

## COSY/Hetcor NMR Guide Spring 2012 Version

A COSY NMR requires a proton NMR to use on the borders. A Hetcor requires a proton and carbon spectra. This user guide will first describe how to take a proton NMR, how to take a proton AND carbon NMR, how to take a COSY NMR and finally how to take a Hetcor NMR. The procedures for COSY and Hetcor are very similar. This guide will be broken down into obtaining the FID with the NMR and PNMR followed by workup of the FID using WinNuts.

### What you should have after running a COSY

- a) proton FID
- b) proton NMR
- c) COSY FID
- d) COSY NMR (if you want to save it)

### What you should have after running a Hetcor

- a) proton FID
- b) proton NMR
- c) carbon FID
- d) carbon NMR
- e) Hetcor FID
- f) Hetcor NMR (if you want to save it)

### **Obtaining a proton FID. (COSY or HETCOR)**

- 1) Attach the spinner to the NMR tube using the depth gauge to correctly set the depth. Put the spinner in the NMR and *make sure the sample is spinning*.
- 2) Start the PNMR program (if not already running). Make sure the nucleus is set for proton. The cursor should read H1. If it does not, type nu H1.
- 3) Set the number of scans to 1. If the number of scans is more than 1, type NS 1.
- 4) Type ZG and then enter. Hit enter again to use the default file name of pnmr fid.
- 5) Check the gain. If the FID is red, the gain is too high. The gain is fine if the FID is yellow. If the FID is red, reduce the gain by typing RG (half of what the number was before). (i.e. if the RG was 100, type RG 50.) Try ZG again using the default name until the FID is yellow.
- 6) Change the NS to 4. This number depends on your concentration. The higher the concentration, the lower the number of scans.
- 7) Type ZG and name the FID as follows: data\organiclab\Monday\dd3\_cosy1H.Fid for a COSY or data\organiclab\Monday\dd3\_hetcorborderH1.fid for a HETCOR. Change to your initials and day of lab. This assumes I am in Monday lab, I used my initials and this was experiment 3. You can name it whatever you want but make sure you can tell it is a FID of a proton because you will need this later.

### **Obtaining a Carbon NMR (HETCOR)**

See appendix at end of this procedure if you need to set the field. Ask your instructor if the field needs to be set.

- 1) Make sure the nuclei is set for carbon (C13). If it is not, type nu C13.
- 2) Now, acquire the C13 spectra. Check the number of scans. For neat samples, the number of scans is usually 12. If the sample is more dilute, add more scans. (64, 128, 256, etc.) Type ZG. Name the FID data\organiclab\Monday\dd3\_hetcorborder13C.fid

### **Obtaining a COSY FID**

- 1) Make sure the nuclei is set for proton (H1). If it is not, type nu H1.
- 2) Type cosy at the H1 prompt and hit enter.
- 3) Type in the name as follows. Data\organiclab\Tuesday\dd3\_cosy.Fid. This assumes I am in Tuesday lab, I used my initials and this was experiment 3. You can name it whatever you want but make sure you distinguish it from the proton taken earlier.
- 4) Set the relaxation delay. Try 2 seconds which is typical.
- 5) Set the number of scans. The number of scans should be set to 4 and then hit enter. There are 256 slices with 4 scans each so the time to complete this task is about 41 minutes.

### **Obtaining a Hetcor FID**

- 1) Make sure the nuclei is set for carbon 13. If it is not, type nu C13.
- 2) Type het and then enter to start the hetcor.
- 3) Give the hetcor FID a name like data\organiclab\Monday\dd3\_hetcor.fid.
- 4) If you are asked to set a relaxation delay, try 2 seconds.
- 5) Set the number of scans in multiples of 4. Start with 4 as the number of scans. The acquisition should take about 20 minutes.

### **Converting the proton FID into a proton NMR (COSY AND HETCOR)**

- 1) Start WinNuts.
- 2) First, process the proton FID you did first.
- 3) Hold down the CONTROL AND F2 buttons at the same time. Use the filename you used earlier for the proton FID. The spectra will come up and save the spectra so it can be later recalled by typing sa (for save as) in WinNuts. Save it as data\organiclab\Monday\dd3\_cosy1Hborder.NMR or data\organiclab\Monday\dd3\_hetcor1Hborder.NMR depending on whether you are doing a COSY or Hetcor. You will use these spectra later.

### **Converting the carbon FID into a carbon spectra (HETCOR ONLY)**

- 1) Start WinNuts.
- 2) Process the carbon FID
- 3) Hold down the CONTROL AND F3 buttons at the same time. Use the filename you used earlier for the carbon FID. The spectra will come up with a range from 220 to -10 ppm. The macro also does an automatic peak pick.
- 4) Remove the peak labels and table displays from the spectra by doing Control B and then doing Control P. It does not matter what order you do these commands.
- 5) Save the NMR by typing sa (for save as) using the following name: data\organiclab\Monday\dd3\_hetcor13Cborder.NMR. Change to your initials and day of lab. You will use this NMR as your border for the Hetcor.

### **Converting the FID into a COSY spectra**

- 1) Process the data by holding down CONTROL AND F5 together. This runs a macro and asks for a filename. Use the filename from obtaining a COSY FID. In this example it was Data\organiclab\Tuesday\dd3\_cosy.Fid. This will bring up an intensity plot.
- 2) Enter the acquisition parameters like name, date, and experiment.
- 3) Add borders. Click on borders (third choice from the left on top). First, click pick top spectrum which is data\organiclab\Monday\dd3\_cosy1H.NMR. Do the exact same steps except pick the left border.
- 4) Change the minimum height for the best spectra. Type mh which will bring up a box to put in minimum heights. Cosy values vary from 0.5 to 2.0.
- 5) Set the amount of spectra to show by using the zoom button. For example, if you sample only has peaks from 4.0 to 0, first click on zo. Then click on f. This will bring up the dialog that will allow you to change the display for both the x and y axis. Finally, double click to get out of zoom.
- 6) Change from an intensity plot to a contour plot by typing c.
- 7) Type pl to plot the spectra.
- 8) Optional. Save the COSY NMR if you do not want to have to process your COSY every time you want to look at it.

### **Converting the FID into a HETCOR spectra**

This procedure is VERY similar to the procedure for the Cosy.

- 1) Process the Hetcor data by holding down the CONTROL AND F6 buttons. This asks for a FID and use the FID obtained earlier for the Hetcor. In this example, it was data\organiclab\Monday\dd3\_hetcor.fid.
- 2) Enter the acquisition parameters like name, date, and experiment number.
- 3) Add borders. Click on borders (third choice from the left on top). First, click pick top spectrum which will be the carbon NMR obtained earlier. In this example, it was data\organiclab\Monday\dd3\_hetcor13C.NMR. Second, click pick left spectrum which will be the proton NMR obtained earlier. In this example, it was data\organiclab\Monday\dd3\_hetcor1H.NMR.
- 4) Change the minimum height for the best spectra. Type mh which will bring up a box to put in minimum heights. Try values between 0.5 and 2 and see which one looks better concerning the CROSS PEAKS.

- 5) Type c to change the intensity plot to a contour plot.
- 6) Type pl to plot the Hetcor spectra.
- 7) Optional. Save the hetcor spectra if you do not want to have to process your FID every time.

## APPENDIX

- 1) Make sure nuclei is set for H1. If not, type nu H1.
- 2) Acquire a proton spectra first by typing zg. Hit enter and then enter again to assign the default name to this FID.
- 3) Once the proton finishes, go to Nuts and click a2. a2 will convert the FID to a spectrum. Find out using the cursor what the chemical shift of the TMS peak is.
- 4) Return to PNMR and type fo. This stands for field offset. Add whatever value you got for the chemical shift of TMS in step # 4 in the first box. Add 0 in the second box. This corrects TMS to 0.