

VNMRJ 4.2 INSTRUCTIONS: VARIAN MERCURY 400 AND VARIAN VNMRS 500

August 9, 2016

1. Sample preparation

- a. Tubes of any length can be used
- b. Make your samples 4 cm deep. They will not shim as well if they are shorter; you will waste deuterated solvent if they are longer
- c. Make sure that the sample is clear: filter it if necessary
- d. Use a pen to label the cap or the top of your NMR tube if needed (do not use labels and do not write below the top few cm of the NMR tube—this is to avoid ink getting into the inside of the spinner and from there getting into the NMR instrument)

2. The Varian Mercury 400 instrument can only run ^1H and ^{31}P experiments.

The spectrometer is good for proton, but poor for directly detected phosphorus. If you do not have much sample, you should consider using either Bruker spectrometer or the Varian 500 (note that the Varian 500 requires special training for phosphorus experiments). The ^{31}P HMBC, which correlates ^{31}P and ^1H signals, gives information about ^{31}P chemical shifts and is a reasonably sensitive experiment.

You can also run basic 2D proton experiments (COSY, TOCSY, NOESY).

3. The Varian VNMRS 500 instrument is set up for ^1H and ^{13}C experiments only.

The spectrometer is capable of doing any nucleus between ^{15}N and ^1H , including ^{19}F , ^{31}P , and ^{15}N , but by default it is only set up to do ^1H and ^{13}C . You need special training to do any nucleus besides ^1H and ^{13}C .

The 500 is sensitive and recommended for both ^1H and ^{13}C . You can run all the 1D and 2D experiments involving these two nuclei on the Varian 500.

3. Experiment preparation

- a. Log in to the walkup NMR software

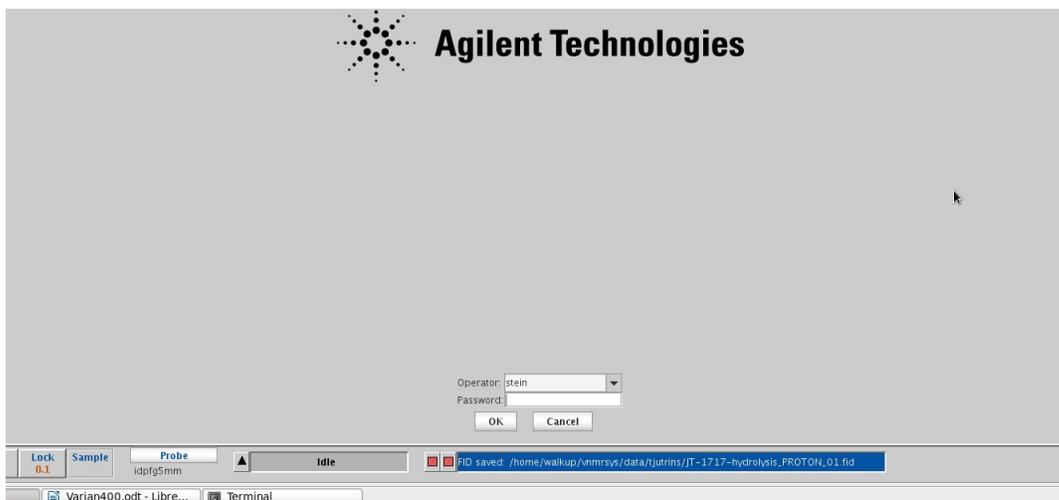


Figure 1: VnmrJ walkup interface login screen. Enter your username and password to log in.

b. Inserting sample

- i. Place sample into spinner (if no spinner is available, then eject the current sample, step v., before continuing with step i)
- ii. Check that the depth gauge is adjusted to 67 mm
- iii. Adjust sample position with depth gauge (the sample should touch the bottom of depth gauge, unless the sample is very short, in which case it should be raised so that it is centered on the box marked at the back of the sample changer)
- iv. Wipe NMR tube with Kimwipe without affecting its position in the spinner
- v. Click the button marked “eject” or type **e**
- vi. Listen for the jet of air. When you hear it, remove the dummy or previous sample and place your sample on the jet of air
- vii. Click the button marked “insert” or type **i**



Figure 2: Sample adjusted properly in the depth gauge.

4. Running in New Study mode

- a. On the bottom left, click on New Study
- b. In the middle of the screen, enter a sample name (usually of the form <initials>_<lab notebook number>; for example, RSS_0216). **Only use letters, numbers,**

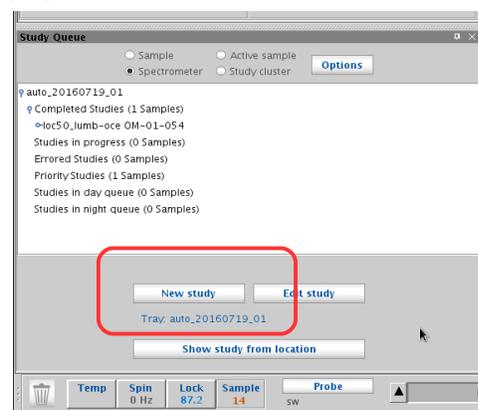


Figure 3: Starting a new study.

hyphens, and underscores as your sample name. Do not use spaces or any special characters such as %, #, !, or anything else. Most special characters will prevent email from working and many will prevent the file from being saved properly.

c. If you want, enter a description of your sample in the “Comments” box. Anything entered here will appear in Mnova or other NMR processing program.

d. Choose one or more experiments from the list on the upper left of the screen, under the McGill tab. See the end of this handout for a description of the experiments available.

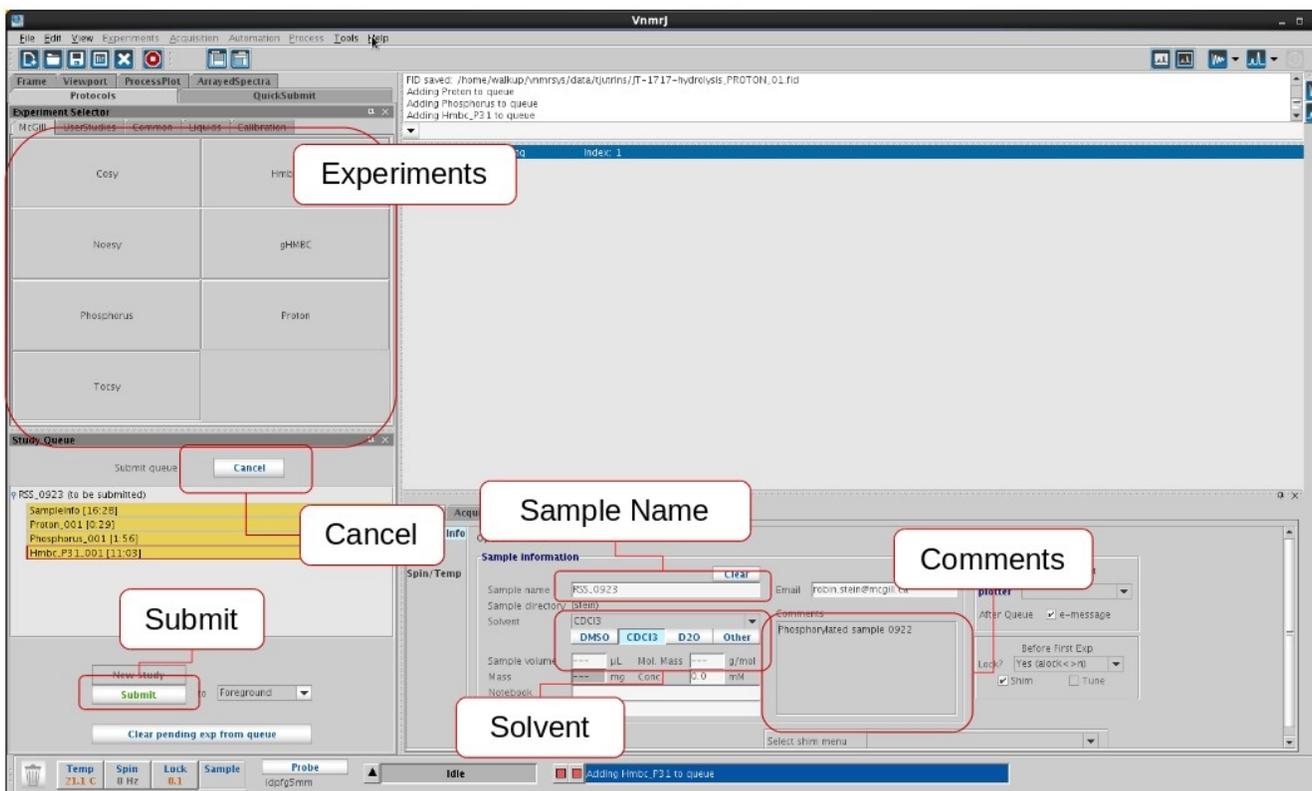


Figure 4: Submitting experiments to the sample queue. At a minimum, choose an experiment, enter the sample name, choose the solvent, choose a sample position, and push submit.

e. To remove an experiment from the list (for example, if you change your mind about running it), click on it and drag it to the trash can below the list

f. To modify an experiment (for example, to add more scans), double-click on it in the list. Then change one or more of the options listed on the right. If you do not see the option you want listed, click on Acquisition (under the Acquire tab, just to the left of the options shown). After customizing your experiment, click on Save (or click on Quit-No save to cancel any changes you have made). Use the “Show time” button to see how long an experiment will take.

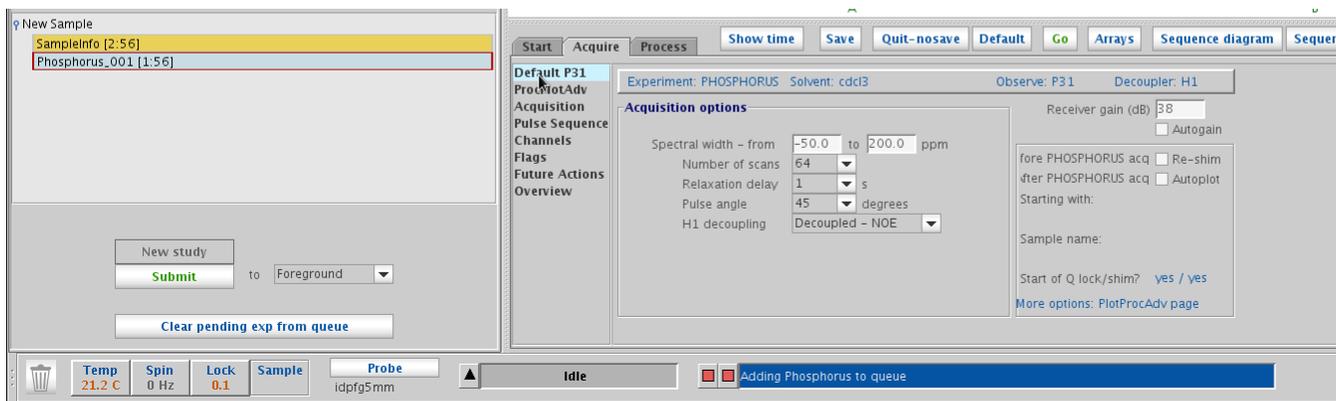


Figure 5: Customizing an experiment. If the parameter you want to change is not displayed, click on "Acquisition" on the left menubar, under "Acquire".

g. Click on "Submit" to submit experiments to the queue, or click on "Cancel" (above the study queue list of experiments to submit) to cancel submitting experiments.

5. Running experiments manually

a. Clear parameters

To make sure that your data are saved in the correct location, click on the "Clear" button (above the Sample Name field) before doing anything else.

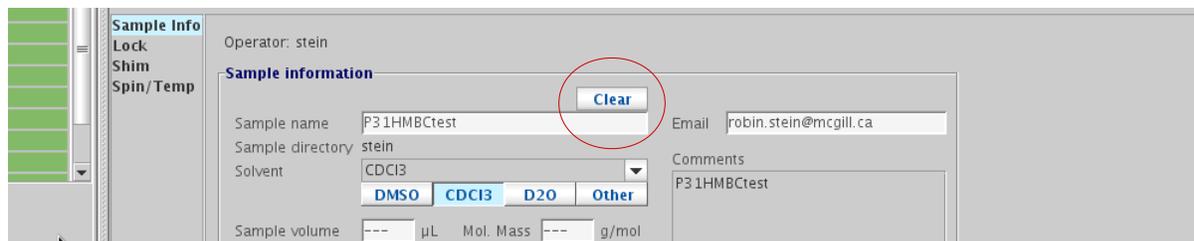


Figure 6: Location of "Clear" button, which should be pressed before doing any manual work, since this sets the sample directory properly.

b. Set sample name

Enter a sample name in the Sample name field.

c. Lock

Click on "Auto lock" (Start panel) to start the Find z0 routine to look for the largest ^2H signal and lock on it

d. Shim

Click on "Gradient shim" (Start panel) to start the gradient shimming routine



Figure 7: Auto lock and gradient shim buttons on the Start panel.

Note: if shims are not good enough, go to the Shim page of the Start panel and click on “Read default shims”, then rerun “Gradient shim.” At this point, you can also adjust the X, Y, XZ, and YZ shims by right- or left-clicking on them so as to maximize the lock level.

e. Choose and customize experiment

Click on the experiment in the “Experiment selector” on the McGill tab (or on another tab, as long as the experiment uses only ^1H and/or ^{31}P) that you would like to run. Modify any parameters you like under the Acquire tab. You might like to set **bs** (block scans), the number of scans after which data is written to disk and you can start processing, to a small number (do this by typing, for example, **bs=2** in the command entry line at the top of the screen. You can also set up kinetics experiments at this point. See section 11 for more information about all experiments, including kinetics experiments.

f. Run the experiment

Use the command **go** (to acquire and process) or **ga** (to acquire without processing) in the command bar or the Go button under the Acquire tab to start acquisition. (**go** is particularly recommended for 2D experiments, because it is easier to process them during acquisition than when **ga** is used.)

If you need to stop the acquisition, click Stop.

6. Saving and emailing data manually

Choose File / Autosave (this will ensure that your data is saved to /home/walkup/vnmrsys/data/<username> and not to some other directory). This will also email you your data. For this to work, you must have clicked “Clear” (step 5a above) and entered a Sample name (step 5b above), and there must be an email address listed in the Email field.

7. Processing data at the spectrometer

a. If your data is listed in the Study Queue on the left of the screen, you can double-click on it to open and auto process it.

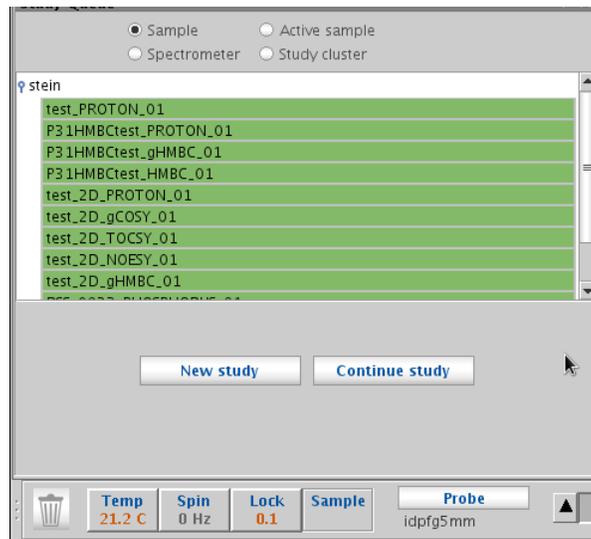


Figure 8: Experiments that have run already (green) and can be viewed by double-clicking on them. Experiments in blue (currently running) can be viewed if the number of completed scans (**ct**) is greater than the number of scans in the block scan (**bs**).

b. You can also go to the File menu and choose Open. Your data will be in /home/walkup/vnmrsys/data/<username>.

c. The basic processing command for 1D data is **wft**; for 2D data, it is **wft2da**. (All typed commands are entered in the command bar at the top of the screen.) Or, you can go to the Process tab and click on the Auto process button. If you try to process a 2D dataset and you get an error message about strflt, go to More 2D (under Process) and deselect F1 under Linear Prediction.

d. For phasing, type **aph** or click on Autophase in the Process tab. For manual phasing, click on the Phase mode icon to the right of the spectrum. Left click on the right side of the spectrum, then click and drag with the left mouse button (coarse control) or the right mouse button (fine control) for 0 order phasing (which affects all peaks across the spectrum). Make sure that the peaks at the right side of the spectrum are phased correctly. Then, left click on the right side of the spectrum. Click and drag with the left mouse button (coarse control) or the right mouse button (fine control) for 1st order phasing (which affects the peaks on the left more than on the right). Make sure that the peaks on the left side of the spectrum are phased correctly. Repeat 0 and the 1st order phasing as many times as needed. Then click on the Redraw icon to quit the phasing mode.

e. To adjust the vertical scale, type **vsadj** or use the mouse wheel. If integrals are displayed, then this command will adjust the integrals, so remove the integrals by clicking

twice on the Integrals icon to the right of the spectrum. To move the baseline of the spectrum, try typing **vp=10** or click and drag the far left side of the spectrum up by treating the mouse wheel as a button.

f. To peak pick, go to the Process / Default panel and click on Find Peaks. Then click on Peak Threshold to show the threshold. Use the left mouse button to position the threshold appropriately (click or click and drag).

g. To integrate, click on the Process / Integration panel and click on the Partial radio button. Click on Clear Integrals and then on Interactive Resets. Left click to start and to stop every integral (ie left click on the left and left click on the right of each peak) and then click on Scale display to fit. If necessarily, type the command **dc** for a baseline correction (which should level the integrals). To normalize the integrals, place the cursor on an integral with known proton number, enter that number in Integral Area, and click on Set integral value (below it).

8. Plotting and generating PDF of data at the spectrometer

a. Zoom in on the region you are interested in and adjust the vertical scale.

b. Go to the Process / Plot panel. Select the items you want to plot (eg Integral lines (Partial), Integral values (Horiz), Peak labels (ppm, top in the drop-down menu), Axis (ppm), etc.

c. To generate a PDF for saving or emailing, click on Preview

d. To print, click on Plot (which will print).

9. Processing data offline

Your data will be emailed to you as a ZIP file. Once you unzip it, you can drag and drop it into Mnova. Or, you can use the `vconv` command in TopSpin to open it there. See the TopSpin notes for more information about reading Varian data in TopSpin (1D data is straightforward, but some 2D data is problematic.)

10. Logging out of walkup interface to VnmrJ

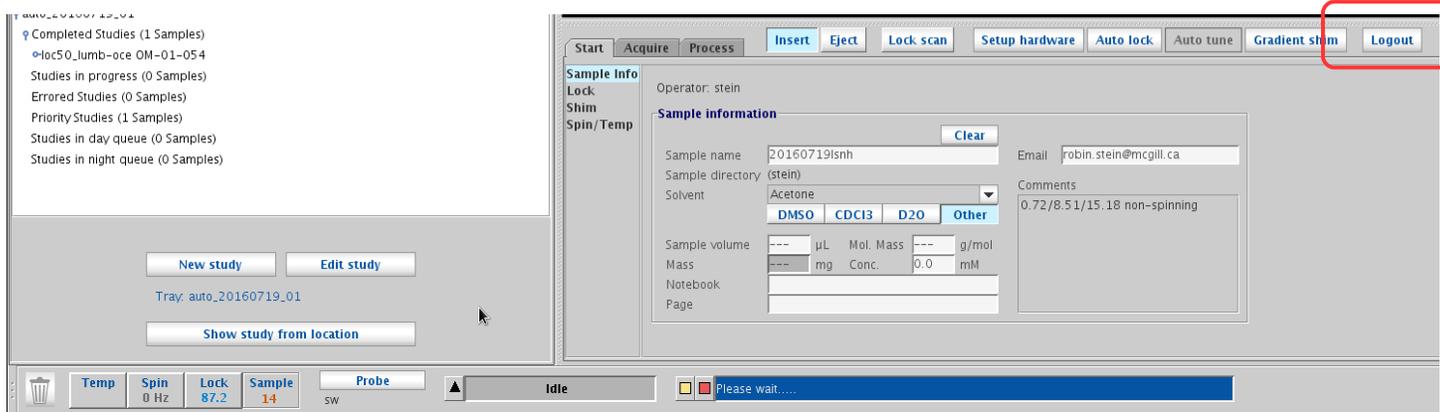


Figure 9: Log out of VnmrJ by selecting the "Logout" button. Do not quit VnmrJ and do not log out of Linux.

Click on Logout (right-middle of the screen, with Start selected) to log out of VnmrJ. Note: do not close the VnmrJ program or log out of Linux. After you log out, you should see the login screen (Figure 2) displayed.