SCA2003-66: Shale Volume Estimates from NMR Core Data

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ABSTRACT

Within reservoir evaluation there is a need to quantify shale volumes. This paper describes methodologies used to formulate a model to estimate shale volumes from NMR core plug data. The NMR signal from outcrop core plugs with a range of clay content and volume, at varying saturation states was measured. The study utilises the differing water adsorption properties of core plugs of varying clay content to calibrate a model that can directly predict shale volumes.

INTRODUCTION

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. Previous studies have described the qualitative effects of clay content and mineralogy on NMR data (Moss and Jing, 2001). The authors are unaware of any use of NMR data to quantify clay volumes. We analysed the NMR measurements of six outcrop core plugs exposed to an atmosphere of 100% relative humidity for a number of weeks. These data were used to formulate an empirical model relating NMR signal to percentage clay content.

NMR Theory

Detailed descriptions of NMR theory and its application to porous rock systems can be found elsewhere (Camden, 1997). For this study a CPMG sequence (Carr-Purcell-Meiboom-Gill) is used to measure NMR T_2 distributions of core plug samples in various saturation states.

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 a continuous range of relaxation times. Each pore-size has a distinctive T_2 value. The echo-

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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 resulting echo-train therefore consists of a continuous distribution of T_2 values each with different signal amplitudes. It is mathematically extremely difficult to deconvolute the data by fitting to a continuous distribution of exponential decays. Thus an approximation is used based on a selected number of T_2 values (say 50) over a specified time range (usually evenly spaced in logarithmic time), used to fit the echo-train, calculating the signal amplitude associated with each one. The result is plotted as a T_2 distribution.

The oscillating 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 either a small pore or wetting film, the nuclei are more likely to interact with the surface, and so the average relaxation will be faster and the time for relaxation shorter than in a large pore.

PROCEDURE

Sample Description

Six outcrop plug samples were used in this study, which covered a range of clay-carbonate-quartz compositions. The mineralogy of each sample was determined using X-ray diffraction (Phillips PW1830 system of CuKa radiation) for both the whole rock and the fines fraction. Helium porosity and grain densities of clean oven dried samples were also measured (de Freitas and Zacharopoulos, 2002). Tables 1 and 2 outline the clay content, distribution and petrophysical properties of each sample.

Sample No.	Quartz (%)	Albite (%)	Calcite (%)	Dolomite (%)	Clay Minerals (%)	Porosity (%)	Grain Density (g/cc)	Max. Water Adsorption (% Pore Vol.)
IST	16	-	49	11	24	39.7	2.71	25.3
TRK	8	-	81	-	10	35.9	2.68	21.1
PRKS	5	-	11	79	6	43	2.81	15.1
IS	21	-	77	-	2	29	2.70	6.6
KLNK	36	5	37	-	22	36.5	2.66	29.3
PROR	45	6	5	-	44	30.9	2.61	100.0

Table 1: XRD Whole Rock and Petrophysical Analysis of Outcrop Samples

Sample No.	Smectite (%)	Illite (%)	Kaolinite (%)	Chlorite (%)	Clay Distribution
IST	9	1	1	13	Clayey calcitic microgranular matrix
TRK	6	4	Trace	-	Scattered calcitic-clayey assemblages
PRKS	3	2	Trace	1	Homogenous trace clay & pockets of interwoven clay flakes
IS	1	1	-	-	Homogenous silty sand
KLNK	11	10	-	1	Silt particles within a clay calcitic matrix
PROR	22	15	5	1	Silt particles within a clay matrix

Table 2: XRD Fines Fraction Analysis and Clay Distribution of Outcrop Samples

NMR Measurements on Samples Containing Adsorbed Water

Samples were prepared for NMR T_2 measurement by exposing each plug to an atmosphere of 100 % relative humidity. Each sample was placed in a separate airtight container, balanced on a thin metal ring so that its cylindrical axis was horizontal. This orientation allows 99.9% of the surface area to be exposed to the atmosphere within the container. The 100% relative humidity in the container was maintained by the addition of a small dish containing distilled water. At no time was the sample allowed to come into direct contact with the water. All containers were maintained at a temperature of 36 °C (the working temperature of the NMR Spectrometer). Water adsorption was monitored by weighing (accurate to three decimal places) the sample every few days. NMR T_2 measurements were taken when no further mass increase was detected. NMR measurements were performed on ResLab-ART's Maran 2 MHz spectrometer.

NMR Measurements on Brine-Saturated Samples

After the NMR T_2 measurements had been made, the samples were dried in a conventional oven at 90 °C and saturated with a complex simulated formation brine of approximately 45,000 ppm. Oven drying may cause clay particle damage but this was essential to allow accurate pore volume measurement. T_2 measurements on the brine saturated samples were measured in order to compare the pore geometry of the sample group. The T_2 distribution can be thought of as a 'pseudo' pore size dsitribution. Short relaxation times equate to small pores and long times larger pores. The pulse sequence settings for NMR measurements on all samples are detailed in Table 3.

Sample State	Echo-Spacing (ms)	No. Echoes	Relaxation Delay (S)
Max. water absorption	200	2,000	3-10
Brine saturated	200	8-16,000	10

Table 3: Summary of CPMG Pulse Sequence Settings

RESULTS

Brine Saturated T_2 Distributions

Previous workers have shown that saturated samples containing high clay content have relaxation times around 1 ms or below, (Moss and Jing, 2001). Figure 1 shows the brine saturated T_2 distributions for each outcrop sample scaled to pore volume. The IS sample has the lowest total clay content (2%) and the peak within the T_2 distribution is centred on long relaxation times of about 30 ms. Visual inspection of the remaining sample T_2 distributions does not reveal which samples have a high clay content (Table 1). The T_2 distributions for samples PROP and IST are both centred on 1.68 ms whilst total clay contents are 44 and 24% respectively. Whilst the brine saturated T_2 distributions supply valuable qualitative information about pore geometry they do not allow a means of quantifying clay content.

Water Adsorbed T_2 Distributions

Figure 2 shows the T_2 distributions for each outcrop sample at its maximum water adsorption saturation. Each distribution is scaled to pore volume and water saturation. The area under each distribution is proportional to the volume of water adsorbed. Thus PROP has the largest peak and highest water adsorption (100% pore vol.). Note that the area under both T_2 distributions for PROP are almost the same, confirming that all the pore space is filled with water after water adsorption. All samples have relaxation times significantly lower than those in the respective saturated T_2 distribution. This indicates that the NMR is measuring water in thin surface films as well as micropores.

It was found that the total NMR signal for the water adsorbed samples (area under the distribution) correlated well ($R^2 = 0.89$) with the percentage of clay in each sample, Figure 3. This relationship (Clay Content = (Total NMR Signal - 0.145)/0.618) may be independent of clay type and could be applied to a wide range of rock types. Smectite is the dominant clay type within these samples. Percentage of smectite against total NMR signal has an excellent correlation ($R^2 = 0.94$), Figure 3.

CONCLUSIONS

- Brine saturated sample T_2 distributions can be used to evaluate differences in pore geometry between core plug samples.
- Water adsorption as vapour at 100% relative humidity and 36^{o}C appears to be directly proportional to percentage clay content.
- For the small number of samples in this test an empirical relationship correlating the total clay content with total NMR signal has been derived. An improved relationship can be obtained by correlating the most abundant clay, smectite, against total NMR signal.
- Core plug samples are often dried in a humidity oven as part of special core analysis programs. Such samples could have T_2 distributions measured. The application of our relationship to this data would provide a non destructive estimate of clay volume within the effective porosity.

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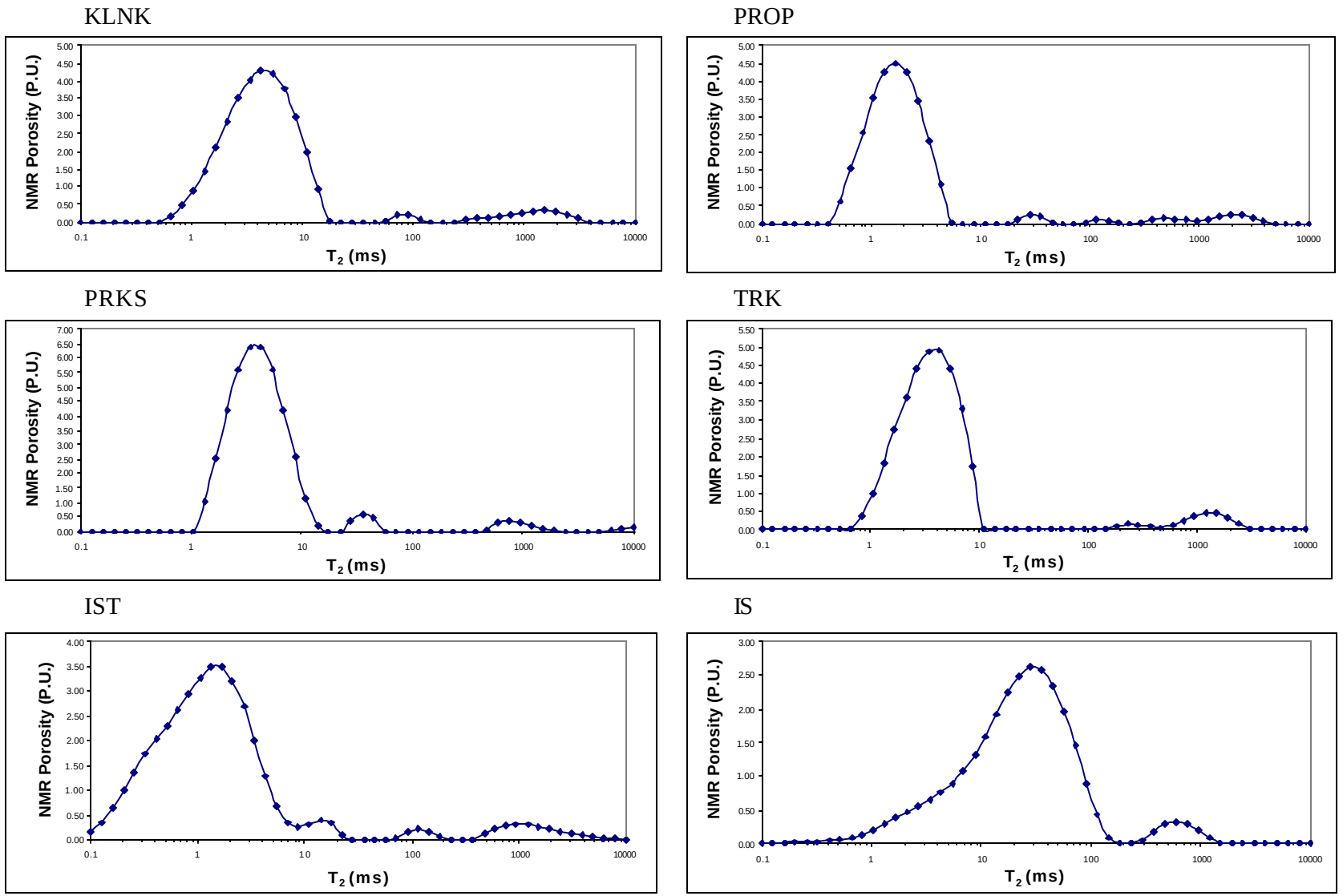

Figure 1: NMR T_2 Distributions for Brine Saturated Outcrop Samples

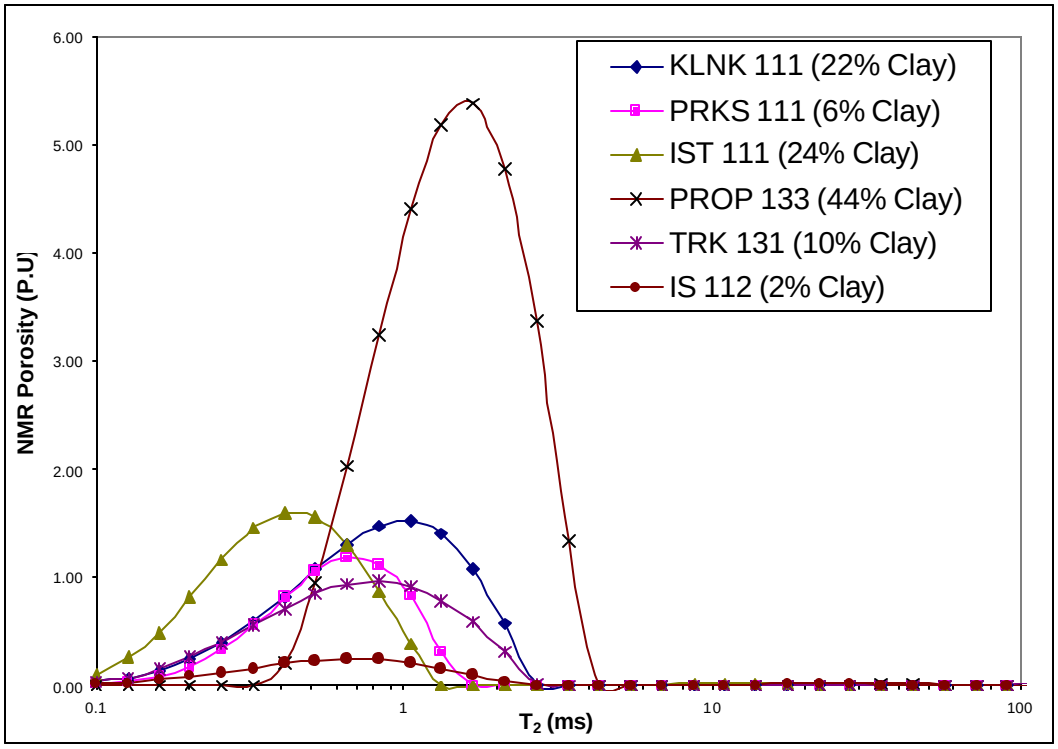

Figure 2: NMR T_2 Distributions for Water Adsorbed Outcrop Samples

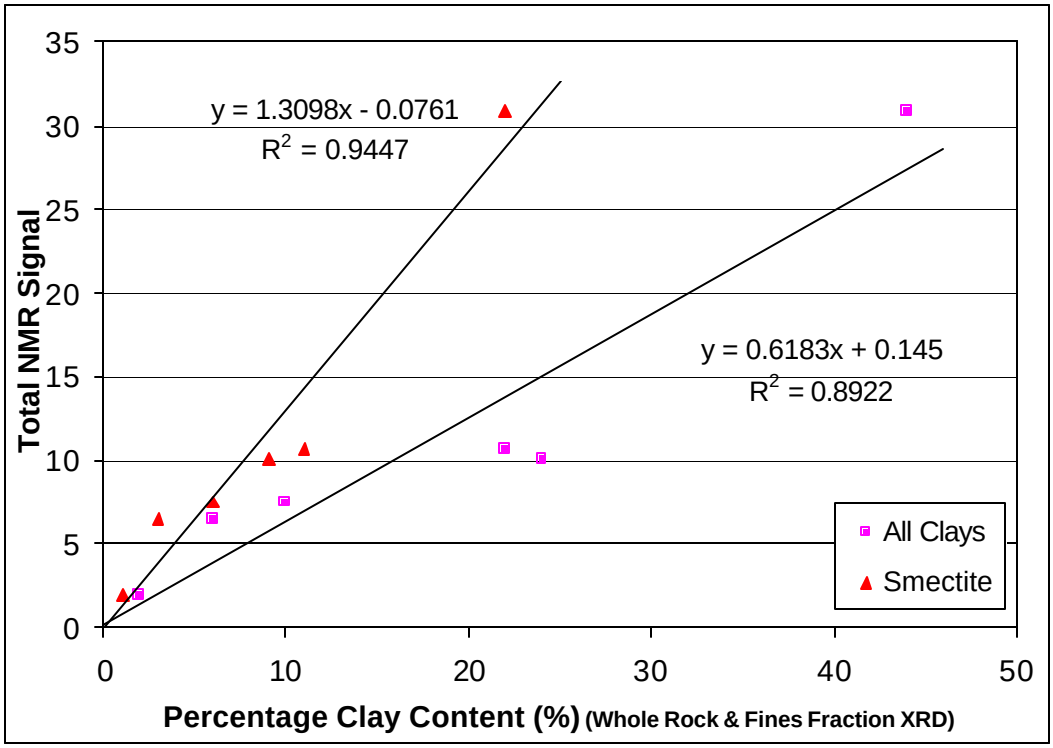

Figure 3: Total NMR Signal Against Percentage Clay Content