# AN INVESTIGATION INTO THE EFFECT OF CLAY TYPE, VOLUME AND DISTRIBUTION ON NMR MEASUREMENTS IN SANDSTONES

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## ABSTRACT
Nuclear Magnetic Resonance (NMR) measurements on reservoir core samples are beneficial for improved interpretation of NMR log data.  Estimates of permeability, pore size distributions, free fluid and bound volumes from NMR logs are significantly improved if the logs are calibrated with NMR measurements on representative and well characterised core samples.

We present results from a study into the effects of clay type and volume on NMR $T_2$ spectra of synthetic sandstone packs. Samples containing varying amounts of randomly distributed montmorillonite or illite clay have been constructed.  The porosity and permeability for each sample were measured at elevated stress. The relationship between these petrophysical properties and the $T_2$ spectra is investigated. An analysis of the applicability of various NMR permeability models is discussed.  Samples were desaturated using the continuous oil injection technique and irreducible water saturations calculated. The effect of clay type and volume on the $T_2$ cut-off between free fluid and bound fluid is investigated.

## INTRODUCTION
### NMR Theory
CPMG sequence (Carr-Purcell-Meiboom-Gill) is used to measure $T_2$. It comprises a 90° pulse, followed by a train of 180° pulses. This pulse sequence eliminates effects due to local variations in magnetic field. Therefore the signal decay is due to interactions with neighbouring spins and surfaces. The CPMG pulse sequence takes only a few seconds to run, and therefore is a more practical measurement both in the laboratory and for downhole logging.

*Pore size effect:* In a porous rock system, there will be a continuous range of pore sizes, rather than several discrete sizes.  This means that the CPMG echo-train comprises of a continuous range of relaxation times.  Each pore-size has a distinctive $T_2$ value.  The echo-train corresponding to one particular pore-size will have a characteristic $T_2$ value and signal amplitude proportional to the amount of fluid contained in pores of that size. The

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resulting echo-train therefore consists of a continuous distribution of $T_2$ values each with different signal amplitudes. It is mathematically too difficult to deconvolute the data by fitting to a continuous distribution of exponential decays. The best way to tackle the problem is to make an approximation, selecting a certain number of $T_2$ values (say 50) over a specified time range (usually evenly spaced in logarithmic time), and fit the echo-train using these values, calculating the signal amplitude associated with each one. The problem becomes mathematically much simpler. The result is plotted as a $T_2$ distribution.

The nuclei diffuse randomly in a fluid, and in a porous system some will come in contact with the pore surfaces, allowing them to relax faster (by energy transfer to the pore wall). In the fast diffusion limit, the relaxation time observed experimentally is an average relaxation time for all the nuclei in the pore. Therefore in a small pore, the nuclei are more likely to interact with the surface, and so the average relaxation time will be faster (shorter) than in a large pore.

*Capillary-bound water:* In a water-wet rock, relaxation of hydrogen protons in the water occupying the smallest pores occurs as a result of interaction with the pore surfaces. Hence the part of the $T_2$ distribution relating to water-filled pores (capillary-bound water) contains information about pore size. The shape of the $T_2$ distribution for bound water (BVI) depends on the range in pore size. The default upper limit for BVI in sandstones is set at 33ms. These values are based on empirical observations relating pore size and $T_2$ relaxation at ~100psi air/brine capillary pressure. Some very short $T_2$ values may also result from water lining the walls of larger, oil-filled pores. In contrast, some very long $T_2$'s may result from water trapped in large pores with narrow throats (e.g. vugs). Clay-bound water (CBW) is associated with very small pores. Oil, in a water-wet rock, does not interact with the pore surfaces and relaxation is governed by the interaction between hydrogen nuclei within the fluid. Hence, the $T_2$ distribution for oil is a function of the density of hydrogen protons. Proton density is governed by viscosity. In an oil-wet rock, the signal for oil and water may switch (i.e. water relaxes at bulk, and oil relaxes in relation to pore size).

*$T_2$ Cutoff:* The typical $T_2$ cutoff for bound fluid in sandstones is 33ms. Surface relaxivity depends in part on the relaxation time of the fluid in a few molecular layers next to the pore surface, thus surface roughness and ionic exchange capacity (CEC) are important factors. Use of a single cutoff implies pores below a certain size are completely saturated (i.e. the wetting phase saturation is confined to smaller and smaller pores as the non-wetting saturation increases). The cutoff need not be constrained to 33ms, and may vary through the formation.

$T_2$ cutoffs are conventionally calculated from the point where the end-point water saturation (Swi) intercepts the $T_2$ curve such that:

$$Sw_{irr} = \sum_{T_2 = 0.1ms}^{T,cutoff} A(T_2) \qquad (1)$$

where  A = signal amplitude in the brine saturated $T_2$ distribution.

   $Sw_{irr}$ = irreducible water saturation after desaturation to end-point.

NMR Response Types are a set of similar NMR distributions that summarise the specific characteristics of the rock (Walsgrove *et al.* 1997).

**NMR PERMEABILITY**
Two permeability equations were applied to the brine-saturated $T_2$ distributions.  These are the Coates dual-water model (Coates et al. 1991) and the Kenyon (or SDR) equation (Kenyon et al. 1986).

*Coates Equation*
The standard Coates equation has the form:

$$KmD = C \cdot \left( \frac{FFI}{BVI} \right)^a \cdot \left( \frac{f}{d} \right)^b \qquad \textbf{(2)}$$

Where:
FFI= Volume of free fluid, where FFI=$\phi_{NMR}$-BVI (%)
BVI= Volume of bound fluid, determined using individual $T_2$ cutoff values
$\phi$ = NMR porosity (%)
C =A constant used to scale NMR permeability to units of milliDarcies
a = A variable exponent, usually set to 2
b = A variable exponent, usually set to 4
d = A variable denominator, usually set to 10

*Kenyon (SDR) Equation*
The standard Kenyon (SDR) equation has the form:

$$k(mD) = C \cdot \left[ (T_{2LM})^a \left( \frac{f}{d} \right)^b \right] \qquad \textbf{(3)}$$

Where:
$T_{2LM}$ = Geometric mean $T_2$ value (ms)
$\phi$ = NMR porosity (%)
C =A constant used to scale NMR permeability to units of millidarcies
a = A variable exponent, usually set to 2
b = A variable exponent, usually set to 4
d = a variable denominator, usually set to 10

**PROCEDURE**

***Sample Preparation***

The synthetic sandstone samples were prepared from random mixtures of sand and clay. Sand used was a washed pure quartz sand of grade 0 - 125 microns. Montmorillonite clay from Surrey, UK, of varying percentage content was used in three of the samples. Illite clay from Cambrian shale in Montana, USA, was used in one sample. Table 1 outlines the clay type and percentage of each sample.

| Sample No. | Clay Type | Percent Mass Clay Content (%) |
|---|---|---|
| Syn 1 | None | 0 |
| Syn 2 | Montmorillonite | 5 |
| Syn 3 | Montmorillonite | 10 |
| Syn 4 | Montmorillonite | 15 |
| Syn 5 | Illite | 10 |

Table 1: Description of Synthetic Samples

The clay was added to the sand to make up the required mass percentage content and thoroughly mixed to allow random distribution of the clay. Each sand/clay mixture is poured into a separate container, sealed and placed on a rolling machine for 24 hours. After rolling the sand clay mixtures were poured into Teflon heatshrink sleeving to create 38mm diameter samples of between 5 and 6cm long. Teflon end screens were used to stop the samples disintegrating.

Samples were vacuum and pressure saturated with a 5% sodium chloride, 1% potassium chloride brine. The unconsolidated nature of the samples means full saturation of all the pore space is achieved but care must be taken when handling the samples to avoid grain lose.

Sample are consolidated in a hydrostatic pressure cell. Each sample underwent three loading and unloading pressure cycles from 100 psi to 4000 psi. Pore volume and resistance change during the stress cycles were measured. The resistances measured during the second and third loading and unloading cycles were very similar for each individual sample. This indicated that the samples were consolidated and no further stress cycles were needed. The permeability to brine at 2000 psi hydrostatic stress was measured for each sample.

***Measurements on Brine-Saturated Samples***

The parameters for the $T_2$ measurements on the brine saturated samples are summarised in Table 2.

| Frequency (MHz) | 2.2 (approx) |
|---|---|
| Number of Scans | 64 |
| Tau (:s) | 320 |
| Recycle Delay (s) | 10 |
| Number of Echos | 8000 |
| P90 (:s) | 14.5 |
| P180 (:s) | 29 |

Table 2: Parameter Settings for NMR CPMG Pulse Sequence on Synthetic Samples

NMR porosity was calculated by comparing the total amplitude of the signal obtained from each sample (by summing the amplitudes in the $T_2$ distribution) to that of a known standard. In this case the reference standard was a sealed glass reference standard containing 20ml of 5% brine.

### *Desaturation by the Continuous Injection Method*
Fully brine saturated samples are loaded into Viton rubber sleeves. A brine saturated capillary contact mat is placed at the ends of the sleeved sample and a water wet ceramic disc (impermeable to oil) mounted at one end. Stainless steel flow platens and stems full of brine are mounted at each end of the sample. Samples are loaded into a multi-sample test cell.

Confining pressure is applied in increments from 100 psi to a maximum of 2000 psi. Pore volume reduction is calculated from the brine volume expelled between the pressure at which the sleeve fully conforms to the sample, typically 200 psi, and the maximum test pressure of 2000 psi. Brine is flowed through the sample to ensure full saturation. Each sample is connected at the upstream end to a dedicated slow rate syringe pump. A mixture of light paraffin oils, trade name Isopar L, is pumped into the samples at a constant rate of less than 0.02 cm$^3$/hour. Desaturation of the samples usually takes 30 days (depending on sample pore volume). Brine volume and temperature measurements are made each day and saturations calculated. The continuous injection desaturation process is analogous to capillary pressure equilibrium desaturation if the flow rate is kept low and capillary force dominates. In our experience if the flow rate is calculated so that desatuartion takes 30 days the residual brine saturation attained by the continuous injection method is the same as the capillary pressure equilibrium method. Indeed a plot of pressure of the oil phase against saturation is almost identical to the capillary pressure curve measured by the equilibrium method.

### *NMR Measurements on Partially Brine Saturated Samples*
When the samples reached residual brine saturation the confining pressure was reduced, the samples removed from the rubber sleeves and platons and the NMR $T_2$ distributions for each sample measured. The parameter settings for the NMR CPMG pulse sequence used on the desaturated synthetic samples are the same as those used on the brine saturated samples, Table 2.

**RESULTS**

The effect of clay on the flow characteristics of sandstones is illustrated by the permeability to brine at 2000 psi confining stress data in Table 3. The sample with no clay, Syn 1, has a permeability of 289 mD. The introduction of 15% clay reduces the permeability to 1 mD. Porosity is also reduced as clay content increases. Residual brine saturation increases with clay content. This is due to a combination of brine trapping due to a reduction in connected pore-space and clay bound water volume increasing as clay content increases. It is interesting to note that the 10% illite sample, Syn 5, has a similar permeability to the 10% montmorillonite but a significantly lower residual water saturation. This may indicate that the two clays form samples having different pore geometries. The porosity at 2000psi confining pressure are significantly lower than those measured by NMR at ambient confining. The synthetic samples are unconsolidated so on removal of confining pressure the pore space opens and pore volume increases.

| | Brine Permeability (mD) @ 2000psi confining pressure | Porosity (%) @ 2000psi confining pressure | NMR Porosity (%) | Residual Brine Sat. (fraction) |
|---|---|---|---|---|
| Syn 1 | 289.7 | 28.15 | 32.7 | 0.200 |
| Syn 2 | 25.6 | 26.86 | 31.7 | 0.358 |
| Syn 3 | 5.8 | 27.54 | 31.3 | 0.501 |
| Syn 4 | 1.0 | 25.69 | 29.8 | 0.642 |
| Syn 5 | 6.6 | 23.64 | 31.0 | 0.206 |

Table 3: Petrophysical Properties of the Synthetic Samples

*$T_2$ Distributions*

The $T_2$ distributions normalised to porosity for the five synthetic samples are plotted in Figures 1a-e. The clay free sand, Syn 1, has a unimodal $T_2$ distribution ranging from 70 to 1000 ms, the geometric mean $T_2$ value is 245.6 ms. The clay free sample contains a range of pore sizes with no significant microporosity. Syn 2 contains 5 % montmorillonite clay, the major peak in the $T_2$ distribution for this sample has shifted to lower relaxation times indicating that the clay has reduced the size of the pores compared to the clay free sand. A small peak is also present at 10 ms and below indicating the presence of microporosity. As percentage clay content increases the $T_2$ distributions move to shorter times showing that the clay is filling the interstitial space between sand grains and forming an increasing volume of microporosity. Chitale and Sigal (2000) measured the $T_2$ response of water saturated montmorillonites. They found that water associated with the exchangable cations between the interlaminar space of the clay particles relaxed at around 1 ms, longer response were measured for inter-aggregate pore waters. Matteson et al. (1998) showed that the T2 distribution for pure clays shifted to the millisecond range when confined at reservoir stresses.

The Sample containing 10 % illite, Syn 5, has an almost unimodal $T_2$ distribution. This is different to the bimodal $T_2$ distribution measured on the 10% montmorillonite sample. The pore space within the illite samples is obviously different to that of the montmorillonite sample.

The montmorillonite structure is composed of units made up of two silica tetrahedral sheets with a central alumina octahedral sheet. In the stacking of the silica-alumina-silica units, oxygen layers of each unit are adjacent to oxygen layers of the neighbouring units. As a result, there is a weak bond and a gap between them so that water and other polar molecules can enter between the unit layers, causing the lattice to expand. In contrast, the illite clay has a more stable structure. Illite clay has a higher charge deficiency due to ion exchanges than smectite and the seat of this charge deficiency is close to the surface of the unit layer and the balancing cation between the unit layers is potassium. Because of these differences, the illite structure is much more stable, so that the polar layers cannot enter readily between them and cause expansion. Since the interlayer balancing cations are potassium, they are not easily exchangable (Grim, 1968). The above analysis of clay structures explain the $T_2$ spectra of the synthetic shaly sample set, in particular the increasing clay-bound water responses for Syn 2 – Syn 4 due to the increasing montmorillonite content (from 5 – 15%).

The $T_2$ distributions for the desaturated samples are plotted on the same axis as the saturated distributions in Figure 1a-e, dotted line. The oil used to desaturated the samples has an NMR response, this can be seen as the peak centred around 700 ms for each sample. The $T_2$ response for the oil is in the same position of each sample. The oil does not wet the pore surfaces, it is located in the centre of the pores surrounded by brine. Because it does not interact with the pore wall the hydrogen protons in the oil relax as if they were bulk oil. The NMR response for a sample of Isopar oil is plotted with the sample $T_2$ distributions for each sample (dashed line). In every case the bulk oil overlays the samples oil response indicating water wet surface character for all the samples. The oil peak in the desaturated samples is wider than that of the oil sample. This is probably due to the exponential curve fitting software combining the oil signal with that of brine trapped in large pores.

The desaturated $T_2$ distribution for the clay free sample, Syn 1, has a peak at the relaxation time associated with bulk oil and a peak between 4 and 10 ms. The short time peak is due to capillary bound brine in wetting films and isolated dead-end pores. As clay content increases a larger response is measured from brine below 4 ms, this brine is clay-bound water either within the clay lattice or bound to the surface of clay aggregates.

The 10% illite sample desaturated down to 0.2 pore volume fraction compared to 0.5 for the 10% montmorillonite sample. The amount of brine trapped in the pore space is a function of pore size and connectivity, illite is not as efficient at reducing these properties as montmorillonite thus a lower residual brine saturation is attained. No $T_2$ response below 1ms was measured for the illite sample indicating that illite does not have interlaminar water, only water bound on the clay surfaces.

*NMR Permeability Modelling*

The permeability of each sample has been modelled using the 'Coates' and 'SDR' equations, Equations 4 and 5 respectively. The exponents in each equation have been adjusted to obtain the closest match of model and core permeability for the five samples as a whole set. The optimised Coates equation obtained was:

$$KmD = 0.008 \cdot \left( \frac{FFI}{BVI} \right)^{2.5} \cdot \left( \frac{f}{10} \right)^{5.8} \qquad (4)$$

The optimised 'SDR' equation obtained was:

$$k(mD) = 0.0015 \cdot \left[ (T_{2LM})^{1.4} \cdot \left( \frac{f}{10} \right)^{4} \right] \qquad (5)$$

The modelled permeability is plotted against sample permeability in Figures 2a and b. Equations 6 and 7 model the permeability of the clay free sample and those containing montmorillonite well. The illite sample is not so successfully modelled indicating that it belongs to a different facies type and could only be modelled using a different set of exponents for the Coates and SDR equations.

*NMR Response Types*

The Applied Reservoir Technology Sandstone NMR Rock Catalogue has been used to match the parameters measured for the synthetic samples with the NMR $T_2$ distributions and petrophysical parameter for reservoir sandstones. Table 4 shows the petrophysical data for those sample selected as analogues to the five synthetic samples.

| Sandstone No. | Ka (mD) | Porosity (%) | Swi (fraction) | Clay Content | Synthetic Sample Match |
|---|---|---|---|---|---|
| Sand 1 | 1700 | 25 | 0.10 | No Clay | Syn 1 |
| Sand 2 | 67.2 | 21 | 0.50 | 6% Illite, 1% Smectite | Syn 2 |
| Sand 3 | 1 | 15 | 0.63 | 6% Kaolinite, 1% Illite 1% Smectite. | Syn 3 |
| Sand 4 | 0.22 | 16 | 0.87 | 9% Illite, 3% Smectite, 1% Chlorite. | Syn 4 |

Table 4: Petrophysical Properties for Reservoir Sandstone Samples

The criteria for selection of a reservoir sandstones analogues were a match of $T_2$ distribution shape and position on the $T_2$ axis and clay content to the respective synthetic sample. Unfortunately no match could be found for Syn 5, the 10% Illite sample.

The ART sandstone rock catalogue contains backscattered electron images of resin impregnated samples. Figure 3 shows the images for the selected sandstone samples. The images show the resin filled pore-space in black, quartz grains in light grey and clays in dark grey. Sand 1 has an open structure with many pores greater than 50 microns diameter. Sand grains within Sand 2 are more tightly packed than Sand 1 and clay particles bridge the pore-space. Sand 3 is well compacted with clay lining and filling pores. Pore-space within Sand 4 is filled with clay and fine particles causing a low permeability and a high residual brine saturation.

The transition from isolated clean sand through to clay particles within the pore space and finally pore blocking clay clusters can be detected within the $T_2$ distributions for the reservoir sandstones, Figure 4. The reservoir sandstones show a similar trend to the synthetic samples. As clay content increases pore sizes are reduced and microporosity is formed. This leads to a shifting of the major $T_2$ peak to shorter times and an increase in the $T_2$ response below 10ms.

**CONCLUSIONS**
• Synthetic sandstone samples have been used to evaluate the effect of clay on NMR measurements. This type of systematic analysis helps to relate NMR response to pore space geometries and aids in the interpretation of NMR core and log data.
• Clay type and content significantly affects the petrophysical properties and NMR relaxation responses of sandstones.
• $T_2$ NMR measurements can detect the presence of clay microprosity and distinguish between free and bound water.
• Both the tuned Coates and SDR equations successfully modelled the permeability of the clay free and montmorillonite containing samples. A different set of model parameters was needed to model the permeability of the sample containing illite clay.

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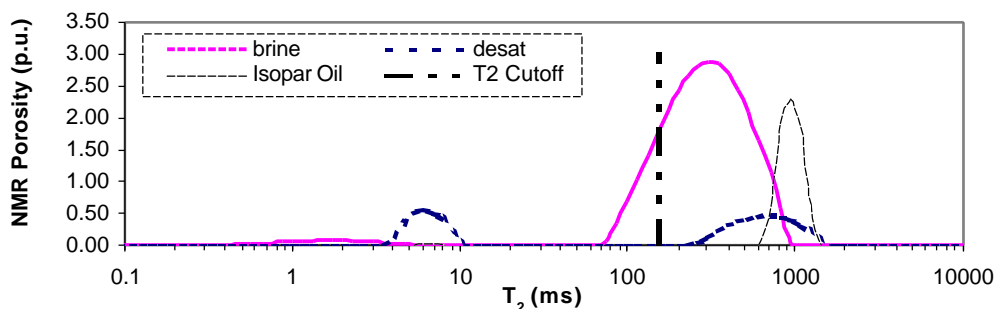
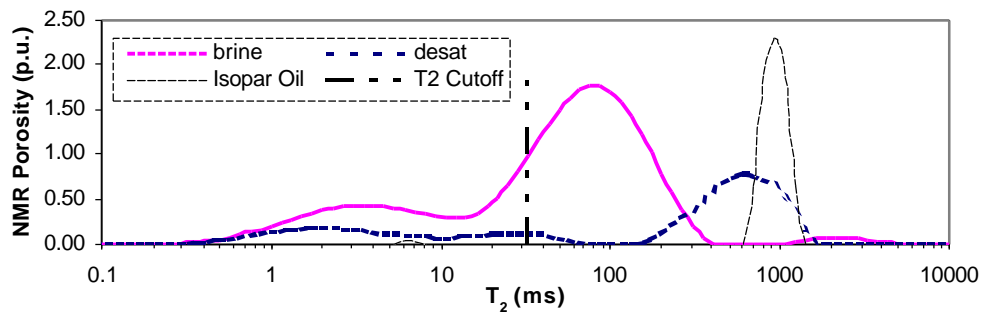Figure 1a: T2 Distribution for Syn 1, Clay free Sand.

Figure 1b: T2 Distribution for Syn 2, 5% Montmorillonite.
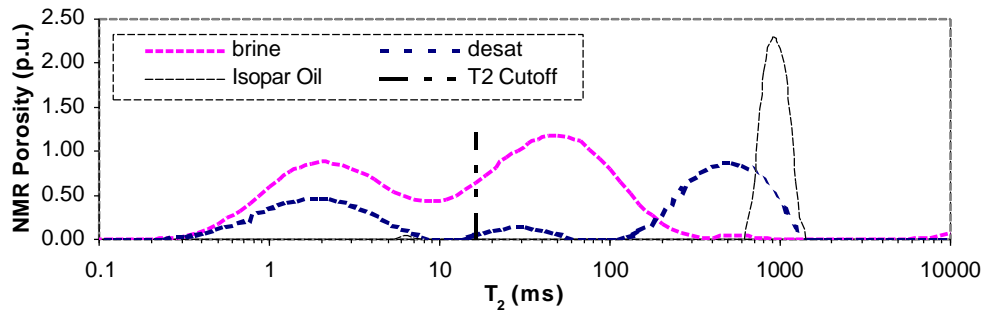
Figure 1c: T2 Distribution for Syn 3, 10% Montmorillonite.
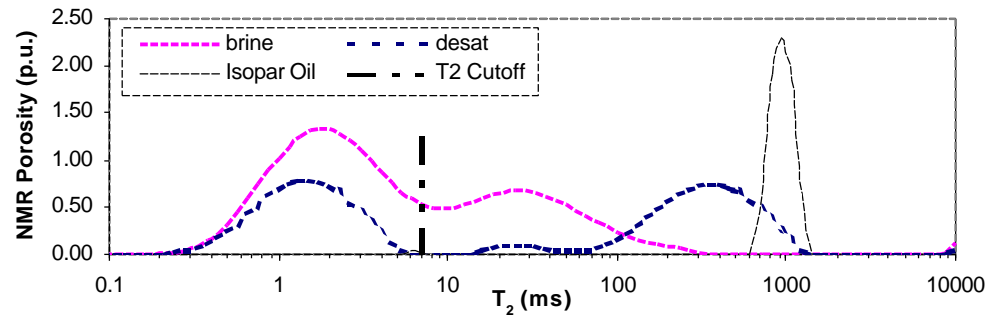
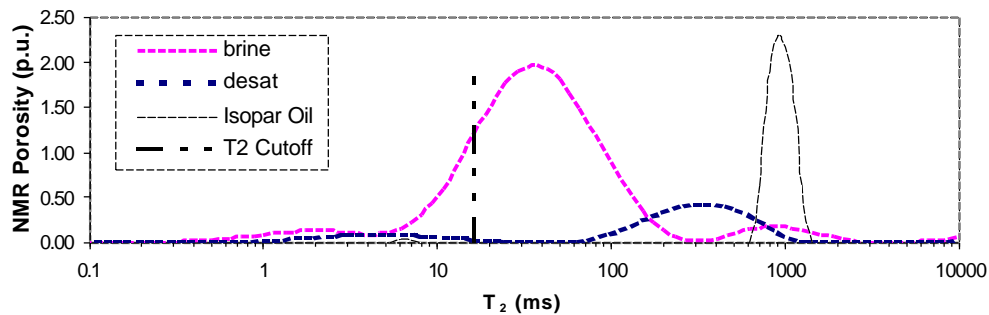Figure 1d: T2 Distribution for Syn 4, 15% Montmorillonite.

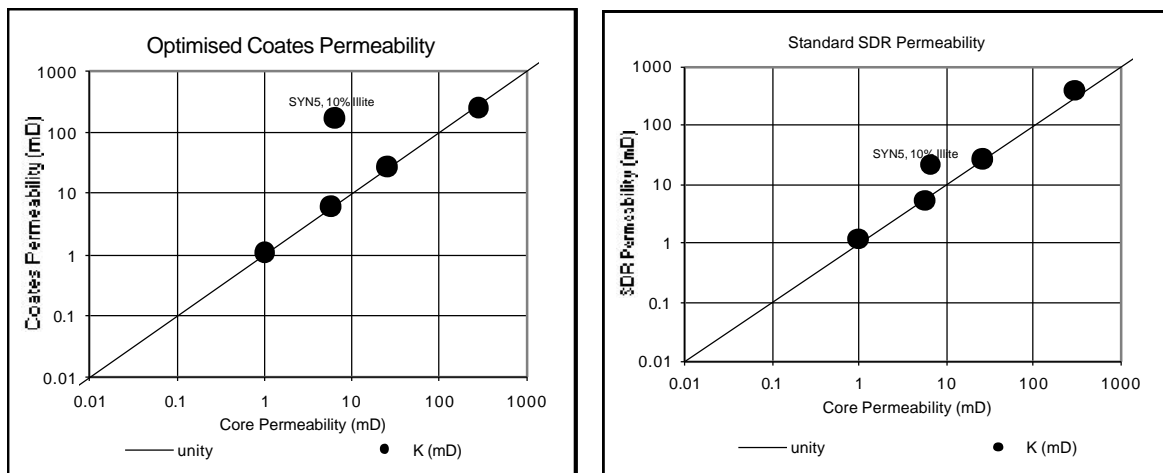Figure 1e: T2 Distribution for Syn 5, 10% Illite.

Figure 2: Modelled NMR Permeability Against Core Permeability.



**100 mm**  **100 mm**  **100 mm**  **100 mm**
**Sandstone 1**  **Sandstone 2**  **Sandstone 3**  **Sandstone 4**
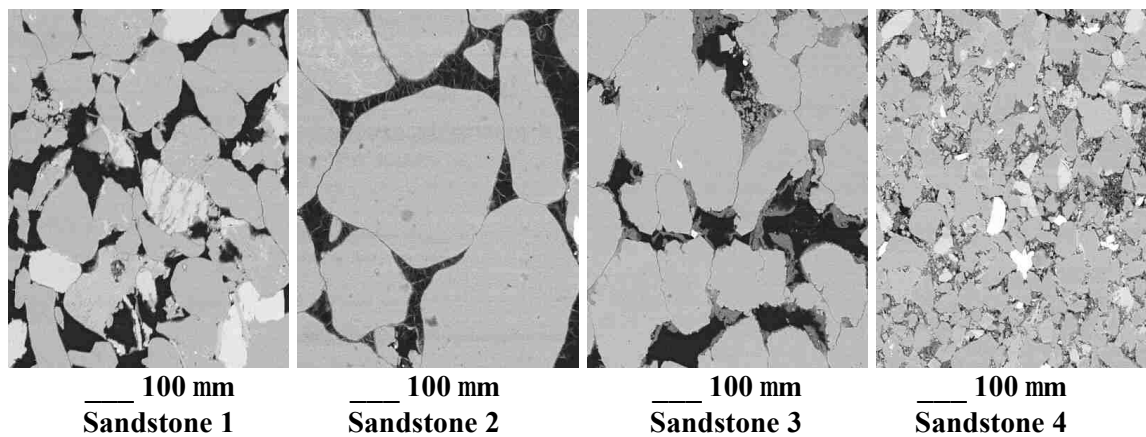
Figure 3: BSEI Images of Reservoir Sandstones from the ART NMR Rock Catalogue
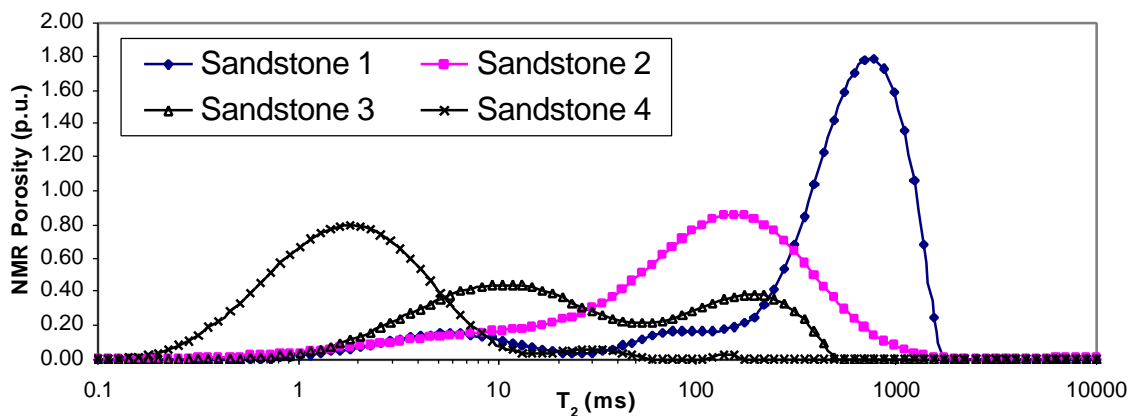


Figure 4: T2 Distributions for Reservoir Sandstones from the ART NMR Rock Catalogue