

## APPENDIX A

### “Instructions & Examples of Experiments”

Source: Borislav Pavlov (2009)



# Teach Spin NMR/CW Spectroscopy Instructions and Experiments

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## 1 Equipment overview

In this NMR setup both pulse and continuous wave experiments are possible. Pulsed NMR is done through pulse length modulation to determine the characteristics of the pulse, and continuous wave NMR is done through magnetic field modulation. Both methods operate at a constant pre-set frequency.

### 1.1 Tuning the spectrometer

To set the frequency of the pulse, use the **F** setting on the **Synthesizer**. It is important to set this parameter to the resonant frequency of the sample. The best way to find and observe that frequency is to calculate it using

$$\omega_o = \gamma B_o \quad (1)$$

Where  $\gamma$  is a property associated with each substance and  $B_o$  is the magnetic field of the magnets (i.e. 0.5 T). After calculating  $\omega_o$ , using

- $\gamma_{proton} = 2.675 \times 10^8$  rad/sec-Tesla
- $\gamma_{fluorine} = 2.517 \times 10^8$  rad/sec-Tesla

the **tuning capacitors** on the magnets are used. This method is used to tune the sample coil to the desired frequency of the synthesizer (sample coil is the name used for the sample chamber).

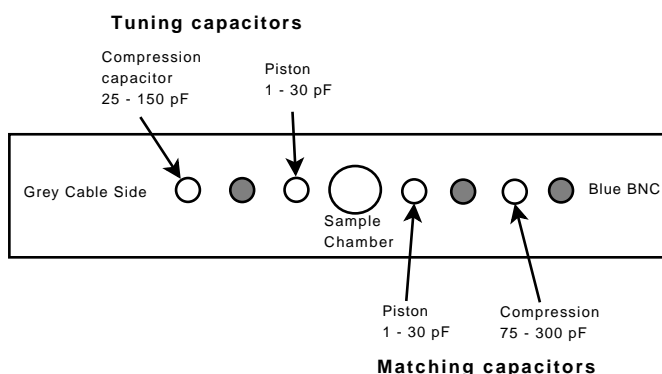


Figure 1: An overhead look of the magnet setup

Following diagram 1, the **plastic screwdriver** provided is used to turn the screws on the variable capacitors. In order to determine if the sample coil is tuned, an RF probe is used. This RF probe is the only measure that can be obtained **DURING** the pulse. NMR spectroscopy is a measure of the effects **AFTER** a pulse is applied. Thus a pulse A has to be applied during the process.

Firstly, connect the RF probe to CH1 of the oscilloscope and insert it in the sample coil (NOTE: the coil of the RF probe must be an equal distance away from the O-Ring stopper, as is a sample in a sample holder; thus compare the RF probe coil with a provided sample holder and put the O-ring on the probe about 39mm away from the coil). Set the frequency on the synthesizer to the previously calculated  $\omega_o$  and observe the oscilloscope. Set the correct oscilloscope dimensions, and also adjust to proper triggering. At the same time, tune the capacitors until a signal is observed on the oscilloscope. NOTE: be very careful with the Compression capacitor as it is very sensitive. Once a small signal is seen on the screen, switch to the fine tuning capacitor (piston) and adjust until the observed signal is  $\approx 45V$  peak to peak.

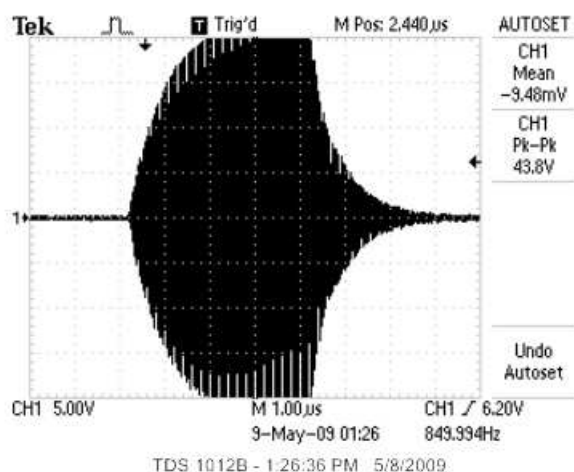


Figure 2: Observed RF probe signal at resonance

The above figure 2 is an example output of a correct setting of the capacitors, matching the resonant frequency set by the synthesizer. Below are further instructions on

how to tune the spectrometer EXACTLY to the desired frequency.

## 1.2 Pulse lengths and more tuning

Since this spectrometer only allows for pulse length modulation and not amplitude modulation, parameters **A.len** and **B.len** (located on the **Pulse Programmer**) are used to specify the properties of the pulse.

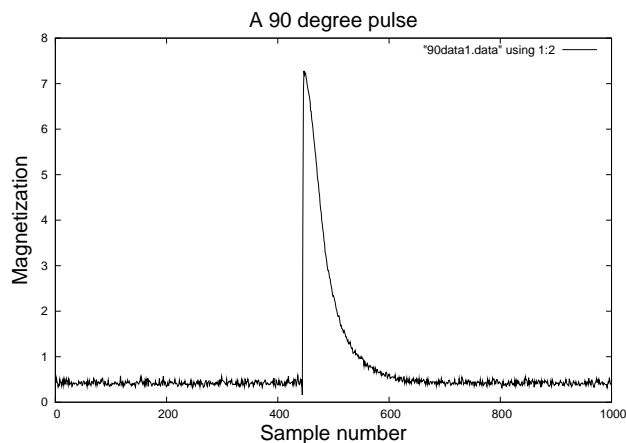


Figure 3: A ninety degree pulse is characterized by the tail

Here in figure 3, the magnetization in the X-Y plane is plotted, after a 90 degree pulse is applied. In order to be classified as a 90° pulse, a 'tail' has to be present. This tail represents the overall magnetization in the X-Y plane, which decays exponentially as the spin becomes random. Initially, when the pulse is applied, the overall magnetization is shifted to the x-y plane, and thus measured on the oscilloscope. At a later characteristic time, the overall magnetization becomes zero, since the vectors return to the initial state.

Where the parameters **A.len** and **B.len** come into play is to determine the type of pulse sent to the sample. By adjusting the length of the pulse (in figure 3 only the A pulse is present), one can determine the amount of magnetization that is applied to the atoms. The following principle is valid:

Start the **A.len** parameter at minimum, and increase it until you find the first 90° pulse (maximum amplitude). In order to make a 180° pulse, double the value of **A.len**. Thus the pulse rotates the magnetization for double the time, allowing a 180° turn.

NOTE: the 90° pulse could be  $\pm 90^\circ$ , thus it could be sometimes thought of as a 270° pulse.

In order to determine the direction of the pulse a more accurate approach is taken. Firstly, let me explain the principle about the description above. The oscilloscope is connected to the **Env Out** on the **Receiver** in order for the above to be observed. What is actually happening is that the Env Out is displaying the envelope surrounding the **I** and **Q** outputs, which are pulses A and B respectively. Those are called **Phase Sensitive Detectors**. In order to perfectly tune the spectrometer and display the pulse orientation, one has to connect the **Ref Out** on the **Synthesizer** to the **Ref In** on the **Receiver**. Then the outputs from **I** and **Q** can be observed. Here, very careful tuning is required.

The spectroscope should always be properly tuned before each experiment.

Firstly, the outputs from either I or Q should be connected to the oscilloscope and carefully examined. If the synthesizer is not tuned to the exact frequency, the outputs from either I or Q will look like this (using a 90° pulse):

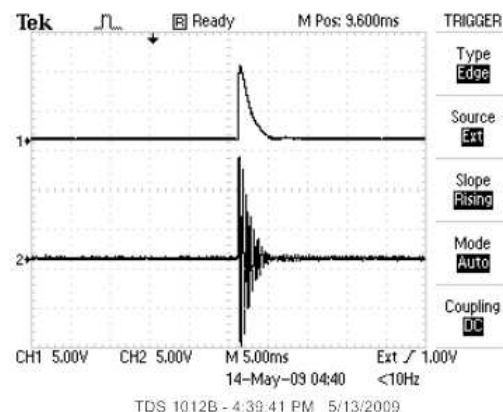
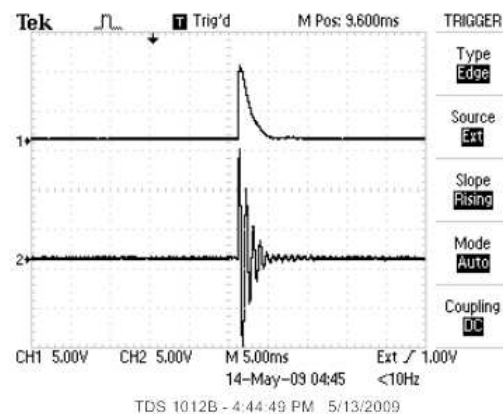
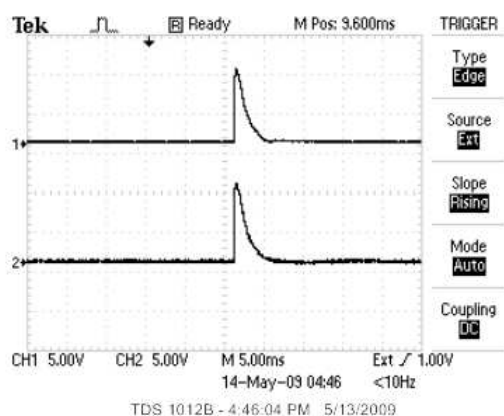
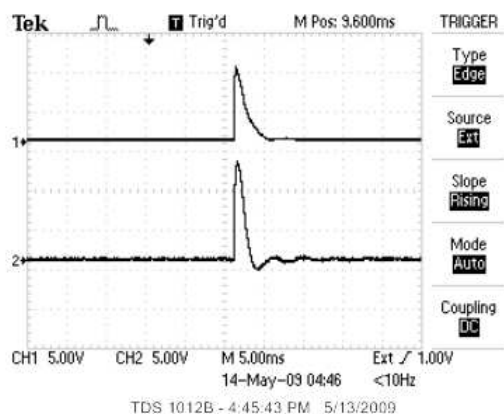
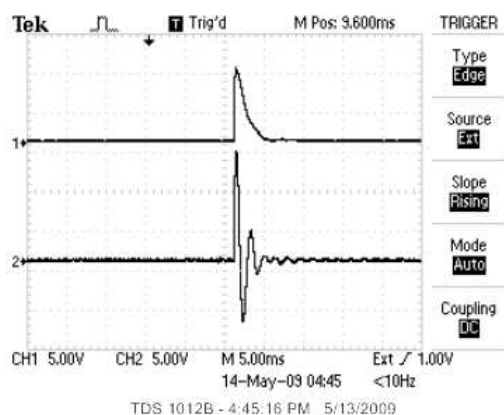


Figure 4: **Channel 1:** Env Out. **Channel 2:** Q Out

By adjusting the synthesizer frequency (in the order of 100Hz variations), the signal gradually evolves.





In the above figures, a time lapse has been shown, presenting the Q out as the synthesizer is gradually tuned. Ultimately, there should not be any oscillations. Now clearly the direction of the output pulse is shown. The absolute direction is irrelative right now, but for later experiments, the relative direction matters.

The next step is to again retune the spectrometer to get near perfect results. Since the  $180^\circ$  does not give a FID signal, the spectrometer has to be tuned so that no signal

is observed. This is necessary, since often the minimum FID amplitude that can be achieved (corresponding to a  $180^\circ$  pulse), is not zero. The way to tune is the following:

1. Tune the spectrometer to resonance (as described above) and obtain the best  $180^\circ$  pulse possible
2. Adjust the fine tuning capacitor in the RF sample probe a small amount (remember which way you changed it!)
3. Change the pulse width and see if the 'tail' of the FID after the  $180^\circ$  pulse is smaller or larger. If it is smaller, continue to adjust the fine tuning capacitor in the same direction. If larger, switch directions.
4. Keep changing the pulse width and the tuning capacitor until you see no tail after the  $180^\circ$  pulse.

Near perfect results can be obtained. This is the best way to tune the spectrometer for all pulsed experiments.

Temperature stability is EXTREMELY important as the precession frequency (discussed above) will drift as the magnets oscillate in temperature. By adjusting the knob to the desired temperature, one can stabilize the magnets to the desired state. The actual temperature reading is unimportant, as long as the magnets remain of stable temperature. Next to the knob is a switch with OPEN and CLOSED written on it. Since the temperature control consists of a feedback loop, those states determine if the feedback loop is open or closed, in other words OFF and ON, respectively.

**THE CONTROL SHOULD BE SET TO OPEN WHEN THE MAGNET IS NOT USED FOR A LONG PERIOD OF TIME, I.E. OVERNIGHT.**

The LED indicator next to each knob represents the current state of the magnet. If the magnets temperature is above the desired temperature, the LED is red. If below, the LED is green. When the desired temperature is reached, the LED is off.

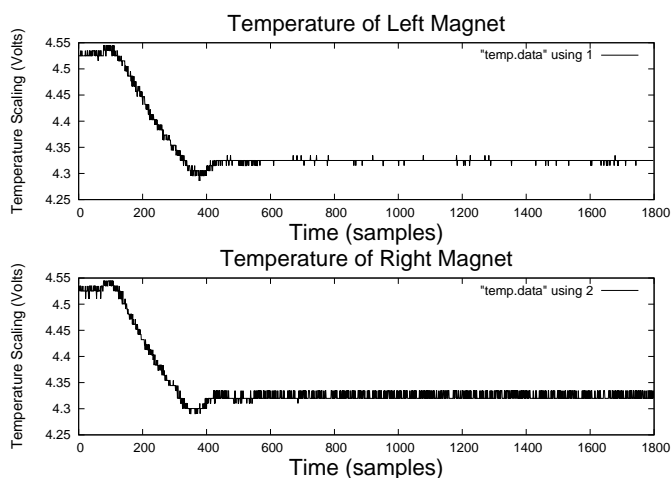


Figure 5: Cooling of the magnets. Note the overshoot

As is seen in figure 5 the temperature needs some time to stabilize. A reasonable amount of time should be allowed for the PID controller to completely stabilize the temperature, 30-40 mins is a good approximation. The dials have an upper limit of 99 but the time taken to reach this setting is unnecessarily long. The following diagram 6 took 40 minutes to produce and shows the heating of the magnets. Overall, the temperature was increased by approximately 40° C.

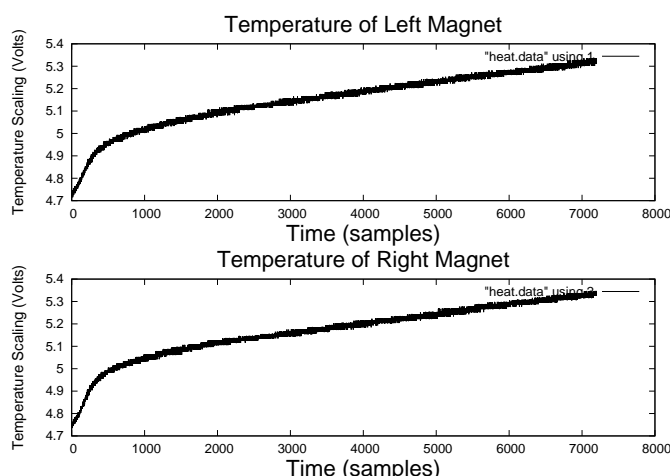


Figure 6: Heating of the magnets for 40 minutes

The user should not go to very high or very low temperatures, since there are heatsinks mounted on the magnets, thus interacting with the environment. If the temperature is set too high/low, the time to stabilize and the quality of the stabilization will be a big source of error.

The precession frequency observed will drift with magnet temperature, thus the more stable the magnet temperature, the better the experiment results will be. So make sure you keep adjusting the frequency on the synthesizer so that no oscillations are observed.

Another setting to be adjusted, which provides for more accurate results is the **Magnetic Field Gradients**. These knobs are located on the **PS2 Controller**. By combining them in different manners, one can obtain an uniform magnetic field, which will avoid abnormalities in the results. When these are set so that the decay time after a 90° pulse is at its longest, and looks like an exponential, then the magnetic field is the most uniform. NOTE: It is preferable to adjust those settings when the temperature is stabilized. Each knob on the PS2 Controller has a lock switch, which locks the dial in place. It is located on the left of each dial.

For the experiments done with one proton atoms the **BAND** switch, located on the **Receiver** must be set to **p**, and for all fluorine experiments to **f**.

### 1.3 Measuring $T_2$

The following figure was obtained while performing the spin-echo experiment, for the Light Mineral Oil sample. Here it is EXTREMELY important to cool the magnets and let them stabilize. **This experiment does not work with the temperature setting on OPEN.** Each magnet was cooled down so that the setting on the temperature knob was  $14 \pm 0.1$ . The experiment consisted of applying a 90° A pulse followed by a 180° B pulse, a time  $\tau$  later. The parameter  $\tau$  is located on the Pulse Programmer.

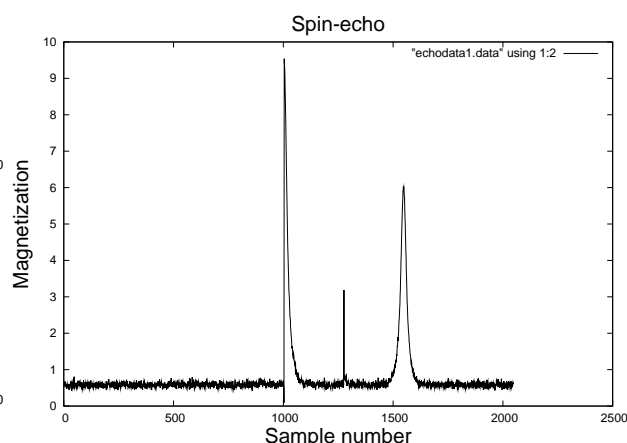


Figure 7: The 180° pulse is applied a time  $\tau$  following the 90° pulse. An echo occurs a time  $2\tau$  later.

The theory behind the phenomenon observed in figure 7 is the following: A 90° pulse rotates the thermal equilibrium magnetization  $M_0$ , initially in the z-direction, to the y-axis. The magnetization in the X-Y plane decreases because some of the particles are in a higher field and some are in a lower field, thus cancelling out. The spins are rotated 180 degrees and the magnetization forms an echo a time  $2\tau$  later. (Refer to the manual I-11)

Applying a sequence of 180° pulses allows us to compute the characteristic time  $T_2$ . This is the time it takes for the FID echo amplitude to drop to 37%. (This is best done by collecting the data and extracting the maxima, and fitting an exponential to it). From the Carr-Purcell principle, we can easily compute the time  $T_2$  by applying a number of B pulses of 180° following an A pulse of 90°. To determine the exact length of the A and B pulses, observe the I or Q outputs. To get a 90° pulse, the amplitude of the FID must be at a maximum.

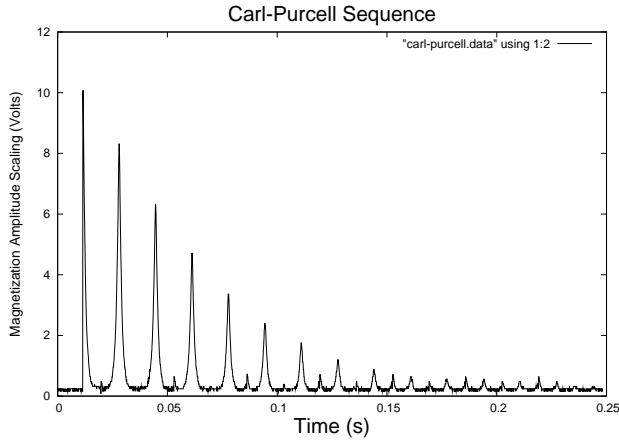


Figure 8: Carr-Purcell principle of multiple echoes. Note the sizes of both the echo and the  $180^\circ$  pulse towards the end of the sequence.

In figure 8 a number of B pulses were applied following the A pulse, and separate echoes are observed. The advantage of such a procedure is big. Here, the user does not need to vary the time  $\tau$  between the pulses to obtain data for the exponential decay of the echo amplitude, outlined in 9.

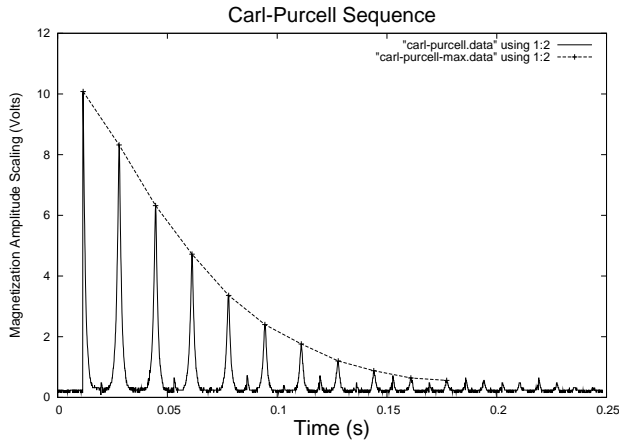


Figure 9: Exponential decay of the echo amplitude

Just as an example, figure 10 demonstrates a variation in  $\tau$ , the separation time between pulse A and B.

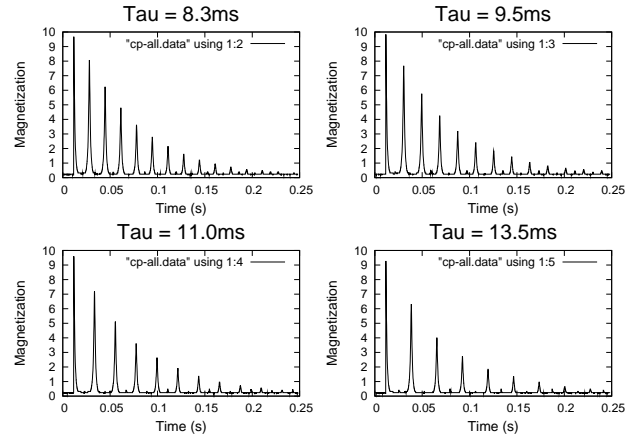


Figure 10: Exponential decay of the echo amplitude

It is clearly justified that the echo amplitude follows the same exponential law, for numerous separation times (maybe a good exercise would be measuring the  $T_2$  for different  $\tau$  and taking an average to minimize the error). This exponential law governing the echo amplitude is given by:

$$M_{xy}(t) = M_o e^{-\frac{t}{T_2}} \quad (2)$$

where  $M_{xy}$  is the magnetization measured. From this equation one can determine the characteristic time  $T_2$ . For the Carr-Purcell sequence, the  $T_2$  measured for Light Mineral Oil was 0.088 seconds. The error on this measure is undefined. It is definitely not the measurement given by the fitting program, as discussed later on.

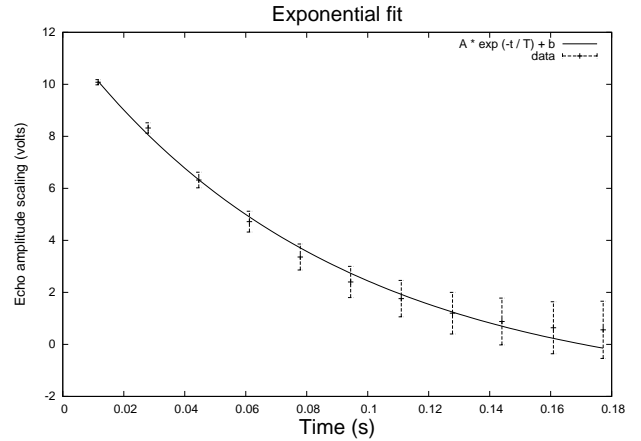


Figure 11: Exponential fit to the Carr-Purcell sequence of echoes

It is essential to notice in figure 11, that the errorbars are increasing. This is a fundamental problem when measuring the echo amplitude while performing the Carr-Purcell sequence. The way the spectrometer works, doesn't allow

us to know precisely at what angle we are rotating the magnetization, thus the measure of  $M_{xy}$  is a measure resulting from an approximate pulse size. This means that we can never be sure if the pulse is exactly  $180^\circ$ , for example. Thus, if pulse B is less than  $180^\circ$  by  $\Delta\theta$ , the difference at the  $n$ -th B pulse will be  $n\Delta\theta$ , resulting in an attenuated echo (maximum echo is achieved at exactly  $180^\circ$ ). Another thing to take into account is the arbitrary error that is selected. The value measured on the oscilloscope, and the data collected, is a result of an approximated rotation due to an applied pulse length and the data is averaged over 16 repetitions. Thus determining the error is not trivial, if we also have to take into account magnet stability, temperature, etc. Here the shown error is derived from the overall oscillations on the  $90^\circ$  pulse after averaging by 16. This is subject to further discussion.

Going back to the accumulated rotation problem, there is a clever solution. If the first  $180^\circ$  pulse (or approximately) is applied in one direction, but the following is applied to the opposite direction, the magnetization will not diverge and there will not be an accumulated error. This is called Carr-Purcell-Meiboom-Gill sequence.

In order to achieve the Meiboom-Gill correction, toggle the **MG** switch to **ON**, located on the **Pulse Programmer**.

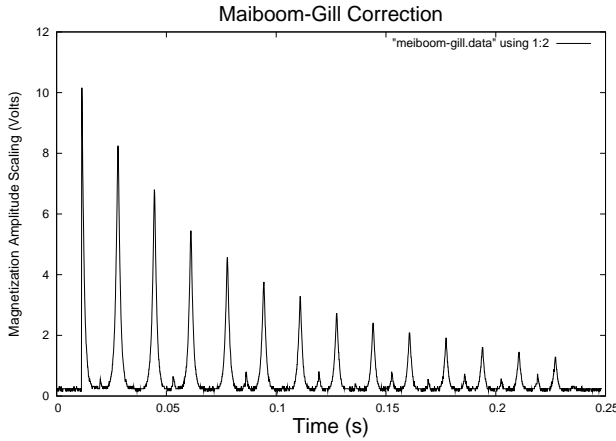


Figure 12: Carr-Purcell-Meiboom-Gill sequence

It is important in both Carr-Purcell and Meiboom-Gill sequences to allow for a long period **P** (located on the **Pulse Programmer**) of repetition. Here a period of 1 second was allowed for the Meiboom-Gill sequence. If a shorter period is applied, attenuation of the initial  $90^\circ$  pulse occurs, due to saturation, i.e. the magnetization is not left enough time to neutralize.

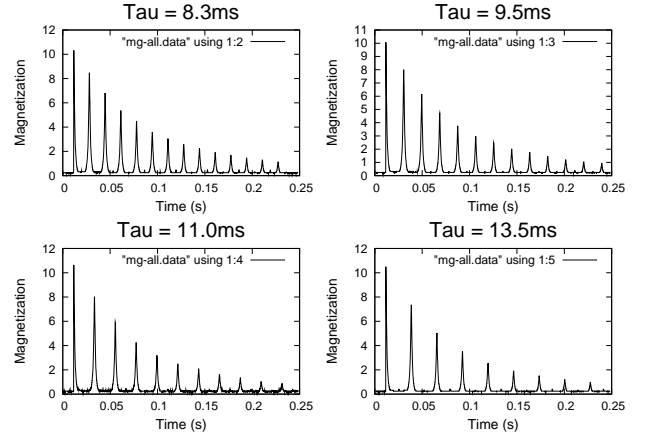


Figure 13: Carr-Purcell-Meiboom-Gill sequence for different  $\tau$  values

Again in figure 13 it is seen that the exponential shape stays constant for different  $\tau$  values. Here the  $T_2$  was measured to be 0.091 seconds. Here, the error is not the same as for the Carr-Purcell sequence. It stays constant, and can be approximated based on the oscillations of the peaks on the oscilloscope. I suggest an error of  $\pm 0.001$ , to account for various factors, such as temperature, pulse length, etc. These values obtained are too high, since usually  $T_2$  is less than or equal to  $T_1$  (seen later), thus the experiment was repeated with a better tuning. The above is just to demonstrate the method of operation.

**For all echo experiments, the Meiboom-Gill sequence should be used, as it provides a more correct value for  $T_2$ .**

Here are the values obtained for all the samples, note that the error that the fitting program is based on the error observed on the oscilloscope, and does not take into account the temperature, gradient and apparatus related errors. Thus the calculated value is meaningless as it is too small. The experiment to obtain this data consists of only a two pulse spin echo sequence and is based on the law:

$$M_{xy}(2\tau) = M_0 e^{-\frac{2\tau}{T_2}} \quad (3)$$

Remember to take the double of the obtained characteristic time (data is  $\tau$  versus echo amplitude) since  $\tau$  is the separation between the A and B pulse. Here we are interested in the separation between the A pulse and the echo occurrence. Thus after obtaining the characteristic time with respect to  $\tau$ , the actual characteristic time is with respect to  $2\tau$ .



| Sample            | $T_2$ (seconds) |
|-------------------|-----------------|
| Heavy Mineral Oil | 0.0273          |
| Light Mineral Oil | 0.0505          |
| Glycerol          | 0.0221          |
| FC-43             | 0.0182          |
| FC-70             | 0.0114          |
| FC-770            | 0.0198          |
| Galden Perfluor   | 0.0321          |
| HT-110            | 0.0312          |

#### 1.4 Measuring $T_1$

In order to measure the other characteristic time, an inverse sequence to that of the spin-echo is used. A  $180^\circ$  pulse is applied followed by a  $90^\circ$  pulse. Here, not a sequence of pulses is used, but only two. The amplitude of the FID occurring as a result of the  $90^\circ$  pulse is plotted as  $\tau$  is varied, and if the spectrometer is properly tuned, and the temperature is perfectly stabilized the following shape is observed on the Q out pulse (the B pulse).

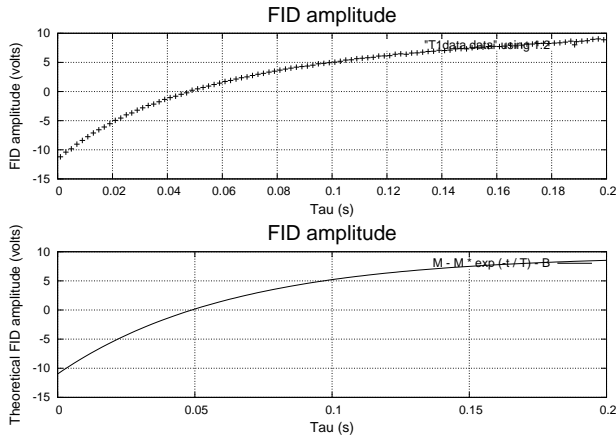


Figure 14: The FID amplitude allows us to measure the Spin-Lattice relaxation time, which is the time constant of the exponential curve shown

The above figure 14 was obtained for Light Mineral Oil. The differential equation governing the exponential curve is the following:

$$\frac{dM_z(t)}{dt} = \frac{M_o - M_z(t)}{T_1} \quad (4)$$

where  $M_o$  is the initial height of the FID (i.e. a  $90^\circ$  pulse alone). Solving with the correct initial conditions, this gives an exponential of the form

$$M_z(t) = M_o(1 - 2e^{-\frac{t}{T_1}}) \quad (5)$$

The characteristic time  $T_1$  can either be computed by a fitting program or a trick can be used. When the FID amplitude crosses the x-axis, this corresponds to  $M_z$  being zero,

thus the following equation is derived [1]:

$$\tau = T_1 \ln(2) \quad (6)$$

From the data obtained, a value for  $T_1$  was found to be 0.063 seconds. Again, here the error depends on numerous things and can be approximated from the oscillation amplitude on the oscilloscope and the display sensitivity. I suggest a value of  $\pm 0.005$ .

When computing the values for  $T_1$  and  $T_2$ , note that the magnets should be set to near room temperature, to minimize precession frequency drifting. Also, let a considerable amount of time before starting the experiment for magnet temperature stabilization. I suggest that all measures be taken with the **AVERAGE** function on the oscilloscope as the magnetization oscillates a considerable amount.

Alternatively, there is a second method to measure  $T_1$ , although this seems to be less precise and would require more programming work. If the  $180^\circ$  pulse is followed by numerous small degree pulses, then the following is traced out:

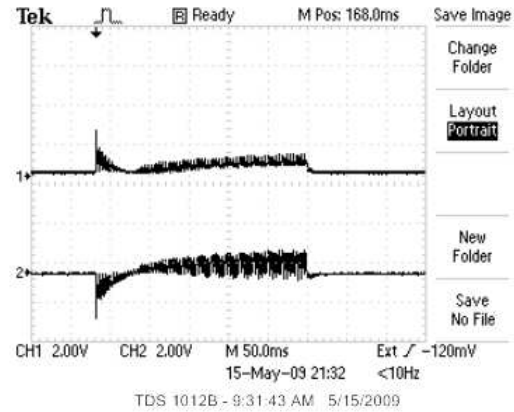


Figure 15: Small-tip sampling method for  $T_1$  measurement

An important factor in the method shown in figure 15 is the choice of  $B_{len}$ . The size of the B pulse should be large enough to be measured, but small enough so that the reduction in  $M_z$  is negligible. If the length of the B pulse can be associated with the exact angle of rotation, then the following formula applies for the error on the measurement:

$$error = (\cos\theta)^n \quad (7)$$

where  $\theta$  is the tipping angle and  $n$  is the number of pulses. The following data was taken for distilled water, notice the difference in timescales:

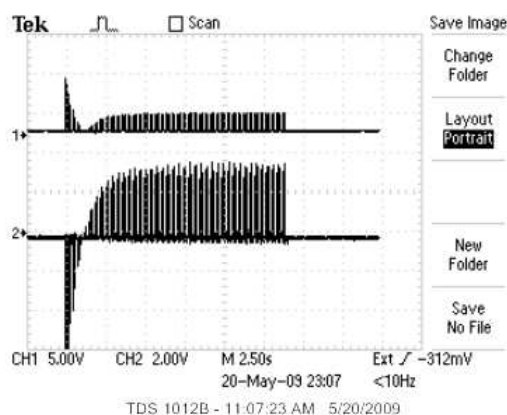


Figure 16: Small-tip sampling method for  $T_1$  measurement, timescale is 2.5s per division

In both cases channel one on the oscilloscope is measuring the Env Out signal and channel two is measuring the Q pulse. Here, fine tuning is also required. After examining the close-up of the above experiment, a time  $T_1$  of distilled water is estimated to be around  $2 \pm 0.2$  seconds. Here I used equation 6 where  $\tau$  is the zero crossing time.

Here is a table of results, summarizing obtained data for  $T_1$  for the provided substances.

| Sample            | $T_1$ (sec)       | $T_1$ (sec)<br>Small-tip |
|-------------------|-------------------|--------------------------|
| Distilled Water   | —                 | $2.0 \pm 0.2$            |
| Heavy Mineral Oil | $0.035 \pm 0.005$ | $0.032 \pm 0.004$        |
| Light Mineral Oil | $0.067 \pm 0.005$ | $0.065 \pm 0.005$        |
| Glycerol          | $0.032 \pm 0.005$ | $0.023 \pm 0.004$        |
| FC-43             | $0.28 \pm 0.02$   | $0.27 \pm 0.04$          |
| FC-70             | $0.13 \pm 0.02$   | $0.14 \pm 0.06$          |
| FC-770            | $0.36 \pm 0.02$   | $0.51 \pm 0.04$          |
| Galden Perfluor   | $0.36 \pm 0.02$   | $0.61 \pm 0.05$          |
| Galden HT 110     | $0.36 \pm 0.02$   | $0.57 \pm 0.04$          |

## 1.5 Fluorine Experiments

In order to achieve correct results for the fluorine experiments, just like all previous ones, a correct setting of the magnetic field gradients is required. This is best done before setting the resonant frequencies to fluorine. Take a 1-proton sample such as mineral oil or distilled water and introduce a  $90^\circ$  pulse. Fine tune the pulse by adjusting the frequency to remove the oscillations in the FID signal. Adjust the gradients so that maximum decay time is achieved (and the best exponential shape). Now the spectrometer is calibrated. After calculating the approximate resonant frequency of fluorine, set the synthesizer frequency from the F parameter. Introduce the Fluorine sample and apply a  $90^\circ$  pulse. Again fine tuning is needed, although this time

it would not be that obvious, because of the nature of fluorine and the FID signal. Switch to a  $180^\circ$  pulse and follow the instructions to obtaining the 'null' FID tail, described earlier. The following is obtained, after careful tuning.

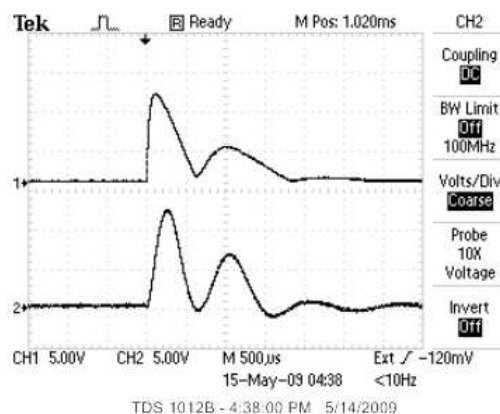


Figure 17: FID signal observed after a  $90^\circ$  pulse applied to fluorine FC-70. CH1: Env out, CH2: I out

The period must be set to a larger value, to avoid saturation. The maximum FID amplitude is plotted as a function of  $P$ , to illustrate the need of a larger value. See figure 18.

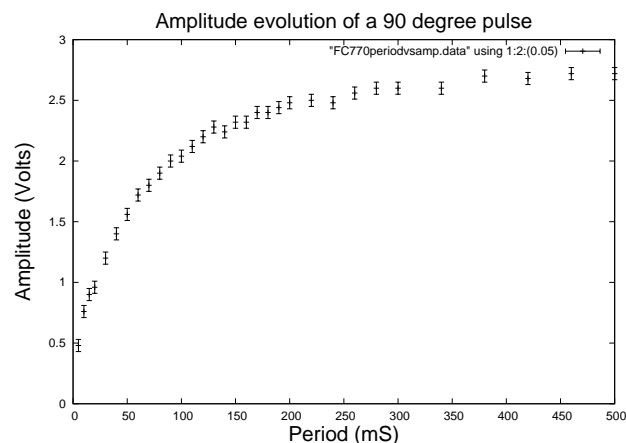


Figure 18: Maximum FID amplitude after a  $90^\circ$  degree pulse varies with the repetition time,  $P$ . Sample: FC-70

The second 'bounce' observed in the FID is due to a **chemical shift**. This is used to determine molecule shape and structure. The best way to describe this chemical shift is to set the synthesizer off resonance (vary only the Frequency parameter) and observe the FID and its FFT on the oscilloscope.

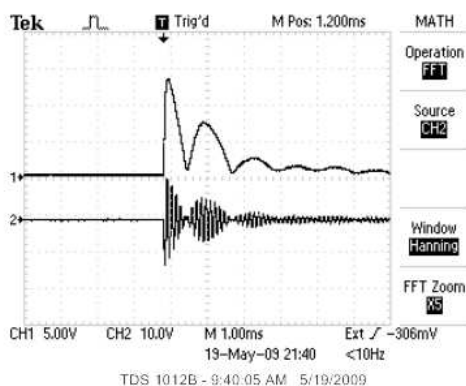


Figure 19: Output observed when the synthesizer is off resonance

The above figure 19 shows the FID signal of the FC-70 sample. As said before, set the frequency of the synthesizer to be off resonance. Now take the FFT of the signal coming out of Q (channel 2 on the oscilloscope).

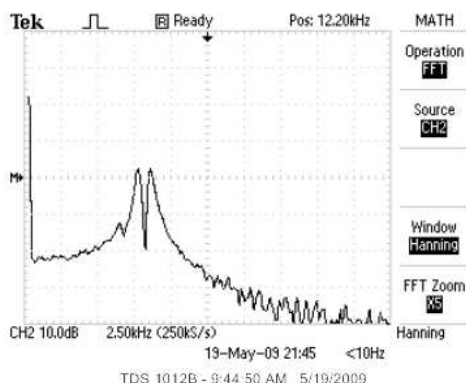


Figure 20: Fast Fourier Transform of the FID signal observed with the FC-70 sample

It is clearly seen that in figure 20, fluorine does not have a single resonant frequency but multiple ones, indicated by the three peaks. The following data was obtained for the FC-43 and is a little bit more exciting.

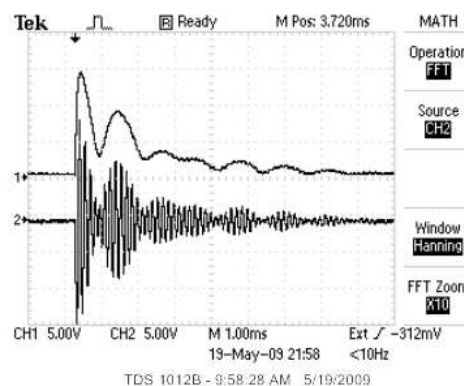


Figure 21: FID signal observed off resonance, FC-43 sample

The signal here is slightly different, and if the FFT is observed even more peaks show up, indicating a clearer picture of what is happening. In figure 22 there are four, maybe five, peaks that can be deduced, indicating multiple fluorine sites, in the molecule.



Figure 22: Fast Fourier Transform of the FID signal observed with the FC-43 sample

## 1.6 Continuous Wave Experiments

In continuous wave experiments, the tuning of the magnet capacitors, both tuning and matching, is extremely important and very hard. Also, the temperature of the magnets must be stabilized. And for a third complication, the BNC coming out of the magnet, proved to be EXTREMELY sensitive to interactions and position. Thus, do not displace it once the experiment has started.

Firstly, tuning the spectrometer takes some time to get used to, and requires a lot of patience. Set the field gradients using a proton sample. Don't forget the selection switch from proton to fluorine if change is needed. If fluorine is the substance of interest, retune the spectrometer to the desired frequency. The next step is crucial and is very specific to

each sample, thus performing it before each sample is important. Connect the I and Q outputs to the channels of the oscilloscope (all pulses must be turned off) and switch to XY mode. A dot should be observed. Connect the **CW OUT (Synthesizer)** to the **CW IN (Receiver)**. You may use one of the cables connecting the pulses as it will not be used. Ground the channels and center them. Then switch the setting to DC coupled. Turn the CW out switch to on and observe the dot. The **A** parameter on the synthesizer should be at -65db. Using both the tuning and matching capacitors, center the dot. After the dot is centered, increase the amplitude A by approximately 15 db. Repeat the centering and keep increasing to about -12db. You will notice that the tuning gets harder as power is increased. Also, make sure that the RF Gain is set to about .75. After the dot is centered connect the x-axis of the oscilloscope to **SWP I/O** located on the **Field Sweep** module. Notice that the variables on that mode are split in two. The first part **G, P, T, M** are for the **Lock-in** Section. For now we are interested in the Sweep section, i.e. **H, S, A, D**. The H parameter represents the constant magnetic field offset, which allows for the observation of the 'resonant' region in case the spectroscope is not perfectly in tune. The S setting is the Sweep mode. This is used for Fast or Slow passage sweeps. Different options should be observed. For very slow, one-time passage sweeps, use the **Trig** option. Thus whenever the user is ready to take data, the **Man Start** button is used to toggle the scan. The next setting is labelled A, and represents the range in amplitude used for the sweep. The sweep amplitude is in Gauss and goes **from -A to +A**. Thus if the setting is at 0.625 G, for example, the total sweep is 1.25G. D stands for the duration of the sweep. This parameter is obsolete for fast passage sweeps.

I recommend using the **Persist** setting on the oscilloscope, especially for slow passage sweeps. This setting is located under **Display**. Also, the x-axis should be set to 2 volts per division.

What we can measure with the continuous wave experiments, gives us an idea of the different sites and molecule positioning, just like the FFT.

The following is sample data for the continuous wave experiment.

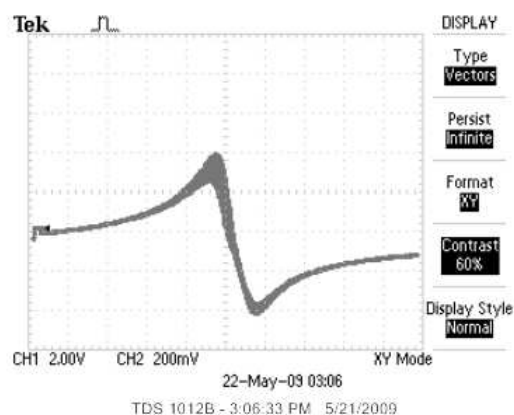


Figure 23: Water sample CW data. Swp Amp = 0.313G. Sweep duration = 200s. Frequency = 20.80120 MHz

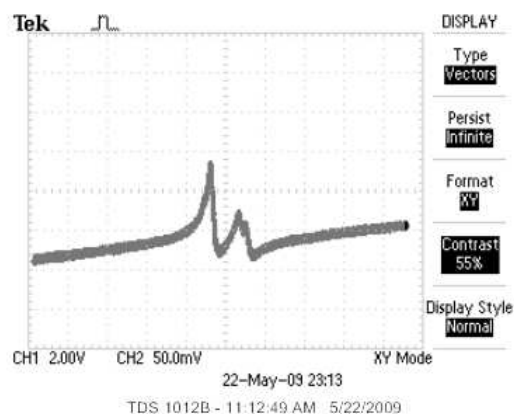


Figure 24: FC-43 CW data. Swp Amp = 1.250G. Sweep duration = 50s. Frequency = 19.57530 MHz

In continuous wave experiments, a fourier transform (an analog to the FFT) is shown [2]. Equation 1 applies here, where  $\gamma$  is known, and  $B$  is swept from zero to amplitude  $A$ . For example, in figure 22 It is clearly seen that there are two major peaks (ignoring the splitting in one of them) separated by 1 KHz. When looking at figure 24, there are two peaks separated by 0.25 G approximately. This shows that equation 1 is satisfied, provided one does the calculation.

The lock-in amplifier is used to amplify the signal from the CW experiments. Connect the Q out cable to channel 1 on the lock-in and view it's output on the oscilloscope. Here, the amplitude setting can be adjusted for desired results, being careful of the overload LED on the lock-in.

## References

- [1] Cowan, Brian. *Nuclear Magnetic Resonance and Relaxation*. Cambridge, UK: Cambridge University Press, 1997
- [2] Abragam, A. *Principles of Nuclear Magnetism*. New York, USA: Oxford University Press, 1961

