NMR

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Concepts

- Nuclear magnetic resonance (NMR); pulsed NMR (PNMR), magnetic resonance imaging (MRI).

Background Reading

- Magnetic Resonance Experiments: Melissinos 7.1-7.5 or equivalent
- Pulsed NMR Fundamentals: Teachspin manual

Special Equipment and Skills

LeCroy 9400 Digital Oscilloscope; High-field permanent magnet; radio frequency (rf) pulses, rf-amplifier, mixer, etc.

Precautions

- The high-field magnet is delicate and dangerous. Most metal objects are strongly attracted to the magnet, potentially causing injury. The rare-earth material of the magnet is brittle, and can be damaged by metal objects that are pulled into the magnet.
- The sample can be traversed between the pole pieces of the magnet, but do NOT force the sample probe past its limits of travel. The carriage should work smoothly, do NOT force it.
- The amplifier can easily overheat if it is operated without being attached to the sample probe, or if the pulse duty cycle exceeds 1 % (time on/time off).

Background and Theory

In this project, you will measure the magnetic resonance and characteristic relaxation times (T_1 and T_2) of nuclei in various samples. The resonance frequency of protons (hydrogen nuclei) depends linearly on the magnetic field, according to the equation f_0 (MHz) = 4.258 B_0 (kilogauss). In the experiment, most of the field comes from a permanent magnet that has a field of about 3.6 kG. Small variations of the field (chemical shifts) come from the local environment of each nucleus.

Free-Induction Decay (FID)

The first set of experiments involves tuning the spectrometer, and examining how the nuclear moments relax towards an external field via the spin-lattice relaxation time (T_1). Mineral oil has a convenient value of $T_1 \sim 10$ ms. Obtain (or prepare) a sample of mineral oil that is roughly spherical; see Fig. 2.3 pg. 30. The O-ring near the top of the vial is used to position the sample in the spectrometer.

I. 90^0 pulse FID (pg. 28-29).
1. Connect equipment as shown in Fig. 1.3 pg. 28. Use settings given at the bottom of pg. 29.
Typical oscilloscope settings: Time/Div 20 μs, Ch1 1V/cm, Ch2 2 V/cm, Trig: Ext, 1 V, POS
2. Adjust "Tuning" until the FID (detector out, channel 1) is maximized.
3. Use the "Frequency Adjust" until the beat oscillations (mixer out, channel 2) are minimized.
4. Adjust "A-width" for a 90^0 pulse (maximum FID).
5. Repeat 3-5 to ensure optimal values.

6. Now readjust the “Frequency Adjust” to observe beat oscillations from channel 2. Sketch these oscillations. Describe the mechanism involved (what do these beat oscillations come from). Estimate the time for the oscillations to diminish to 1/e of their initial amplitude (this is a crude estimate of the spin-spin relaxation time T_2).

II. Other pulse lengths (pg. 31).

In Part I-5, the “A-width” is adjusted to give a 90^0 pulse. Now you will examine the behavior for other pulse lengths.

1. Start with the settings for the 90^0 pulse, with beat oscillations, from I-6.
2. Increase the “A-width.” Describe the behavior of the oscillation from channel 2. How do you identify a 360^0 pulse width?
3. Sketch diagrams of the oscillations from channel 2 for key values of the pulse lengths: 90^0, 180^0, and 360^0. Explain the observed behavior.

Spin-lattice relaxation time (T_1)

(section G, pgs. 31-33).

III. Single Pulse

1. Start with the settings for the in-tune 90^0 pulse from I-5. Increase the “Repetition Time” to ensure that the initial amplitude (from “Detector Out” channel 1) is maximized.
2. Then decrease this “Repetition Time” and examine the reduction in amplitude.
3. A crude estimate of T_1 is given by the “Repetition Time” that gives an initial amplitude of about 1/3 (1/e) of its maximum value.

IV. Two Pulses, Zero Crossing

1. Return the repetition rate to ~100 ms, and use the settings for the in-tune 90^0 pulse from I-5.
2. Turn on the B pulse (B on, B pulses=1); start with a small delay between pulses (e.g. 0.2 ms).
3. Adjust the oscilloscope until both A and B pulses can be observed.
4. Adjust the “A-width” until the amplitude from the first pulse is minimized (180^0 pulse).
5. Adjust the “B-width” until the amplitude from the *second* pulse is *maximized* (90^0 pulse).
6. Switch “Sync” to B to focus on the amplitude from the second pulse.
7. Adjust the “Delay Time” (τ). The value for T_1 is the time that minimizes this amplitude.
8. Obtain an accurate value for T_1 by measuring the amplitude of the second pulse as a function of τ around the minimum, and fit the curve to a quadratic function to find the minimum.

Spin-spin relaxation time (T_2)

(section H, pgs. 33-34).

V. Two-pulse spin echo

1. Start with both A and B pulses as in section IV, but with opposite types of pulses as follows.
2. Adjust the “A-width” until the first amplitude is maximized (90^0 pulse). Note that the initial value of the second amplitude should be minimized.
3. Adjust the “B-width” until the second amplitude is maximized at a *later* time (180^0 pulse).
4. Obtain values for the second amplitude as a function of total “Delay Time” (2τ). Use the cursor for Channel 1 to obtain accurate values of the amplitude.
5. Fit your data to an exponential decay to obtain the spin-spin relaxation time T_2.

VI. Carr-Purcell multiple pulse spin echo sequences
1. Start with the settings of part V, but with three B pulses. Be sure the CPMG switch is off. Diminish the repetition rate to avoid overheating the rf amplifier. Adjust the "Delay Time" and oscilloscope settings until you can observe a sequence of large-amplitude, time-separated pulses.
2. Add more B pulses, and adjust the oscilloscope until you can see the decay in their amplitudes.
3. Again obtain values for the second amplitude after each pulse as a function of total "Delay Time" (from the very first 90^0 pulse).
4. Fit your data to an exponential decay to obtain the spin-spin relaxation time T_2.

VII. Meiboom-Gill sequence.
1. Turn ON the Meiboom-Gill switch (CPMG), and repeat sections VI-2-4. How and why does the Meiboom-Gill value for T_2 differ from the Carr-Purcell value you obtained in section VI.

VIII. Choose one of the "Experiments" (e.g. T_1 and T_2 in B glycerin, or D petroleum jelly).