VNMRJ ^{1}H Operating Instructions

Login

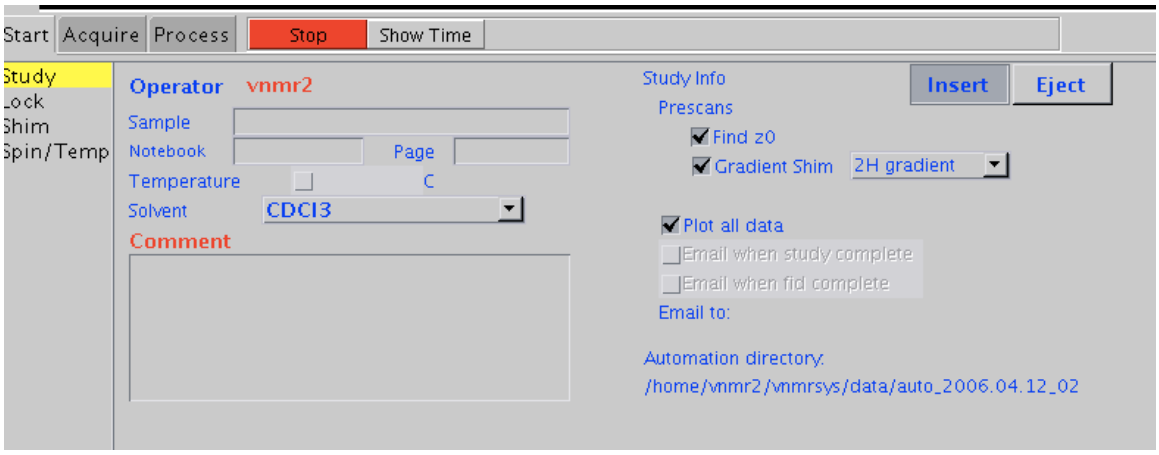
Login to the system by entering your group's user name and password.

Starting vnmrj

1. Start vnmrj by clicking the vnmrj spectrum icon at the top of the screen.
2. Choose to run a ^{1}H experiment by dragging the proton icon on the left of the screen to the black area.

Inserting your sample

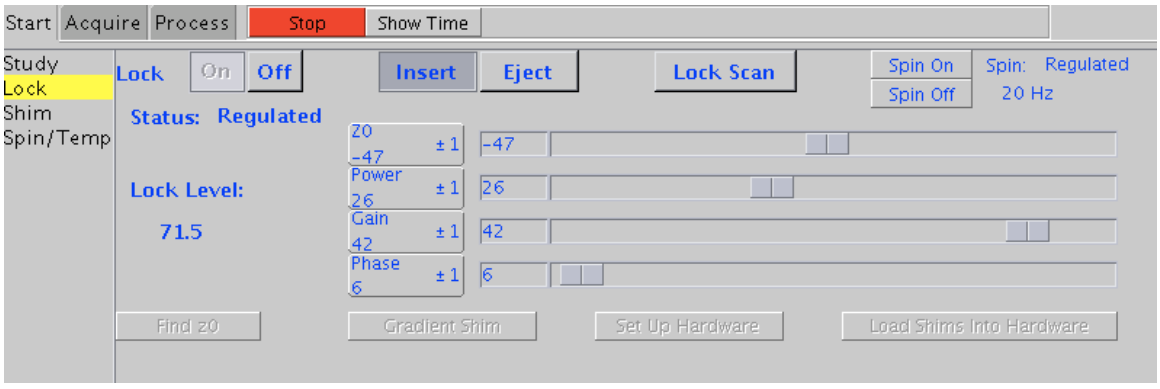
1. Click *eject* to remove the standard.
2. Replace the standard with your sample and click on *insert*. Note that there is no need to turn the spin or lock off before ejecting.
3. If you wish to use a solvent other than $CDCl_3$, select which solvent you would like to use with the drag-down box on this page.
4. If you would like to print a sample name/ID on your spectrum, insert this text into the comment box here (will print later by the command **pap**).



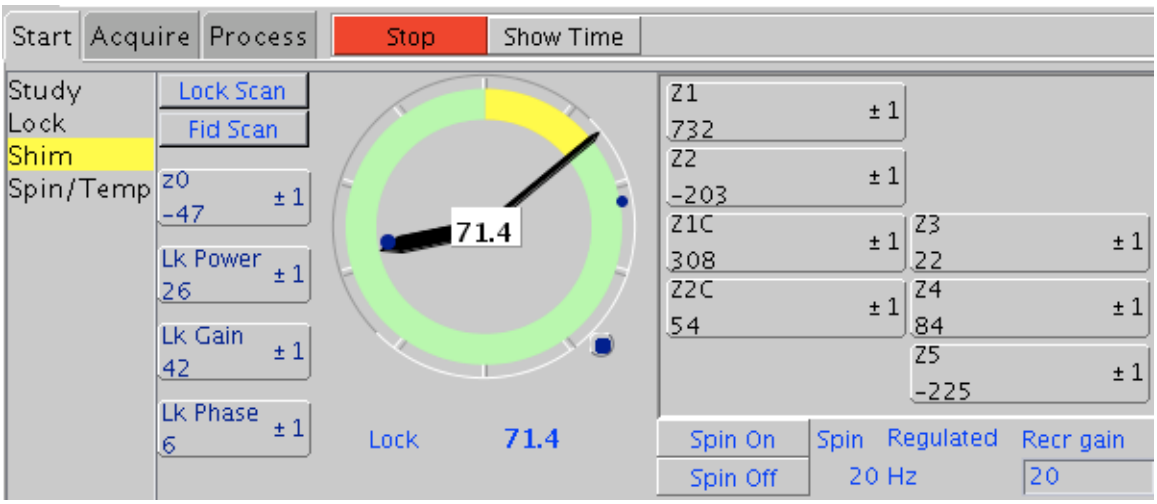
Shimming and locking your sample

1. If you are using a solvent other than $CDCl_3$, click on the lock tab and change Z0, Power, Gain, and Phase as necessary to lock your sample. The values can be adjusted with the left and right mouse buttons as in the case with the other instruments, or by typing in the desired value and pressing enter.
2. Shim your sample by first clicking on the shim tab, then by adjusting the Z1 and Z2 values.

Locking…



Shimming…



Acquiring your ^{1}H spectrum

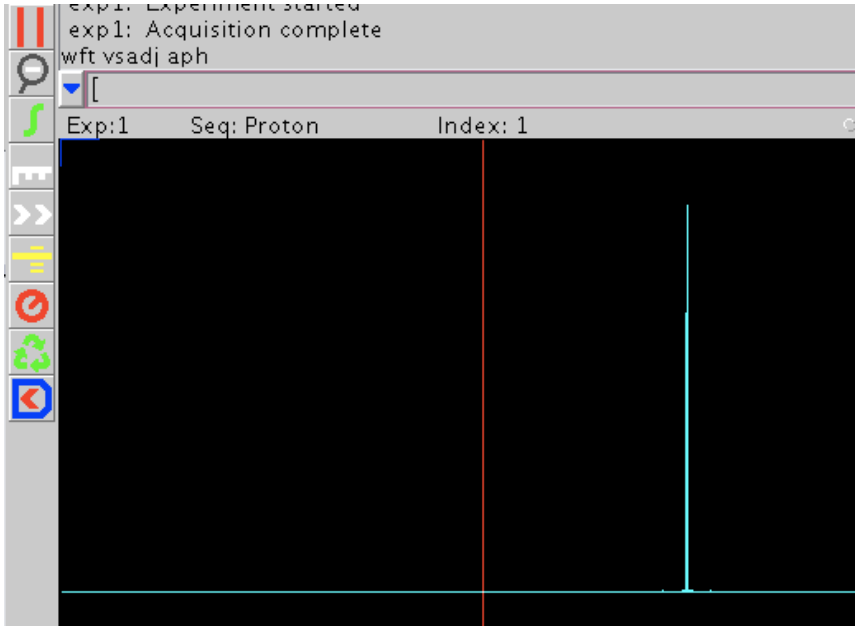
1. Check the current acquisition parameters by clicking on the process tag, then by typing **dg** in the input box above the black box.
2. To change any parameters, click the acquire tag and make adjustments as neccesary by typing in the input box. Use the format **"parameter = value"** to make changes.
3. Alternatively, you may use the default parameters and simply change the number of scans by typing in the command input box (i.e., type **nt = 16** for 16 scans).
4. Start the experiment by typing **ga**.

Processing your spectrum

1. After acquisition is complete, the spectrum should be displayed on the screen.
2. Apply the same commands as used on the other instruments to process the spectrum (i.e., type **wft vsadj aph** in the input box for fourier transform, vertical scaling of peaks, and autophasing, respectively).
3. ***Displaying the scale***: Note the set of processing icons to the left of the spectrum. Display the scale by clicking on the white ruler icon.
4. ***Expanding the spectrum***: To expand the spectrum, select the top icon containing 2 red lines. Place the cursors where desired, then expand the region by chosing the zoom in/out (magnifying glass)

icon. Use this icon to expand the spectrum and also to return to normal size.

5. ***Setting the solvent reference peak***: As in the case with the other instruments, place the red cursor on your solvent peak, then type **rl(x.xxp)** in the input box to place the cursor directly on the peak and set the reference value (i.e., **rl(7.27p)** for $CDCl_3$).
6. ***Integration***: Click on the green integral icon, then take note of the two additional integration icons that appear just below it. Click the middle integration icon and use the mouse to cut the line, then return to the spectrum by typing **ds** in the input box. If necessary, you can also click on the top integral button or type **cz** to display the full integral.
7. ***Setiing a peak integral reference value***: To set a specific peak integration value, click on the process tab, then the cursors/integration subtab. Place the cursor on the desired peak and type in the desired number of protons in the *normalization value* box. Save changes by clicking on the *set integral value* icon.
8. ***Set a precise spectrum width***: To set a specific numerical range for your spectrum, use the same commands as used previously on other instruments (i.e., typing **cr = 9.5p delta = 10p** then clicking the expand icon will set the window from -0.5 to 9.5 ppm).
9. ***Setting the peak threshold***: To choose the level of peaks for peak picking, press the icon labeled by the yellow threshold line and adjust the line accordingly.



Plotting and printing the spectrum

To plot and print, type any combination of the following commands into the input box. Note that these are the same commands as the ones used for other instruments, but are listed here as a reminder.

- pl : print spectrum
- pir: print integrals
- pscale: print scale
- pltext: print text label (from the comment box)
- pap: print acquisition parameters
- pll: print peak locations (ppm and Hertz) and peak height
- ppf: print peak frequencies above each peak
- page: eject page from the printer

Saving the data

1. If you do not yet have a directory in your group's folder, make a new directory by typing the command **mkdir('directory_name')**. Alternately, you can open your group's folder (top left of the desktop) and create a new directory by selecting *File* → *Create folder*.
2. To save a file in an existing directory, first choose the directory by typing **cd('your_directory')**. Next, save the file by typing **svf('file_name')**.

3. To retrieve your data later, find your file in your group's folder on the desktop and drag it to the black processing window. Process as described previously.

Finishing the experiment

1. Eject your sample and replace with the standard sample.
2. If you used a solvent other than $CDCl_3$, adjust the lock parameters back to the required ones listed under the monitor.
3. Click on the *loadshim* icon at the top of the page to load the standard shim file.
4. Manually shim the sample to reach the required lock level.

5. Exit the system by choosing *File*→*Exit vnmrj* or by typing **exit** into the input box.
6. Logout by choosing *Actions*→ *logout*, then click *OK*.

Carolyn Leverett, October, 2006