# Quickstart Guide to Acquiring Data on the Agilent 600

- **Getting Started**
- **Common 1D and 2D Vnmrj commands**
- **Troubleshooting**

## *Getting Started*

1. Insert your sample into a sample holder. Make sure that the sample is appropriately positioned. The minimum sample volume in a normal NMR tube is 500 µl. Typical sample depth is 61 mm.
2. Eject the lock sample from the magnet. The sample should be put back when you are done acquiring data on your sample.
3. Insert your sample into the magnet.
4. Set temperature using the **temp** command; be sure to use appropriate settings on the FTS water bath (the temperature of the water bath should be 10 °C lower than the desired probe temperature).
5. Set up the appropriate nuclei that you will detect.
    - The usual setting is: **tn='H1'**, **dn='C13'**, **dn2='N15'**.
6. Once the probe temperature has stabilized, you may tune the probe.
    - The usual setting is: Chan #1: H1, Chan #2: C13, Chan #3: N15.
    - It is not necessary to tune the probe every time you change the sample unless your sample is really salty (salt concentration is >150 mM). You can check the tuning quality in Vnmrj but the measured 90° pulse-widths are the best reflection of tuning (for $^{1}$H pulses at high power, this should not exceed 9 µs).
    - The tuning and matching rods are extremely delicate; if you meet with any kind of resistance, **stop turning the rods immediately** and turn the rod in the **opposite** direction a couple of times. This way the next person to tune the probe will not accidentally break the rod if it has reached its limit.
7. Optimize lock signal.
    - Open the "Acqi" or the 'lock window'; if button is absent on the menu, just type **acqi**.
    - Adjust $z^0$ to maximize the lock signal (**use extreme caution while changing this - note the $z^0$ value before you start!**)
    - Click "Lock" button.
    - Adjust "lock gain" (maximum 40) and "lock power" (maximum 34 in 10% $D_2O$ samples and 20 in 100% $D_2O$) so that the lock level is around 70.
        - Whenever you change "lock gain" be sure to re-optimize the lock level using "lock phase".
8. Shim on your sample starting from an appropriate shimming file.

- **rts('yourshimfile') su. If you do not have a shim file that is okay too. In this case shim from wherever the previous user left off. Be sure to check the value of $z^0$ (as of August 2004, it should be between +15500 and +16000).**
- Manually shim the axial shims $z^1$, $z^2$, $z^3$, $z^4$ and then the radial shims x, y, xz, yz, $xz^2$, and $yz^2$. Reshim the axial shims.
- You may also set up a gradient shimming protocol. Before doing so, I highly recommend saving your shims using the command **svs('giveashimfilename')**. Go to exp2 (for $H_2O$ samples) or exp3 (for $D_2O$ samples). Be sure **pw=1**, **gain=0**, **gzsize=5** for $H_2O$ samples. Type **gmapsys** and you will get a new set of menus. Select "Make Shimmap". After this finishes, set **gzsize=4** and click on "Autoshim on Z". Repeat but set **gzsize=5**.

9. Calibrate proton pulse widths in exp5 (for $H_2O$ samples) or exp6 (for $D_2O$ samples).
   - Set **tpwr=54, tof=0**, **pw=1**, type **go**. After it finishes, type **wft** to apply Fourier transform to the time-domain data and phase the spectrum. Expand the spectrum zooming in on the water resonance. Place the cursor on top of the water resonance. Then type **movetof full f**. Note the value of **tof** - you may need it for your experiments later. Type **ga**. Zoom in on the water resonance.
   - Use the **array** command to array **pw**. Start with number of elements in array: 7, starting value of 32 and array increment of 2. Type **ga**. Note the value of **pw** at which the water resonance goes through a null. Repeat the experiment with a more rational starting value and array increment of 0.4.
10. Set up appropriate parameters in your experiment. These typically include **pw** and **tof** (determined from step 9) but can include a host of other parameters. Please start from a set of parameters that are known to give satisfying spectra. Consult with Yongbo or Ishwar about any new experiments.
11. Finally, you do not need to change any acquisition parameters for titration experiments. Spend time optimizing parameters for your 1st experiment and use identical experimental parameters thereafter.

## *Commonly Used Vnmrj Commands*

1. Start a 1D experiment and fourier transform and phase using previously set parameters: **ga**. If you do not want any processing after acquisition, just type **go**.
2. Start a2D or 3D experiment: **go** (**never type ga for multi-dimensional experiments!**)
3. Save the acquired data: **svf('giveanappropriatefilename')** - be sure you are in the correct directory (type **pwd** to check). To navigate between directories, use the "Set Directory" button. The "Parent" button takes you up one level.
4. Process 2D data and correct dc offset: **sinesq(2,2*ni,'f1') wft2da dc2d('f2')**

5. Plot a 2D spectrum in contour mode on the screen: **dpcon('pos', 20, 1.2)**
6. Plot a 2D spectrum from printer (be sure to choose the appropriate region you want to plot): **pap pcon('pos', 20, 1.2) page**. Before you do be sure you have set the the plot parameters appropriately using the command **sc=0 wc=150 sc2=0 wc2=150**.
7. Plot a 1D spectrum from printer: **pl pap pscale page**
8. Plot arrayed spectra: **plww page**

## *Commonly Encountered Problems*

1. The "Acqi" button is missing from the main menu
    - To resolve this issue, just type the **acqi** command to get the lock window.
2. Occasionally, the acquisition processor locks up (esp. when you abort an acquisition immediately after starting it), which manifests itself in many ways:
    - the spectrometer is unresponsive and does not execute any command
    - the Status in the Acquisition Status window is set to "Inactive"
    - you cannot open the lock window using the **acqi** command
        - To resolve this issue, follow the steps below:
            1. Open a Unix window and type **su acqproc**. You will get the following message: "Killing acqproc"
            2. Open the left door of the console. Locate and push the small, red RESET button on the left-most board marked as the CPU board.
            3. On the Magnet/Sample regulation board (seven boards to the right of the CPU board), the fail light should turn red.
            4. Wait until the fail light turns off and the READY AP light turns green. Concomitantly, the green lights on the pulse/probe temperature status panel (next to the computer monitor) should flash and when ready should turn steady. This will take a couple of minutes - so be patient!
            5. Type **su acqproc** in the Unix window.
            6. The Status in the Acquisition Status window should now revert to "Idle". If you do not get the lock window, type **acqi** in vnmr.

**This is by no means a comprehensive command reference guide!** For more commands and details about the commands given in bold above, please see the manual "VNMR Command and Parameter Reference". Furthermore, "User Guide: Liquids NMR" and "VNMR User Programming" also contain very useful information on setting up typical 2D experiments and understanding pulse sequences. We highly recommend those manuals to advanced NMR users. Talk to Ishwar or Yongbo if you have questions.