

Routine 1D Acquisition on Varian Inova-500 & VNMRS-500

GENERAL PROCEDURE FOR DATA ACQUISITION

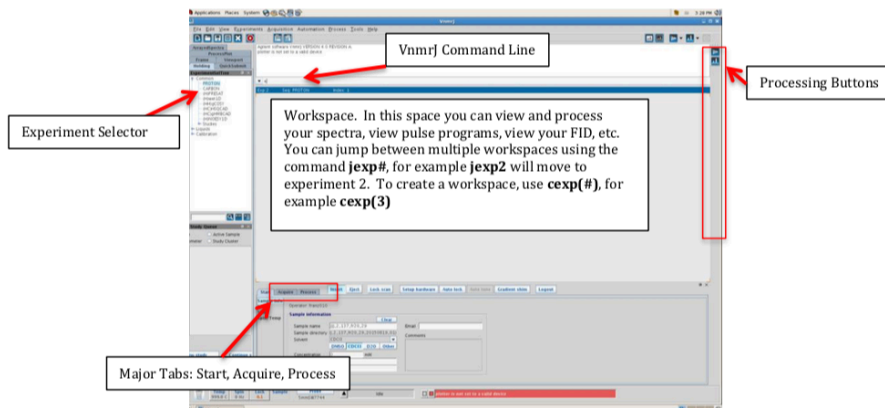
The general procedure for acquiring NMR data on all spectrometers is similar, but commands differ depending on the software. For every sample, you must perform all of these steps. However, if you are running multiple experiments on the same sample (e.g., first ^1H , then ^{13}C , then HSQC), you do not need to re-lock or re-shim unless you change the probe temperature or remove the sample from the instrument.

- 1) Log into the computer and double click VnmrJ icon
- 2) Insert sample (type **e** to remove the dummy sample; set depth of your sample; **i** to insert your sample)
- 3) Set-up initial acquisition parameters (Select **Experiment** -> Exp. to Run)
- 4) Lock on to deuterated solvent (select your **solvent**; hit **find Z0**)
- 5) Tune the probe (Pro-Tune on Inova, Q-Tune on the VNMRS)
- 6) Shimming (Gradient Shim or manual)
- 7) Check acquisition parameters (**Acquire** -> check your parameters)
- 8) Set the receiver gain (**autogain**)
- 9) Acquire data (**go** or **ga**)
- 10) Process data (**wft** ↵ **aph** ↵ **bc** ↵)
- 11) Remove sample (**e** to remove your sample; **i** to insert dummy sample)
- 12) Log off spectrometer

LOGIN AND INSERT YOUR SAMPLE

If no one was logged into the spectrometer computer, there will be no data acquisition active. Login with your username and password. Double-click on the VnmrJ icon to launch VnmrJ.

- 1) First you will need to remove the dummy sample from the instrument. Eject the dummy sample with the command **e**. Alternatively you can click the **Eject** button (by the major tabs). Place the sample in the sample handling area.
- 2) Check to make sure that the spinner is clean. Avoid touching below the black and white tape with your bare hands.
- 3) Wipe the outside of the sample tube with a clean KimWipe and insert it into the spinner. Make sure the tube fits snugly in the spinner. If a tube is loose in the spinner, do NOT use it. Please use the other spinner. Loose tube/spinner combinations can get stuck in the instrument or easily break in the probe, which will cause extended downtime and require expensive repairs.
- 4) Set the depth using the depth gauge. After you set the depth, remove the sample and spinner from the depth gauge, and wipe the outside of the sample tube again.
 - a. If your sample height is not ≥ 45 mm, make sure to center the sample around the line marking the center of the *rf*-coils when using the depth gauge.
 - b. Improperly-positioned samples can get stuck in the instrument or easily break in the probe, which will cause extended downtime and require expensive repairs.
- 5) Carefully place your sample into the instrument. It should float on a bed of air.
- 6) Type **i** or hit the **Inject** button to insert your sample. You should hear a clicking sound when the spinner settles properly into the instrument.



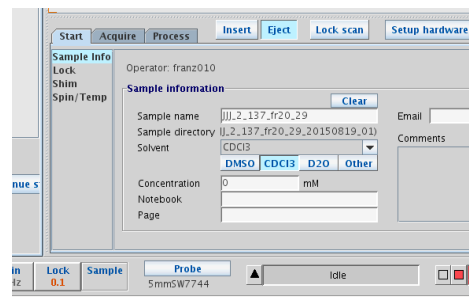
SET UP INITIAL ACQUISITION PARAMETERS

Read in a generic parameter set. From the menu at the top, select **Experiments** -> **PROTON** (or other experiment).

LOCK ONTO DEUTERATED SOLVENT

The NMR lock is a ^2H (deuterium) lock, which compensates for small changes in the magnetic field that might occur during data acquisition. The NMR lock will only work if you use a deuterated solvent. Automatic locking is available on all instruments.

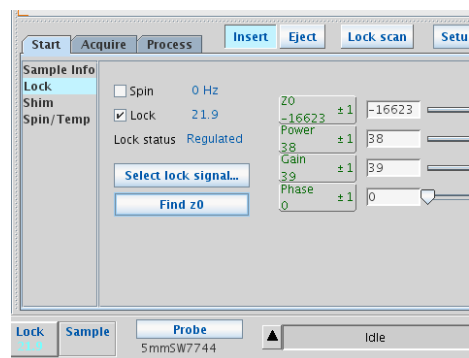
- 1) Under the **Start** main tab, there is an option for **Sample information**. Select your solvent using the icons, or using the drop-down list.
 - a. You can skip the solvent selection menu and call the solvent directly by invoking its "short" name, which appears in the drop-down. For example, **solvent='cdcl3'**
- 2) Then under the **Start** tab, select **Lock**, and hit the **Find z0** button.
 - a. If you have difficulty locking, please read in the most recent shim file. Under the **Start** tab, select **Shim**, then hit **Read default shim**.
- 3) The **Find z0** button does not always set the Lock **Phase** correctly. Check the whiteboard to see the most recent lock phase value and enter the value into the box and hit the enter key on your keyboard.



TUNE THE PROBE

Inova: Hit the **Pro Tune** button. This will take a few minutes and you will hear the instrument make a bunch of sounds. A window will pop up, and notify you when the tuning procedure is finished.

VNMRS: Tuning on the VNMRS is done by hand. Be careful not to overtighten the tuning rods as you will break the probe. Hit the **Q-Tune** button and walk over to the probe. Turn the appropriate tuning rods while watching the monitor's wobb curve.



SHIMMING

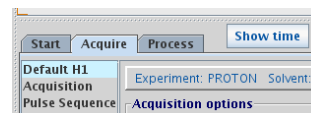
To produce an NMR spectrum with good resolution, the magnetic field must be homogeneous throughout the volume of sample. Shimming is a way of minimizing the magnetic field inhomogeneity by using magnetic field gradients. Refer to the handout entitled "Adjusting Magnetic Field Homogeneity" for a more comprehensive discussion of shimming.

All of the Varian instruments are equipped with gradient shimming. For a quick scan, it is often much faster to manually adjust the field gradients to maximize the lock level for optimal shims. In order to do this, select the **Shim** tab. A list of the different shims will appear. The idea is to maximize the lock level by adjusting your Z1 and Z2 shims iteratively.

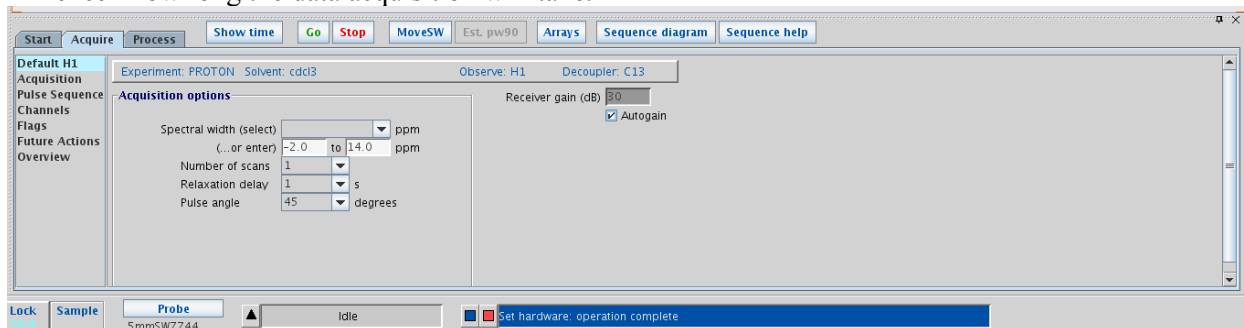
In order to utilize gradient-shimming, select the **Shim** tab, then hit the **Gradient Shim** button.

CHECK THE ACQUISITION PARAMETERS, SET THE RECEIVER GAIN & ACQUIRE DATA

- 1) Go to the **Acquire** tab. Check the acquisition parameters as needed. Do not change any acquisition parameters that you do not understand. For routine data acquisition, you can use the default parameters.



- 2) We suggest using **nt** as a multiple of 8. If you change the number of scans, you can type **time** to check how long the data acquisition will take.



For all nuclei except natural abundance ^{13}C , you will need to set the receiver gain (**gain**). (The receiver gain is already set to the maximum for ^{13}C). If the receiver gain is too low, you will miss small signals. If it is set too high, you will get artifacts in your spectrum.

- 3) Type **autogain** or select box next to the autogain option in the Acquire tab.
- 4) Type **go** or **ga**, or click on the **Go** button to start the data acquisition.
- 5) The Varian spectrometers do not automatically save your data like the Bruker spectrometers do. Click File -> Save or hit CTRL+S to save your data. Save the data in "*your_username/vnmrdata*" folder.

PROCESS DATA

- 1) The command **wft** will perform an exponential multiplication and Fourier transform.
 - a. To perform Fourier transform without apodization function, type **ft**.
- 2) **aph** will automatically adjust the phase of the spectrum. You can type use the manual phasing routine if automatic phasing does not work for you.
- 3) **bc** will correct the baseline.

REMOVE SAMPLE + LOG OFF SPECTROMETER

- 1) Type **e** or hit the **Eject** button to remove your sample.
- 2) Carefully replace your sample with the dummy sample, then type **I** or hit **Inject**.
- 3) Close VnmrJ by either clicking the "X" in the upper right corner or type **exit**.
- 4) Log off the spectrometer. Be sure to "Confirm Logout" (do not use "switch user"). A keyboard shortcut you can use is **CTRL + Alt + Backspace**.