RUNNING SPECTRA ON THE JOEL 500 MHZ NMR SPECTROMETER

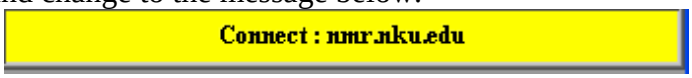
1 **Sign-in** in the logbook

2 **Log-in** [each research group is provided with has its own password. The login ID is the last name of the faculty member, all lower case]

3 Click once on the **Delta software icon** on the lower status bar. The icon is a big, blue Δ with a tennis ball circling it. [You open the ***Delta window***.] Do not close this window as long as you are running the spectrometer. You can monitor the status of all functions in this window.

4 

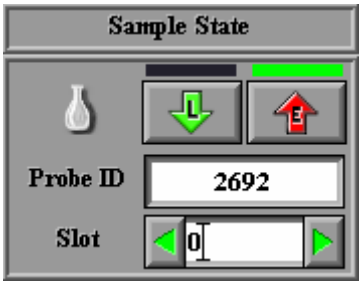
You will now connect the Dell computer to the spectrometer.
Select the **NMR magnet icon** (second row, 4th icon to the right). [You open the ***Spectrometer Control window***]. You will very quickly see "No Current Link" in a white background change to the message below.



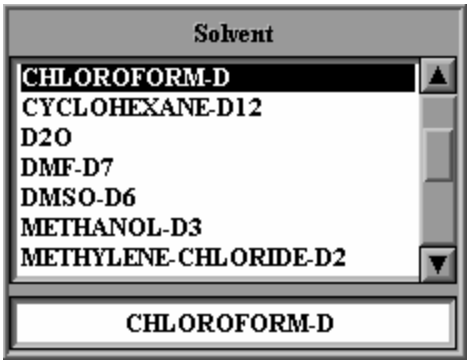
5 

Click the **Sample** button, found on the bottom row of buttons. [You open the ***nmr.nku.edu window***]

Note: When using Delta software you must have the cursor in the writing area for you to type.

6 

Find the **slot** number, located in the *Sample State* panel (upper left panel of the ***nmr.nku.edu window***). Place your sample and spinner in the sample changer in any empty slot other than the slot number in the window. Change the slot number in the *Sample State* panel to the slot number where your sample is in the sample changer. When you move the cursor out of the **slot** text area the changer will change the sample.

7 

Select your **solvent** from the list found in the *Solvent* panel (located below the *Sample State* panel). You may do this while your sample is loading.

Only when flask icon is full (Sample State panel), the top icon is standing up (Spinner panel), and your solvent has been selected is your sample is ready for the next step


becomes

8 

Select the **gradient shim / lock** button (2nd row, far right) on the *Lock Control* panel (located to the right of the *Solvent* panel and below the *Temperature* panel).When the spectrometer has completed this process (about two or three minutes) the two bars on the lower right of the *Lock Control* panel will read *LOCK ON* and *IDLE* in a green background.

You can monitor this process in the ***Delta window***.

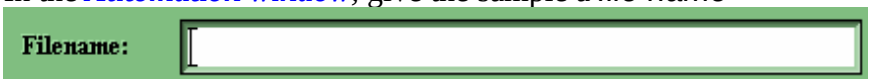
On occasion, due to a software problem, the lock will remain off after gradient shimming (the two bars above remain red). Check the ***Delta window*** to see if gradient shimming is complete. If gradient shimming is complete and the bars remain red click the **autolock** button (1st row, far right; directly above the **gradient shim / lock** button) on the *Lock Control* panel. This will complete the process.

You Are Now Ready To Run Your Spectrum

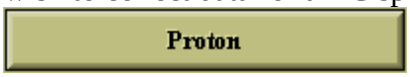
9 

Select the **Auto** button from the bottom of the ***Spectrometer Control window***. [You open the ***Automation window***]

10 In the ***Automation window***, give the sample a *file name*



11 Slect the experiment you wish to run from the bottom half of the window (typically **Proton**; select Carbon if you wish to collect data for a ^{13}C spectrum).



If asked, acknowledge. Before you know it your spectral data will be collected

RETRIEVING YOUR SAMPLE AND LEAVING THE NMR

12 

To retrieve your sample, go back to the ***nmr.nku.edu window*** and change the slot number from where your sample was back to the standard sample (see steps 6 and 7). Remember to change the solvent back if your sample was different. The standard uses ***chloroform***. [Note: to bring a window to the front you will need to place the cursor on the edge or head of the window and click] When the standard is ready, select the **auto-lock** button from the *Lock Control* panel (1st row, far right button) and wait until the two bars on the lower right of the panel read *LOCK ON* and *IDLE* in a green background. (see step 8)

13 

Once the sample is locked you may close all of the windows except the ***Delta window*** and the ***Spectrometer Control window***. Go to the ***Spectrometer Control window*** and click the red **Unlink** button. Once the spectrometer is FREE, you may close this window.

14 Close the ***Delta window*** and click the EXIT button.

15 Logout by selecting **logout** from the K icon on the lower status bar or by using the right mouse button.

16 **Sign out** in the logbook

17 **Remove your sample** from the sample changer and carefully take it out of the spinner. **Return the spinner** to the spinner container.

PROCESSING YOUR DATA AND MANIPULATING YOUR SPECTRUM

If a ***1D Processor window*** automatically opens after collecting data, you may begin with step 2. If you wish to reprocess old data, begin with step 1.

1 
Select the **Data Processor** button from the ***Delta window*** (1st button, top left). This will open the ***Open Data for Processing window***. From this window double click on the file you wish to open. If there are multiple files with the same name, the file with the largest number will automatically be selected. If you wish another simply double click on that number. This will open the ***1D Processor window***

2 If the ***1D Processor window*** opens with only a "typical" spectrum (no FID), you may skip this step.

or

If the ***1D Processor window*** opens with a FID above a "typical" spectrum you may close the FID by deselecting the ***Show FID*** box from the **Preferences** menu found on the top menu bar.

or

If the ***1D Processor window*** opens with two FIDs,.you must convert the bottom FID into a spectrum by first selecting the ***^{1}H button*** (for a ^{13}C spectrum select the ***^{13}C button***) and then pressing the ***Process button.***. You can hide the top FID by deselecting the ***Show FID*** box from the **Preferences** menu found on the top menu bar.

3 **Choosing the viewing range**

1) Select ***View Control,*** the last choice under the **Options** menu found on the top menu bar. This will open the ***View Control window***. Type in the range you want to view.

Or


2) Use the Zoom feature. You can change the cursor to the zoom data cursor by pressing the shift key at any time. Sweep out the area you wish to expand. Hints: If you do not like what you have zoomed use the backspace key to go to the previous plot(s). Use the End key to make the tallest peak expand to the top of the spectrum.

3 **Picking Peaks and Displaying Chemical Shifts and Integrations**

In the upper right hand corner of the spectrum you will have either a bar with a small dot or an expanded menu. Click on the bar with the small dot and

becomes

You may not start with the Zoom menu, but it does not matter. If you click on **Zoom** meun, or whatever the far right button is, you will see a number of other menus. Select the **Peak** menu.

To set the peak threshold, first select the ***peak threshhold button***. You will see a green line at the bottom of your spectrum. Use the mouse to drag the line to a position where you want the threshhold. You will not get chemical shifts or integration for any peaks which are below the threshhold.

To see the chemical shifts and integration for the peaks above the threshhold select the auto ***peak pick and integration button*** from the top row of buttons found above the spectrum. If you are not satisfied with the peaks and integration, you can change the threshhold (as above) and re-peak pick and integrate. Theses steps also can be done separately by using setting the peak threshhold, selecting the peak pick button, going the integration menu, resetting the threshhold and the selecting the integration button.

Note: The action of any menu button can be determined by clicking the right mouse button while the cursor is over the button of interest. When a menu button is selected its function appears in the right hand corner under the spectrum. By selecting the  button you can find a list of all the shortcut keys for a particular menu.

4 **Printing your spectrum**

Click on the printer icon, found in the top row of buttons above your spectrum. How easy is that?

When you are finished return to step 12 in the first section of this document and continue as appropriate.