

Time of Water

Studying the spin-lattice relaxation time of water sample
using Earth's Field Nuclear Magnetic Resonance

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Abstract

This paper deals with studying the magnetization of 125ml water sample in the external magnetic field, using the Earth's-field free precession technique. This technique employs the use of two perpendicular magnetic fields, namely Earth's Field and polarizing field produced by the solenoid. By studying the amplitude of the free induction decay with varied polarizing current or polarizing time it was possible to study spin-lattice relaxation time and Curie's law. The results of the study have shown that the spin-lattice relaxation time of water is 2.552 ± 0.0060 s which fits the predictions. In addition it was shown that the Curie's law for magnetization is satisfied

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1 Introduction

1.1 Magnetism

All the magnetic phenomena in the universe are due to the motion of charges. The magnetic field created by current-carrying wires is due to the moving charges, and the magnetic field produced by Earth is due to the convection of a liquid iron alloy in the outer core, which drives currents, resulting in the production of the magnetic field (Nigel 2002)³. However, the motion of charges is not apparent in the compass needles or refrigerator decorations. The magnetism of these objects also comes from the motion of the charges inside the atoms.

Each particle has a property called spin, denoted by \mathbf{S} . The spin is an internal angular momentum generated by a circulating flow of energy in the wave field of the particle (Ohanian 1984)⁴. The spin of a particle can either be an integer or a half-integer. This paper deals primarily with atoms, where protons, neutrons and electrons have $\mathbf{S} = 1/2$. Due to angular momentum, a charged particle acts as a magnet and has an associated dipole moment $\boldsymbol{\mu}$. Ordinarily, particles' spins are randomly aligned and cancel each other's magnetic fields out. However, once submerged in an external magnetic field \mathbf{B} , all the dipoles align, and the material becomes magnetically polarized. The total magnetization of the material is called \mathbf{M} . Depending on the structure of the material, the dipoles can either align parallel to field \mathbf{B} (these substances are called paramagnets) or opposite to field \mathbf{B} (diamagnets). Due to the random motion of the molecules (or atoms), most substances do not maintain the polarization after the external field is switched off. However, there are substances called ferromagnets that keep being magnetized after the field is turned off (magnets used in compass needles and refrigerator decorations are ferromagnets) (Griffiths, 2017, pp. 266-293)⁵. This paper focuses on studying the materials that lose magnetic properties once the external field \mathbf{B} is switched off.

1.2 Magnetization of the paramagnets

The motion of the charged particle produces loops of currents that give rise to magnetic moments. Classically, a loop of area A and current I produce magnetic moment

$$\boldsymbol{\mu} = I\mathbf{A}$$

However, for particles with spin \mathbf{S} , the magnetic moment is

$$\boldsymbol{\mu} = g \frac{q}{2m} \mathbf{S} \tag{1}$$

where g is the dimensionless gyromagnetic ratio, q and m are the charge and the mass of a particle, respectively (McIntyre, 2012, pp. 1-50)². However, it is impossible to determine the value of each magnetic dipole inside the substance. Therefore, it is common to be working with the total magnetization \mathbf{M} . This paper focuses on studying the magnetization of 125 ml sample of water, which has two hydrogen and one oxygen atom. ^8O has eight electrons in configuration $1s^2 2s^2 2p^4$, meaning it has 2 electrons in the valence shell, eight protons and eight neutrons. ^1H has 1 electron and 1 proton. Since two hydrogen atoms combine with a single oxygen atom, electrons of hydrogen fill in the electron configuration of oxygen, leaving no free electrons. Since the electron configuration is

complete, the net dipole moment of electrons is zero (due to the Pauli exclusion principle (McIntyre, 2012, pp. 410-443)²). The dipole moment of the oxygen nucleus (protons and neutrons) is zero due to the parity effect of interacting nucleons (since protons and neutrons have spin-1/2, the spins "cancel out")(Evans, 1955, pp. 140-177)⁶. Therefore, the only contribution to the magnetization comes from two hydrogen nuclei - protons.

To study the magnetization of water in external magnetic fields, consider what happens to the magnetic dipoles. Some of them align parallel to \mathbf{B} , call them "up," and some anti-parallel, call them "down" (Schroeder, 1999, pp. 74-110)¹. The energy of a dipole $\boldsymbol{\mu}$ in magnetic field \mathbf{B} is

$$U = -\boldsymbol{\mu} \cdot \mathbf{B} \quad (2)$$

Let the total number of particles in the system be N , so there are N_{\uparrow} particles in the "up" state and N_{\downarrow} in the "down" state. So the net energy of the system (from equation 2) is

$$U = \mu B(N_{\downarrow} - N_{\uparrow}) = \mu B(N - 2N_{\uparrow}) \quad (3)$$

The total magnetization is $M = \mu N$, which, from equation 3 becomes

$$M = \mu(N_{\uparrow} - N_{\downarrow}) = -\frac{U}{B} \quad (4)$$

Therefore, to find the magnetization of this system, its energy needs to be found first. Since each particle in the system can have only two states, the total number of possible "orientations" of N particles is

$$\Omega(N_{\uparrow}) = \binom{N}{N_{\uparrow}} = \frac{N!}{N_{\uparrow}!N_{\downarrow}!} \quad (5)$$

The energy of the system can be found from the entropy, which is given by $S = k \ln \Omega$ (k is Boltzmann's constant), or, using equation 5 and considering a large number of particles, entropy becomes

$$S/k \approx N \ln N - N_{\uparrow} \ln N_{\uparrow} - (N - N_{\uparrow}) \ln (N - N_{\uparrow}) \quad (6)$$

When entropy is differentiated with respect to energy, the result is the dependence of the inverse temperature (in Kelvin) on energy, magnetic field and number of particles. When that equation is rearranged for energy and divided by $-B$, it produces the relation between magnetization M , number of particles N , temperature T and magnetic field B :

$$M = N\mu \tanh\left(\frac{\mu B}{kT}\right) \quad (7)$$

If $\mu B \ll kT$, then equation 7 is approximated as

$$\boxed{M \approx \frac{N\mu^2 B}{kT}} \quad (8)$$

This result is known as Curie's law and shows that magnetization is proportional to the strength of the magnetic field and inversely proportional to temperature

1.3 Spin-Lattice relaxation time

Consider how magnetization changes with time as the magnetic field is turned on or off. Dipoles in molecules need time to rearrange themselves in the presence of the field \mathbf{B} . Curie's law (equation 7) does not account for this process and describes the final value. As shown in Figure 1, the magnetization of a system evolves exponentially with instantaneous change in magnetic field. Magnetization then can be expressed as

$$M(t) = M_0 \left(1 - e^{-\frac{t}{T_1}} \right) \quad (9)$$

where M_0 is the magnetization predicted by Curie's law (equation 7) and T_1 is the spin-lattice relaxation time, which is defined as the time it takes for the magnetization to reach 63% of M_0 . T_1 is a significant value in medicine. Since humans consist mainly of water, different tissues can be detected by observing the different relaxation times of various materials. As a result, scientists have learned how to scan and examine the internal organs of a human without surgery. The technique that produces this scan is called Magnetic Resonance Imaging (MRI) and operates by applying a strong magnetic field and detecting its spin-lattice relaxation time.

1.4 Experiment

As mentioned in Section 1.2, this paper focuses on studying the magnetization of water and its spin-lattice relaxation time. The study is conducted using the Earth's-field free precession technique. In this method, the net magnetic field is a vector sum of Earth's magnetic field \mathbf{B}_e and polarizing field \mathbf{B}_p , perpendicular to \mathbf{B}_e . The polarizing magnetic field is induced by current I_p in a coil and is much stronger than B_e , so the magnetic field in equation 8 is approximately \mathbf{B}_p . Therefore, once the polarizing field is activated, the dipoles of water start aligning with it, and magnetization changes according to equation 9. However, when B_p is switched off, dipoles do not randomly disarrange but start precessing around \mathbf{B}_e (similarly to a classical gyroscope) at Larmor frequency (Levitt, 2008 Ch 2.5)⁷, which is then detected as the Free Induction Decay (FID) signal. The FID signal shows the precessing frequency, and by studying the change in the amplitude of this signal with changing polarizing time t_p (time during which polarizing field is on), the spin-lattice relaxation time T_1 can be determined. It is expected that T_1 for water in room temperature with $I_p = 3A$ is approximately 2.5 seconds (TeachSpin, 2002)⁸. In addition, by changing the polarizing current I_p , Curie's law can be demonstrated.

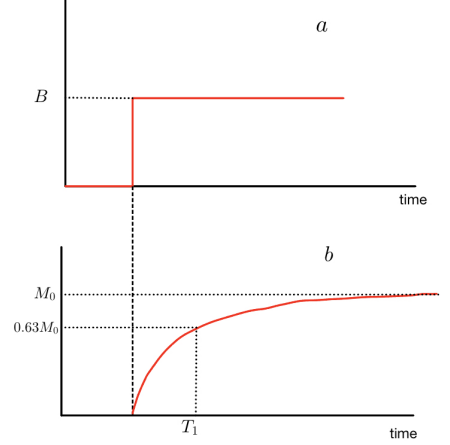


Figure 1: Magnetic field (a.) and magnetization (b.) vs time.

2 Method

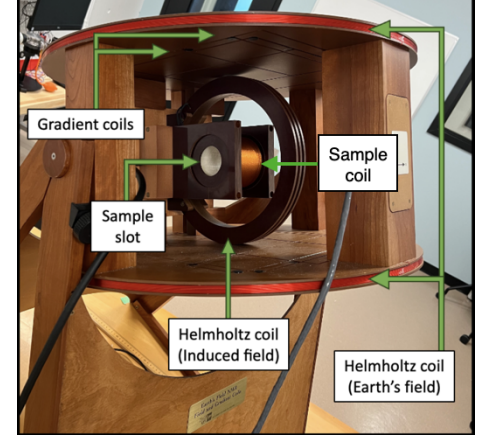
2.1 Apparatus

To use the Earth's-field free precession technique, TeachSpin's Earth's Field Nuclear Magnetic Resonance (EF NMR) apparatus was used. Figure 2a shows the positioning of the coils in the EF NMR apparatus.

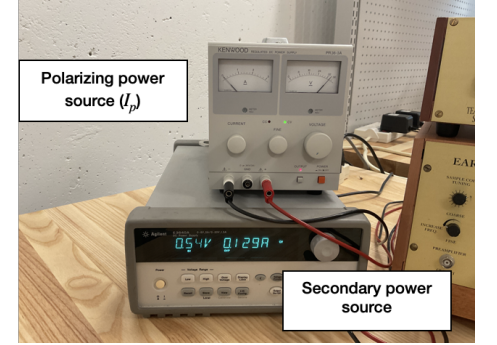
In this setup, the sample (water) is placed in the sample slot, surrounded by a solenoid called the sample coil. The polarizing power supply in Figure 2b supplies the sample coil with direct current I_p (can be varied from 0 A to 3 A), which creates a magnetic field B_p inside a sample slot. This magnetic field is proportional to the polarizing current since the magnetic field inside a solenoid is given by $B = \mu n I$, where μ is the permeability of a sample, n is the number of turns per unit length and I is the current in the coil. Note that $I_p \neq I$ because of the internal control circuit.

The Helmholtz and gradient coils create the magnetic "noise-compensating" fields. Since the experiment was conducted inside the building, the enforced concrete walls reduce the strength of Earth's magnetic field. Helmholtz coil (Earth's field) in Figure 2a compensates for that loss and produces a uniform magnetic field B_e perpendicular to B_p . The Helmholtz coil (induced field) and gradient coils cancel any external magnetic fields that come from wires, lamps, or any other circuit (it was observed that mobile phones create vast amounts of noise due to the internal LC circuit) and homogenize the field around the sample. The current is supplied to these coils by the secondary power supply in Figure 2b and controlled by the Gradient Coils controller (Figure 2c 1.).

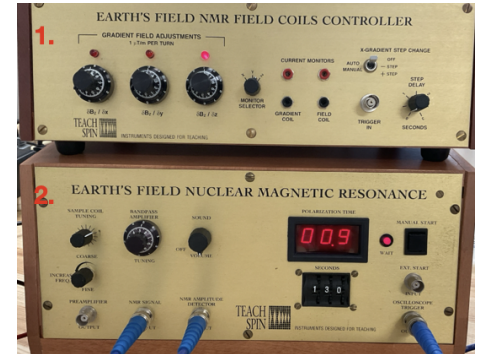
The gradient coil controller (Figure 2c 1.) has three knobs that control the magnetic field produced by the gradient coils. These three knobs adjust the strength of the magnetic field along the x, y and z axes which allow them to cancel out most of the noises. Meanwhile, the Timing and Control circuit (Figure 2c 2.) controls the apparatus and the detected signal by allowing the tuning of the sample coil. It has four knobs: Two knobs on the left of Figure 2c 2. are the coarse and fine adjusters of sample coil tuning; they control the capacitance of a variable capacitor in Figure 3. By tuning the sample coil to the correct value, one should achieve the



(a)



(b)



(c) 1.-Gradient Coils controller; 2.-Timing and Control Circuit.

Figure 2: EF NMR setup. 2a - Coils; 2b - power sources for coils; 2c - Coils control units.

resonant frequency of the LC circuit ($\omega_0 = (LC)^{-1/2}$) that matches the Larmor precession frequency of a sample. The knob on the right (closest to the digital screen) controls the volume of the speaker, which turns the detected precession frequency into sound (since Earth's magnetic field is weak, the precession frequency is in an audible spectrum). The last knob is the bandpass amplifier tuning that adjusts the frequency of the blocked high and low-frequency noise from the preamplifier (the preamplifier, bandpass amplifier, full-wave rectifier and the low pass filter together serve for amplitude detection and are schematically shown as a tuned amplifier in Figure 3). The Timing and Control circuit has three outputs: the preamplifier output, the NMR signal output and the NMR amplitude detector output. The output from the preamplifier is used to monitor the signal while adjusting the coarse and fine controls of the sample coil tuning. When the circuit is correctly tuned to the frequency of the free precession signal, the amplitude of this signal will be maximum. The NMR signal output is the output from the bandpass amplifier. This signal is used to observe the free precession signal. Lastly, the NMR amplitude detector outputs a signal from a full-wave rectifier followed by a low-pass filter. This signal is essentially a free induction decay signal, and the conducted experiment focused on studying its amplitude.

Figure 3 shows the simplified block diagram of the operation of NMR. Timing and Control Circuit (Figure 2c 2.) activates and controls the apparatus. Before the start of the procedure, the polarizing time t_p is adjusted using the thumbwheel switches (below the digital screen in Figure 2c 2.). After the "Manual Start" button is pressed, the relay connects the polarization power supply (Figure 2a) with the sample coil and maintains this position for time t_p . After that, the control circuit triggers the oscilloscope and dictates the relay to connect the sample coil to the sample coil tuning (variable capacitor) and the amplifier, leading to the oscilloscope input.

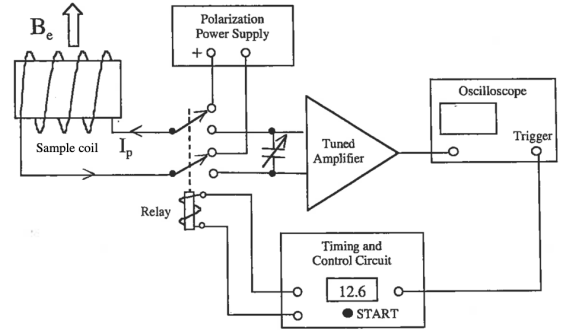


Figure 3: Simplified block diagram of the apparatus.

2.2 Procedure

The sample coil and the gradient field needed to be tuned to give a maximum amplitude with minimum pickup noise to obtain the most accurate results. This was achieved by positioning all the knobs in the gradient coils controller and timing and control circuit to their middle values. To observe the signal, the preamplifier output was monitored in the oscilloscope (yellow signal in Figure 4a). With each working period of the NMR apparatus, only one knob could be adjusted for a maximum output signal. As a result of this process, it was determined that the best settings of the gradient field are $\partial B_z/\partial x = 5.95$, $\partial B_z/\partial y = 0.5$, $\partial B_z/\partial z = 1.9$ with the secondary power supply maintained at 0.54 V. The best settings for the sample coil tuning are two positions clockwise in the coarse adjuster and five counterclockwise in the fine adjuster. In the end, it was determined that the optimal value of the bandpass amplifier was 5.3.

The two experiments were conducted to determine the spin-lattice relaxation time and demonstrate Curie's law. In the first experiment, the polarizing current was maintained at 3 A, while the measurements were conducted using the polarizing time t_p 1s, 3s, 5s, 8s, 10s and 13s. For each of these times, the experiment was repeated three times (for accuracy), and the amplitude of the FID signal was measured (yellow signal in Figure 4b). To demonstrate Curie's law, the polarizing time was kept constant at 13s while the polarizing current was changed. The experiment was run five times for each of the values of the polarizing current: 3A, 2.5A, 2.2A, 2.0A, 1.5A, 1A. By detecting the amplitude of the FID signal, it was possible to demonstrate the linear dependence between the polarizing current and magnetization.

3 Results

3.1 Determining spin-lattice relaxation time

Figure 5 shows the measurements of the amplitude of the FID signal while the polarizing time is being varied (blue dots) and the exponential fit of this data. Note that the obtained points follow closely to the fit, demonstrating the correctness of equation 9. Moreover, it was determined that for $I_p = 3.0A$ and room temperature $T \approx 293K$, the magnetization, predicted by Curie's law was $M_0 = 1.740 \pm 0.010V$ where the uncertainty is just 0.5% and the spin-lattice relaxation time was $T_1 = 2.552 \pm 0.060$ s, where the uncertainty is 2.5%.

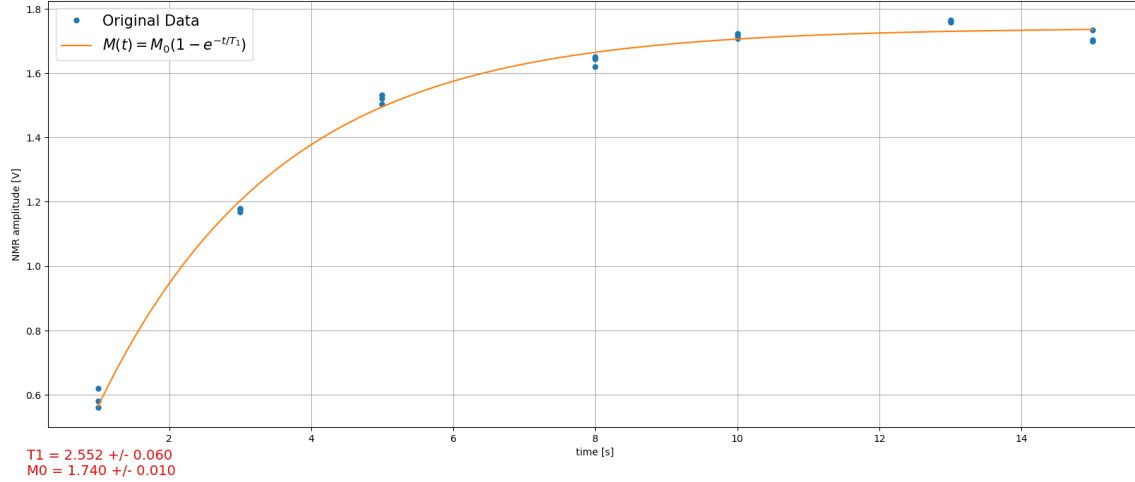


Figure 5: NMR amplitude vs t_p . Data points (blue) and the exponential fit (orange)

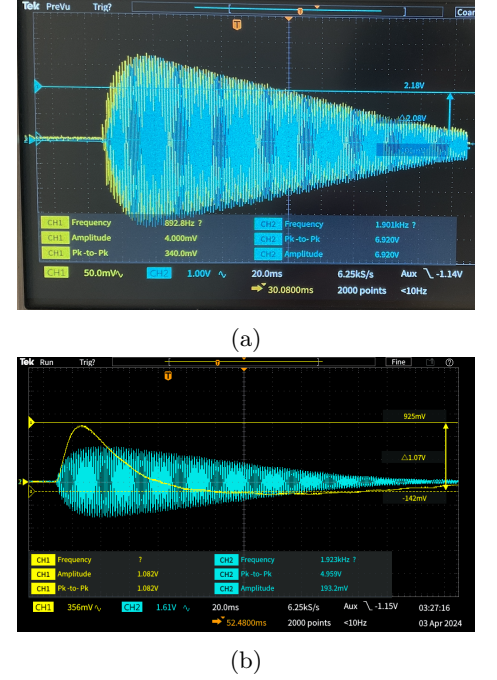


Figure 4: Signals in the oscilloscope for properly tuned and adjusted coils. In 4a yellow - pre-amplifier output, blue - NMR signal output; In 4b yellow - NMR amplitude detector output (FID), blue - NMR signal output

3.2 Demonstrating Curie's law

In the second experiment, the polarizing current was varied while the polarizing time was kept at 13.0s. The measurements of the amplitude of the FID signal obtained in this experiment, together with the linear and hyperbolic tangent fits, are shown in Figure 6. The errors in data were calculated by averaging the data points at a given polarization current. Figure 6 demonstrates how closely Curie's law (equation 8) follows the "true" magnetization law (equation 7), demonstrating its validity.

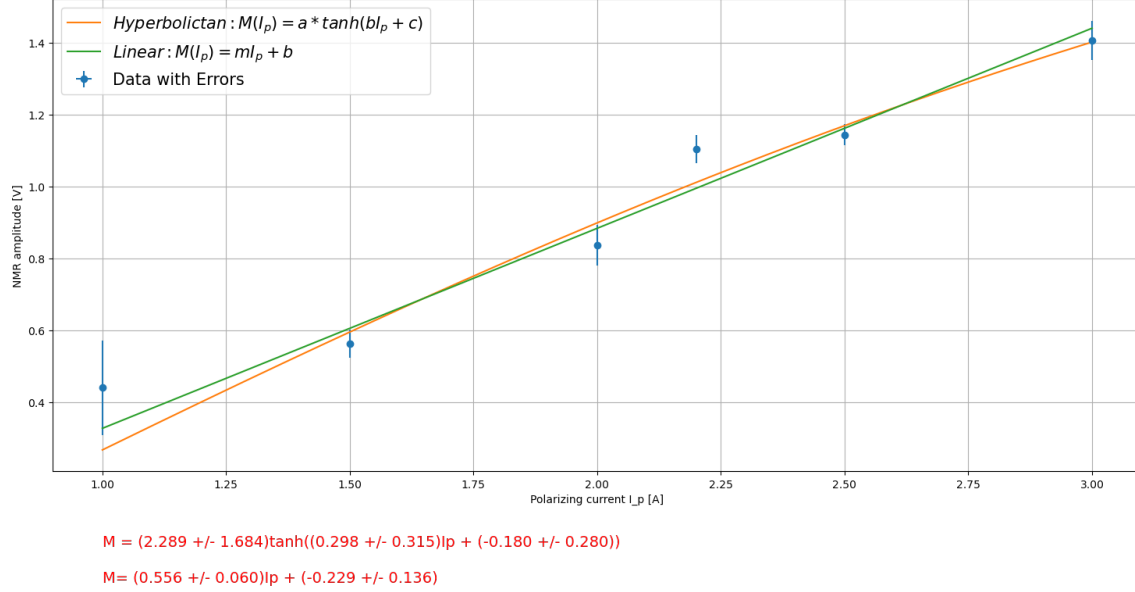


Figure 6: NMR amplitude vs I_p . Data points (blue), linear fit (green) and hyperbolic tangent fit (orange)

4 Discussion

The results for spin-lattice relaxation time demonstrate how closely the data fit with the predicted law (equation 9). Since it follows so closely, it was possible to determine the spin-lattice relaxation time with only 2.5% uncertainty. Note that the predicted value of spin-lattice relaxation time ($\approx 2.5s$) is within the uncertainty of the obtained result $T_1 = 2.552s$. Moreover, it was possible to determine the "terminal" magnetization M_0 that is predicted by Curie's law just with 0.5% uncertainty. The uncertainties' main reasons are the thermal and external magnetic noise. Thermal noise comes from the changing temperature of the sample coil. Since the measurements were repeated multiple times, the coil did not have enough time to cool down, so it was heating the water sample.

Equation 8 dictates that the magnetization value is inversely proportional to the temperature of the sample. This implies that equation 9 also becomes temperature-dependent and M_0 slightly

changes in the process of measurement. However, note that the uncertainties for magnetization and T_1 are minuscule and insignificant. The explanation is that for the thermal noise to become significant, the temperature should drastically increase, and for that to happen, the number of measurements should be much larger than used in this experiment. On the other hand, the magnetic noise comes from the electronics surrounding the apparatus. Most noises are cancelled by the correct adjustment of the gradient coils, but the slightest change in external noise requires readjustments. During this experiment, the gradient coils were tuned to the correct values, and the noise fields had minimal changes. Therefore, the uncertainties in the measurements come mainly from the thermal noise. Since most of the noises are due to the heating of coils, the obtained results can be further improved by waiting some time between the measurements to allow the sample coil to cool down. Another way to improve it without needing extra time is to use some cooling. Cooling with fans might have drawbacks due to the electric motors inside them, but this effect might be corrected by properly adjusting the gradient coils.

The results from the experiment with varying polarizing currents demonstrate how closely Curie's law fits with the theoretical equation 7 in the limit when $\mu B \ll kT$. Note that equations 7 and 8 do not explicitly depend on current. However, as mentioned in section 2.1, the strength of the magnetic field B_p is directly proportional to the polarizing current I_p , so one can rewrite Curie's law as

$$M \approx C_1 \frac{N\mu^2 I_p}{kT} + C_2 \quad (10)$$

where C_1 and C_2 are proportionality constants. In this experiment, it was found that $C_1 N\mu^2/kT = 0.556 \pm 0.060$ and $C_2 = -0.299 \pm 0.136$. This result closely fits with the hyperbolic tangent fit, demonstrating the accuracy of 8. However, the uncertainty for C_1 is 10.8% and the uncertainty for C_2 is 45.5%. These uncertainties come from relatively large errors in the data in Figure 6 and the fact that the points do not perfectly fit the straight line. These errors come partially from the thermal noise and the oscilloscope precision. This experiment was conducted after tuning, implying that the coils were already warm. Repeated magnetization measurements with varying currents further increased the temperature, resulting in large uncertainties around the data points. Moreover, note from Figure 6 that the largest uncertainty is around the point with the lowest polarization current. The amplitude of the FID signal is smallest at that point, meaning that the adjustments of the precision of the oscilloscope used were not perfect and could not properly detect low amplitude signals. To improve the uncertainties caused by the thermal noises, one could give some time for the coils to cool or use the cooling system. To account for the uncertainties caused by the oscilloscope, it needs to be properly adjusted to detect low-amplitude signals.

5 Conclusion

This paper focused on studying the magnetization of water in the external magnetic field. To do this the Earth's-field free precession technique was used. In this technique the net magnetic field is a vector sum of Earth's magnetic field \mathbf{B}_e and the polarizing magnetic field \mathbf{B}_p , produced in a coil by the polarizing current I_p , which is much stronger than Earth's field. This technique works by detecting the precession of molecules about \mathbf{B}_e when the polarizing field is turned off. The magnetization of the system depends on the polarizing time t_p (i.e., the time during which the polarizing field is on) and was shown to change with time according to the exponential equation 9 (Figure 5). The time constant in this equation is called the spin-lattice relaxation time which is defined as the time it takes for the magnetization to reach 63% of its terminal value. By measuring the amplitude of the free precession signal while changing the polarizing time and keeping the polarizing current constant at 3.0A, the spin-lattice relaxation time for water at the room temperature was found to be 2.552 ± 0.0060 s which fits the expected value of 2.5s. This result is essential for the Magnetic Resonance Imaging of living tissues, since the living organisms mainly consist of water and by distinguishing the spin-lattice relaxation time of different tissues it is possible to scan the internal organs without doing any surgery.

The terminal magnetization of water in the external magnetic field was shown to follow Curie's law (equation 8). The study was conducted with varying polarizing current and the constant polarizing time maintained at 13.0s, which ensures that the magnetization reaches 99.5% of the terminal magnetization as predicted by equation 9 and T_1 found in the first experiment. As a result, it was shown that magnetization linearly depends on I_p and, since the magnetic field produced by the coil is proportional to the current, it follows that magnetization linearly depends on the strength of the polarizing magnetic field, as predicted by Curie's law. However, the uncertainties in the fitting values in Figure 6 are large. It was discussed that the primary sources of these uncertainties are the increase in temperature of the solenoid, due to the repeated measurements, and the improper tuning of the oscilloscope which resulted in large uncertainties near the points corresponding to low currents. Nonetheless, the results fit with the prediction of Curie's law. This result is also sufficient, as it allows for good approximations of magnetization of water in relatively weak magnetic fields.

In future, the experiment can be modified by obtaining the values with less uncertainties by controlling the temperature of the coils. In addition the dependence between the temperature of water and its magnetization can be established by conducting the above-described experiments with different temperatures of water.

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