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A PETROLEUM GEOCHEMICAL EVALUATION
OF THE INTERVAL 980m TO 2999.5m
OF THE 30/9-6 WELL,
DRILLED IN THE NORWEGIAN NORTH SEA

by

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1 SUMMARY

A petroleum geochemical evaluation, comprising maturity and kerogen type determinations, of the interval 980m to 2999.5m of the 30/9-6 well, drilled in the Norwegian North Sea, has been made for Norsk Hydro.

Maturity gradients increase steadily with depth, and the data suggest that the Tertiary and Cretaceous unconformities do not represent significant amounts of missing section. Tertiary sediments, between 980m and 1700m, are immature for hydrocarbon generation. Lower Tertiary and Upper Cretaceous sediments, from 1700m to 2500m, are early mature for oil generation. Middle maturity for oil generation is reached between 2500m and TD at 3034m.

The organic matter comprises mainly humic kerogens throughout the analysed section with minor amounts of algal material present in the Lower Jurassic section.

2 INTRODUCTION

This report presents a petroleum geochemical evaluation of the interval 980m to 2999.5m of the 30/9-6, drilled in the Norwegian North Sea.

The study was requested by Norsk Hydro in a telex dated 26th May 1987. The 23 sidewall core samples, 6 ditch cuttings samples and 4 samples of conventional core were already in-house for the biostratigraphic study of this well, of which 26 samples were used for geochemical studies. The quality of the samples was fair and the quantity was good.

Maturity has been assessed by spore colouration and vitrinite reflectivity studies. A visual assessment of kerogen types has also been made. Initially, samples not being processed for biostratigraphical studies were processed for maturity analyses. Further samples were processed for vitrinite reflectivity analysis, by selecting from the palynological slides the samples rich in vitrinite. Initially, it was hoped that the spore colouration work could be done using the unoxidised mounts of the palynological studies. These, however, proved to be unsuitable for SCI work owing to a difference in mounting technique; the sample material in the palynological slides takes on a brownish stain and is of little use for SCI analysis. Thus further samples, (already processed for VR analysis) were prepared for SCI. Analytical procedures and techniques are described in Appendix 2 of this report.

The number of analyses carried out were:

| | | |
|------------------------|---|----|
| Sample preparation | : | 26 |
| Kerogen preparation | : | 26 |
| Spore colouration | : | 26 |
| Vitrinite reflectivity | : | 26 |

Stratigraphic data used in this report are taken from the Robertson Research International Report 3668/Ia, issued in June 1987.

Robertson Research personnel involved in this study are:

| | | |
|-----------------------|---|------------------------------------|
| Project co-ordination | : | P.C. Barnard |
| Report preparation | : | F. James |
| Microscopy | : | S. Coleman, A.G. Collins, F. James |
| Sample preparation | : | supervised by M. Jones |

3 RESULTS AND INTERPRETATION

3.1 MATURITY EVALUATION

The estimation of spore colour indices (SCI) and kerogen colour in transmitted light and the measurement of the reflectance of vitrinite and associated organic matter in reflected light form the basis of maturity evaluation in this well. Supplementary maturity data is gained from the fluorescence of the organic matter under UV/blue light. Visual estimations of the relative abundance of vitrinite, inertinite and sapropel were also made.

3.1.1 Spore Colouration (Table 1, Figure 1)

Measurable palynomorphs are moderately frequent to abundant in the kerogen preparations. In the Tertiary and Maastrichtian samples, darker coloured palynomorphs, probably stained, were also present. Moderately frequent secondary populations of bleached palynomorphs are present in the Middle and Lower Jurassic samples.

Spores, unlike vitrinite, are relatively similar to oil-prone kerogens and the interpretation of maturity levels is based on spore colouration index data rather than vitrinite reflectivity data. Between 980m and about 1700m, Oligocene and Eocene sediments are immature for hydrocarbon generation. Below 1700m to about 2500m, Lower Tertiary and Upper Cretaceous sediments are early mature for oil generation. From about 2500m to total depth at 3034m, Lower Cretaceous, Middle and Lower Jurassic sediments are middle mature and oil generation could occur if suitable source rocks were present. Spore colour indices increase steadily with depth and the data suggest that Tertiary and Cretaceous unconformities do not represent significant amounts of missing section.

3.1.2 Vitrinite Reflectivity (Table 1, Figure 2)

Measurable vitrinite particles are moderately frequent to occasionally sparse. Secondary populations of higher reflecting material, either reworked vitrinite or inertinite, are common in many of the samples. Low reflecting vitrinite is often present in Middle and Lower Jurassic samples below 2634m.

In general, reflectance values increase steadily with depth although the data do show some scatter. The data offer some corroborative evidence for maturity

levels established from the spore colour indices; interpreted vitrinite reflectivity is Ro 0.27% at 980m, increasing to Ro 0.5% (widely accepted value for the onset of oil generation) at about 2100m, and increasing to an interpreted value of Ro 0.66% at total depth.

3.1.3 UV/blue Light Fluorescence

The fluorescence of oil-prone kerogen and palynomorphs in UV/blue light can provide a general guide to the level of maturity. In the immature Tertiary samples, the miospores show bright yellow to moderate yellow or yellow-orange fluorescence whilst the fluorescence of the dinocysts is more variable from bright yellow to yellow-orange to orange. In the early mature Lower Tertiary and Upper Cretaceous samples the palynomorphs have poor fluorescence, possibly due to slight staining as a result of oxidation during transport and deposition. Palynomorphs in the middle mature Jurassic samples show moderate to dull orange fluorescence.

3.2 KEROGEN TYPING (Table 1)

Visual estimations of the relative abundance of the broad groups of vitrinite, inertinite and sapropel are made on the total kerogen slide mount but reference is also made to the >20 μ sieved fraction to assist in identification. The scheme of identification is shown in Appendix 2.

Between 980m and 1600m (Oligocene and Eocene sediments) organic matter mainly comprises orange-brown non fluorescent amorphous material, probably of humic origin, with occasional degraded light orange-brown vitrinite fragments and rare inertinite fragments. Algae, possibly of Botryococcus type, showing yellow fluorescence were noted at 2235m.

From 2346m to 2475m (Upper Cretaceous sediments) the organic matter comprises mainly small inertinite fragments with light orange-brown humic amorphous matter present at 2346m.

Below 2600m to 2999.5m, the organic matter is humic with frequent inertinite fragments and degraded dark orange/orange-brown vitrinite fragments, often grading into humic amorphous matter. Tasmanites type algae with bright yellow fluorescence are present at 2634.8m and 2844m. Botryococcus type algae with bright yellow fluorescence are present at 2829m, 2870m and 2905m.

4 CONCLUSIONS

A petroleum geochemical evaluation of the interval 980m to 2999.5m of the 30/9-6, drilled in the Norwegian North Sea, has been made for Norsk Hydro and the following conclusions have been drawn:

1. The maturity gradients increase steadily with depth, and the data suggest that the Tertiary and Cretaceous unconformities do not represent significant amounts of missing section.
2. From 980m to about 1700m, Oligocene and Eocene sediments are immature for hydrocarbon generation. Below 1700m to about 2500m, Lower Tertiary and Upper Cretaceous sediments are early mature for oil generation. Sufficient maturity for oil generation is reached in the Lower Cretaceous and Jurassic sections between 2500m and total depth at 3034m.
3. Humic kerogens, mainly degraded vitrinite and amorphous humic matter, dominate the organic matter from 980m to 1600m and from 2600m to 2999.5m. Inertinitic kerogens predominate between 2346m and 2475m. Algae, of Tasmanites and Botryococcus type, are present in the Lower Jurassic section.

| SAMPLE DEPTH (METRES) | SAMPLE TYPE | GENERALISED LITHOLOGY | SPORE COLOUR INDEX (1-10) | VITRINITE REFLECTIVITY R _{oil av} % | KEROGEN COMPOSITION (%) (by microscopic examination) | | | KEROGEN COMPOSITION (%) (by calculation from pyrolysis data) | | | | |
|--------------------------|----------------|---|------------------------------|--|---|---------------|----------------------|---|-----------|-------------------|------------------|--|
| | | | | | INERTINITE | VITRINITE | SAPROPEL | INERTINITE | VITRINITE | ALGAL SAPROPEL | WAXY SAPROPEL | |
| 980 | Ctgs | MDST, lt brn-gy + mnr CMT | 2.5 - 3.0 | .31(16) .53(2)R | 10 | 20 inc Am | 70 | | | | | |
| 1200 | " | MDST, brn-gy+ 20% MDST, lt brn-gy, calc + 20% SND | 2.5 - 3.0 | .30(12) .54(2)R | 10 | 90 Am | Mnr Sp + Di | | | | | |
| 1400 | " | MDST, brn-blk+ 20% MDST, lt brn-gy | 3.0 - 3.5 | .31(5) .59(2)R | 10 | 90 Am | Mnr Sp + Di | | | | | |
| 1600 | " | MDST, ol-gy+ tr MDST, pal yel-brn, calc+ tr DOL, dk yel-brn | 3.0 | .40(4) .27(3)C .96(2)R | 20 | 80 | Mnr Sp + Di | | | | | |
| 1800 | " | MDST, dk gy+ tr DOL, pal yel-brn | 3.5 | * | 70 | 20 | 10 Sp + Di | | | | | |
| 2000 | " | MDST, dk gy+ 30% MDST, dsk brn+ tr DOL, pal yel-brn+ tr CHK | 3.5 | .44(8) .36(5)C | 20 | 70 inc Am | 10 Sp + Di | | | | | |
| 2165 | Swc | CLYST, dk gn-gy + tr SLTST, brn-blk | 3.5 - 4.0 | 1.42(8)R | 80 | 10 | 10 Sp + Di | | | | | |
| 2235 | " | CLYST, dk gn-gy, mic+ tr pyr | 3.5 | .51(24) .67(16)R | 90 | Mnr | 10 Sp + Di | | | | | |
| 2275 | " | CLYST, ol-blk | 4.0 | .53(3) .75(3)R | 90 | Mnr | 10 Sp | | | | | |
| 2305 | " | CLYST, gn-blk+ tr CLYST, brn-gy calc | 3.5 - 4.0 | .54(2) .74(7)R | 90 | 10 | Mnr Sp + Di | | | | | |
| 2346 | " | CLYST, gy-brn, calc+ tr CLYST, lt ol-gy, mic | 4.0 | .51(2) .65(1)R | 10 | 90? Am | * | | | | | |
| 2400 | " | CLYST, med-dk gy calc+ tr pyr | 4.0 | .57(10) .68(3)R | 80 | 10 | 10 Sp + Di | | | | | |
| 2441 | " | CLYST, med-dk gy + tr pyr+ tr glc + tr SST, wht | 4.5 | .61(28) .85(13)R | 80 | 10 | 10 Sp + Di | | | | | |
| 2475 | " | CLYST, dk gn-gy + 20% CLYST, gn-blk | 4.5 | .60(5) .87(5)R | 80 | 10 | 10 Sp + Di | | | | | |
| 2600 | Core | CLYST, med gy, mic | 4.5 - 5.0 | .69(39) | 40 | 60 | * | | | | | |
| 2634.8 | " | CLYST, med-lt gy mic | 5.0 | .62(16) .48(38)L | 20? | 70? inc Am | 10? | | | | | |
| 2706 | Swc | SST, lt brn-gy+ 20% CLYST, brn-gy | 5.5 | .60(12) .88(42)R | 20 | 70 | 10 Sp + Cu | | | | | |
| 2735 | " | CLYST, brn-gy | 5.0 | .66(20) .97(30)R | 30 | 70 | * | | | | | |
| 2785 | " | CLYST, dk gn-gy | 5.5 | .63(10) .99(43)R | 50 | 30 | 20 Sp, Cu + Am | | | | | |
| 2829 | " | CLYST, brn-gy, mic | 5.0 - 5.5 | .64(1) .64(1) 1.25(4)R | 30 | 50 | 20 Am + Sp | | | | | |

Maturity and Kerogen Composition Data

TABLE 1A

COMPANY: NORSK HYDRO

WELL: 30/9-6

LOCATION: NORWEGIAN NORTH SEA

| SAMPLE DEPTH (METRES) | SAMPLE TYPE | GENERALISED LITHOLOGY | SPORE COLOUR INDEX (1-10) | VITRINITE REFLECTIVITY R oil av % | KEROGEN COMPOSITION (%) (by microscopic examination) | | | KEROGEN COMPOSITION (%) (by calculation from pyrolysis data) | | | |
|--------------------------|----------------|---|------------------------------|---|---|-----------|-----------------|---|-----------|-------------------|------------------|
| | | | | | INERTINITE | VITRINITE | SAPROPEL | INERTINITE | VITRINITE | ALGAL SAPROPEL | WAXY SAPROPEL |
| 2844 | Swc | CLYST, brn-gy, mic | 5.0 - 5.5 | .77(7) .45(24)L 1.18(8)R | 40? | 40? | 20? Sp | | | | |
| 2870 | " | CLYST, ol-gy+ mnr SST, brn-gy, calc | 5.0 - 5.5 | .59(5) .44(28)L .86(7)R | 20 | 70 | 10Sps Mnr Al | | | | |
| 2905 | " | CLYST, med-dk gy + tr SLIST, blk | 5.5 | .55(9) .46(25)L .99(20)R | 40 | 50 | 10Sps | | | | |
| 2935 | " | CLYST, ol-gy, aren+ tr SLTST, blk | 5.0 - 5.5 | .67(3) .45(15)L .97(14)R | 70 | 20 | 10Sps | | | | |
| 2977 | " | CLYST, brn-gy, aren+ tr SLTST, blk | 5.5 - 6.0 | .59(28) 1.06(21)R | 70 | 20 | 10 Sps | | | | |
| 2999.5 | " | CLYST, brn-gy, carb | 5.5 | .53(7) .44(14)L 1.04(31)R | 70 | 30 | * | | | | |

Maturity and Kerogen Composition Data

TABLE 1B

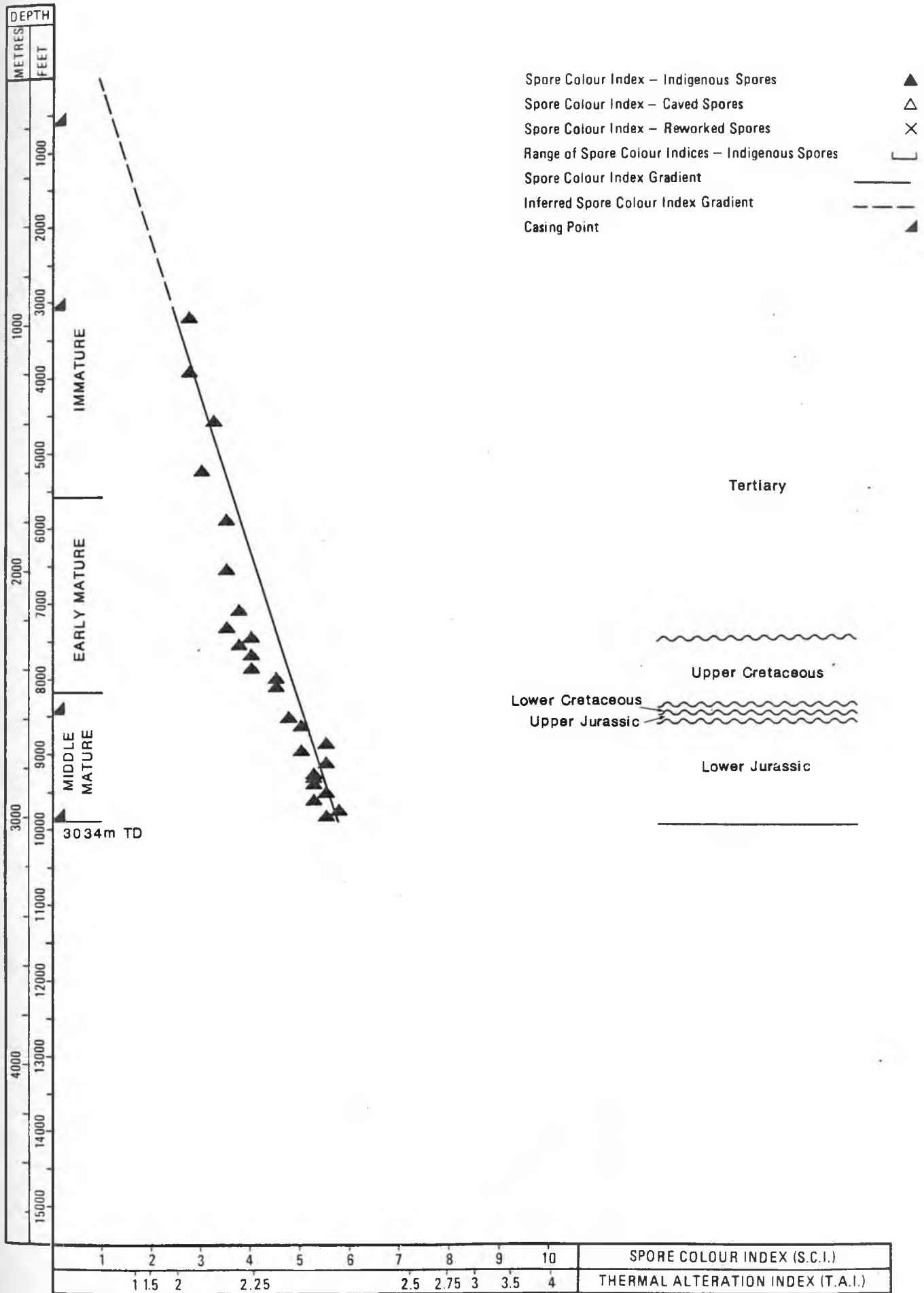


FIGURE 1 Spore Colour Indices against Depth

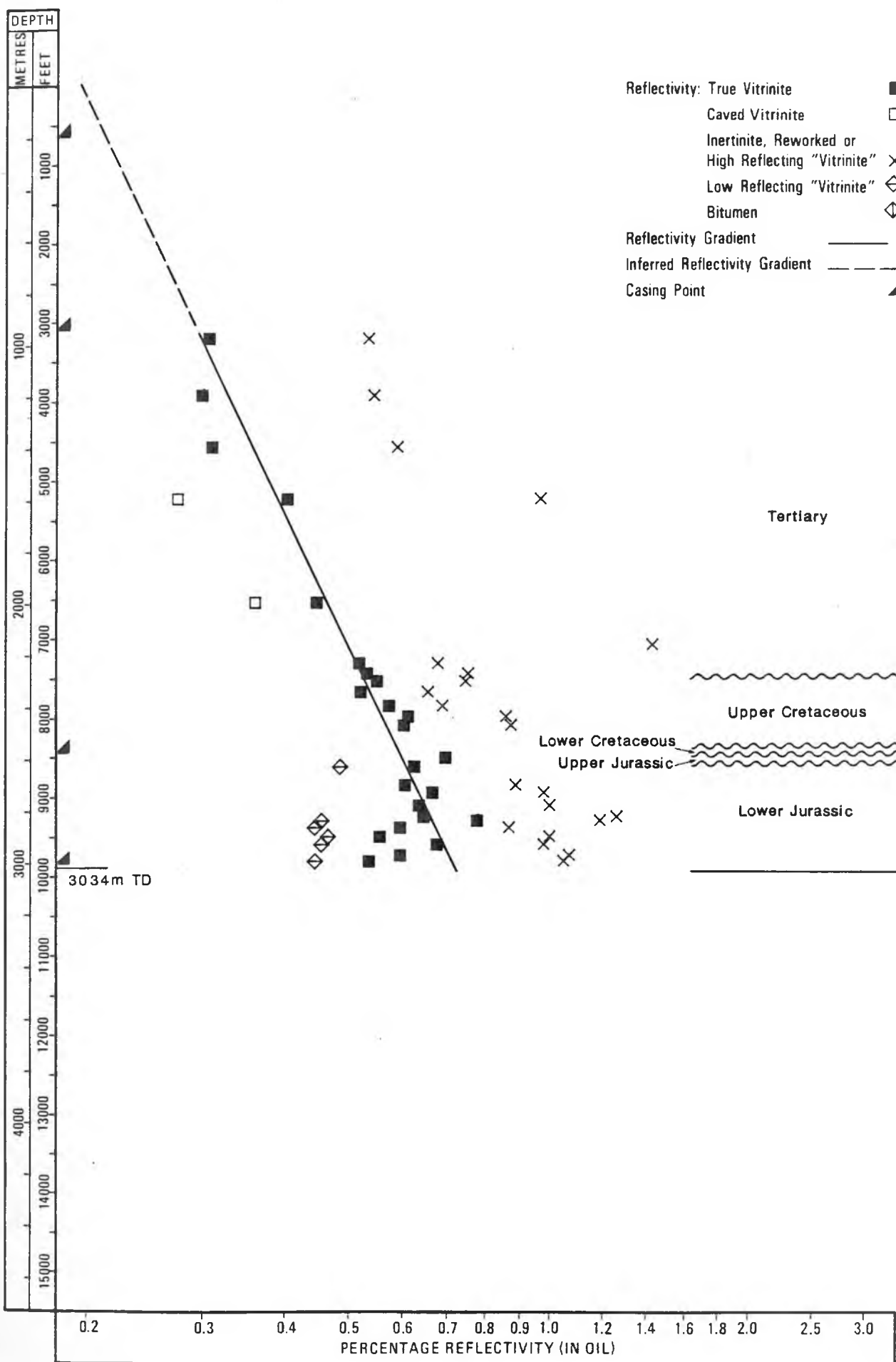


FIGURE 2 Vitrinite Reflectivity against Depth

APPENDIX 1
ABBREVIATIONS USED IN ANALYTICAL DATA SHEETS

| | | | | | |
|---------|---|---------------------------|---------|---|-------------------------------|
| a/a | - | as above | MDST | - | mudstone |
| Ac | - | acritarchs | med | - | medium |
| ADD | - | mud additive | MET | - | metamorphic rocks |
| Al | - | algae | mic | - | mica/micaceous |
| Am | - | amorphous | micr | - | micritic |
| ang | - | angular | min | - | mineral |
| ANH | - | anhydrite | mnr | - | minor |
| aren | - | arenaceous | mod | - | moderate |
| arg | - | argillaceous | ntl | - | mottled |
| BAS | - | basalt | n- | - | normal |
| bd | - | bedded/bedding | NA | - | not available |
| B(IT) | - | bitumen/bituminous | nod | - | nodule/nodular |
| bl | - | blue | NS | - | no sample |
| bld | - | bleached | occ | - | occasional |
| blk | - | black | ol | - | olive |
| bri | - | brilliant | ool | - | oolitic |
| brn | - | brown | orng | - | orange |
| calc | - | calcareous | OS | - | oil stain |
| CALT | - | calcite | P | - | picked lithology |
| carb | - | carbonaceous | pal | - | pale |
| CGL | - | conglomerate | Ph | - | phytane |
| CHK | - | chalk | pnk | - | pink |
| CHT | - | chert | por | - | porous/porosity |
| CLYST | - | claystone | pp | - | purple |
| CMT | - | cement | Pr | - | pristane |
| Comp | - | composite | pred | - | predominantly |
| crs | - | coarse | Prt | - | present |
| CSG | - | casing point/shoe | PYR/pyr | - | pyrite/pyritic |
| Ctgs | - | ditch cuttings | QTZ(T) | - | quartz(ite) |
| Cu | - | cuticle | Re | - | resin |
| C(vd) | - | caved | R(ew) | - | reworked |
| decarb | - | decarbonated | rnd | - | round(ed) |
| Di | - | dinocysts | Sap | - | sapropel |
| dk | - | dark | sbng | - | subangular |
| DLT | - | dolerite | sbrd | - | subrounded |
| DOL/dol | - | dolomite/dolomitic | SCI | - | spore colour index |
| dsk | - | dusky | Sf | - | semifusinite |
| Ex | - | exinite | sft | - | soft |
| Exs | - | exsudatinite | SH | - | shale |
| extr | - | extracted | shly | - | shaly |
| f | - | fine | sil | - | siliceous |
| fel | - | feldspathic | sks | - | slickenside surface |
| fer | - | ferruginous | SLA | - | slate |
| flu | - | fluorescence | SLT(ST) | - | silt(stone) |
| fm | - | formation | slty | - | silty |
| foss | - | fossils/fossiliferous | SND | - | sand |
| fr | - | friable | sndy | - | sandy |
| frac | - | fracture | Sp | - | spores |
| frags | - | fragments | SST | - | sandstone |
| Fu | - | fusinite | st | - | stained |
| GLC/glc | - | glauconite/glauconitic | stks | - | streaks |
| gn | - | green | suc | - | sucrosic |
| grd | - | graded/grading to | surf | - | surface |
| grns | - | grains | SWC | - | side wall core |
| gy | - | grey | TD | - | total depth |
| GYP | - | gypsum | TOC | - | total organic carbon |
| HAL | - | halite | tr | - | trace(s) |
| hd | - | hard | trns | - | transparent |
| hor | - | horizontal | v | - | very |
| H(RV) | - | high reflecting vitrinite | vgt | - | variegated |
| i- | - | iso- | Vit | - | vitrinite |
| I/b | - | inter-bedded | vn | - | vein |
| IGN | - | igneous rocks | VOLC | - | volcanic rocks |
| inc | - | including | VR | - | vitrinite reflectivity |
| Inert | - | inertinite | wht | - | white |
| lam | - | laminae/laminated | xln | - | crystalline |
| LCM | - | lost circulation material | yel | - | yellow |
| LIG/Lig | - | lignite/lignitic | - | - | no analysis carried out |
| lns | - | lens(es) | * | - | analysed but no data obtained |
| L(RV) | - | low reflecting vitrinite | gy-gn | - | greyish green |
| LST | - | limestone | gy/gn | - | grey-green (gradation) |
| lt | - | light | gn-gy | - | greenish grey |
| mass | - | massive | | | |

Note: (Maturity data tables only). Number in brackets refers to number of reflectivity values averaged to give quoted result. Preferred values for indigenous phytoclasts are listed first.

APPENDIX 2

ANALYTICAL PROCEDURES AND TECHNIQUES

This appendix summarises the main steps in the analyses carried out in the Robertson Research International Ltd. petroleum geochemistry laboratories. Analytical pathways are shown on the flow chart (Appendix Figure 1) and details of laboratory procedures and techniques are given in the text. These may in certain circumstances be adapted to suit particular samples or conditions. Interpretation guidelines are also defined.

1. Sample Preparation

General

Samples are received into the laboratories in the forms of well-site canned ditch cuttings, bagged ditch cuttings in various stages of preparation from wet, unwashed to dried, washed; sidewall cores, conventional cores, outcrop samples, crude oil samples and gas samples. Each sample is assigned a number which is entered into a computer system to monitor sample selection and progress. Preparation techniques are directed towards obtaining clean samples, free of drilling mud and mud additives, obvious caving contamination and indeterminate fine material. Washing with cold water is standard but further washing with solvent (dichloromethane, DCM) is carried out if oil-based mud is present, after which samples are dried, described and individual lithologies hand-picked where practicable. Samples are rough crushed to approximately pea-sized fragments for kerogen preparation or finely milled for chemical analysis.

Kerogen Preparation

Kerogen concentrates for microscopic examination and elemental analysis are prepared using standard palynological procedures but omitting oxidation or acetolysis. Acid maceration involves the use of hot hydrochloric acid (HCl) to remove carbonates and hot 60% hydrofluoric acid (HF) to remove or break down silicates. Mineral residues are separated from the kerogen by a combination of ultrasonic vibration and zinc bromide flotation. Kerogen samples for spore colour and kerogen typing are mounted on glass slides in glycerin jelly, those for vitrinite reflectivity are dried and mounted in epoxy resin. Kerogen residues are stored in methanol.

2. Maturity Evaluation

The techniques employed for interpreting maturity and thermal history in these laboratories are based mainly on spore colouration and vitrinite reflectivity measurement, supplemented by data obtained from airspace gas and gasoline analysis, pyrolysis Tmax, and hydrocarbon analysis including gas chromatography and gas chromatography-mass spectrometry.

Spore Colouration

Sporomorph colour is assessed using a >20 μ sieved kerogen fraction viewed in transmitted light on a standard palynological microscope. Unusual hues are checked using incident blue/UV light fluorescence. Measurement is made by eye against reference sets of single grain spore mounts and trained operators achieve a high degree of accuracy and reproducibility. The 1 to 10 Spore Colour Index (SCI) scale was designed for linearity with increasing depth and temperature and correlates approximately with the following zones of oil generation: 1.0 to 3.5, immature; 3.5 to 5.0, early mature, generation of low gravity oils (28 to 35 °API); 5.0 to 7.0, middle mature, generation of medium gravity oils (35 to 42°API); 7.0 to 8.5, late mature, generation of light oils (>42°API) and condensates; 8.5 to 10, post mature, generation of condensate, wet gas and, ultimately, dry gas. Linearity of scale is of great value in prediction, by extrapolation, of the depth to any part of the oil generation sequence. The value of SCI measurement lies in the objective selection of measured grains, so minimising problems of caving and reworking, and in its more direct correlation against oil generation than vitrinite reflectivity measurement. Limitations in its use concern the difficulty of correlation against other colour scales and the insensitivity of the scale in the late to post mature region. Anomalous colours may result from bleaching or staining during deposition and diagenesis. The correlation of SCI against Thermal Alteration Index (TAI) given on the SCI versus depth plot in the reports was made by direct comparison of Staplin's standard slides with SCI standard slides.

Vitrinite Reflectivity

The majority of preparations examined under reflected light in these laboratories are made using >20 μ sieved kerogen, mounted in resin blocks and polished with carborundum and alumina although total kerogen may be used when sample size is

limited. Picked coals, organic-rich shales or limestones containing solid bitumen are mounted directly in resin blocks and polished in the usual way. Measurement is made on a Leitz Orthoplan microscope fitted with an MPV Compact photometer which feeds values direct to a desk top computer for data processing from each sample. The system is calibrated against glass standards and reflectance values are expressed as arithmetic means of measurements taken in oil immersion (R_o or $R_{m \text{ oil}}$). R_{max} and R_{min} may be measured and quoted in certain circumstances but the difference is insignificant below about R_o 1.0%. Some operator selection of particles during measurement is essential and obvious contaminants or non-vitrinitic material are noted but not necessarily quoted. The value quoted on data tables is that which is interpreted as most appropriate, but other possibilities may also be given. Plotted figures assume a logarithmic increase of reflectance with depth. R_o 0.5% is a widely accepted threshold value for the onset of oil generation, although as the kinetics of oil generation may not be identical to those of vitrinite reflectivity development this must be seen only as a general guide. The floor for oil generation is characterised by a reflectance value of about 1.3%. Wet gas generation peaks at a value of about 1% and ceases at the 2% level. Dry gas generation peaks at a reflectance of about 1.5% and ceases at the 3% to 4% level. Correlation of reflectance values with other maturity parameters may not be universal because of time-temperature factors and is best made on a local basis.

Reflectivity measurement is a widely used and versatile tool which may be readily calibrated against easily obtained standards. It is applicable over a wide range of maturity stages from immature to post mature (0.2% to 5% R_o). High surface intercepts on plotted figures and discordances at faults and unconformities can give realistic estimates of the amount of section missing. It is of limited value in Early Palaeozoic sections where land plant material is absent, although a general guide to maturity may be obtained from chitinous organic matter. Even a skilled operator may have difficulty in distinguishing indigenous vitrinite from some forms of inertinite, anomalously reflecting "pseudovitrinite", cavings and reworked fragments.

Airspace Gas Analysis

Wet cuttings are collected at the well site and sealed in partly full cans containing bactericide. In the laboratory, the airspace (headspace) gas is extracted using a can piercer fitted with a septum and analysed by gas chromatography. The proportions of methane, ethane, propane, iso- and n-butane are calculated from integrated peak areas by comparison with a standard mixture of these gases. Methane is the dominant gas in immature and post mature sediments, comprising 90-100% of total gas, falling to 30-70% in mature sediments. The onset of maturity for oil generation (SCI 3.5) is characteristically marked by an increase in wet gas (C_2-C_4) to between 10 and 20% with further increases in maturity indicated by a decrease in the ratio of iso- to n-butane. Ratios of >1.0 are typical for immature sediments and <0.5 are usual in mature sediments. Departures from composition versus depth trends may be useful in indicating migrant gas at faults, unconformities or reservoir rocks but limit the method as a reliable maturity indicator. Airspace gas analysis is an inexpensive and rapidly executed method of screening samples for further maturity and hydrocarbon content determinations.

Gasoline Analysis and Cuttings Gas Analysis

Cuttings samples received wet, preferably in sealed containers, are suitable for gasoline and cuttings gas analysis. A portion of the washed cuttings sample is retained wet, pulverised in a sealed shaker and warmed to expel the C_1 to C_7 hydrocarbon components into the shaker airspace. A sample of this airspace gas is then removed and analysed by gas chromatography either for cuttings gas (C_1 to C_4) or gasolines (C_4 to C_7). Up to 28 hydrocarbon components are identified in the C_4 to C_7 range and their relative proportions calculated from integrated peak areas with reference to standard mixtures. Immature source rocks yield low total abundances and limited numbers of components whereas mature source rocks usually contain a full complement of identified hydrocarbons with the onset of maturity indicated by a rapid rise in total gasoline abundances with depth. Anomalous amounts of gasolines may mark the presence of oil stain. Gasolines may be used in oil to oil or oil to source rock correlations but the concentration of some of the measured components is not only a function of source but also depends on maturity, migration and alteration in the reservoir. Using the most stable compounds, pairs with similar chemical structure and boiling points are reduced to pair ratios and compared with the same pair ratios in other oils or possible source rocks. Gasoline analysis is a valuable tool in that it measures directly the hydrocarbons being generated from a sediment but its sensitivity in detecting traces of oil places constraints on its use as a maturity indicator.

Rock-Eval Pyrolysis, Gas Chromatography (GC) and Gas Chromatography-Mass Spectrometry (GC-MS) in Maturity Analysis

These three analytical processes measure parameters which are functions of both maturity and kerogen type. Data from them may give a general guide to maturity but if the kerogen types are known, more specific conclusions may be drawn. From Rock-Eval data, the temperature of maximum rate of pyrolysis, T_{max} , is the most useful datum; gas chromatograms of alkanes, separated from source rock extracts or oils, yield carbon preference indices (CPI) and isoprenoid ratios; GC-MS quantitative fragmentograms provide abundance ratios for specific compounds which are particularly useful in assessing the level of maturity at which source rock hydrocarbons or oils have been generated. All these supplementary data may be used to confirm results from visual analysis or supplant them if poor or unavailable.

3. Source Rock Evaluation

Total Organic Carbon Content (TOC)

Organic carbon values are obtained by treating 0.1g of crushed rock sample with hot, concentrated HCl to remove carbonates. The washed residue is filtered on to a glass fibre pad and ignited in a Leco carbon analyser. For screening purposes, samples are analysed singly but where further analyses, such as pyrolysis or solvent extraction are anticipated, a duplicate sample is run. Blanks and standards are run as routine and where values from duplicated samples do not concur within strict accuracy limits, they are rerun. Where samples are heavily stained with oil, either from natural deposits or drilling mud, TOC is repeated on the dried, solvent extracted sample.

TOC measurement is fundamental in assessing source rock quality since when combined with kerogen type and maturity, a full description of the potential to generate oil may be given. It is found in practice that sediments containing less than 0.3% TOC are unlikely to have any source potential, those containing between 0.3% and 1% may be marginal sources but the better quality sources contain in excess of 1% TOC. Screening by TOC is therefore an inexpensive and rapid method of selection of samples for further analysis in source potential evaluation.

Rock-Eval Pyrolysis

Pyrolysis data are obtained using the IFP-Fina Rock-Eval apparatus. 100 mg of crushed, whole rock either from bulk sample or picked lithology is weighed accurately into a crucible and introduced into a furnace at 250°C. Free hydrocarbons (roughly equivalent to solvent extractable hydrocarbons) are volatilised and quantified by flame ionisation detector (FID) to give Peak 1 (S_1 , ppm). The furnace temperature is increased to 550°C at 25°C/minute and within this range, kerogens crack to give hydrocarbons, measured by FID to give Peak 2 (S_2 , ppm) and carbon dioxide, measured by thermal conductivity detector (TCD) to give Peak 3 (S_3 , ppm). The temperature at the maximum rate of evolution of cracked volatiles (T_{max}) is measured automatically but can also be monitored visually. The instrument is calibrated daily using standards both at the beginning of the work period and at regular intervals thereafter and crucible blanks are run as routine. The tabulated data in reports comprise the following parameters:

- T_{max} °C - temperature of maximum rate of Peak 2 hydrocarbon evolution.
- Hydrogen Index (HI) - S_2/TOC (mg/g) or ratio of released hydrocarbon to organic carbon content. This is a measure of the hydrocarbon generating potential remaining in the kerogen as opposed to that of the whole rock.
- Oxygen Index (OI) - S_3/TOC (mg/g) or ratio of released carbon dioxide to organic carbon content.
- Production Index (PI) - S_1/S_1+S_2 , or ratio of the amount of hydrocarbons released in the first stage of heating to the total amount of hydrocarbons released and cracked during pyrolysis.
- Potential Yield (PY) - S_2 (ppm) or total of hydrocarbons released during cracking of kerogen compared to original weight of rock.

T_{max} , hydrogen index and oxygen index are each functions of both maturity and kerogen type. Using published and empirical data, it has been possible to assemble a model to show the relationships of these factors to maturity as measured by spore colouration and vitrinite reflectivity for a selection of pure kerogen types. The kerogen types used are algal sapropel (type I), waxy sapropel (type II), vitrinite (type IIIA) and inertinite (type IIIB) and a computer program has been devised by which the amounts of these components may be calculated from the HI, OI, T_{max} and maturity

data for any sample. These are the values expressed in the "kerogen composition by calculation" columns tabulated in the reports.

The hydrogen index is a measure of the hydrocarbon generating potential of the kerogen and is analogous to the atomic H/C ratio. Immature, organically rich source rocks and oil shales give values above 500, mature oil source rocks give values between 200 and 550. For a given kerogen type, these values progressively diminish with increasing maturity.

The temperature of maximum rate of pyrolysis depends partly on the kerogen type but the transition from immature to mature organic matter is marked by temperatures between 415° and 435°C. The maturity transition from oil and wet gas generation to dry gas generation is marked by temperatures between 455° and 460°C. In practice, greater variation than these ideal temperature ranges may be seen, but they are nevertheless useful as general guides to the level of maturity attained by the sediment.

The production index increases with maturity from values near zero for immature organic matter to maximum values of 0.15 during the late stages of oil generation. Anomalously high values indicate the presence of oil or contaminants. The potential yield is an indication of the predicted yield of hydrocarbons from the source rock at optimum maturity and is a measure of the quality of the source rock. For immature sediments, values of 0 to 2000 ppm of hydrocarbon characterise a poor source rock, 2000 to 6000 ppm fair, 6000 to 20 000 ppm good and above 20 000 ppm very good.

Pyrolysis techniques have in recent years provided a major advance in the assessment of source rock quality and generating potential. Hydrocarbon yields from immature source beds examined on-structure may be translated into actual oil productivity from the same beds in mature basinal, off-structure situations. Models relating maturity and kerogen type may be used to define original source rock quality grades which are of great value in mapping organic facies. Amorphous kerogen types, indistinguishable in microscopic preparations over a wide range of chemical properties, may be readily differentiated by pyrolysis. The problem of analysing bulk samples containing mixed kerogens has been largely overcome by the kerogen type/maturity model and anomalous results arising from the presence of caving contamination and drilling mud additives can usually be explained by inspection. High oxygen indices sometimes occur as a result of the presence of metastable carbonates and in such cases the sample is acid decarbonated and re-run.

Visual Examination of Kerogen Concentrates

All palynological preparations on which SCI determinations are made are also examined for kerogen type. Visual estimations of the relative abundance of the broad groups vitrinite, inertinite and sapropel are made on the total kerogen slide mount but reference is also made to the >20 μ sieved fraction to assist in identification. The scheme of identification is shown in Appendix Table 1. Full use is made of incident blue or UV light in distinguishing immature or early mature oil-prone kerogen from gas-prone kerogen.

Extract Analysis

The soluble organic materials present in rocks can be extracted with organic solvents, fractionated and analysed. The type and amount of material extracted depends largely upon the nature of the contained kerogen and its maturity, although the presence of migrant oil or drilling contamination may be the determining factors.

A maximum of 40g of crushed sample is extracted for a minimum of 12 hours in a Soxhlet apparatus using laboratory redistilled DCM. The solvent and the more volatile components (approximately up to n-C₁₅) are lost by evaporation in an air flow and the resulting total extract is weighed, dissolved in hexane and separated into alkane (saturate) hydrocarbon, aromatic hydrocarbon, resene and asphaltene (polar) fractions by silica adsorption chromatography in the Iatroscan process.

Larger fractions, suitable for further analysis, are obtained by column chromatography. The extract is run through a short glass column packed with silica and alumina and eluted with hexane (to give the saturate fraction), (3:1 hexane: toluene mixture (to give the aromatic fraction) and methanol (to give the polar, or resene and asphaltene, fraction). A small proportion of non-eluted polar compounds usually remains on the column.

The data tabulated in reports comprise the following parameters:

Total extract - soluble organic matter, heavier than about $n-C_{15+}$, expressed as ppm of weight of rock.

Hydrocarbons - sum of alkane and aromatic hydrocarbons, expressed as ppm of weight of rock.

Extract % of organic carbon (EPOC) - $\frac{\text{total extract ppm}}{\text{TOC} \times 100}$; the extractability.

Hydrocarbons mg/g of organic carbon - total hydrocarbons normalised to 1g of organic carbon.

Hydrocarbons % extract - total hydrocarbons as a proportion of total extract.

Alkanes % hydrocarbons - the proportion of alkanes (saturates) in the total hydrocarbons. The proportion of aromatics is (100 minus this value) expressed as a percentage.

The extractability of oil-prone sapropelic organic matter increases rapidly in the oil generation zone and diminishes to very low values in post mature sediments. Overall the extractability of sapropelic organic matter is greater than that of gas-prone humic organic matter for similar levels of maturity. Samples with extractabilities of greater than 20% generally contain migrant oil or are contaminated with mud additives.

As maturation proceeds in the oil generation zone the proportion of hydrocarbons in the total extract increases from less than 20% to a maximum in the most productive horizons of around 60%. This trend is reversed as the oil-condensate zone is entered. The relative proportions of alkanes to aromatics can be used as a check for low levels of contamination. Fractions of the extract, separated by column chromatography are retained for further analysis by gas chromatography or for stable carbon isotope determination.

Capillary Gas Chromatography of C_{15+} Alkanes

A portion of the Soxhlet extract is eluted with hexane through a short silica column to yield the saturate hydrocarbon fraction. This fraction is evaporated in a stream of dry nitrogen at room temperature. A small portion of the fraction is then taken up in hexane and introduced into a 25 metre, wall-coated, open tubular glass capillary column coated with OV-1, or equivalent, mounted in a Carlo Erba gas chromatograph which is temperature programmed from 70°C to 270°C at 3°C/minute.

C_{15+} chromatograms are inspected for the distributions of n -alkanes, and the presence and abundance of isoprenoids (particularly pristane and phytane), steranes and triterpanes and unresolved envelopes of naphthenic compounds. The ratios pristane:phytane and pristane: $n-C_{17}$ are calculated. Carbon Preference Index (CPI) values quoted are those as defined by Philippi as the ratio $2C_{29}$ to $(C_{28}+C_{30})$ unless otherwise stated. Chromatography may reveal information about the kerogen type of the source rock, its maturity and condition of deposition and, if migrant oil is present, whether this has been water-flushed or biodegraded. Contaminant drilling mud additives may be identified.

Capillary Gas Chromatography of Aromatic and Branched/Cyclic Alkanes

The aromatic portion of the Soxhlet extract is eluted from a short silica/alumina column by a hexane/toluene mixture. The dried fraction is taken up in DCM and introduced into a 25 metre, wall-coated, open tubular glass capillary column coated with OV-1, or equivalent, mounted in a Carlo Erba gas chromatograph which is temperature programmed from 70°C to 270°C at 3°C/minute.

Branched chain alkanes are separated from normal alkanes by urea adduction and treated as for total alkanes.

Gas Chromatography-Mass Spectrometry

Mass spectrometry is a technique in which molecules are bombarded with high energy electrons causing ionisation and fragmentation of the molecules into ions of varying mass(m) and charge(z). The way in which a molecule fragments into ions of various m/z value is known as its fragmentation pattern, or mass spectrum and is unique. When linked to a gas chromatograph the mass spectrometer can be used in two different modes:

1. Full Scan Mode: A mass spectrum is obtained of each peak eluting from the gas chromatograph and a structural identification of the compound producing that peak can be made.
2. Multiple or Single Ion Monitoring Mode: The mass spectrometer is tuned to certain m/z values to detect whether a compound, eluting from the gas chromatograph, fragments to give an ion at that value. Certain fragmentations are indicative of specific compound types and the most commonly monitored fragment ions used in petroleum geochemistry are those with m/z values of 191, 217 and 259 which are the principal fragment ions obtained from groups of alkanes known as triterpanes, regular steranes and rearranged steranes respectively. These are compounds containing 27 to 35 carbon atoms arranged in a polycyclic, normally 4 or 5 ring, structure, occurring in the n-C₂₆ to n-C₃₅ region of a gas chromatogram. The basic molecular skeletons of these compounds are very similar to those of the original organic matter deposited in the sediment and so these 191, 217 and 259 distribution plots, known as mass fragmentograms or mass chromatograms, form a pattern characteristic of the source material. This technique of "fingerprinting" is also one of the more exact methods of correlating an oil to its source, or to another oil.

Carbon Isotope (¹³C/¹²C) Ratio Analysis

Carbon has two stable isotopes, the more abundant ¹²C isotope and the heavier ¹³C isotope, which in nature forms about 1% of carbon. Deviations from the ¹³C/¹²C ratio are extremely small and carbon isotope ratios, as measured by mass spectrometry, are expressed as deviations from a standard, the Pee Dee Belemnite carbonate (PDB standard) in parts per thousand (parts per mil; ‰). Positive deviations indicate ¹³C enrichment and conversely, negative deviations indicate ¹³C impoverishment.

While the carbon isotope ratios of oils and rock extracts can range from -20 to -32 ‰ depending on the source organic matter type, the difference between a specific oil and its source is small. Measurements are usually made on the C₁₅₊ alkane and aromatic hydrocarbon fractions separately and there should be no more than 1 ‰ difference between the oil and its source for either fraction. If there is any doubt that the source rock extracts are not indigenous to the source rock kerogen, the carbon isotope ratio of the extracted source rock kerogen can be measured.

Pyrolysis-Gas Chromatography

The hydrocarbon pyrolysate derived from thermal, anhydrous cracking of kerogen is analysed by capillary gas chromatography. A few mg of rock, kerogen or asphaltene is heated to 600°C for 20 seconds in the injector of a gas chromatograph. The chromatograph oven is kept at -30°C during pyrolysis and then raised to 300°C at a programmed rate of 7.5°C/minute. Chromatograms produced this way are often very different from those of source rock extracts or oils in that branched and cyclic isomers are generated freely giving numerous, closely spaced peaks, along with unsaturated, alkene (olefin) hydrocarbons. The "doublet" peaks often observed in these chromatograms comprise alkene-alkane pairs, the first eluting, and usually smaller peak, being the alkene. The chromatograms range from C₁ to C₃₀ or above and although variable, are broadly characteristic of source rock type. Gas-prone kerogen cracks to give a more limited molecular weight range of products, concentrated towards the light ends, whereas oil-prone kerogen gives more prominent alkene-alkane doublets in the C₁₂ to C₃₀ region. The largest peak from both types is usually methane.

Elemental Analysis

Total (unsieved) kerogen is prepared as described in Section 1. The dried material is combusted in oxygen in an elemental analyser and the oxides of carbon, hydrogen, nitrogen and sulphur are measured. The unburnt residue is the ash content. Oxygen is usually calculated by difference but can be determined separately if required. Results are quoted as percentage weights of C, H, O, N, S and Ash with the atomic ratio H/C and O/C calculated and plotted on the standard van Krevelen diagram. The relative amounts of C, H and O present in organic matter are dependent on both source and maturity. At known maturity levels, some measure of source quality may be determined. Limitations of the method in source rock assessment involve the difficulty of obtaining pure kerogen (in particular, free from pyrite) and the lack of a simple, direct determination of oxygen content.

4. Oil Analysis

RRI laboratories offer a wide range of oil analyses both for geochemical purposes and industrial use. Physical property determinations are based mainly on IP methods and are available for lubricating oils, fuels and greases as well as crudes. Frequently measured properties of crude oils presented in geochemistry reports include: API gravity, pour point, viscosity and contents of water, sulphur, wax, asphaltene, nickel, vanadium and other metals. Chemical analysis of oils involves the following:

Whole oil gas chromatography - using split syringe injection and a temperature programme from -20°C or -30°C up to 270°C at 4°C/minute.

Associated gas - if oil has high gas/oil ratio.

Gasoline analysis - as for gasolines in rock samples but a weighed quantity of oil is used.

Topping of the oil - this is equivalent to the removal of the fraction boiling below about 210°C and gives a more standardised product for comparison of gas chromatograms of the C₁₅₊ fraction.

Column chromatography and gas chromatography - as for solvent extracts. Analysis is carried out on topped oil.

5. Gas Analysis

The hydrocarbon gases, C₁ to C₄, may be collected from the airspace of sealed canned samples or may be received from well-site tests in a special sealed gas cylinder (gas mouse). Chromatographic separation of the C₁ to C₄ gases is effected as described under airspace gas analysis. In addition, the separated gas components may be analysed for stable carbon and hydrogen isotope composition which may provide valuable clues to the origin of the gas.

6. Solid Bitumen Analysis

In some oil fields, problems are encountered where bitumen developments form continuous or patchy layers within reservoirs, dividing the pay zones and acting as barriers to natural fluid movement or inhibiting enhanced oil recovery techniques. Integrated geochemical and sedimentological studies aim to produce geological models capable of predicting the occurrence of bitumen layers and their likely thickness and ability to act as permeability barriers. Of further concern are the past or present relationships between the bitumen and reservoir oil, their source rocks and the timing of bitumen formation.

Analysis schemes involve screening of samples by assessing the amount of bitumen in polished core pieces using reflected light microscopy, followed by solvent extraction of control samples to estimate the proportion of solvent soluble bitumen. Different phases of bitumen formation are differentiated by reflectance measurement as described for vitrinite reflectance measurement. Soluble extracts are fractionated to give alkane, aromatics, asphaltene and resene components. Separated bitumens may be subjected to elemental analysis.

Kerogen Typing Scheme for Transmitted White and Incident Blue/U.V. Light

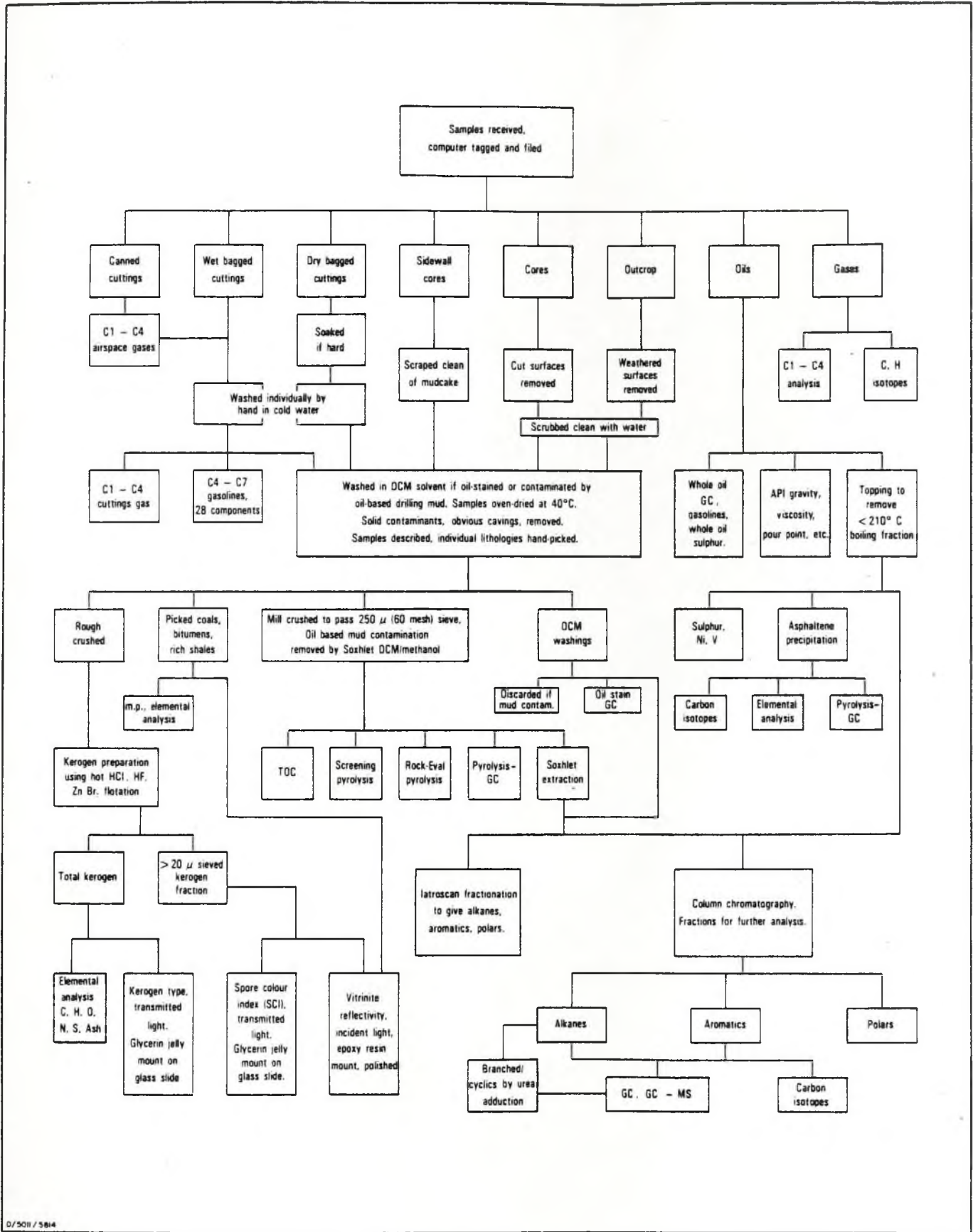
| | | |
|--|------------------------|-----------|
| General Properties | RRI Report Data Tables | Type * |
| Sapropelic (Oil-prone gas-prone at high maturity) | Algal Sapropel | Type I |
| | Waxy Sapropel | Type II |
| Humic (Gas-prone) | Vitrinite | Type IIIA |
| | Inertinite | Type IIIB |

| Amorphous | | Structured | |
|--|---|---|--|
| Non-Fluorescent | Fluorescent | Non-fluorescent | Fluorescent |
| Type I/II at high maturity (SCI >7.5) | Type I Sapropel Type II (degraded spores) Soft bitumens | Vitrinite (Type IIIA) brown/black, woody tissue | Cuticle Spores Pollen Dinocysts (Type II) |
| Type IIIA/B | | | |
| Oil residues (bitumens) Mineral (undigested) Grease contamination Mud additives | | Inertinite (Type IIIB) very dark brown/black, woody tissue | Resinite Algae (Tasmanites, Botryococcus etc.) |
| | | Solid bitumen - brown/black (oil residue) often with crystal imprints | (Type I) |
| | | Microforaminifera, chitinozoa etc. (Not usually important) | |
| | | Spores, cuticle etc. at high maturity levels | |
| | | Mud Additives - walnut etc. | |

* Types I, II, III approximately sensu Tissot et al but Type III subdivided into IIIA (vitrinite) and IIIB (inertinite)

APPENDIX TABLE 1

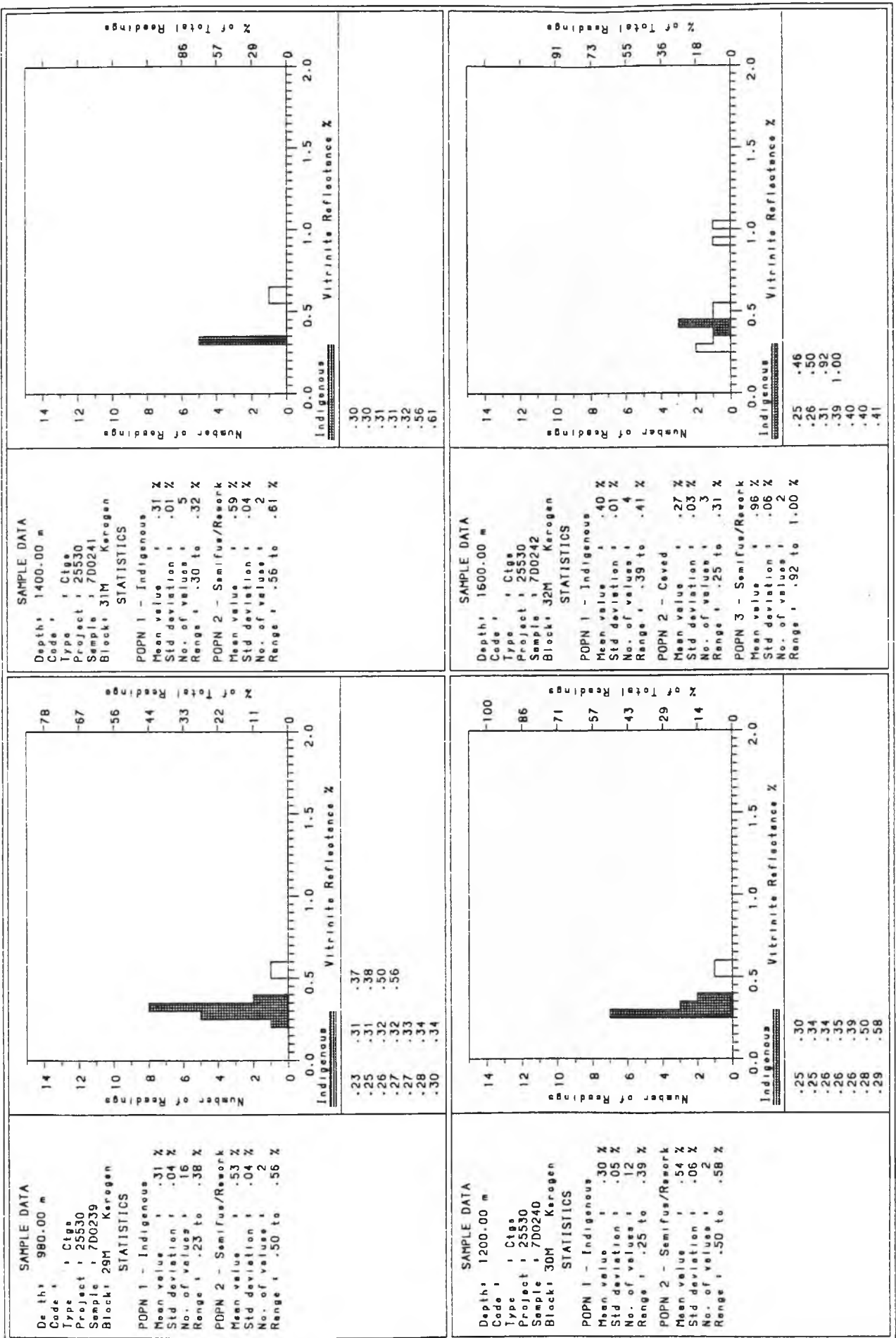
FLOW CHART FOR GEOCHEMICAL ANALYSIS

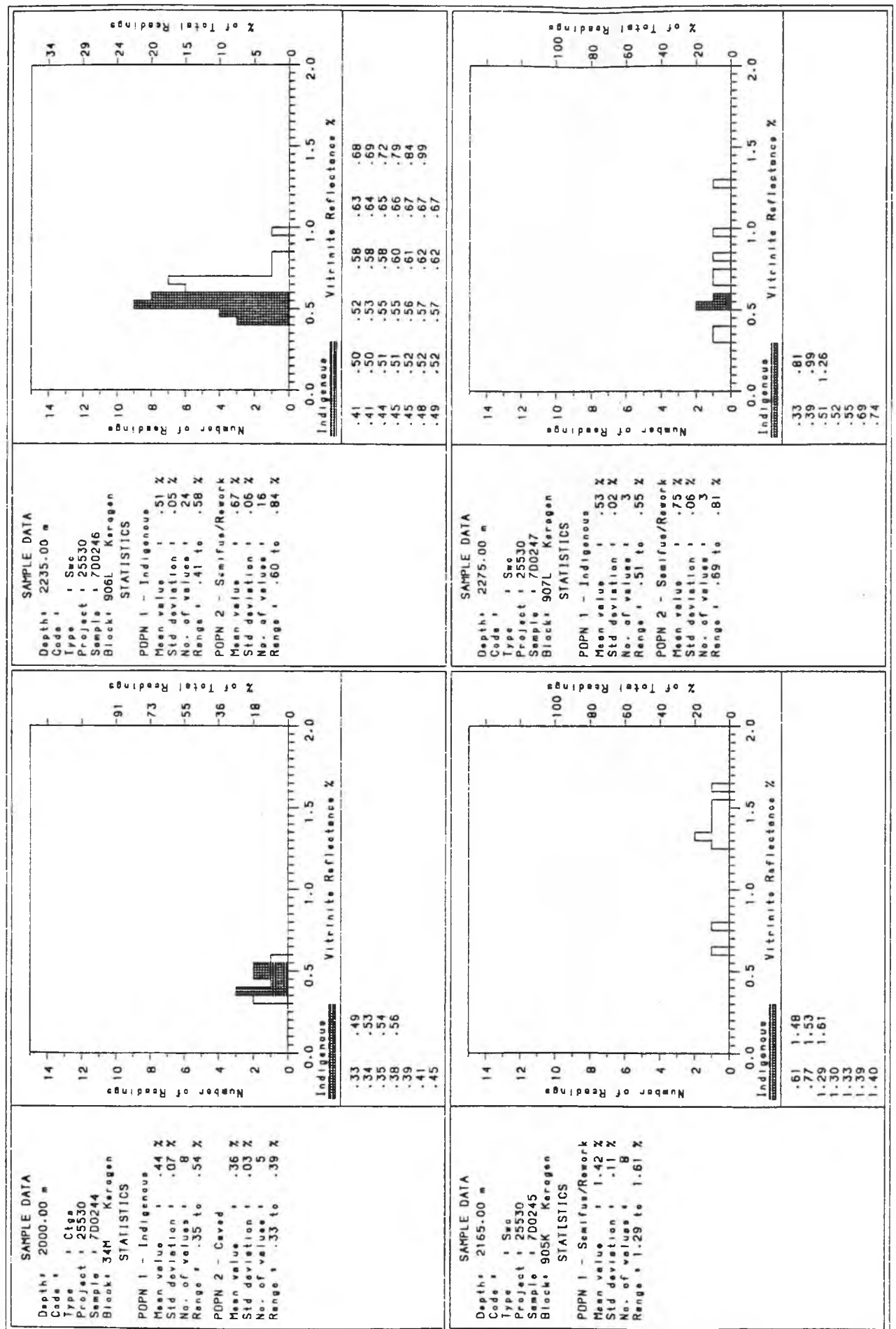


APPENDIX FIGURE 1

COMPANY : NORSK HYDRO
 WELL : 30/9-6
 LOCATION : NORWEGIAN NORTH SEA

HISTOGRAMS, DATA AND STATISTICS
 FOR VITRINITE REFLECTIVITY

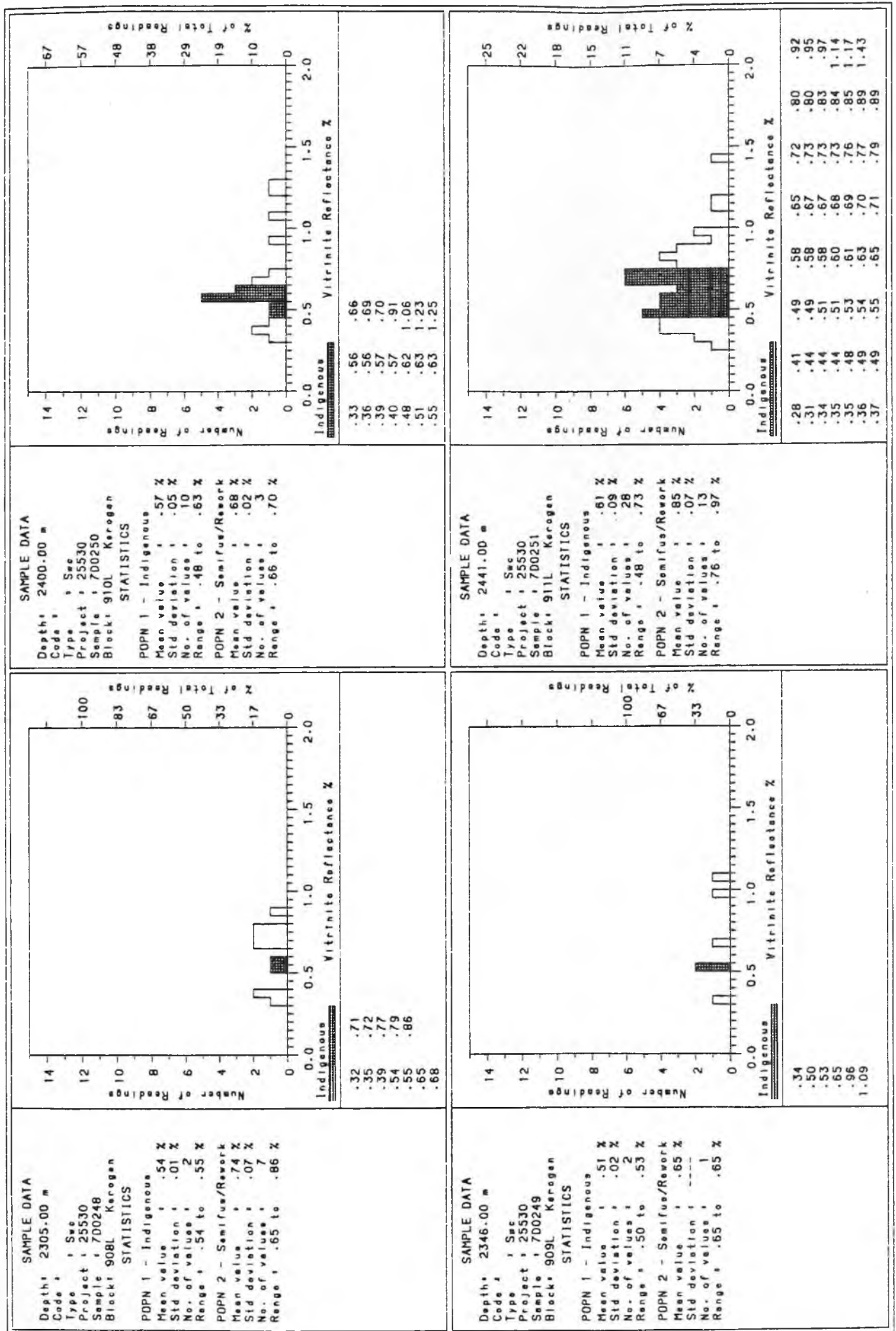




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HISTOGRAMS, DATA AND STATISTICS
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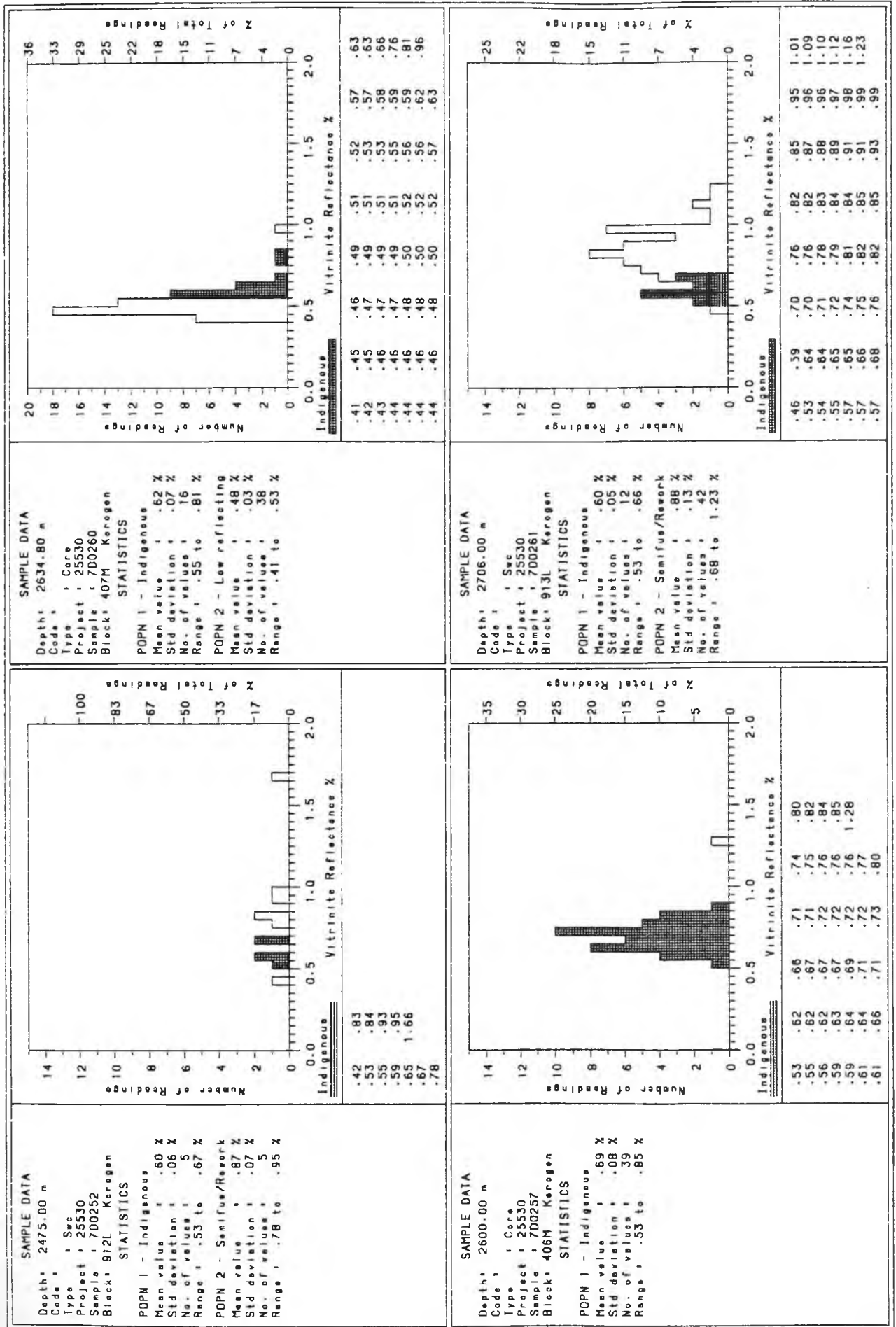




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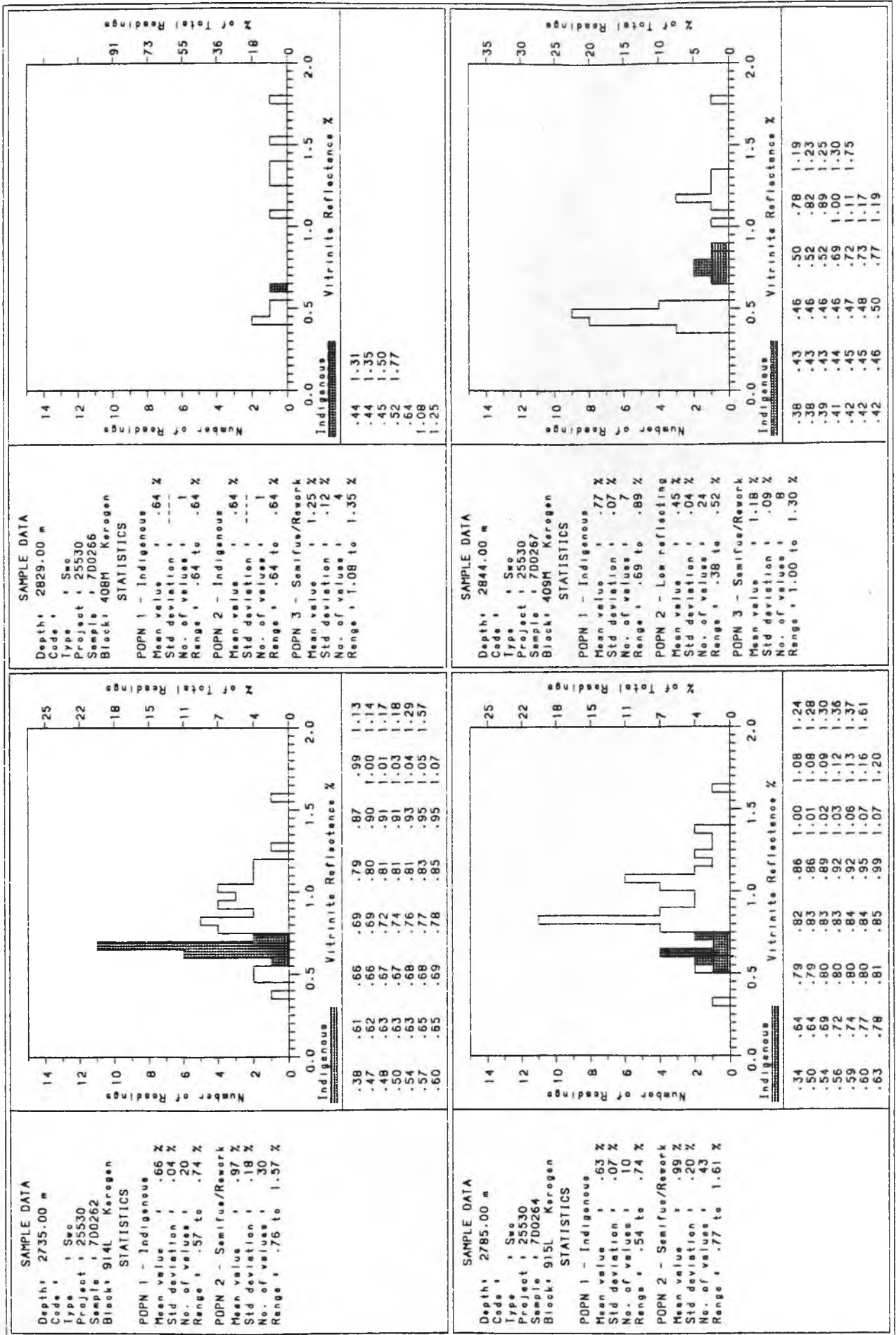




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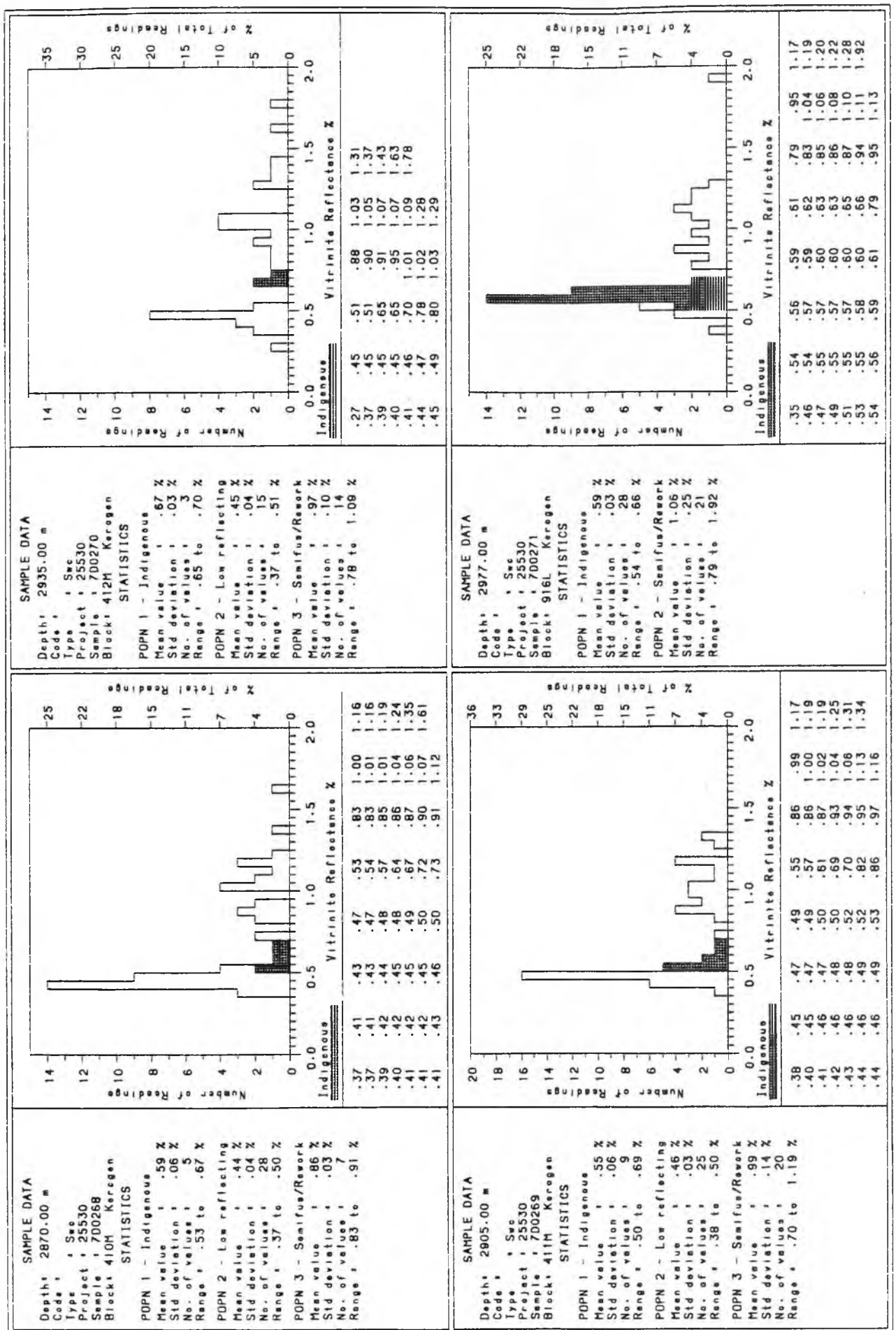




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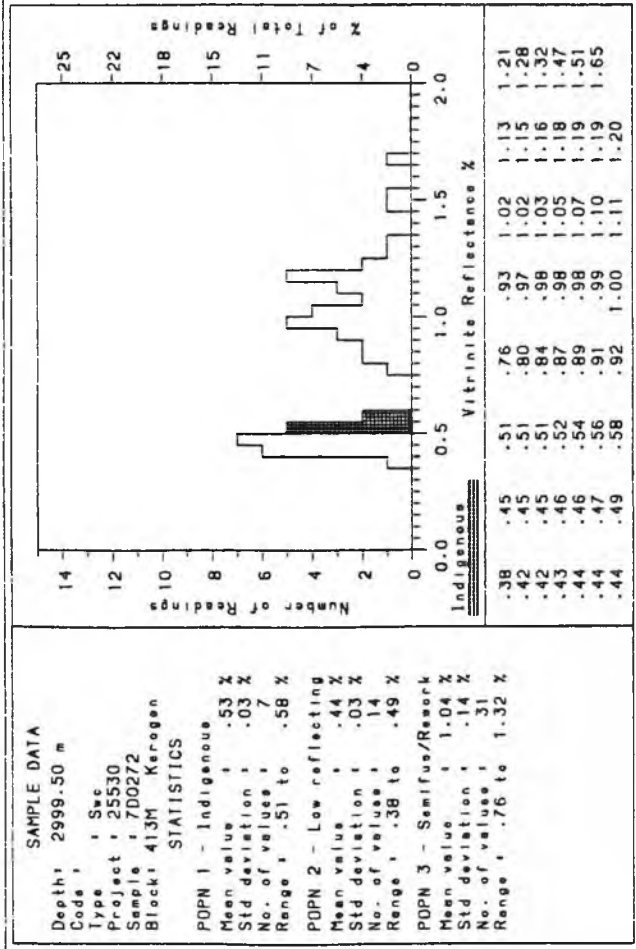




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HISTOGRAMS, DATA AND STATISTICS
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