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## NMR $T_1$ and $T_2$ Relaxation in Oil-Based Mud Filtrates at Reservoir Temperatures

Nitesh Kumar, James Jacob\* and Lorne A. Davis, members SPE, Magnetic Resonance Imaging Petrophysical Applications Center, Department of Petroleum Engineering, Texas Tech University (\* now at Temco, Inc.)

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#### Abstract

Knowledge of the nuclear magnetic resonance (NMR) properties of the near-wellbore fluids at reservoir temperature is needed for planning and interpretation of an NMR logging program. If the oil-based mud filtrate (OBMF) is the non-wetting pore phase, it will exhibit long, bulk fluid-like relaxations that can strongly affect the NMR response of the flushed zone. Knowledge of the NMR T<sub>1</sub> and T<sub>2</sub> relaxations at reservoir temperature is needed, since these parameters have significant temperature dependences. In this work, the bulk-fluid NMR T<sub>1</sub> and T<sub>2</sub> responses of six oil-based mud filtrates and three drilling fluid base oil samples were measured over a range of elevated temperatures. The present work also has application to NMR lab measurements on core taken with low invasion oil-based coring fluids.

All samples were degassed using a standard helium sparging technique, since dissolved paramagnetic oxygen can greatly reduce the NMR relaxations of refined oil products. The experimental temperature range was approximately 30-100 C (546-670 °R). The temperature dependence of the bulk viscosity of each sample was measured using a rolling ball viscometer. The NMR relaxation data was collected on a 2 MHz permanent magnet spectrometer.

There were no important NMR relaxation differences between base oils and their derived mud filtrates. Sample viscosity had an approximately linear inverse relationship with temperature. The sample relaxations varied approximately as the inverse cube power of viscosity, steeper than has been observed in dead crude oils. For all samples,  $T_1$  and  $T_2$  increased approximately linearly with temperature, tripling from 30 to 100 C (546-670 °R).

#### Introduction

The modern nuclear magnetic resonance (NMR) log can measure different sets of petrophysical and pore fluid properties depending upon how the basic NMR physical parameters are collected and contrasted with one another.<sup>1, 2</sup> Fluid typing, the differentiation between water, oil and gas, relies upon the contrast in the observed T<sub>2</sub> distribution generated by changes implemented in the tool wait time and the inter-echo spacing. These tool parameter changes generate contrast in  $T_1$  with  $T_2$  and in diffusivity with  $T_2$ , respectively. These contrasts, in turn, identify fluids by these differences in their NMR relaxation rates and in diffusivity. There is currently a stream of development in oil/gas discrimination using the NMR log.<sup>3, 4, 5, 6, 7</sup> Other fluid typing research and development has been toward the determination of oil viscosity.<sup>8, 9</sup> Relaxation data on the pore fluids is required for pre-planning and interpretation. Sophisticated timedomain/relaxation-domain data analysis algorithms have been constructed that combine standard inversions with forward modeling.<sup>10</sup> Relaxation data is a prerequisite.

Even the relatively simple NMR measurement of porosity requires that the wait time between consecutive pulse sequences be three to five times the longest  $T_1$  value for any of the fluids in the pore space. The longest wait time will thus set the logging speed. In the water-wet invaded zone of a well bore or of a core that has been drilled/cored with oil-based muds, the OBMF will be the fluid with the longest  $T_1$  value, probably significantly so. Marschall, Gardner and Curby have discussed the importance of supporting lab measurements toward quality formation outcomes and recommend that the OBMF be analyzed in the lab for the NMR parameters specific to the job.<sup>11</sup> Kleinberg and Vinegar have assembled a valuable compendium of NMR physical data for reservoir fluids, but OBMF and base oil data were not included.<sup>12</sup> The present study is an effort to develop such NMR data for the open literature.

It is standard coring practice to use bland muds with low concentrations of surfactants in order to minimize wettability alteration. In some cases, invert oil emulsion drilling muds, with their high surfactant emulsifier concentrations, may alter wettability and shift the  $T_1$  and  $T_2$  relaxations to lower values than those reported for the invaded OBMF bulk liquid.<sup>13</sup> It

may sometimes be necessary to model such systems in core material using OBMF with reconstituted surfactant agents.<sup>13</sup>

#### **Experimental Procedures**

Three base oils for oil-based drilling fluids and six oil-based mud filtrates were contributed by Schlumberger. **Table 1** relates the physical characteristics of the samples as measured during this study.

**NMR T<sub>1</sub> and T<sub>2</sub> Relaxation Measurements.** The NMR relaxation data were measured using a 2 MHz Resonance Instruments Ultra tabletop spectrometer with gradient capability and a coil bore diameter of 1.50 inch. Experimental pulse sequence and hardware parameters for the spectrometer are provided in **Table 2**. The CPMG T<sub>2</sub> pulse sequence used an 800  $\mu$ sec inter-pulse time spacing. From published predictions of the effect of diffusion on T<sub>2</sub> relaxation in oils, there is a negligible effect of diffusion on T<sub>2</sub> measurement for this pulse spacing in a uniform field for a bulk sample.<sup>12</sup>

The samples were pumped into a Peek plastic liquid chromatography cell of 1.0 cm diameter and 2.2 ml volume. The cell was rated at 4000 psig and 100 C. A computer-controlled positive displacement pump, Quizix QL-700-5K, ran in constant pressure mode to maintain a cell pressure of 300 psig.

The sample cell was surrounded by a counter-wound, double-helical heat exchanger immersed in a cylindrical bath in the bore of the spectrometer. The heat transfer fluid was circulated through the helical heat exchanger by a hightemperature circulating bath. To prevent interference with the NMR measurements, the heat transfer fluid and the bath fluid were a non-hydrogen perfluoropolyether, Galden HT-270, from Ausimont. This particular fluid was chosen for its combination of low viscosity at room temperature, low vapor pressure at elevated temperatures and very low water solubility (14 ppm).

A Luxtron Fluoroptic thermometer, using a very small fluoroptic sensor at the end of a glass fiber light pipe, measured the temperature of the bath fluid to  $\pm$  0.1 C. This thermometer probe is minimally invasive, non-conductive electrically and remains accurate in high magnetic fields and in high RF fields. NMR data was not acquired until after the bath and cell temperatures had stabilized. The maximum variation in temperature and pressure was approximately  $\pm$ 0.1 C and  $\pm$ 0.2 psig, respectively. To generate a constant magnetic field from the spectometer permanent magnet, the magnet cabinet and coil temperature must be controlled at a point slightly above ambient room temperature. For this work, the set point temperature in the instrument cabinet was 30 C (545.7 °R).

**Sample Preparation for NMR.** Oxygen is paramagnetic. When dissolved, the oxygen increases the  $T_1$  (and  $T_2$ ) relaxation in refined hydrocarbon fluids and water. A helium sparging technique was used to remove the oxygen from solution in the samples. After sparging, the sample fluid was

maintained under a positive-pressure helium atmosphere while being pumped into the sample cell. The cell was a flowthrough design and was flushed prior to the lower end being sealed and a constant pressure being applied.

Sparging efficiency was determined using the least viscous base oil, sample S1. Helium was bubbled through a 100 ml volume sample at rate of 1 l/min. The  $T_2$  was periodically measured over several hours. The results are displayed in **Figure 1**. After two hours, no further appreciable removal of oxygen was observed. For each sample, sparging was conducted for a minimum of three hours prior to NMR data collection.

As with core preservation issues, the question of representativeness can be raised. What is the dissolved oxygen content in a fresh OBMF in the reservoir? The standard assumption is that the reservoir is at a reduced condition and that solution oxygen will be bound relatively quickly.

**Viscosity Measurement.** The temperature dependence of viscosity was measured using a Ruska rolling ball viscometer. Prior to measuring the viscosity of the OBMF samples, the viscometer was calibrated using the N100, S6, and S3 Cannon viscosity standards. After determining the calibration constant for the viscometer, the viscosities of the OBMF samples were measured. The NMR relaxation measurements were performed on a fresh sample after the viscosity determination.

**Interpolation of Viscosity-Temperature Data.** Since the viscosity and relaxation data are not taken simultaneously, one of the data sets must, in general, be interpolated. For each sample, viscosity values were linearly interpolated with respect to the inverse absolute temperature  $(1/T_R)$ . To determine the viscosity value at the temperature of the relaxation data, slight or negligible interpolation adjustments of the original viscosity-temperature data were needed. **Figure 2** illustrates the linear relations used in these interpolations.

**Calculation of T<sub>1</sub> and T<sub>2</sub> Relaxation Distributions.** A commercial relaxation inversion software package, WinDXP from Resonance Instruments, generated the relaxation distributions. The inversion procedure produces the style of multi-exponential logarithmically spaced data that has been shown to be successful for NMR applications in rock.<sup>14</sup>

The mean log values,  $T_{1ML}$  and  $T_{2ML}$ , were calculated from the distributions derived from this software. The component weight  $f_i$ , the software-generated signal intensity for the i<sup>th</sup> component normalized with respect to the number of scans in the measurement, was input data for determining the mean log  $T_2$ ,  $T_{2ML}$ :

$$T_{2ML} = \exp\left(\frac{\sum f_i \ln T_{2i}}{\sum f_i}\right)....(1)$$

An analogous expression determines  $T_{1ML}$ .

#### **Results and Analysis**

The viscosity and the mean log relaxations are provided as a function of temperature for each sample in **Table 3**.

**Temperature Dependence of T**<sub>2ML</sub> and T<sub>1ML</sub> Relaxations. The temperature dependence of T<sub>2ML</sub> and T<sub>1ML</sub> relaxation data for each sample are plotted in Figure 4 and Figure 5, respectively. Note the general linear dependence and the fact that the slopes are similar for all samples. T<sub>2ML</sub> and T<sub>1ML</sub> exhibit similar slopes also. The relaxation-temperature slopes showed a small, but consistent tendency to increase with the initial sample relaxation value at ambient lab conditions. This was never modeled well enough to warrant a representation.

The basic linear dependence of the relaxation on absolute temperature  $T_R$  is expected. The NMR relaxation in simple non-polar liquids is proportional to the translational diffusion, an intramolecular interaction.<sup>15</sup> In practice, this can be assumed to be the infinite-dilution binary diffusivity constant,  $D^0_{AB}$ , which is often assumed to have a linear temperature dependence. However, the theory for liquid state diffusion coefficients is quite idealized and fails to fully represent even simple non-polar low-viscosity hydrocarbon liquids.<sup>16</sup> While a linear dependence can fit a restricted range of absolute temperature, a broad temperature range usually requires that the diffusivity be represented by two constants A and B,<sup>16</sup>

$$D_{AB}^{0} = A \exp\left(\frac{-B}{T_{R}}\right).$$
 (2)

A functional relationship of the form of equation (2) did not significantly improve the correlation of relaxation with absolute temperature over the range of this work.

**Viscosity Dependence of T**<sub>2ML</sub> and T<sub>1ML</sub> Relaxations. The viscosity dependence of mean log T<sub>2</sub> and T<sub>1</sub> relaxation data for each sample is plotted in **Figure 6** and **Figure 7**, respectively. The lower trend line in **Figure 6** represents the correlation developed by Straley *et al.* for sixty-four crude oils at ambient conditions,<sup>17</sup> that later successfully represented the T<sub>2</sub> dependence on viscosity over a temperature range similar to the present study,<sup>18</sup>

$$T_{2ML} = 1.2/\mu^{0.9}$$
 .....(3)

As shown in **Figure 6**, these base oils and OBMF samples had a much steeper dependence of mean  $\log T_2$  on viscosity,

A typical dependence of  $D^0{}_{AB}$  on viscosity  $\mu$  for such materials has a power law dependence on viscosity with exponent ranging from –0.5 to –1. $^{16}$ 

If the  $T_{1ML}$  dependence had not been measured, this cubic dependence on viscosity might be blamed on convection

currents within the sample cell. As **Figure 7** shows, the viscosity dependence for  $T_{1ML}$  is essentially identical to that of  $T_{2ML}$ . But a  $T_1$  measurement would not be influenced by such convection, for the same reasons that it is not influenced by diffusion. A  $T_1$  measurement is an energy transfer measure, not a phase coherence measure like  $T_2$ . Moreover, the cell has a small diameter/length ratio and measurements show that the counter-wound double helical heat exchanger windings impose a negligible temperature gradient in the cell bath.

**Temperature Dependence of the T**<sub>1ML</sub>/T<sub>2ML</sub> **Ratio.** A minor dependence of T<sub>1ML</sub>/T<sub>2ML</sub> on temperature is portrayed in **Figure 8**. Over the range of explored temperature, the T<sub>1ML</sub>/T<sub>2ML</sub> ratio increases 10-20%. The slope seems to be sample dependent. This might represent a change in coupling into intermolecular interactions or be related to a rapidly increasing diffusivity with temperature.

#### Comparison of Relaxations in Base Oil versus in OBMF.

There was no consistently discernible NMR relaxation behavior difference between a base oil and its corresponding lab filtrate, S1-S7 and S2-S9, as evident from **Figures 4**, **5**. Both base oil-OBMF pairs showed small (< 10%) offsets in viscosity, as illustrated in **Figure 2** and **Table 3**. This suggests that a base oil might be used with some confidence in lab tests if OBMF is unavailable and if forward modeling is not planned. As discussed earlier, wettability alteration can be the dominant representativeness issue for lab relaxation measurements in core with multiple phases present.

#### Conclusions

Degassing of dissolved oxygen is necessary before reproducible lab  $T_1$  and  $T_2$  measurements can be made on OBMF and base oils. This, of course, raises the question as to what is the dissolved oxygen content in a fresh OBMF in the reservoir.

There were no significant or consistent differences in the  $T_1$  and  $T_2$  relaxations as a function of temperature between a base oil and an OBMF derived from that base oil.

Both  $T_1$  and  $T_2$  relaxations increased approximately linearly with the absolute temperature over the range studied (30-100 C, 546-670 °R).

The viscosity of each sample varied approximately linearly with the inverse of absolute temperature. However, the OBMF and base oil samples did not fit standard correlations that relate  $T_1$  and  $T_2$  to dead crude oil viscosity. The OBMF and base oil samples had a steeper dependence on viscosity, approximately as the inverse 3.36 power.

The  $T_1/T_2$  ratio trended upward with temperature.

It is recommended that NMR parameters of a degassed OBMF be measured in the lab at reservoir temperature as part of any comprehensive evaluation program. On the basis of the present work, a base oil could be substituted if wettability shifts were not an issue.

#### Nomenclature

fi	= signal amplitude of i <sup>th</sup> component of a relaxation	i
	distribution	
-0	2	

- $D_{AB}^{0}$  = infinite-dilution binary diffusivity, cm<sup>2</sup>/sec
- $\log = \log base 10$
- $\ln = \log base e (natural log)$
- $T_1$  = longitudinal relaxation time constant, sec
- $T_2$  = transverse relaxation time constant, sec
- $T_{1i}$  = i<sup>th</sup> distribution component of longitudinal relaxation time, sec
- $T_{2i}$  = i<sup>th</sup> distribution component of transverse relaxation time, sec
- $T_{1ML}$  = logarithmic mean of the set of  $T_{1i}$ , sec
- $T_{2ML}$  = logarithmic mean of the set of  $T_{2i}$ , sec
- $T_R$  = sample temperature in ° Rankine
- $\mu$  = sample viscosity, cp

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TABLE 1. Sample Description at 545.7 ° R (30 C)								
Sample	Description	Туре	<b>μ</b> (ср)	T <sub>2ML</sub> (sec)	T <sub>1ML</sub> (sec)			
<b>S</b> 1	HDF 2000	Base oil	3.84	0.658	0.674			
<b>S</b> 2	ESCAID 110	Base oil	3.43	1.21	1.22			
<b>S</b> 3	<b>BIOBASE 130</b>	Base oil	3.32	0.947	0.948			
S4	EMNL 8ppb	Filtrate	4.02	0.716	0.799			
S5	EMNL HT 8ppb	Filtrate	3.19	0.792	0.846			
<b>S</b> 6	EMNL HT 4ppb	Filtrate	3.54	0.740	0.778			
<b>S</b> 7	HDF 2000	Filtrate	3.73	0.630	0.655			
<b>S</b> 8	-	Filtrate	3.49	1.15	1.25			
S9	ESCAID 110	Filtrate	3.19	1.07	1.11			

Table 2. NMR Spectrometer Settings					
Acquisition:					
Nucleus	$^{1}H_{1}$				
Spectrometer Frequency	2.03 MHz				
Filter Width	100 kHz				
Number of Echoes	2000 - 8000				
Number of Repetitions	64				
T <sub>1</sub> Pulse Sequence	Inversion Recovery				
T <sub>2</sub> Pulse Sequence	CPMG				
Timing:					
90° Pulse Length	24.6 µsec				
180° Pulse Length	48.3 µsec				
CPMG T <sub>2</sub> Sequence Inter-Pulse Spacing	800 µsec				
CPMG Experiment Recycle Delay	5 T <sub>1</sub>				

TABLE 3.	NMR Relax	ation Data		
Sample	T <sub>₫</sub> (°B)	T <sub>2ML</sub> (sec)	T <sub>1ML</sub> (sec)	μ (cp)
S1	545.7	0.658	0.674	3.84
51	607.2	1 20	1 27	3 23
	641.4	1.20	1.27	3.02
	668.4	1.05	2.07	2.89
	008.4	1.95	2.07	2.09
	545.7	1.21	1.22	3.43
S2	576.6	1.58	1.66	3.12
	612.6	2.36	2.59	2.91
	641.4	2.87	3.23	2.77
	673.8	3.20	3.71	2.65
	545.7	0.947	0.948	3.32
\$3	569.4	1.24	1.20	3.13
~~~	605.4	1.82	1.82	2.90
	639.6	2 39	2 59	2.90 2.74
	675.6	3.05	3.32	2.61
	5157	0716	0.700	4.02
<b>C</b> 4	545.7	0.710	0.799	4.02
54	504.2	0.870	1.00	3.95
	609.3	1.06	1.35	3.46
	043.2	1.00	2.08	5.25
	670.2	1.98	2.61	3.10
	545.7	0.792	0.846	3.19
S5	558.6	1.03	1.10	3.34
	605.4	1.65	1.79	2.94
	634.2	2.20	2.45	2.77
	664.8	2.63	2.92	2.63
	545.7	0.740	0.778	3.54
<b>S</b> 6	567.6	0.948	1.00	3.28
	607.2	1.51	1.62	2.92
	641.4	2.02	2.27	2.71
	668.4	2.49	2.88	2.58
	545.7	0.630	0.655	3.73
S7	578.4	0.912	1.00	3.28
	616.2	1.37	1.49	2.96
	639.6	1.72	1.90	2.82
	672.0	2.29	2.58	2.65
	545 7	1 1 5	1 25	3 49
58	578.4	1.15	1 70	3 22
50	600.6	1 97	2.16	2 97
	639.6	2 73	3.00	2.27
	668.4	3.39	3.92	2.59
	5157	1.07	1 1 1	3 10
50	572 1	1.07	1.11	2.19
39	5/5.4	1.40	1.30	2.94 2.67
	627.8	2.01	2.30	2.07
	057.8	2.44	2.94	2.30
	663.5	2.63	3.23	2.47



**Figure 1.** Helium sparging for three hours removes dissolved paramagnetic  $O_2$  from the samples, thereby increasing  $T_2$ .



**Figure 3.** Example  $T_1$  and  $T_2$  relaxation distributions for sample S3 @ 605.4 °R. The  $T_{1ML}$  and  $T_{2ML}$  values are calculated from these amplitudes.



**Figure 2.** The temperature dependence of sample viscosity as measured by a rolling ball viscometer. The viscosity-temperature data was linearly interpolated using these plots.



**Figure 4.** The NMR  $T_2$  relaxation for each sample is approximately linear with absolute temperature.



**Figure 6.** The OBMF samples had a stronger dependence of  $T_2$  on viscosity ( $T_{2ML} = 64.8 \ \mu^{-3.36}$ ) than is predicted by a correlation developed for crude oils, lower trend line ( $T_{2ML} = 1.2 \ \mu^{-0.9}$ ).



**Figure 5.** The NMR  $T_1$  relaxation for each sample is approximately linear with absolute temperature.



**Figure 7.** The T<sub>1</sub> relaxation dependence on viscosity (T<sub>2ML</sub> =  $69.3 \mu^{-3.30}$ ) tracked the behavior exhibited by T<sub>2</sub>.



**Figure 8.** The  $T_1$  and  $T_2$  relaxations were approximately equal, as is typical for these liquids. There was a trend of increasing  $T_1/T_2$  ratio for increasing temperature.