Pulsed Nuclear Magnetic Resonance: Free Induction Decay and Spin Echoes

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ABSTRACT

This laboratory explores two phenomena of Biological Physics: Free Induction Decay (FID) and Spin Echoes. The decay graph of the spin echo is used to determine the spin-spin relaxation time (or the Transverse relaxation time) of glycerin. The first experiment involves using an on-resonance one pulse sequence of magnetic field to obtain a graph of FID over time for the glycerin sample. The second experiment is done in order to measure spin echo signals which must be done with two pulses. With the obtained conditions, different observation can be made for both on-resonance pulses and off-resonance pulses. The primary objective of these experiments is to find the T_2 relaxation time of Glycerin.

Pulsed Nuclear Magnetic Resonance Experiment

1 Introduction

In 1946, Felix Bloch and Edward Purcell independently discovered Nuclear Magnetic Resonance. However, NMR was still a slow process, in which the normal time for processing an image taken by MRI took more than two days. Later Richard Ernst in 1991 discovered the Fourier-Transform Nuclear Magnetic Resonance (FT-NMR) which resolved slower spectrum sample times, producing images in a matter of minutes. NMR was one of the most prolific and influential discoveries of the 20th century and changed the course of modern medical techniques of diagnosing various illnesses and diseases.

NMR operates on the general principle of nuclei of particles possessing an intrinsic spin and magnetic moment; spin is manipulated by applying magnetic field to a sample in order to control the direction of the magnetic moment of the nucleus. NMR relies on the concept of the gyro-magnetic ratio to differentiate between different species of material to relatively a high accuracy. The following equation describes the gyro-magnetic ratio, the proportionality constant of the intrinsic spin and magnetic moment: $f_0 = \gamma B_0$, where f_0 is the frequency of precession, B_0 is the magnetic field applied to the nuclei & γ is the gyro-magnetic ratio of the nuclei. Free Induction Decay is the process by which the excitation of the nucleus of a particle returns to its original equilibrium magnetization after being perturbed by a magnetic field. Maximum intensity of the signal produced is when the direction of the magnetic moment is perpendicular to the pulse magnetic field as is experiences more torque shown by Equation 22 .

T_2 is the time of relaxation of the particles back to their original position of magnetization. The time of relaxation can be determined by the equation below, where A is the magnetization of the nuclei and $\beta = \frac{-2t}{T_2}$.

$$A = A_0 e^{\beta} \tag{1}$$

Since the moment of the nuclei is not perfectly aligned with the magnetic field, the sample undergoes torque:

$$\tau = \vec{\mu} \times \vec{B} \tag{2}$$

where $\vec{\mu}$ is the amgnetic moment and $\vec{B}$ is the transverse magnetic field.

2 Experimental Setup

Figure 1 displays the required equipment for the experiments. NMR spectrometer, probehead and electromagnet are utilized to determine Glycerin's free induction decay (FID) as well as on and off resonance of spin echo. In addition, data is collected through a control program which is shown in Figure 2.

Figure 1. General Setup (from Left to Right): NMR Probehead and Electromagnet, NMR Spectrometer,

2.1 Obtain on-resonance FID

In order to obtain on resonance FID of Glycerin sample, modify the parameters in the "PS 15 Control and Processing" program as shown in Figure 2. The position of the sample might need to be adjusted by hand to maximize the signal. The gain and Δf_0 can be changed also.

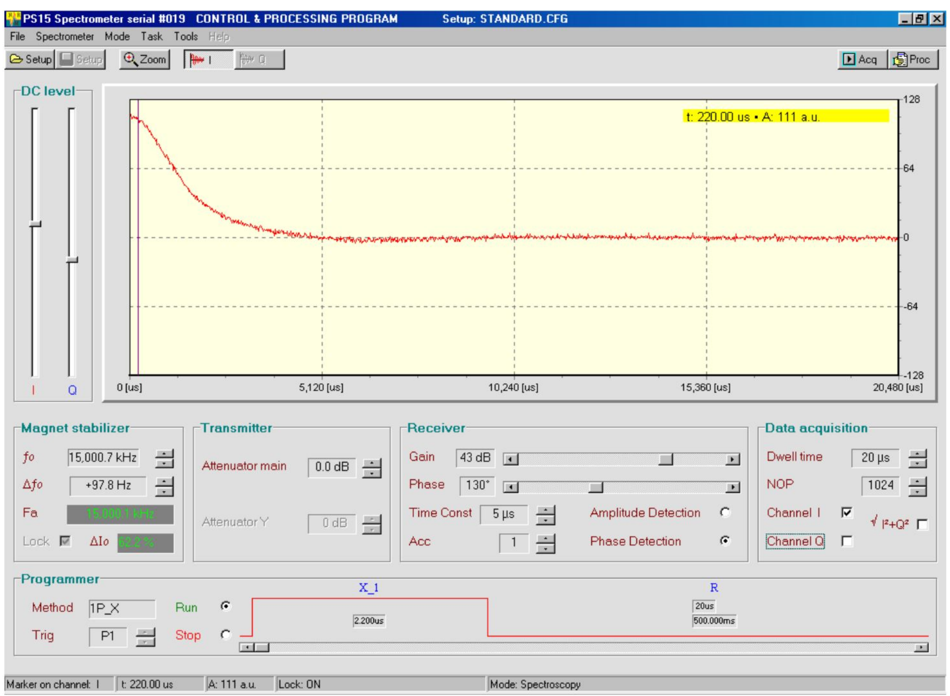

Figure 2. PS 15 Control and Processing Program Window

2.2 Determine Pulses

After successfully obtaining the on resonance FID signal from the previous step, move the cursor on the live-time plot and drag the marker vertical bar so that "t" shown on the graphing area reads 25 μs or more (exceeding the 'dead' time for the instrument). Set pulse width to minimum 0.2 μs (Programmer $\rightarrow X_1$ = 0.2 μs). Use the DC level slider to adjust the DC offset to zero. Read and record the FID amplitude in the marker position. Repeat the measurement with 0.2 μs increments and record the FID amplitude.

2.3 Obtaining Off-resonance Spin Echo

By setting our Method to 2P_X_D we can use the setting for a two pulse sequence. Use the determined conditions from the previous experiment for this step. Add or subtract 500 Hz to Δf_0.Set the program to 'Run' if it is in "Stop" mode. Adjust "Acquisition time," "Dwell time (DW)," or "Number of Points (NOP).

2.4 Obtaining On-resonance Spin Echo

To obtain on-resonance spin echo, use the previous parameter from above (2.3). Adjust the values of Δf_0, pulse length, phase, and gain.

2.5 Measuring T_2 Time Manually Using Simple Spin Echo Method

To start, use one-pulse sequence, in this case 1P_X, to determine the correct conditions that are necessary to acquire an on-resonance FID after a $\frac{\pi}{2}$ pulse.

3 Result

Figure 3 shows on-resonance one pulse of Glycerin, this later helps to determine the pulse phase in Figure 4. Figure 4 determines that at $\frac{\pi}{2}$ pulse happens at t = 2.4 μs, π pulse is at t = 4.4 μs, $\frac{\pi}{2}$ pulse occurs at t = 6.4 μs, 2π pulse is at t = 8.4 μs, and $\frac{5\pi}{2}$ pulse happens at t = 10.4 μs. By determining the phases of pulse, the off and on resonance of spin echo can be found. Due to instrumental errors, the second pulse (π pulse) length shown in Figure 4 is not exactly 2 times of the first pulse ($\frac{\pi}{2}$ pulse). Figure 5 presents the obtained off resonance spin echo signal. Figure 6 displays the collected on resonance spin echo signal from the experiment above. However, the obtained signal in Figure 6 is not fully on resonance. This can be seen around the area where t = 10,240 μs. The relaxation time of a sample is the time it takes for the transverse magnetization to fall to 37% of its initial value. Relaxation time T_2 of echoes amplitude decay can be calculated by equation 1 or using "Curve Fitting Tool" from Matlab. Figure 7 below is obtained by utilizing Matlab. The collected equation is

$$A = 84.77e^{(-0.04416t)} \implies \beta = \frac{2t}{T_2} = -0.04416t \implies T_2 = \frac{2}{|-0.04416|} = 45.3\mu s$$

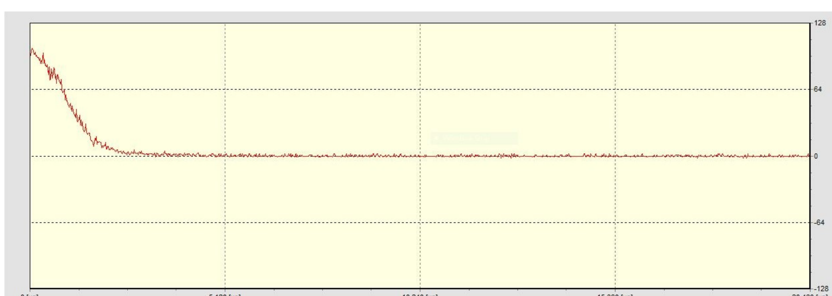

Figure 3. On-Resonance One Pulse of Glycerin

4 Conclusion

Collected decay signal shown in Figure 7 and the calculated relaxation time T_2 for Glycerin was within the range of experimentally measured and widely accepted decay times. This signal can be improved by adjusting the parameter of the on resonance one pulse signal. Additionally, in order to obtain a better on-resonance signal for spin echo, different adjustments can be made, such as physically adjusting position of the sample. Besides, the position of Glycerin sample can be consider a source of error. The signal can be minimized or maximized depending on where the sample is in the electromagnetic field. Proper positioning of the sample is a crucial step to ensure the signal is clear.The two methods provided, black o-rings and white plastic tube insert proved inferior to the human hand. Moreover, collected data represented in some figures have unexpected data points due to reading sensitivities during measurement, perhaps even the interaction of the magnetic field of the instrument with surrounding equipment.

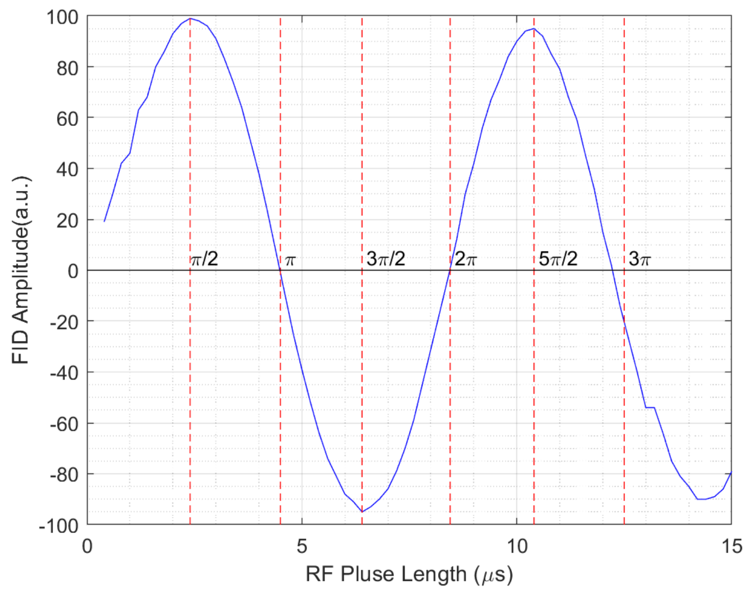

Figure 4. The RF Pulse Phases

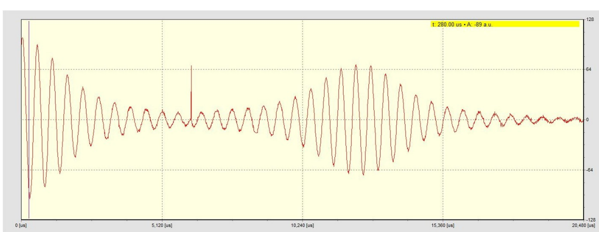

Figure 5. Off Resonance Spin Echo

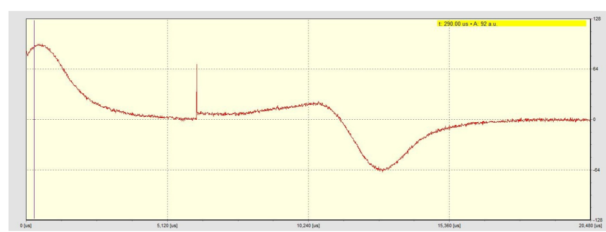

Figure 6. On Resonance Spin Echo

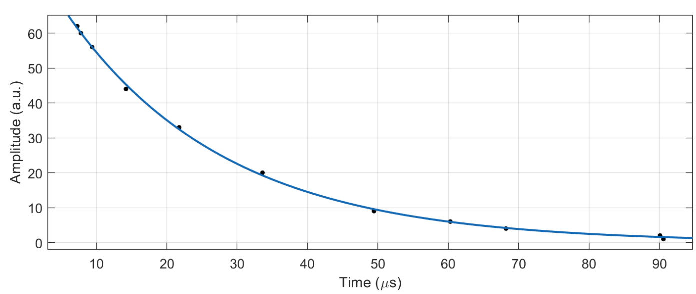

Figure 7. Echoes Amplitude Decay

References

1. "Free Induction Decay (FID)." Questions and Answers in MRI, mriquestions.com/free-induction-decay.html
2. "Nuclear Magnetic Resonance." Wikipedia, Wikimedia Foundation, 4 Feb. 2019,