

## Troubleshooting Guide for Varian/Agilent NMR Spectrometers running VnmrJ

---

### Problems during acquisition

The vast majority of VnmrJ acquisition problems are due to interrupted communications between the spectrometer's console and the host (Dell) computer. Some of these problems include:

- The acquisition doesn't start when you give the **go** or **ga** command or when you click on the [Acquire] button.
- The spectrometer doesn't obey commands or buttons like [Find Z0], [Gradient autoshim], [Acquire], etc.
- The sample is not ejected with the [Eject] button or the **e** Command.
- The message "Setup complete" does not appear after you type **su**
- The message **Active** or **Inactive** or **Interactive** appears continuously on the bottom of the Vnmrj window instead of the normal green **Idle** even when no acquisition or shimming is in progress.
- The message: "Cannot set hardware during interactive acquisition" or "Cannot do {some command} when an acquisition is active or queued" or "sethw cannot proceed, another user's experiment is already active" or "Acquisition system is not active" appears on Vnmrj's info bar.
- The lock display is slower than normal (it refreshes the image less than two times per second).
- The red RCVR OVFL light on the status unit adjacent to the monitor of the Inovas is continuously lit even when no acquisition is in progress (it is normal for it to flash during pulses or to be on when the [Lock Scan] button is depressed).

To solve these problems, try the following steps, in order, until you resolve your problem:

1. Close out of VnmrJ and re-open. If that doesn't work:
2. type **abortallacqs** on the command line of Vnmrj, wait not less than 1 minute and then type **su**. If successful, the message "Setup complete" should appear. You may need to reload the **sw** shims and lock parameters and re-shim after this.
3. If the above doesn't work, you should perform an **su acqproc**
  - a. close out of VnmrJ
  - b. right click on the desktop and open up a terminal window
  - c. type **su acqproc** in the terminal window. It should say "Stopping Acquisition Communications". Wait until this process is complete (usually about 10 seconds)
  - d. type **su acqproc** again to restart console communications. A new window with a message like "Acquisition Console at 600 MHz ready" might appear
  - e. Close out of the Terminal window and Open up VnmrJ.
  - f. Be sure to read in the shims: **rts** "enter", **sw** "enter, **su** "enter"
4. If you are still having issues, you may have to **reset the console**.
  - a. Close out of VnmrJ and open up a terminal window again.
  - b. Stop communications by typing **su acqproc** wait 30 seconds.
  - c. Go to the console and find the reset button. On the 600 MHz it is inside the right door of the console, near the bottom and to the right. On the 400 MHz it is inside the left door of the console, near the center and to the left. On the 300 MHz spectrometers it is on the back of the console. Press this reset button, and wait about 1 minute.
  - d. In the terminal window, type **su acqproc** again.
  - e. Close out of the Terminal window and Open up VnmrJ.
  - f. Be sure to read in the shims: **rts** "enter", **sw** "enter, **su** "enter"
5. If you are still having issues, leave a note in the logbook, and contact NMR Facility staff via phone call or email immediately so we can resolve the issue. Please consult the call-sheets posted at each instrument to determine who to call.

### Locking and Shimming problems

- a) I cannot find the lock signal or lock the sample.
- b) The lock signal is unstable.
- c) Shimming is difficult.

The majority of Locking and Shimming problems come from one of two things:

1. You forgot to load in the default shims. Make sure you do this before locking. To load in the default sw shim set, type `rts "enter" sw "enter" su "enter"` in the VnmrJ command line.
2. Your sample preparation is poor. If you expect your lineshape to look good after simply touching up the Z1 and Z2 shims, you must make sure your sample is prepared correctly. Here are a few suggestions for a properly prepared sample:
  - a. Make sure you are using a deuterated solvent. Yes, that sounds silly but... it has happened.
  - b. Make sure your sample height is about 5 cm. Eject the sample and verify that you have enough solvent to fill the window in the depth gauge.
  - c. Make sure you don't have any undissolved material in your sample. If you still have residual solid material, go back to your lab and try to remove this material with a simple filtration or centrifugation. Often using a bit of kimwipe wadded up in the base of a Pasteur glass pipette is enough to filter out undissolved material.

If everything is correct, it is possible that something in your solution or tube may be making the lock signal so broad that the lock level is too low to be noticed. This includes paramagnetic metals, particles in suspension, high viscosity or a bad tube. The lock parameters; Z0, lockpower, lockgain and lockphase depend on solvent characteristics and is different for the various solvents. For example, acetone requires very low lock power while chloroform uses higher power. Using high power with acetone leads to signal saturation which in turn produces instability in the lock signal. These parameters depend also on the instrument and probe.

Also if your sample is not the "typical" organic sample, i.e. your solvent is not common or has a high concentration of salts, you may also need to adjust the lock phase as well as the other lock parameters.

If the sample is locked but the lock level is unstable you are probably using a lock power that is too high for your solvent. Reduce it until the level is stable. You can increase the gain to the maximum if needed.

### VNMRJ is "locked by active process"

This can happen if you have more than one VNMRJ window open at one time. To fix this, simply close the most recent VNMR window that you have opened.

### "variable DN undefined" error message and VNMRJ appears all greyed out

This happens when VNMRJ is exited in an unorthodox way. Look above the command line (you may have to scroll up) and you should see something that says unable to unlock experiment # (an actual number). In the command line type `unlock(#)` and press enter. The screen should appear normal now

### Receiver overflow/ADC overflow

This happens if the gain is set to high for your sample. It will result in "clipping" of the fid which will cause your spectra to look funny. Simply lower the receiver gain and recollect your spectra, or use a lower tip angle (for example, use a 30 degree pulse instead of a 45). Note, the receiver gain and the lock gain are different! Lowering your lock gain will have no effect. You also can select the Augogain option in the Acquisition parameters.

If your receiver gain is set as low as possible and you still see ACD overflow errors, this is probably because you have too much signal, either because your sample is crazy concentrated (10 Molar), or you are not using a deuterated solvent.

**When issuing the go or ga command, the message "Auto gain failure, gain driven to 0, reduce pulse width" appears and no acquisition takes place.**

This message appears when the sample is very concentrated or contains large amounts of non deuterated substances (solvents or water). In all these cases, the signal it produces is so strong that it overloads the digitizer. To reduce it to manageable levels, you can reduce either the pulse length or the power. As the message is trying to tell you, try reducing the pulse width. Type `pw=1` and try again. If this doesn't work, type `tpwr=tpwr-10` and try again. If this doesn't work, take your sample out of the magnet, go to your lab and dilute it or prepare a new, diluted sample with good deuterated solvent.

**The [Lock Scan] button is frozen. It remains depressed and doesn't show the lock signal.**

To unlock it, on `vnmrj`'s command line type "lock scan"

**Z0 is off the scale for some solvents**

Please let someone from the NMR facility know so that we can reset the lock frequency.

**Spectra appears to be all noise**

It's likely you are not tuned correctly. If you're on the 600, check the tuning for proton and carbon using the `trtune` command. If you're on another instrument, check the Probe Tuning guide, or ask for help from the NMR TA's or NMR Facility staff

**The temperature increases to a value above room temperature even though I didn't attempt to change it.**

Go to the "Start, Spin/Temp" panel and make sure that "Control temperature from this panel only" is disabled. Type `temp='n' su`. The temperature should start to decrease and the green light on the status unit on the Inovas should go off indicating that the heater is turned off. If it doesn't happen, press the [Reset VT Controller] button in this parameter panel. If the temperature still doesn't go down, reset the communication with the console with `abortallacqs` as explained previously in this document and perform a hard reset on the VT unit as follows. On top of the console on all spectrometers, you will see the power plug of the VT unit (it has a red tag labeled "VT") plugged to a power strip. Simply switch off the power to the strip, wait 10 seconds and switch it back on. Then type again "temp='n' su"

**The Sample doesn't spin.**

First, we don't recommend spinning because of possible damage to probes, and many pulse programs will now work if you are spinning your sample. However if you feel you must spin your sample, you may. In general, most spinning problems are due to grease, dirt or sample residues accumulated in the spinner and in the spinner housing located inside the magnet. The first one is easy to clean, but the latter requires removal of the housing and reshimming of the probe which is very time consuming. So please, always handle the spinners with clean hands, holding them from the black-painted band only and don't drop them. When a spinner is dropped by accident or negligence it may become permanently unbalanced, giving rise to what looks like severe "spinning sidebands" or spurious signals around all your peaks and difficulty spinning the sample. Spinners cost around \$200 to replace.

Go to the "Start, Spin/Temp" panel and turn spinning off by clicking the [Spin Off] button. Then, turn it on with the [Regulate Spin] button. A click should be heard around the magnet's legs. If this doesn't work, eject your sample and clean the outer surface of the spinner with a Kimwipe. Clean also the lower rim. If this doesn't work, try a different tube.

Please let us know if you can't get the sample to spin, but keep in mind that you can still get a perfect spectrum as long as the sample is properly shimmed. In fact, most routine 2D spectra is done with spinning turned off.

**I broke a sample**

Please, oh please, try not to break your samples. Around 99% of these "accidents" happen because the user is careless or too much in a hurry to handle the samples with due care.

If you do break a sample, immediately clean the area where the solution was spilled. Inspect the spinner on the outside and inside and make sure it has no sample residues or glass pieces. If sample was spilled inside the spinner, alert the NMR facility staff. If the sample broke inside the magnet or some of the solution or glass pieces went into

the magnet, notify the staff immediately. Failure to follow these rules may result in costly repairs and termination of your user privileges. Place a "Do not use, sample broken" message in the computer.

**I dropped an empty spinner in the magnet and now it does not eject it.**

Well, it is probably too late to tell you but, don't do it! You won't be able to get the spinner out without some kind of trick or tool and the spectrometer will be out of service until we go to the lab to do it for you. Contact someone from the NMR facility and hang a sign on the monitor of the computer.

**I dropped a sample tube without spinner in the magnet**

Notify the staff immediately. Place a "Do not use..." message describing the problem in the computer. Go to one corner of the lab, facing the wall and stay there for one hour until you learn your lesson.

**All my peaks, even singlets, appear as fine doublets or multiplets.**

This problem is usually due to poor shimming or a weak lock signal. Shim your sample carefully and make sure the lock is on and the lock level is at least 50%.

**All my peaks show "waves" (sinusoidal distortions) on both sides.**

The acquisition time **at** is too short for your sample. Increase it, for example **at=at\*1.5** and use some kind of apodization, for example **lb=1/at** and measure your spectrum again. The appropriate values for **at** and **lb** can vary greatly depending on your sample, nucleus, relaxation times, etc.

**I logged in and the screen is black**

Press ctrl-alt-backspace (NOT ctrl-alt-delete) to log out and let the NMR facility staff know that this has happened.

**I got something stuck to the magnet**

Please inform someone who works in the NMR facility immediately. Stand outside the NMR room, as an object stuck on the magnet can cause a quench. Do not try to pull the object off by yourself, you can permanently damage the magnet.

**Software problems**

**When attempting to join an existing workspace (experiment) or when starting vnmrj, the program prints "experiment locked by active process" or "experiment X locked" and it is impossible to access that experiment.**

This happens when some process in vnmrj aborts unexpectedly but vnmrj thinks it is still alive. It also happens if you are running more than one instance of Vnmrj. If you can, in vnmrj's command line type **unlock(x, 'force')**, where x is the experiment number to unlock. If this fails, quit vnmrj, open a terminal window and type "**rm ~/vnmrsys/lock\***".

**Some of Vnmrj's parameter panels (where buttons and parameters are found) are incomplete, appear completely empty or are missing.**

This can be corrected by simply exiting vnmrj and starting it up again, or from the main menu select "Edit, Parameter pages..." and simply close the window that appears. If this does not fix the problem then contact NMR facility staff.

**I cannot see error messages or status information.**

The lower edge of the vnmrj window contains a Status Bar that displays error messages and other very useful information. This bar may be hidden from view by the Linux Task Bar (the lower bar on the screen, similar to MS-Windows' task bar). It is important to make sure the status bar is always visible. You can do this by resizing or maximizing the window by clicking the middle button on the upper right corner of the vnmrj window.

**Where is Vnmrj's command line?**

Put the mouse on top of the spectrum and slowly move it up (not pressing any button) until it goes just past the top edge of the spectral area and changes into a vertical double arrow. Now press the mouse button and drag down the command line and notification area.

#### **The computer is frozen or Vnmrj is frozen.**

This may be due to several causes. Despite Varian's claims, Vnmrj is a complex, old, heavily patched and buggy program, and may be very slow to load if its locator's database is used. Vnmrj may appear like it is frozen, but it may be just "thinking" hard. Click the "close" button on the window title bar (the rightmost button) several times and wait a few (20-30) seconds. This usually kills vnmrj. If vnmrj is not responding, but Linux is working (you can open the main Red Hat menu), open a terminal and type "xkill". Then click anywhere inside the vnmrj window to kill it and run vnmrj again. If this doesn't work, log off, log on and try again. If the problem continues please let us know to help you. If Linux is not responding (keyboard and mouse are dead or extremely slow) press ctrl-alt-backspace. You should get back to the Log In window. Log in again. If the problem persists, log off or press ctrl-alt-backspace and select "Reboot" from the login screen. If you get a black, character-only screen with a login prompt, login and type "reboot". This behavior has been observed on a few occasions on the Mercury 300. On fewer occasions, the computer is not frozen but the mouse is inactive. The computer and console remain active. Unplug the mouse from its USB port and plug it back in to reactivate the mouse. Unless it is an emergency, do not power off the computer as this may corrupt the computer's file system.

#### **A previous user left the computer locked with a screen saver**

If it is your time to use it, and the previous user does not appear to be coming back, and there is no acquisition going on (no lights flickering on the status unit of the Inovas), and we are not around, then press ctrl-alt-backspace (not ctrl-alt-delete!). You should get the Log In window. Do this only as a last resort, as it may corrupt the files of the previous user or harm the communication with the spectrometer's console.

#### **The printer won't print**

Go to Process→Plot and make sure that you have a printer selected where it says "Send this plot to:"

**Contact information for NMR facility staff and NMR TA's are posted at each instrument.**