

VNMRJ 4.2 INSTRUCTIONS: MERCURY 300 WITH AUTOSAMPLER

August 8, 2016

1. Sample preparation

a. Use only 8" tubes in the sample changer

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 (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. Rules for using the sample changer

a. Do not put a sample underneath the sample changer grip

b. Do not use position 100 for your sample (position 100 is for the dummy sample)

c. Do not use any position labeled "Do not use"

d. Never get in the way of the sample changer: stand back if you hear the air come on, indicating that it will move

3. Rules about time and experiments

a. Each sample can be run for 45 minutes during the day

b. Each sample can be run for as much time as you like during the night

c. The Mercury 300 autosampler instrument can **only** run ^1H , ^{19}F , and ^{13}C experiments.

4. Running experiments

a. Sample insertion

- i. Wipe NMR tube with Kimwipe
- ii. Place sample into spinner (from box under sample changer)
- iii. Adjust sample depth 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). The depth gauge should be adjusted to display between 68 and 70 at the bottom.



Figure 1: Sample of standard length inserted into spinner and properly positioned in depth gauge.

- iv. Put sample in tray, respecting all notes in point 2, above

b. Log in to the walkup NMR software

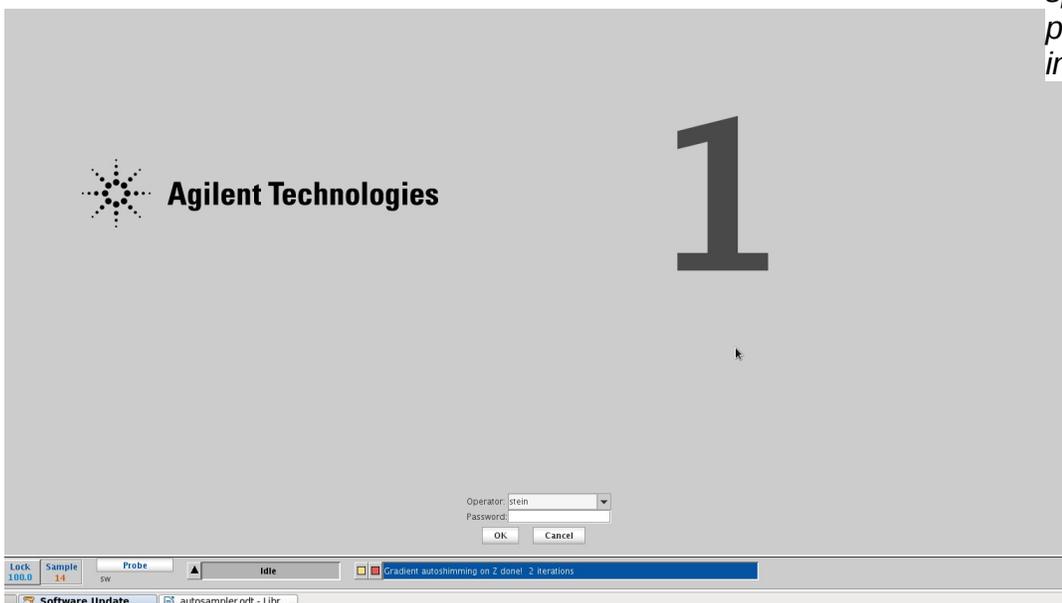


Figure 2: Login screen for NMR software. Enter your username and password to log in.

c. 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, 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.

d. 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.

e. Choose one or more experiments from the list on the upper-left of the screen. You can line up multiple experiments by clicking on them, one at a time. By default, the experiments will run during the day (first-come, first-served). However, if you exceed the permitted time for your sample (usually 45 minutes), subsequent experiments will be run at night. If you want to force an experiment to run at night, you must select the NightQ button below the list of samples before adding that experiment to the list.

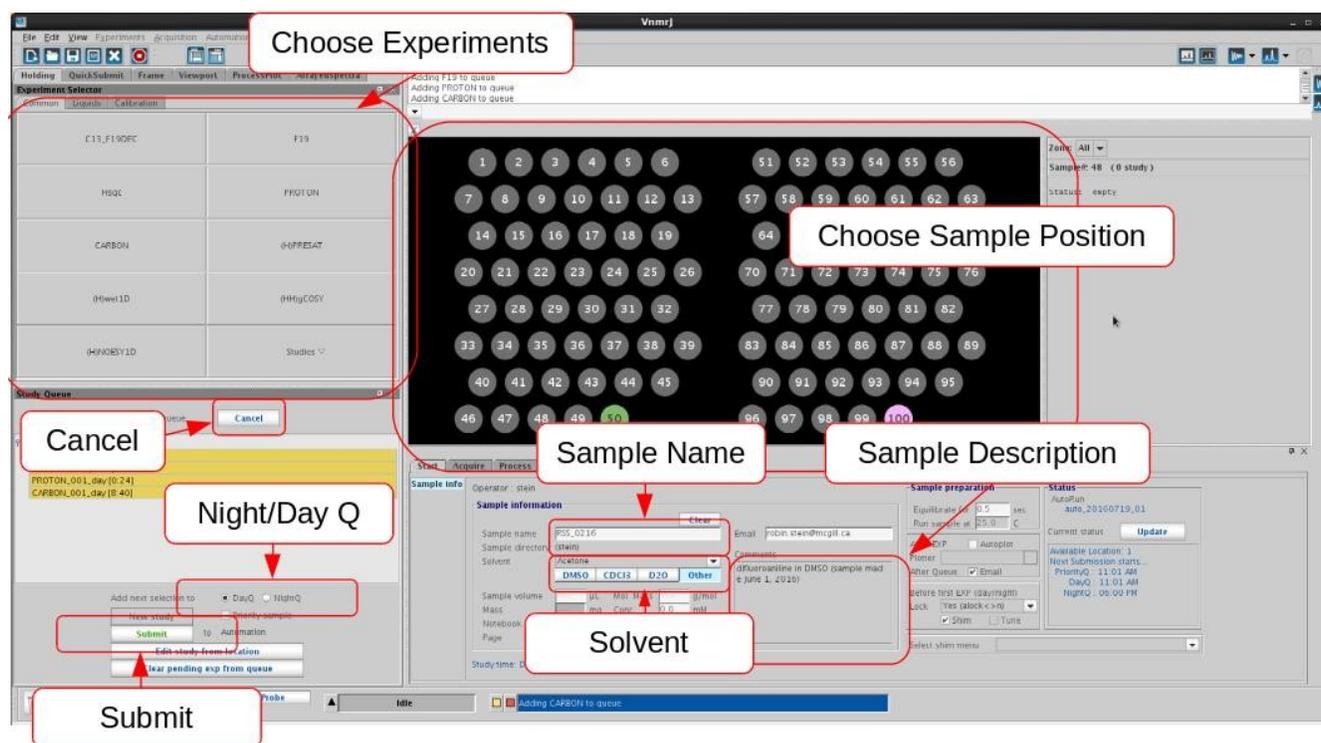


Figure 3: Submitting experiments to the sample queue. At a minimum, choose an experiment, enter the sample name, choose the solvent, choose a sample position, and push submit.

f. 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

g. 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.

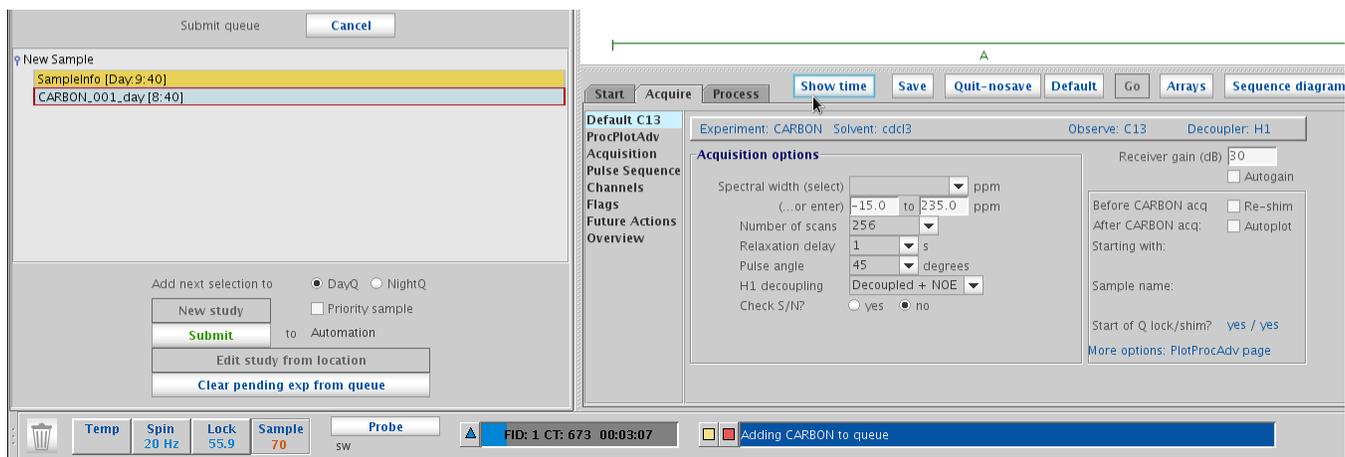


Figure 4: Customizing an experiment. If the parameter you want to change is not displayed, click on "Acquisition" underneath "Default C13" (currently selected).

h. 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.

i. To run an experiment on a second sample, repeat steps c through h. Note that the queue will contain the previously chosen experiments: you can further customize the list if you want.

j. Click on Logout (right-middle of the screen) 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.

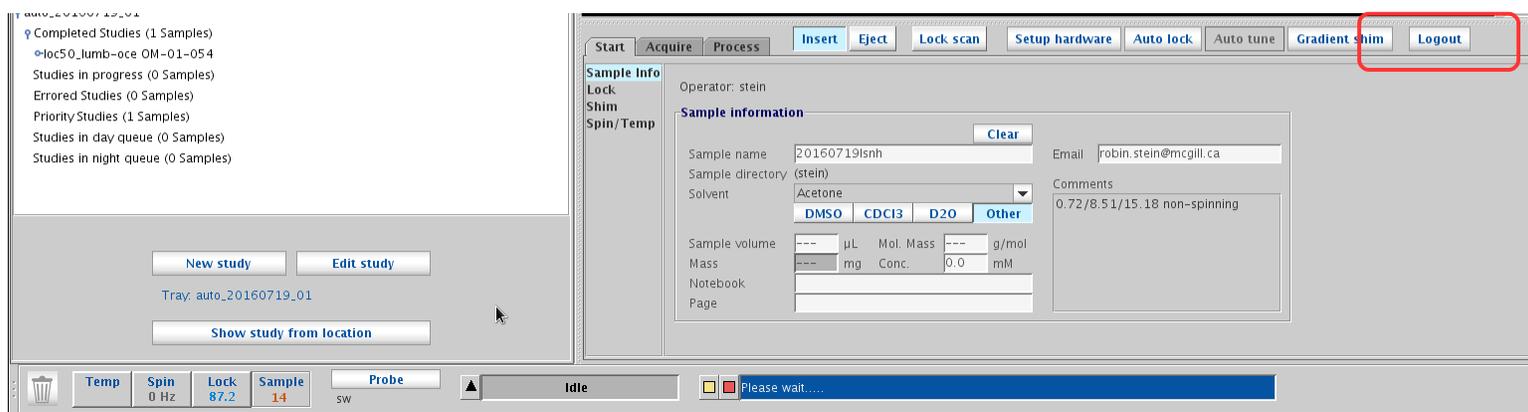


Figure 5: Log out of VnmrJ by selecting the "Logout" button. Do not quit VnmrJ and do not log out of Linux.

5. Deleting a submitted study (experiment)

a. Click on the Tray location (if you cannot see the Tray, click on the circle icon in the upper left of where the tray would normally be)

b. From the menu bar, select Automation / Tray actions / Delete Pending Study from a Location

- c. Click on DayQ, NightQ, or DayQ and NightQ, as appropriate

6. Modifying a submitted study (experiment)

- a. Click on the Tray location (if you cannot see the Tray, click on the circle icon in the upper left of where the tray would normally be)
- b. From the menu bar, select Automation / Tray actions / Recall and Edit Study from a Location
- c. Make appropriate modifications, select the position again, and click on Submit

7. Processing data

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.)