

SHALY SANDS: MULTIPLE SALINITY AT VARYING BRINE SATURATIONS.

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ABSTRACT

The combination of Continuous Injection (CI) and Multiple Salinity (CoCw) tests, leading to what the authors have termed CtCw plots, has given rise to a more comprehensive technique for measuring the BQv of Shaly Sandstones, than the traditional CoCw test which only uses fully brine saturated samples.

Utilising recent advances at Imperial College, which allow the preparation of synthetic core samples containing specific clay types, disseminated throughout the sample, it is possible to calibrate the CtCw data to give more representative excess brine conductivity data for reservoir core samples. Synthetic samples, containing measured percentages of kaolinite, montmorillonite, chlorite and illite, are used to provide the calibration data for multiple salinity measurements on reservoir cores.

The use of multiple CIs on each sample, using different brine concentrations, generates a series of multiple salinity plots at decreasing brine saturations. Overlaying the plots for decreasing water saturations, derives the effective BQv for each saturation, and this data may be used to obtain a more representative calibration of downhole resistivity logs.

Results show that the shale effect can vary with brine saturation. In particular, the shale effect can be considerably higher at low brine saturation. Whilst the results for kaolinite seem anomalous and require further examination, montmorillonite, chlorite and illite all produce greater BQv measurements at lower brine saturations.

INTRODUCTION

The problem of calibrating downhole resistivity logs has never been solved satisfactorily. In the laboratory there are various traditional methods used to measure shaliness:

Cation Exchange Capacity (CEC)

This is a wet chemistry method which utilises a crushed rock sample. The crushing mechanism can sometimes break up clay minerals, thus exposing more shaly sites to the chemicals than may be available in the whole rock sample. If this happens, the shaly measurement will be erroneously high.

Multiple Salinity (Co/Cw)

This method measures the electrical conductivity of a rock sample saturated with a series of brines of different salinities. However, the measurement is only ever made on fully

brine saturated samples. This is an ideal method for anyone interested in reservoirs full of water and containing no oil.

Membrane Potential (Qv)

This method uses a rock sample as a membrane between two brines of different salinity in an electrochemical cell; Thomas [6]. The measurement can be made on fully brine saturated samples and although it can use rock chips or sidewall samples, mud contamination could compromise the results.

RELATED LITERATURE

Ever since Archie [1] described how the downhole resistivity log could be used for determining the fluid saturation of a clean sand formation, researchers have attempted to find ways of applying similar formulae to resistivity measurements of shaly sands. It was found by Hill and Milburn [2] that clay minerals in a sandstone would affect the electrical properties such that, at low brine saturations, downhole resistivity logs were much less reliable. The same researchers measured the cation-exchange capacities of their core samples in an attempt to quantify the shaliness of the cores. Waxman and Smits [3] developed an equation from careful core measurements relating the electrical properties of brine saturated shaly sand with its clay content and extended the model to oil bearing shaly sands. Waxman and Thomas [4], while confirming that the Waxman and Smits equation held true for most resistivity logs in hydrocarbon-bearing shaly sands, also agreed with Hill and Milburn that the effective shaliness of a sandstone increases proportionally with decreasing brine saturation, and these finds were corroborated by Koerperich [5], although his experiments were on unconsolidated cores. Thomas [6] produced Qv from membrane potential data measured on drill cuttings and sidewall cores, to confirm the Waxman-Smits model. Worthington [7] stressed that shale effects on electrical properties are governed by the concentration of the formation brine as well as its degree of saturation of the core, and pointed out that a formation with a “clean” water zone may turn out to have a “shaly” oil zone. Glennie [8] described how diagenetic clay formation can cease as the reservoir fills with hydrocarbon, thus leaving the water zone with a larger shale effect than the oil zone. Worthington [9] confirmed this, showing how early assumptions of equal porosity in the oil and water zones could be seriously flawed. In carefully controlled laboratory experiments, Campos and Hilchie [10] showed that measurement of cation exchange capacity (C.E.C.) by crushing the sample must be treated with great caution, as the smaller the size of the ground particles, the greater the surface area of any clays, and this statement was upheld by Hardwick [11], who described a new technique for measuring the shale parameter more accurately than, and as efficiently, as C.E.C. analysis. Another non-destructive method of C.E.C. analysis is given by Austin and Ganley [12], but this has been disputed. Hardwick also showed that the shale effect was less when the core was saturated with fairly dilute brine solutions, but pointed out that as the brine concentration was reduced the shale effect became more important. This was in contradiction to an earlier paper by Patnode and Wyllie [13], who deduced that the conductivity of shaly rock was independent of the salinity of the brine. Several researchers, including Yuan [14], point out that clay conductivity generally decreases with increasing dilution of the formation brine, but

stress that this depends on the clay type. Jing [15] described a modification of the Waxman-Smits model, incorporating a temperature dependant coefficient and clay distribution coefficient which is dependant on whether the clay is in laminations, or evenly distributed, but much of Jing's work was on synthetic cores. Jing and Archer [16] also measured the electrical properties of shaly rocks at reservoir pressures and temperatures and concluded that both parameters should be taken into account when these measurements are made.

Brine saturation is of fundamental importance during this research programme and several researchers have considered this parameter during electrical property measurements. Dunlap et al. [17] performed resistivity measurements on core samples during porous plate capillary pressure measurements, where the saturation of the cores was changed by stepwise changing of the capillary pressure, while Diederix [18] measured resistivity index at low brine saturations with an effective overburden of 70 bar and concluded that anomalies were caused by roughness of the sand grains. Maerefat et al [19] gave guidelines on saturating and desaturating techniques for use during resistivity measurements and recommended that where possible, these measurements should be done at reservoir conditions. Worthington et al [20] insist that laboratory studies are necessary to obtain reliable values of saturation exponents since rocks with varying pore types and size can have saturation exponents which change with varying brine saturation. Sondena et al [21] describe, how in certain circumstances, water-wet samples give the same saturation exponents at room conditions as at reservoir conditions.

OBJECTIVE

The objective of this research was to present a more comprehensive multiple salinity technique, which measures shaliness at varying brine saturations, and aims to identify reservoirs where the shaliness effect changes with saturation. These measurements were made at the lowest brine saturations achievable with the equipment available, as it is at these very low saturations that the electrical properties of the clays cause the greatest anomalies in the downhole resistivity logs, and that an undetected shale effect could cause a productive oil bearing zone to be overlooked. The technique allows measurements to be done at representative reservoir overburden pressure. This, in turn, should allow better calibration of downhole resistivity logs.

TECHNIQUE

For this research synthetic shaly sand core plug samples have been manufactured at Imperial College in London, by a technique pioneered by Dr. X.D. Jing [22]. These synthetic samples contain different percentages of montmorillonite (smectite), chlorite, illite and kaolinite and, only one clay type is present in each sample. The method for preparing these samples is given in Appendix A.

The experimental procedure is as follows:-

- Saturate sample in synthetic brine
- Measure Formation Resistivity Factor (FRF) at ambient and overburden pressure

- Run Continuous Injection Formation Resistivity Index (FRI)
- Flush clean (cold solvent) to prevent possible clay damage
- Saturate with second brine of different salinity
- Repeat FRF and FRI
- Flush clean and repeat FRF and FRI using third brine
- Repeat using fourth brine

Using the data from the four continuous injections, the sample conductivity is plotted against the brine conductivity for 100% saturation, 90%, 80%, 70%, etc., down to the lowest saturation achievable. This gives a series of plots, of which the 100% brine saturation plot is the CoCw. All lower brine saturations give what the authors have termed CtCw plots.

For each sample, the plots of decreasing water saturation, are superimposed to show how the shaliness measurement, BQv, varies with saturation for each clay type and how it varies with the percentage of clay in the sample. Superimposition of the four FRI plots for each sample to give a composite FRI plot is used to give confidence in the associated CtCw data. The data is considered most accurate when the values of the four saturation exponents are the same or very close.

It is hoped that later Environmental SEM (ESEM) measurements on each of the synthetic samples will show these synthetic samples to be comparable with natural rock samples of similar clay content. Magnetic Susceptibility measurements are also included for comparison with natural rocks and are given in Table 1.

Table 1. Magnetic Susceptibility for each Clay type, measured at Heriot-Watt (for 5% illite, 8% montmorillonite, 8% chlorite and 5% kaolinite)

Sample	Mass Magnetic Susceptibility ($10^{-8} \text{ m}^3 \text{ kg}^{-1}$)
SI5	0.75
SM8	1.08
SC8	1.64
SK5	-0.10

DISCUSSION

This phase of the project is now complete and although further work is required, a pattern has emerged for each clay type tested.

Examples of composite FRI and CtCw plots are given in Figures 1 - 12 for the four different clay types. On the CtCw plots, montmorillonite, chlorite and illite all show larger BQvs for lower brine saturations. In particular, the data for montmorillonite (Figures 1 – 6) show a measurable increase in BQv with increase in clay content and with decrease in brine saturation, whilst the plots for the chlorite sample (Figures 7 – 8) and the illite sample (Figures 9 – 10) are showing a consistent increase in BQv with decrease in saturation. It is our intention to deduce calibration data for each clay type, which can then be used during the measurement of reservoir core samples to correct BQv measurements for downhole resistivity tool calibration. To do this, it will be necessary to

make CtCw measurements on a larger number of samples, especially for chlorite and illite.

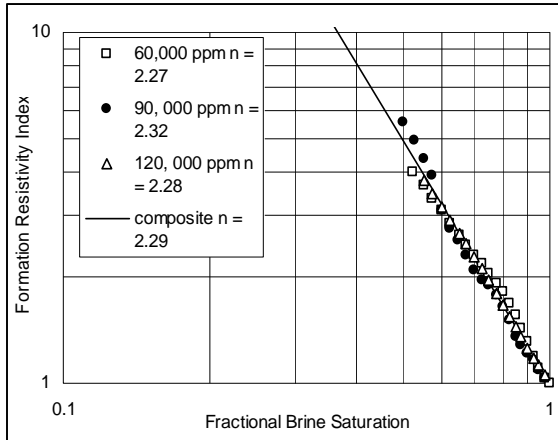


Figure 1:
Composite FRI for 3% Montmorillonite

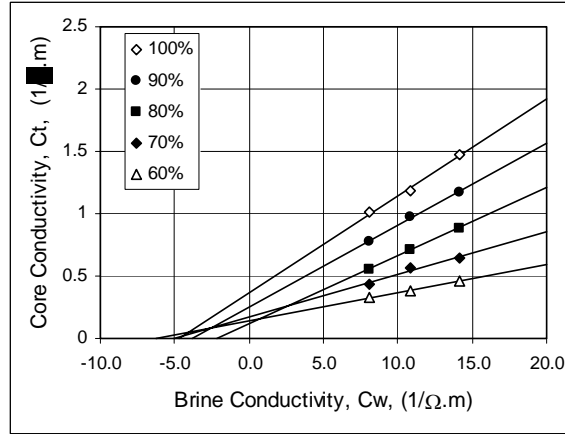


Figure 2:
Multiple Salinity for 3% Montmorillonite

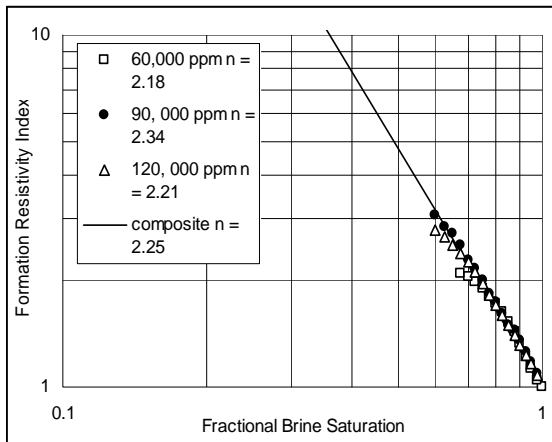


Figure 3:
Composite FRI for 8% Montmorillonite

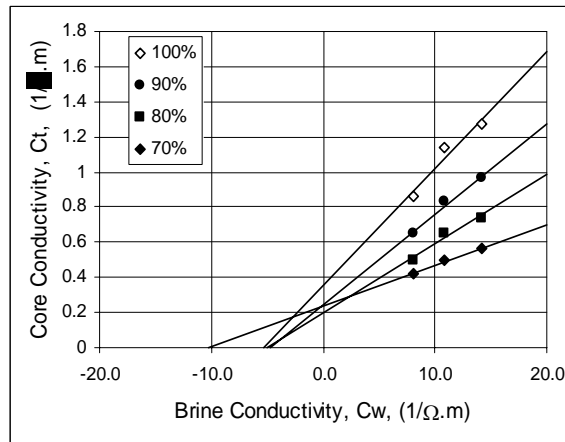


Figure 4:
Multiple Salinity for 8% Montmorillonite

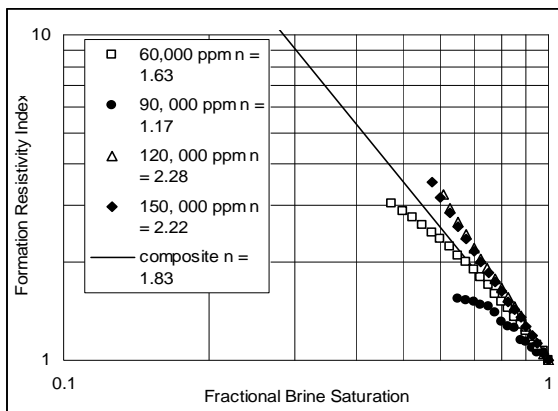


Figure 5:
Composite FRI for 10% Montmorillonite

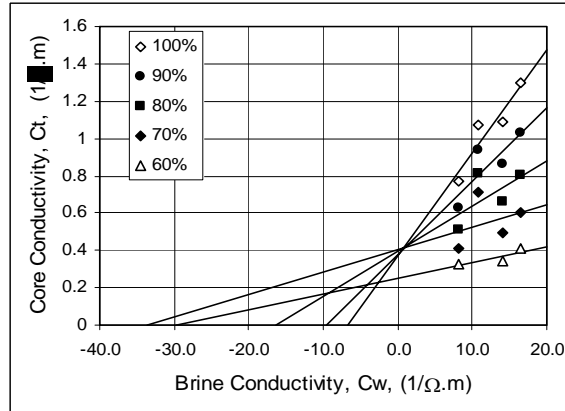


Figure 6:
Multiple Salinity for 10% Montmorillonite

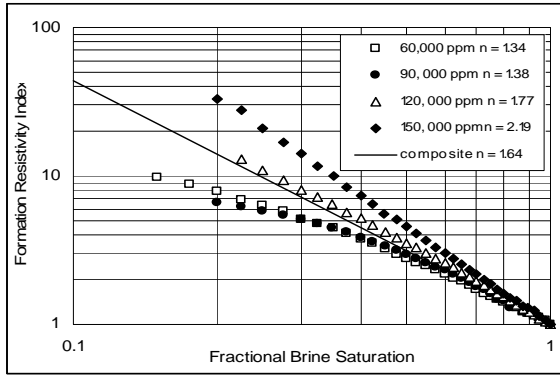


Figure 7:
Composite FRI for 8% Chlorite

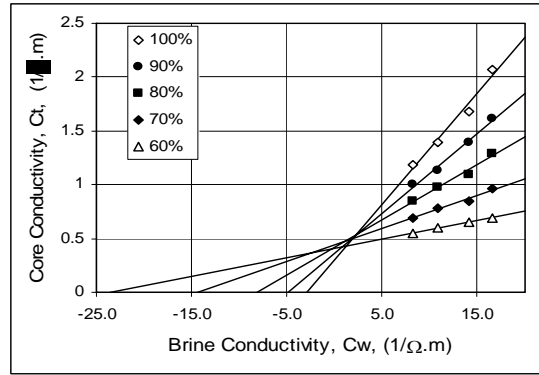


Figure 8:
Multiple Salinity for 8% Chlorite

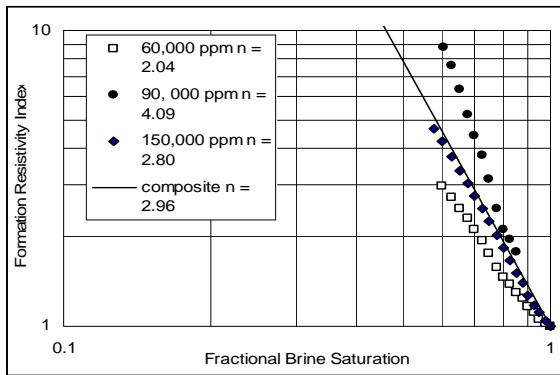


Figure 9:
Composite FRI for 5% Illite

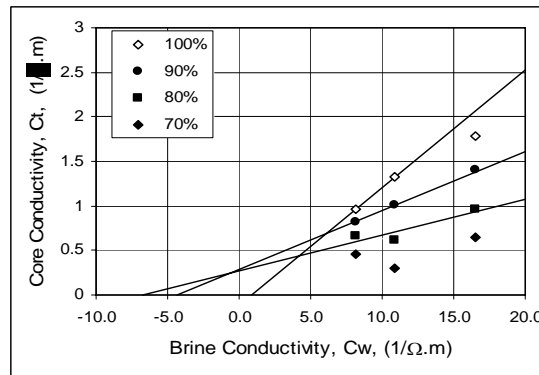


Figure 10:
Multiple Salinity for 5% Illite

The kaolinite samples produced anomalous results. As the sample saturations decreased, the BQv, rather than showing an increase, or, as was expected, no change at all, showed a decrease (Figures 11 - 12). This was felt to be an artefact of the measuring technique, as samples containing this clay, in particular, appeared to be more affected by overburden hysteresis than samples containing the other clay types. It was also pointed out to us that kaolinite was always considered to be neutral to shale effect. However, the results of magnetic susceptibility measurements on a selection of these synthetic clay samples have pointed to the possibility that, rather than being neutral to the shale effect, samples containing kaolinite may have a negative shale effect, which becomes greater as the brine saturation is decreased. There is insufficient data as yet to support this, as the magnetic susceptibility measurements were only made on four samples, each containing a different one of these four clay types. However, since the magnetic susceptibility and the CtCw measurements for kaolinite seem to point in the same direction, this is clearly an area for further research. Later ESEM data may help to resolve this seeming anomaly. Further work would be useful in evaluating other less common clay types and the results will be analysed using the Waxman-Smiths model.

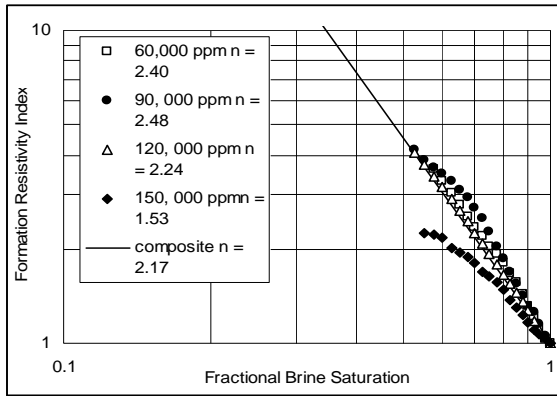


Figure 11:
Composite FRI for 5% Kaolinite

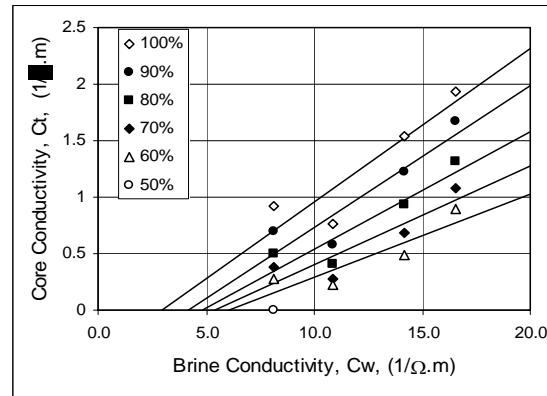


Figure 12:
Multiple Salinity for 5% Kaolinite

CONCLUSIONS

Combining Formation Resistivity Index (FRI) with Multiple Salinity (CoCw) tests gives a series of linked CoCw plots for each core sample, which give a shaliness measurement (BQv) at several different brine saturations, and show that the shale effect can vary with brine saturation. In particular, the shale effect can be considerably higher at low brine saturation, for rocks containing the main clay types found in shaly sandstones. Kaolinite, however, would appear to buck this trend, as it seems to give lower BQv measurements at lower brine saturation. Correctly, the conductivity of a partially brine saturated sample is termed Ct (Co being reserved for fully brine saturated samples). The authors have thus called these new plots CtCw plots.

The methodology described in this paper should produce a more accurate measurement of Excess Clay Conductivity.

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Synthetic shaly sandstone samples for this project were manufactured at Imperial College, London.

NOMENCLATURE

- B = Equivalent conductance of the clay exchange cations (sodium), as a function of C_w .
- Q_v = Concentration of sodium exchange cations associated with the clay.
- C_w = Conductivity of brine.
- C_o = Conductivity of the fully brine saturated core sample.
- C_t = Conductivity of the partially brine saturated core sample.
- FRF = Formation Resistivity Factor.
- FRI = Formation Resistivity Index.

REFERENCES

1. ARCHIE G.E. "The Electrical Resistivity Log as an Aid in Determining Some Reservoir Characteristics", Trans., AIME (1942) Vol. 146, pp 54-62.
2. HILL H.J., and J.D. MILLBURN, "Effect of Clay and Water Salinity on Electronical Behaviour of Reservoir Rocks", Trans., AIME (1956) Vol. 207, pp 65-72.
3. WAXMAN M.H., and L.J. SMITS, "Electrical Conductivities in Oil - Bearing Shaly Sands", Soc. Pet. Eng. J. (June 1968), pp 107-122.
4. WAXMAN M.H., and E.C. THOMAS, "Electrical Conductivities in Shaly Sands - (I) The Relation between Hydrocarbon Saturation and Resistivity Index: (II) The Temperature Coefficient of electrical Conductivity", J. Pet. Tech. (February 1974), pp 213-225.
5. KOERPERICH E.A., "Utilisation of Waxman-Smits Equations for Determining Oil Saturation in a Low-Salinity, Shaly Sand Reservoir", J. Pet. Tech. (October 1975), pp 1204-1208.
6. THOMAS, E.C., "The Determination of Q_v from Membrane Potential Measurements on Shaly Sands", JPT, (September 1976), pp 1087-1096.
7. WORTHINGTON P.F., "Influence of Shale Effects Upon the Electrical Resistivity of Reservoir Rocks", Geophys Prospect. 30 (1982), pp 673-687.
8. GLENNIE K.W., "Introduction to the Petroleum Geology of the North Sea", Sec. Ed. (1984) 3, pp 63-85.
9. WORTHINGTON P.J., "The Relationship of Aquifer Petrophysics to Hydrocarbons Evaluation", Quart, J. Eng. Geol., Lon. (1986) Vol. 19, pp 97-107.
10. CAMPOS J.C., and D.W. HILCHIE, "The Effects of Sample Grinding on Cation Exchange Capacity Measurements", SPWLA, Ann. Log. Symp. (1980).

11. HARDWICK A., "XIs : A new Shale Parameter for Resistivity Log Evaluation", SPE 19576 (1989).
12. AUSTIN S.P. and S.M. GANLEY, "Measurement of the Cation Exchange Capacity of Core Plugs by a Non-destructive 'Wet' Chemical Method", Advances in Core Evaluation II, Ed. Worthington and Longeron (1991).
13. PATNODE H.W. and M.R. WYLLIE, "The presence of Conductive Solids in Reservoir Rocks as a Factor in Electric Log Interpretation", Trans. AIME (1950) Vol. 189, pp 47-52.
14. YUAN H.H., "Salinity Dependence of Shaly Sand Parameters from Membrane Potential Measurements", SCA Conf. (1991).
15. JING X.D., "The effect of Temperature and Clay Distribution on Shaly Sands Conductivity", SPE Europe Student Paper Contest (1990).
16. JING X.D., and J.S. ARCHER, "Special Core Analysis Considerations in the Determination of electrical Properties of Shaly Rocks at Reservoir Conditions", Advances in Core Evaluation II, Worthington and Longeron (1991).
17. DUNLAP H.F., H.L. BILHARTZ, E. SHULER, and C.R. BAILEY, "The Relation between Electrical Resistivity and Brine Saturation in Reservoir Rocks", Trans. AIME (91949), TP 2711 (1940).
18. DIEDREX K.M., "Anomalous Relationships between Resistivity Index and Water Saturations in the Rotliegend Sandstone (The Netherlands)", SPWLA Ann. Log. Symp. (1982).
19. MAEREFAT N.L., B. BALDWIN, A. CHAVES, G. LATORRACA, and B. SWANSON, "Guidelines for Saturating and Desaturating Techniques of Core Plugs during Electrical Resistivity Measurements", sub. comm. SCA Elec. Work. Comm. (1988).
20. WORTHINGTON P.F., N. PALLATT, and J.E. TOUSSAINT-JACKSON, "Influence of Microporosity upon the Evaluation of Hydrocarbon Saturation", SPE 14296, (1985).
21. SONDENA E., F. BRATTELI, H.P. NORMAN, and K. KILLTVEIT, "The Effect of Reservoir Conditions on Saturation Exponent and Capillary Pressure Curve for Water-Wet Samples", Advances in Core Evaluation I, Ed. Worthington (1990).

22. JING X.D., “The Effect of Clay, Pressure and Temperature on the Electrical and Hydraulic Properties of Real and Synthetic Rocks”, Ph.D. thesis, Imperial College, London (1990).

23. DENICOL P.S., “Effect of Pore Geometry, Clay Content and Fluid Saturation on the Complex Impedance of Reservoir Rocks”, M.Phil. thesis, Imperial College, London (1996).

APPENDIX A

Synthetic Core Sample Preparation At Imperial College, London

The use of synthetic samples allows the systematic control of clay content and clay type. Fifteen synthetic samples with various clay concentrations and clay types were prepared for this study. As the main clay types found in shaly sandstones are montmorillonite, chlorite, illite and kaolinite, these four clays were used in the synthetic sample manufacture, with one clay type in each sample.

Two quartz sands with different sizes were used in the preparation of the samples, 45-425 microns and 0-125 microns. Equal weight of both sands were mixed with the required amount of clay in order to produce the desired clay concentration. The mixture was placed on a mixing roller for 12 hours, to a homogeneous mixture between the quartz and clay. Subsequently, the mixture was poured into a PTFE shrink sleeve, then filter paper and stainless steel meshes were placed at the top of the sample to prevent any fines migration, after which the sample was capped and the grain volume and density determined by He expansion. Subsequently, the sample was saturated with brine (5% sodium chloride and 1% potassium chloride) and the initial water volume of the samples was determined by weight. After saturation the samples were placed into Viton sleeves and loaded into a multi-sample pressure vessel, which has a maximum working pressure of 69 MPa. The confining pressure in the vessel is controlled with a hydraulic pressure intensifier. The samples are connected to a pore pressure system, which contains five calibrated glass burettes, partially filled with oil and brine. The burettes are used for volume displacement measurement during application of stress, in order to calculate the sample pore volume at each stress point.

The samples were repeatedly loaded and unloaded over successive cycles covering a hydrostatic pressure range from 500 psi to 2000 psi, following the procedure outlined previously by Jing [22] and Denicol [23]. During the cycles the volume of brine exiting the sample, as well as the electrical resistance were recorded. Each confining pressure was kept constant until no changes in pore volume and resistivity were detected. The pressure was then increased or decreased depending on the cycle. A large amount of hysteresis between the loading and unloading paths was observed during the first two cycles, however, the hysteresis was greatly reduced in the subsequent cycles. Consequently, the stress cycles were stopped when subsequent paths almost tracked each other.