^{1}H NMR in Willet Science Center

Before making a sample and acquiring a spectrum, be familiar with ^{1}H NMR theory from your textbook and the practice of making a sample from Padias.

Samples are normally placed in solution with a solvent that has no protons or in special solvents where protons have been exchanged with deuterium (i.e.- $CDCl_3$, CD_3OD, etc.)

Normally we would determine the solubility of your compound in a non-deuterated solvent first; chloroform is a good place to start. Put a tiny amount (think: a few grains of sand) into a sample vial and add a few drops of chloroform. See if the sample is soluble. If not, see your professor. However, assume your sample is soluble in chloroform.

After identifying a suitable solvent (chloroform), make up your sample. You can do this a.) in the NMR tube (checked out from the stockroom) or b.) in a sample vial and then transfer to an NMR tube. Since this is your sample for the NMR, you must use a deuterated solvent here. (Deuterated solvents are expensive, so use sparingly.) The deuterated solvent should contain TMS (tetramethylsilane), which serves as an internal standard.

The graphic to the left is a to-scale model of our NMR tubes for the 60 MHz NMR spectrometer in Willet. You should make up a 3-4% solution for obtaining a FT-NMR spectra. This is nearly equivalent to one drop of liquid compound in about 30 drops of solvent **or** a small pinch of solid compound in 30 drops of solvent. Under no circumstances should your NMR tube be filled more than an inch high. Be sure you don't try to make it too concentrated. Many substances are soluble in dilute solutions and not in concentrated solutions.

Before you go to acquire your ^{1}H NMR, you should have estimated what the spectrum will look like: number of signals, shifts, integrations, splitting. Take your notebook with you to the magnet. Be aware that the integrations are just ratios and you will have to make a determination of how those ratios correspond to your expected numbers of protons.

When you have finished acquiring your spectrum, you can recover your NMR sample (ask your professor) or you can dispose of the liquid in the waste container, accounting for the waste on your waste sheet.

solvent to ~ 1.3 inches

sample (same depth as the cap covers)
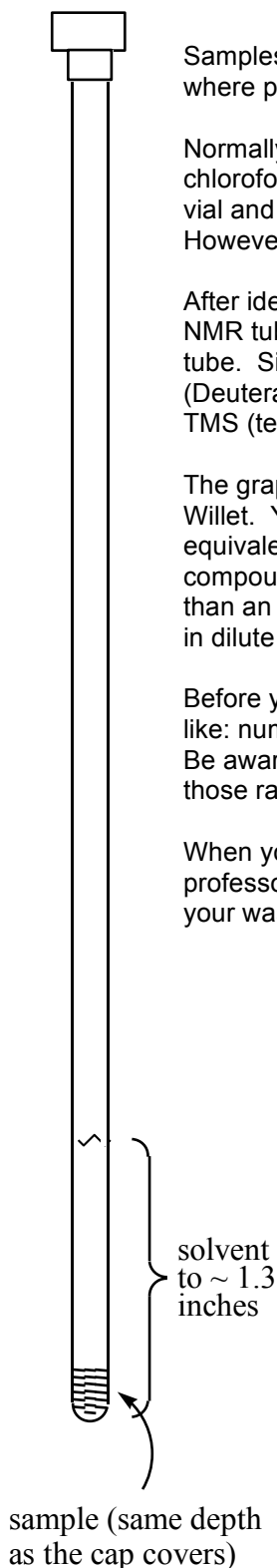
Operation of the EFT-60 MHz NMR

Sample insertion:

1. Eject the sample in the probe by pushing in the button marked eject. A stream of air will float the sample to the top.
2. Remove the sample tube and place your tube in the spinner with the rubber O-ring oriented to the top of the spinner.
3. Use the depth gauge to set your NMR tube to the correct height (in the hole located on the front part of the magnet). The spinner should rest on the top of the plastic guide.
4. Insert your sample in the probe (make sure you have a spinner on the NMR tube). You may need to push the sample down as the receiver for the signal is about a foot down inside the tube. You are ready to obtain an NMR spectrum.

Two programs control the NMR. The first, PNMR, is a Windows based acquisition program. The second, Nuts, is a Windows based data manipulation program, which converts your spectrum from an FID form to something you can interpret. To toggle between the two, use the command ALT-TAB (hold down the ALT key, and hit the TAB key).

To acquire a spectrum:

1. Type "acq" (without the quotes) and hit <enter>.
2. Hit <enter> again to accept the name of the file (pnmr.fid or some such – don't change it here because that is the name that WinNuts will be looking for later).
3. The computer will do a quick shim, set the gain and then acquire your spectrum.
4. Wait for the acquisition to be complete (hit <enter> when the box pops up saying it is done).

Now is the time to process your FID signal to an NMR spectrum (most of the commands do not require you to hit the <enter> key):

1. ALT-TAB will take you to the WinNUTS program, in Windows.
2. Before processing, prepare the program by hitting the <enter> key a couple of times and a CTRL-F.
3. Type a2. This command does a number of different things including, transferring your FID to NUTS (same as the ZZ command), correcting the baseline (BC), Fourier transform (FT), and default phase (PL).
4. Now you may need to touch up the phasing, getting the baseline on either side of a peak to be at the same height. Type ZO to enter the zoom routine. You need to select two peaks (or set of peaks) at either end of the spectrum for the phasing. Hold down the left mouse button and draw the red window around a set of peaks on the left side. Hit the '1' key. This defines that peak as number 1. Now draw a red box around a peak on the right side of the spectrum. Hit the '2' key. This defines that peak as number 2. Hit the <enter> key. Now type PE. Hold down the left mouse button and move the mouse side to side until the baselines on either side of peak are of even height on the screen. Hold down the right mouse button and do the same. When you are satisfied, hit the <enter> key.
5. Type ZO. Select the TMS peak by drawing a red window around the rightmost peak. Hit CTRL-E to expand that red region to full screen, and hit <enter>. Now type SZ to set the TMS peak to zero. Type ZO and hit CTRL-F and draw a red box around your region of interest (ie a region that contains all your peaks (usually between 10 and 0 ppm or smaller). Hit CTRL-E, <enter>.
6. Flatten the baseline by typing FB, and then 'L' to calculate. When the calculations are finished, <enter>
7. Now is the time to integrate. Type 'ID' (the i key). 'C' clears any previous integrals. Starting at the left side of a peak or peaks, place the cursor and hit the left mouse button twice. Move the cursor to the right side and hit the left mouse button once. Repeat for all of the peaks you are interested in. You should see numbers displayed for each integral. If you don't see any, or aren't happy with their values, you can adjust them somewhat. Click once on a peak (choose a peak that you think you know how many protons it is worth). Type 'V' and enter a value in the box provided. Click on 'OK'.
8. You are ready to print. Type 'P'. Click on the OK button.
9. Hit <enter> to exit the integrate routine, and CTRL-F to expand the spectrum to full screen. Remove your sample from the probe and replace it with one of the standard samples.