

Pulsed Nuclear Magnetic Resonance

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Abstract

*We use pulsed nuclear magnetic resonance to determine the spin-spin relaxation and spin-lattice relaxation for glycerin and samples with Fe^{3+} ionization through observing free-induction decay. We determine magnetic inhomogeneity to be approximately $1.678 * 10^{-4} \text{ kG}$ from varying local magnetic field. With spin-echo amplitude measurements depending on variance of τ and T_D , we calculate T_2 to be $2400 * 10^{-6} \pm 0.00088 \text{ s}$ and T_1 to be $2.25 * 10^{-3} \pm 0.00053 \text{ s}$, respectively, for glycerin. From comparison to water, glycerin's higher viscosity increases T_2 . In addition, paramagnetic impurities from testing two concentrations of Fe^{3+} ionized samples demonstrated an increase in T_2 , with $1600 * 10^{-6} \pm 0.00293 \text{ s}$ for a $1 * 10^{16}$ ions/mL concentration and $1600 * 10^{-6} \pm 0.01984 \text{ s}$ for a $1 * 10^{19}$ ions/mL concentration.*

I Introduction

Pulsed nuclear magnetic resonance (PNMR) is a method used to determine the magnetic moment of nuclei, finding their distinct energy levels based on their nucleus spin and splitting. Nuclear magnetic resonance was an original technique using oscillating electric and static fields' resonance frequencies to find a suitable resonant condition for various samples. [1]

Deriving from this idea, PNMR has changed the measurement efficiency through implementing the oscillating electromagnetic fields as short pulses of radio frequency signals. The angle between the application of these fields is altered when the oscillation frequency is at Larmor frequency. With a perpendicular oscillating magnetic field applied with a static field, proton magnetization in a sample placed between two magnetic poles is slowly tipped through the applied torque to 90° to show as a $\frac{\pi}{2}$ pulse. [1] The exponential decay following this signal is known as free-induction decay (FID), which we can evaluate characteristic time, T^* , from

$$\frac{1}{T^*} = \frac{1}{T_2} + \gamma \Delta B \quad (1)$$

where T_2 is spin-spin relaxation, ΔB is the distribution of applied magnetic field, and γ is the magnetogyric ratio relating magnetic moment μ to angular momentum $\hbar \mathbf{I}$. [1]

With spin echo measurements, we eliminate magnet inhomogeneity represented by ΔB in Equation 1 caused by areas of instrumental error caused by an inconsistent magnetic field through a relation

given by Bloch's equations:

$$\frac{dM_z}{dt} = \gamma(\mathbf{M} \times \mathbf{B})_z + \frac{M_0 - M_z}{T_1} \quad (2)$$

where $(\mathbf{M} \times \mathbf{B})_z$ gives the Larmor frequency from the magnetic field \mathbf{B} and a collective of nuclear moments \mathbf{M} . We determine the z component of \mathbf{M} , M_z from the following:

$$M_z = M_0[1 - 2e^{-\frac{T_D}{T_1}}] \quad (3)$$

I.1 Motivation

In this experiment, we measure resonant frequencies with PNMR. Each of the tested liquid samples—water, glycerin, and two concentrations of Fe^{3+} ionized solutions—contains protons in the nuclei which are measured for collective resonant adsorption, determined by the finely tuned resonant condition. This is done through observation of the free induction decay, or FID, telling the different relaxations of the spins—spin-spin relaxation (T_1) and spin-lattice relaxation (T_2)—which we can use to calculate magnetic moments of the nuclei. [1]

II Experimental Procedure

The experimental setup (Figure 2) consists of several main components. A frequency generator produces a sinusoidal signal at 30.000 MHz and $1V_{rms}$, which is then split to the gate, allowing control from the *PulseBlaster* software to adjust TTL pulses. The variable attenuator is set to 5dB

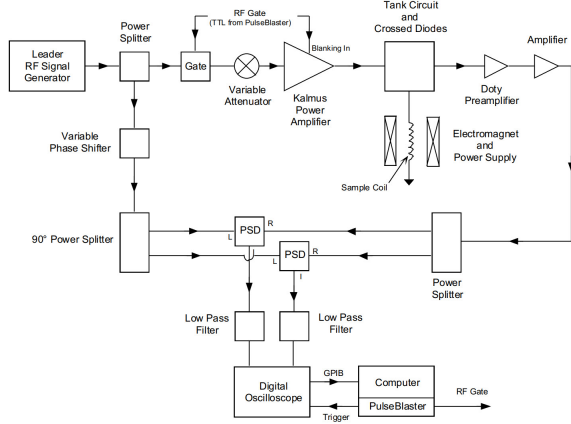


Figure 1: Diagram of pulsed nuclear magnetic resonance spectrometer setup, from RF signal generator to oscilloscope measurement [1]

and the signal is amplified to be inputted through a sample coil wrapped around each liquid sample. With additional adjustments on the tank circuit and cross diodes, it is split again into two phase sensitive detectors (PSD), where a variable phase shifter can help adjust the mixed signals and observed on the oscilloscope. [1]

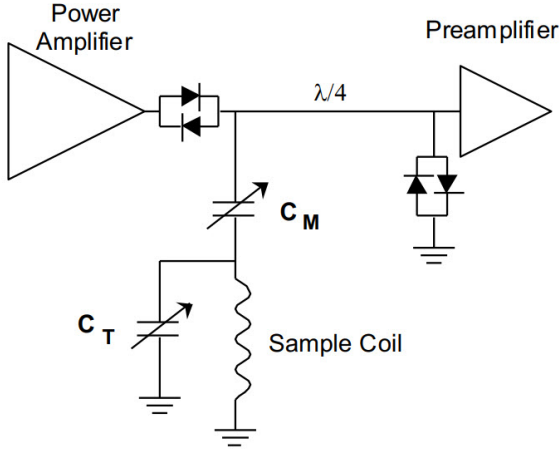


Figure 2: Diagram of the tank circuit and crossed diodes [1]

For each of water, glycerin, and both concentrations of Fe^{3+} ionized solutions, the NMR tube is changed out and knobs controlling the capacitance values in Figure 2 of the tank circuit are adjusted so that there is production of a minimized sinusoidal signal. With the expression for H^1 -based samples,

$$f(\text{MHz}) = 0.42577B(\text{kG}) \quad (4)$$

we determine the applied magnetic field to be approximately 7.0461kG from resonant frequency f , 30MHz. Then, we measure spin-lattice relaxation

T_1 through input of the following: a 1s initial delay time, $20\mu\text{s}$ π pulse, 5ms delay time T_D , $10\mu\text{s}$ $\frac{\pi}{2}$ pulse, 2ms delay time τ , and finally a $20\mu\text{s}$ π pulse. For spin-spin relaxation T_2 , the following sequence is inputted into the *PulseBlaster* software: 1s initial delay time, $10\mu\text{s}$ $\frac{\pi}{2}$ pulse, 2ms delay time τ , and a $20\mu\text{s}$ π pulse. Both sequences are copied for 1000 cycles.

Nuclei travel and become out of phase depending on magnet inhomogeneity causing a different magnetization. The π pulse reverses the moments of nuclei so that they are back in phase from the result of the preceding $\frac{\pi}{2}$ pulse. [2] The maximum resulting magnetization produces a spin echo which can reliably determine T_2 in solutions, even with a high viscosity causing a low diffusion in spin. [1]

We note the amplitude of the signal echo according to the variation of τ in T_2 and T_D in T_1 . With the water solution, an example of a typical FID (Figure 3) can be visualized, with the leftmost peak indicating a $\frac{\pi}{2}$ pulse decaying at e^{-t/T^*} , the middle peak as a π pulse decaying at $e^{-\tau/T_2}$, and the rightmost being the spin echo. [2]

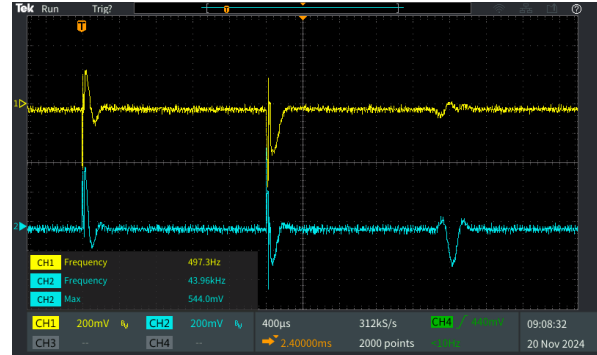


Figure 3: Typical time trace of a FID and spin echo of water sample; Signal in Ch. 2 (blue) is a phase shift of signal in Ch. 2 (yellow); Peaks indicate the following from left to right: π pulse, $\frac{\pi}{2}$ pulse, spin echo

III Results and Analysis

We first insert a water sample into the sample coil to display a FID displaying measurements from which we can determine magnet inhomogeneity ΔB using Equation 4 and

$$\omega = \gamma B \quad (5)$$

T_2 as $200\mu\text{s}$ and T^* as 2ms, giving an inhomogeneity of $1.678 * 10^{-4}\text{kG}$.

III.1 Glycerin

Following, we measure T_1 (Figure 4) for glycerin, adjusting T_D and noting the spin-echo amplitude.

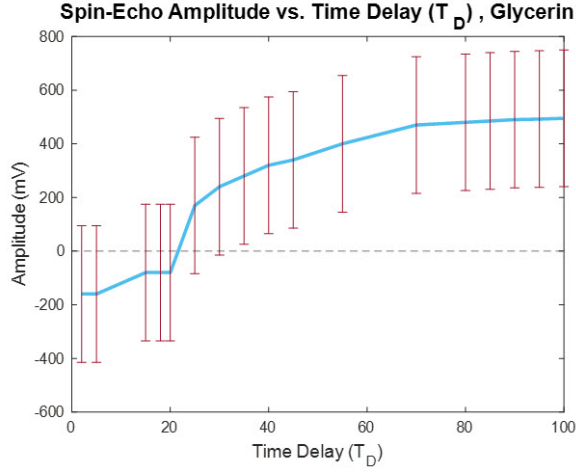


Figure 4: Spin-echo amplitude vs. time delay for glycerin sample

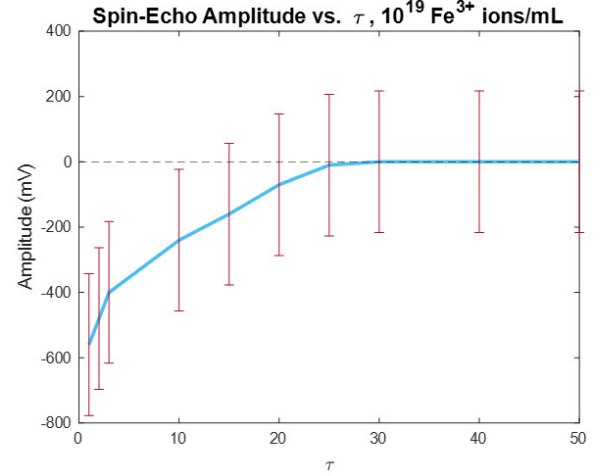


Figure 6: Spin-echo amplitude vs. τ for highest concentration Fe^{3+} (1×10^{19} ions/mL) sample

There is an initial, exponential increase which then plateaus at around 470mV. Using equations 2 and 3, T_1 , or the inversion recovery of M_z of glycerin, is $2.25 \times 10^{-3}s$ with an uncertainty of $\pm 0.00053s$. T_2

samples 1×10^{19} ions/mL (Figure 6) and 1×10^{16} ions/mL (Figure 7). Using the same method for calculating spin-spin relaxation, T_2 for the lower concentration sample is $1600 \times 10^{-6} \pm 0.00293s$ and $1600 \times 10^{-6} \pm 0.01984s$ for the higher concentration sample, with uncertainties calculated by the least-squares fit.

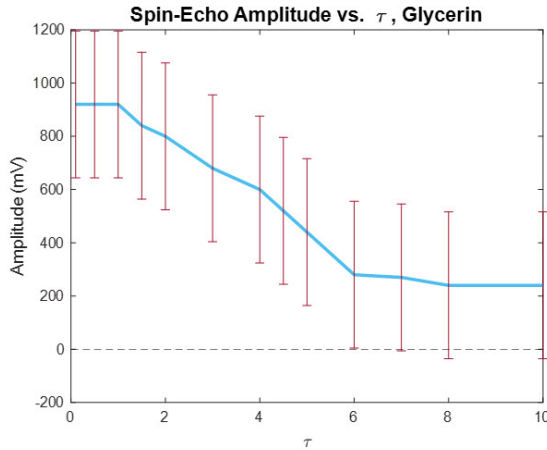


Figure 5: Spin-echo amplitude vs. τ for glycerin sample

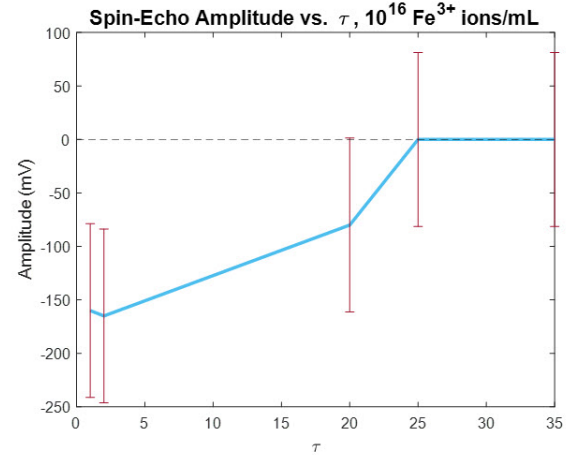


Figure 7: Spin-echo amplitude vs. τ for lower concentration Fe^{3+} (1×10^{16} ions/mL) sample

involving adjusting τ over 0s to 10s gives a generally decreasing spin-echo trend. We calculate T_2 to be approximately $2400 \times 10^{-6} \pm 0.00088s$ with Equation 1 and observation of the change in the speed of nuclei over time and the difference in speed itself through T^* . [2] [3].

Glycerin has a higher viscosity than water [4], and has demonstrated a higher T_2 than water. Therefore, higher viscosity is predicted to have an increased spin-spin relaxation.

III.2 Paramagnetic Samples

We measure spin-echo amplitude for multiple values of τ for two concentrations of Fe^{3+} ionized

IV Conclusion

We successfully used water to determine magnet inhomogeneity, $1.678 \times 10^{-4} \text{ kG}$, later used in calcula-

tions to determine spin-spin and spin-lattice relaxation for glycerin and paramagnetic samples, with $1 * 10^{16}$ ions/mL and $1 * 10^{19}$ ions/mL concentrations of Fe^{3+} . Data from the glycerin has shown a trend that higher viscosity leads to a higher spin-spin relaxation, a time value of $2400 * 10^{-6} \pm 0.00088\text{s}$. Following the trends of spin-echo amplitude with respect to τ for paramagnetically impure samples, we find an increase in spin-spin relaxation time, T_2 , from $1600 * 10^{-6} \pm 0.00293\text{s}$ at $1 * 10^{16}$ ions/mL concentration to $1600 * 10^{-6} \pm 0.01984\text{s}$ at $1 * 10^{19}$ ions/mL concentration.

IV.I Improvements and Future Directions

Improvements can be implemented to this experiment through even higher efforts to minimize the sinusoidal signal when calibrating the tank circuit by fine-tuning the inner applied capacitance. We are also able to gauge the trends of viscosity and paramagnetic impurities with spin-spin relaxation with additional liquid samples with varying viscosity and perform more dilutions of the Fe^{3+} solution. This will provide additional data to form a more well-rounded conclusion and minimize uncertainty, as some calculations resulted in a relatively large uncertainty, such as T_2 for the higher concentrated paramagnetic sample.

References

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