NMR Laboratory

Session #1: Measurement of Spin-Lattice Relaxation Time T_1 and Spin-Spin Relaxation Time T_2

Goals: understand and apply simple pulse sequences to determine the T_1 and T_2 relaxation times in proton nuclear spins in glycerol.

Measuring T_1 by the *Inversion Recovery* method

1. Read sections 2.1.1-2.1.4 of the experimental manual to understand the concepts of a free-induction decay, the static field B_0, the rf field B_1, the equilibrium magnetization M_0, the longitudinal magnetization M_Z, the transverse magnetization M_{xy}, Π and $\Pi/2$ pulses, and the T_1 and T_2 relaxation times.

2. The TA should have set-up the NMR instrument so that the magnetic field is locked and the software is running in the spectroscopy mode. (Losing the locking may cause the software stops responding. During the whole lab, always remember to make sure the lock on light is on.)

3. Insert a NMR tube with a large volume of glycerol into the magnet. We are using glycerol for these experiments because glycerol has a large number of protons and a relatively short T_1 relaxation time.

4. Load the 1P_X method. The parameters should be Receiver Gain=50 dB; Time Const=30 microseconds; Acc=4; Phase Detection; Dwell Time=20 microseconds; NOP=512; Channel I and Channel Q; X_1=3.4 microseconds; R=600 msec. Adjust the phase of the receiver so that the in-phase (I) signal is at a maximum at short times and the quadrature (Q) signal is at a minimum. . Follow the experiment on p. 48 of the Experimental Handbook, and refer to pages 16-18 for procedures to obtain an on-resonance FID and determining the lengths of the Π and $\Pi/2$ pulses. Be sure to save all raw data (data before performing IR routine) and processed data in both the default format, as well as an exported text file.

5. Save the data of this off-resonance free-induction decay (FID) and record the value of Δf_0. Adjust the magnetic field to obtain an on-resonance FID. Save the data record the value of Δf_0. Does the beat frequency in the off-resonance FID match the <u>difference change</u> in Δf_0?

6. Read section 3.1, 3.1.1-3.2.1 and sections 3.2.1 of the experimental manual on the *inversion recovery* method. (We will be using MatLab to do the data analysis, not the Data Processing parts of the NMR software package.) Use the Π and $\Pi/2$ pulses you obtained in part 3.

7. Use the *inversion recovery* method to measure collect data that you can use to determine T_1 in glycerol by analyzing how rate at which the magnetization recovers. Remember, repetition time T_R should be five times longer than expected T_1. If you don't know T_1 beforehand, increase T_R to see if the signal increases at the same time. If yes, then the current T_R is too small so that when you apply the next pulse, the neutrons don't have enough time to restore their original configuration. Use 'Acc' to acquire data and use 'Proc' for data process.

Measuring T_2 with the *spin echo* method

1. Read section 2.1.5 and section 4.1 on spin echoes and the measurement of T_2 free from artifacts introduced by inhomogeneities of the magnetic field.

2. Follow the procedure in section 4.2.2 on measuring T_2 using the simple two-pulse sequence and collect data of the type shown in Fig. 48 for glycerol. Fit the data to an exponential decay to determine T_2 using equation on page 65.

Session #2: Using the spin-spin relaxation time T_2 to probe polymerization kinetics

Measure T_2 for monomer and polymer

1. Read the brief discussion at the beginning of section 2.2.5 on the difference between liquid-like and solid-like samples.

2. Measure T_2 for a dicyclopentadiene (DCPD) monomer sample using the same procedure as above.

3. Measure T_2 for a cross-linked (polymerized) DCPD sample using the same procedure as above. If needed, get assistance from the TA on optimizing the parameters for the data acquisition.

Monitor changes in T_2 during polymerization

1. At this point, ask the TA for assistance in preparing a sample. To prepare the sample, we add a "Grubb's Catalyst" to the neat DCPD monomer, which begins the polymerization reaction. Once the catalyst is dissolved in the monomer, start a timer and place the sample in the NMR.

2. At t=0, i.e. before the polymerization reaction begins, pick a delay time τ in the two-pulse sequence and record the amplitude A of the echo.

3. As the polymer reacts, keep the same τ and measure how the amplitude of the echo changes. Approximately once every minute, record the value of A and the time since the beginning of the reactions.

4. Plot A vs. time and relate that to the change of T_2. As viewed by the NMR spins that are precessing at 15 MHz, how long does it take for the polymerization reaction to finish?