CORE PRESERVATION - AN ALTERNATIVE APPROACH

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Abstract The preservation of consolidated core samples for petrophysical and petrological analysis in an effective and efficient manner is of particular importance for the production of accurate and representative reservoir data.

To provide an alternative to the more conventional method of "wrap and seal", the Core Preservation Container (CPC) has been developed. The new method rapidly isolates selected whole core samples in an environment which minimizes physical and chemical alteration of the mineral grains and pore fluids, by the prevention of oxidation, evaporation and drying.

The design of the CPC draws upon the recommendations made in the literature as to the effectiveness of preserving core under anaerobic conditions. This is achieved by immersing the core sample in a compatible liquid, contained within a sealable glass vessel.

Initial screening in the laboratory suggests the technique will preserve key core parameters such as the insitu clay fabric and wettability during transportation and storage.

Field trials have been used to establish a suitable methodology and to test whether the technique is practical and safe at the wellsite. Modifications have been made to address the problems encountered, for example fracturing of the glass body while in transit from the wellsite to the laboratory.

INTRODUCTION

A special core analysis programme aims to provide a data base that is directly applicable to the reservoir formation under investigation. These data will be used in the estimation of reserves and understanding the most efficient method of hydrocarbon recovery (1). In order to generate representative values it is necessary to preserve the selected core material in an effective manner. The purpose of the core preservation is to maintain the inherent physical and chemical properties of the whole core sample and its pore fluids, as close as possible to the reservoir data.

Failure to provide a competent, impermeable barrier around the sample can result in the alteration of the wettability (2), the loss of the interstitial water (3), salt precipitation, and damage to the clay mineral fabric (4). Of

these, wettability (5, 6) and clay morphology (4) are regarded as the parameters most liable to change. The alteration of the hydrocarbons through the loss of the light ends and/or the deposition and oxidation of the heavy ends, with the resultant formation of polar products which can be surfactants, are the primary causes for wettability change. This may be accompanied by the deposition of an oil-wet residue on the grain surfaces as the core dries out (7 - 9). Illite bearing rocks have the potential to increase in permeability if cleaned, dried and subsequently measured. This is due to the collapse of the illite clay mineral structure by the passage of an interface to the grain surfaces (4).

This paper summarizes the preservation methods which are currently available to the oil industry. It also proposes an alternative method, which allows the core to be stored anaerobically by immersion under a compatible liquid. The results of the field trials and preliminary laboratory assessment performed on the Core Preservation Container are presented. This alternative technique has been based on the work done by Bokek *et al* (10). They recommended the use of glass lined steel tubes filled with de-oxygenated formation brine to maintain the wettability of core material over an extended period, prior to laboratory analysis.

METHODS OF PRESERVATION

In 1960, a variety of accepted practices for preserving cores was catalogued in the American Petroleum Institute's Recommended Practice of Core Analysis Procedure (11). The API RP 40 listed six preferred methods which they suggest should be used as experience dictates, given the nature of the rock, the projected storage time and the testing required. These six methods were :

1. Sealing in air-tight metal cans.
2. Sealing in steel, aluminium or plastic tubes, using suitable couplings, pipe caps, or O-ring seals.
3. Sealing in plastic bags.
4. Freezing with dry ice.
5. Wrapping in metal foil or plastic tape.
6. Coating with plastic.

Monicard (12) in 1980 produced a listing of preservation techniques in his review of core analysis, which were broadly similar to those of the API RP 40 and illustrated a lack of significant advancement in the intervening 20 years. The listing was as follows :

(a) Quick freezing method: the cores are stored in a dry ice chamber.

(b) Wrapping of cores with thin sheets of aluminium and paraffin wax coating.

(c) Use of plastic sacks which may be joined.

(d) Use of fitted boxes.

(e) Use of sealed tubes.

Monicard observed that (a) and (b) gave excellent results, while (c) was a "very special method".

The technique mentioned in both listings which has been most frequently used in the 1970's and '80's has been that of Hunt and Cobb (13) described as the "wrap and dip" method. This requires the core to be wrapped in layers of plastic film and aluminium foil, and sealed with a hot-melt, strippable plastic coating. However, laboratory screening in the late 1980's (13, 14) concluded that the technique was not wholly effective in maintaining an impermeable barrier between the core and the atmosphere, which resulted in the ingress of oxygen and the loss of pore fluids.

Hunt and Cobb proposed the use of a laminated heat-sealed package which their experimentation found was superior in providing high oxygen and water vapour barrier properties, as well as being resistant to chemical attack by core fluids. Auman (14) confirmed the suitability of these polypropylene laminates formed into heat-sealed packages and produced data which demonstrated the significant reduction in daily weight loss experienced by samples preserved in these laminates.

Despite the findings of these authors it is worthy of note that the "wrap and dip" method remains the dominant preservation technique in the North Sea Oil and Gas Province in early 1990.

RATIONALE FOR THE DEVELOPMENT OF AN ALTERNATIVE PRESERVATION TECHNIQUE

The introduction of the laminated package technique has advanced the wrap and seal approach to core preservation to a point where the possibility of core and pore fluid degradation has been restricted to an acceptable minimum. However, the technique does suffer from a number of apparent flaws. For example the extended delay between core recovery and final preservation in long cored sections, a quasi-anaerobic environment and the high possibility of puncturing the outer protective skin. These problems promoted the development of a viable alternative.

The criteria which the alternative technique had to satisfy may be summarised as follows :

1. Safety at the wellsite, with the removal of the need for a hot work permit as would be necessary for a heat sealer or a hot-melt strippable plastic bath.

2. Ease of handling at the wellsite, with the possibility of the selected core sections being preserved within minutes of recovery from the barrel, thus minimising the exposure to the atmosphere with the attendant problems of evaporation and contamination.

3. The provision of an anaerobic environment.

4. The prevention of evaporation and drying by liquid immersion.

5. The use of an inert preservation material to prevent an adverse reaction with the pore fluids, which may produce surfactants that could have an impact on the core's wetting preference.

6. The ability to view the core on demand, without cutting away the layers of preservation material which typify the wrap and seal techniques.

7. The facility to store the core over an extended period.

The Core Preservation Container (CPC) was the solution which appeared to satisfy these criteria.

CORE PRESERVATION CONTAINER - DESCRIPTION AND SPECIFICATIONS

The Core Preservation Container (hereafter referred to as a CPC) is a glass vessel with a sealable lid, which has the capacity to store core in an anaerobic environment.

The main body of the CPC is an elongated cylinder, closed at one end and constructed from toughened safety glass, with a low sodium content (Figure 1). The selection of glass was made in preference to a less fragile material such as polypropylene, due to its ability to withstand prolonged exposure to both oils and brines, without chemical alteration and degradation, and the subsequent generation of possibly harmful by-products. It became apparent during the field trials that the glass body required a degree of protection, other than that offered by the crate packaging, and an outer sleeve of "safe-glass" has been added. This particular coating is effective in absorbing impact shock generated by poor handling practises.

The glass body has at its open end, a formed lip, with a flattened upper surface, to complement the lid design which requires sealing by U ring.

The maximum carrying capacity of the body is a core of 11 inches in length by 5.25 inches in diameter.

Placed within the body is a free standing sample cradle made from a single piece of 316 stainless steel. The use of a single length of steel precludes the need to weld, which eliminates points of possible corrosion. The cradle has been designed to fit snugly into the body to prevent the lateral movement of the sample and there are Viton base platelets to absorb the shock caused by vertical travel of the core. In addition, folding handles have been attached to the cradle to allow for the rapid loading and unloading of the core.

The third component of the CPC is the moulded ABS composite lid with locking ring and U ring. The design of the lid is such that a positive pressure can be introduced into the CPC via a one-way inlet valve, and it can be vented as required through a separate release valve. The pressure within the vessel can be monitored by means of a gauge attached to the lid. Experimentation has shown that a pressure of between 15 and 20 psig can be maintained without significant loss over a six month period, although periodic checks are required to ensure any drop is corrected. Nitrogen is used as the pressure blanket gas to exclude oxygen and to maintain a buffer between the lid and the immersion liquid.

Research is currently being undertaken to investigate the suitability of argon as the blanket gas, given the nutritional value of nitrogen to bacteria, especially sulphur reducing bacteria. The growth of such organic matter can impair permeability should they invade the pore system of the preserved whole core.

IMMERSION LIQUID

The selection of an immersion liquid to be used in the CPC, should be primarily concerned with the maintenance of the wettability of the core material.

Bokek *et al* (10) and Richardson *et al* (15), suggested deoxygenated formation or synthetic brine, while Mungan (9) recommended that cores be stored in degassed crude oil. However, McGhee *et al* (16) and Morgan and Gordon (17) proposed the core is immersed in its wetting fluid, either formation brine or crude oil. The wettability being determined at the wellsite by a droplet imbibition test.

The recommendations made by McGhee *et al*, and Morgan and Gordon, in conjunction with experimentation performed for a number of clients, have been used as the basis for a set of guidelines, designed to assist in the choice of a suitable liquid.

1. Core from a Gas Well or the Water Leg - A deoxygenated (with added biocide) formation brine or simulated formation brine*.

2. Core from an Oil Leg, Water or Intermediate Wet (the wettability is determined by a droplet imbibition test for an exploration well, see Appendix No.2, or by previous wettability testing on core from an earlier well) - As recommendation No.1.

3. Core from an Oil Leg, Oil Wet (wettability determined as in recommendation No.2) - Preferably Crude Oil to minimize pore oil chemical alteration. If no crude oil is available a depolarized refined mineral oil with a viscosity/density identical to the crude oil to minimize diffusion†.

* If the formation brine or a brine analysis are not available, the preservation brine can be constructed based on the parameters of the liquid intended for use in the Special Core Analysis programme, such as a log Rw.

† If the viscosities/densities of the crude oil and the refined mineral oil are not matched, diffusion may result in the deposition of plugging compounds. In the absence of sufficient crude oil or the knowledge of its viscosity/ density, it is recommended that a high viscosity (40-50 cp.) depolarized refined mineral oil is used.

In addition to the maintenance of the wettability of the core material it is important to ensure the liquid selected is compatible with the mineralogical content of the core, especially the clay fraction. Recent work by Vaidya and Fogler (18) has demonstrated the inter-relationship between formation damage, pH and ion exchange and an earlier study by Mungan (19) attributed permeability reduction to changes in salinity and pH. Given the possibility of formation sensitivity linked to "water-shock", the immersion of cores in brines with salinity values significantly lower than the formation brine is not recommended.

The use of the drilling mud is also unacceptable as a preservation liquid, due to the possibility of the imbibition of the mud filtrate into the core's pore system. Such an invasion can cause emulsion blocking or surfactant contamination. Oil based muds are particularly problematical, because of the oil-wetting agents and the emulsifiers used in their construction and they should be avoided from the standpoint of the CPC.

FIELD TRIALS

A series of field trials were undertaken in both onshore and offshore locations to test the durability and suitability of the CPC's in a potentially hostile environment and to determine which packaging and handling method would minimise the possibility of the glass body being fractured during transport. In addition, the field trials were used to ascertain the most efficient routine for preserving a core in a CPC.

Following the initial trials it became apparent that while the CPC could be transported to the wellsite without difficulty and it did not suffer damage during the core preservation process, given judicious handling practices, vessel failure occurred between the rig and the laboratory. The source of the problem was found to be the unrestricted vertical and lateral movement of the core which caused impact fracturing of the glass.

To reduce the frequency of this type of damage a rigid crate with moulded foam packaging was introduced. This was complemented by a change in the design of the sample cradle to increase grip and the use of a foam insert, placed between the bottom of the cradle and the glass base. The insert is intended for shipping purposes only and is removed after the arrival of the CPC in the laboratory. Current investigation

is concerned with the replacement of the foam insert with glass wool or a similar inert material, to remove any possibility of an adverse reaction between the foam and the immersion liquid.

To further protect the glass from breakage, a "safe-glass" sleeve has been fitted around the body of the CPC. The sleeve minimises impact fracturing, and should failure occur, it prevents the complete loss of the immersion liquid.

Subsequent trials have shown the modifications to be successful, although it remains necessary to provide the shipping personnel with precautionary handling instructions.

The methodology for core preservation in a CPC was established during the course of the field experimentation. The recommended procedure is presented in Appendix (1).

The wellsite screening also confirmed two basic assumptions made when the concept of the CPC was originally discussed. Firstly the willingness of onsite safety personnel to allow preservation to be undertaken in areas normally off-limits to the portable heating bath and the heat sealer needed for the "wrap and seal" techniques. Secondly core dehydration and pore fluid oxidation/evaporation following core recovery can be minimised by the quickness of the preservation technique in reducing atmospheric exposure time.

EXPERIMENTAL ASSESSMENT

The potential of the CPC technique is currently being assessed through a series of long term screening experiments. These have been designed to determine such parameters as wettability alteration, crude oil degradation, pore water loss and clay fabric collapse. The initial findings, where available, are presented in this paper.

Reference is also made to a laboratory study performed by Statoil A/S, Norway, which has kindly been made available.

CLAY FABRIC PRESERVATION

If a core is allowed to dry, because of poor preservation practices, damage to the clay fabric can be caused either by the movement through the sample of an air-brine interface or the loss of the bound and interlayer water.

The particular mechanism is directly related to the clay minerals present. The Smectites (expandable clays such as montmorillonite) are susceptible to the loss of their bound and interlayered water through core dehydration. While illite, which has no interlayered water, can suffer gross morphological changes as the pore water is lost through drying. The increasing surface tension, associated with the air-brine interface, on the filaments and platelets, promotes the collapse of the clay on to the pore walls (4).

To test the effectiveness of the CPC in preserving the clay fabric, a core rich in expandable clays (XRD - 8% of Bulk Rock, Clay Fraction, 60% Smectite, 40% Illite and Illite/Smectite) was immersed in formation brine at the wellsite. Prior to immersion a chip was removed and placed

in a cloth bag. In the laboratory, after 60 days storage, this chip in conjunction with material from the preserved sample were subjected to SEM examination, following miscible cool solvent cleaning and Critical Point Drying preparation.

As shown in Figure 2, Photograph 1, the smectites in the preserved sample form honeycomb assemblages of connected platelets. No visible damage to this clay fabric could be determined.

The smectites in the unpreserved sample (Figure 2, Photograph 2) had suffered considerable damage, with the collapse of the honeycomb structures. This collapse is due to the passage of an interface during drying combined with the removal of interlayer water.

It is recognised that this comparison considers only the extremes of preservation and further work is being done to compare the CPC with the advanced forms of wrap and seal currently available.

WETTABILITY

The alteration of a core's wettability caused by its exposure to air and drying has been demonstrated by a number of authors. Amott (20), Treiber *et al* (8), Bartell and Niederhauser (21), and Richardson *et al* (15) present experimental evidence which suggests that through the oxidation of crude oil, there is a general movement in water-wet cores towards an oil-wet preference. However, Chilinger and Yen (22), and Mungan (9) show a reverse trend, with cores becoming more water-wet on exposure to air. Anderson (2), when reviewing these data sets, concludes that "it is impossible to predict how the wettability will be altered by the oxidation of the crude".

Given this interplay between wettability alteration, atmospheric exposure and drying, it was necessary to establish whether the CPC method of preservation could maintain a core's wettability during storage. A series of tests were initiated to provide the data for such an assessment. The testing was designed to investigate the relationship between possible wettability alteration and the length of time a core is stored in a CPC. This was combined with samples stored in other preservation materials to allow comparisons to be made.

The screening which has been completed to date and is available for publication is limited. It is restricted to testing performed on two cylindrical lengths of quarried sandstone from North East Scotland. Previous experimentation had shown this material to be strongly water wet. These cores were cut into two sets of three plugs. They were saturated with a compatible brine, flushed to irreducible water saturation with a typical North Sea crude oil and aged under the same crude for 500 hours at 200°F.

The wettability of one plug from each set was determined by the Amott (20) technique, to act as a control.

A second plug from each set was wrapped in a thin plastic film, aluminium foil and dipped in strippable plastic. The remaining two plugs were immersed in the brine, as the controls indicated a water-wet to intermediate wettability.

Both plugs were stored for 90 days, prior to the wettability of the four plugs being assessed by the Amott Technique.

The generated data from this initial screening exercise is presented in Table 1. The range of values is clearly limited, however, it is suggested that the first data set (1A/B/C) appears to indicate the CPC technique has maintained the wettability successfully, although the coated samples have not shown excessive alteration. An examination of the second data set (2A/B/C) does not provide any easily definable variation between the techniques.

It is recognised that the data base has to be expanded to allow for a more meaningful assessment of wettability maintenance and the CPC. The testing currently being performed should provide standard sandstones and reservoir core material and is encompassing the CPC, laminates and strippable wax.

TABLE 1 AMOTT WETTABILITY INDICES

SAMPLE	WETTABILITY INDEX WATER	OIL
1A	0.586	0.027
1B	0.490	0.088
1C	0.612	0.016
2A	0.703	0.014
2B	0.663	0.025
2C	0.677	0.034

SAMPLES 1A/2A = Controls
SAMPLES 1B/2B = Preserved in strippable wax.
SAMPLES 1C/2C = Immersed in brine sealed in a CPC.

Wettability Index = $\frac{\text{Liquid Spontaneously Imbibed}}{\text{Liquid Removed by Spontaneous Imbibition and Dynamic Displacement.}}$

CAPILLARY PRESSURE - IRREDUCIBLE WATER SATURATION (AFTER STATOIL, NORWAY)

The Production Laboratories of Statoil A/S performed an investigative examination of the influence of a range of preservation and cleaning techniques on selected Special Core Analysis parameters. A section of the study is presented in this paper to illustrate the variation in irreducible water saturation, derived by centrifugal displacement, which can occur when different preservation methods are employed.

Eight plugs were cut from a sandstone formation with a moderate to high clay content (kaolinite predominantly, illite and illite/smectite approximately 50% of the less than 2 micron fraction), taken from the oil leg of the reservoir. The base parameters of Klinkenberg Permeability, Porosity and

Pore Size Distribution of the 8 plugs, determined as part of the testing sequence, was broadly similar. The plugs were divided into 4 pairs, in accordance with the particular method used to preserve the whole core from which they were drilled: (A) immersed under refined oil in a CPC, (B) aluminium/ plastic laminate, (C) strippable plastic and (D) no protection.

The plugs were cleaned by cool solvent miscible displacement and dried by the Critical Point method. A gas-brine capillary test using the centrifuge technique was performed, followed by refluxing extraction with toluene and methanol, and drying in a humidity controlled oven. The gas-brine capillary pressure test was then repeated. From the two data sets, the final water saturation value (Swi) for each plug run was taken and the percentage variation between each pair was calculated. These values are presented in Table 2, with the Klinkenberg Permeability and the Porosity for each plug.

The aim of this measurement cycle was to investigate whether the choice of the preservation method could influence the effectiveness of these two commonly used cleaning and drying techniques. The variation in Swi was used as an indication of a possible relationship.

TABLE 2 IRREDUCIBLE WATER SATURATION

PLUG NO.	PRESERVATION TECHNIQUE	IRREDUCIBLE WATER SATURATION * (% VARIATION)	Kl (mD)	ϕ (%)
1	No Coating	-37	0.23	10.5
2	No Coating	- 8	0.26	11.7
3	Strippable Plastic	+ 3	1.40	13.6
4	Strippable Plastic	+19	0.61	12.9
5	Laminate	- 1	0.79	13.1
6	Laminate	- 3	0.68	12.9
7	CPC (Oil)	+ 2	0.91	13.6
8	CPC (Oil)	+ 1	0.80	13.7

* Swi (pre-soxhlet, run 1) - Swi (post-soxhlet, run 2).
Kl = Klinkenberg Corrected Gas Permeability, millidarcies.
ϕ = Porosity, percent.

An examination of the percentage variation between the two sets of irreducible water saturations shows the Swi values for the cores stored in strippable plastic and those without any coating to vary significantly between the measurement cycles. This is in contrast to the cores preserved in either the CPC under oil or the aluminium/plastic laminate, which show only minor variation between runs. Given the similarity between the 8 plugs, in terms of permeability, porosity and pore size distribution, it would seem probable that the plugs' Swi values would show only minor variations between

runs. This view presumes the plugs were sufficiently robust not to be unduly altered by soxhlet cleaning and humidity oven drying. This would seem to be the case for the 4 plugs which were preserved in the CPC and the laminate.

For the other 4 samples the significant variation in Swi would suggest the manner of preservation may be a contributing factor. The presumption being made is that the cool solvent miscible flushing and critical point drying could not remove the products of drying, evaporation and oxidation, as successfully as the hot solvents. These products being the result of ineffective preservation. Although there is only limited data available it may be for the samples with no coating, the negative trend indicates drying and evaporation was dominant, with salt precipitation and residual oil reducing the pore system and increasing capillary retention. For the 2 samples preserved in wax, oxidation of the crude oil may have been more important with a tendency to create slightly oil wet plugs. If this did occur the hot solvents may have been more successful in reversing the wetting preference towards water. Thereby increasing water retention after the hot solvents and causing a positive trend in the Swi.

CONCLUSIONS

The Core Preservation Container provides the means by which the potentially damaging effects of the drying, evaporation and oxidation of the core and its pore fluids, which may occur between the time of recovery and laboratory analysis, can be eliminated or at least minimized.

It is a rapid method which can be used successfully at the wellsite. The containers can be recycled and they allow for long term storage of core in a closed, controlled environment.

Further work is required to assess the long term storage effects of this method.

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APPENDIX 1. RECOMMENDED PROCEDURES FOR CORE PRESERVATION CONTAINER USAGE

WELLSITE

1. The selected immersion liquid should be prepared in sufficient quantities and made ready for use.

2. The foam insert should be located in the base of the glass vessel, beneath the sample cradle.

3. Label each CPC with all the available information, including company, well number, and core number. The exact depth of the preserved whole core sample can be added when the data is available.

4. Each CPC should be two-thirds full of selected immersion liquid and the lid loosely fastened.

5. The CPC's must be located either on the rig floor and at the core packing location, and marked sequentially.

6. Cores for preservation should be selected by the client's representative or removed by the catcher on a statistical basis. Exposure to the atmosphere should be kept to a minimum.

7. The core is placed in the CPC body and if necessary additional liquid is added until a slight gap exists between the liquid and the lid. The core should be consistently inserted with deepest surface at the base of the container.

8. The lid should be attached and the locking ring engaged in the direction indicated by the arrows on the tabs.

9. If core inspection or measurement is required this can be done prior to the introduction of the nitrogen blanket. A pressure of 15 psig is recommended.

10. The CPC's should be placed in the foam lined crates, the lid secured and the assembled units made ready for shipment.

11. The freight agent must be made aware of the contents of the crates and suitable care should be taken during loading and unloading.

ARRIVAL IN THE LABORATORY

1. All data pertaining to the CPC's and their contents should be cross checked and verified.

2. The foam insert should be removed.

3. The liquid level should be adjusted if necessary and the nitrogen blanket must be re-established pending possible analysis.

STORAGE

1. Periodic checks at 3 monthly intervals should be made of the pressure and liquid levels. Adjustments should be made as necessary.

2. The immersion liquid should be changed at regular intervals. It is recommended that the brine is replaced yearly, while crude oil and refined mineral oil can be changed once every 3-4 years.

APPENDIX 2. DROPLET IMBIBITION TEST.

A rapid assessment of a core's wettability can be achieved by the use of a simple droplet test. The results are qualitative and are intended only as a crude estimation.

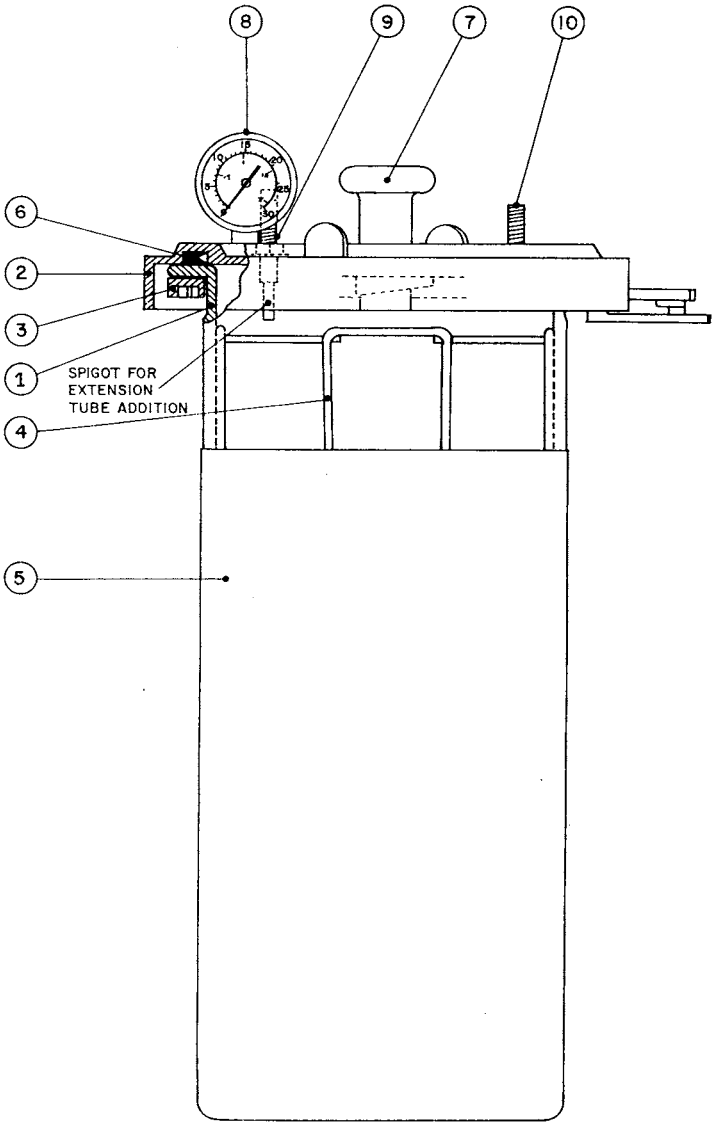
1. Remove fragments of rock from the centre of the core at representative sampling points.

2. Divide fragments into two equal parts ensuring a freshly exposed faced is present on both.

3. Place under a low-powered binocular microscope.

4. Via a pipette, deliver a single droplet of formation brine to one piece of core and a droplet of crude oil or refined mineral oil to the other. Estimate the initial contact angle and record the time required to allow for the spontaneous imbibition of the droplets.

5. Compare the information for the tests and make a qualitative judgement with regard to water, oil or intermediate wetting.

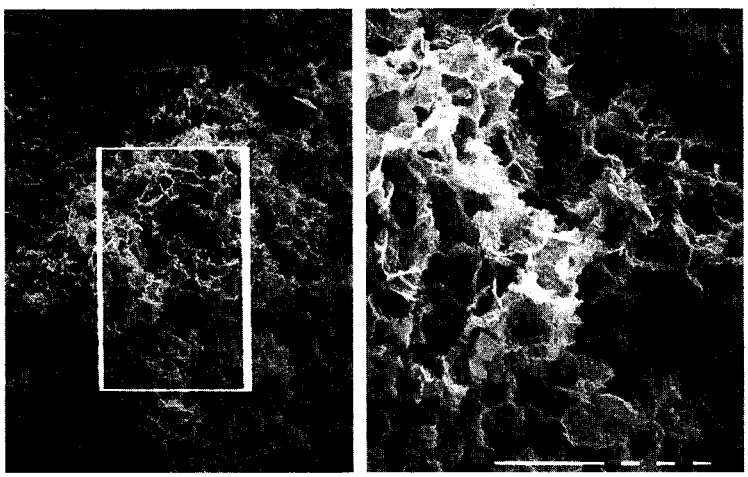


LEGEND

(1)	CORE SAMPLE JAR	(6)	HORIZONTAL 'U' RING
(2)	CORE SAMPLE JAR LID	(7)	HANDLE
(3)	CORE SAMPLE JAR LOCKING RING	(8)	PRESSURE GAUGE
(4)	CORE SAMPLE BASKET	(9)	INLET VALVE
(5)	PROTECTIVE SAFETY SLEEVE	(10)	RELEASE VALVE

FIGURE 1 CORE PRESERVATION CONTAINER ASSEMBLY

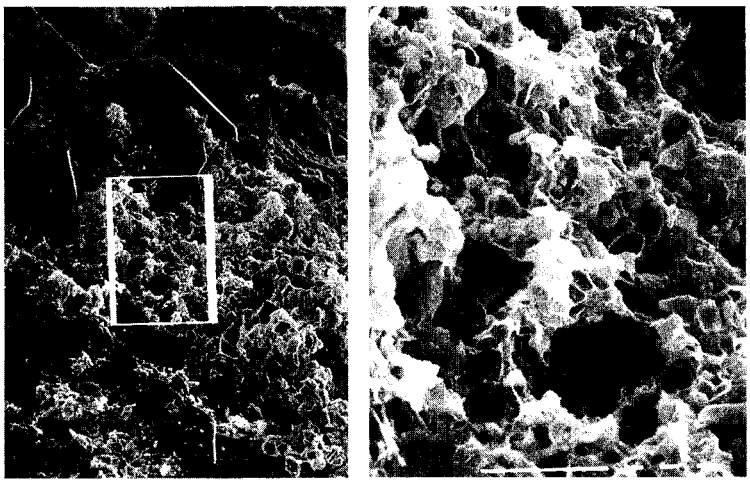
FIGURE 2 SEM PHOTOMICROGRAPHS - CLAY FABRIC PRESERVATION



Photograph 1

After Critical Point Drying and Preservin in a CPC

Left Scale Bar = 100 microns



Photograph 2

After Air Drying on an unpreserved sample

Left Scale Bar = 100 microns