

## Fall 2011 NMR Experiment Guide

### 1) Sample Preparation

- a) If a solid sample, dissolve in a solvent. Choices for the solvent include  $\text{CDCl}_3$  (Preferred solvent), Acetone- $\text{d}_6$ , DMSO- $\text{d}_6$ , and  $\text{CD}_3\text{OD}$ .
- b) If a liquid, no solvent required.
- c) Put sample in an NMR tube. Add enough liquid so that there is about 2.5 inches (length of your pinky) in the tube.
- d) Add a standard if necessary. Tetramethylsilane is a great standard and stored in the refrigerator due to a low boiling point. A VERY small amount is required.

### 2) Acquiring the Free Induction Decay

- a) Make sure the air pump is running and that the NMR is tuned correctly. **Tuning should not be required of students and done only by faculty.**
- b) Eject the standard sample in the NMR and put the spinner on your sample.
- c) Adjust the height of the spinner on the NMR tube using the depth guide.
- d) Insert the sample into the probe and **insure the sample is spinning.**
- e) Possible spectra to be run include a  $^1\text{H}$ ,  $^{13}\text{C}$ , Dept, or Cosy NMR. This guide will only discuss  $^1\text{H}$  NMR but see the notebook at the NMR to run different spectra.
- f) Start WinPNMR (if not already running.)
- g) Change the number of scans to 1 by typing NS followed by a space followed by the number 1. Then hit enter.
- h) Type ZG and then hit enter.
- i) A window will pop up asking you to name the file. Just hit enter to name the file as the default file name.
- j) See if the FID is red or yellow. If the FID is red, turn down the receiver gain to a lower number. Values can be from 1 (the lowest which is used for concentrated samples) to 100 (the highest which is used for dilute samples).
- k) Type ZG again and hit enter again. Again name the spectra as the default file name. See if the FID is red or yellow.

l) Once the FID is yellow, change the number of scans to 4 by typing NS and then a space and then 4 followed by hitting enter.

m) SAVE THE FID by clicking on data.new and then your lab day. An example of a file name would be ddmundayexp6\_1H.fid. This name involves my initials, my lab day, the lab number and what type of experiment I carried out (a proton in this example). The extensions are fid for the data and nmr for the nmr.

n) WAIT UNTIL SCANNING IS COMPLETE and then remove sample when scanning is complete and return standard with spinner back into probe.

### 3) Obtaining a spectra from a FID.

#### *Option 1*

a) Start the Nuts software. (NMR Utility Transform Software)

b) Run the macro on the displayed FID (in WinPMR) to obtain spectra. This will be different for different spectra.

To obtain the  $^1\text{H}$  NMR, **hold down the control button and hit F2.**

To obtain the  $^{13}\text{C}$  NMR, hold down the control button and hit F3

Other spectra – See notebook.

c) The spectra will be displayed. Type SA (for save as) and a window will pop up (USUALLY displaying your folder with your FID that you named earlier.) Find your FID that you named earlier and then single click on it. Change the extension to .nmr and then click enter.

#### *Option 2*

a) Start the Nuts software. (NMR Utility Transform Software)

b) Click on file and then open. Open your fid by finding the FID in the folder and with the name you used earlier.

c) **THIS IS NOT YOUR SPECTRA.** This is your fid. Click on a followed by 2. This will run the macro to convert your fid into your spectra.

d) Type AP. This stands for automatic phasing and will make your spectra look better.

e) Type ZO or double click the left mouse button. This stands for zoom. If you click and hold the left mouse button down while dragging to the right, the region should

turn orange. Right clicking the mouse button will zoom the area. Right click again will return you to the full spectra.

\* If you are in ZO, ID, PP, or something else and want to get out of it, just hit return.

f) Type PP. This stands for peak pick. If too many peaks are picked, click and hold down the left mouse button. Move the horizontal line up or down to add or delete picks. Anything above the line will be peak picked. When you like where the line is, type M (while still holding down the left mouse button.)

g) Type ID. This stands for integrate data. This will include a non broken integral over the whole spectra. Double click the left mouse button to the left of a peak. A green line should appear. Now, single click the left mouse button to the right of the peak. The integral should now appear broken. Repeat this on every peak you want to integrate. Click on the left mouse button while ON any of the integrals and type V. This will allow you to assign a relative value to this integral.

h) Print the spectra in landscape format.

Any questions can be answered with the notebook or any chemistry faculty member.