

QUANTITATIVE ANALYSIS OF OIL BASED MUD FILTRATE INVASION

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ABSTRACT Mud filtrate invasion into core material taken from oil reservoirs may cause problems for future core analysis studies due to the potentially damaging effect of the mud filtrate upon the rock's wettability. It is therefore desirable in these situations to minimise the degree of invasion and accurately measure the success of this minimisation. There is much discussion in the literature of tracer techniques but all of these require well site activity as well as tracer purchase and handling. A quantitative technique is presented which requires no tracer to be used and is quick and easy to perform. The process involves Gas Chromatography finger print analysis of both reservoir crude and mud filtrate. High contrast characteristics are identified and quantified for known ratios of mud and crude thus generating a calibration curve. Hydrocarbons are then solvent extracted from the core trims which may be as small as 1.50" diameter and 0.25" thick. The effluent is then boiled down (taking care to minimise carry over) to concentrate the hydrocarbons extracted. The residue is then analysed using GC and related to the calibration curve generated previously. Experimentation to date has suggested that the technique is accurate to within approximately +/- 2 wt%. The resolution also depends to some extent on the hydrocarbons present.

INTRODUCTION

Mud filtrate invasion to some degree will always occur during the coring process with both oil and water based systems. In some cases this invasion may compromise any analysis which is carried out on the core at a later date. The seriousness of this problem very much depends upon the application. Where water based mud systems are used then filtrate influx will affect S_w from core determination studies. Where oil based systems are used then the wetting characteristic of the rock may also be significantly affected by the emulsifiers present in the mud. This would obviously affect measurements dependant upon the wettability of the rock where native state core was to be utilised. This paper discusses a technique for quantifying the degree of oil based mud filtrate invasion and steps that can be taken to minimise its presence.

AN OVERVIEW OF TECHNIQUES

The literature contains many references to the use of tracers added to the mud system on site to track filtrate invasion into core material. These methods may use chemical tracers such as iodide, bromide, chloride, dextrose, acetone, etc (Ref 1,2,3) or isotope and radioactive tracers such as deuterium, tritium or radioactive vanadium (Ref 1,2). The tracer technique requires that known quantities are added to the mud system at regular intervals on site during the coring process and that the mud system is regularly sampled for calibration purposes. The phase occupied by the tracer must then be extracted in the laboratory and analysed for tracer content which can then be related to mud filtrate invasion. These methods typically aim to trace the water phase although oil phase tracers could also be used (Ref 4). Whilst in principle these methods can be accurate they are relatively expensive due to the cost of the tracers themselves and the on site manpower costs on top of which the cost of analysis must also be added. The advantage of these tracer techniques is that they are well suited to the tracing of the water phase. With the possible gradual phasing out of oil based mud systems in the North Sea this may become an increasingly significant factor in their favour.

as the pressure drops during the rise to surface will act as an opposing force to invasion. Upon reaching the surface the core barrel was cut into 1M sections and placed on drainage racks. Selected intervals were removed for inspection by the site geologist and suitable plugging points identified. Five 1.5" diameter plug samples were taken from each depth interval with minimum separation.

The plugging procedure itself is important. By plugging as soon as possible the opportunity for continued diffusion of mud filtrate into the centre of the core is minimised. This core was therefore plugged on site immediately after inspection. Conventional plugging techniques use a positive head of lubricant - normally simulated formation brine - to carry away fines from the drill bit. However there is a danger, particularly with permeable core that this lubricant will be forced into the plug as it is drilled. This will inevitably force filtrate on the outside of the core into the plug. To prevent this it is necessary to plug with no lubricant. However this can cause overheating and seizure of the bit resulting in both structural and chemical damage to the sample. Hydrocarbons present may decompose due to the heat thus possibly affecting the wettability of the rock.

Compressed air as a lubricant proved ineffective since the positive air pressure at the top of the plug was sufficient to drive fluids into the sample. Instead a technique was developed whereby a vacuum was pulled on the drill bit. This has the advantage that air is drawn down the outside of the bit past the cutting edge and up the annulus between the bit and the plug. This removes enough of the fines and has sufficient cooling effect. It should be noted that this technique may prove ineffective with tight core but here the rock was a highly permeable and relatively soft sandstone.

The plugs were trimmed to a length of 2" and wrapped in cling film and aluminium foil and sealed in individual plastic bags. The central plug in each depth interval was then sliced and each piece similarly preserved. Figure 1 shows a schematic of the 2" plugs taken for future core analysis (below) and the centre sample cut into 11 slices (above). Note that six trims were also taken from the core analysis plug samples.

Where oil based mud systems are used the technique described here is an attractive alternative. The fact that it can be performed retrospectively may also be of significance. Although it is entirely possible that it has been used before, surprisingly no evidence could be found in the literature search conducted for this paper.

Water based tracing exercises are normally performed to correct S_w from core measurements whereas oil based tracer exercises would normally be undertaken to identify the potential alteration filtrate may have made to rock native wettability. This would have important implications for any native state relative permeability analysis subsequently performed.

A CASE STUDY

An oil based mud system was used to core an oil bearing sandstone reservoir with a view to performing an extensive relative permeability study. In order to optimise the validity of the data obtained the operator was concerned that representative native state core samples should be used. There were therefore two primary goals ;

- 1) To minimise the degree of mud filtrate invasion into the core prior to sample analysis.
- 2) To quantify the degree of mud filtrate invasion if it had occurred in order that those samples affected could be discarded from the work programme.

Minimising Invasion

Consideration was given to coring techniques that would minimise filtrate invasion during the coring process. One important factor is the coring rate since this effects the residence time of the core in the core barrel. In this case a rate of 90 ft/hr was achieved using a DBS CD93 drill bit. This bit is designed to minimise the degree of filtrate invasion. As the core enters the fibre glass core barrel it is in contact with a filter cake and filtrate. This remains in the annulus between the barrel wall and the core for the duration of the trip to surface. Outgassing in the reservoir fluids

All plug samples and trims were flown off the rig by helicopter at the earliest opportunity. This was normally within 12 hours of the core coming to surface. Within 3 hours of arriving onshore the plug samples were removed from their packaging and placed in individual glass sample jars under de-polarised kerosene. Filtrate invasion analysis was then carried out on the trims and slices.

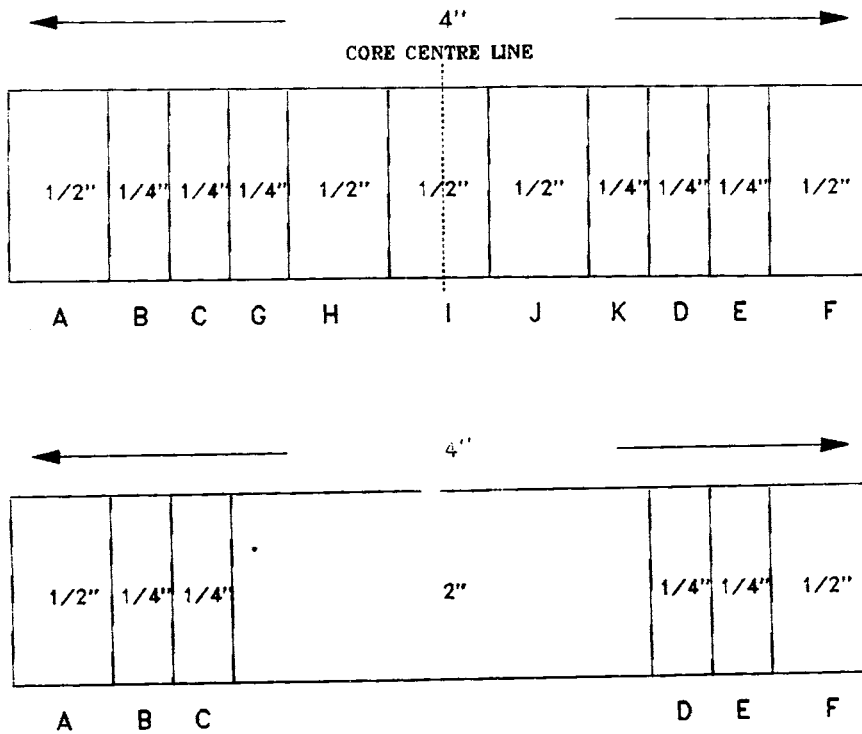


Figure 1

Filtrate Invasion Analysis

Hydrocarbons were extracted from each sample using boiling toluene. In some cases the toluene became black or dark brown with leached hydrocarbons but in others only a very small quantity of hydrocarbons were recovered leaving a yellow effluent. The residue was then boiled down to concentrate the sample and analysed using Gas Chromatography (Appendix 1). This yielded a fingerprint analysis from which the weight fraction of mud filtrate present in the hydrocarbons extracted was determined.

By plotting this fractional content of mud filtrate in the hydrocarbons extracted from the sample trim vs distance along the plug an invasion profile was produced - Figures 2-9.

Quantification of Filtrate Content in Extracted Hydrocarbons

Fingerprint analysis is a well established technique to characterise the composition of crude oils. The technique described here compares the crude oil fingerprint with that of the mud filtrate. The hydrocarbon content of the mud system originates from a refined oil and hence has quite a different fingerprint. Figure 10 shows the fingerprints for the crude oil in this well and the mud filtrate along with intermediate known mixtures. Visual inspection shows a significant difference. However if these known weight ratios of mud and mud filtrate base oil are to be used in a quantitative way further analysis is required.

Each component on the chromatograph is represented by a peak of finite area and height. Figure 11 shows a blown up region of two fingerprints derived from high and low base oil content ratios respectively. The absolute area and height will depend upon the concentration of sample used and hence is not of value. However the ratio of two areas or heights is independent on sample concentration and is directly related to the composition. In order to optimise the accuracy of this analysis characteristics of greatest contrast are required. The C16 Isoprenoid:nC15 peak areas ratio (R) gives a good level of contrast between pure crude (R=0.38) and pure mud filtrate (R=0.76). More

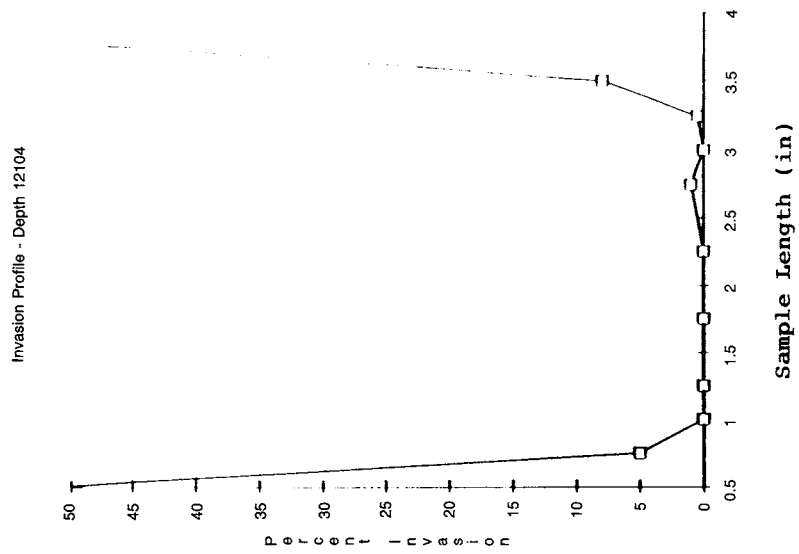


Figure 3

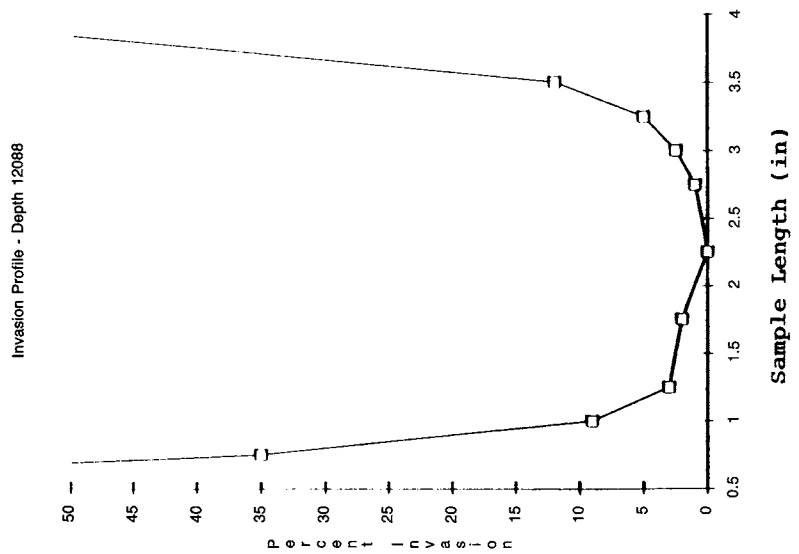


Figure 2

Invasion Profile - Depth 12227

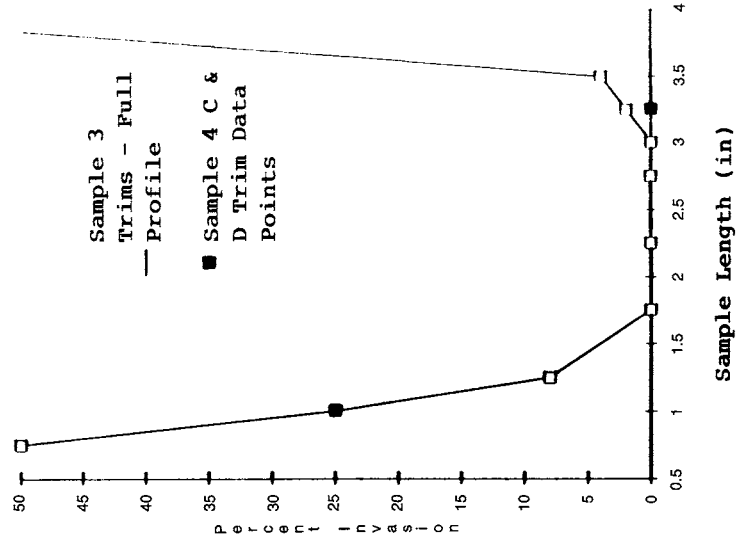


Figure 5

Invasion Profile - Depth 12115

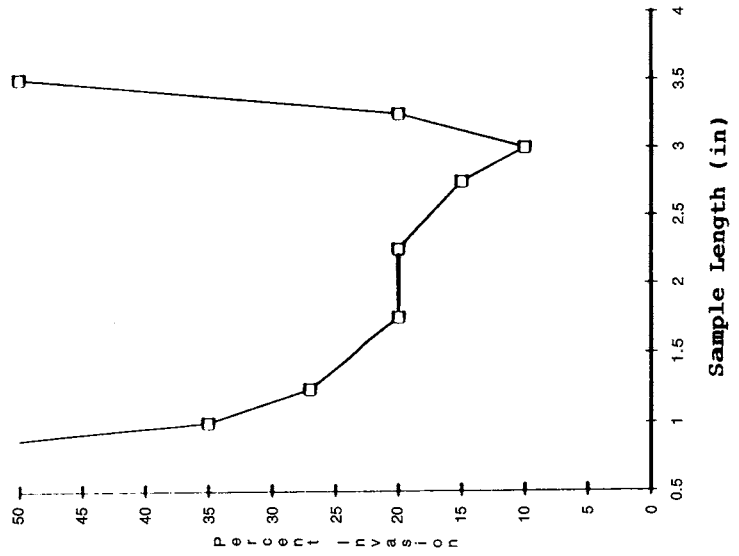


Figure 4

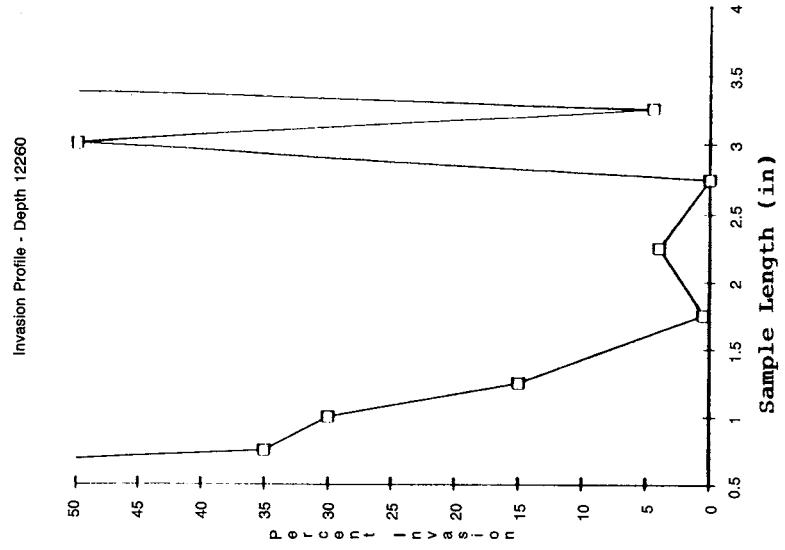


Figure 7

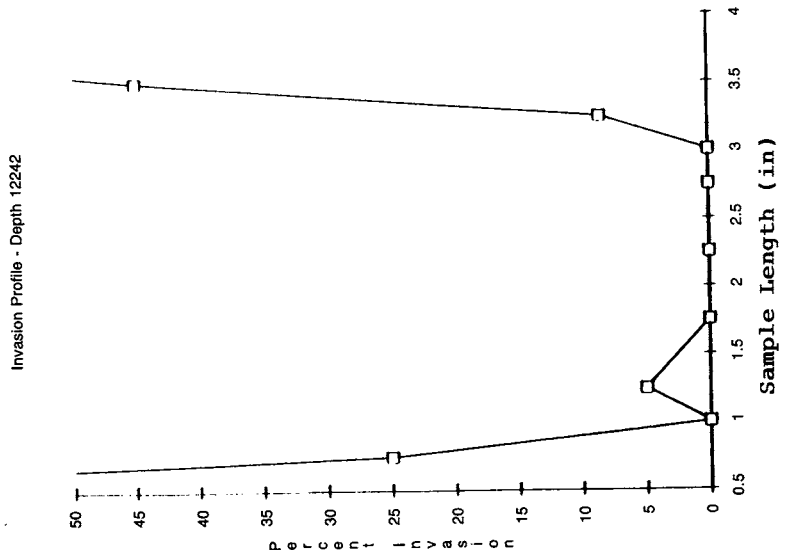


Figure 6

Invasion Profile - Depth 12273

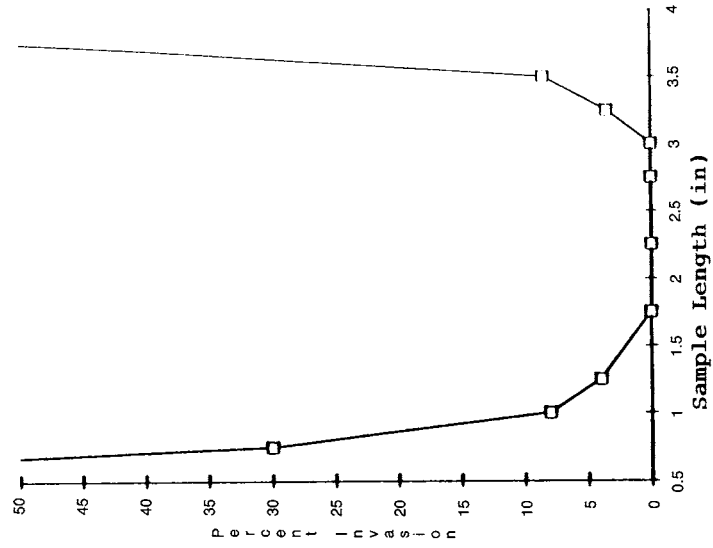


Figure 9

Invasion Profile - Depth 12288

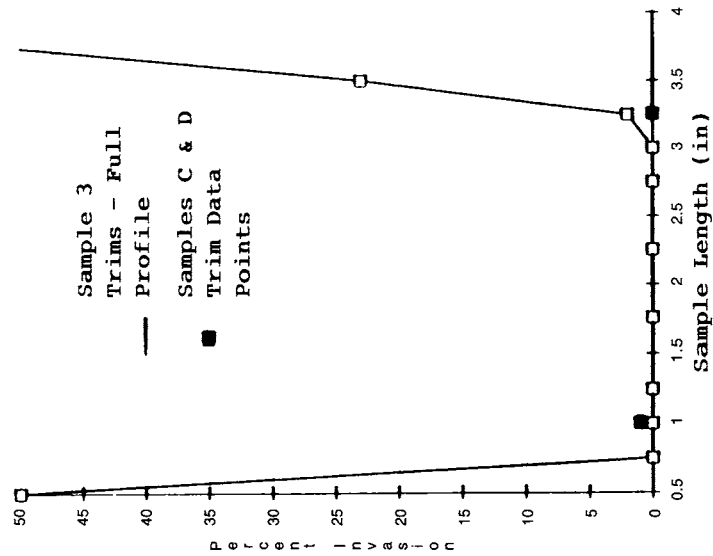


Figure 8

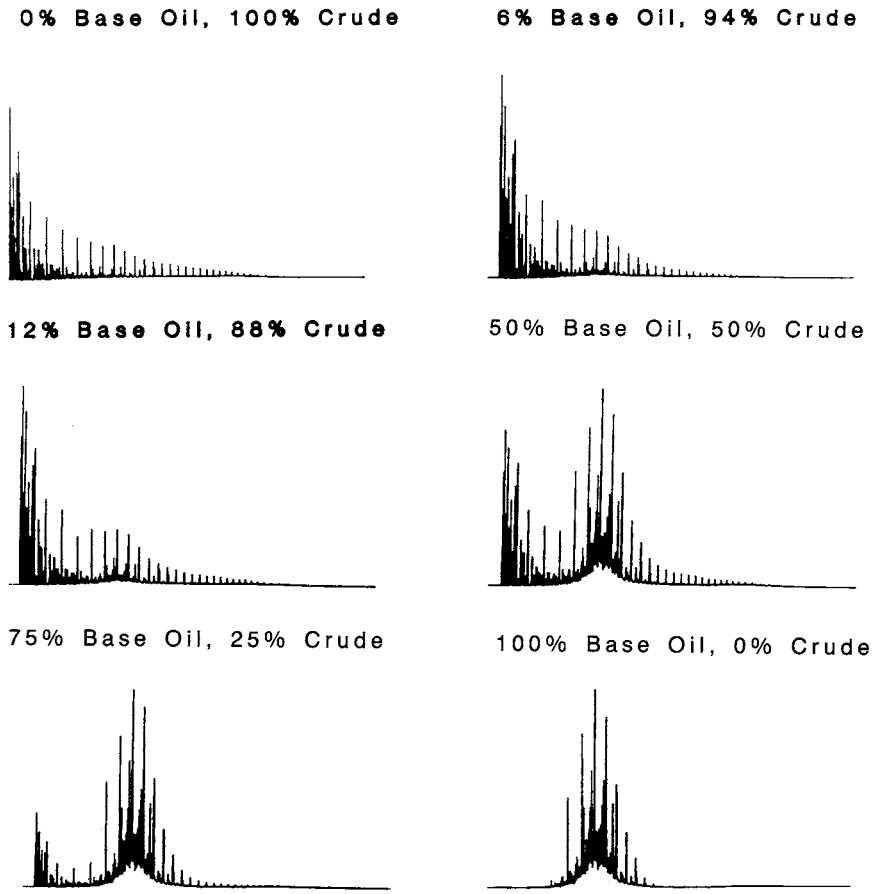
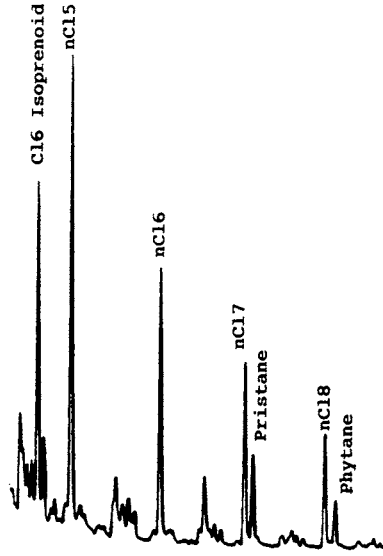


Figure 10

High Base Oil Content



Low Base Oil Content

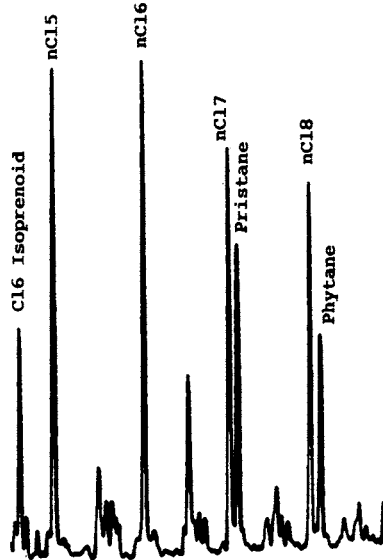


Figure 11

importantly the region of greatest change in R with respect to composition is at the low levels of base oil content which is the area of primary interest. By preparing known weight fractions of the two components and deriving the associated ratios a calibration curve relevant to **this** well can be generated - Figure 12. The ratio derived from the samples themselves can then be related to this calibration curve and a fractional mud filtrate content derived. Since we are not particularly concerned with very high levels of invasion (>20wt%) the data for this calibration curve is concentrated below 20wt% filtrate content. There is a certain amount of scatter here which indicates the degree of error in the final measurement. The sources of error and ways of minimising it are discussed in Appendix 1.

To check that the crude composition in C16 Isoprenoid and nC15 did not vary down the well data from the 3 central trims were plotted vs depth - Figure 13. There appears to be no correlation showing this not to be a problem here. It has been assumed that R for the mud filtrate did not vary significantly during the coring process.

From the calibration curve it can be seen that the resultant error in fractional filtrate content is approximately +/- 2wt% in the low base oil content region (<20wt%). Sample concentration and injection volume affects the amount of noise in the measurement. Figure 14 shows the standard deviation of measurements made with given injection volumes for one of the weakest samples used in this study. All of the injection volumes used in this case study exceeded 2 micro-l.

Results

Using this technique it was found that 6 out of 8 samples analysed had suffered negligible invasion beyond 1" into the core. However the assumption is that this lack of invasion was a consequence of the characteristics of the rock itself and the physical processes that occurred whilst that depth interval was being cored. This must be true if the adjacent plug sample is to be assumed similarly safe from invasion. To check this trims C and D were analysed from the adjacent sample. Figures 5 and 8 show the data over plotted on the profile for the adjacent plug. From this we can conclude that the adjacent sample is showing the

same invasion profile - as we would expect. This then provides us with samples which we can be confident have suffered no significant filtrate invasion.

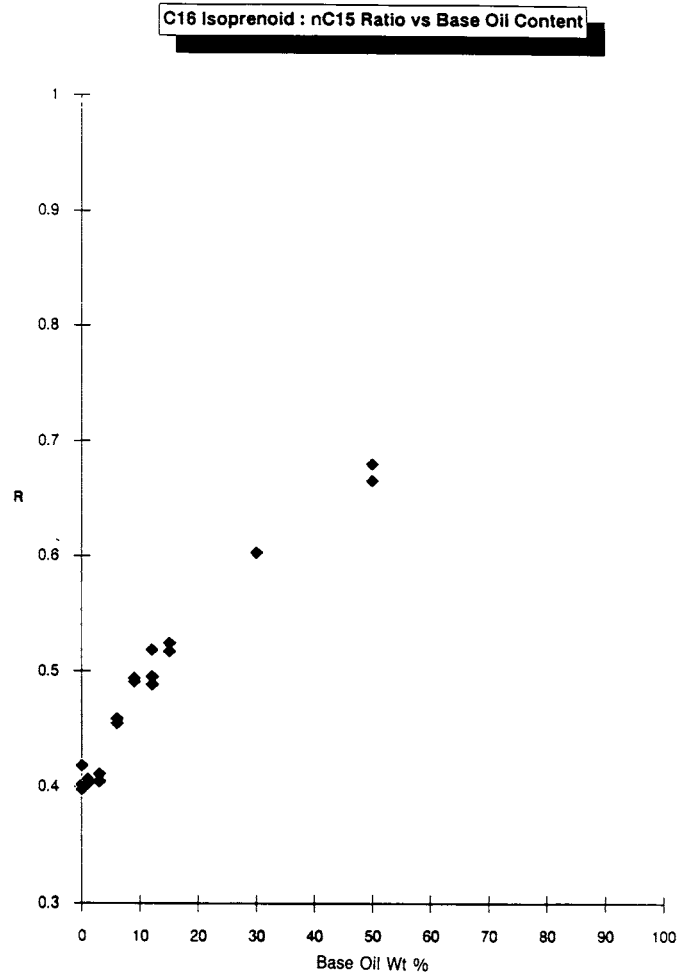


Figure 12

Depth vs C16 Isoprenoid : nC15 Ratio for Central Core Region

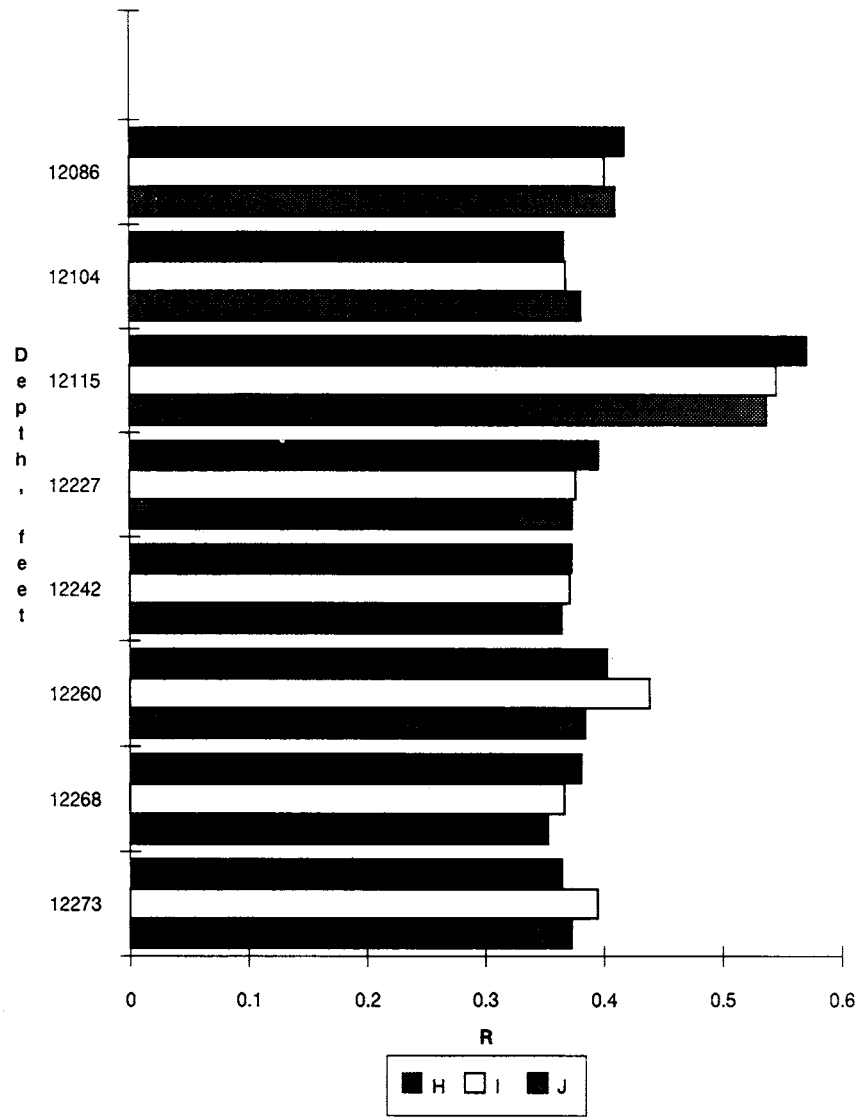


Figure 13

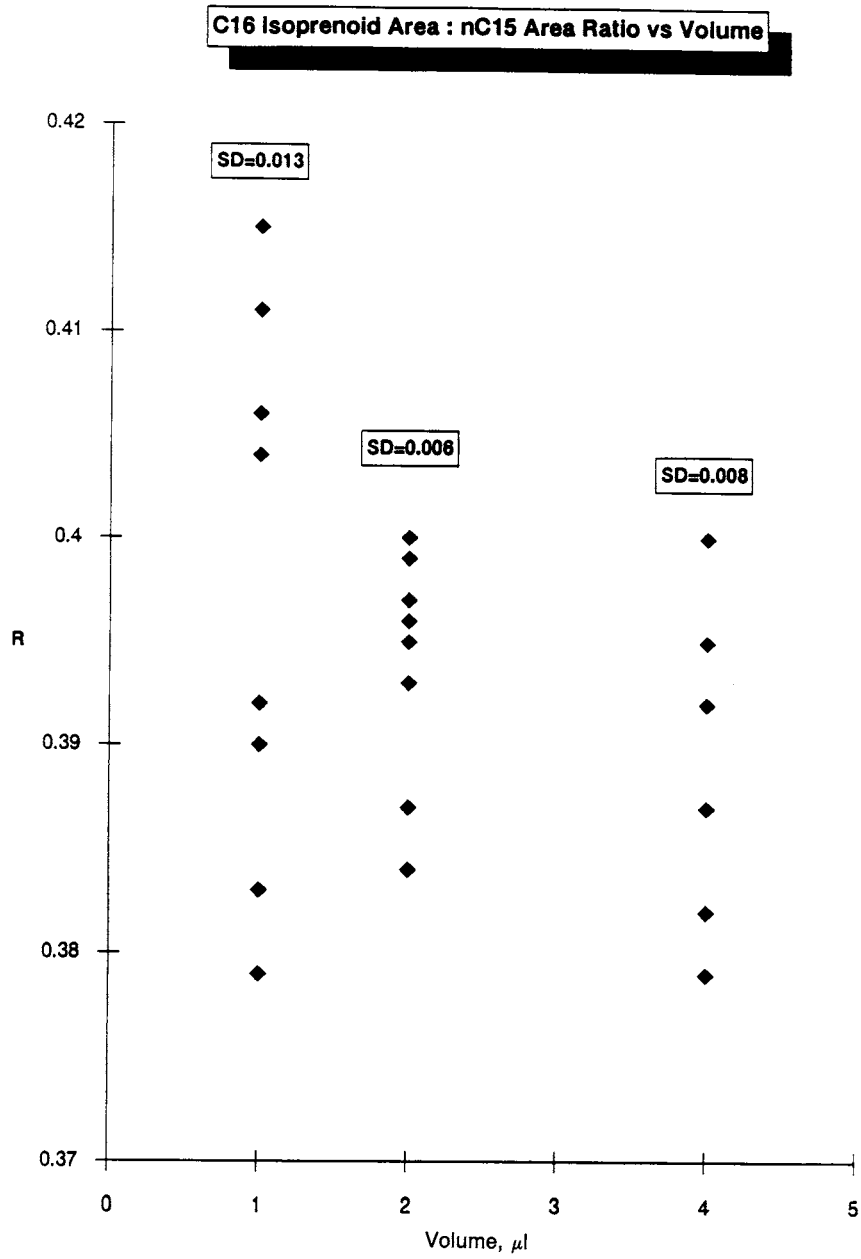


Figure 14

DISCUSSION

Whilst good results have been obtained in this case study there still remains room for improvement in the GC analysis. The biggest single improvement would probably be in using an automated temperature programmable injection system to eliminate any source of error in the sample injection process.

Further work could be undertaken to more accurately quantify all sources of error and their relationship to controllable parameters such as sample size. However this has to be put in perspective to the consequent financial gain of improved accuracy. This technique was developed for use in a commercial venture and not for the sake of research. It should be viewed in that light.

The technique has proved sensitive and simple to apply. The main advantages over tracing techniques is that it can be done retrospectively and that the expense of using tracers in the mud system on site is avoided.

Although the nC15 and C16 Isoprenoid peak areas were used in this case other parameters could have been selected. In the time available these appeared to offer the best contrast. In other studies this may not be the case. It is presumably possible that in some cases no parameters at all could be identified to give adequate contrast between mud and crude oil but this seems unlikely.

Although it has not been rigorously investigated here, previous work has indicated that the diffusion process operates rapidly over the first few days. Hence it was felt that we should not take the risk of delay in transportation of the core by boat followed by plugging in the laboratory. However the rate of diffusion could be investigated further to clarify the seriousness of the problem.

CONCLUSIONS AND RECOMMENDATIONS

- 1 Oil based mud filtrate invasion into core can be accurately quantified as a function of distance into the core using GC fingerprint analysis.
- 2 This technique should be used whenever an oil based mud is used to core a well and native state relative permeability measurements are to be performed on the rock obtained.
- 3 No on site effort is required and it can be performed retrospectively.
- 4 Trim samples as small as 1.5" diameter and 0.25" thick are adequate to give good results.
- 5 Analysis is very quick and can be performed with readily available equipment.
- 6 Use of automated temperature programmable injection systems in the GC equipment may help to make the technique more accurate.
- 7 Coring parameters such as the type of drill bit used and the speed of drilling should be optimised to minimise mud filtrate invasion.
- 8 Samples should be plugged on site to minimise the time available for filtrate diffusion into the centre of the core unless there is evidence to show that insignificant diffusion of mud filtrate occurs over the boat trip time.

ACKNOWLEDGEMENTS

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APPENDIX 1

GC Fingerprint Analysis

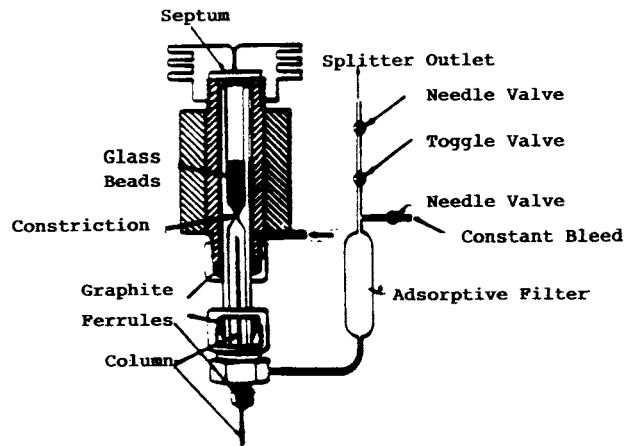
Gas Chromatography analysis basically allows a mixture of molecules in gaseous form to be separated and their fractional content in the original mixture to be quantified. Liquids such as crude oil are vaporised shortly after entering the equipment to make this possible.

The GC system itself comprise of 3 basic parts. INJECTION, COLUMN and DETECTION.

Injection

The basic parts of the injection system are shown in Figure 15. For the purposes of this study, a sample not exceeding 5 microlitres was injected. This is a relatively large injection necessitated by the dilute nature of the samples. There are many different designs of injection system, but in this case the system is designed to vaporise the liquid sample at a temperature of 325 degC and to allow an inert carrier gas (Helium) to sweep a homogeneous sample of the vapour on to the column. The vaporisation temperature used is below that at which thermal decomposition of the sample would occur. The narrow bore capillary column used has a very limited sample capacity and only a very small portion of the vaporised sample is passed on to the column. This is achieved by Split Injection of the vapour, in this case at a ratio of 450:1. It is in the Split Injection system that the largest potential for error exists. If the vapour produced in the injection system is not entirely homogeneous at the time that the split takes place then a sample which is not representative of the whole will be passed on to the column and subsequently quantified. It is known that this molecular weight discrimination is likely to occur in samples of wide boiling range such as the type under study. More sophisticated equipment, using wide bore capillary columns and temperature programmable on-column injection systems, are available which can automatically inject the entire sample directly into the column using a fine fused silica needle. With this method the entire sample is vaporised inside the front portion of the column so

Injection System



FID Detection System

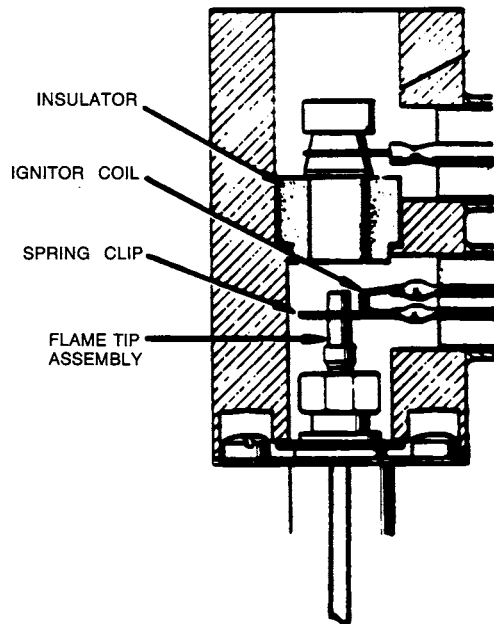


Figure 15

there is no possibility of molecular weight discrimination taking place. This technique is technically more difficult since very small samples must be injected in order not to overload the column. There is little doubt that this would significantly improve the repeatability of the final analysis which is of particular importance to the technique discussed in this paper.

Column

Chromatographic separation can be achieved using various techniques but the one used here is PARTITION. The vaporised sample is carried through a long (30m) narrow bore capillary column (0.25mm i.d.) which is coated on the inside wall with a non-polar silicone fluid (Polydimethylsiloxane). This liquid phase is chemically bonded to the column wall.

Figure 16 diagrammatically shows the separation process in 6 stages. In the column used over 100,000 such equilibrium stages take place. The higher molecular weight components have a longer residence time in the liquid phase than do those of lower molecular weight and hence take longer to travel the length of the column. Separation is thus achieved.

Detection

The end of the column passes the effluent into a detector. Various detection systems can be used but all are extremely accurate and would contribute little error to the analysis here. The system used for this analysis was of the Flame Ionisation Detection (FID) variety and is suitable for detection of hydrocarbon components. Figure 15 diagrammatically shows the technique. As the components emerge from the column they are ionised in a hydrogen flame. These ions are then attracted to the ANODE where their presence is detected as a small current. This current is amplified and output from the system to a data acquisition system. The magnitude of the current is proportional to the mass of ions reaching the detector in each time slice. It is important to note that the molecules are completely oxidised and so larger molecules produce many more ions than smaller ones. This current is therefore proportional to mass and not a molar quantity.

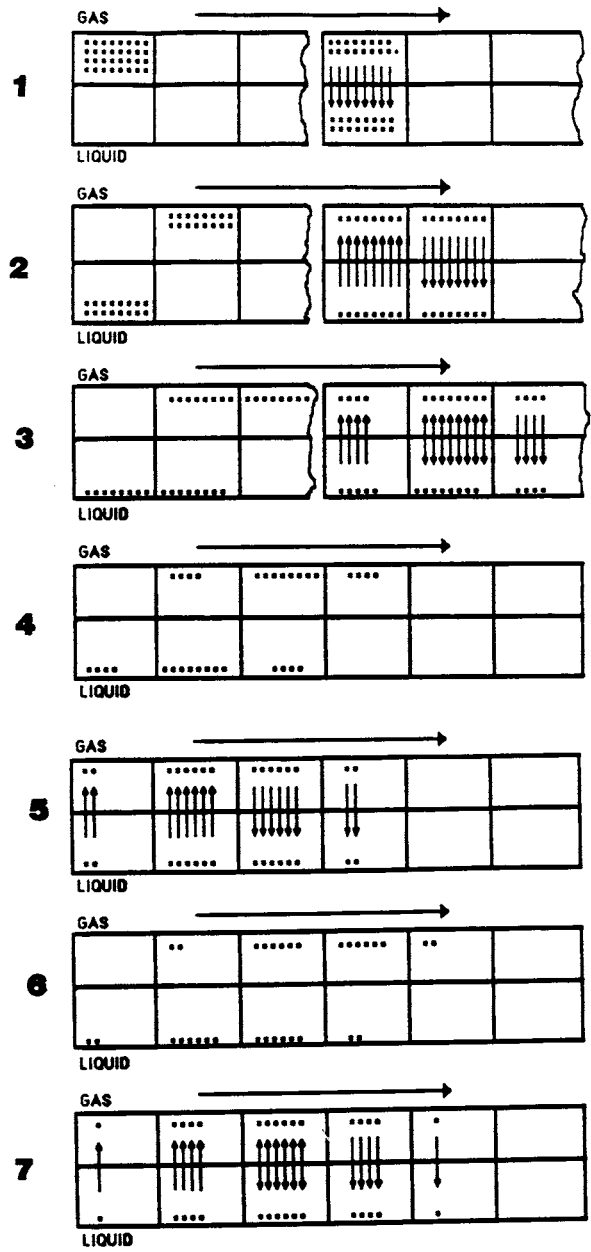


Figure 16