

# Method of Nuclear Magnetic Resonance

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Using Nuclear Magnetic Resonance techniques on a sample of mineral oil it is possible to observe the free induction decay rate of spin states excited away from their magnetization ground states in order to determine the relaxation time of  $T_1$  and  $T_2$  for an oil sample. Theory predicts  $T_2 < T_1$  and in experiment  $T_1$  was found to be  $40.8 \pm 0.4$  ms and  $T_2$  was found to be  $14.0 \pm 0.3$  ms

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## 1. INTRODUCTION

### A. Spin

Nuclear Magnetic Resonance (NMR) is the exploitation of the quantum mechanical effect of spin: an intrinsic angular momentum that all particles possess. Spin has no classical counterpart however, a useful analogy is that spin is supposed to be proportional to the gyration of the particle about the center of mass ???. Although this spin has direction in three degrees of freedom, the method of NMR will constrain it to one. The spin will be classified as *positive* if the spin lies along a chosen axis (Chosen to be  $\hat{Z}$ ) or *negative* if it lies anti-parallel to the axis. Using Dirac notation these spins are classified as being in  $S_z$  with state  $|+\rangle$  or  $|-\rangle$ , respectively. Subjecting our particles to a magnetic field, The  $S_z$  population can be flipped from  $|+\rangle$  to  $|-\rangle$  or vice-versa so long as the magnetic field is oscillating at or close to the Larmor frequency  $\omega_0$ . This behavior was exploited by I.I. Rabi to determine the magnetic moment of nuclei and can be used for spectroscopy because the Larmor frequency is dependent on the energy difference of the two states given by

$$\Delta U = \gamma \hbar B_0 = \hbar \omega_0 \quad (1)$$

where  $\gamma$  is the gyro-magnetic ratio of the sample. This leads to a differentiation technique between different materials leading to spectroscopic use. NMR is not without limitations of course, due to the effect that is being exploited the sample of interest has to have a spin:  $s \neq 0$ . Examples of elements that produce no NMR response are  $^{12}\text{C}$ ,  $^{18}\text{O}$ , and  $^{32}\text{S}$  (Even number of proton and neutrons).

### B. Magnetic Resonance

Placing a sample within a controlled magnetic field allows the determination of the Larmor frequency  $\omega_0$  to induce the state transition described earlier. Sending pulses of this magnetic field for a short duration allows the populations of spin-up and spin-down to go through *Rabi oscillations*???. The analogy of precession of the spin magnetisation around the magnetic field in a reference frame rotating about the Larmor frequency. ?? a  $\pi/2$  pulse is one which flips the net spin magnetization,  $M_0$  to the transverse plane ( $90^\circ$  to  $B_0$ ), a pulse twice as long, a  $\pi$  pulse flips  $M_0$   $180^\circ$  with respect to  $B_0$

### C. Relaxation

Measuring the population relaxation of nuclear spins that return to equilibrium after magnetic resonance gives  $T_1$ ,  $T_1$  is also known as spin-lattice relaxation. This equilibrium state is a

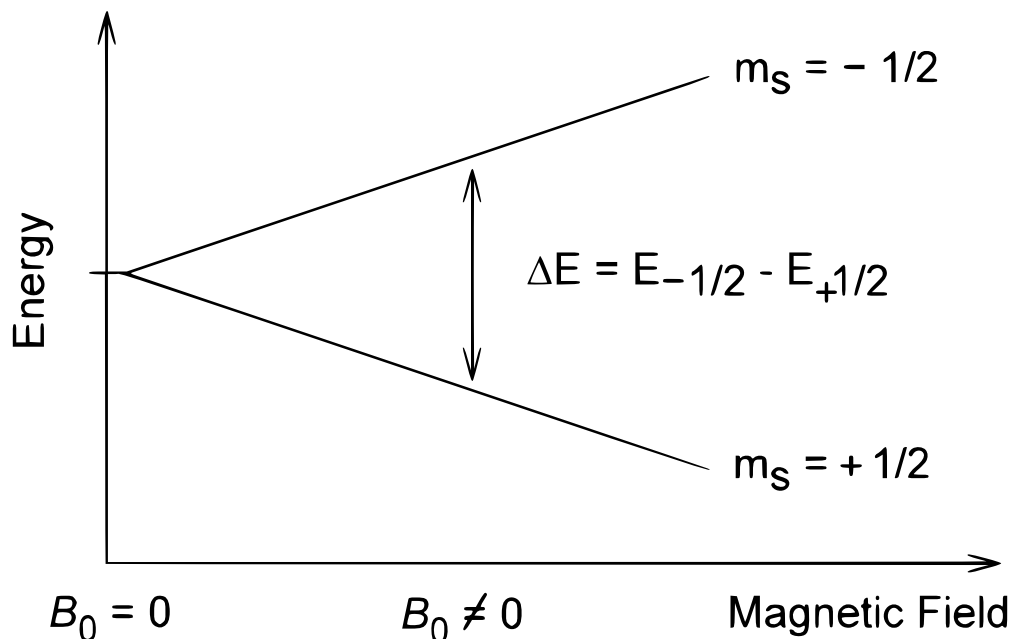


Fig. 1. Splitting of nuclei spin energies in an external magnetic field

superposition of states. Occasionally precessing nuclei fall out of alignment with each other and gradually stop producing a signal. This decay is called  $T_2$  which is the rate of decay of excited states, also called Free-Induction Decay (FID).

#### D. Hahn Echo

Due to inhomogeneous effects from the magnetic field and other elements in experiment, different spin in the sample precess at different rates. This concern for different phases causing dephasing is removed when the  $180^\circ$  pulse is applied however, this inhomogeneous state will rephase some time later to create what is called a Hahn Echo. A technique to measure dephasing time is used and is related to  $T_2$  by exponential decay equation  $e^{-2t/T_2}$  where the inversion pulse is applied after a period  $t$ .

These properties are all combined to give information on the dipole-moments of molecules and other spectroscopic properties of the sample.

## 2. SET-UP AND CALIBRATION

### A. Set-up

For this experiment a study of an oil sample containing  $\text{CuSO}_4$  using a TeachSpin Pulsed NMR Spectrometer. The Spectrometer contains a permanent magnetic capable of producing a magnetic field,  $\vec{B}_0$  of 0.49 T ??; Additionally, it contains a set of coils perpendicular to the magnetic field capable of producing a secondary magnetic field  $\vec{B}_1$  which allow measurement. These fields, and more, are variably controlled by the "mainframe" ?? Lastly, the mainframe is connected to an oscilloscope where the signal peak can be measured automatically and recorded.

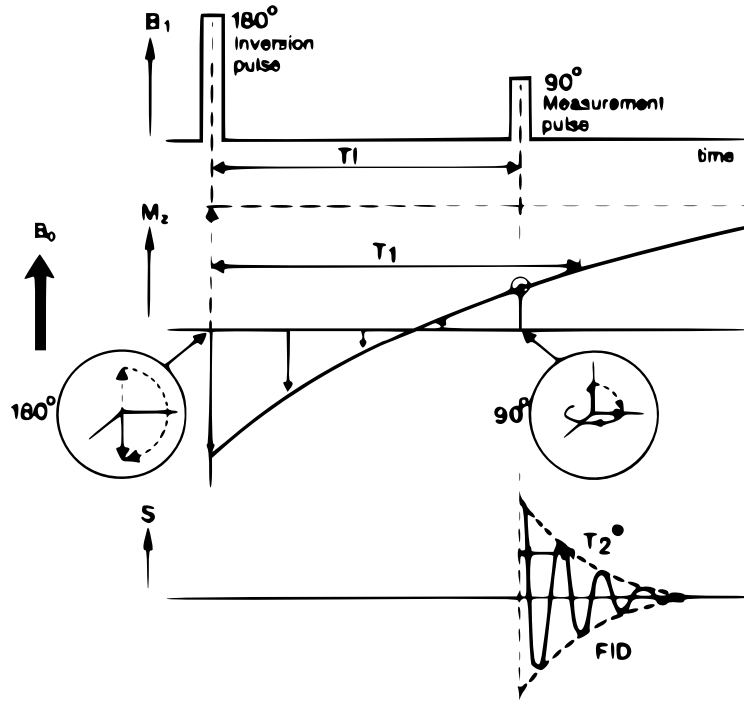
### B. Calibration

When the magnetic field is not at the Larmor frequency, a beat frequency can be seen on the oscilloscope, this beat frequency is minimized to reach the Larmor frequency. The inhomogeneous effect of the magnetic field is reduced by adjusting three degrees of freedom for  $\vec{B}_1$ . Lastly, the  $\pi/2$  pulse is found by maximizing the response of the sample (The  $\pi$  pulse is double  $\pi/2$ ).

## 3. MEASUREMENTS

### A. Measuring $T_1$

An easy way to measure  $T_1$  is by using a two-pulse sequence. First a  $\pi$  pulse is applied to invert the net Magnetization  $\vec{M}$  from  $M_0$  to  $-M_0$ . Then, a  $\pi/2$  pulse is used to induce the transition



**Fig. 2.** The delay of the FID signal produced by the  $\pi/2$  pulse is proportional to  $T_1$

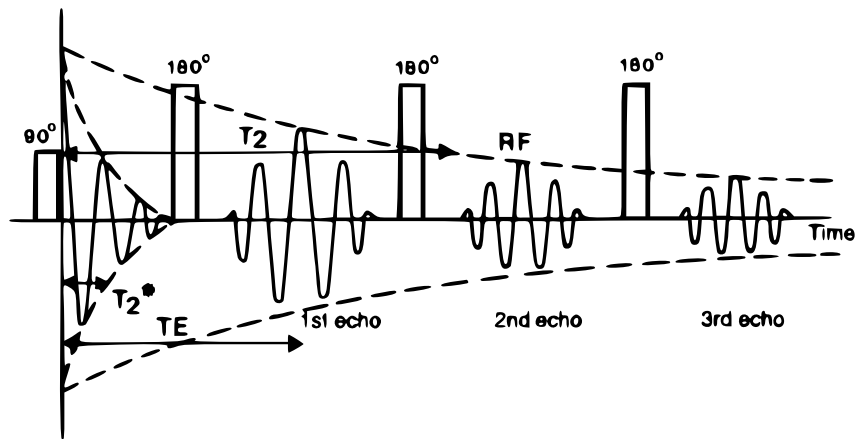
state which will naturally decay. By varying the delay between pulse A ( $180^\circ$ ) and pulse B ( $90^\circ$ ) it is possible to determine  $T_1$  since

$$M_z(t) = M_0 - (M_0 - M_i)e^{-t/T_1} \quad (2)$$

The peak of the FID decay is measured as a function of delay time,  $t$ , and  $T_1$  is determined.

### B. Measuring $T_2$

To easily measure  $T_2$  the Hahn echo effect described earlier is exploited. Following the same measurement technique for  $T_1$  where A is now a  $90^\circ$  and B is now  $180^\circ$ , the echo peak is measured as a function of delay time and  $T_2$  is determined by using  $e^{-2t/T_2}$ . An example using repeated  $\pi$  pulses instead is illustrated in fig. 3, however, delaying the pulse constitutes the same effect.



**Fig. 3.** Multipulse spin echo sequence. Later  $180^\circ$  ( $\pi$ ) pulses refocus the spins to produce additional spin echos following on the first echo. The envelope of the decay follows an exponential in terms of  $T_2$

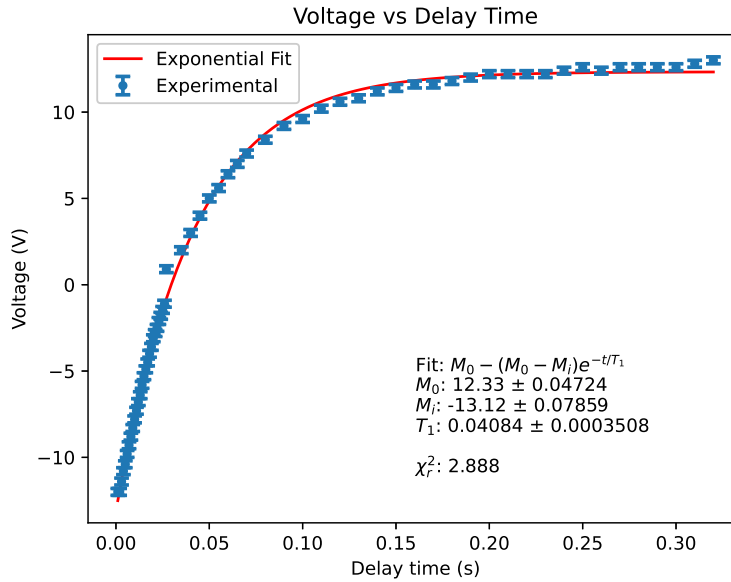
## 4. RESULTS AND CONCLUSION

### A. Calibration

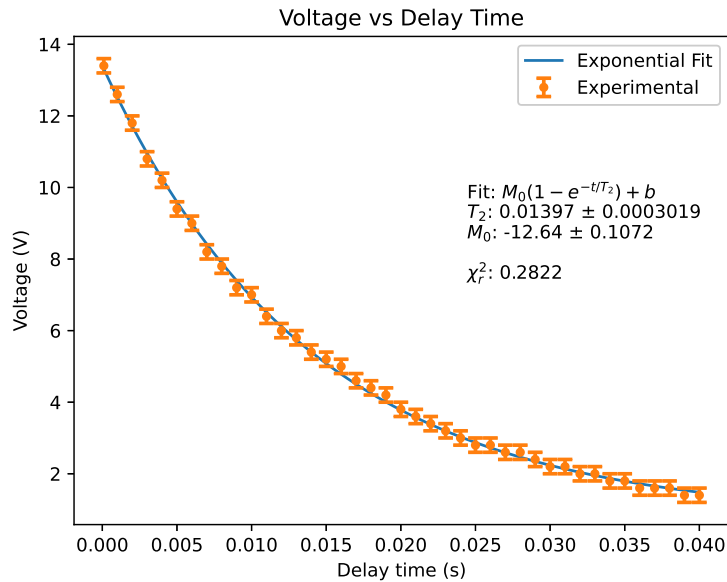
The Larmor frequency was found to be approximately 21.13268 MHz. When finding  $T_1$  Pulse A was found to have a time,  $t$ , of  $\approx 3.22 \mu\text{s}$  while pulse B was  $\approx 6.44 \mu\text{s}$ .

### B. Results

Using the calibration, the methods of measuring  $T_1$  and  $T_2$  were followed to create the following plots.



**Fig. 4.** Voltage vs Delay time to calculate the relaxation time  $T_1$  after a  $\pi$  and  $\pi/2$  pulse respectively



**Fig. 5.** A graph plotting Voltage vs Delay time to calculate the relaxation time  $T_2$  after a  $\pi/2$  and  $\pi$  pulse respectively

### C. Conclusion

The experimental results of the determination for  $T_1$  is  $40.8 \pm 0.3\text{ms}$  while  $T_2$  is  $14.0 \pm 0.3$ . These relaxation times can be used to confirm the molecular make-up of any sample and give information on dipole-dipole interactions and more.

### REFERENCES

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