

# ACQUISITION OF CORE CAPILLARY PRESSURE DATA BY IN-SITU SATURATION MONITORING – A COMPARATIVE EVALUATION

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## ABSTRACT

Accurate quantification of hydrocarbon saturation (1-  $S_w$ ), a key volumetric parameter in reservoir characterisation, has a profound impact on the economic development potential of oil and gas fields. Drainage capillary pressure ( $P_c$ ) data acquired from core are required to model initial water saturation ( $S_w$ ) distribution in the reservoir as a function of height above the free water level. Imbibition ( $S_w$  increasing)  $P_c$  is not considered in this paper.

$P_c$  data can be acquired from core by a variety of methods – traditionally by ultracentrifuge, porous plate and air-mercury injection. Each technique has its own advantages and disadvantages – in terms of how well the  $P_c$  curve is characterised. The ultracentrifuge offers several advantages including achieving higher capillary pressure and much shorter test duration than porous plate. A disadvantage is that the  $P_c$  curve is constructed indirectly, based upon  $S_w$  calculated from a theoretical model(s)[15]. Although the accuracy of  $P_c$  curve modelling improved significantly during the 1990's based upon the work of such authors as Forbes [6], the capacity to form a robust but very detailed characterisation of the  $P_c$  curve remains elusive. If  $S_w$  distribution within the core plug can be directly visualised using In Situ Monitoring (ISSM) techniques then it should be possible to utilise ultracentrifuge based  $P_c$  data with greater confidence.

ISSM methods historically encompassed the use of X- and gamma-rays and have routinely been associated with relative permeability experimentation. More recently the ISSM portfolio has been expanded with the inclusion of applied NMR, in conjunction with  $P_c$  profiling. Whilst several papers have been authored by the principal proponent of the NMR based ISSM method, Green Imaging Technology (GIT), an independent verification of this method had not been performed. To address this issue a set of core plugs was obtained for which conventional  $P_c$  data was available. This suite of plugs underwent  $P_c$  acquisition by ultracentrifuge, combined with ISSM. The testing was conducted at wholly independent laboratories, with all reference data held only by the principal author, to ensure the complete integrity of the subsequent evaluation. The effectiveness of the combined ISSM centrifuge method is investigated by comparative analysis of the derived data sets.

## INTRODUCTION

Correct prediction of water saturation in the reservoir model has a profound impact on reservoir economics, drilling and completion strategies, EOR etc. The shape of the drainage  $P_c$  curve across the transition zone is of particular importance.

This was exemplified by Abu Shiekah et al [1] for Shuaiba Transition Zone fields where most of the Hydrocarbon Initially In Place (HIIP) is in the capillary transition zone, often in low-dipping structures spread over extensive areas. The amount of recoverable oil in a transition zone depends on the distribution of initial oil saturation ( $S_{oi}$ ) as a function of depth, as well as the relative permeability and  $P_c$  characteristics. This vividly illustrates the importance of accurately defining the initial water saturation distribution.

The purpose of this study is to investigate ISSM based methods for improved definition of the drainage  $P_c$  curve from centrifuge. ISSM has been available as an analytical tool in core analysis for over 60 years. Initially, X-Ray systems provided the means for saturation visualization [3, 7, 12 & 14], whilst gamma radiation has also been employed. Of particular relevance to the current study is work by Maloney et al [13] and Spindler & Maloney [16] – in the latter,  $P_c$  from centrifuge was acquired by X-Ray ISSM. NMR has provided an additional means of monitoring saturation in conjunction with  $P_c$  determination. The use of NMR was reported in 1991 by Baldwin et al. [2] and much more recently in 2005 by Chen & Balcom [4]. Green et al [9 & 10] presented the first attempts at commercial application of the technology – specifically for  $P_c$  measurement. Whilst Green Imaging Technology provided evidence of the validity of their patented NMR technology, by comparison of GIT  $P_c$  data with operator provided data, a completely independent verification has not been conducted.

Helix RDS, who are an oil and gas consultancy, orchestrated an independent investigation of drainage  $P_c$  data acquired by ISSM. It was arranged that a set of test samples were subject to centrifuge drainage  $P_c$  measurement in conjunction with ISSM by both X-ray and NMR. In addition pre-existing  $P_c$  data was available for the same samples.

All analyses were proposed and arranged by Helix RDS without any communication between the laboratories at which the testing was conducted – X-Ray ISSM (Corex UK) and NMR ISSM (GIT). Corex were aware that the X-Ray ISSM work that they were performing was to be compared directly with the NMR ISSM data. Corex were unaware that they themselves had performed the conventional  $P_c$  measurements on the test samples, several years prior to the current study (2004) - measures were taken to ensure sample anonymity prior to the current study commencing. The primary author held all original and new data none of which was released to the labs until after final data had been reported. Ensuring data confidentiality is of primary importance for an independent consultancy.

It should be noted that all of the laboratory analyses performed during the study were performed free of charge for the purposes of verification of the centrifuge ISSM procedure. This represents a considerable investment of time and resources for the advancement of the principles of data acquisition and potential commercial utilization of ISSM technologies.

The objective of this paper, building upon the work of previous authors, is to investigate the application of ISSM in conjunction with centrifuge for improved  $P_c$  determination.

### **Core Materials**

The Forties sandstone core plugs employed in this study had previously comprised part of a SCAL study conducted on core acquired from a UK North Sea oil reservoir. The operator, Talisman Energy Limited, kindly donated 5 plugs for use in this study. The Forties sandstone, of Palaeocene age, is a submarine turbidite which hosts many of the most well known oil and gas fields in the UK North Sea, including Forties (5 BBN bbl OIIP) and Montrose (UK's first producing offshore field). The quality of the sandstone varies from outstanding to poor depending upon proximity to the main sand body and the distance from sand source.

The samples incorporated in this study exhibit medium - high porosity (range 17.4% - 25.5%) accompanied by low to moderate permeability (1.4 – 158 mD) – Figure 1. Most samples exhibit a bi-modal or broad pore throat size distribution (PTSD) with abundant micro porosity generally associated with authigenic clay. They are described as fine grained poorly sorted sandstones with occasional coarser grained laminations.

The broad PTSD's typical of this sand body resulted in an extended transition zone in the reservoir from which the test samples were acquired - an important issue when planning field development, well placement etc.

All samples were physically robust and free of defects such as chips or fractures.

### **Methods**

The conventional Pc tests performed during the initial SCAL study consisted of:

- Air/Brine Porous Plate (soil moisture cell) at ambient (unconfined) conditions,
- Air/Brine Ultracentrifuge (Beckman L8), and
- Air/Mercury high pressure mercury injection (Micromeritics Autopore IV).

ISSM was used to characterise the centrifuge induced Pc curve as fully discussed in the following sections. During the ISSM Pc determination, conventional centrifuge data was recorded, i.e. end-face Pc and average saturation, however, this was only available for 3 spin speeds and so is insufficient to fully characterize the Pc curve conventionally (typically requires  $\geq 5$ ). In theory, a single centrifugation cycle (spin speed) could be sufficient to capture the properties of a Pc curve, however, in reality this is difficult to achieve and a more practical approach is to perform at least 3 (Green et al. [9]). This still represents a significant time saving over the conventional centrifuge method, as well as the advantage of directly measuring saturation. Performing more than 3 cycles of centrifugation would begin to erode the time saving advantages of the combined centrifuge ISSM method.

On completion of the centrifuge ISSM by NMR a trim (off-cut) was removed from each sample upon which high pressure mercury injection capillary pressure (MICP) was performed.

It should be noted that the combined centrifuge ISSM procedure has potential limitations:

- Saturation – re-distribution occurring between removal of sample from centrifuge, prior to ISSM – studies conducted by GIT [9&10] concluded this effect is minimal assuming the scanning is performed rapidly (minutes) after centrifugation;

- NMR – sample cannot be confined at stress – a particular issue for poorly consolidated formations;
- NMR – for oil/brine systems use of D2O is required to discriminate oil from water.

A general assumption for centrifuge based Pc determination is that the test sample is uniform (homogeneous) due to the 1-D nature of the interpretation. Although the Forties sandstone is a turbidite with significant local heterogeneity, CT scanning of the core prior to SCAL ensured the test samples were largely homogeneous.

### **Generic Sample Preparation and ISSM Procedures**

The core plugs had previously been fully cleaned using hot refluxing solvents (methanol followed by toluene) using Soxhlet extraction, then dried at 105<sup>o</sup>C. These methods had been proven to be non-damaging based upon a sample preparation pre-study. Helium porosity and air permeability at ambient conditions were re-measured to validate the sample condition by comparison to the original values. The following step-by-step generic ISSM procedures were carried out.

1. A baseline ISSM scan was performed at 0% brine saturated condition.
2. The samples were evacuated, then pressure saturated with brine.
3. Brine saturation was validated by comparing the gravimetric saturated pore volume with the helium pore volume.
4. A baseline ISSM scan was performed at 100% brine saturated condition.
5. The sample was placed in an ultracentrifuge and rotation initiated. The rotor was spun at the lowest selected speed for 48 hours. The volume of expelled brine was monitored throughout the period and the final volume.
6. The sample was removed from the centrifuge and scanned at partially de-saturated conditions using the appropriate ISSM method. Average saturation confirmed gravimetrically.
7. Steps 3 & 4 were repeated at 2 further (higher) centrifuge speeds.
8. On completion of the final (3<sup>rd</sup>) centrifugation / ISSM cycle, the sample was extracted in hot methanol and oven dried. Helium pore volume was re-measured to ensure calculated saturations were accurately reported.
9. The ISSM data was transformed to saturation by the appropriate procedure for each of the experimental stages.
10. The 3 segments of the Pc curves were amalgamated and the appropriate procedure applied to derive a composite drainage Pc curve.

The centrifuge rotational speeds employed were calculated based upon the original Pc data available for the test samples – initially 2000 revolutions per minute (RPM), followed by 3000 RPM and finally 5500 RPM. The centrifuge geometries differed between the 2 laboratories, resulting in different sample mid-point effective Pc values – for Corex this approximated to 14, 30 and 107 psi air-brine (at the 3 increasing speeds) and for GIT, 23, 50 & 170 psi. Maximum Pc at the sample end-face for the final centrifuge speed approximated to 180 and 300 psi respectively. The original conventional Pc data was derived at end-face Pc values close to 5, 10, 30, 70, 120 and 180 psi (samples 2 & 3 only). The ISSM technique should provide data equivalent to centrifugation at a large number of conventional centrifugation cycles, in a single or small number of ISSM cycles. The Pc's achieved were sufficient to derive a detailed characterisation of the Pc curves when considered in conjunction with the ISSM data.

### Corex ISSM Procedure

The samples were saturated with brine containing an X-ray dopant (sodium iodide) to provide the necessary level of contrast between the brine and gas phases during the experiment. Each plug was transferred to a holding vessel, weighed and loaded into the X-ray ISSM apparatus. The system had been previously calibrated for each sample in a dried and fully brine saturated condition. The plug sample was marked with a reference point to ensure consistent orientation relative to the X-ray beam. A scan was performed along the length of the plug, utilising defined slice size, slice frequency and a slice scanning time. A further scan was performed after repositioning the plug after a 90° rotation along the plug axis relative to the X-ray beam – the purpose of this procedure was to ensure that variation in saturation due to rock heterogeneity was fully visualised by the X-ray beam (Figure 2 left, based upon sample 1). The scanning system incorporates a fixed start position for consistent sample location and a precision drive system, ensuring that removal and replacement of the sample from the scanning system has minimal impact on the measured saturation profiles.

The data recorded in the form of X-ray counts were converted to fluid saturations in association with the calibration output through the application of the formula:

$$S_b = 1 - (\text{Log}C_x - \text{Log}C_{100}) / (\text{Log}C_0 - \text{Log}C_{100}) \quad (1)$$

Where:  $S_b$  = saturation of blocking fluid (brine), fraction  
 $C_x$  = intermediate X-ray counts.

When the saturation of the blocking fluid ( $S_b$ ) is 1.0,  $C_x = C_{100}$ . When  $S_b$  is 0.0,  $C_x = C_0$ .

The saturation values for the twin scans from each stage of centrifugation were averaged to produce a single saturation vs. Pc dataset.

The Pc was calculated from the each rotational speed, at each position along the sample length using the industry standard procedure. The composite drainage Pc curve was derived by Helix RDS, using a best fit curve fit to combined saturation vs. Pc data for the 3 experimental phases.

### Green Imaging Technology ISSM Procedure

The NMR experiments were carried out on a 0.18 Tesla Bruker LF90 MiniSpec (Bruker Optics, Houston, TX) and proprietary software and hardware from GIT. The LF90 MiniSpec is a low-field NMR instrument and the effective spin-spin relaxation time ( $T2^*$ ) is much longer than the phase encoding time ( $t_p$ ). This ensures the SPRITE NMR profile is a simple fluid saturation with no other NMR relaxation dependencies (Figure 2, centre – based upon sample 1 – note when comparing to Figure 2, left,  $S_w$  displayed as fraction of Bulk Volume).

The fluid content profiles along the length of the core before and after centrifugation were determined by one-dimensional double half k-space SPRITE NMR with a phase encoding time of 50  $\mu$ s, field of view of 7cm, flip angle of 15 degrees and a resolution of 64 points. This technique was presented by Green et al. [9 & 10] as a robust method for producing quantitative saturation images of sedimentary rock. The number of signal averages was varied to achieve the desired signal to noise ratio of 100. This results in more data points on the capillary pressure curve.

Saturation was measured as a function of position along the sample using NMR. The saturation profile measured after centrifugation was divided by the 100% saturated profile, giving the saturation as a function of radius,  $S_w(r)$ . This, together with  $P_c$  as a function of radius,  $P_c(r)$ , was plotted to produce the  $P_c$  curve (Green et al. [9 & 10]).

Concerns with regard to removal and replacement of the sample from the test equipment – a concern for X-Ray ISSM – impacting upon measure saturation is inapplicable for NMR. The NMR signal is measured multiple times with different linear magnetic field variation (gradient) across the sample. Each acquisition however measures all the water in the rock and positional information is 'encoded' into the phase or frequency of the signal. Avoidance of moving source or sample avoids scattering which may occur in Gamma or X-Ray systems. In addition, NMR specifically measures fluid components of the system and matrix effects are minimised by the SPRITE techniques, whilst X-Ray systems for example are dominated by matrix effects and relatively insensitive to fluids.

The QUICK-CAP (Green et al [7]) procedure as established by GIT was used to derive a composite drainage  $P_c$  curve. QUICK-CAP uses established  $P_c$  models (Brooks-Corey [3] and van Genuchten [17]) to parameterize the data combined with standard and patented (Orthogonal Least Squared Fit™) error summation and minimization procedures.

The NMR T2 data was also used to extract pore throat size data from the  $P_c$  curve by computing the change in saturation at each pressure (i.e. the reverse of the mercury intrusion method). Then the pore throat size data was converted to NMR equivalent  $P_c$ , based upon the capillary bundle model (Wasburn [18]):

$$P_c = (2 * \sigma * \cos(\theta) / r \text{ (um)}) = 144 / r \text{ (um)} \quad (2)$$

Where:  $\sigma$  is Interfacial Tension,  $\theta$  is the Contact Angle and  $r$  is the pore throat radius.

### **Comparison of $P_c$ Data from Various Methods**

The principal objective of this study was to independently evaluate the integrity of the ISSM technique for  $P_c$  determination. As ISSM is a direct measurement, this also allows us to determine if the “true”  $P_c$  curve matches that from the original centrifuge models [11].

In order to evaluate the ISSM data, reference or “benchmark”  $P_c$  data is required - available from centrifuge (samples 2 & 3), porous plate (samples 1, 4 & 5) and MICP (all samples).

A principal advantage of the ISSM technique for characterizing the centrifuge  $P_c$  curve is the avoidance of assumptions that are inherent in the conventional centrifuge method. From this perspective the ISSM curve could be considered to be the “benchmark” relative to a conventional interpretation. In this respect it would be preferable to achieve a good match between the  $P_c$  curves derived from X-ray and NMR ISSM, rather than to the conventional centrifuge data (Forbes [6] interpretation). A further limitation upon conventional centrifuge data is selection of centrifuge rotation speeds – whilst these can be reasonably estimated (from MICP for instance) the level of success is unknown until the experiment is complete.

Porous plate Pc, when conducted in a coherent and consistent manner, should provide a good reference Pc curve. Often (as in this case) the technique provides limited detail on the Pc curve shape due to the practical (time) limit on the number of de-saturation pressure increments applied (typically 5-6). If the pressure increments are inappropriately selected this may further compromise the derived data. Porous plate data for the study samples was obtained using a Soil Moisture Corporation “1500f1 Pressure Plate Extractor” with a 15 Bar extractor plate. Measurements were conducted at unconfined conditions, requiring the samples to be removed from the test cell at each pressure step (5, 10, 30, 70, 120 & 180 psi). Care was taken to ensure capillary contact was re-established between the test sample and the de-saturation media on completion of each gravimetric saturation determination.

MICP provides a very detailed characterisation of the drainage Pc curve (typically up to 200 data points). As the MICP test is only performed upon part (trim) of the full core plugs, there is an inherent assumption when comparing to Pc data acquired on the plug, that the same is homogeneous and the off cut is representative of the whole sample. A closure (surface conformance) correction was applied to raw MICP data. Greder et al [8] concluded that MICP data is generally more optimistic than data from porous plate, depending upon the sample properties – in clean sandstones for instance MICP data was found to agree very well with conventional oil/brine Pc data. The test samples were determined from XRD (whole rock and clay fraction) to contain  $\leq 10\%$  clay – typically 25% Kaolinite & 75% chlorite. Chlorite was observed in thin section and SEM to commonly form a grain coating and pore filling cement. Based upon the work of Geder, the observed clay content would be expected to result in a MICP being more optimistic than Pc from other methods.

Although the centrifugation speeds applied during the original and new data acquisition were consistent, differing centrifuge geometries meant that average saturations achieved at each speed were not comparable. It was also not straightforward to define an “absolute” reference Pc curve, given potential uncertainties associated with the original Pc data from centrifuge (samples 2 & 3 only) and ambient porous plate (samples 1, 4 & 5 only). A qualitative visual comparison of the data displayed in graphical form serves as an one method of evaluation of the ISSM Pc data. In most cases, clear correlations are clearly visible between the available conventional Pc data, the ISSM Pc data. In addition, for each core plug, the two ISSM Pc curves and a “reference Pc curve” (centrifuge or porous plate) were sampled and error bars plotted (Figures 3 - 7) of the ISSM data relative to the “reference data”. Arguably, certainly where the ISSM Pc are in good agreement, they could be considered to be a more robust “reference” data.

The author aims to highlight the principal conclusions of this comparative evaluation in the following section, however, the reader is invited to examine the data and form their own opinion. This study was logistically limited to 5 samples.

## **RESULTS & DISCUSSION**

The results of the experimental procedures described in this paper can be examined in Figures 3-7. Summary observations are provided below:

- X-ray and NMR ISSM Pc curves generally agree well at air/brine Pc > 50 psi. Below 50 psi the NMR Pc curve generally appears more pessimistic predicting a higher entry pressure (P<sub>ce</sub>) than the X-ray ISSM or the conventional Pc curves.

- The GIT curve fitting procedure appears to result in higher Pce than would be inferred from a simple curve fit to the best fit raw saturation vs. Pc data (Figure 2, right).
- Following on from the previous observations, X-ray ISSM appears to better characterise the lower part of the Pc curve (< 50 psi air-brine), based upon the observation that there is good general agreement with conventional Pc data (Figures 3-7).
- Conventional centrifuge characterisation of the Pc curve (samples 2 & 3) is in good general agreement with the X-ray ISSM Pc.
- At higher capillary pressures (100 psi air-brine) the air-brine porous plate is more optimistic (Figures 6 & 7).
- MICP supports the Pce implied from X-ray ISSM and the Pc curves from centrifuge and porous plate (as far as Pce can be estimated from the latter 2 methods). At higher Pc (> 50 psi air-brine) the mercury injection Pc curve is more optimistic than the majority of the other Pc curves (excepting porous plate in 2 cases) – potentially due to the clay content of the samples [8].
- The NMR T2 based Pc curve is a poor match to any conventional or ISSM based Pc curves – especially at low Pc. The T2 Pc curve has very low Pce, is pessimistic and is rather featureless (Figures 3 – 7).

## CONCLUSIONS

1. In this work, we have compared ISSM based Pc measurement techniques with conventional Pc data. The combined ISSM centrifuge technique requires fewer centrifuge equilibration steps and thus decreases the measurement time significantly. In addition, it is believed that the ISSM measurement should be inherently more accurate because the water saturation is directly measured in the rock.
2. We conclude that the results of the current study have validated the combined ISSM centrifuge technique for drainage Pc determination.
3. Observed agreement between the X-ray ISSM and conventional Pc data suggests that the NMR Pc interpretation methodology may benefit from some refinement at low Pc.
4. Accurate quantification of HIIP benefits from as detailed a characterisation of the drainage Pc curve as can practically be achieved. ISSM based Pc by centrifuge has the potential to accurately quantify HIIP when applied to create a suitable saturation height equation.
5. The potential for the combined ISSM centrifuge method to rapidly characterise the drainage Pc curves suggest that it merits wider adoption by the core analysis community. Utilisation of robust and verified new procedures is key to ensuring that core analysis remains central to modern, dynamic and integrated reservoir description. No technique is exclusive and the combined centrifuge ISSM procedure is one of range of potential methods for determining drainage Pc depending upon the specific application.

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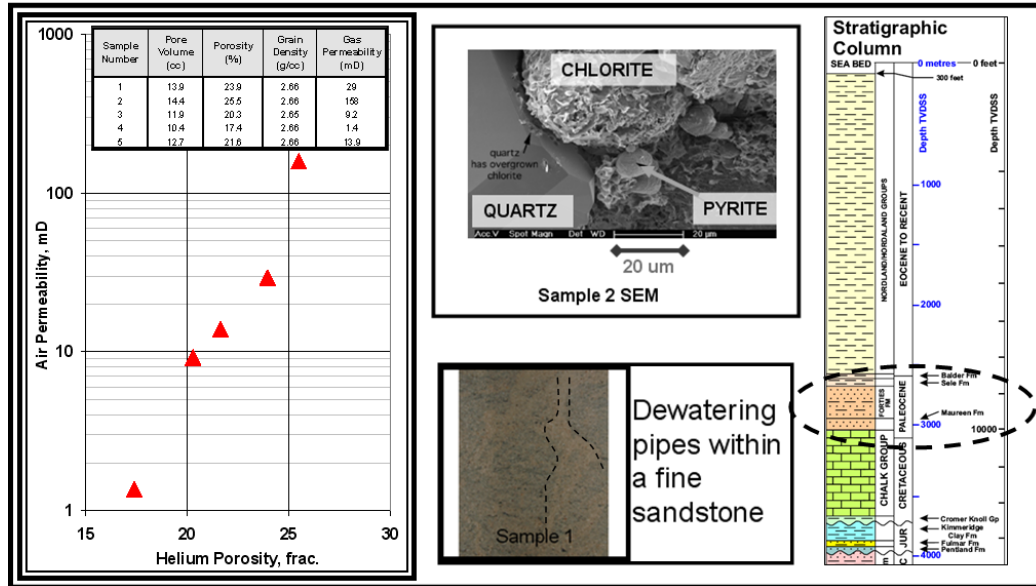


Figure 1: Petrophysical Properties of Test Samples at Ambient Conditions

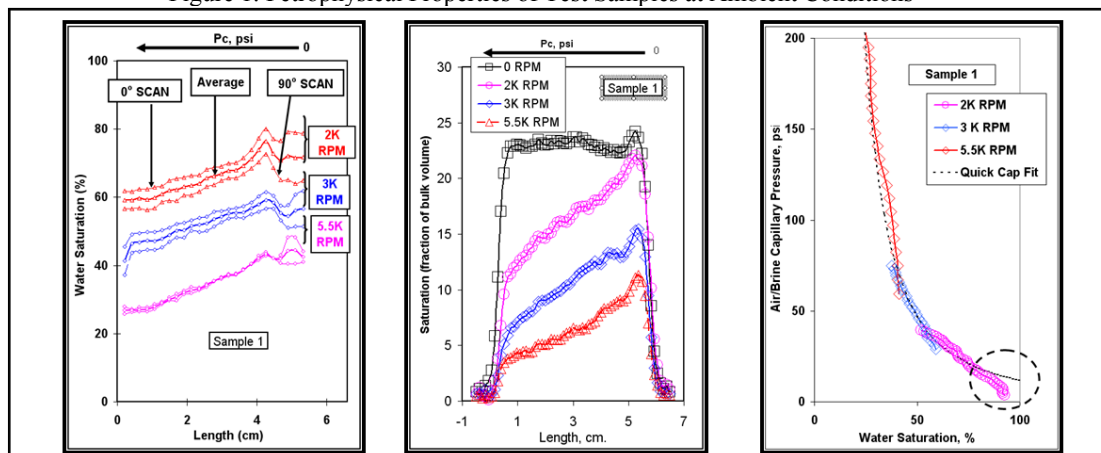


Figure 2: X-Ray Profiles (left), NMR Profiles (centre) & Construction of Pc curve (right)

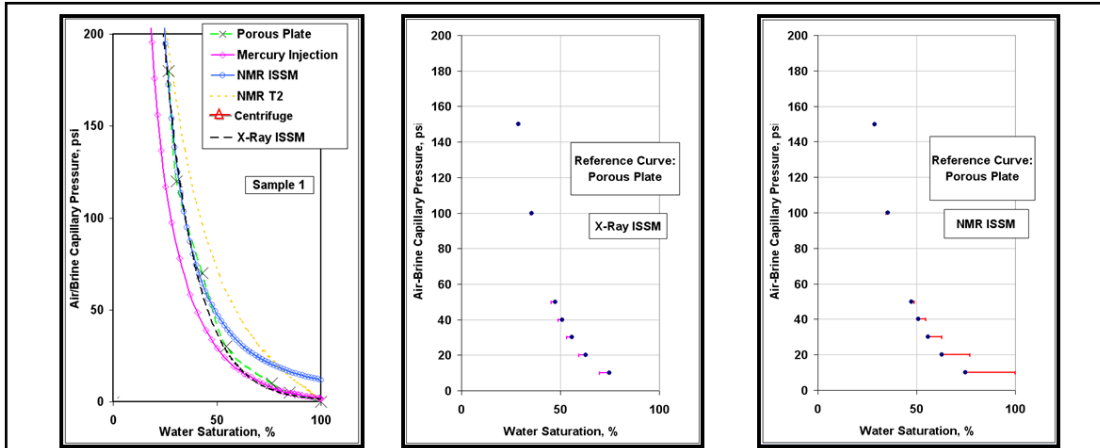


Figure 3: Spl. 1 - All Pc curves (left), X-Ray (centre) & NMR error (right) vs. reference Pc

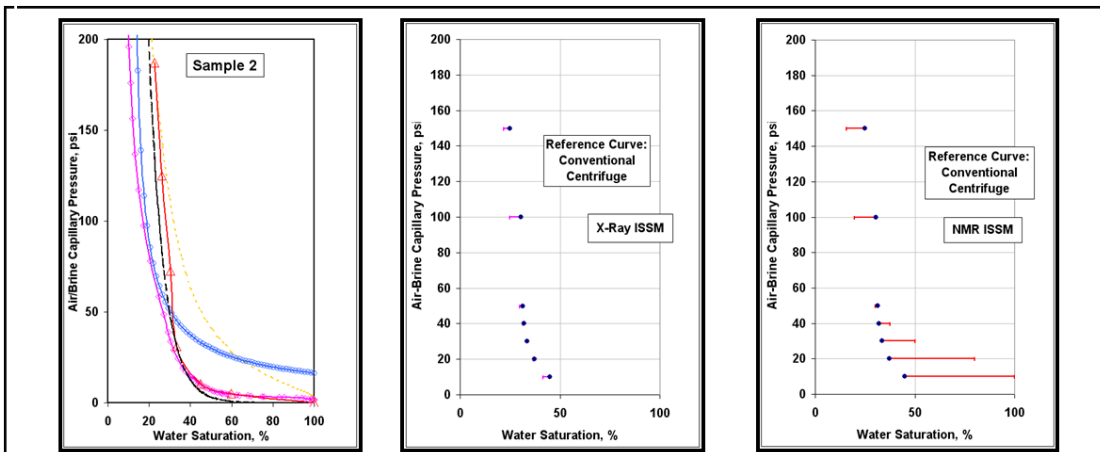


Figure 4: Spl. 2 - All Pc curves (left), X-Ray (centre) & NMR error (right) vs. reference Pc

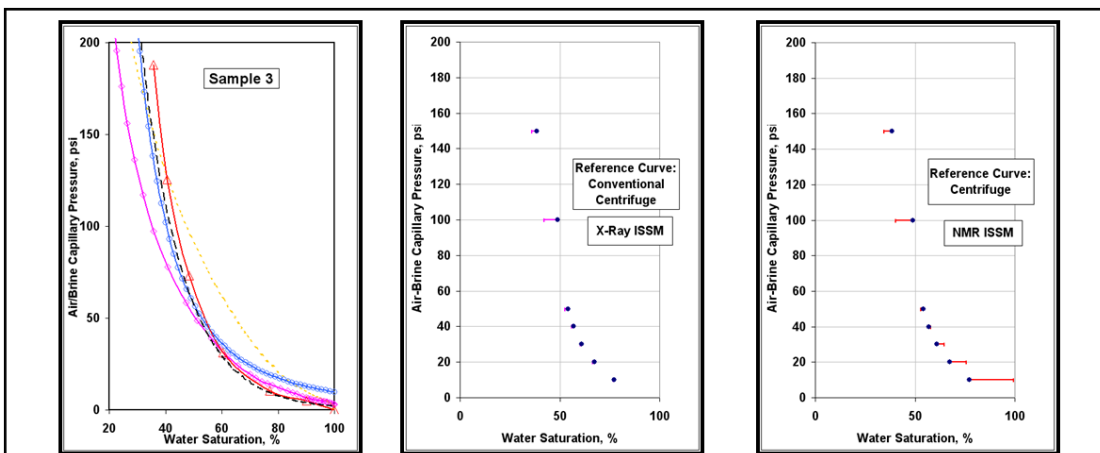


Figure 5: Spl. 3 - All Pc curves (left), X-Ray (centre) & NMR error (right) vs. reference Pc

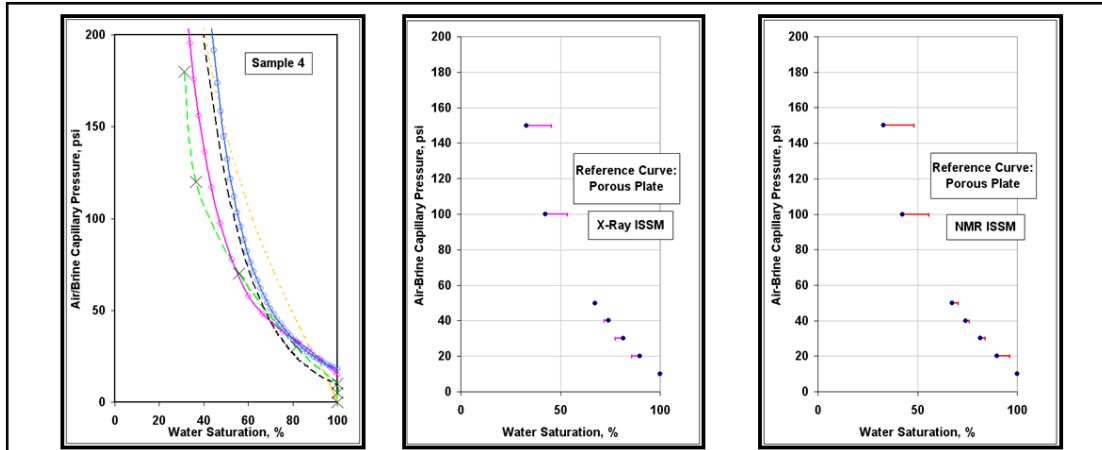


Figure 6: Spl. 4 - All Pc curves (left), X-Ray (centre) & NMR error (right) vs. reference Pc

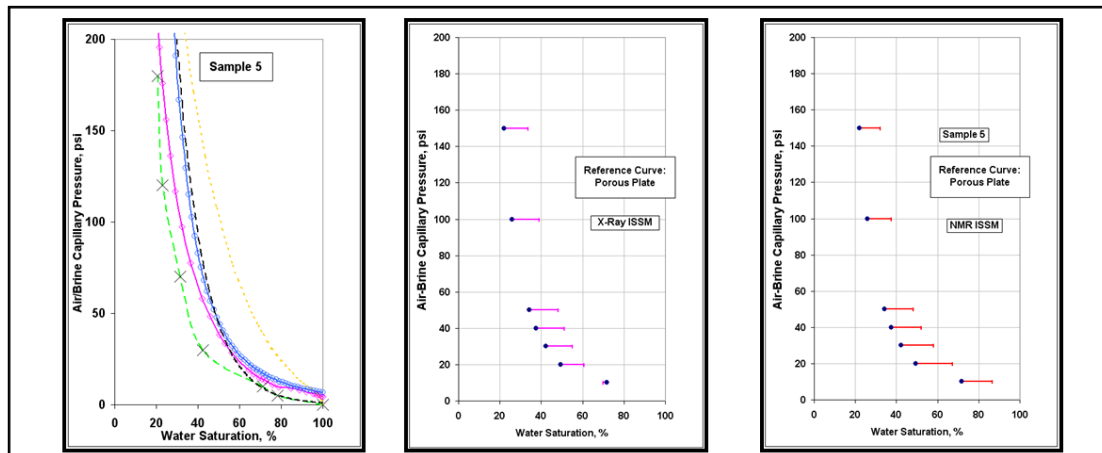


Figure 7: Spl. 5 - All Pc curves (left), X-Ray (centre) & NMR error (right) vs. reference Pc