

A 12 MHZ ROCK CORE ANALYZER FOR IMPROVED CHARACTERISATION OF 1.5" ROCK CORE PLUGS

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ABSTRACT

A new 12 MHz bench top rock core analyzer has been developed aiming at improving the characterisation of 1.5" rock core plugs at laboratory conditions.

INTRODUCTION

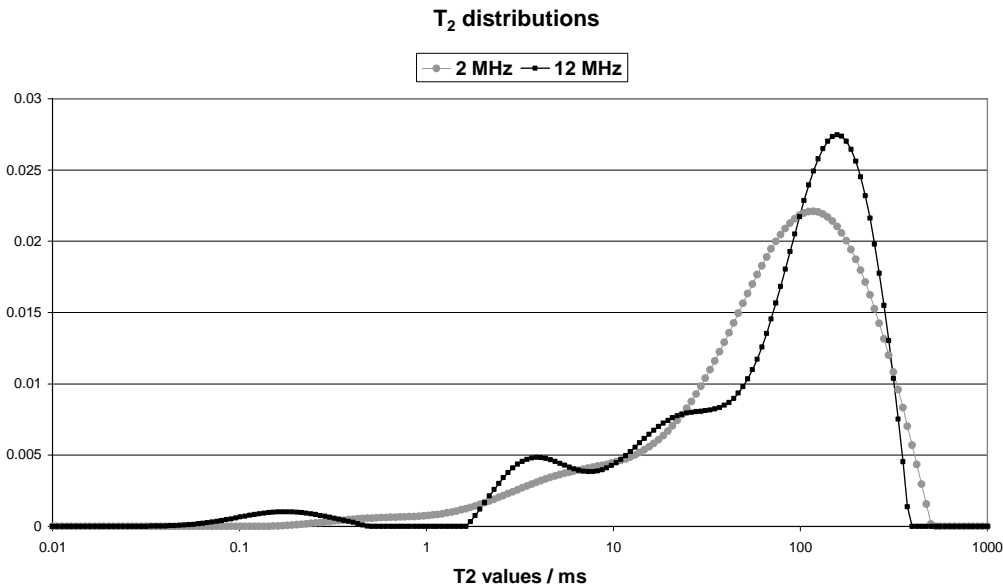
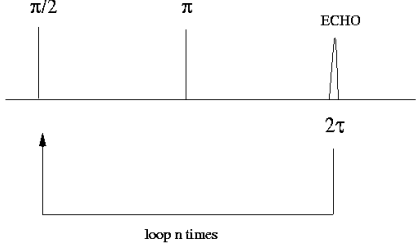
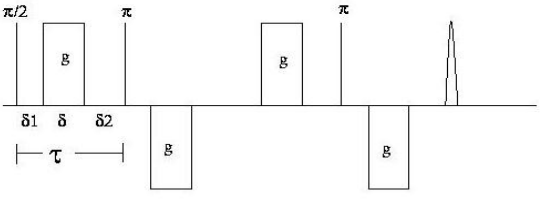
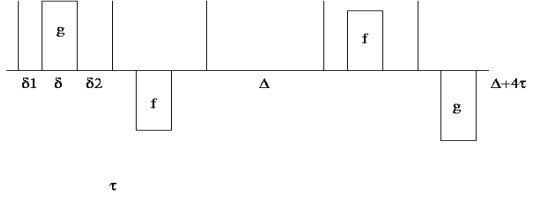
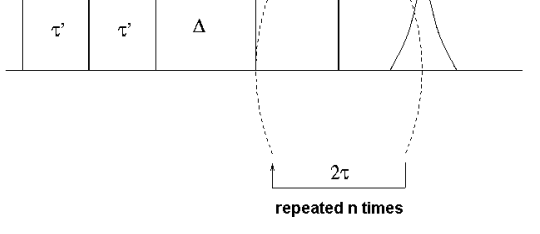
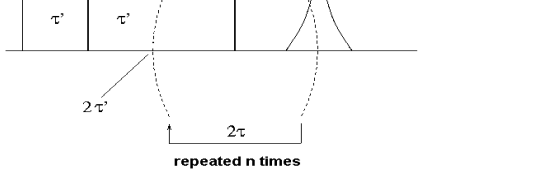


Figure 1: T₂ distributions from a rock core plug measured at 2 MHz and 12 MHz

When working with NMR SCAL methods at laboratory conditions, it is evident that there are limitations in sensitivity when measuring at an operating field of 2 MHz. Thus we have cooperated with Oxford Instruments in developing a 12 MHz rock core analyzer that has its signal to noise significantly improved. Figure 1 indicates the improvement achieved when going from a 2 MHz to a 12 MHz rock core analyzer: While the T₂ distribution from the 2 MHz returns a major peak with a left shoulder, the 12 MHz data returns a narrower main peak and indicating 3 other distinct regions where the one with shortest T₂ is completely resolved from the other regions. In a T₁-T₂ map all these regions will be resolved with the 12 MHz data. In addition to improved signal to noise ratio, the rock core analyzer comprises a pulsed field gradient option where the maximum strength of the

Table 1: A summary of the NMR pulse sequence applied on the rock core plugs.

NMR sequences	Corresponding attenuations
<p style="text-align: center;">CPMG</p>  <p style="text-align: center;">loop n times</p>	<p>The ordinary CPMG</p> $I = I_0 e^{-n \cdot \left[\frac{2\tau}{T_2} - \frac{2\tau^3}{3} \gamma^2 G_i^2 D \right]}$ <p><i>yields a T_2 distribution</i></p>
<p style="text-align: center;">PFGSE</p> 	<p>The Pulsed Field Gradient Spin Echo</p> $I = e^{-\left(\frac{4\tau}{T_{2ik}} + \gamma^2 \delta^2 (2g)^2 D_{ik} \left(\frac{3}{2}\tau - \delta \frac{1}{6} \right) \right)}$ <p><i>yields the diffusion values of the fluid components</i></p>
	<p>The Pulsed Field Gradient Stimulated Echo</p> $I = I_0 e^{-\frac{4\tau}{T_2} - \frac{\Delta}{T_1} - \gamma^2 g^2 D (2\delta)^2 (\Delta + 3/2\tau - \delta/6)}$ <p><i>yields the surface to volume ratio at short observation times</i></p>
 <p style="text-align: center;">repeated n times</p>	<p>A combined Stimulated Echo CPMG</p> $I = I_0 e^{-n \cdot \left[\frac{2\tau}{T_2} + \frac{2\tau^3}{3} \gamma^2 G_i^2 D \right]} e^{-\left[\frac{2\tau'}{T_2} + \gamma^2 G_i^2 D \frac{2\tau'^3}{3} \right] + \frac{\Delta}{T_1}}$ <p><i>correlates the transverse relaxation time to the longitudinal relaxation time</i></p>
 <p style="text-align: center;">repeated n times</p>	<p>A Spin Echo CPMG</p> $I = I_0 e^{-n \cdot \left[\frac{2\tau}{T_2} + \frac{2\tau^3}{3} \gamma^2 G_i^2 D \right]} e^{-\left[\frac{2\tau'}{T_2} + \gamma^2 G_i^2 D \frac{2\tau'^3}{3} \right]}$ <p><i>probes the pore sizes (the degree of sorting) of the porous rock</i></p>

magnetic field gradient is approximately 225 Gauss/cm. To avoid instrumental artefacts the coils generating the magnetic field gradient are mounted on the magnetic poles. The use of active shielding to reduce the eddy current dead time reduces the maximum gradient strength obtainable (Turner(1986)). Instead of active shielding we apply preparatory gradient pulses to saturate the eddy currents induced. We also apply sinusoidal shaped gradient pulses as the pulsed gradient option has shown to induce instability effects from the gradient amplifier. With these settings we have achieved eddy current dead times of $\sim 300 \mu\text{s}$ at full power on the gradient amplifier, which is competitive to the more expensive actively shielded gradient option with less gradient strength available. In the following sections we will show some results indicating the capabilities of the 12 MHz rock core analyzer. The NMR sequences used are summarized in Table 1.

T_1 - T_2 MAPPING

As shown in Figure 1, even the ordinary CPMG experiment returns a T_2 distribution where several regions are more resolved at 12 MHz than at 2 MHz. In order to improve this resolution one may combine a stimulated echo sequence with a train of spin echoes (CPMG).

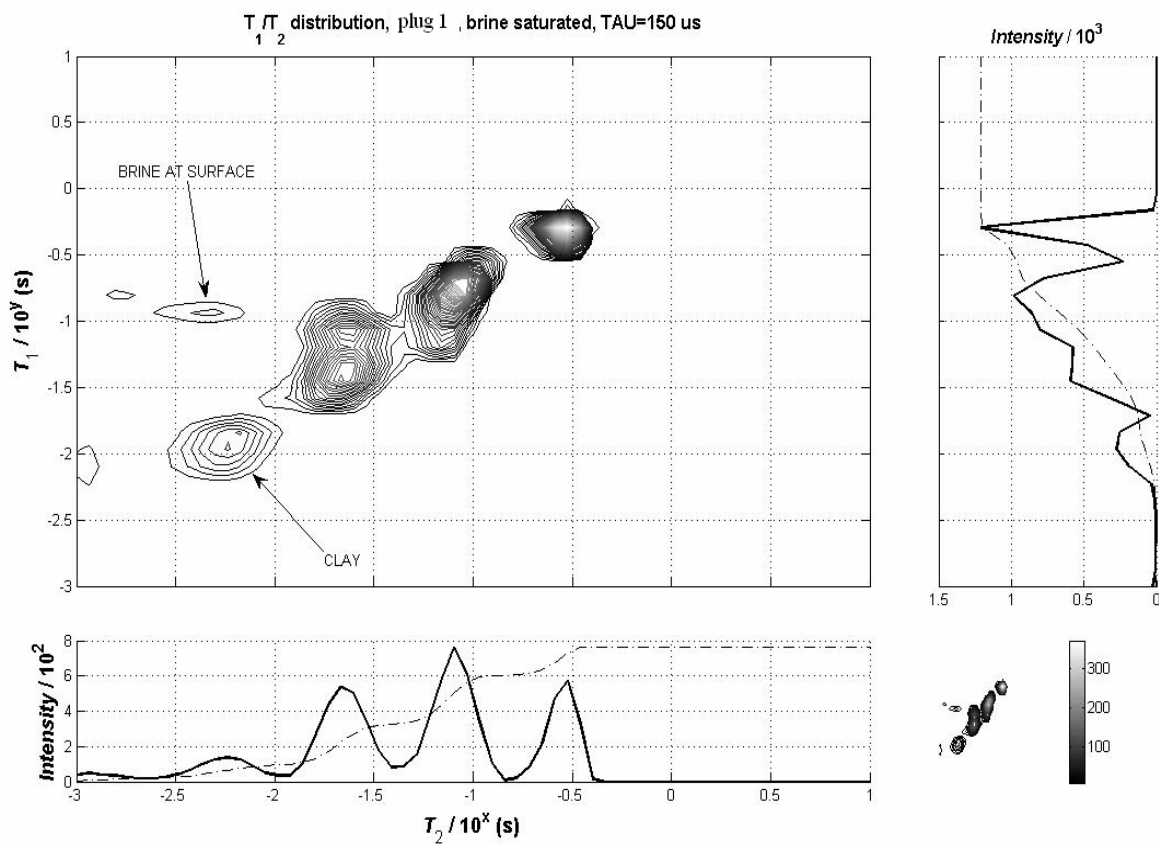


Figure 2: T_1 - T_2 map of a brine saturated rock core plug containing significant amounts of clay. The scale bars yields the intensity level for the contour lines in the T_1 - T_2 map.

In Figure 2 the T_1 - T_2 map of a brine saturated rock core plug is shown. From SEM microscopy pictures we know that this rock contains significant amounts of clay, and both the low T_1/T_2 ratio and diffusion experiments confirm that we have two brine components. One component has a diffusion coefficient close to the bulk value $2.9 \cdot 10^{-5} \text{ cm}^2/\text{s}$ while another exhibits a diffusion coefficient approximately 5 times slower than the bulk value. Thus we find that the clay bound brine has a diffusion coefficient of the order of $10^{-6} \text{ cm}^2/\text{s}$, which is in accordance with that found by Nakashima (2001). When recording T_1 - T_2 maps at different inter echo spacing we also notice that the T_1/T_2 ratio increases more for the brine located outside the clay than the clay bound brine. The reason for this behaviour is the presence of strong and rapidly fluctuating internal magnetic field gradients within the clay. During the inter echo delay the dephasing due to this gradient is averaged out as the clay bound brine is probing internal gradients of both polarities, thus leading to a T_2 value that does not change much as a function of the inter echo spacing. For the brine outside the clay the situation is different as the brine is diffusing in the presence of approximately constant internal gradients during the inter echo delay. Thus the T_2^* value is reduced as the τ value increases.

THE PULSED FIELD GRADIENT OPTION

Diffusion Measurements

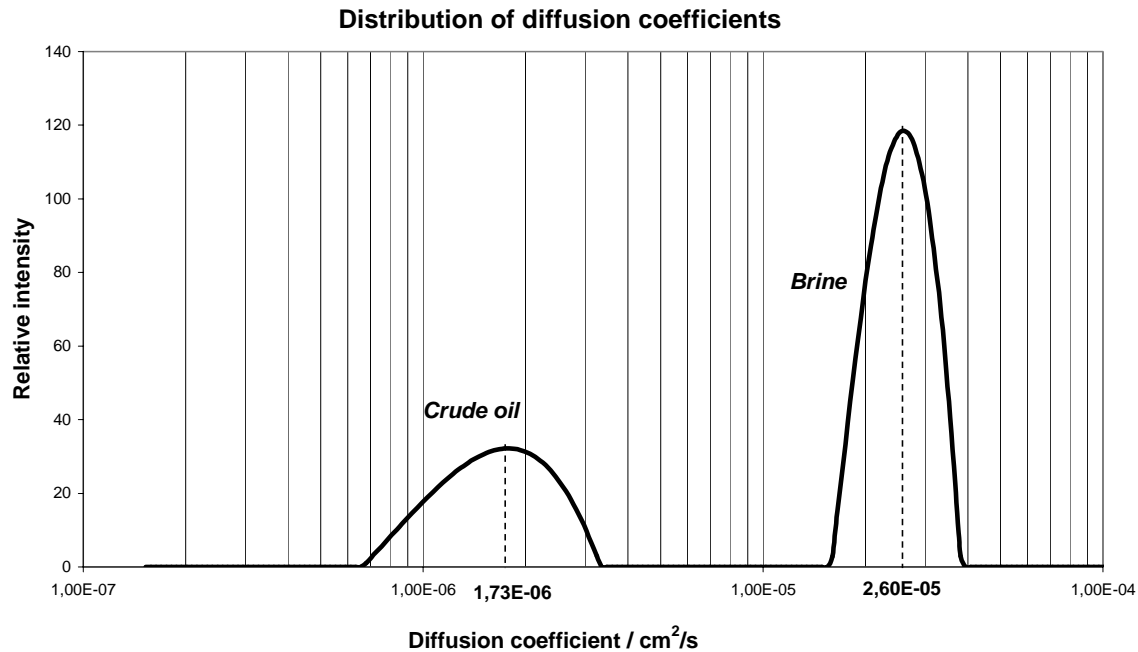


Figure 3: Inverse Laplace Transform (ILT) of data from a PFG diffusion experiment

When characterising rock core plugs, either in fresh state or cleaned state, and saturated with different fluids, the pulsed field gradient option may be used to resolve the contribution from the fluid components (Sørland et al (2004)). Figure 2 shows the result from a diffusion experiment performed on a rock core plug at fresh state, where the

motivation was to, if possible, identify crude oil. The NMR sequence applied was the 11 interval PFGSE sequence (Sørland et al (1999)). From the ILT analysis of the diffusion data (Figure 3) we do find two fluid components with distinct differences in molecular mobility, more than a factor of 10. While the component with the highest mobility coincides with the diffusion coefficient found for brine confined in a porous rock, the mobility of the slowest component is as expected from a crude oil. As the diffusion coefficient also is a measure for the size of the molecules investigated, the broader distribution of the crude oil like fraction indicates variations in the molecular size of the hydrocarbons. We therefore conclude that the rock core plug contains significant amounts of a fluid component which can be characterised as a crude oil component.

Pore Size Distributions

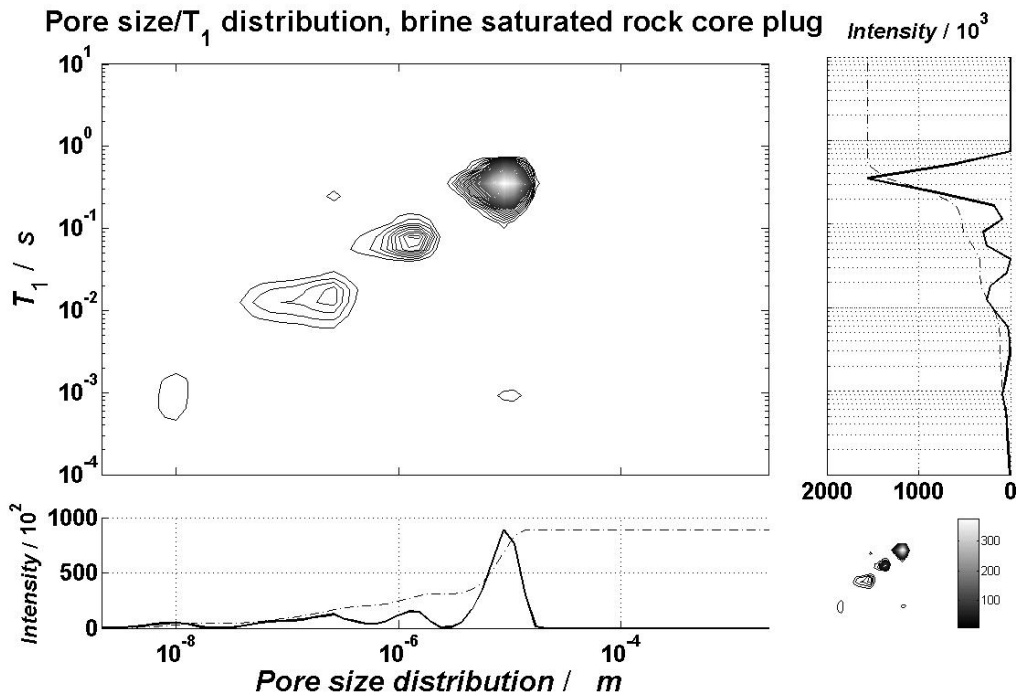


Figure 4: Combined pore size and T_1 distribution of a brine saturated rock core plug. The scale bars yields the intensity level for the contour lines in the T_1 - T_2 map.

One of the strong features in the 12 MHz rock core analyzer is the possibility of measuring diffusion coefficients at short observation times (~ 1 ms). Then we can apply the short observation time expansion of the measured diffusion coefficient by Mitra et al (1993)

$$\frac{D(t)}{D_0} = 1 - \frac{4}{9\sqrt{\pi}} \sqrt{D_0 t} \frac{S}{V} \quad (1)$$

where $D(t)$ is the time dependent diffusion coefficient, D_0 is the bulk value of the

diffusion coefficient and S/V is the surface to volume ratio. Combining the S/V found from such a set of measured $D(t)$ at short t values with a combined $T_1 - T_2$ experiment may return a combined pore size/ T_1 distribution as shown in Figure 4. The basic assumption leading to the combined pore size / T_1 distribution is the assumption of a surface relaxivity that is not dependent on the pore sizes within the porous rock. Then we may apply the measured S/V from the diffusion experiment in the T_2 relaxation time experiment and thus a measure for the surface relaxivity.

$$\rho \approx \frac{\overline{1}}{T_2} \times \left[\frac{\overline{S}}{\overline{V}} \right]^{-1} \quad (2)$$

where we may apply the average values as we assume the surface relaxivity to be independent of pore size.

CONCLUSION

The 12 MHz rock core analyzer is a competitive option to the 2 MHz analyzer when one is aiming at optimised characterisation of rock cores. Signal to noise improves the resolution and reduces the experimental time significantly. Thus two dimensional experiments as T_1 - T_2 map may be conducted within reasonable amount of time. The pulsed field gradient options makes it possible to record absolute pore size distributions and returns details in the distributions (Figure 4) that is not obtainable from Mercury intrusion techniques.

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