

Finding Spin Lattice and Spin Spin Relaxation Times Using Pulsed NMR

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We use nuclear magnetic resonance (NMR) to measure the free induction decay (FID), spin-lattice relaxation times (T_1) and spin-spin relaxation times (T_2) for mineral oil and glycerin. Systematic sequences of RF pulses are used to measure these relaxation times. We find T_1 values of 20.0 ms and 40.0 ms for mineral oil and glycerin respectively with certainty to $\pm 5\%$. We find T_2 values of 15.9 ms and 37.1 ms for mineral oil and glycerin respectively with certainty to $\pm 5\%$.

I. INTRODUCTION

Nuclear Magnetic Resonance is a technique that exploits the intrinsic magnetic properties of atoms by using external magnetic fields and radio frequencies to systematically align the spins of their nuclei. The development of this technique occurred in 1946 by both Purcell and Bloch independently and has since had a major impact on the scientific world. NMR introduced a novel form of spectroscopy and a new way of studying quantum and nuclear phenomena. It's significance reaches far beyond the physics community, however. NMR has proven to have many uses which span various fields of research including chemistry, geology, biology, and medicine. A practical use of NMR is the common MRI machine (magnetic resonance imaging), which uses NMR techniques to produce images of human tissue with amazing detail and allows doctors to easily diagnose certain medical issues.

The present experiments use NMR techniques to measure the spin-lattice and spin-spin relaxation times in mineral oil and glycerin. By using an external magnetic field to align the spins of the nuclei within these samples and subsequently supplying a radio frequency pulse to the sample, we are able to alter the alignment of the spins in such a way that allows for measurement of the above mentioned times. The physics background necessary for the understanding of our techniques is explained in the following section. The apparatus used to carry out the experiments will be diagramed and explained in the "Apparatus" section of the paper. Following, a detailed description of the procedure and results of each experiment will be given. The results and the implications of these results will be summarized in the conclusion of the paper.

II. THEORY

The nucleus of an atom is a little spinning ball of charge with an angular momentum \mathbf{J} and a magnetic moment μ . It's angular momentum, J , is given by

$$J = I\hbar. \quad (1)$$

where I is the spin of the nucleus. For a single free proton, $I = 1/2$ since it is a Fermion. The spin of any

nucleus will break into different energy states due to its magnetic nature. There are $2I + 1$ of these states, each denoted by m_I . For our free proton, there are two such states: $m_I = \frac{1}{2}, -\frac{1}{2}$. For simplicity, we can think of the low energy state as spin up and the high energy state as spin down. In thermal equilibrium, there will be similar numbers of spin up and spin down protons. The net magnetization of the entire system is proportional to the difference between the number of spin up (N_1) protons and number of spin down (N_2) protons and is given by

$$M = (N_1 - N_2)\mu. \quad (2)$$

So in thermal equilibrium, net magnetization will be close to zero since $N_1 \approx N_2$.

In the presence of external magnetic field \mathbf{B} , the protons will align their spins in the direction of the magnetic field and a net magnetization in the system will appear. This is because more protons enter the low energy state and much fewer occupy the high energy state. This magnetization does not occur instantaneously, however. The time it takes for a system to reach its maximum net magnetization after being placed in an external magnetic field is called the spin-lattice relaxation time and is denoted T_1 .

Suppose we supply an external magnetic field in the z -direction. This process of attaining magnetization is given by the following differential equation:

$$\frac{dM_z}{dt} = \frac{M_0 - M_z}{T_1}. \quad (3)$$

This characteristic time, T_1 is called the spin-lattice relaxation time because when the system is placed in the \mathbf{B} field, the energy that leaves the protons as they transition from the high energy state to the low energy state flows into the other atoms surrounding them. This "lattice" of surrounding atoms gains the energy that the protons lose.

If we take Equation 3 with initial conditions such that at $t = 0$, $M_z = 0$, and integrate, we get the following equation:

$$M_z(t) = M_0(1 - e^{-\frac{t}{T_1}}). \quad (4)$$

This will be used to calculate the value of T_1 from the data obtained from the experiments.

Before introducing the idea of spin-spin relaxation time, we need to introduce the different kinds of pulses needed to observe the spin-spin relaxation time. Suppose we have a sample in an external magnetic field with the net magnetization in the z -direction. Then suppose we supply another external magnetic field in the x -direction. If this field is supplied for just the right amount of time, the result is a net magnetization fully in the x - y plane. This introduction of an orthogonal B field to alter the alignment of the spins is called a *pulse*. It is created using a short burst of radio frequency (RF). An RF pulse that transforms a magnetization in the z -direction into a magnetization in the x - y plane is called a $\pi/2$ pulse. If this pulse is applied for twice the amount of time, the net magnetization resides in the $-z$ -direction, and is called a π pulse.

If a particle with magnetic moment μ and spin I is displaced from the z -direction in a field B_z , it will begin to precess around the field direction with what is called the Larmor frequency.

$$\omega = \frac{\mu B_0}{\hbar I} \quad (5)$$

(expand on this!)

When a collection of protons in thermal equilibrium with M_{net} in the z -direction is supplied a $\pi/2$ pulse, the net magnetization will transform into the x - y plane. Once in the x - y plane, the spins of the protons will begin to de-phase with one another, and the net magnetization will return to thermal equilibrium after some time. As de-phasing is occurring, the magnetization precesses around the z -axis in the x - y plane. Refer to Figure 1 for a graphical representation of this process. The time that elapses from the initialization of the $\pi/2$ pulse until the net magnetization in the x - y plane is reduced by a factor of e is called the spin-spin relaxation time. It is denoted T_2 .

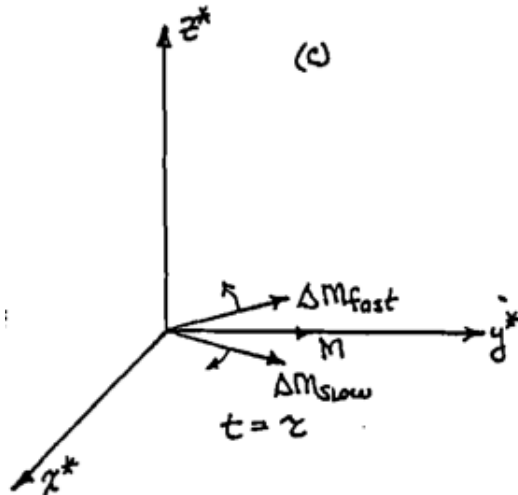


FIG. 1. After a $\pi/2$ pulse has been applied, the spins begin to de-phase with another and the magnetization precesses around in the x - y plane. As de-phasing progresses, the net magnetization in the x - y plane decreases.

In measuring T_2 directly, however, one finds a problem. The permanent magnet used does not provide a perfectly uniform magnetic field throughout the entire sample. (Explain spin echo here.)

Mineral oil and glycerin are used in the present experiments because of their collection of "free protons." Each hydrogen atom on its own has a single proton nucleus and one orbiting electron. This atom cannot be thought of as a free proton because the electron, with a charge of its own, affects the magnetic characteristics of the proton. The reason we can treat hydrogen as a free proton in this experiment is due to the covalent nature of the bonds between the hydrogen and carbon atoms. Mineral oil is a hydrocarbon. Since each hydrogen is covalently bonded to a carbon, their electrons are shared, and each hydrogen effectively holds two electrons. Since both electrons are in the same orbital, the Pauli Exclusion Principle prevents them from having the same spin. Otherwise, they would occupy the same energy state. This implies that there is one spin up electron and one spin down electron. Their spins cancel each other out and the result is no net effect on the proton. The hydrogen is essentially a free proton and can be treated as such. Glycerin behaves in the same way as each of its hydrogen atoms are covalently bonded.

III. APPARATUS

The apparatus used in the experiments consists of several key components. A diagram of the apparatus is shown in Figure 2.

The permanent magnet provides a steady B field in the z -direction, and the sample sits inside this B field.

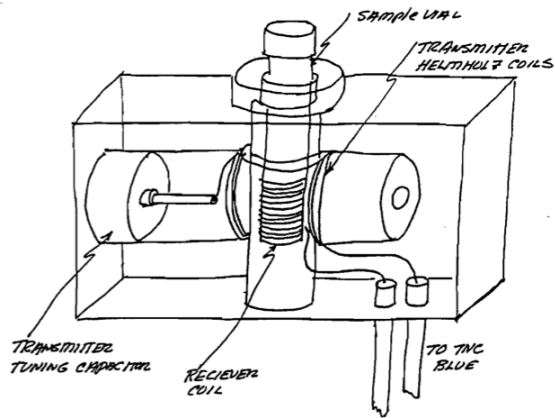


FIG. 2. Setup

A set of Helmholtz coils runs along side the sample that produces a B field in the x-direction. These coils are fed by an RF synthesized oscillator and carry the RF pulses that resonate with the sample and flip the spins into the x-y plane. Another set of coils surrounds the sample itself. These are called the pickup coils and are responsible for measuring the magnetization in the sample. They are positioned in such a way that magnetic fields from the sample create a current in the wire due to Faraday induction. It is important to note that only magnetizations of the sample in the x-y plane can be measured using this apparatus. The signal through these coils is picked up by a receiver which sends the signal to a mixer and an RF detector. Both of these signals are then sent to an oscilloscope which allows us to view the voltage detected.

The signal that goes into the mixer from the pickup coils is mixed with the signal coming from the RF synthesized oscillator. If the two signals are out of phase, the oscilloscope will show beats that result from the incoherence of the two sources. If, however, the two signals are in resonance with each other, a smooth signal will appear, and the beats will disappear.

IV. EXPERIMENT I

The goal of this experiment was to measure T_1 for two kinds of materials: mineral oil and glycerin. Since the pickup coils only measure the magnetization in the x-y plane, we needed a way to observe the change in magnetization that occurs in the z-direction in the x-y plane. In order to do this, a π pulse is first applied to the magnetized sample. This flips the net magnetization into the -z-direction. Once pulsed, the M_{net} will immediately begin to decay back into the +z-direction. Once there are equal numbers of spin up and spin down protons, M will go to zero. Eventually, M will begin to increase in magnitude in the +z-direction. To determine the magnitude of this M at any given moment, we then supply a $\pi/2$ pulse after some time τ which turns M into the x-y plane. This M_x can then be measured by the pickup coils. By per-

forming this series of pulses for several different values of τ , we can plot the corresponding voltages on a graph which allows for calculation of T_1 . The inverse of the exponential constant for the graph represents T_1 .

The procedure for carrying out the measurement was as follows. First, the sample was placed into the permanent magnetic field. The A pulse was set as a $\pi/2$ pulse and resonance between the RF and the sample was found. Once resonance was found, the A pulse was set to a π pulse and the B pulse was set to a $\pi/2$ pulse. A delay time of 1 ms was then applied, and the voltage of the peak was recorded. The voltage was then recorded for a series of increasing delay times. The graphs for each sample are shown below.

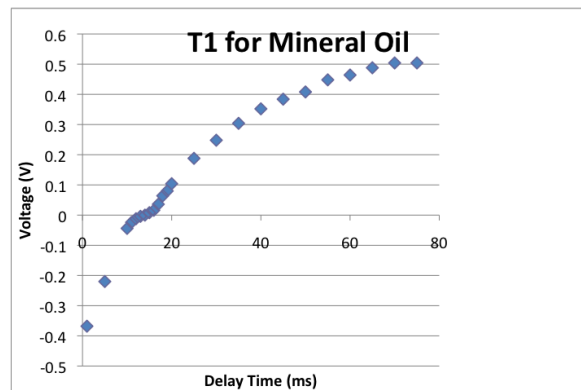
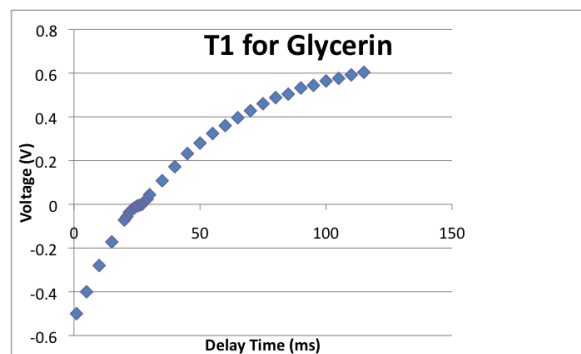


FIG. 3. Graph of voltage vs. delay time for mineral oil.

FIG. 4. Voltage vs delay time for glycerin. The inverse of the exponential constant gives T_1 .

To calculate T_1 , begin with Equation 4. At the point where the voltage on the oscilloscope goes to zero, we know that the net magnetization in \hat{z} is zero. We know this because when the $\pi/2$ pulse is given, no current is being produced in the pickup coils which implies that there is no net magnetization. If we look at it with the initial conditions that at $t=0$, $M_z=0$, then at the point where there is no voltage, M_z is actually $\frac{1}{2}M_0$. If we substitute this value into Equation 3 and solve for T_1 , we obtain

$$T_1 = \frac{t}{\ln 2} \quad (6)$$

where t is the delay time. For mineral oil, Voltage=0 at $t=14$ ms. Therefore, T_1 for mineral oil is 20 ms ± 1 ms. For glycerin, Voltage=0 at $t=27$ ms. Therefore, T_1 for glycerin is 40 ms ± 1 ms.

V. EXPERIMENT II

The goal of this experiment was to measure the spin-spin relaxation time (T_2) for mineral oil and glycerin. We measured the voltage of the echo at different delay times and plotted a graph of the points. The data is shown in the graphs below.

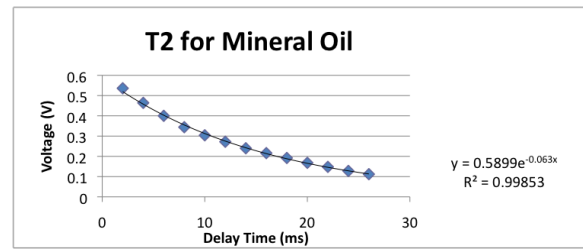


FIG. 5. The decay of net magnetization in the x-y plane indicative of spin-spin relaxation. The inverse of the exponential gives T_2 .

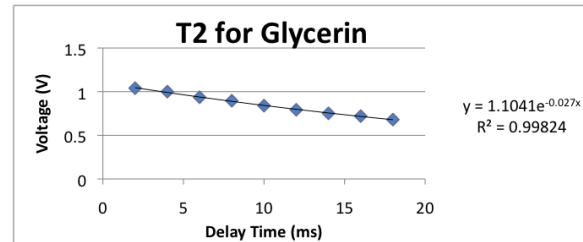


FIG. 6. The decay of net magnetization in the x-y plane. The inverse of the exponential gives T_2 .

T_2 is simply calculated by taking the inverse of the exponential constant of the trend line. For mineral oil, $T_2 = 15.9$ ms ± 1 ms. For glycerin, $T_2 = 37.1$ ms ± 1 ms.

Error is a result of several factors. The magnetic field produced by the magnet varied slightly with changing temperature. The resonant frequency increased an average of 10 Hz every ten minutes due to these fluctuations. Another source of error was reading the voltage of the peaks from the oscilloscope. Accuracy could be attained to about two orders of magnitude.

VI. CONCLUSION

For mineral oil, we obtained values of T_1 and T_2 of 20.0 ms and 15.9 ms respectively. For glycerin, we obtained values of T_1 and T_2 of 40.0 ms $\pm 5\%$ and 37.1 ms $\pm 5\%$ respectively.

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