Pulsed NMR

Nicolas Renaud

Lab Partners: Jacob Letsinger, Chris Ogunsami

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Abstract

This report, investigating Pulsed Nuclear Magnetic Resonance, entails the dynamics of nuclei precessing magnetic field that is applied for various lengths of times, called pulses. This study looks at the relation between the magnetic field's strength and the two relaxation times, spin-lattice relaxation(T_1) and spin-spin relaxation(T_2). This report found that $T_1 = 27.440 \pm 4.983$ ms and $T_2 = 9.920 \pm 0.5393$ ms. An apparent spin-spin relaxation (T_2^*) was also measured to have an interval of 54.98 ± 4.049 μs or 0.5498 ± 0.004049 ms. This time is much smaller than 1 ms showing inconsistency in measurement, coming from sensitivity. The real spin-spin relaxation time also showed some discrepancy, since the mineral oil sample was known to have relaxation times between 10-30 ms. The external magnetic field applied to the sample was also calculated to be $B_o = 3.66 \pm 0.03663$ kG. These findings confirm the nuclear magnetic resonance phenomenon and the pulsed method.

1 Introduction

Nuclear Magnetic Resonance (NMR) is a technique, developed by Edward Purcell and Felix Bloch in 1946, for analyzing the molecular structure and dynamics of a sample of interest. There are different types of NMR methods. This report will investigate the Pulsed NMR, introduced in 1950 by Erwin Hahn, which offers an analysis of the nuclear spin behavior within that sample by using pulsed oscillations rather than continuous-waves. This investigation aims to analyze the Pulsed NMR method on a mineral oil sample, focusing on the apparent and real spin-spin relaxation time as well as the spin-lattice relaxation times.

2 Background Information

Pulsed Nuclear Magnetic Resonance (NMR) is a method to provide insight on the interactions between a material and an external magnetic fields using spectroscopy. The technique involves applying a short pulse, being in the radio frequency scale, at precise intervals to manipulate the nuclear spin within the sample. The precession in the nucleus can be measured in terms of relaxation times. The first relaxation time is the spin-lattice relaxation time. This represents the time for which the nuclei return to their equilibrium alignment with the external magnetic field. This time interval is influences by the nuclear spin and its surroundings, such as other molecules in the sample [2]. The second relaxation time is the spin-spin relaxation time. This signifies the time interval for which the phase coherence among the nuclei dissipates [2]. This dissipation is shown to be an exponential decay over increasing time interval. The apparent spin-spin relaxation time happens when other environmental factors cause additional phase changes in the nucleus's spin behavior, which is typically much shorter than the other relaxation times.

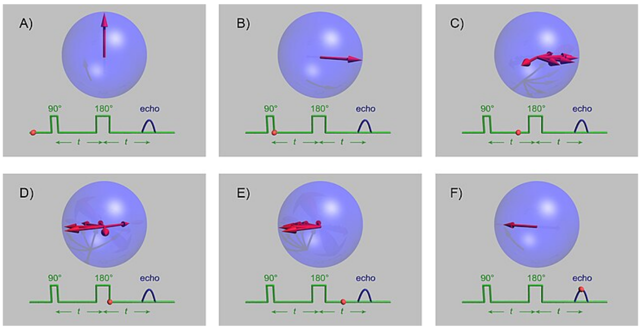
3 Theory

The precession rate of protons depends linearly on the magnetic field applied to it, according to the equation:

$$f_o = 4.258 B_o \tag{1}$$

Where f_o is the frequency in Megahertz, which is in the radio frequency range of the electromagnetic spectrum, and B_o is the force magnetic field applied on the sample in Kilogauss.

The magnetic field used comes from a permanent, rare-earth, magnet that has a field strength of about 3.6 kG, with small variations from the local environment of each nucleus, which will be calculated. As explained in the Background Information, the two main relaxation times will also be calculated. The relationship between the spin-spin relaxation time and the voltage amplitude given by the nucleus's reaction to the magnetic field is an exponential decay, while the spin-lattice relaxation time and the voltage amplitude has a quadratic relationship. The following schematic shows how the nuclear moments are rotated then refocused using the Hahn spin-echo sequence. [3]



4 Experimental Procedure

First the spectrometer is calibrated by minimizing beat oscillations and maximizing the FID (free-induction decay) signal. Next, the FID amplitude is measured as a function of time. Using this calibration, the spin-lattice relaxation time(T_1), and the spin-spin relaxation time (T_2) can be measured and calculated.

Once calibrated, the apparent spin-spin relaxation time (T_2^*) can be calculated by measuring the FID voltage amplitude. The spin-lattice relaxation time (T_1) was calculated by varying the time delay between the two pulses. Finally the real spin-spin relaxation time (T_2) was calculated by measuring the spin-echo and fit to an exponential fit.

5 Results

The following figures show the results of this experiment.

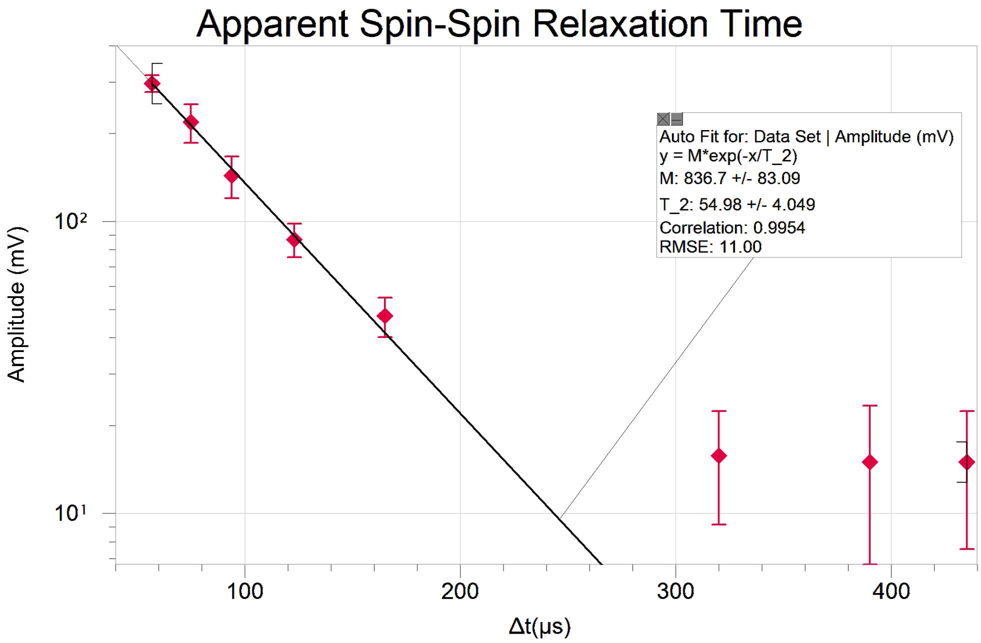


Figure 1: Apparent Spin-Spin Relaxation Time (T_2^*)

The graph shows the behavior between the pulsed frequency and the free induction decay voltage amplitude. The sample is placed in the strong external magnetic field, B_o, aligning the proton's magnetic dipole with the magnetic field. The pulse is applied, causing the magnetic dipole to precess in the direction of the B_o and then relax with respect to the frequency. This spin-spin relaxation time is followed by the FID signal, created by the interaction between magnetic fields. Fitting the data to the exponential equation: $V = Me^{-t/T_2}$, the apparent spin-spin relaxation time, T_2^*, is calculated to be $54.98 \pm 4.049\mu s$. the magnetic field can also be calculated, since f_o was held constant throughout the experiment at 15.6 ± 0.156 MHz. Thus, using equation (1), $B_o = 3.66 \pm 0.03663$ kG.

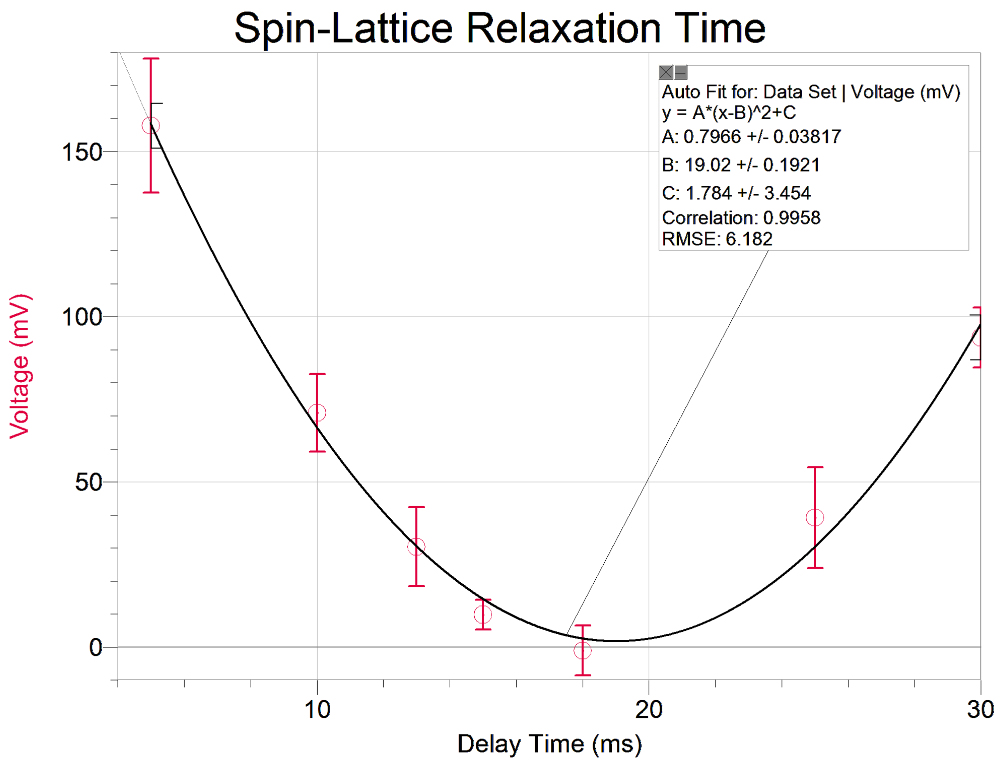


Figure 2: Spin-Lattice Relaxation Time (T_1)

This graph depicts the nucleus' amplitude of magnetization following the B-pulse as a function of delay time. The spin-lattice relaxation time(T_1) is the rate at which the magnetization returns to equilibrium. The first pulse causes the spin to precess in the magnetic field and the second causes it to rate the spin back , creating the spin echo. This amplitude is minimized at T_{min}, representing the time required for the magnetization to record in the longitudinal plane, and aligning with the external magnetic field. These measurements can be fit to a quadratic function. Using the line of best fit T_{min} was measured to be 19.02 ± 3.454 ms, and using the equation $T_1 = T_{min}/ln(2)$, $T_1 = 27.440 \pm 4.983$ ms. An approximate $T_1^{'}$ was also measured through the oscilloscope of 10.72 ms. This value is much smaller, which can be a factor of not accounting for the decay amplitude's proper start and end point.

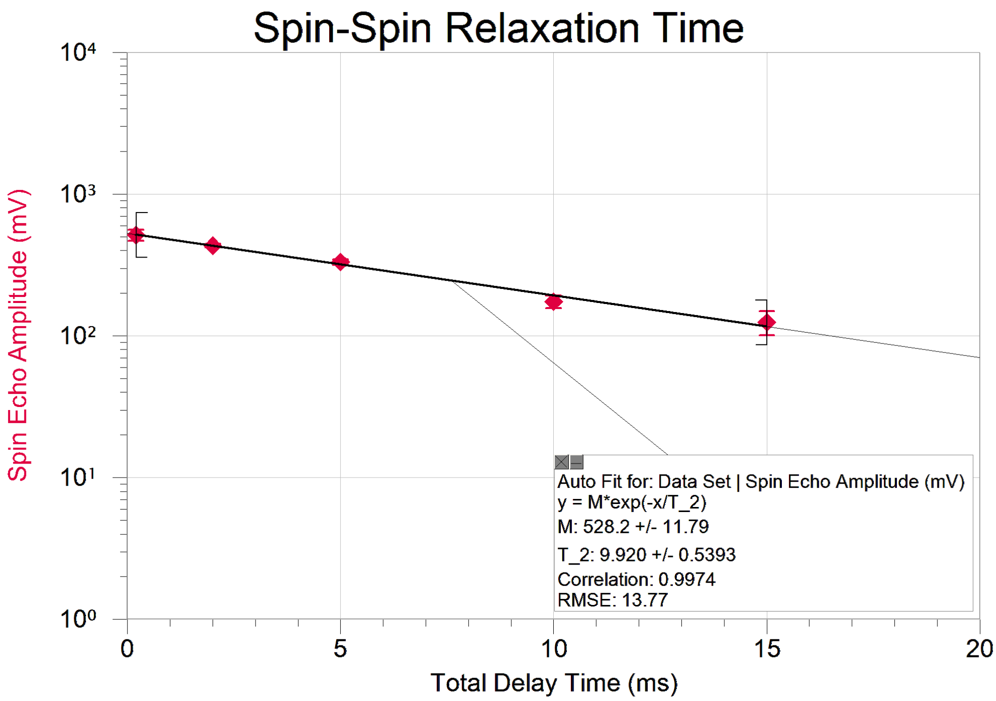


Figure 3: Spin-Spin Relaxation Time (T_2)

This figure illustrates the decay of the spin-echo amplitude (in mV) as a function of the total delay time (2τ), with a semi-logarithmic relation. As explained earlier, the first pulse causes a precession in the direction of the magnetic field, and the second rotates the spin back, creating a coherent signal called spin echo. As delay time because the pulses increases, the spin-spin interaction decreases, decreasing the spin-echo amplitude. This decay can be fitted to an exponential function, giving the real spin-spin relaxation time (T_2), which was calculated to be 9.920 ± 0.5393 ms. Comparing this to the apparent spin-spin time from figure 1 of $54.98 \pm 4.049 \mu s$, the values are much different by a factor of 3. This is because the apparent reflects on the FID and, thus, more affected by the spin-spin interaction as well as the magnetic fluctuations in the environment and the mineral oil sample.

6 Discussion

The mineral oil was known to have T_1 and T_2 values between 10-30 ms. The value measured for T_1 fit in this range giving 27.440 ± 4.983 ms. However, the value for T_2, measured at 9.920 ± 0.5393 ms, was slightly below this range, meaning there is error. This error must come from the measurements being averaged over a small range of values, measuring only five data points, as well as improper interval measured. The range of delay time was only between 0 to 15 ms, if more data was collected over a larger range, this value would be more precise. The apparent spin-spin relaxation time was calculated to be 0.5498 ± 0.004049 ms which is lower than 1 ms, as expected. This is much lower than the real spin-spin relaxation time because of measurement discrepancies coming from sensitivity, pulse imperfections, environmental conditions, as well as field inhomogeneities. This is why the real spin-spin relaxation is measured over the spin-echo, removing sensitivity issues.

7 Conclusion

This report on Pulsed Nuclear Magnetic Resonance (NMR) investigated the behavior of nuclei precessing in magnetic fields applied for varying time intervals, called pulses. The experiments focuses on understanding the relationship between the magnetic field strength and the two relaxation times: spin-lattice relaxation (T_1) and spin-spin relaxation (T_2). Experimental findings reveal that T_1 was determined to be 27.440 ± 4.983 ms, while T_2 was found to be 9.920 ± 0.5393 ms. Additionally, an apparent spin-spin relaxation time (T_2^*) of 0.5498 ± 0.004049 ms was measured. The external magnetic field applied to the sample was calculated to be $B_o = 3.66 \pm 0.03663$

kG. These results confirm the phenomenon of nuclear magnetic resonance and validate the pulsed method. By employing pulsed oscillations rather than continuous waves, the analysis examines the behavior of nuclear spins within a mineral oil sample, providing crucial insights into relaxation processes and molecular dynamics. The discrepancy observed between the real and apparent spin-spin relaxation time values suggests a need for broader data collection, removing sensitivity factors. In conclusion, the investigation offers valuable insights into the dynamics of nuclear spins and relaxation processes, contributing to a deeper understanding of NMR phenomena.

8 References

[1] Adrian C. Melissinos. Experiments in modern physics. *New York, Academic Press,* ,1966

[2] Levitt, M. H. . Spin Dynamics: Basics of Nuclear Magnetic Resonance (2nd ed.). *John Wiley & Sons.*

[3] Wikimedia Foundation. (2024, January 5). Spin echo. *Wikipedia*. wikipedia.org/wiki/Spin_echo