

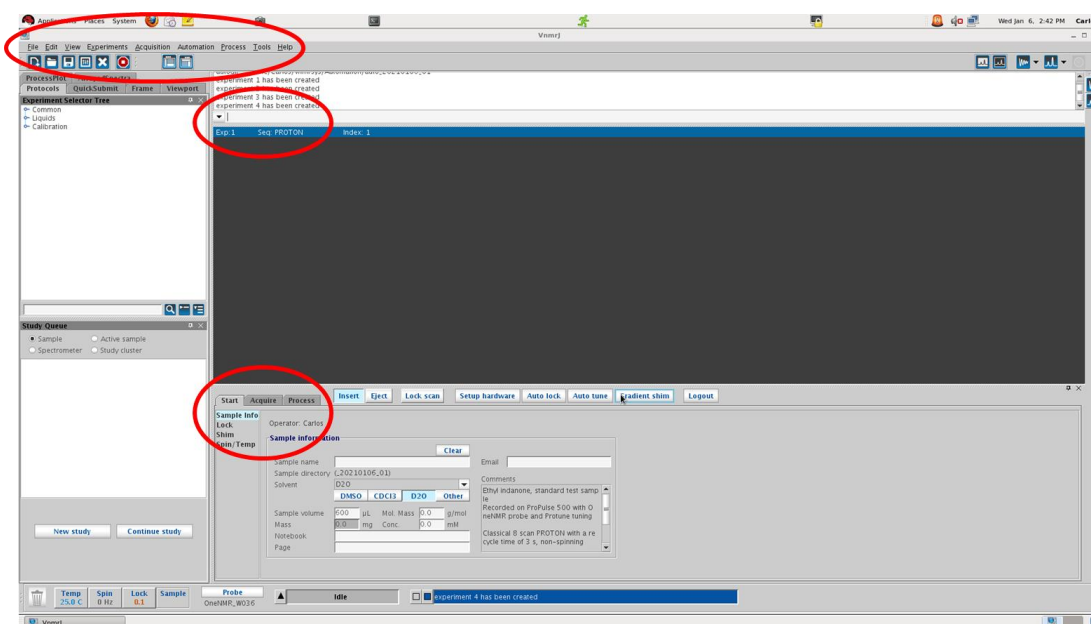


# Department of Chemistry NMR Facilities

## Varian 500 Tutorial. Acquisition of 1D $^1\text{H}$ and $^{13}\text{C}$ spectra

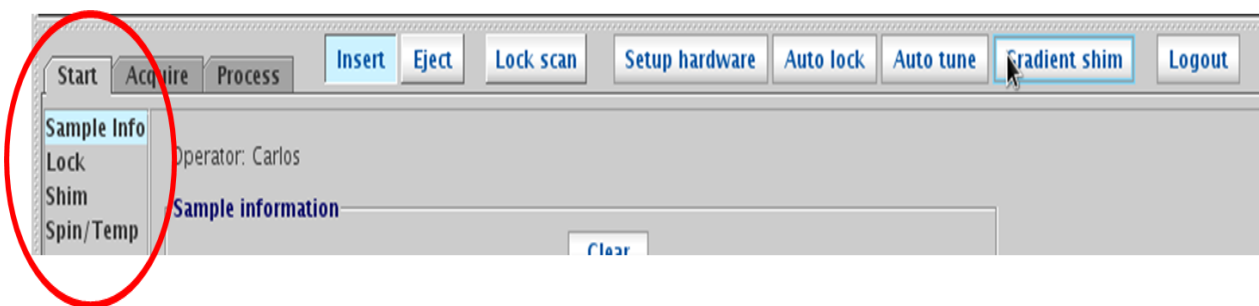
### The VNMRJ layout.

VNMRJ window. Main menus, command line and tabs are highlighted.

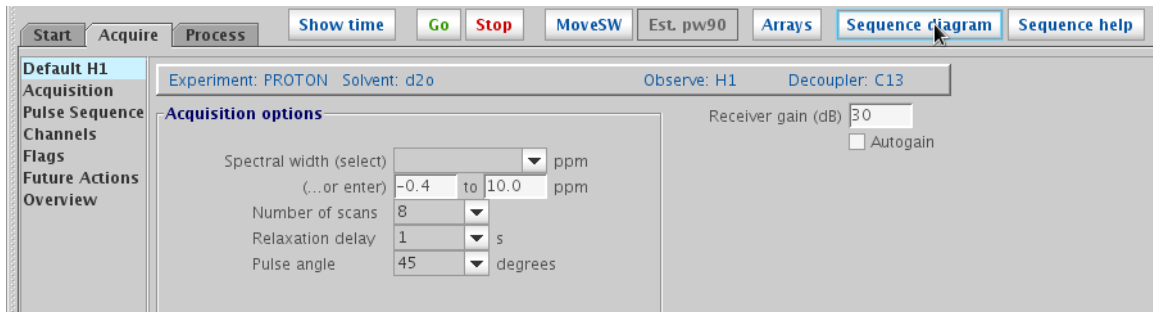


All the commands to operate the NMR spectrometer are within three tabs; **Start**, **Acquire** and **Process** and, the lines below each tab.

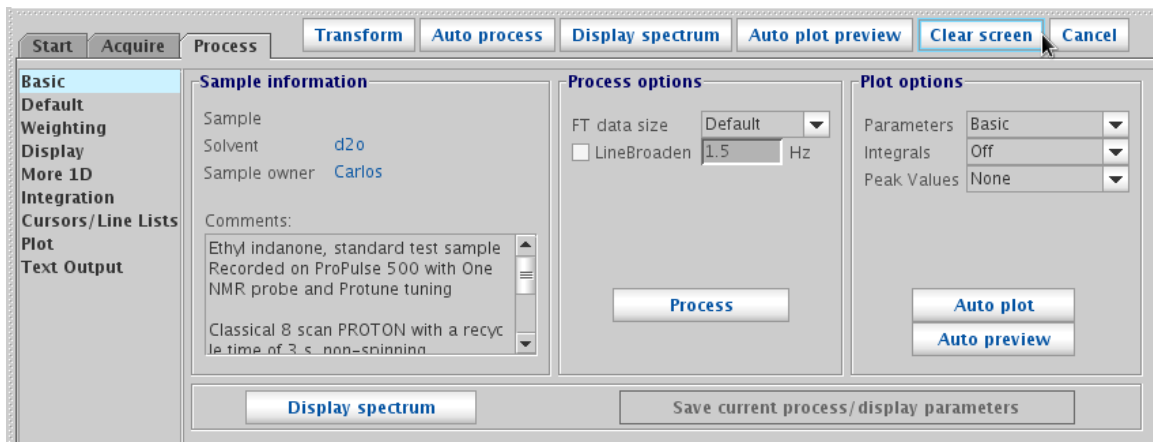
### Start



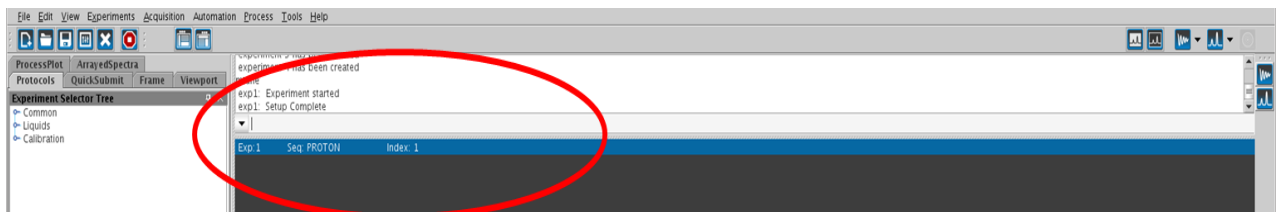
## Acquire



## Process

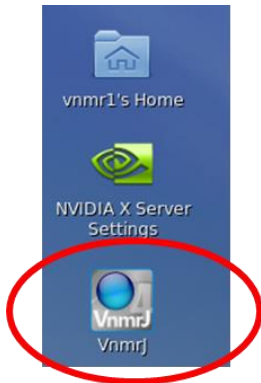


Commands can also be entered in the “command line”.

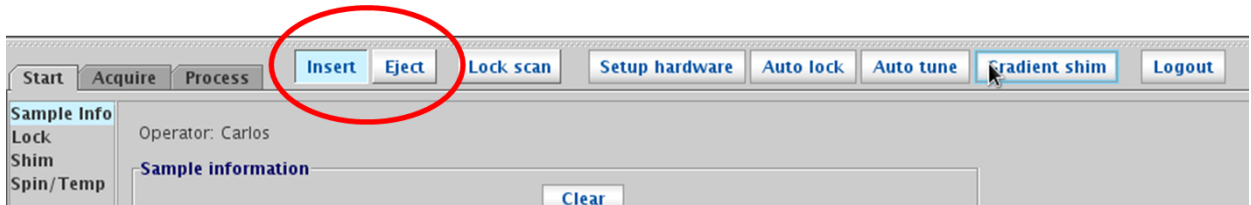


**Starting the program and setting up the sample.  
Tuning, locking and shimming the sample.**

- 1) Log on into the linux account.
- 2) Click on the VNMRJ icon to open the program.



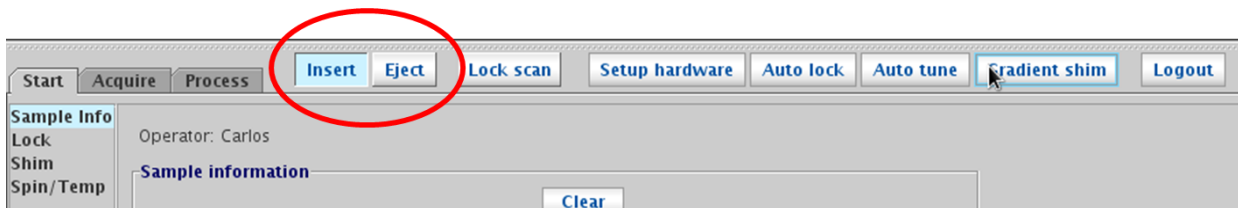
- 3) On the tab Start, click on <Eject> to eject the sample from the magnet.



- 4) Clean the sample.
- 5) Insert the tube in the spinner. Use the gauge to measure the depth.

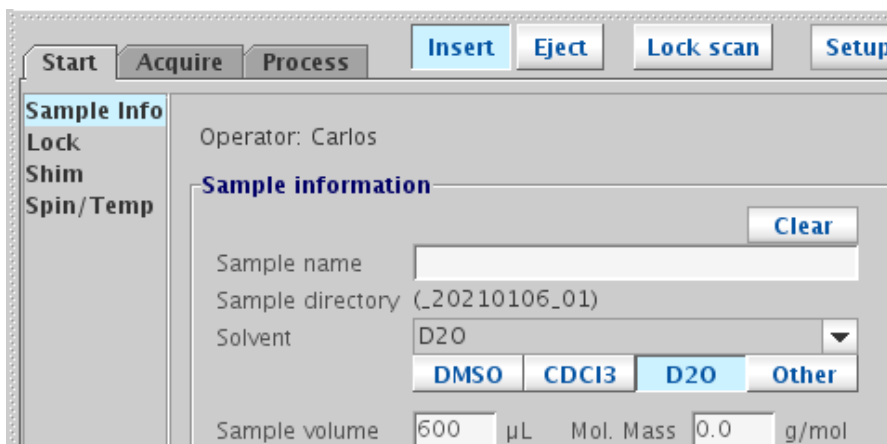


3) On the tab Start, click on <Eject> to eject the sample from the magnet.

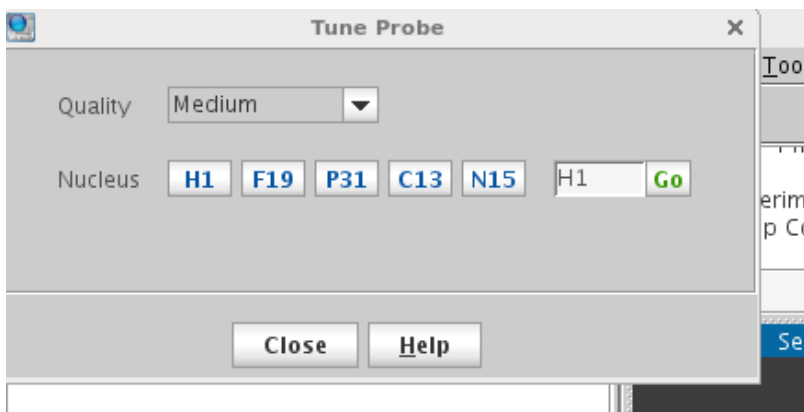


6) Place the new sample on top of the magnet. Click on <Insert> to insert the sample into the magnet.

7) Select the solvent on tab “Start/Sample Info”

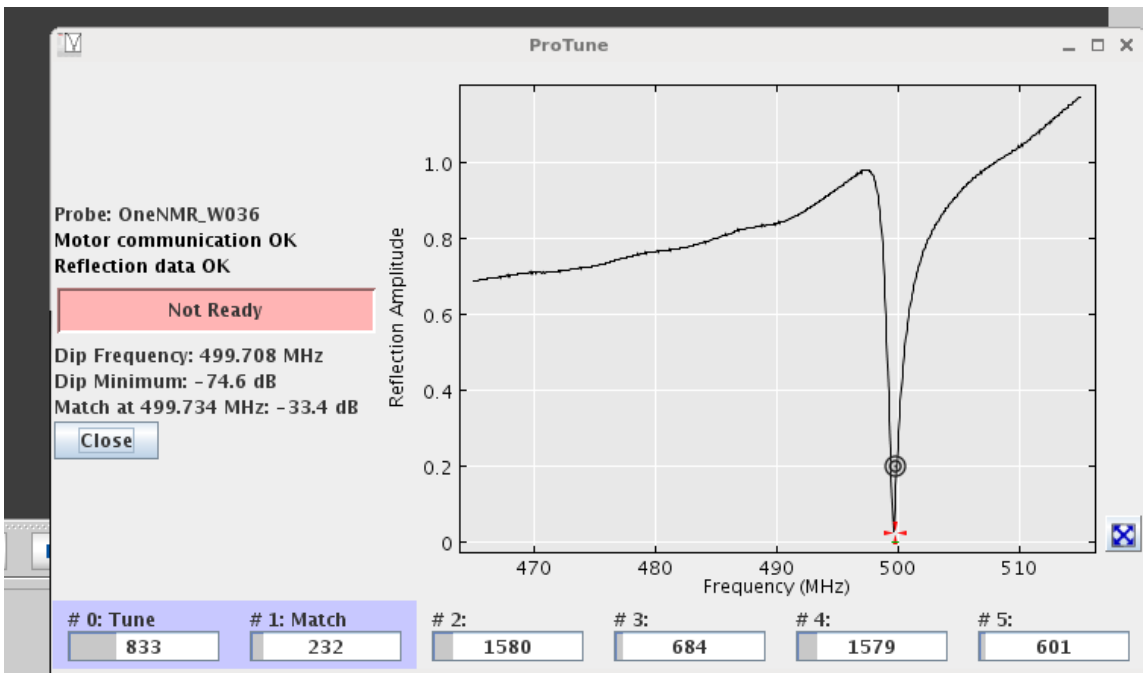


8) Type “protune” on the command line and then press <enter>. The window shown below pops up.



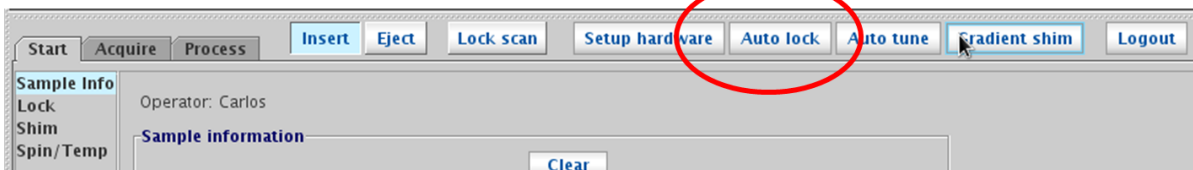
9) Click on the nucleus or select the nucleus and click on <Go>. Another window pops up showing the tuning process (shown in the next page).

**Attention:** If running  $^{13}\text{C}$  or any other nucleus, except  $^{19}\text{F}$ , first tune that particular nucleus and then tune  $^1\text{H}$ . Tuning of  $^1\text{H}$  is needed because the 1D experiment of any nucleus (except for Fluorine) is run with proton decoupling.



10) Wait until the whole tuning process is finished.

11) Click on <Auto lock> to lock the sample.



12) Click on <Gradient shim> to shim the magnet.

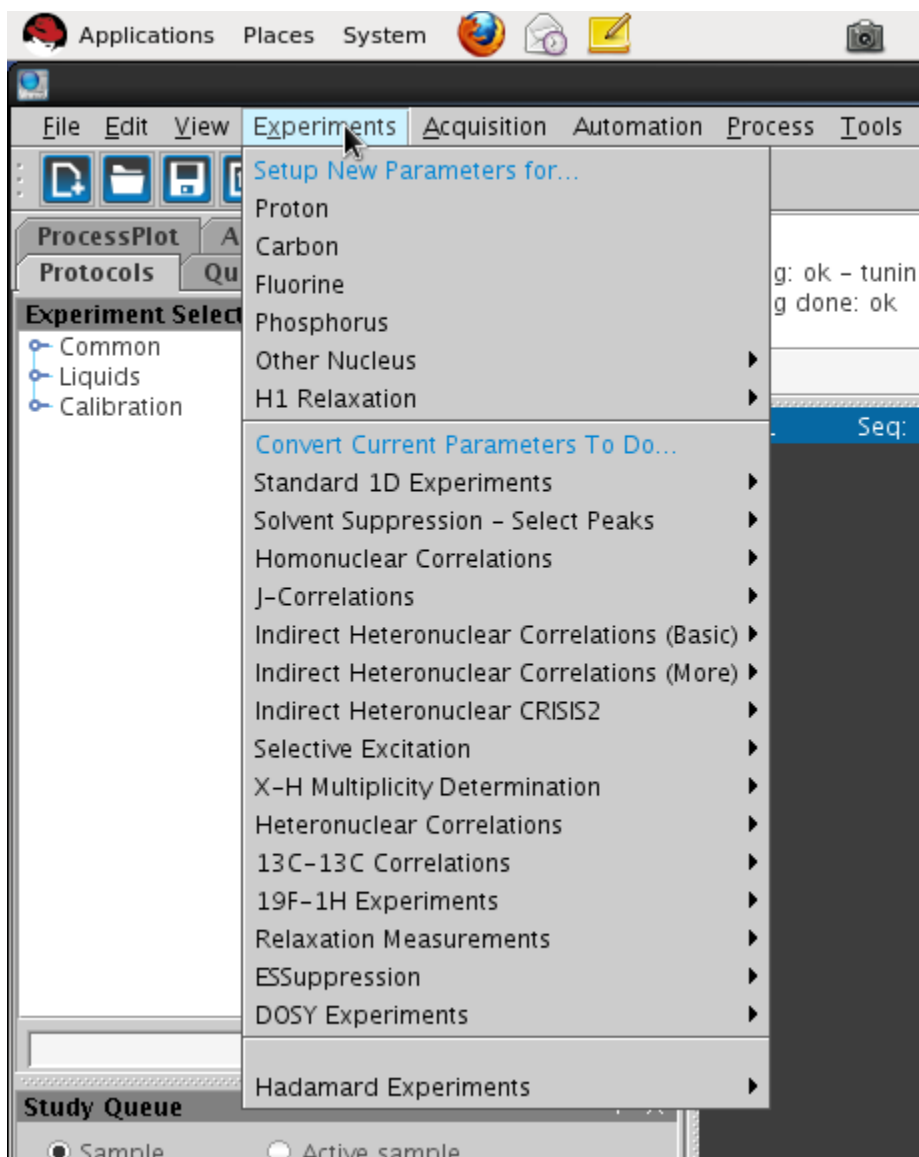


When doing Gradient shim and Auto lock, the solvent will temporarily switch to D<sub>2</sub>O and spinning will turn off.

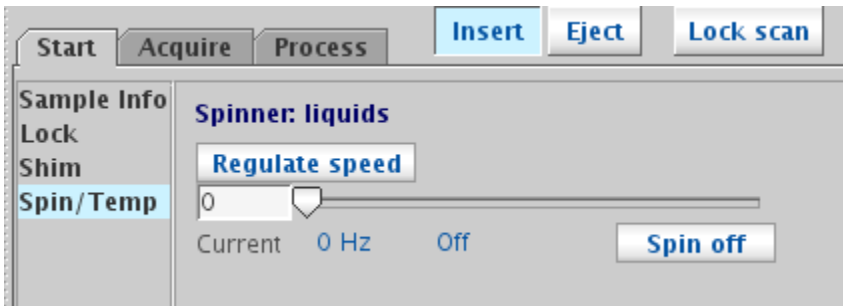
**Tuning, locking and shimming must be done only once for each sample, right before running any NMR experiment.**

### Proton (<sup>1</sup>H) experiment.

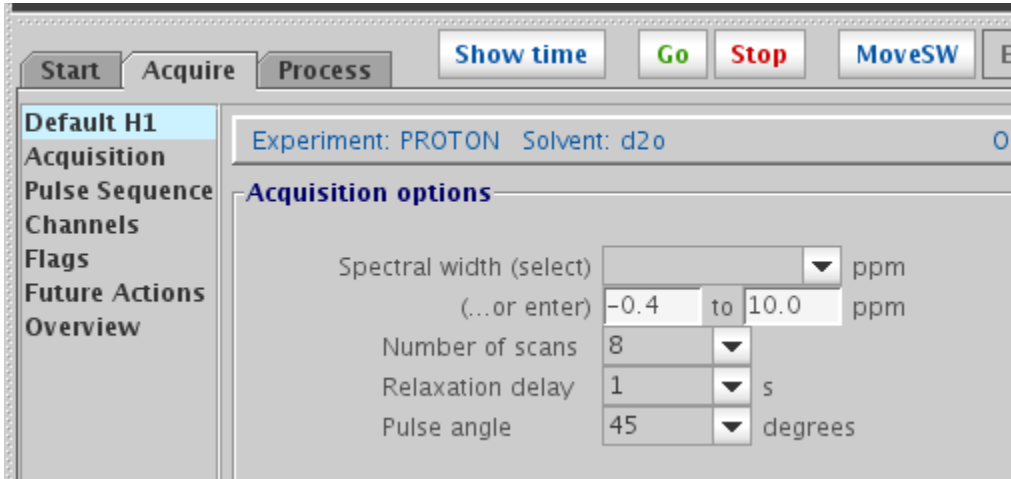
1) Select an experiment; click on the menu “**Experiments**” and then click on “**Proton**”, as shown below.



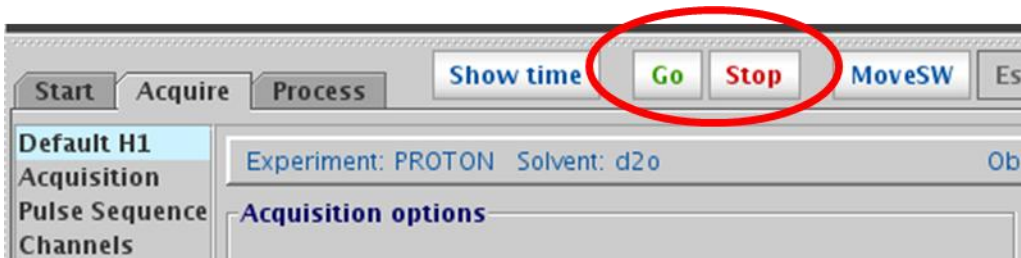
2) Regulate the spinning or enter “spin=0” in the command line.



3) On the tab “Acquire/Default H1” the experimental parameters can be modified; Spectra Width, Number of scans and Relaxation delay.



4) To run the experiment, click on <Go> on the Acquire tab.



5) To display the spectrum, type “wft” on the command line and then press <enter>.

6) Usefull commands: **wft**: to refresh the spectrum, **aph0**: automatic phasing, **vsadj** : adjust the high of the spectrum, **f**: brings back the full spectrum after it was zoomed.

7) Saving files: select “save as” in the “File” menu (top left side of the window).

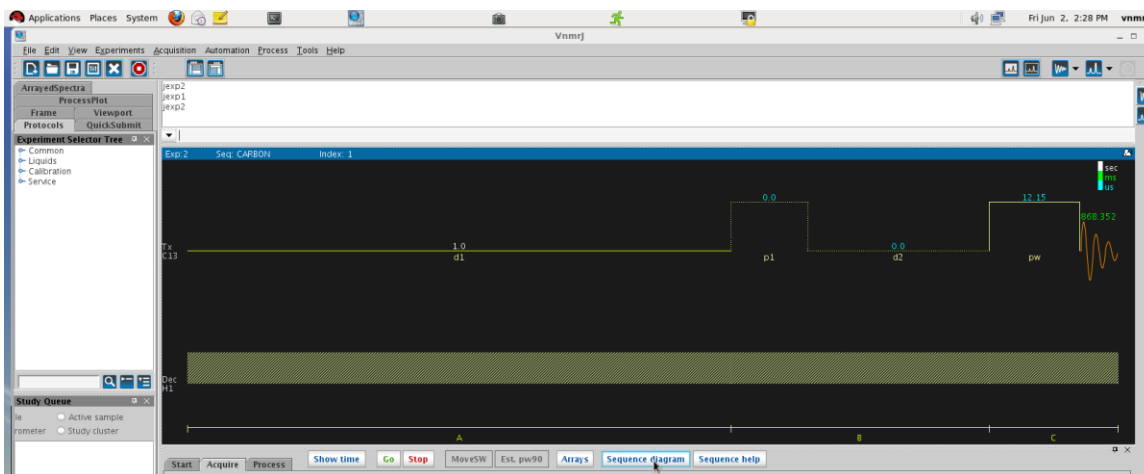
### Carbon ( $^{13}\text{C}$ ) experiment.

1) Switch to experiment 2; type “**jexp2**” on the command line and then press <enter>.

2) Select the solvent on tab “**Start/Sample Info**”. **Solvent must be set for each jexp.**

3) Open the file with the  $^1\text{H}$  spectrum saved in the previous experiment.

4) Select the experiment “**Carbon**” from the “Experiments” menu.



5) Type “**bs=8**” in the command line and press <Enter>.

6) Regulate the spinning or enter “**spin=0**” in the command line.

7) Click on <**Go**>.

8) After the acquisition is done, save the file.



### **Once you are done;**

- 1) Insert the dummy sample back in the magnet.
- 2) Close VNMRJ; select “Exit Vnmrj” from the menu “File”.
- 3) Log out from your account.
- 4) Log in the log book.

### **Retreiving data from the spectrometer computer using WinSCP**

- 0) Open WinSCP program.
- 1) Click on <NEW>.
- 2) Fill in the window that pops up: host name: juliet.chem.utk.edu, user name: .... , and password:.....
- 3) Click on <save>.
- 4) Click <OK> on next window.
- 5) When trying to connect for the first time, a window pops up. Just click on <yes>.
- 6) The right panel is the host computer. Change the directory to vnmrsys/data.
- 7) Download the data.
- 8) Once you are done. Click on <Quit> to disconnect from the host computer.