

NMR Instructions for the Superlab User

Preparing your NMR Sample

1. Load your sample into a long NMR tube with a green cap. (the short tubes with yellow caps are only for use in the Bruker 200 instrument)
2. Find a Varian spinner for your sample. These are in the top drawer behind the magnet or on the bench behind the computer table for the 500.
3. Use the depth gauge (the gold cylinder on the bench behind you) to insert your sample into the spinner. Adjust the sample so liquid covers the entire dotted rectangle marked on the inside of the depth gauge, and so that the liquid is roughly symmetrical around this rectangle. (The rectangle indicates where the RF coil will fit around your sample.)
4. With the spinner in hand, climb the ladder and insert the sample carefully into any free sample slot **except for position 1** (which is reserved for the resting lock sample) **or the active sample position** (which is the one that is currently oriented to drop straight into the magnet).

Instrument Setup

5. Go to the computer and check the status of the computer.
 - a. If a user is already logged in and the VNMR software is running, click the Start tab below the spectrum or sample changer ring and check that the operator is the correct one for you (should be "spring11"). If the current operator is a research group user, shut down VnmrJ and log out of the system (go to System: Log Out at the linux desktop). Then follow step b. below.
 - b. When you see the linux login screen, log in as "superlab" with password "*slr2010!*". Then start VnmrJ and select your semester from the drop down list and log in with no password.

Now you are logged in as the correct user and ready to run a sample.

- a. If you are running the first sample of the day and the lock sample is sitting in the magnet (this is the case if the sample changer is at position 1 and the yellow light is on), go to "***File: New Automation Run***" to start a new queue. Go to step c.
 - a. If you are not running the first sample of the day, go to the next step.
 - b. If a spectrum is showing, click on the small circle at the top left corner of the spectrum to display the sample changer control (a ring of 12 large dots).
 - c. Click once on the sample changer position containing your sample: it should now appear with rainbow colors around it.
 - d. At left, click on the kind of experiment you want to run on your sample (e.g. Proton) and a new entry should appear in the Study box at bottom left. This new entry should have three levels: "New Sample", a sequence, and a signal area (in green). Double-click on the third entry (the one with the smallest type). The text will turn black and become italicized.

Experiment Setup

Adjust desired experimental settings at right below the sample changer window:

At the **Start** tab:

- i. Under the **Study** tab:
 1. Enter a file name with your name and any other sample information needed to identify the sample. (The date, nucleus, pulse sequence, and solvent will be automatically appended to the file name.)
 2. Select the solvent.
 3. Make sure that the boxes for AutoLock, Tune and Gradient Shim (2H gradient) are all checked.
 4. Check the Plot box if you want an immediate printed output of your spectrum as soon as it finishes.
 5. Leave all other tabs as is: these are for manual control of the instrument.
- a. At the **Acquire** tab, change any parameters as desired for your spectrum. Pay particular attention to the following items:
 - . Spectral width
 - i. Spinning (check the box)
 - ii. Number of scans

When everything is the way you want, go to bottom left and click “Submit DayQ”. Your sample position should turn yellow on the screen, then blue when it becomes the active sample. Watch the sample changer move to your position. At any time you may return to step 8 and input a new experiment at a different or the same position.

How to Eject Your Sample

If there is a sample changer error due to a poorly shaped spinner, your run will abort with an error and your sample position will turn red. If this happens, delete your run by dragging it from the “study” window to the trash can in the lower left and go back to step 6d. If this happens repeatedly and the sample position remains on your sample, type “e” in the command line (above the sample changer picture) to eject your sample, change spinners and put your sample in a new slot. *Then go back to step 6d.*

Data Processing

When your experiment has completed, the position of your sample will turn green. At this point you can process your data by double-clicking on the data file and going to the **Process** tab. This gives you lots of options for integrals, plotting, etc.

If another run is not immediately active and you are the last user, your sample will stay in the magnet. ***BEFORE YOU LEAVE: Go back to step 6d and select slot 1: then run an automated proton spectrum of the lock sample. Close down VnmrJ and log out of linux by going to System: Log Out.***