Frequency Sweep NMR Calibration Using Polarized Water Cells

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from the College of William and Mary
by
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Abstract

The College of William and Mary is able to produce polarized ^{3}He target cells and test their polarization using a nuclear magnetic resonance system (NMR). These polarized cells can then be used by scientists at the Thomas Jefferson National Accelerator Facility to study the spin properties of the neutron. For an upcoming experiment, a NMR system is being constructed inside a magnetized iron box at Jefferson Lab in order to cancel out any external magnetic fields. There are two types of NMR used to study the cells, frequency sweep and field sweep. Frequency sweep NMR involves producing a constant magnetic field in the system and varying the frequency, while field sweep holds the frequency constant and varies the magnetic field. Traditionally field sweep NMR can be used to analyze the target cell, but due to constraints put on us by the magnetized iron box, frequency sweep NMR must be used instead. The problem is that the background present in the system is dependent on frequency. As a result, as the frequency is varied the background changes and cannot be subtracted by the system's instrumentation. This background does not affect the data when running field sweep NMR on a polarized ^{3}He cell because the ^{3}He signal is significantly larger than the background. But, in order to measure the absolute polarization of the ^{3}He cell, the system must first be calibrated with a water cell whose polarization is well known. The problem is that the signal produced by the water cell is much smaller than the background and as a result is buried in it. The magnitude of the background is on the order of milivolts, while the magnitude of the water signal is on the order of microvolts. The goal of my senior project is to reduce the background picked up by the NMR system so that it does not dominate the signal produced by a water cell, enabling us to calibrate the system.

Acknowledgements

I would like to thank Dr. Todd Averett, my research advisor, for intensifying my fascination with physics as both an advisor and a teacher. Without his help this research project would not have been possible. I would also like to thank Aidan Kelleher. His help explaining the workings of the lab and assistance getting me started on my research were invaluable.

1 Introduction

Scientists at the Thomas Jefferson National Accelerator Facility are currently conducting electron scattering experiments to study the spin properties of the neutron. In order to conduct these experiments a target cell must first be filled with neutrons. Unfortunately, no practical free neutron target has yet to be produced because free neutrons radioactively decay to protons. However, it is possible to create a cell that demonstrates the properties of a free neutron target. Helium-3 (^{3}He) has been found to behave much like a single neutron when polarized inside a target cell, and serves as a satisfactory substitute for a free neutron target. ^{3}He's nucleus contains two protons and one neutron. When polarized, the spins of the protons will anti-align 90% of the time and essentially cancel each other out, leaving just the spin of the neutron aligned with the spin of the nucleus.

A nuclear magnetic resonance system (NMR) can be used to measure the polarization of the ^{3}He using a process known as Adiabatic Fast Passage (AFP), which will be discussed later. All subatomic particles have a resonant frequency known as the Larmor Frequency. By inducing a frequency equivalent to the Larmor Frequency it is possible to cause the spin of the particles to flip and induce an electromotive force (EMF). One problem with doing any NMR sweep is the frequency dependent background in the system that must be accounted for. In NMR experiments previously conducted in the lab and at Jefferson Lab the magnetic field of the system was varied during the NMR sweep, a process known as field sweep NMR. Since the frequency of the RF is constant during this type of sweep, the background is also constant and can easily be measured and subtracted by the system's instruments. This has worked extremely well in the past, but a new NMR system that is being constructed at Jefferson Lab requires the use of a different type of sweep, frequency sweep NMR. Frequency sweep NMR varies the frequency instead of the magnetic field, ramping the frequency up through the system's resonant frequency and then back down past it. Due to the constraints imposed by an upcoming project at Jefferson Lab, the new NMR system is being constructed inside an iron box that will effectively cancel all external magnetic fields. The box is also magnetized to produce a uniform target field inside. But, because of eddy currents and the hysteresis effect of iron, the magnetic field inside the box cannot be swept precisely enough for NMR. As a result this system may only use frequency sweep NMR experiments, which keep the magnetic field constant. This presents a problem because the background of the system is dependent on the frequency. Ramping the frequency causes the background to change, and since the background is not constant it cannot be accounted for by the system's instrumentation. The goal of this year's research was to find a way to reduce the amount of frequency dependent background picked up by the NMR system so that frequency sweep NMR may be used inside the iron box being constructed at Jefferson Lab.

2 The Target Cell

The target cells used in our lab and at Jefferson Lab are made of aluminosilicate glass whose surface is free of microfissures and paramagnetic impurities. Depolarization can occur when nuclei interact with the cell wall, and the surface characteristics of this glass minimize this depolarization.

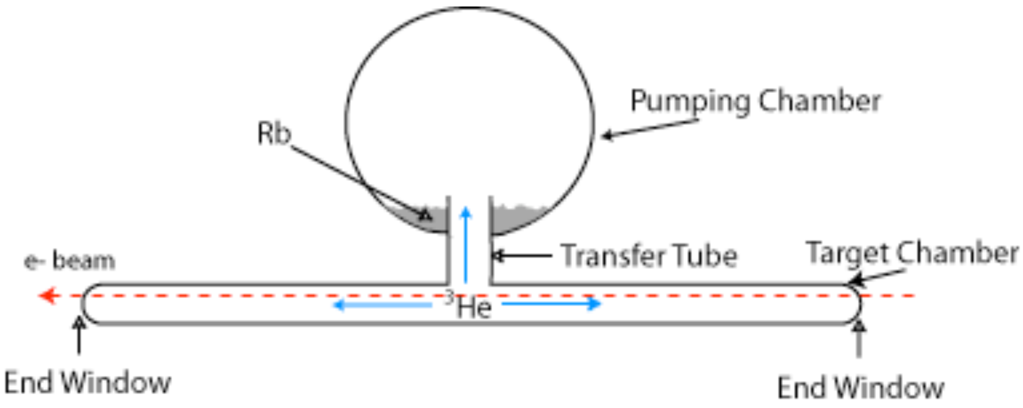

Figure 1: Schematic of a ^{3}He target cell. The electron beam passes through the end window of the target chamber, collides with he ^{3}He nuclei, and exits out the other end window. The pumping chamber contains a small amount of rubidium to be used in the optical pumping process. The transfer tube allows ^{3}He to travel between the pumping chamber and the target chamber.

The cell is comprised of two main components: the pumping chamber and the target chamber (see Figure 1). The pumping chamber is the upper, spherical chamber of the cell and contains a small amount of an alkali metal, typically rubidium. When the cell is placed in the NMR system, the pumping chamber sits inside an oven that heats the cell. Vaporizing the alkali metal makes it available for use in the optical pumping process, which will be discussed in the next section. The target chamber is the lower, rod like chamber of the cell. The electron beam enters the target chamber through one of its end windows and then begins interacting with the ^{3}He inside the target chamber. The chamber must be made extremely long to maximize the interaction between the electron beam and the ^{3}He. This part of the cell remains outside the oven and is positioned between two pickup coils which measure the induced EMF signal generated by the ^{3}He nuclei's spin procession (see Figure 2) [1].

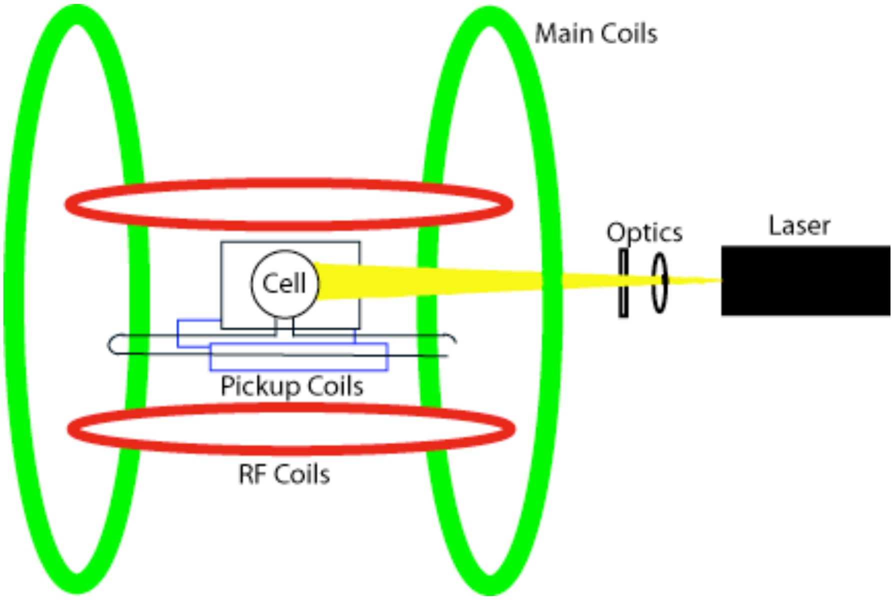

Figure 2: Schematic of the NMR system. This system is used to polarize the ^{3}He and then to perform and record NMR measurements. The green coils are the Helmholtz (main) coils that generate a uniform holding field around the system. The red coils are the RF coils, which generate an oscillating magnetic field (H_1). The blue coils are the pickup coils that measure induced EMF. The cell is placed inside the system and the electron beam enters it through one of the end windows of the target chamber.

3 Polarization

Polarization (P) is defined as:

$$P = \frac{N\uparrow - N\downarrow}{N\uparrow + N\downarrow} \tag{1}$$

where N↑ is the number of spins aligned and N↓ is the number of spins anti-aligned with the magnetic holding field.

Before either a field or frequency sweep NMR experiment may be run on a target cell, the cell's ^{3}He must first be polarized. This is done through a two-step process that involves first optically pumping the alkali metal (Rb), and then the rubidium's spin exchange with the ^{3}He. For this process to occur the target cell is placed inside the NMR system and will remain there for the duration of the experiments conducted on it.

3.1 Optical Pumping

In order to polarize the ^{3}He in the target cell, the alkali metal (Rb) must first be optically pumped so that it may exchange its spin with the ^{3}He. In the NMR system a magnetic holding field (H_o) produced by the Helmholtz (main) Coils shown in Figure 2 is used to separate and distinguish the otherwise spin-degenerate energy states [2]. This causes Zeeman splitting in the atomic spin states of Rb, splitting both the ground state, $5S_{1/2}$, and the first excited state, $5P_{1/2}$, into the m=+1/2 and m=-1/2 states. A diode laser then emits a 795nm beam of circularly polarized light, exciting the electrons from the $5S_{1/2}$ m=-1/2 state to the $5P_{1/2}$ m=+1/2 (see Figure 3) [1]. From the $5P_{1/2}$ m=+1/2 state the electrons then decay back to the ground state with equal probability of decaying to the m=+1/2 and m=-1/2 states. The electrons that decay to the m=+1/2 state can remain in this state or decay back to m=-1/2. All the electrons that decay back to the m=-1/2 state can be optically pumped again, and with continuous pumping a very high percentage of electrons wind up in the $5S_{1/2}$ m=+1/2 state [3]. Since the optical pumping process generates a high percentage of rubidium electrons with the same spin direction, the rubidium is considered polarized.

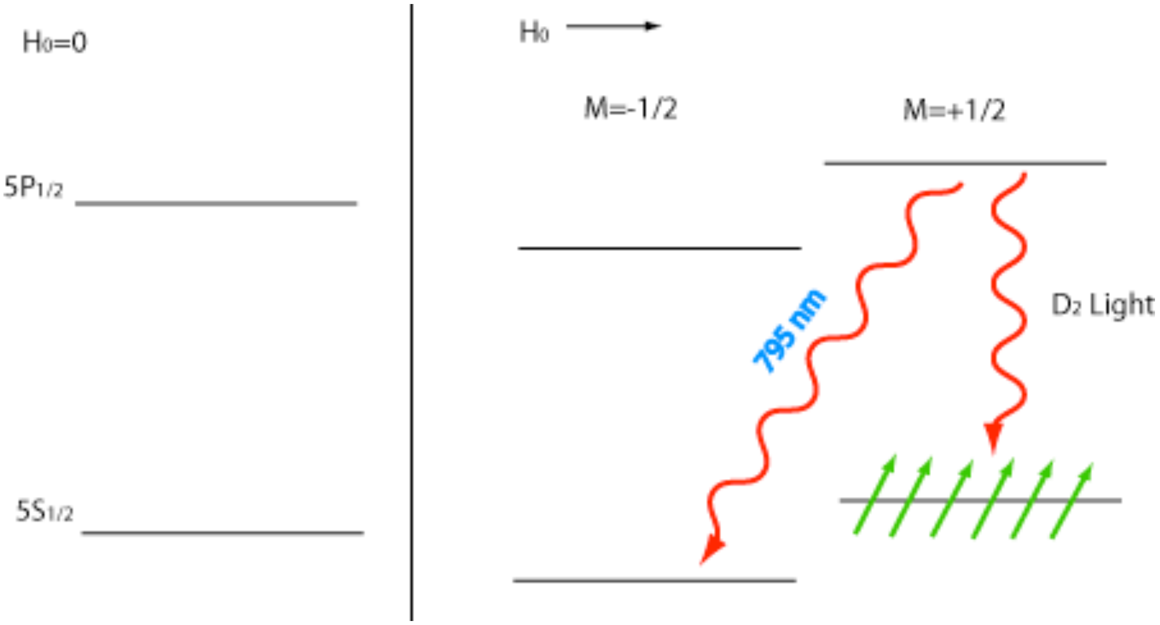

Figure 3: Optical Pumping. A magnetic holding field (H_o) causes Zeeman splitting in the atomic spin states of Rubidium, splitting both the ground state, $5S_{1/2}$, and the first excited state, $5P_{1/2}$, into the m=+1/2 and m=-1/2 states. A diode laser then emits a 795nm beam of circularly polarized light exciting the electrons from the $5S_{1/2}$ m=-1/2 state to the $5P_{1/2}$ m=+1/2. From the $5P_{1/2}$ m=+1/2 state the electrons then decay back to the ground state with equal probability of decaying to the m=+1/2 and m=-1/2 states.

3.2 Spin Exchange

Once polarized, the rubidium can exchange its spin with the ^{3}He in the cell, polarizing the ^{3}He nuclei. This spin exchange is possible because of hyperfine-like interactions between the Rb and ^{3}He that occur during binary collisions between the two [4]. Because Rb has been optically pumped to the m=+1/2 state, it may only transfer an angular momentum of +1 to ^{3}He in the m=-1/2 state. This polarizes the ^{3}He nucleus, leaving it in the m=+1/2 state and the Rb in the m=-1/2 state. The Rb is now ready to be optically pumped again [4]. The maximum polarization yielded by this process is approximately 45%, and is dependent on the spin exchange rate between Rb and ^{3}He, the average polarization of Rb, and the depolarization rate of the target cell's ^{3}He.

4. Polarization Theory

Nuclear magnetic resonance is used to measure the polarization of the ^{3}He target cell during its polarization using a process known as adiabatic fast passage (AFP) [5]. The NMR system, shown in Figure 4, consists of the Helmholtz (Main) coils, the RF coils, pickup coils, electronics, and computer programs designed to execute tests and record data.

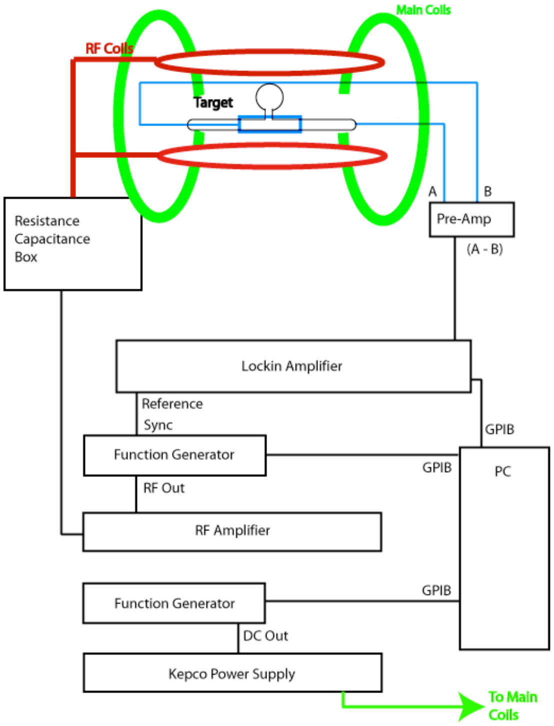

Figure 4: Schematic of the NMR system's electronics, which are responsible for running NMR experiments and collecting data from them.

4.1 Adiabatic Fast Passage

At the beginning of the adiabatic fast passage (AFP) process, the spin of the ^{3}He nucleus is anti-aligned with the main holding field created by the Helmholtz coils [2]. An RF field is then applied orthogonal to the main holding field by the RF coils. At this point the resonant frequency, or field of the system, is determined using the equation:

$$\nu = \frac{2\mu B}{h} \tag{2}$$

where h is Planck's constant, v is the frequency, μ is the magnetic moment of ^{3}He, and B is the magnetic field. In our system, the field generated is 28 Gauss, which results in a frequency of approximately 91 kHz. If a field sweep NMR measurement is being taken, then it is necessary to choose a resonant frequency whose magnetic field is in the middle of the sweep range, and if a frequency sweep measurement is being taken, it is necessary to chose a resonant field whose frequency is in the middle of the frequency range to be swept.

In either case, when the sweep is run it causes a passage through the resonance of the nuclei known as adiabatic fast passage [6]. This acts to reverse the magnetization direction of the ^{3}He nucleus, causing the spins of the nucleus to flip. The sweep then ramps back down to its starting point, restoring the magnetization to its original direction and once again causing the neutron spins to flip. As the spins of the neutrons process a time dependent magnetic flux is generated inside the pickup coils, which lie parallel to the cell's target chamber [7]. This magnetic flux then induces an electromotive force (EMF) in the pickup coils which can then be read by the system's instrumentation.

5. Experimental Setup

Figure 4 shows all the components of the NMR system and the electronics that control them. The Helmholtz (main) coils generate a uniform holding field around the system, and are controlled by a KEPCO Bipolar Operational Power Supply. This power supply is controlled by an Agilent 33120A Function Generator, which is operated via a computer program. The function generator is used to ramp the current if field sweep

NMR is being conducted, or output a DC current for field sweep NMR. The RF coils are used to generate a 100mG oscillating magnetic field (H_1), which is generated with frequencies ranging from 80 to 100 kHz. This field is generated using a function generator that produces a sinusoidally oscillating voltage, the RF voltage. An RF amplifier then amplifies the function generator's signal before it is sent to the RF coils. The amplifier is controlled by a Hewlett Packard 3324A Synthesized Function/Sweep Generator that is programmed by the lab's computer to either generate a constant frequency for field sweep NMR, or vary it for frequency sweep NMR. When the ^{3}He nuclei's spin proceeds, an EMF signal is induced in the pickup coils. The pickup coils are connected to a Stanford Research Systems SR560 Preamplifier, which amplifies the signal and sends it to a Stanford Research Systems SR 844 RF Lock-In Amplifier. The lock-in amp is controlled by the computer, which also collects data during measurements. The lock-in amp serves to isolate the frequency specified by the RF function generator and measures the amplified voltage in the pickup coils of the signal with this frequency.

6 The Problem

The frequency dependent background present is caused by the system's frequency response. When taking a field sweep NMR measurement, the frequency remains constant, and therefore the frequency dependent background also remains constant. As a result the instrumentation can subtract out the background so that it does not affect the signal. However, when taking a frequency sweep NMR measurement, the frequency dependent background varies as the frequency is swept, making it impossible for the system to subtract it out. If the signal is significantly larger than the background, then the background can be considered negligible. But, if the background is large the signal can become buried in the background.

The frequency sweep NMR of a polarized ^{3}He cell generates a signal (~100mV at preamp gain=1) that is large enough to be unaffected by the background (2-20mV or less), but doing frequency sweep NMR on a ^{3}He cell only yields the cell's relative polarization. Therefore, it is essential to achieve absolute polarization of the cell, which means the entire system must first be calibrated. This can be done by using a cell filled with water instead of ^{3}He, because the polarization of water is well known and can easily be used to calibrate the cell. The only problem with this is that the signal it produces (~10μV at gain=100x) is significantly smaller than the background (2-20mV) in the system. As a result, the water signal is lost in the background and the system cannot currently be calibrated.

7 Pickup Coil Theory

The NMR system's pickup coils along with the wires that connect them to the NMR preamplifier form an LRC circuit. As is true for any LRC circuit, the pickup coils have a resonant frequency (ω_o), which generates a natural ringing within the system.

$$\omega_o = \frac{1}{\sqrt{LC}} \tag{3}$$

Figure 5 is a quality curve (Q-curve) of the old set of pickup coils' frequency response that were originally used in the NMR system. This graph represents the induced EMF picked up by the pickup coils when a frequency sweep is run. The old coils reach resonance around 190 kHz, which is not the same as that of the system, but the ringing generated by the coil's resonance is large enough to mask the small signal produced by the water cell.

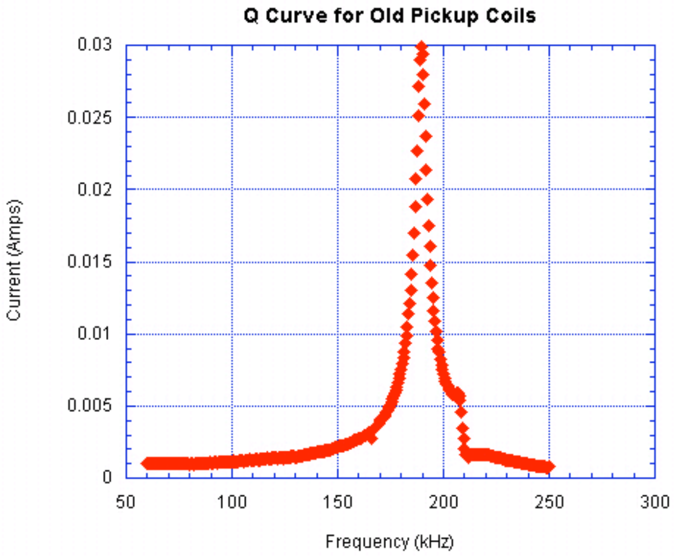

Figure 5: Graph of the quality curve for the old pickup coils. Note that resonance occurs at approximately 190 kHz, and that the large resonance response of the pickup coils is masking the small water signal in the 91 kHz region.

7.1 Fixing the Faults of the Previous Pickup Coil

The previous set of pickup coils that were in the NMR system had wire wound in a groove within the pickup coil (see Figure 6). The problem with this design was that the coils themselves could not be put directly next to the cell. Even when the pickup coils were put directly next to the target cell, the coils of wire themselves were still 1.2 mm away. When a NMR experiment was run, a changing magnetic flux was created in the target cell, which induced an EMF signal in the pickup coils. This signal acted as a magnetic dipole and accordingly its signal strength fell off as $1/r^3$, where r is the distance from the source. Since the closest the coils could get to the cell was 1.2 mm away, signal strength was lost. This was especially problematic when considering the water signal the pickup coils were looking for is on the order of 10μV. This signal loss was remedied by constructing pickup coils with a removable faceplate that when attached to the coil, formed the groove in which the wire could be wound. Once the wire was wound, epoxy was applied to the windings so that they would remain in place and then the faceplate was removed. This allowed the coils to be placed directly next to the target cell.

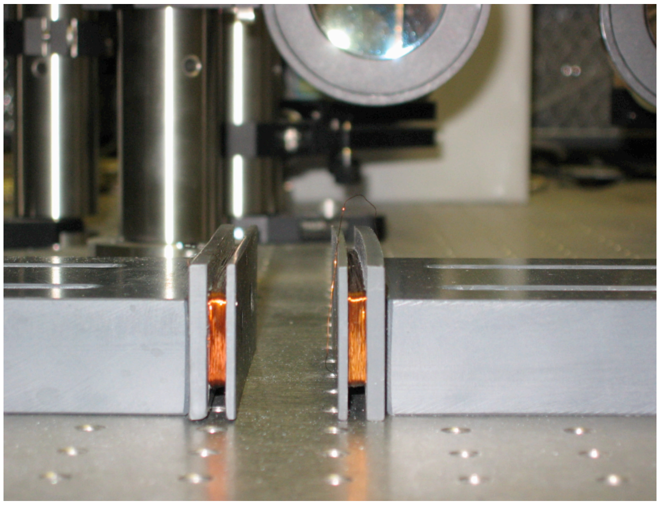

Figure 6: Side profile of the old pickup coils. Note that the wire is wound in a groove that limits the coil from being closer than 1.2 mm away from the target cell.

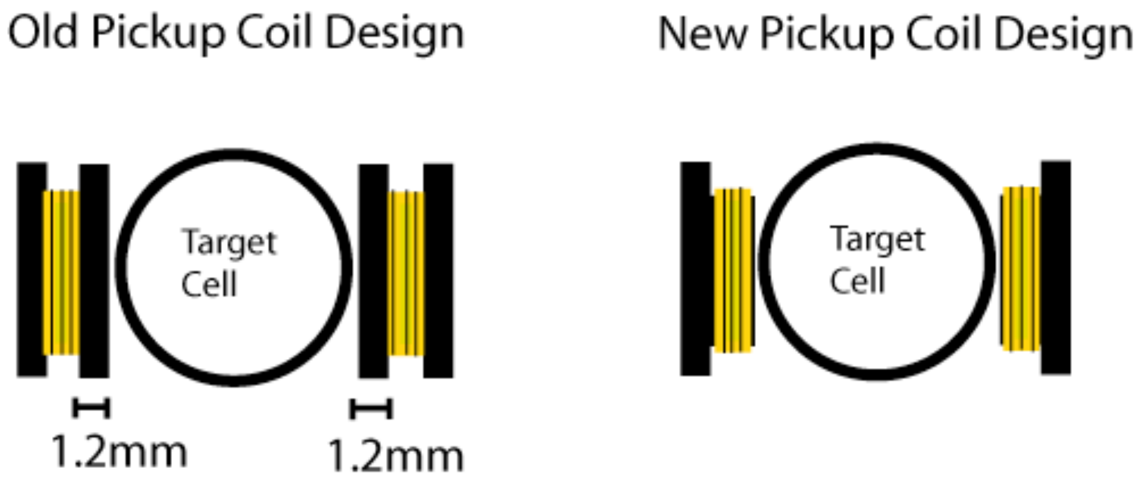

Figure 7: Schematic diagram of the old coil's proximity to the target cell verses the new coil's proximity to the target cell.

Another problem with the previous set of pickup coils was the way in which the coils connected to the NMR preamplifier. Soldering plugs were epoxied to the pickup coils and then the wire from the coils and twisted pair wire were soldered together on the plugs. Unfortunately, the soldering plugs often came loose and detached from the pickup coils. In addition, every time the pickup coils were removed from the system the wire had to be unsoldered from the plugs. This put a tremendous amount of stress on the coils, repeatedly resulting in a broken wire. When this occurred, a turn of wire would often have to be removed from the coil so that enough wire was present to reestablish the connection, and then the two coils needed to be impedance matched once again. This problem was remedied by attaching the coils to a male BNC terminal, which was mounted on the pickup coil itself. A BNC terminal is a type of terminal used to connect coaxial cables to electronic devices. This allowed the pickup coils to be connected to the NMR preamplifier by simply connecting a BNC cable to them, eliminating the risk of breaking the wire. This was especially important since the coils were epoxied in place and turns of wire could not be removed if a wire broke.

Additionally, the previous set of pickup coils in the system had only 100 turns of wire on each of them. With only 100 turns on each coil, there was not enough EMF induced in the pickup coils to read the relatively small water signal. If the inductance of the coils is increased, the EMF induced in the coils would also increase. This was accomplished by adding more turns of wire to the coils to increase their inductance. But, the resonant frequency (ω_o) of the coils is inversely proportional to inductance (Equation 3), so as inductance is increased the resonant frequency of the coils decreases, bringing it closer to 91kHz. As a result it was also necessary to decrease the capacitance of the pickup coils by wiring additional capacitors in series with the coils to again move the resonant frequency away from the 91 kHz range.

7.2 New Pickup Coil Design

Two new sets of pickup coils were fabricated in an attempt to reveal a water signal. The first was a set of dual coils that contained two coils of wire on each pickup coil. Each of these coils had 100 turns of wire on them, creating pickup coils that had a total of 200 turns of wire on each. The second set of coils was designed with only one coil of 250 turns on each pickup coil. Both sets of coils were constructed with the shortcomings of the old pickup coils discussed above in mind.

8 Dual Coils

The dual coils (Figure 8) were designed with the goal of eliminating background from the system. The two coils of wire on each of the pickup coils were wound so that the signals they picked up were 180 degrees out of phase. For signals far away from the system, *i.e.* the background we were attempting to eliminate, the two coils were effectively at the same distance away and therefore canceled one another. But, when extremely close to the coils, *i.e.* the signal from the target cell, the EMF generated by the two coils of wire were no longer equal and produced a non-zero result. This configuration resulted in a signal that was actually smaller than past signals, but the background was also smaller and by a larger degree. As a result the signal to noise ratio of these coils was much higher than the old coils. This is extremely important because the water signal will only be revealed when the signal to noise ratio of the pickup coils can be increased enough that the background no longer masks the signal.

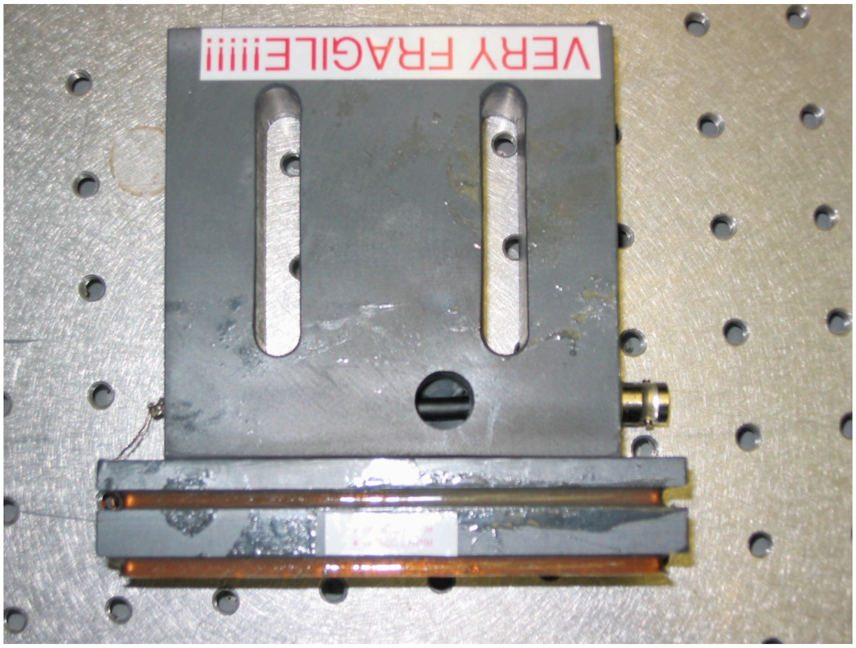

Figure 8: Top view of one of the dual pickup coils. Notice the two coils of wire epoxied in place and the male BNC terminal.

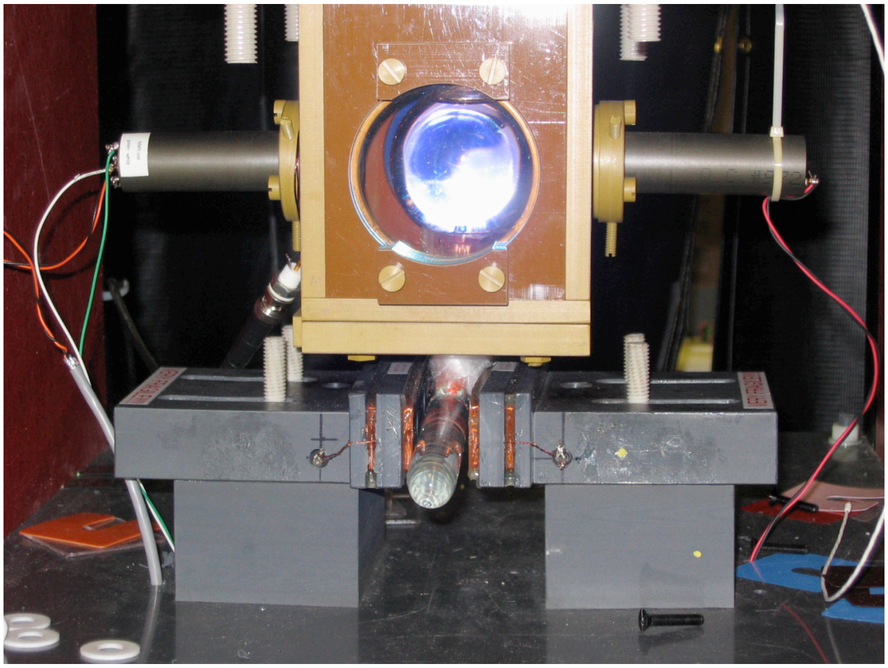

Figure 9: Dual coils mounted inside the NMR system.

8.1 Response of the Dual Coils

The quality curve of the dual coils can be seen in Figure 10. This plot shows that resonance of the pair of dual coils occurs in the vicinity of 250 kHz, although it is unclear from the graph exactly where resonance appears. Obviously, this is not near 91 kHz, but the ringing of the dual coils at resonance is still significant enough to continue to mask the signal.

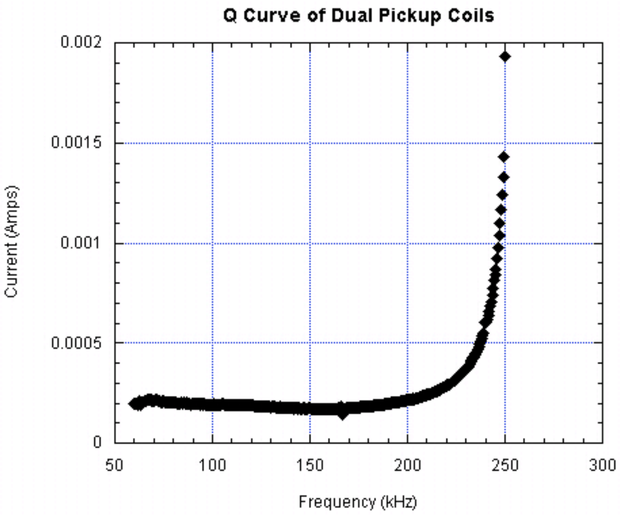

Figure 10: Quality curve of the dual coils. These coils have a resonant frequency of approximately 250 kHz, but continue to mask the extremely small water signal in the 91 kHz region.

8.2 Frequency Sweep NMR with the Dual Coils

Figure 11 shows frequency sweep NMR of a water cell with the dual coils in the system. Although reduced, frequency dependent background is still present in the system and the water signal continues to be buried. It appears as though there may be the start of a water signal around 95.6 kHz, but the graph sharply increases with a variation in frequency because of the background. When the graph is blown up around 95.6 kHz (Figure 12) it appears as though a water signal may be starting to appear, but there is still too much frequency dependent background present to know for sure.

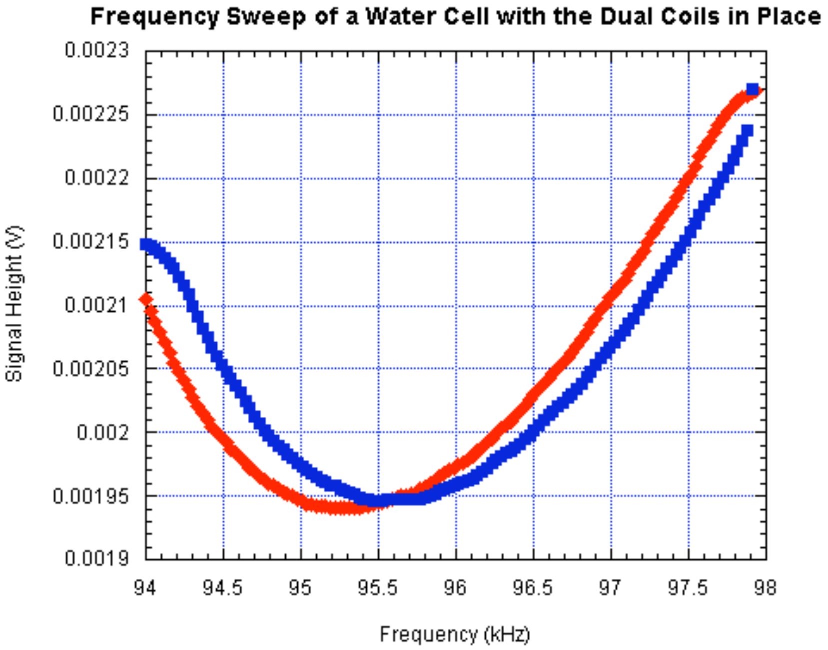

Figure 11: Field Sweep NMR on a water cell using the dual coils. Note the tremendous amount of background (on the order of milivolts) picked up by a system looking for a microvolt water signal.

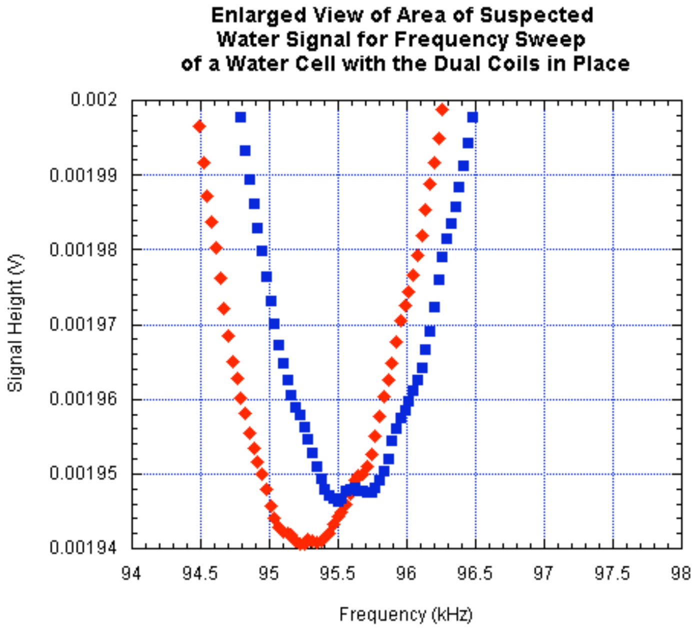

Figure 12: Enlarged view of Figure 11 examining the possibility of a water signal being present in the 95.6 kHz region.

8.3 Field Sweep NMR with the Dual Coils

Although the dual coils did not effectively shift the resonant frequency enough to obtain a water signal, they did succeed in reducing the background in the system. Figure 13 shows a NMR Field Sweep on a water cell with the new dual coils. The peaks in the graph are the water signal and the residual to each side is the background picked up by the pickup coils. In the this field sweep run, the background is significantly smaller, with much less fluctuation than with previous pickup coils. Reducing the background picked up by the pickup coils allows a much clearer field sweep signal to be generated, allowing for more accurate measurements.

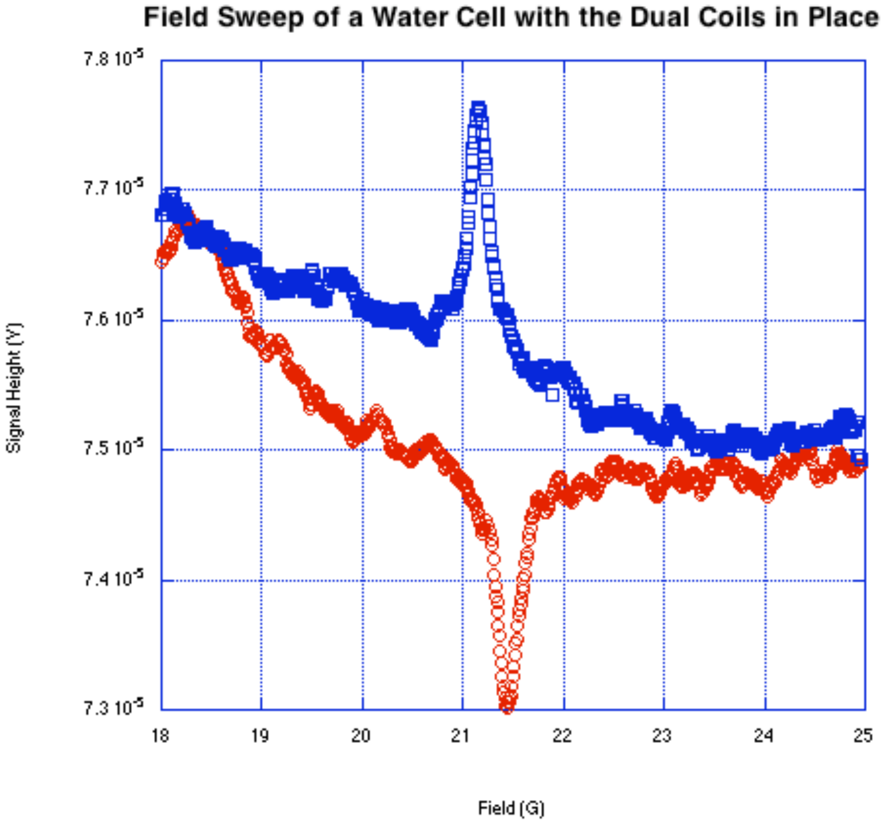

Figure 13: Field Sweep NMR done on a Water Cell with the dual coils in place. Notice that the noise is extremely low and that a clear signal is visible

8.4 Dual Coil Conclusions

Although the Dual Coils did not successfully generate a water signal when running frequency sweep NMR, they were a step in the right direction. They proved that with a dual coil design and increased inductance, the background in the system could be significantly reduced, allowing us to see field sweep NMR data of a water cell much more clearly that in the past. The only shortfall of the dual coils was that they did not move the resonant frequency of the pickup coils far enough away from our sweep range to allow a water signal to be seen when performing frequency sweep NMR.

9 New Single Coils

A second new set of coils was constructed in an attempt to move the resonant frequency (ω_o) away from the 91 kHz region. We chose to do this in favor of adding capacitance to the dual coils so that we would not risk breaking the wire on the dual coils. These pickup coils were referred to as the single coils because they contained only a single winding of wire on each coil. Each single coil was wound with 250 turns of wire in order to increase the signal strength even more than had previously been accomplished with the dual coils. In addition, a 5pF capacitor was added in series to each coil in an attempt to increase the resonant frequency (ω_o) enough so that it would not mask the signal at 91 kHz (see Equation 3). This configuration resulted in a resonant frequency of only 156 kHz for the single coils, which was lower that that of the dual coils (see Section 8.1), even with extra capacitance added. This was because winding 250 turns of wire on each coil increased the inductance so much that we could not add enough capacitance to effectively shift the resonant frequency (ω_o). As with the dual coils they were constructed with a removable face plate and a male BNC connection terminal.

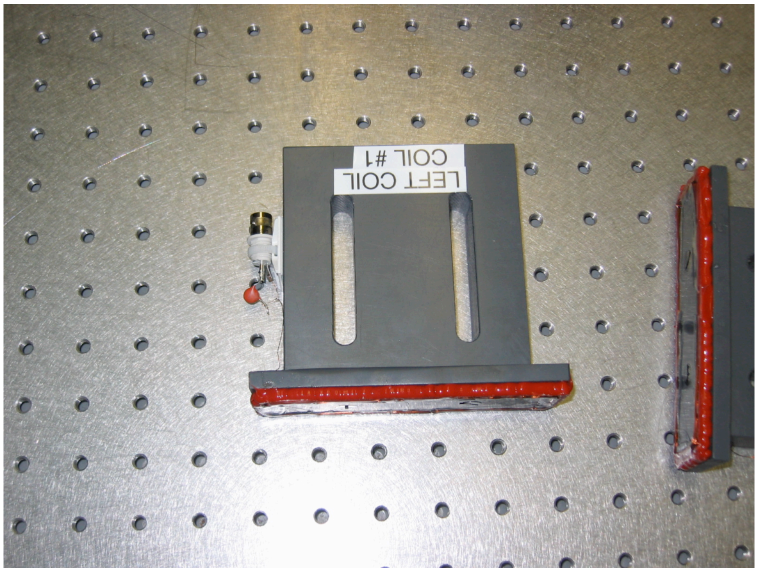

Figure 14: Top view of the single pickup coils. Notice the single coils of wire epoxied in place, the male BNC terminal, and the capacitor wired in series with the coil.

Unfortunately to date the single coils have yet to yield a water signal. More testing with these coils will be needed in the future.

10 Conclusions and Further Research

Although we were unsuccessful in revealing a water signal, we did succeed in successfully reducing the background present in the system. The dual coils successfully did this and as a result yielded a much better field sweep signal than had previously been attained. The only shortcoming of these coils was that they were not successful in eliminating the effect of the pickup coil's resonant frequency (ω_o) at 91 kHz. We attempted to remedy this problem by constructing a new set of coils with capacitance added to them.

Unfortunately the single coils have yet to yield a water signal, but there is a tremendous amount of work still to be done with them. The next step is to determine exactly why these coils are not yielding a water signal and remedy the problem. My guess at this point is that we may need to experiment with different capacitances wired in series to hopefully shift the resonant frequency (ω_o) of the coils. Once we have established that adding capacitance can successfully shift the resonant frequency (ω_o), a new set of dual coils will need to be constructed with capacitors wired in series so that the benefits of both coil designs may be achieved by one set of pickup coils.

References

[1] B. Hahn, Frequency Sweep NMR on Polarized ^{3}He Target Cells, College of William and Mary, (2004 REU Report)

[2] D.E. Milkie, Polarization and Polarimetry of ^{3}He, College of William and Mary, (2002 Senior Honors Thesis).

[3] J.L. Knowles, Investigation of Techniques for Producing High Polarization ^{3}He Gas Targets, College of William and Mary, (1999).

[4] T.G. Walker, W. Happer, Rev. Mod. Phys. **69**, 629, 634 (1997).

[5] J.R. Johnson *et al.*, Nucl. Instr. And Meth. In Phys. Res. A **356**, 150 (1995).

[6] J.G. Powles, Proc. Phys. Soc., **71**, 496.

[7] M.V. Romalis, Laser polarized ^{3}He target used for a precision measurement of the neutron spin structure, Princeton University, (1997).