

Varian 500MHz NMR instructions for Organic students

Preparing Your NMR Sample

1. Pipette 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 magnetic 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. The VNMRJ software should be running and the operator should be "chem221".
6. 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).
7. Click once on the sample changer position containing your sample: it should now appear with rainbow colors around it.
8. 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 All Data box for 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. Under the **Acquire** tab, change any parameters as desired for your spectrum (this is usually not necessary). 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. The next student may now return to step 4 and input a new experiment at a different position.

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, or just take the printed spectrum from the printer

Run a ‘Lock Sample’

1. The last person to run a spectrum should do this before they leave.
2. Make sure the ‘lock sample’ is inserted in slot #1 in the autosampler.
3. Run a proton NMR on the lock sample
4. Print out lock sample spectrum and put in log book binder to monitor the performance of the instrument over time.
5. Log out of WNMRJ and log out of the computer.
6. Clean up any mess and glassware.