

Vnmrj's common problems and solutions

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These notes have been collected for our users in the NMR Facility of the Department of Chemistry, The University of Michigan. Some, particularly those referring to our custom-made macros, may not apply to users of other facilities. Updated for Vnmrj 2.2C and 2.2D with Chempack 4.1.

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1. Problems during acquisition

- 1.1 The acquisition doesn't start when you give the `go` or `ga` command or when you click on the [Acquire] button.
- 1.2 The spectrometer doesn't obey commands or buttons like [Find Z0], [Gradient autoshim], [Acquire], etc.
- 1.3 The sample is not ejected with the [Eject] button or the `e` Command.
- 1.4 The message “*Setup complete*” does not appear after you type `su` or `setlock`.
- 1.5 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.
- 1.6 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.
- 1.7 The lock display is slower than normal (it refreshes the image less than two times per second).
- 1.8 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).

All the above problems, and most other problems related to acquisition are due to interrupted communications between the spectrometer's console and the host (Dell) computer. To solve any of these problems, 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 shims and lock parameters with `setlock` and re-shim after this.

If the above doesn't work, open up a terminal window (click the “Terminal” icon on the Linux Desktop or select it from the Linux task bar menu: Red Hat>System Tools>Terminal) and type “`su acqproc`” to kill the

communication with the console. When the command prompt returns, type “su acqproc” again to restart the communications. A new window with a message like “Acquisition Console at 500 MHz ready” might appear; close that window. Finally, go to Vnmrj and type “su” to reinitialize the console. You may need to reload shims and lock parameters with `setlock` and `re-shim` after these procedures.

On other occasions you may need to do the following. First, in a terminal window type “su acqproc”, then reset the console and finally type “su acqproc” again. To reset the console, if you are on an Inova spectrometer, open the left door of the console by pulling it from its upper edge and press the little red button shown in the figure below (left image); if you are in the 400MR instruments (*Gallium* and *Cobalt*) or the vnmrs instruments (Tellurium-500, Ytterbium-700), open the console's front door (the right door on the vnmrs) and push the reset button on the rightmost controller card as shown in the figure below (right image); if you are on the Mercury spectrometer, look in the back of the Mercury console and press the reset button (the location is similar to the one shown below, at left, for the Inovas).



If nothing works, leave us a message or send us an email.

1.9 After a power failure, the spectrometer is dead.

All spectrometers have a power breaker that trips after a power failure or a power surge. If recently there was such a failure, and the spectrometer seems to be completely dead (after you open the door there are no lights turned on or blinking on the console, and all the fans in the back of the console are off), press the reset button on the back of the console. The button is green and labeled PUSH-ON and is located in the lower part on the back of the console.

1.10 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`.

1.11 When I start an acquisition the lock is lost.

Go to the “Start, Standard” panel and verify that “Autolock, When:” is set to “Not Used”. And “Lock Autoshim” is set to “Never”.

1.12 Signal to noise on the 400MR spectrometers (*Gallium* and *Cobalt*) is very low (the spectrum shows no signals or is noisier than expected).

One major difference between the new 400MR spectrometers and our older Inovas and Mercury spectrometers is that the former are equipped with protune accessories that allow it to automatically tune the probe to the desired nucleus and solvent. In our older instruments the default probes do not need tuning (Inova 400, Mercury 300 are equipped with probes pre-tuned to ^1H , ^{19}F , ^{13}C and ^{31}P), or we do not allow users to tune the probe (Inova 500 is always tuned to ^1H and ^{13}C in CDCl_3). In the newer instruments, users are allowed to, and should always tune the probe to the nucleus of interest and solvent. This happens automatically when the acquisition is started by clicking on the green **[Acquire]** button; but if the acquisition was started with the `go` or `ga` commands, as was done traditionally on the older instruments, it won't happen. The problem with that is, for example, that if the previous user was doing ^{19}F and you want a proton spectrum, after you type "`go`" the spectrum will contain only noise because the probe will still be tuned to fluorine. Instead, you should click on the green **[Acquire]** button on the "Acquire" panel (or type `cpgo` on the command line). This should automatically tune the probe to the nucleus you have already selected before starting the acquisition. You can also explicitly request that the probe be tuned to a specific nucleus or sample. Go to the menu "Tools, Probe Tuning" and select "Tune probe". In the window that appears, select the appropriate nucleus under "Quick Tune" or enter a nucleus under "Advanced Tune" and click **[Tune to Criterion]**. You should now hear a screeching sound from the protune box and the cables to the probe should turn and stop after a few seconds. This is the normal behavior.

It is very useful to verify that the tuning process was successful, especially before starting a long acquisition! Click on the **[Probe]** button on the lower status bar of Vnmr's window; then in **[Tune Sweep]**. You will now see a new panel under the "Acquisition" tab. If you want to tune to H1 or F19 select channel 1 ("Tune RF channel") and enter the nucleus in "Center Frequency" (remember that Gallium can not be tuned to F19). If you want to tune to C13 or anything else, select channel 2 and enter the nucleus (letters first, e.g. C13 and not 13C). Now click on **[Start Probe Tune]**. You should see a graph with a deep "V" shape and a blue vertical line at the nucleus' frequency. When properly tuned, the blue line should be in the lowest 30% of the V well. When you are done looking at the graph, click on **[Stop Probe Tune]** and **[Quit]**.

Notice that some nuclei, particularly Boron-11 and Mercury-199 are difficult to tune to. These nuclei are outside Varian's official capabilities for the 400MR consoles. However, in practice, at least in our spectrometers, we can usually tune the probes to their frequencies. But the V shape in the tune sweep graph may be severely distorted and difficult to recognize.

1.13 Automatic probe tuning on the 400MR spectrometers (*Gallium* and *Cobalt*) doesn't work.

If, when tuning the probe or starting an acquisition you see any of the following:

- the cables of the protune box don't turn (no sound)
- the tuning process takes forever (the cables turn back and forth for more than 1 or 2 minutes)
- the tuning process aborts unexpectedly
- the "tune sweep display" shows that the probe is not tuned even after 1 or 2 attempts

then the protune unit may require recalibration. Unfortunately this happens from time to time, more frequently than desired. To recalibrate it type "`protune('calibrate')`" in Vnmrj's command line. In the window that opens, in the `Cmd:` field located at the middle left of the window type "`index all`" followed by the enter key. The status line on this window will change to "Running" and all the motors in the box will turn in sequence. Wait a couple of minutes until the status changes to "Ready" and now click on **[Quit]**. The protune unit is now calibrated and you can try tuning the probe again.

If you still have problems, please send us a message or leave a message on the desk. Notice that if you had problems with the protune unit, most likely all succeeding user will have the same problems.

1.14 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.

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

This is a common problem, especially in the Inova 400. 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`.

1.16 I have problems controlling the temperature

If the temperature controller doesn't appear to respond to commands, reset it by clicking on the **[Reset VT Controller]** button on the “Start, Spin/Temp” parameter panel. Set the desired temperature and type `su`. Check the styrofoam bucket setup and make sure everything is ok (there is enough liquid nitrogen for cooling, all the hoses are securely connected, etc). Sometimes, the rigid tube connecting the bucket to the probe pops out at the top of the bucket. If that is the case, gently reconnect the silicone “septum” to the mouth-shaped opening in the bucket's lid by pressing it to the opening. The black, rigid tube connecting the bucket to the probe is double walled glass with vacuum insulation and is very expensive. Be very careful not to exert any force on it or it may break.

Keep in mind that when you are controlling the temperature in one of `vnmrj`'s workspaces and switch to another to do a different experiment, the temperature can change when you start a new acquisition, that is because the temperature in a workspace is a parameter (`temp`) independent from other workspaces. To avoid this behavior, you can check “Control temperature from this panel only” in the “Start, Spin/Temp” parameter panel, or just set the temperature to the same value in all workspaces you are using for acquisition.

1.17 The message “VT airflow threshold exceeded” appears when an acquisition starts.

This happens from time to time on our 400MR systems (*Gallium* and *Cobalt*). Go to the “Start, Spin/Temp” panel and click on **[Reset Pneumatic Router]**; then check that “VT Air Flow” is set to 10 L/min.

1.18 On the Inova 500, my ^{13}C spectrum shows strange peaks around 200 ppm.

Unfortunately, there is a signal with a frequency very close to 125 MHz from an unknown external source that is picked up by the probe. This shows up as spurious signals in the $\text{C}13$ spectrum. Fortunately, they disappear as you collect more scans. Some days the signal is strong while others it is not noticeable.

1.19 The Sample doesn't spin.

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 or tissue wet with isopropanol. Clean also the lower rim, taking care not to use solvent on the black-painted band with the white dots. If this doesn't work, try a different tube.

The Inova 500 is especially sensitive to the spinner and may spin easily with one spinner but not with another. If you are using the Inova 500, make sure you are using the spinner labeled “500”. Occasionally the Mercury 300 does not spin the sample. It appears that although vnmrj turns the air flow on, the electronics fails to regulate the spinning. This happens only from time to time and we have been unable to pin point the cause. Please let us know when this happens again to continue with the diagnostics.

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.

1.20 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, remove the o-ring that holds the NMR tube in place using a fine spatula and wash it with isopropanol. Wash and clean the groove where the o-ring lies with the tip of a wet Kimwipe and the inside of the spinner with more isopropanol taking care not to spill solvent on its black-painted band or the white dots. 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.

1.21 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. Fortunately, after many inexperienced users did just that, we designed a tool to extract empty spinners from the magnets. Before using it, make sure the spinner was indeed dropped empty and there is not a sample tube or pieces of a broken tube in the magnet. If there is, do not continue and wait until the staff comes to retrieve the spinner or you might damage the probe. The tool is located in the NMR lab (room 3500) on a little rack on the wall behind the 400 “cobalt”. Carefully and slowly insert the pointy end in the magnet until you feel that the tip is touching the spinner, then press a little so the tip is inserted in the spinner's hole. Now slowly withdraw the tool and the attached spinner from the magnet. Put the tool back in the rack.

1.22 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.

1.23 I cannot find the lock signal or lock the sample.

1.24 The lock signal is unstable.

1.25 Shimming is difficult.

If you can't find the lock signal, make sure you are using a deuterated solvent. Yes, that sounds silly but... it has happened. Eject the sample and verify that you have enough solvent (at least 3 cm) and that the tube is properly positioned in the spinner. The solution column should be centered on the dotted box of the sample gauge, with equal amounts of solvent above and below the box. 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. To facilitate entering appropriate parameters, a macro called `setlock` was created. This macro sets the lock field, power, gain and phase appropriate for your solvent. Additionally, it reads the latest shims for the probe in use. The `solvent` parameter, selected from the “*Start, Standard*” panel, must have been previously set correctly for `setlock` to work properly. It may still be necessary to adjust slightly Z0 or to increase momentarily the gain or power to kick the lock in, but only minor adjustments should be needed for most samples.

If after loading the lock parameters the lock does not engage, display the “Start, Lock” or “Start, Shim” panel, notice the current lock power and increase it by 10 or 20 units. Turn the lock off and increase the lock gain to the maximum. Move Z0 one way or the other until you see the lock signal and keep moving it until you get to the correct Z0 (flat signal with no sinusoidal shape). Then turn the lock back on and decrease the lock power to its previous value. You can also simply press **[Find Z0]**; it works on all instruments except the Mercury 300 and usually works fast and reliably. Solvents with more than one peak in their spectrum like THF-d8 and toluene-d8 will not give you a flat lock signal and in consequence finding the correct Z0 will be more challenging and will require some experience. 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 lock signal is still difficult to find it may be because the shims are very far from optimum. Use gradient shimming to attempt to shim the sample so the lock signal can be seen more clearly. The sample does not have to be locked in order to do it, but Z0 should be correct for your solvent. Gradient shimming is available in all instruments except the Mercury 300. Click on **[Gradient shim]** or type `gradshim` and try to find the lock signal as described above again. You can even shim a non deuterated solvent with `gradshim('H1')`. In this case make sure the lock is off.

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.

1.26 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%.

1.27 All my peaks show a shoulder or tail on one side

This is probably due to poor shimming. If the shoulder starts near the the middle of the peak's height, it is probably due to Z2. If it is near the base, it is probably Z4. First shim Z1 and Z2 as usual, then optimize Z4, go back to Z1 and Z2 and re-optimize Z4. Repeat as necessary. Z3 may also need adjusting.

1.28 The baseline of my spectrum is distorted.

This may be due to improper phase corrections. Type “`rp=0 lp=0`” on vnmrj's command line and phase the spectrum again. If you still have this problem see below.

Pulse breakthrough sometimes happens, especially with some nuclei like ^{195}Pt or ^{19}F where large spectral widths and/or short acquisition times are used. One way to eliminate it is with backward linear prediction. Go to “*Process, More 1D*” and select “Linear Prediction” and the “Back” option. Click on the [Auto] button to calculate appropriate parameters and re-transform the spectrum. If the distortion is severe, you may need to increase the parameters. Use for example: “starting at=9, predicted pts=8, starting at=8”.

You can also use a baseline correction to fix the baseline. First, you must integrate the spectrum and cut the integral in pieces to define the regions where real peaks are located. Anything outside the integral regions will be flattened. Type `bc (5)` to perform the baseline correction.

Severe baseline distortion that may look like very “noisy” or weird looking spectra may be caused by digitizer overloading. Check the `gain`. If it is set to any value other than 'n', set it to 'n' and take a new spectrum. Otherwise, type `gain=gain-6` and take a new spectrum.

1.29 All my peaks show “waves” (sinusoidal distortions) on both sides.

The acquisition time 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.

2. Software problems

2.1 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)`, where `x` is the experiment number to unlock. If this fails, quit vnmrj, open a terminal window and type “`rm ~/vnmrsys/lock*`”.

2.2 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.

2.3 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.

2.4 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.

2.5 The computer is frozen.

2.6 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. Loading times are much shorter with versions 2.2C and above. 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.

2.7 A previous user left the computer locked with a screen saver

If it is a workstation, and no other free workstation is available and it appears that the previous user is not coming back then press ctrl-alt-backspace (not ctrl-alt-delete!).

If it is a spectrometer, and 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.

3. Plotting and processing problems

3.1 I cannot plot my spectra.

If you get the error message “*no plotter on system or plotter undefined*” type “`user_setup`” on the command line of `vnmrj` to set the default plotter and other common parameters. If you get no error messages, check the printer to make sure it has paper and it is online. Turn the printer off, wait 30 seconds and turn it back on. If everything is ok but you still can't get `vnmrj` to plot, try another workstation but please send us a message.

3.2 I didn't find what I was looking for in this document.

3.3 The solution to a problem is not clear.

3.4 I have a suggestion to make.

Please send me a message (`ealva_at_umich.edu`) describing the problem you had.