**PHYS 4007** 

Lab Report

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#### **ABSTRACT**

This Lab report focuses on Nuclear Magnetic Resonance (NMR) in half spin particle. For the experiment glycerin and mineral were used. The experiment aims at measuring the spin-lattice and spin-spin relaxation time for both using resonance frequency (RF) spectroscopy methodology. A permanent magnet setup is used to induce magnetic resonance in the samples. The spin-lattice ( $T_1$ ) relaxation time measured for glycerin and mineral respectively is (40.9 + 1.1) ms and (22.45 + 1.1) ms respectively. When compared to expected values for the same using a T-test they gave results that depict accuracy and precision. The spin-spin ( $T_2$ ) relaxation time measured for glycerin and mineral oil respectively is (30.0 + 1.1) ms and (10 + 1.1) ms respectively. When compared to expected values for the same use a T-test they gave results that depict that these values are not the best outcomes. One of the major reasons for imperfect results was the high noise sensitivity of the oscilloscope and the spectroscope. It was observed that even by plugging a laptop charger around the system resulted in causing a huge amount of noise in the signal.

#### **THEORY**

Nuclear magnetic resonance (NMR) spectroscopy is used to determine characteristics of molecular structure at atomic level. NMR spectroscopy comes from the fact that nuclei are electrically charged and have multiple spins. When an external magnetic field is applied it creates a possibility for energy transfer. This energy transfer results in change in the spin of the nuclei which further results in change in the magnetic moment. After the application on this external field the nuclei spin returns to its base position (Un-magnetized state). The same frequency of energy is released as was absorbed and thus it is measured by using the spectroscope setup [1].

$$\vec{\mu} = \gamma \vec{I} \tag{1}$$

For this experiment the focus has been on magnetism and *spin angular momentum*. The relation between the two can be observed in equation 1. In the given equation  $\vec{\mu}$  represents the magnetic moment,  $\gamma$  represents the *gyromagnetic ratio* and  $\vec{l}$  represents the *spin angular momentum* [2].

Since the spin is quantized, it will either align parallel or anti-parallel to the *applied magnetic* field  $(B_0)$  as in this case only spin half substances were used. The quantization of the *spin angular momentum* can be written such as in equation 2. With  $I_z$  representing *spin angular momentum* in the direction of the applied magnetic field, which in this case has been assumed to be Z-direction, m is the *magnetic quantum number* which can take integral values between (-I and +I), which in this case would be (+0.5 or -0.5) and  $\hbar$  is *modified plank's constant* [2].

$$I_z = m.\hbar \tag{2}$$

The energy due to the applied magnetic field can be calculated using equation 3. When the nuclei is aligned along the magnetic field the value of m is +0.5 and when it is aligned against the magnetic field it is -0.5. Figure 1 shows the splitting of the energy level when an external

magnetic field is applied, this effect is also known as Zeeman splitting. In the absence of an external magnetic field these energy states are degenerate [3].

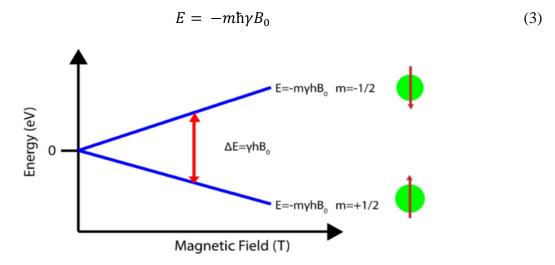


Figure 1: Zeeman Splitting: Energy level splitting in the Presence of Magnetic Field

When at equilibrium when the overall magnetic moment is oriented parallel to the applied magnetic field it results in a macroscopic magnetic moment  $M_0$ . To detect this magnetic moment Another magnetic field is applied in the Y-direction. This is done using a radio frequency (RF) signal that is at resonance with the magnetic moment of the nuclei. This concept provides us with the Larmor equation, i.e., equation 4 [4]. On applying this frequency as a magnetic signal, the magnetic moment of the sample gets unaligned from the Z axis and gives an output signal proportional to the RF precession signal. The relaxation of the magnetic moment of the sample here results in measurement of the *spin-lattice relaxation time* ( $T_1$ ). This process has been illustrated in figure 2 [2].

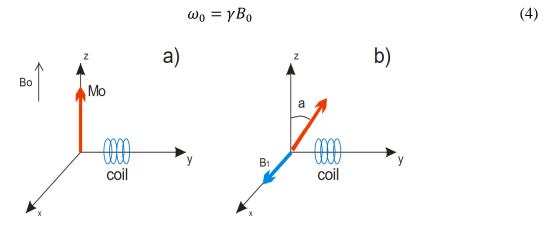


Figure 2: Magnetic Moment Orientation on the nuclei a) In static Magnetic field b) RF Magnetic Field

#### **Spin-Lattice Relaxation Time**

Under the static magnetic field, the sample get magnetized to a certain degree. The magnetization under static magnetic field can be called  $M_0$ . When an RF signal is applied to the sample the magnetization of the sample changes to  $M_Z$ . The magnetization of the sample changes instantaneously when the RF signal is applied, and it starts to decay instantaneously too. The time taken for the magnetization to reach back to the initial magnetization state is called the spin-lattice relaxation time ( $T_1$ ). Figure 3 depicts the manner of magnetization, while equation 5 and 6 depict the formulae [5]. The variable  $T_1$  as seen in equation 5 and 6 is the lattice relaxation time for the given substance.

$$\frac{dM_z}{dt} = \frac{M_0 - M_z}{T_1} \tag{5}$$

$$M_z(t) = M_0 \left( 1 - e^{\frac{-t}{T_1}} \right)$$
 (6)

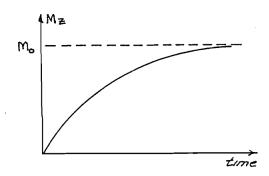


Figure 3:Re-Magnetization of Sample under Static Magnetic Field

#### **Spin-Spin Relaxation Time**

Spin-spin relaxation time can be defined as the time taken to reduce the transverse magnetization by a factor of e. A factor of e is chosen as such because there is no magnetization in the transverse direction at the equilibrium. The equation 7 and 8 represent the spin-spin relaxation time formulae [2].

$$\frac{dM_y}{dt} = \frac{-M_0}{T_2} \tag{7}$$

$$M_{y}(t) = M_{0}e^{\frac{-t}{T_{2}}} \tag{8}$$

In terms of signal the spin-spin relaxation time is represented by the echo signal obtained and the time taken by it to fade away. The result is expected to look like that in figure 4 and 5. In figure 5 there are multiple echoes that are observed and the amplitude of each keeps decreasing This decrease is caused due to subsequent demagnetization of the nuclei. The peaks of each of the echoes are recorded and plotted to fit equation 8. Thus measuring the spin-spin relaxation time  $(T_2)$  [2].

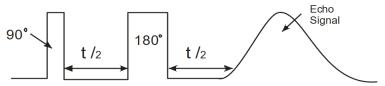


Figure 4: Echo signal as obtained from the perspective of the pulses

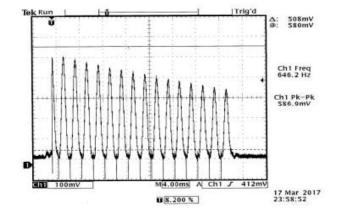


Figure 5: Multiple echoes with reducing amplitude

#### **APPARATUS**

This section lists all the apparatus used for the NMR experiment with brief details on their working and setup.

#### TeachSpin's PS1-A

PS1-A is the NMR spectrometer used for this experiment. Most parts of the spectrometer in this case is embodied in one box but is separate in configuration and does not alter the working of one another. For example, the working of the transmitter does not interfere with the receiver [6].

The major follow of parts of the spectrometer is depicted in figure 6.

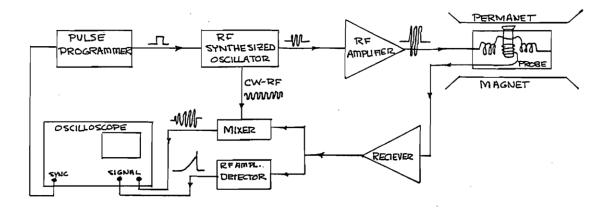


Figure 6: Spectroscope Flowchart

#### -Pulse Programmer:

The pulse programmer is used to create a 0-4V logic level pulse (digital) the purpose of this pulse is to control the RF oscillator. Part of the spectrometer that delays with A-B width is what constitutes of the pulse programmer. It is depicted in Figure 3 [7].

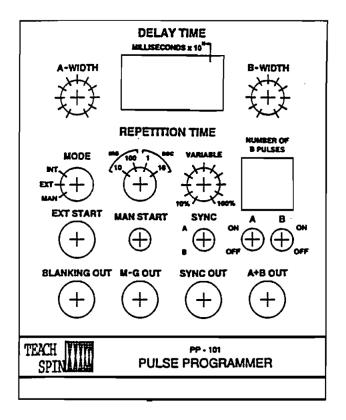


Figure 3: Connections Examining Pulse Programmer

#### -RF Oscillator

Based on the pulses received by the RF Oscillator it generates analog oscillatory pulses. The frequency of the output here is to be set by the user [6].

#### -RF Amplifier

It amplifies the signal received from the RF oscillator and passes on the amplified signal to Transmitter coils [6].

#### -Receiver

In figure 3 the red wire depicts the receiver wire than pick the emf induced and the RF signal. This signal is then sent to the receiver of the spectroscope [6].

#### -Mixer

This is an inbuild part of the spectroscope, it uses the initial signal from the RF Oscillator as reference signal to analyze the signal received from the receiver and then gives an output of only the emf induced signal. In simpler words it subtracts the RF signal from the receiver signal and leaves us with only the emf signal.

#### -RF Amplitude Detector

It receives the signal from the receiver and rectifies it such that it is proportioned as per the peak amplitude of the RF oscillator signal. It focuses on recording free induction decays and spic echo signals [6].

#### **Oscilloscope**

The oscilloscope is used to ready the signal from mixer and the RF amplitude detector. The oscilloscope is synchronized using the pulse programmer.

#### **Probe Assembly**

The probe consists of transmitter coil, receiver coils and a port for the sample vial. The horizontal position of the sample coil can be controlled by scale at the front and the vertical position of the coil can be controlled by the scale at the side of the probe. Figure 4 depicts the probe setup [6].

The transmitter coils are setup in Helmholtz configuration (Figure 5) which result is creating a magnetic field in the direction perpendicular to the sample vial and the receiver solenoid. The receiver solenoid is placed in between the transmitter coil such that an emf is generated on them due to the magnetic field of the transmitter coil [6].

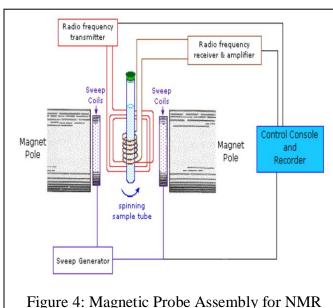
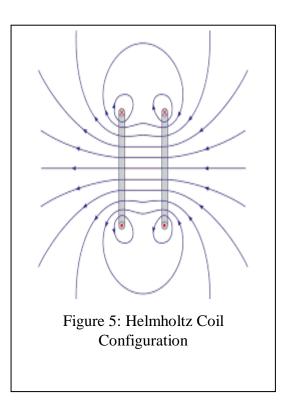


Figure 4: Magnetic Probe Assembly for NMR Spectroscope



#### **PROCEDURE**

#### **Spin Lattice Relaxation Time**

To measure the spin lattice relaxation time the apparatus was connected as depicted in figure 6. The test sample (glycerin or mineral oil) was inserted in the permanent magnet setup and its position and resonance frequency is adjusted until no beats were observed on the oscilloscope. Once the system had been adjusted to get a clear noise free signal the gain was adjusted such that the output beat had its peak at 4 Volts. The other specifications on the spectroscope was setup as depicted in table 1.

Characteristic	Value
A,B Pulse Width	0.25
Mode	Int
Number of B pulses	01
State of pulse A and B	ON
A&B Output	Vertical input on scope
Delay Time	100 us
Repetition time	100ms, 100% Variability
Sync	Pulse A
Sync Out	Extended to input on scope

Table 1: Spectroscope Specifications for Spin Lettice (T<sub>1</sub>) Measurements

The specifications in table 1 were altered to get a clear signal. Such that the width of pulse A is setup in such a manner as the minimum width that produces the maximum amplitude in the absence of pulse B. For pulse B it is the width that produces the minimum amplitude in the absence of pulse A (Zero pulse width does **NOT** count). Then both the pulses were switched on that the measurements were taken with an increment of 2ms for delay time. The RF frequency was retuned at regular intervals to avoid the variation in RF frequency due to the heating of the magnets.

#### **Spin-Spin Relaxation Time**

The setup of the spin-spin relaxation time was the same as for spin-lattice relaxation time, except the number of B pulses is set to 15 with 1ms delay for each pulse. The amplitude of each pulse was them measured.

#### RESULTS

#### Glycerin

Spin-Lattice Relaxation Time  $(T_1)$ 

This section will discuss the results obtained by analyzing the data for spin-lattice relaxation time for glycerin. The results calculated here are compared to expected values from the internet. MATLAB was used for analyzing the data. The code used in MATLAB can be found in the appendix. Figure 6 depicts the decay of the magnetization of the sample with time and the fit from the plot was used to calculate the spin-lattice relaxation time.

Spin-lattice relaxation time calculated (40.8 +/- 1.1) ms for glycerin is extremely close to that found online (40 +/- 2) ms. By using a T-test to compare the value, t = 0.89 is observed for t<2. It deems that the measured value is acceptable. The detailed calculation is provided in appendix. The reduced Chi square value for this fit was found to be 2.1535. Since closer it is to two, better is the fit, hence this is a goof fit.

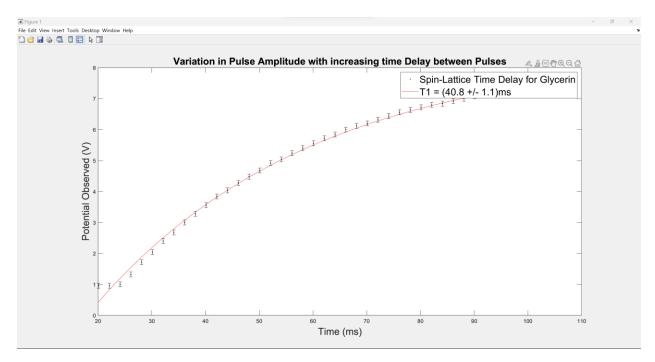


Figure 6: Pulse Amplitude increase for Glycerin fit to obtain the spin-lattice relaxation time Spin-Spin Relaxation Time (T<sub>2</sub>)

This section will discuss the results obtained by analyzing the data for spin-spin relaxation time for glycerin. The results calculated here are compared to expected values from the internet. MATLAB was used for analyzing the data. The code used in MATLAB can be found in the appendix. Figure 7 depicts the decay of the magnetization of the sample with time and the fit from the plot was used to calculate the spin-spin relaxation time.

Spin-spin relaxation time calculated (30.0 +/- 3.5) ms for glycerin is not so close to that found online (35 +/- 2) ms. By using a T-test to compare the value, t = 3.33 is observed for t>2. It deems that the measured value is to be considered not assigning perfectly with the expected value. The detailed calculation is provided in appendix. The reduced Chi square value for this fit was found to be 0.0083. A chi square value so much smaller than 2 indicates that this is not a good fit. Figure 8 depicts the data extracted from the oscilloscope plotted using MATLAB for glycerin's spin-spin relaxation time.

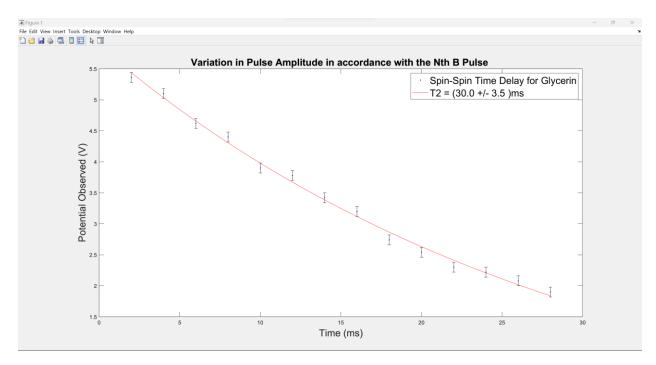


Figure 7: Pulse Amplitude decrease for Glycerin fit to obtain the spin-spin relaxation time

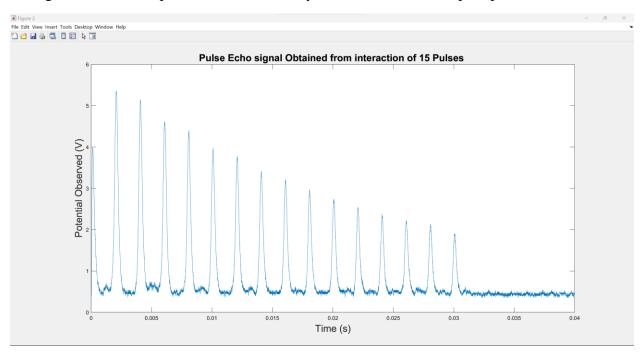


Figure 8: Echo Signal Extracted from Glycerin Sample

#### **Mineral Oil**

Spin-Lattice Relaxation Time  $(T_1)$ 

This section will discuss the results obtained by analyzing the data for spin-lattice relaxation time for mineral oil. The results calculated here are compared to expected values from the internet. MATLAB was used for analyzing the data. The code used in MATLAB can be found in the appendix. Figure 9 depicts the decay of the magnetization of the sample with time and the fit from the plot was used to calculate the spin-lattice relaxation time.

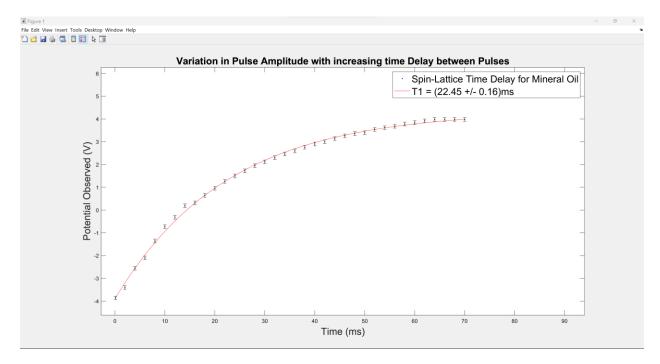


Figure 9: Pulse Amplitude increase for Mineral Oil fit to obtain the spin-lattice relaxation time

Spin-lattice relaxation time calculated (22.45  $\pm$ 0.16) ms for mineral oil is extremely close to that found online (20  $\pm$ 0 ms. By using a T-test to compare the value, t=1.26 is observed for t<2. It deems that the measured value is acceptable. The detailed calculation is provided in appendix. The reduced chi square value for this fit is 1.3428. A value less than 2 for the reduced chi square means that the errors have been over estimated.

#### Spin-Spin Relaxation Time (T<sub>2</sub>)

This section will discuss the results obtained by analyzing the data for spin-spin relaxation time for mineral oil. The results calculated here are compared to expected values from the internet. MATLAB was used for analyzing the data. The code used in MATLAB can be found in the appendix. Figure 10 depicts the decay of the magnetization of the sample with time and the fit from the plot was used to calculate the spin-spin relaxation time.

Spin-spin relaxation time calculated (10 +/- 26) ms for glycerin is not so close to that found online (15 +/- 2) ms. By using a T-test to compare the value, t = 0.2 is observed for t>2. It deems

that the measured value aligns with the expected value for the given amount of uncertainity. The detailed calculation is provided in appendix. The reduced Chi square value for this fit was found to be 0.0019. A chi square value so much smaller than 2 indicates that this is not a good fit. Figure 11 depicts the data extracted from the oscilloscope plotted using MATLAB for mineral oil's spin-spin relaxation time.

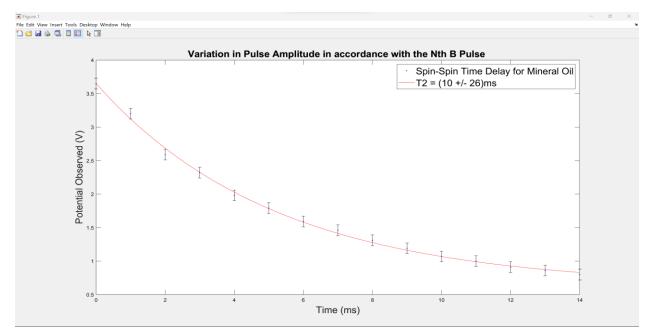


Figure 10: Pulse Amplitude decrease for Mineral Oil fit to obtain the spin-spin relaxation time

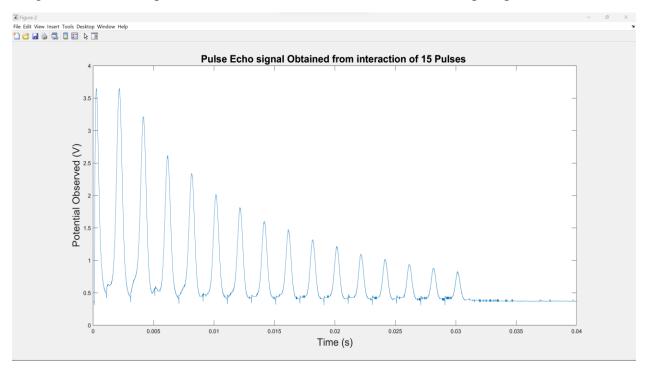


Figure 11: Echo Signal Extracted from Mineral Oil Sample

## **DISCUSSION**

The experiment studied Nuclear Magnetic Resonance (NMR) in half spin particle. Glycerin and mineral oil were the molecules of interest. The experiment measured the spin-lattice and spin-spin relaxation time for both using resonance frequency (RF) spectroscopy methodology. A permanent magnet setup was used to induce magnetic resonance in the samples. The spin-lattice ( $T_1$ ) relaxation time measured for glycerin and mineral respectively is (40.9 + -1.1) ms and (22.45 + -0.16) ms respectively. These results were compared to the expected values using T-test method and the were found to be accurate. The spin-spin ( $T_2$ ) relaxation time measured for glycerin and mineral respectively is (30.0 + -3.5) ms and (10 + -26) ms respectively. On testing these values with standard values using T-test methods, it was found that they were not accurate enough.

There were multiple sources of error that could have interfered with the results. One of the examples of that is the that the fact that the noise on the oscilloscope would drop significantly on unplugging a nearby laptop charger as it's magnetic field would otherwise interfere with the setup. Since the experiment was being performed in equipment heavy laboratory. There must have been other unknown factors that were tampering with the results. A second source of error could be the fact that the resonance frequency of the setup by the nature of it changes continuously even on adjusting it at short intervals does leave room for error and uncertainty. Furthermore, the coaxial cable that was carrying the signal between the permanent magnets and the spectroscope did not have a perfect isolation, even though it was covered by metal foil to help with grounding even a small move on it had been altering the signal and the noise significantly.

NMR has heavy applications in the field of material studies and the medical scanning for Magnetic Resonance Imaging (MRI) is also based of the concept of NMR. In the field of material studies, it is used to determine the structure and other characteristics of organic molecules solutions, crystals etcetera by applying the same concept of spectroscope as was applied in this experiment. Food quality and control uses NMR spectroscopy to identify carotenoids and quantify metabolites. Other extremely useful application of NMR spectroscopy lies in identifying human disorders, by using the techniques on cellular metabolism of an individual biomarkers are identified and are used to diagnose a wide range of conditions, including tuberculosis, pneumonia, and malaria. The technique can also be used to investigate Parkinson's disease, cardiovascular diseases, and a range of neuropsychiatric disorders such as bipolar, schizophrenia, major depression, and autism-spectrum disorders. NMR spectroscopy is also used to analyses environmental factors and study the contaminants in the environment. Over and all NMR has a wide range of extremely useful applications and many of its utilizations are still being discovered [8].

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#### APPENDIX I: MATLAB CODE

#### **Spin-Lattice Relaxation Time**

```
% This file is for analysing T1 for Mineral Oil
% Time is in ms, Voltage is in volts
time = MoilT1data(:,1);
time = table2array(time);
err time = (2E-08)*ones(size(time));
voltage = MoilT1data(:,2);
voltage = table2array(voltage);
err = 0.08*ones(size(voltage));
%F = fit(time, voltage, '(a*(1-exp(-x/b)))+c', 'Start', [100, 2,
0], 'Weight', 1/0.02^2+zeros(length(time), 1));
[F,goF,Fit_output] = fit(time,voltage,'(a*(1-exp(-x/b)))+c','Start',
[100, 2, 0], 'Weight', err.^(-2));
plot(F,time,voltage)
xlabel('Time (ms)', 'FontSize',18);
ylabel('Potential Observed (V)', 'FontSize', 18);
title ('Variation in Pulse Amplitude with increasing time Delay
between Pulses', 'FontSize', 18)
legend('Spin-Lattice Time Delay for Mineral Oil', 'T1 = (22.45 +/-
0.16)ms','FontSize',18)
hold on
errorbar(time, voltage, err, "k.", 'HandleVisibility', 'off')
hold on
errorbar(time, voltage, err time, 'horizontal',
"k.", 'HandleVisibility', 'off')
hold off
% Extract weighted jacobian
J = Fit output.Jacobian;
%Get the covariance and curvature matrix and extract the errors on F
%parameters from there.
curvature matrix = J'*J;
covariance matrix = inv(curvature matrix);
% Calculate CHI squared
min_chi2 = goF.sse;
```

```
dof = goF.dfe;
reduced chi2 = min chi2/dof;
Spin-Spin Relaxation Time
% This file is for analysing T2 for Mineral Oil
% Time in ms Volatge in Volts
% This is measured data analysis
time measured = MoilT2(:,1);
time measured = table2array(time measured);
voltage measured = MoilT2(:,2);
voltage measured = table2array(voltage measured);
[F,goF,Fit output] = fit(time measured, voltage measured, '(a*(exp(-
x/b)))+c');
err = 0.08*ones(size(voltage measured));
errorbar(time measured, voltage measured,
err,'vertical',"k.",'HandleVisibility','off')
hold on
plot(F,time measured,voltage measured)
xlabel('Time (ms)', 'FontSize',18);
ylabel('Potential Observed (V)', 'FontSize', 18);
title ('Variation in Pulse Amplitude in accordance with the Nth B
Pulse ','FontSize',18)
legend('Spin-Spin Time Delay for Mineral Oil', 'T2 = (10 +/-
26)ms', 'FontSize', 18)
% Extract weighted jacobian
J = Fit_output.Jacobian;
%Get the covariance and curvature matrix and extract the errors on F
%parameters from there.
curvature matrix = J'*J;
covariance_matrix = inv(curvature_matrix);
% Calculate CHI squared
min chi2 = goF.sse;
dof = goF.dfe;
reduced chi2 = min chi2/dof;
%%%%%
```

```
MoilextractT2 = 'MoilT2extracted1527825.isf';
MoilextractT2 = isfread(MoilextractT2);
time_extracted = MoilextractT2.x;
voltage_extracted = MoilextractT2.y;

figure(2)
plot(time_extracted, voltage_extracted)
xlabel('Time (s)','FontSize',18);
ylabel('Potential Observed (V)','FontSize',18);
title ('Pulse Echo signal Obtained from interaction of 15 Pulses
','FontSize',18)
```

## APPENDIX II: DATA COLLECTED

#### Glycerin

Spin-Lattice Relaxation Time

frequency (MHz)	Delay(ms)	Voltage (V)
15.26853	0.1	6.68
15.26853	2.1	6.16
15.26853	4.1	5.36
15.26853	6.1	4.6
15.26853	8.1	3.88
15.26853	10.1	3.2
15.26853	12.1	2.56
15.26853	14.1	2
15.26853	16.1	1.48
15.26853	18.1	1
15.26853	20.1	0.96
15.26853	22.1	0.96
15.26853	24.1	1
15.26853	26.1	1.32
15.26853	28.1	1.72
15.26853	30.1	2.04
15.26853	32.1	2.4
15.26853	34.1	2.68
15.26853	36.1	3
15.26853	38.1	3.28
15.26853	40.1	3.56
15.26853	42.1	3.84
15.26853	44.1	4.04
15.26853	46.1	4.28
15.26853	48.1	4.48
15.26853	50.1	4.68
15.26853	52.1	4.92
15.26853	54.1	5.04

15.26853	56.1	5.24
15.26853	58.1	5.4
15.26853	60.1	5.56
15.26853	62.1	5.72
15.26853	64.1	5.84
15.26853	66.1	6
15.26853	68.1	6.12
15.26853	70.1	6.2
15.26773	72.1	6.32
15.26773	74.1	6.44
15.26773	76.1	6.56
15.26773	78.1	6.64
15.26773	80.1	6.72
15.26773	82.1	6.8
15.26773	84.1	6.84
15.26773	86.1	6.92
15.26773	88.1	7
15.26773	90.1	7.08
15.26773	92.1	7.12
15.26746	94.1	7.2
15.26746	96.1	7.24
15.26746	98.1	7.32
15.26746	100	7.32
15.26746	101	7.32

# Spin-Spin Relaxation Time Time (ms) Voltage (V)

Time (ms)	Voltage (V)
2.00E+00	5.36
4	5.1
6	4.62
8	4.4
10	3.9
12	3.78
14	3.42
16	3.2
18	2.74
20	2.54
22	2.3
24	2.22
26	2.08
28	1.9

## **Mineral Oil**

Spin-Lattice Relaxation Tin		
Time (ms)	Volatge (V)	
0.1	-3.86	
2	-3.4	
4	-2.56	
6	-2.1	
8	-1.36	
10	-0.728	
12	-0.316	
14	0.192	
16	0.316	
18	0.644	
20	0.952	
22	1.26	
24	1.5	
26	1.72	
28	1.95	
30	2.12	
32	2.3	
34	2.46	
36	2.6	
38	2.76	
40	2.9	
42	3	
44	3.14	
46	3.26	
48	3.36	
50	3.4	
52	3.54	
54	3.62	
56	3.68	
58	3.78	
60	3.84	
62	3.92	
64	3.98	
66	3.98	
68	3.98	
70	3.98	

Spin-Spin Relaxation Time

Time (ms)	Volatge (V)
0	3 65

Nuclear	Magnetic	Resonance	(NMP) in	Half Snin	Particles
Nuclear	Magnetic	Resonance	(INIVIE) III	г пан эрш	ranneles

1	3.2
2	2.59
3	2.32
4	1.98
5	1.79
6	1.59
7	1.46
8	1.31
9	1.19
10	1.07
11	1
12	0.91
13	0.86
14	0.8

## **APPENDIX III: Sample Calculation for T-Test**

$$t = \left| \frac{\overline{x1} - \overline{x2}}{\sigma_{x1} - \sigma_{x2}} \right|$$

$$t = \left| \frac{40.8 - 40}{1.1 - 2} \right| = 0.89$$

For Spin-Lattice relaxation time the value measured was (40.8 + /- 1.1) ms and that obtained from the internet was (40 + /- 2) ms. Using these values, the above was calculated. All other T-tests follow the same format.

## **APPENDIX IV: EXTRA FIGURES**

### Glycerin

Spin-Lattice Relaxation Time



Figure 12: Pulses A & B an Oscilloscope Screen Capture for Glycerin

## Spin-Spin Relaxation Time

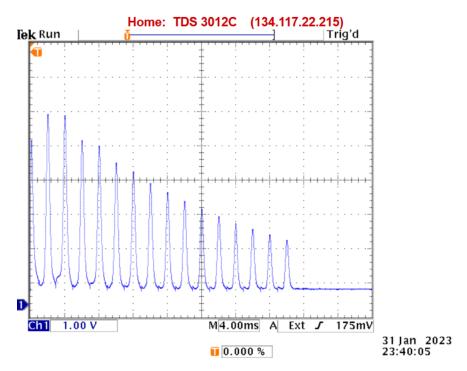


Figure 13: Echo Signal as Captured on the Oscilloscope

#### **Mineral Oil**

Spin-Lattice Relaxation Time

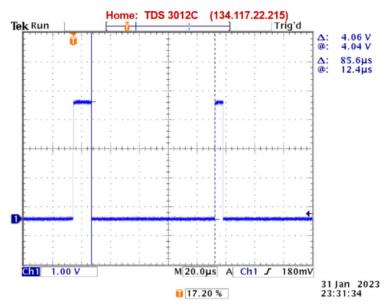
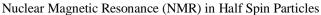


Figure 14: A & B Pulse an Oscilloscope Screen Capture for Mineral Oil



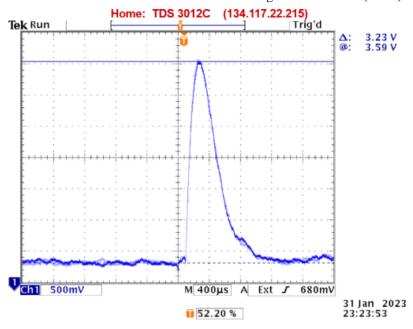


Figure 15: First Free Induction Decay (FID)

## Spin-Spin Relaxation Time

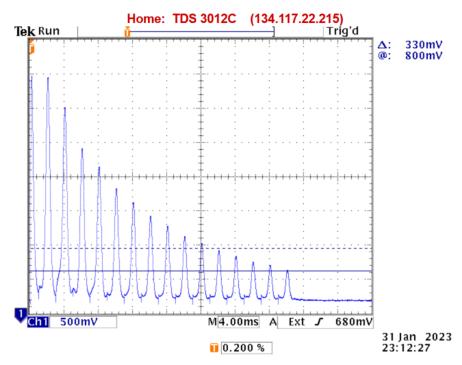


Figure 16: Echo Signal as Obtained on the Oscilloscope