Pulsed NMR Experiment Guide

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1. **Introduction**

NMR or nuclear magnetic resonance occurs when nuclei are placed in a magnetic field. It is a physical phenomenon where the nuclei absorb and then re-emit electromagnetic radiation. NMR allows for the observation of specific quantum mechanical properties of the atomic nucleus.

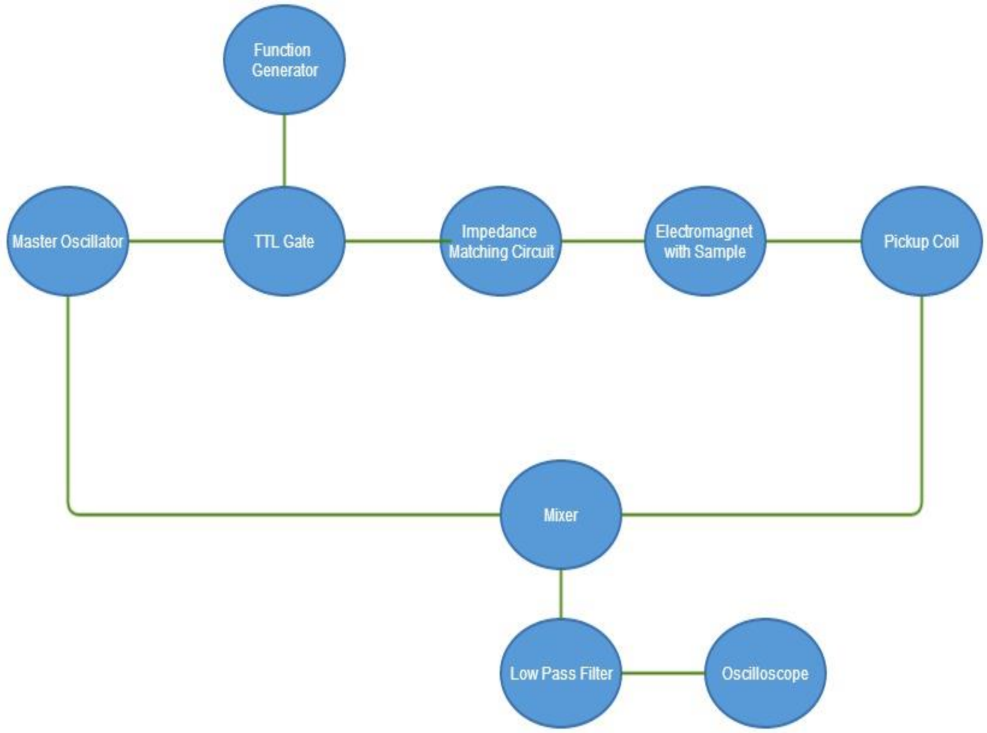
In order to use NMR, it is necessary to have nuclei with non-zero angular momentum and a magnetic moment. When the nuclei are placed in a magnetic field, the magnetic moments either align (paramagnetic) or oppose (diamagnetic) the magnetic field. This occurs due to the magnetic field breaking the degeneracy of the nuclei, causing them to go into either the spin up or spin down state. In addition to the differing spin states, the nuclei will also precess around the magnetic field at a frequency known as the Larmor Frequency.

The relevant information gained from pulsed NMR comes from applying short radiofrequency pulses in succession. As a result, the precession of the nuclei are then shifted by a certain amount depending on the applied signal. The nuclei are then allowed to relax back to their thermodynamic equilibriums where measurements are taken. The important quantities that are obtained from pulsed NMR and the T1 and T2 relaxation times.

The T1 relaxation time (spin-lattice relaxation) is the relaxation of the z-component of the net magnetization. T1 is measured by applying a series of $\pi/2$ pulses to the sample. The first pulse shifts the magnetization 90 degrees and the spins decay, the other pulse shifts the magnetization another 90 degrees, leave a small amount in the x-y plane. Then the free induction decay can be measured, which is an observable NMR signal from non-equilibrium spins. This is then used to determine the T1 relaxation time.

The T2 relaxation time (spin-spin relaxation) is the relaxation in the x-y component of the magnetization. The T2 relaxation time can be measured by initially applying a $\pi/2$ pulse to shift the spins into the x-y plane. After the pulse, the spins in the x-y plane begin to dephase due to the inhomogeneity in the magnetic field. A π pulse is then applied in order rotate all the spins over the x-y plane. The spins that were moving slower are now going to be ahead of the spins going faster and eventually they catch up to each other to create a large spin-echo signal. The spin echo is then used to determine the T2 relaxation time. However, because the magnetic field used is not completely uniform, the dephasing of the spins is not completely natural and is influenced by the irregularities, so a short T2* time is used as the transverse relaxation time constant.

2. Experimental Setup



Electronics

Master Oscillator: The oscillator provides a signal that will perturb the nuclei in the sample. The oscilloscope should be set to deliver a signal with frequency equal to the Larmor frequency $\omega = \gamma B_0$, where γ is the gyromagnetic ratio and B_0 is the magnitude of the constant magnetic field.

TTL Gate: This circuit cuts up the signal from the oscilloscope governed by the function generator. The logic the circuit uses is given by: $V_{out} = \begin{cases} V_{in}, & V_{in} \geq 5V \\ 0, & V_{in} < 5V \end{cases}$. It is important that no negative voltages are applied to the circuit.

Function Generator: The function generator is used in conjunction with the TTL circuit and the oscilloscope that will create pulses to shift the nuclear spins. It is important that the duration of the pulses be $\approx 5V$ and non-negative. The duration of the $\pi/2$ pulse is given by: $t_{\pi/2} = \frac{\pi}{2\gamma B_1}$ where γ is the gyromagnetic ratio and B_1 is the magnitude of the rotating magnetic field. The duration of the π pulse can be found by multiply the duration of the $\pi/2$ by 2. In order to produce both types of signals, a second function generator or a computer should be used.

Rf Amplifier: The RF amplifier converts the signal from the oscilloscope into a larger signal with more power. It is important to make sure the amplifier is not turned on unless the TTL circuit is operational. If too long of a signal is passed through the amplifier, it may result in burning out the diodes in the impedance matching circuit.

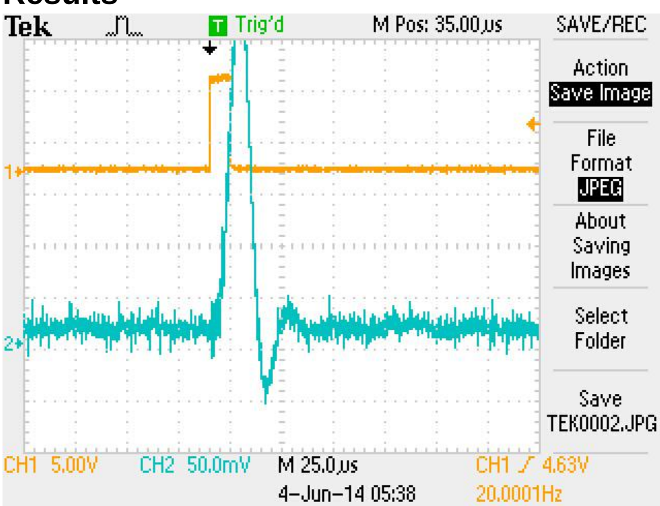
Impedance Matching Circuit: As the name suggests, the purpose of this circuit is to match the impedances of the source and the load. Once the impedances are matched the signal reflection will be minimized.

Electromagnet with sample: The sample of nuclei is placed in the magnetic field. The magnetic field can be adjusted using a DC power supply with current regulator. This can be used with the oscilloscope in order to fine-tune the frequency of the oscillator. The magnet strength can be altered in order to get the best response by changing the current until the oscilloscope shows the largest response to the pulses.

Pickup Coil: The pickup coil measures the signal from the precessing spins that are perturbed. The pickup coil allows for the measurement of the free induction decay and the spin echo. Because the signal from the nuclei are so small, the signal must be amplified in order to become measureable.

Mixer: The purpose of the mixer is to clean the signal from the sample in order for it to be used with the oscilloscope. The mixer receives two inputs, one from the master oscillator and another from the pickup coil. The mixer subtracts the signal from the master oscillator from the signal from the pickup coil resulting in a measurable signal decay from the sample.

3. Results



After setting up the electronics for the experiment, data was finally able to be taken. The picture above shows the output of the oscilloscope of a sample of light mineral oil. Looking at the blue output from the bottom, it can be determined that the sample did respond to the radiofrequency pulse; however, it is impossible to determine the amplitude of the free induction decay of the response.

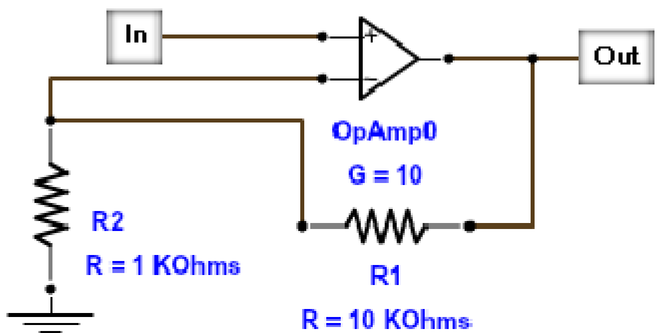
It was later found out that the reason why the free induction decay was immeasurable was due to the leakage from the impedance matching circuit creating noise in the signal. The leakage occurs due to the pickup coil on the sample. The pickup coil is designed to amplify the small changes in voltage from the perturbed spins which is on the order of nanovolts. In order for these changes to be measurable on the oscilloscope, they must be amplified to the millivolt scale. The problem arises because any noise that gets picked up from the coil will also be amplified. As stated earlier, due to the poor shielding on the impedance matching circuit, the signal passing through the circuit ends up getting picked up and amplified by the coil, making the results incomprehensible.

4. Additional Circuits

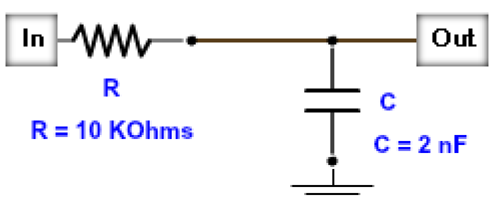
Even though the experiment was unsuccessful in obtaining meaningful results, there were still some modifications made in order to optimize the overall setup.

The circuits below are used in conjunction with the function generator in order to optimize the electronics in the NMR setup:

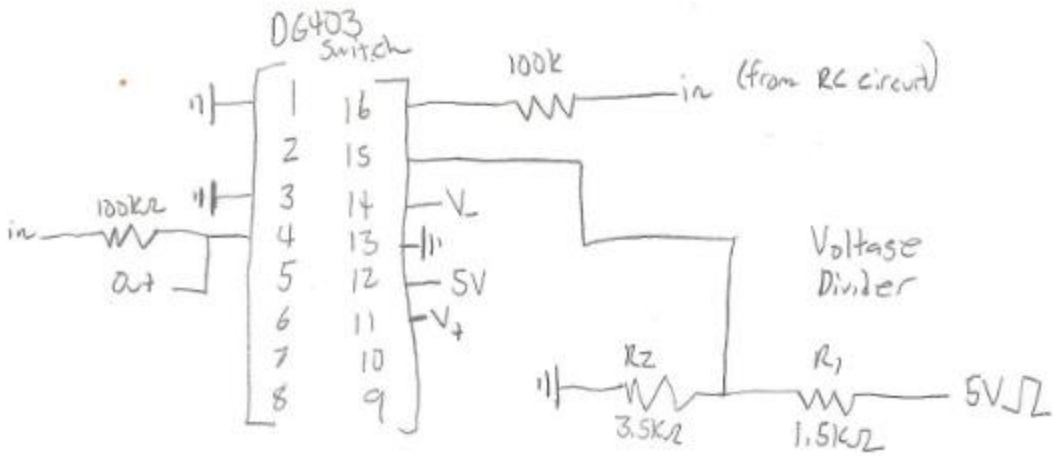
1) *Non-inverting amplifier*:



2) *RC Circuit:*



3) *DG403 Switch:*

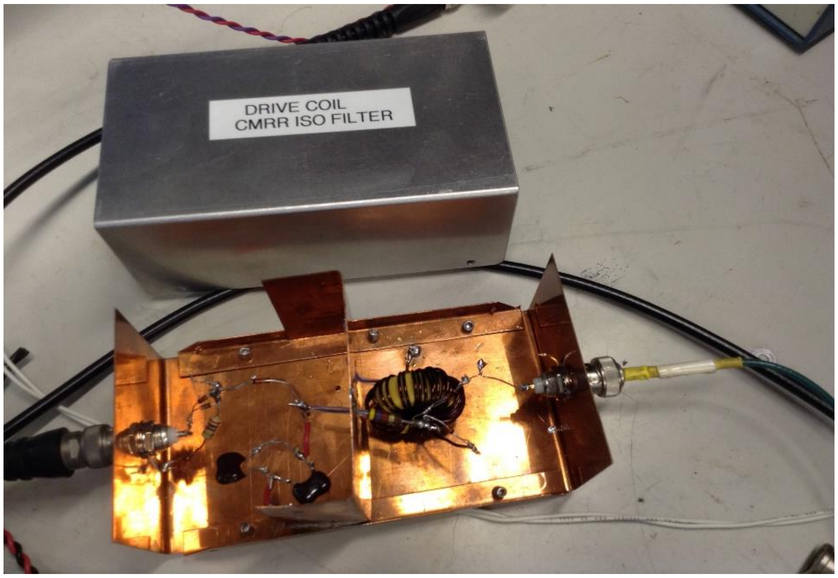


When these three circuit are combined, they create an additional control for the RF amplifier. The RF amplifier needs additional control because if the pulse is too long, the power of the signal will be too great and burn out the diodes in the impedance matching circuit. Upon further development of the setup, it is expected to eventually have a computer control the output and due to the natures of computers, it is possible for the generation of a signal that is long enough to burn out the diodes.

The function of the circuit is shut off the signal if the signal length is too long. The purpose of the non-inverting amplifier is to create a signal large enough to be processed by the RC circuit. The RC circuit charges based on the values of the resistor and capacitor which is then fed into the DG403 switch. If the signal applied to the TTL circuit is too large, the RC circuit will charge to a certain voltage which will in turn reach the required voltage to shut off the DG403 switch. This same logic on a different pin controls another unaltered pulse train which will Regulate the desired pulse length.

The following circuit can serve as a replacement if a computer cannot be used to control a function generator. Instead, two function generators can be used to generate the 90 and 180 degree pulses. The purpose of this circuit is to utilize an op-amp in order to generate a cleaner signal with less triggering issues.

5. Conclusion



Although the experiment was unsuccessful in obtaining useful data for determining the T1 and T2 times for various samples, the information and circuits made may prove to be useful for future experiments. The biggest obstacle to overcome is whether or not the current setup will actually enable the measurement of the free induction decay and the spin echo of the samples used. In order for the experiment setup to become fully operational, the impedance matching circuit must be addressed. The leakage from the circuit could be fixed by possibly rebuilding the circuit to have much better shielding. Upon fixing the impedance matching circuit, further troubleshooting can be made possible.

References:

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