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INSTITUTT FOR KONTINENTALSOKKELUNDERSØKELSER

# CONTINENTAL SHELF INSTITUTE

| CLIENT<br>Norsk Hydro       | REPORT TITLE<br>ROCK-EVAL PYROLYSIS OF SAMPLES FROM<br>WELL 30/7-7. |             |  |  |  |
|-----------------------------|---|-------------|--|--|--|
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SUMMARY

Rock-Eval pyrolysis has been undertaken on a number of samples from well 30/7-7 to evaluate the type of kerogen and the maturity.

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OLJEDIREKTORATE

KEY WORDS

Pyrolysis

| Source | Ruck |
|--------|------|
|        |      |

#### EXPERIMENTAL

100 mg of fresh crushed sample was analyced on a Rock-Eval pyrolyses instrument.

#### ROCK-EVAL PYROLYSES THEORY.

#### ABSTRACT

A procedure and apparatus have been developed for rapid source-rock characterization. Based on the pyrolysis of small rock samples, it makes possible the determination if:

- various types of source rocks
- their degree of evolution (immature, oil zone, gas zone)
- their petroleum potential

The method can be performed on cores or cuttings either in the laboratory or at a well site (especially aboard offshore drilling vessels), and in this case it can be used to dedect oil shows.

Generally the procedure uses ground rock, but it is also possible to operate directly with small cuttings without any prior treatment.

Aboard the GLOMAR CHALLENGER (LEGS 48 and 50) the apparatus was especially used as a safety tool to reval rapidly the presence of oil shows.

#### INTRODUCTION

In the field of petroleum exploration, one of the most important aspects is to be able to recognize series which, under the effect of increasing temperature during burial, produce petroleum compounds.

As a matter of fact, the quantity and character of the hydrocarbons which have been produced depend on the character of the organic matter originally deposited in these source rocks as well as on subsequent thermal evolution, i.e. temperature and geological time. Recent research 1, 2, 3 on kerogens (insoluble organic matter) from sediments sampled in various basins has shown that the physico-chemical analysis of these kerogens makes it possible to classify the various types of organic matter and to estimate their oil and gas potential together with their degree of evolution.

However these methods are long and costly and do not fulfull the requirements of petroleum exploration which requires the analysis of a large number of sediment samples by simple, cheap and rapid methods. The basic knowledge acquired from the study of various types applied to characterize the different qualities (types) of organic matter directly on sedimentary rocks and to estimate their oil potential and their degree of evolution.

This method is based on the selective detection of hydrocarbon compounds and of one of the principal oxygenated compound  $(CO_2)$  produced by pyrolysis under normalized conditions of organic matter contained in sediments. The pyrolysis technique has already been used by various authors  $({}^4$ ,  ${}^5$ ,  ${}^6$ ,  ${}^7$ ,  ${}^8$ ) for studying organic matter in sedimentary rocks.

#### CHARACTERIZATION OF VARIOUS TYPES OF KEROGEN

Elementary analysis of kerogens shows that the major atomic constituents are carbon, hydrogen and oxygen. The H/C and O/C atomic rations utilized in a diagram of the type established by Van Krevelen for coal shows that the samples corresponding to the same quality of organic deposits are situated on a curve called "evolution path". (fig. 1).

In this diagram, kerogens of type I (algal kerogen's Green River Shales, etc.) shows a high H/C atomic ration and a low O/C atomic ration. In IR analysis the less evolved samples of this type (sample Ao, fig. 2) show  $CH_2$ ,  $CH_3$  aliphatic bands which are very large as compared with C=O carbonyl and carboxyl

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band. In thermogravimetric analysis there is a high percentage of weight loss (up to 85%) at  $600^{\circ}$ C. In Mass Spectrometry Analysis, the pyrolysis products produced by heating at programmed temperature show moderate CO<sub>2</sub> and H<sub>2</sub>O formation below  $400^{\circ}$ C, followed by a high production of hydrocarbon compounds between 400 and  $500^{\circ}$ C. These kerogens belong to a family that can produce large quantities of oil and gas during catagenesis.

On the other hand, kerogens of type III (Upper Cretaceous of the Douala Basin in the Cameroons, Lower Mannville shales of Lower Cretaceous age in Alberta) show a low H/C atomic ratio ans a high O/C ratio. For the less mature samples in this series (sample Co, fig. 2) IR analysis shows that  $CH_2$ ,  $CH_3$  aliphatic bands are smaller than the C = O carbonyl and carboxyl band and the C = C aromatic band. In T.G. analysis the final loss of weight reaches only 30%. Mass Spectrometr shows that the amounts of  $CO_2$  and  $H_2O$  obtained below  $400^{O}C$  are hight and the quantity of hydrocarbon compounds is low.

The origin of these kerogens is principally upper plant detritus and humic terrestrial matter, and they have a low petroleum potential.

Kerogens of type II (Lower Toarcien from the Paris Basin, Silurian frim the Sahara) are in an intermediate position. They have the qualities of a good source rick.

The position of kerogen samples along each evolution path depends on their maturation level. First, the oxygen content decreases. This is the immature stage where mostly  $CO_2$  and  $H_2O$  are generated. Later, the hydrogen content decreases. This correspond to the main phase of oil generation, then to the phase of cracking and gas generation.

#### RAPID METHOD FOR CHARACTERIZING ORGANIC MATTER IN ROCKS

Using the basic knowledge presented above IFP have developed a method and apparetus for the rapid analysis of organic

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matter directly in rocks.

#### Principle

The method consist if the selections, detection and quantitative analysis of oxygenated and hydrocarbon type compounds issuing from the pyrolysis of sedimentary rocks in an inert atmosphere and with programmed temperature.

#### Development of the Method

While the oxygenated compounds produce by the pyrolysis of kerogens isolated from their mineral matrix are due exclusively to the decompisition of organic matter, this is no longer true when the organic matter is in its mineral matrix because of minerals whose thermal dissociation is known to give off water (case of clay minerals, hydroxides etc...) and CO<sub>2</sub> (carbonates).

A study of the thermal dissociation of the principal minerals found in sedimentary rocks has therefore been made in order to delimit the temperature intervals within which only oxygenated compounds will ve liberated by decomposition of the organic matter.

It has been shown that only  $CO_2$  analysis can reasonably be considered in the study of organic matter in rocks, in a temperature interval below  $400^{\circ}C$  (interval where there is no interference with the thermal dissociation of carbonates).

### Description of the Apparatus (fig. 3)

The apparatus consist of a micropyrolysis oven (1) in which heating is controlled by a temperature programmer. The sample (2) is heated in an intern atmosphere (helium) up to  $550^{\circ}$ C.

At the top of the oven there is a heated splitter (3) whose temperature is regulated at  $550^{\circ}C$ . It divides the gaseous pyrolysis flux into two approximately equal parts :

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First, towards a flame ionization detector (FID) (4) for the detection of hydrocarbons (free hydrocarbons present in the rock, and hydrocarbon compounds produced by the cracking of kerogen).

Second, towards a device for trapping  $CO_2$  (5) during the programmed heating, in the temperature interval selected by the operator. At the end of the temperature programming, the  $CO_2$  trap is reheated for the desorption of the  $CO_2$  which is then drawn to a Porapak column (6) and detected by a thermal conductivity detector (7).

A thermocouple (8) situated at the level of the sample inside the oven makes it possible to know the temperature T corresponding to the maximum of hydrocarbon type compounds produced by the cracking of kerogen during pyrolysis.

#### Operating Procedure

A small quantity (50 to 100 mg) of previously ground rock is put into a boat whose bottom and cover are made of sintered steal.

The boat is put on the top of the piston and then the operator starts the automatic analysis cycle.

The various analysis phases are completely automatic. They are all carried out in accordance with impulses given at predetermined times by the temperature programmer.

The duration of the analysis is 15 minutes, plus 5 minutes for cooling, i.e. 20 minutes for the full cycle.

#### Parameters Given by the Apparatus

The analysis of a rock sample is characterized by the recording in Figure 4.

1. Peak  $P_1$  corresponds to the free hydrocarbons present in the rock and which are volatilized at a temperature below  $300^{\circ}C$ . Its area  $S_1$  gives the quantity of free hydrocarbons

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(oil + gas) contained in the rock expressed in mg Hydrocarbons per gram of rock.

2. Peak  $P_2$  corresponds to hydrocarbon type compounds produced by the cracking of the kerogen up to 550°C. Its area  $S_2$  gives the residual petroleum potential of the rock, expressed in mg of hydrocarbons per gram of rock. The ratio  $S_2$  (mg Hydrocarbon)/Total organic carbon of the rock, is called the "Hydrogen Index".

3. Peak  $P_3$  corresponds to the  $CO_2$  produced by the pyrolysis of the organic matter in the rock. Its area  $S_3$  gives the quantity of  $CO_2$  expressed in mg per gram of rock. The ratio  $S_3$  (mg  $co_2$ )/Total organic carbon of the rock is called the "oxygen index".

4. Temperature T corresponds to the maximum hydrocarbon type products by the cracking of kerogen during pyrolysis. This temperature is characteristic of the evolution level of the organic matter.

5. The Production Index is given by the ratio Sl(Sl + S2). It characterizes the evolution level of the organic matter and makes possible the detection of oil shows.

#### APPLICATIONS. SOURCE ROCK CHARACTERIZATION

#### Types of organic matter.

- - -

It has been shown that the "hydrogen index" and "oxygen index" reveal a good correlation with the atomic rations H/C and O/C obtained by the elementary analysis of kerogens isolated from rocks.

These two indices enable us to draw a diagram (fig. 5) which is similar to the diagram established for kerogens from the aromic rations H/C and O/C (fig. 1).

Three types of organic matter can be defined directly from the rock samples. The samples situated on path I have a high

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"Hydrogen Index" and a low "Oxygen Index" : they characterize excellent source rocks containing algal organic matter (kerogen type I). On the other hand for samples on path III, the index values are revoresed : they have a comparatively lower source rock potential ; the organic matter is of terrestrial original (kerogen type III) and is generally more favourable for gas than for oil. Between these two extreme types, path 2 characterizes good source rocks whose organic matter is from marine deposite (kerogen type II).

#### Petroleum Potential

The concept of the quality of organic matter should be completed by the petroleum potential of the rock. This petroleum potential represents the maximum quantity of hydrocarbon compounds (oil + gas) which can be provided in nature by a rock if has been buried at sufficient depth for a sufficiently long time. It corresponds to the sum  $S_1 + S_2$ .

S<sub>1</sub> gives the quantity of hydrocarbons (oil + gas) currently generated in the rock.

S<sub>2</sub> gives the quantity of hydrocarbons that the rock could still produce should burial and maturation continue.

Quantitatively, this petroleum potential can be expressed as kg of hydrocarbon compounds (oil + gas) per metric ton of rock.

Petroleum Potentiel =  $(S_1 + S_2)$  kg Hydrocarbons/metric ton of rock.

Maturation stages of Source Rocks They can be defined in different ways:

First, by the position of samples on their specific evolution path in the diagram of the "hydrogen index" - "oxygen index" (fig. 5).

If we start from a sample containing immature organic matter, at the beginning of maturation a rapid decrease of the "oxygen index", appears, while the "hydrogen index" remains nearly constant. When the main zone of maturation (oil zone) is reached, only the "hydrogen index" will decrease.

Second, by the temperature corresponding to the maximum of peak  $P_2$ . This temperature increases with the degree of evolution. Generally temperatures between  $400^{\circ}C$  and  $435^{\circ}C$  correspond to the immature zone. The oil zone or nature zone is characterized by temeratures between 435 and  $460^{\circ}C$ . Up to  $460^{\circ}C$  it is the gas zone or metamorphosed zone (fig. 6).

Third, by the "production index", which is given by the ratio  $S_1/S_1 + S_2$ . The value of this index increases with depth (fig. 6 and 7). During their evolution upon increasing burial, sedimentary rocks show an increase in the  $P_1$  peak (free hydrocarbons present in the rock) correlatively with a decrease in the P2 peak (hydrocarbon products from kerogen cracking). This observation is particulary revealed by the examples in Fig. 6, in which pyrograms obtained by this method are given for samples from the Tertiary of West Africa from depths of 800 m to 2770 m.

#### Oil shows

Oil shows are detected by evaluaring the amount of hydrocarbons present in the rock  $(S_1)$  in association with the "production index".

In a geological series when there is no accumulation the plot of the production index versus the corresponding depth is as shown in fig. 7. The "production index" increases regulary with the depth. On the other hand, if there is an accumulated zone in a series, the "production index" increases very rapidly in this zone A (Fig. 8), and then decreases until the base line is reached.

In the same way if there is a draines zone the "production index" will decrease suddenly in this zone B (Fig. 8) and then it increases until the base line is also reached.

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#### Presentation of Results. Geochemical Log

The various parameters given by our apparatus can be used to draw a geochemical log. The sample analysis may be done on the site in the well logging unit itself as soon as the cuttings are recovered. The log is very simple to draw up and gives a fast picture of the various geological zones encountered during the drilling.

Figure 9 shows a geochemical log example for an exploration well from 1200 meters to 3500 meters. This log shows three main zones. The first one down to 2100 meters corresponds to the immature zone. It is characterized by temperatures T below 435°C, an "oxygen indewx" high principaly in the first part, the oil and gas content and the production index are low and they increase gradually with depth. The "Hydrogen index" shows a good source rock between 1500 - 1700 meters, but it contains immature organic matter which has not yet generated oil.

The oil zone is located between 2100 and 3000 meters. At the beginning of this zone temperature T reachers 435°C and at the end 460°C. The "oxygen index" remains low. The "Hydrogen index" shows a good source rock level between 2250 and 2500 meters, which has produced oil. The oil and gas content column indicates two oil shows, the first corresponding to the good source rock level and another one slightly below. The "Production index" shows that the second oil show between 2600 and 2750 m corresponds to an accumulation of oil (reservoir rock) probably coming from the upper source rock.

After 3000 meters the gas zone begins and is characterized by high temperature T (more than 360<sup>O</sup>C). The hydrogen index is low. The gas content and the "Production index" increase rapidly.

Aplication as a safety tool (IPOD PROGRAM)

The apparatus was used on the GLOMAR CHALLENGER for Legs 48 and 50. An important aspect of its use on board was as a safety tool because the method can rapidly reveal the presence of oil show during drilling. In assition it has provided information on the different types of organic matter encountered and on their degree of evolution.

#### RESULTS AND DISCUSSION

Thirty-one samples from well 30/7-7 were analysed, nine samples from the upper 3800 m and twenty-two samples from the lower part. The hydrogen index is low for all the samples indicating that none of the analysed samples will be good quality source rocks for oil. The  $T_{max}$  values indicate the border between immature and mature to be at approximately 2500 m and between mature and condensate window at approximately 4400 m.

By examination the oil and gas content  $(S_1 + S_2)$ , together with the production index  $(S_1(S_1 + S_2))$ , the area between 3900 and 4300, and at approximately 4800 m have indications of migrated hydrocarbons.

#### CONCLUSION.

When these results are compared with the other source rock results, (Report 0-204/1/79) it is noted that there is a discrepancy in the maturity estimation. Vitrinite reflectance and visual kerogen examination found the border between moderate mature and mature to be of 2900 m and between oilwindow and condensate window at 4400 m. The reason for this discrepencies are, we believe, due to a finer division of the maturety stages, in using the vitrinite reflectance and visual kerogen (immature, moderate mature, mature, oilwindow maturity, condensate window, gas window, compared to the Rock-Eval method (immature, oil zone and gas zone).

When the source rock potential is examined, the Rock-Eval method does not show any source rock potential for oil in

the examined samples, while the visual herogen examination shows some of the samples to consist almost completely of amorphous kerogen. It is, however, noted that almost all the examined samples contain particles of coal. This will give a high reading on the total organic carbon measurements which will result in a low hydrogen index. If these coal particles are reworked, which the vitrinite reflectance measurements indicate, this would decrease the amount of "true" organic carbon, and the hydrogen index would be higher. These reworked particles would in volume only take a small proportion compared to the amorphous material.

When the Rock-Eval results are taken into account we feel that the source rock potentials given for the various zones in this well should be changed in the following places.

Zone D : Poor to fair potential as a source rock for oil and gas, oilwindow maturity.

Zone E : Rich potential as a source for gas (oil), oil window maturity.

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- Fig.10 Geochemical log of samples from well 30/7-7.

Sample Depth 3450-350( 3350-75 3725-50 3875-390( 3900-392( 3950-60 4030-40 4100-411( 4140-50 4180-90 2400-50 3350-3401 2950-3000 3]5**0-**320 4450-60 4370-80 4300-10 4240-50 2700-50 2250-230 0.36 0.40 0.40 0.75 0.75 1.14 1.12 3.36 1.83 1.83 1.83 2.71 1.22 2.96 1.46 1.97 1.64 0.42 0.22 0.17 Ś  $\begin{array}{c} 1.22\\ 0.03\\ 0.02\\ 0.90\\ 0.90\\ 0.15\\ 0.06\\ 0.66\\$ Š 0.847 0.541 2.076 0.540 0.449 0.449 0.442 0.583 0.583 0.583 0.583 0.485 1.644 1.8300 1.8300 1.8300 1.8300 1.8300 1.8300 1.830 1.523  $\begin{array}{c} 0.99\\ 0.71\\ 0.72\\ 1.14\\ 0.62\\ 0.69\\ 0.69\\ 0.86\\ 0.86\\ 2.56\\ 2.56\\ 2.56\\ 2.56\\ 2.69\\ 3.16\\ 2.69\\ 2.69\\ 3.21\\ 2.92\\ 4.59\\ 4.59\\ 3.21\\$ Corg Hydrogen Index 42.25 27.78 78.86 23.71 7.25 7.25 77.09 90.79 90.79 90.79 90.79 90.79 90.79 109.37 56.47 95.17 105.31 41.05 66.58 65.41 34.72 123.23 1153.84 1119.20 75.14 182.11 87.110 64.06 63.77 67.79 92.15 10.57 10.57 10.57 10.39 52.03 68.03 68.03 45.51 47.43 47.43 19.11 19.11 19.11 0xygen Index 0il of 9ascontent  $(S_1 + S_2)$ 0.25 0.19 1.26 0.55 1.62 1.62 1.55 3.42 6.07 6.07 6.17 2.74 4.54 3.85 5.10 1.351.64 Production Index Sl S1 + S2 0.88 0.90 0.29 0.73 0.73 0.59 0.57 0.57 0.36 0.36 0.36 0.37 0.37 0.44 0.37 0.44 0.37 0.32 0.52 0.26 T<sub>max</sub> <sup>O</sup>C

TABLE I

Rock Eval. pyrolysis

| Sample | Depth     | s <sub>1</sub> | s <sub>2</sub> | S <sub>3</sub> | C <sub>org</sub> | Hydrogen<br>Index | Oxygen<br>Index | Oil of <b>b</b> as<br>content<br>(S <sub>1</sub> + S <sub>2</sub> ) | Production<br>Index<br>Sl<br>Sl + S2 | T <sub>max</sub> C |
|--------|-----------|----------------|----------------|----------------|------------------|-------------------|-----------------|---|--------------------------------------|--------------------|
| K360   | 4540-50   | 1.15           | ?1.26          | 0.44           | 2.34             | 53.89             | 18.72           | 2.41  | 0.48                                 | ?                  |
| К368   | 4570-80   | 0.98           | 0.93           | 0.56           | 2.17             | 42.86             | 25.81           | 1.91  | 0.52                                 | 489                |
| К375   | 4640-50   | 0.75           | 0.35           | 6.43           | 2.02             | 17.48             | 318.07          | 1.10  | 0.68                                 | 484                |
| K382   | 4710-20   | 0.24           | 7.05           | 6.47           | 10.6             | 66.47             | 61.07           | 7.29  | 0.03                                 | 435                |
| K387   | 4760-70   | 1.31           | 9.62           | 2.60           | 5.17             | 186.05            | 50.23           | 10.93   | 0.12                                 | 412                |
| К390   | 4790-4800 | 2.01           | 1.91           | 0.48           | 2.43             | 78.77             | 19.63           | 3.92  | 0.51                                 | 487                |
| К393   | 4820-30   | 1.71           | 1.92           | 0.18           | 2.59             | 73.94             | 6.76            | 3.63  | 0.47                                 | 481                |
| К442   | 4900-10   | 0.79           | 0.45           | 0.44           | 2.15             | 20.79             | 20.28           | 1.24  | 0.64                                 | 479                |
| K447   | 4950-60   | 0.65           | 0.40           | 0.62           | 2.60             | 15.19             | 23.96           | 1.05  | 0.62                                 | 494                |
| K457   | 5050-60   | 1.07           | 0.42           | 0.28           | 2.50             | 16.64             | 11.20           | 1.49  | 0.72                                 | 484                |
| К463   | 5110-20   | 0.96           | 0.58           | 0.25           | 2.41             | 24.23             | 10.42           | 1.54  | 0.62                                 | 480                |
|        |           |                |                |                |                  |                   |                 |   |                                      |                    |
|        |           |                |                |                |                  |                   |                 |   |                                      |                    |
|        |           |                |                |                |                  |                   |                 |   |                                      |                    |
|        |           |                |                |                |                  |                   |                 |   |                                      |                    |
| 1      |           |                |                |                |                  |                   |                 |   |                                      |                    |

### **CLASSIFICATION OF THE VARIOUS TYPES OF KEROGEN** IN AN ATOMIC H/C\_O/C DIAGRAM

Fig.1



- Algal Kerogens (Botryococcus, etc...)
- Lower Toarcian , Paris Basin
- Silurian, Sahara\_Libya
- Upper Paleozoic\_Triassic, Spitsbergen .
- ☆
- Upper Cretaceous, Douala Basin Cretaceous, Persian Gulf (Oligostegines limestone)
- Lower Mannville shales, Western Canada Θ
- Others ا∆

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### **OPERATIONAL FLOWSHEET OF THE APPARATUS**



## SCHEMATIC EXAMPLE OF RECORDING OBTAINED WITH A CRUDE ROCK





Fig.4

CLASSIFICATION OF THE VARIOUS TYPES OF SOURCE ROCK IN A HYDROGEN INDEX / OXYGEN INDEX DIAGRAM

1 . . . .



- ★ Green River shales
- Lower Toarcian, Paris Basin
- 🛦 Silurian\_Devonian , Sahara \_ Libya
- Upper Paleozoic , Spitsbergen
- ☆ Upper Cretaceous, Douala Basin
- Cretaceous, Persian Gulf (Oligostegines limestone)
- Upper Jurassic , North Aquitaine

Increasing maturation



**RAPID CHARACTERIZATION OF THE DEGREE OF EVOLUTION** 

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# VARIATION OF THE PRODUCTION INDEX WITH DEPTH WHEN THERE IS NO MOBILIZATION AND MIGRATION OF HYDROCARBONS

Fig.



## VARIATION OF THE PRODUCTION INDEX WITH DEPTH WHEN MOBILIZATION AND ACCUMULATION OF HYDROCARBONS HAVE BEEN EFFECTIVE



## RAPID CHARACTERIZATION OF THE DEGREE OF EVOLUTION OF SOURCE ROCK IN A SEDIMENTARY SERIE OF TERTIARY AGE

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| DEGREE<br>OF<br>EVOLUT. | DEPTH<br>(m.) | T <sup>o</sup> C<br>400 430 460 | HYDROGEN INDEX<br>mg CO <sub>2</sub> /g org carbon<br>200 400 600 | OXYGEN INDEX<br>mg CO <sub>2</sub> /g org. carbon<br>50, 100, 150 | OIL B GAS CONTENT<br>KgHC/metric ton of rock<br>4 B 12 | PRODUCTION INDEX<br>S1/S1+S2<br>0.4 0.6 0.8 |
|-------------------------|---------------|---------------------------------|---|---|--|---|
| MMATURE ZONE            | 1500          |                                 | Good source<br>rock,  |   |  |   |
|                         | -<br>2000 -   |                                 | immature  |   |  |   |
| DIL ZONE                | 2500 -        |                                 | Good source<br>rock,<br>mature                                    |   | Oil detection  | ¢<br>Oil<br>accumulation                    |
| GAS ZONE                | 3000 -        |                                 |   |   |  |   |



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