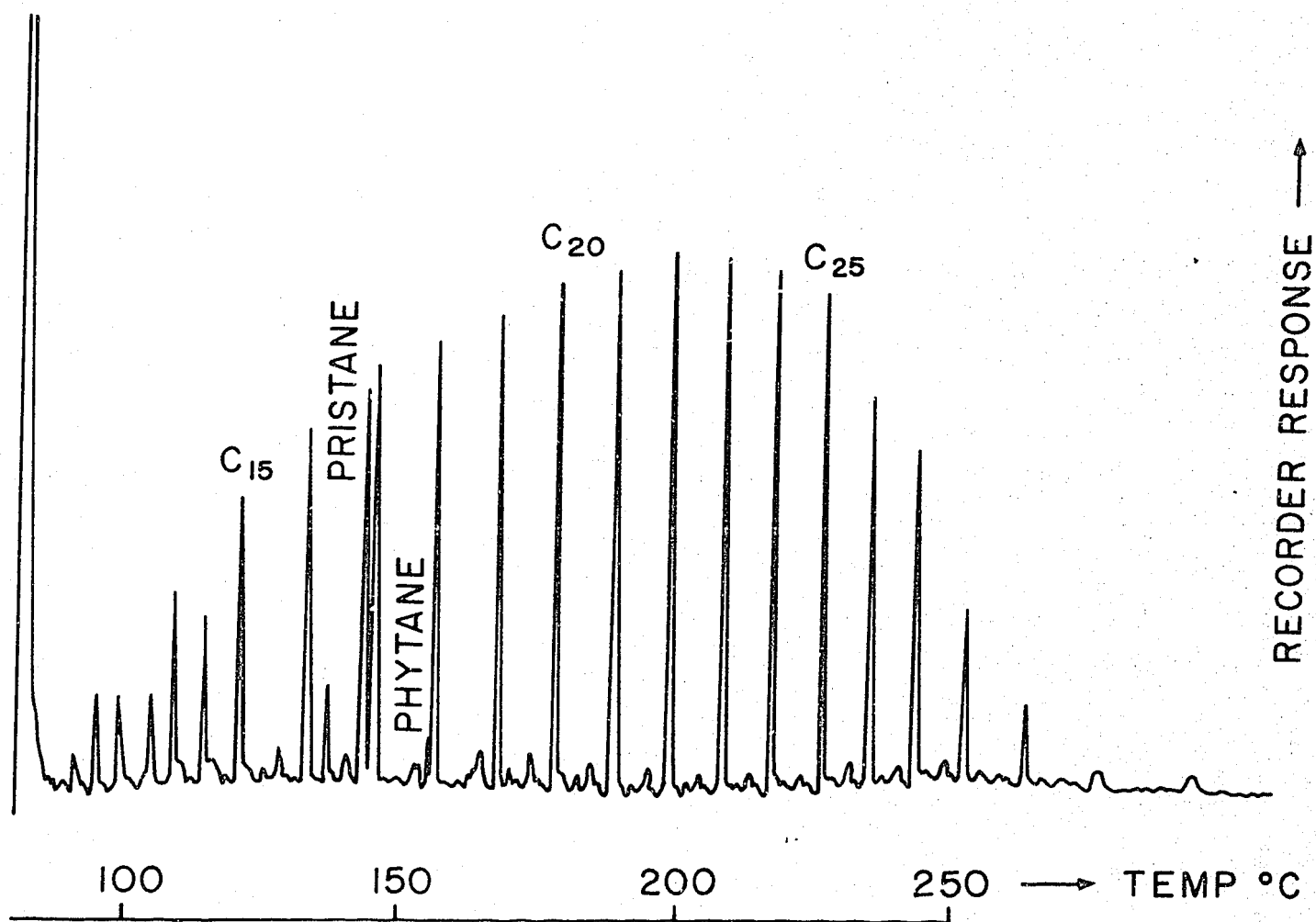


Fig 5. Gas chromatogram of the C_{15+} alkanes in Halibut oil

M(PII)24

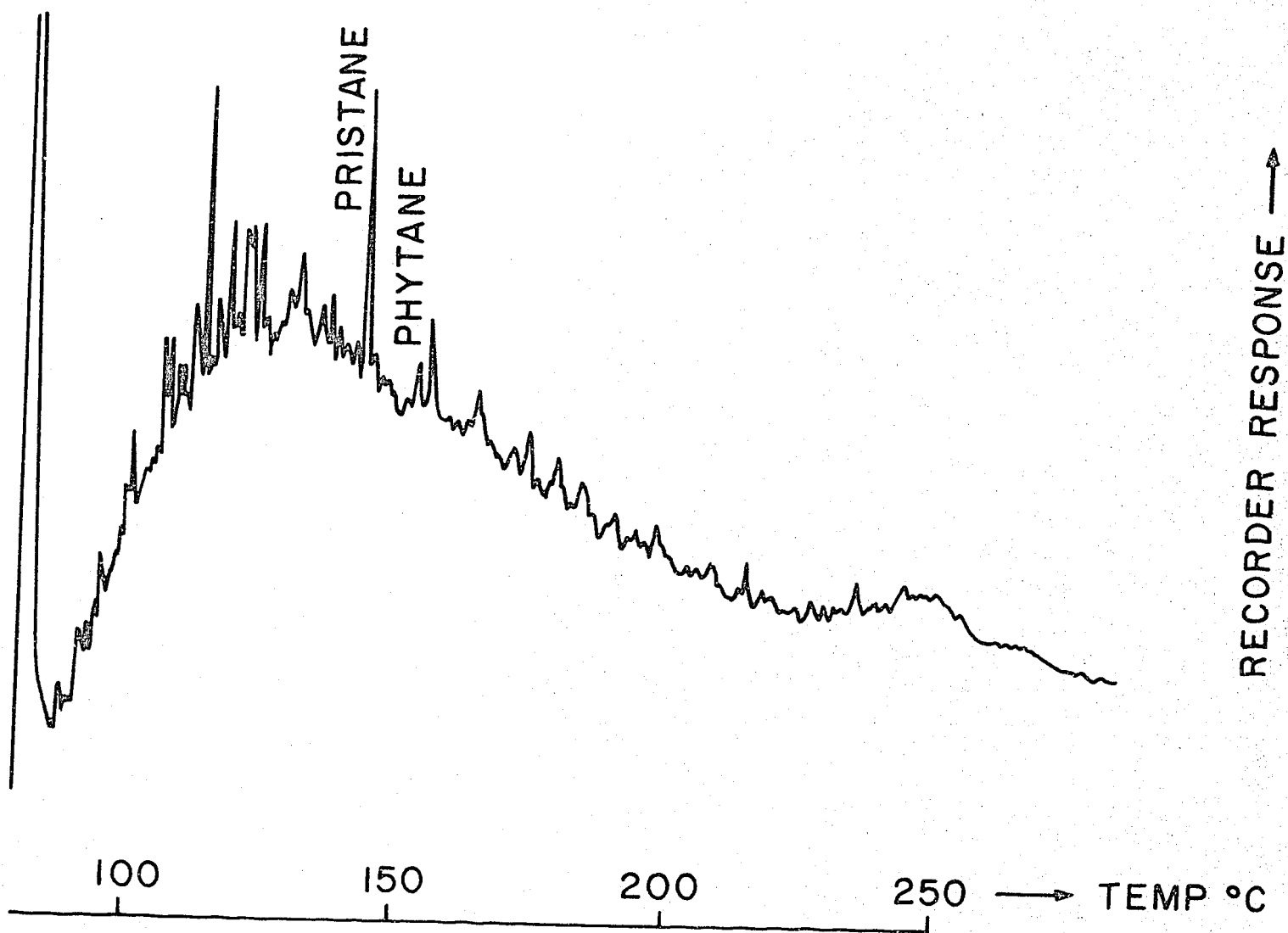


Fig 6. Gas chromatogram of the $C_{15}+$ alkanes in Conloi oil

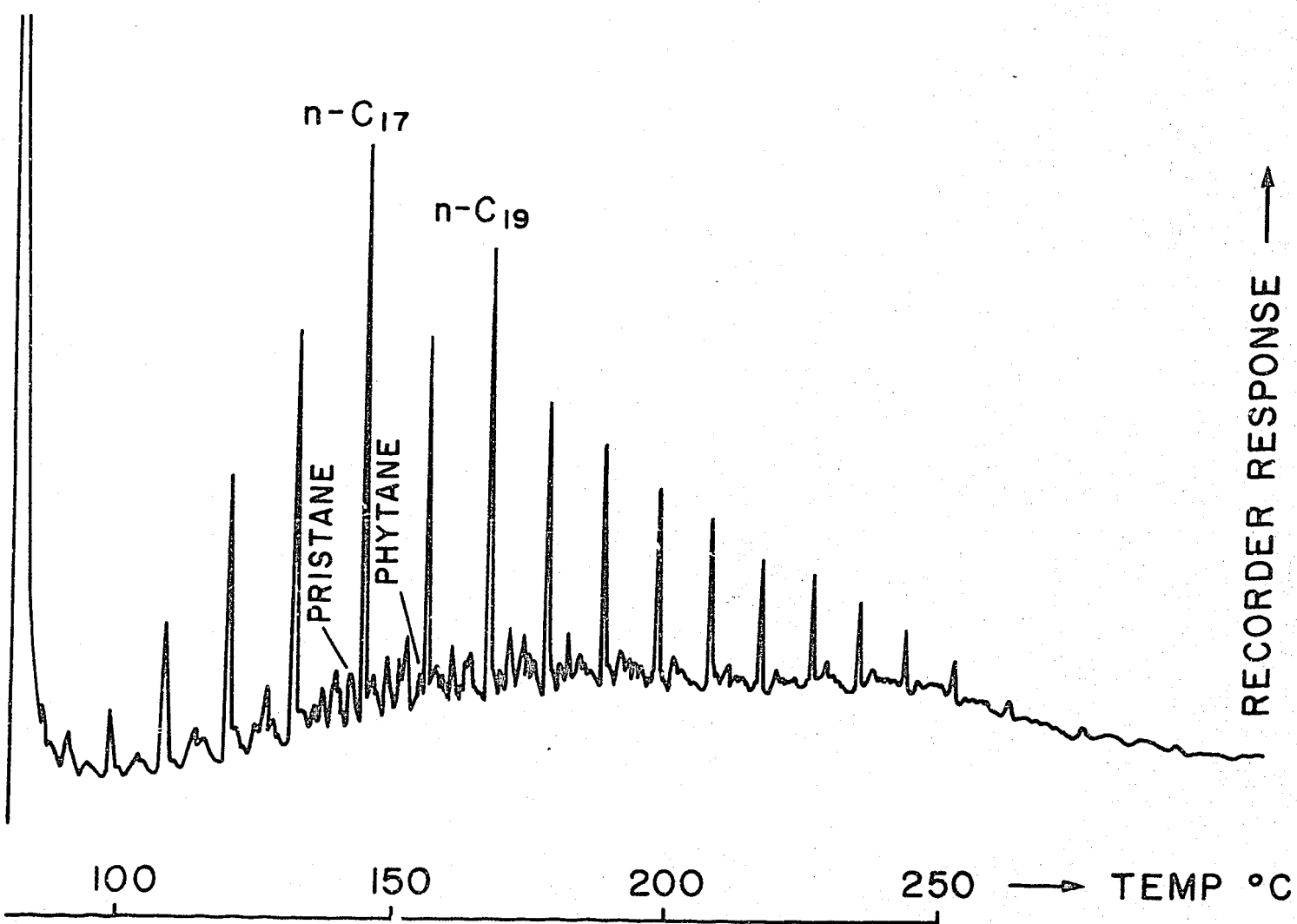


Fig 7. Gas chromatogram of the C₁₅+ alkanes in East Mereenie oil.

	Rank of associated coals (% C daf)	Percent < 200°C fraction in oil
COOPER BASIN, S.A.	84-90	> 50
GALILEE BASIN, QLD	80	< 20

M(P11)27

Fig8. Effect of thermal alteration on the gasoline content of crude oil.

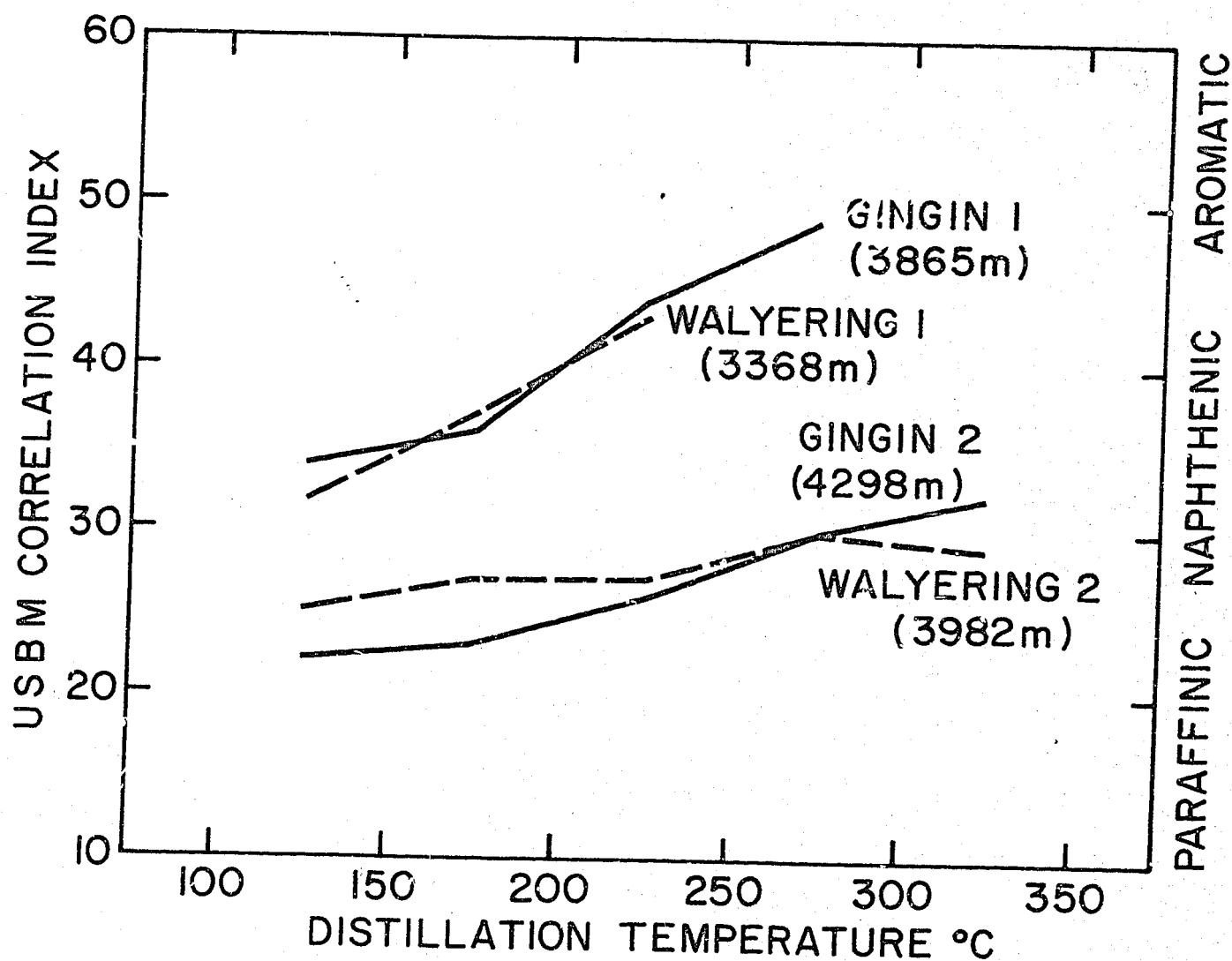


Fig 9. Use of correlation curves to identify 'oil families'

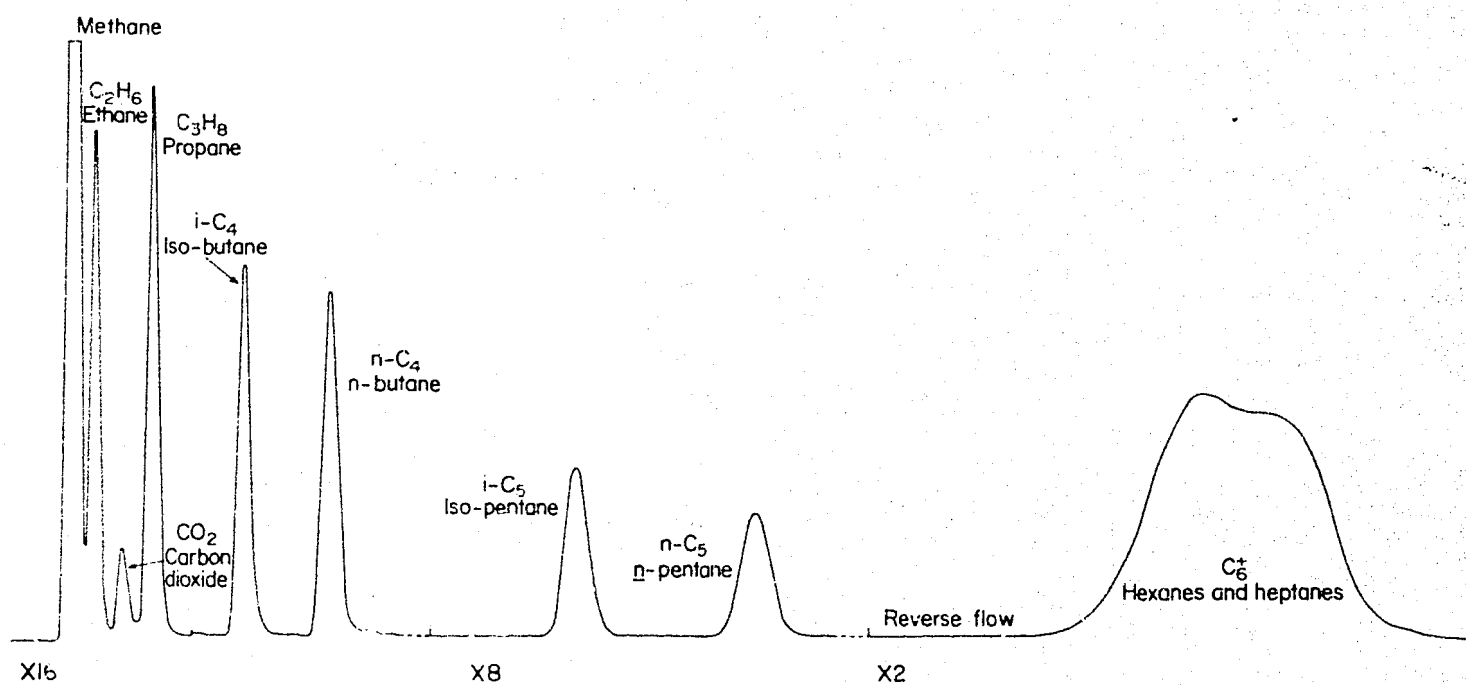


Fig 10. Gas chromatogram of a typical natural gas

1 Tirrawarra — Fly Lake — Moorari Fields

67% wet gas OIL / CONDENSATE AREA

2 Gidgealpa Field

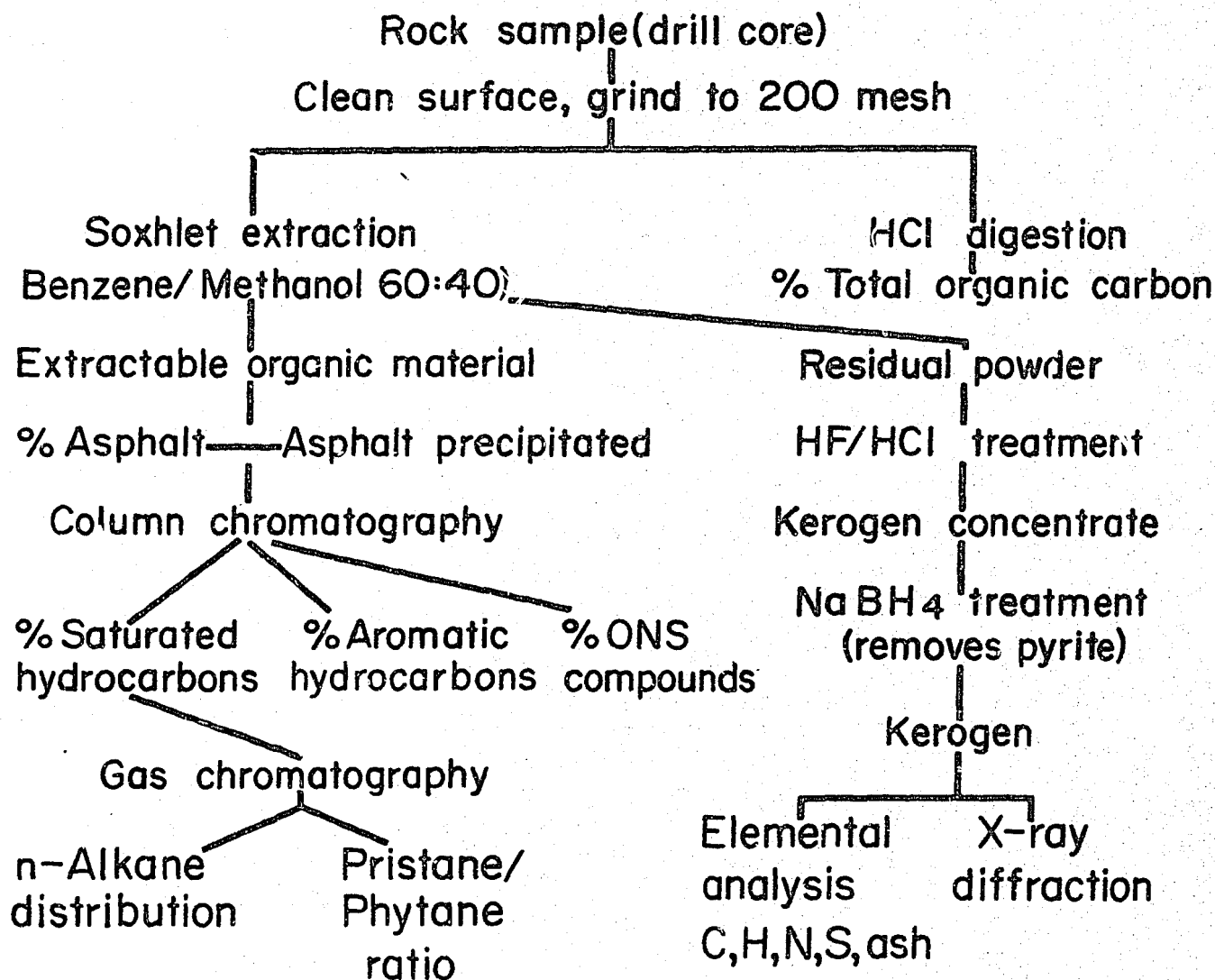
26% wet gas GAS / CONDENSATE AREA

3 Moomba Field

6% wet gas DRY GAS AREA

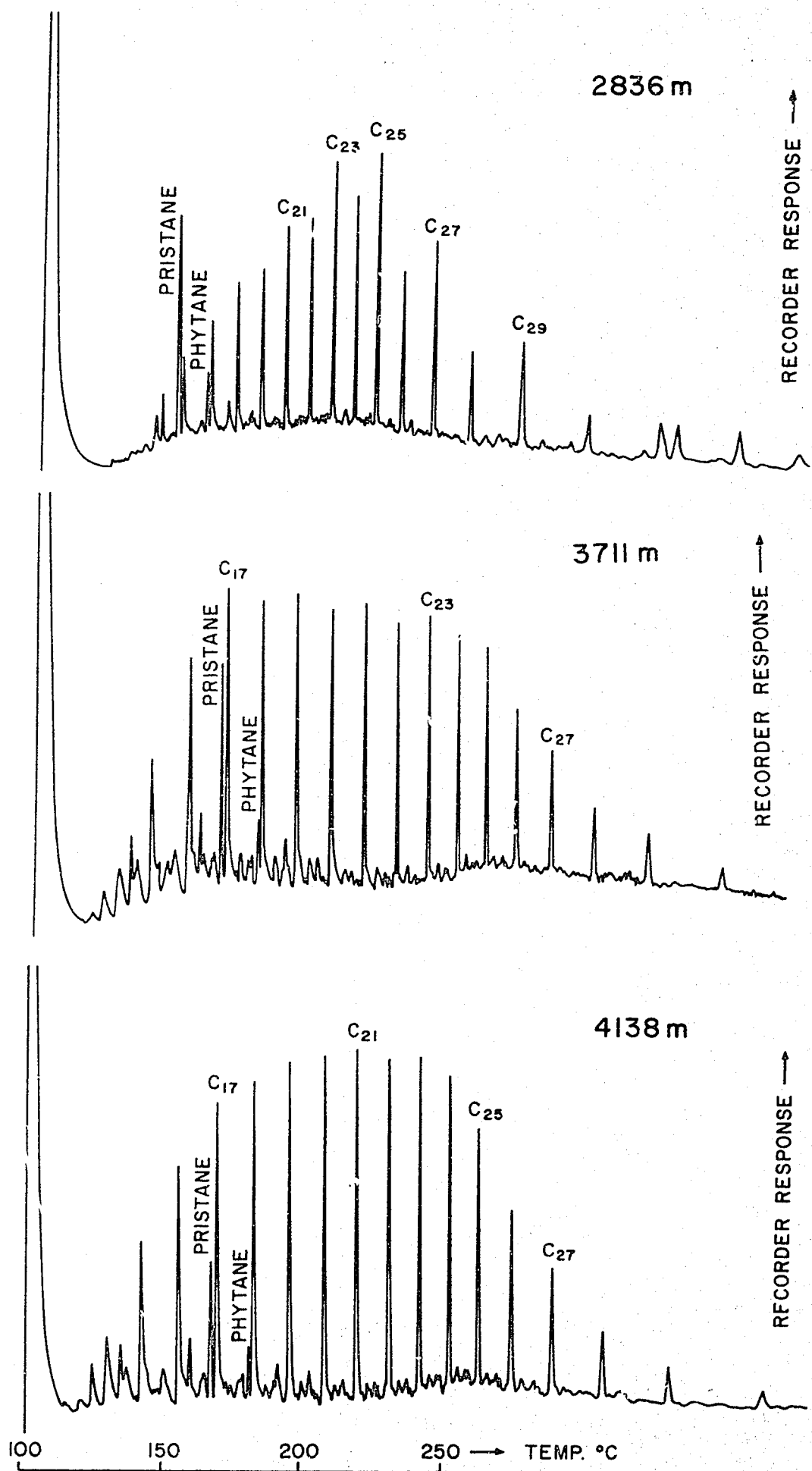
M (PII) 30

FigII. Use of wet gas as a maturation index in the Cooper Basin, S.A.



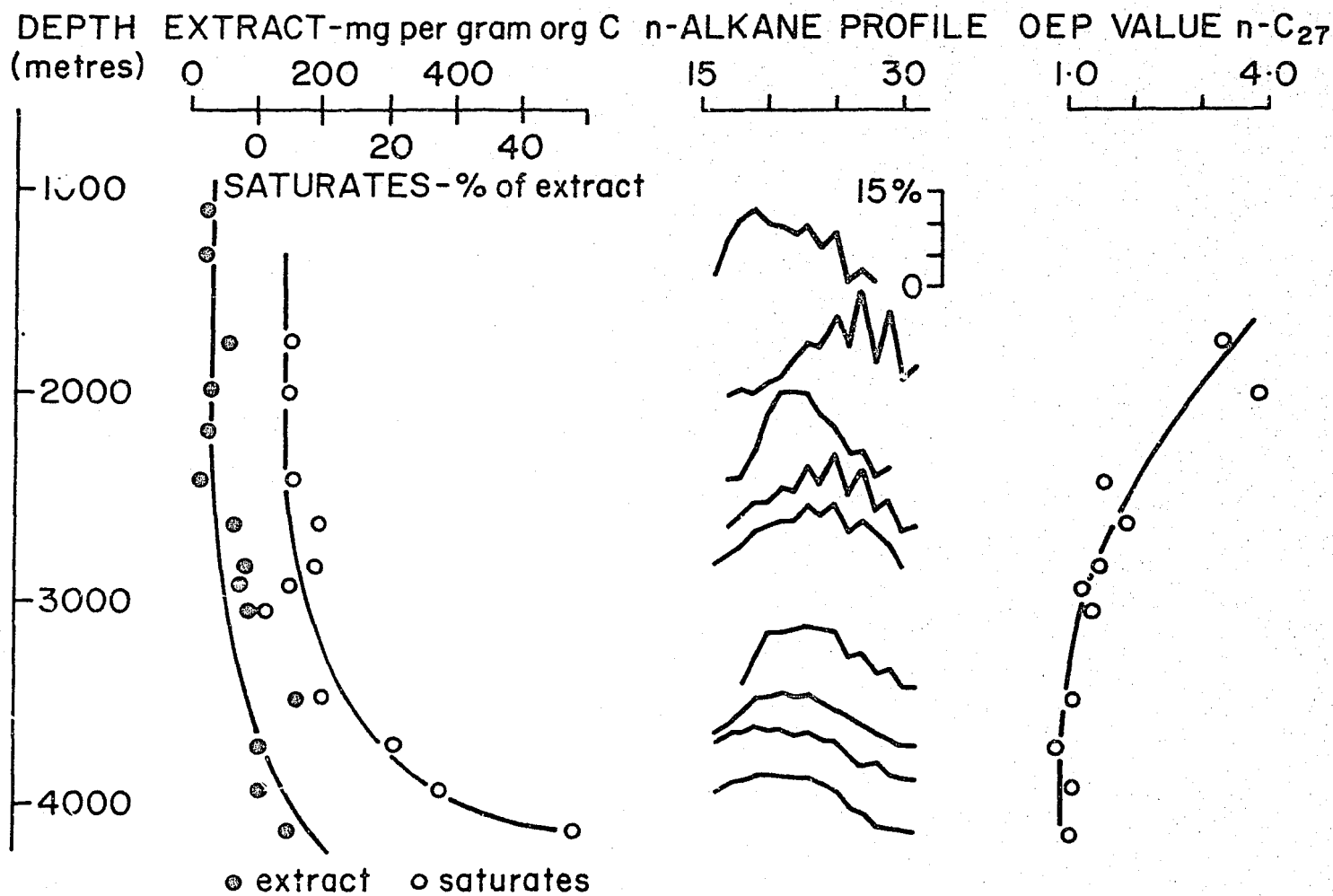
M(P11) 31

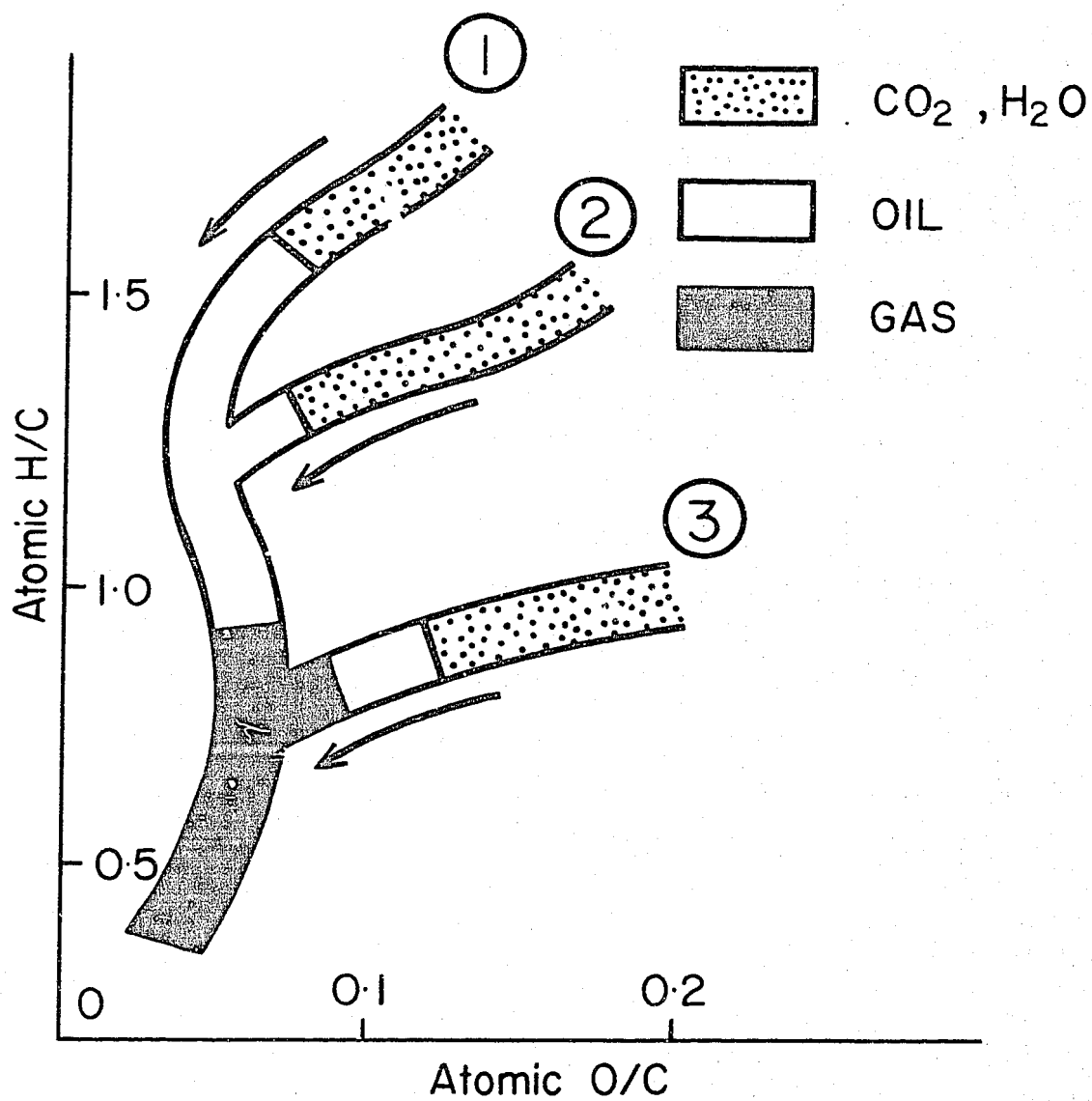
Fig 12. Analytical scheme for source rocks



M(Pit)32

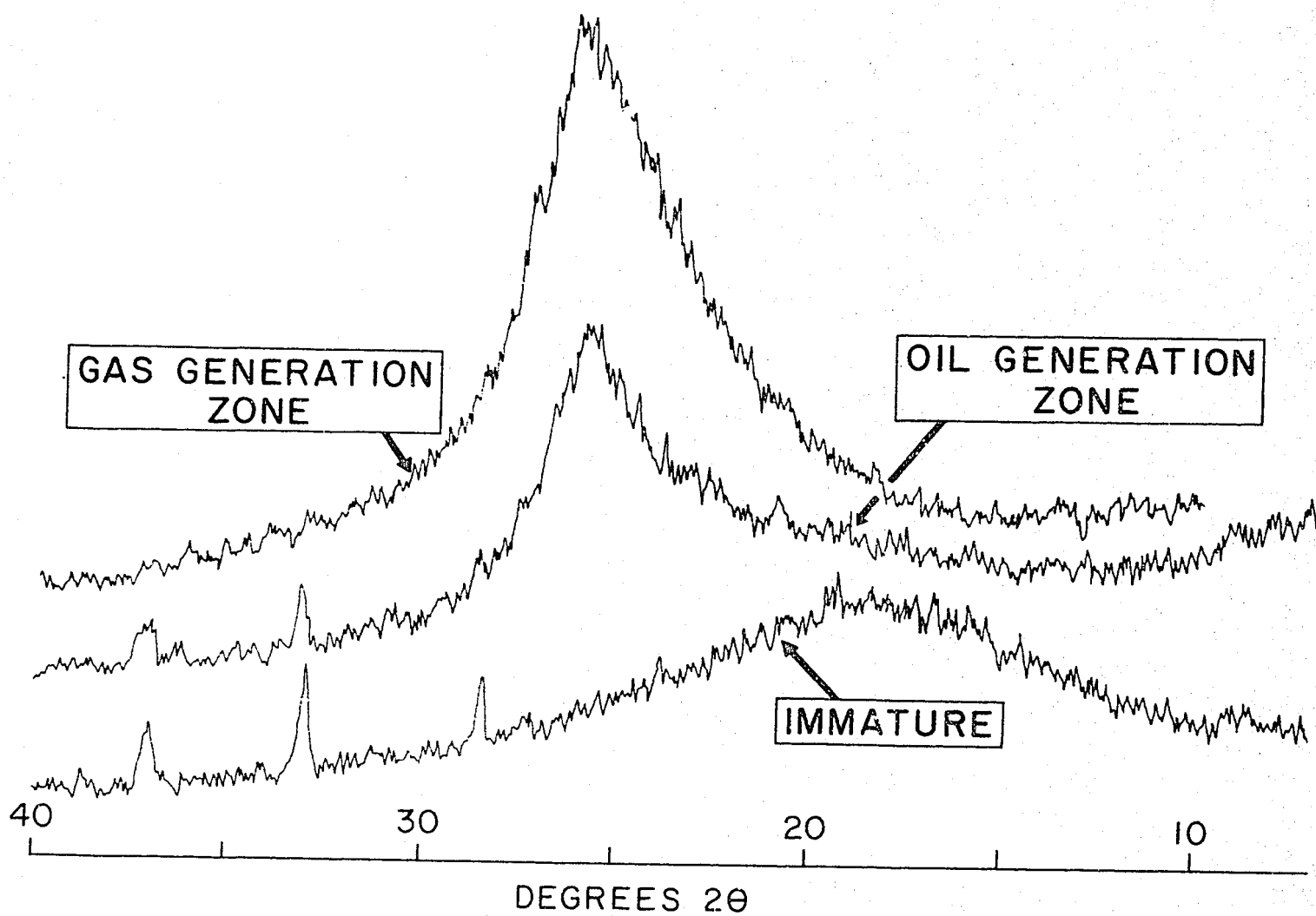
Fig I3. Change in C₁₅+alkane patterns with depth in an exploration well.





M(PH)34

Fig15. Maturation paths and hydrocarbon generation potential of different kerogen types (after Tissot et al., 1974)



M(Pit)35

Fig16. X-ray diffractograms of algal kerogens at different stages of maturation