Troubleshooting Tips for VNMRj's Common Problems

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This document was prepared to help users understand common problems they may encounter when using the CIC-NMRs and what they can do to prevent these from occurring. However, only those that have been trained to reset the NMR consoles and the computers are authorized to do so. If you have not been trained please contact Paul Ralifo at <ralifo@bu.edu> when you encounter a problem that requires rebooting of the computers and the consoles.

Problems During Acquisition

- ***The acquisition doesn't start when you give the go or ga command.***
- ***The spectrometer doesn't obey commands or buttons like [Find Z0], [Gradient autoshim], [Acquire], etc in the m300 or m500.***
- ***The sample is not ejected with the [Eject] button or the e command on the m300 or m500.***
- ***The message "Setup complete" does not appear after you type su or setlock.***
- ***The message "Active" appears continuously on the bottom of the Vnmrj window instead of the normal "Idle" even when no acquisition or shimming is in progress.***
- ***The message "Inactive" appears on the bottom of the Vnmrj window on a yellow field instead of the normal green "Idle".***
- ***The message "Cannot set hardware during interactive acquisition" or "Cannot do {some command} when an acquisition is active or queued" appears on Vnmrj's info bar.***

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. Only persons listed in the NMR emergency contact list are allowed to reset the console. Please contact any of these individuals or email Paul Ralifo to fix this problem.

- ***The [Lock Scan] button is frozen. It remains depressed and doesn't show the lock signal on the m300.***

To unlock it, on vnmrj's command line type lock_scan.

- ***When I submit a sample I get an error, "Sytem Reported Failure" or something to that effect.***

This problem could be due to a number of things, including sample was not detected inside the magnet, etc. Email Paul Ralifo to report this.

- ***Automatic shimming on the v400 doesn't work.***
- ***Signal to noise on the spectrometers is very low (the spectrum is very noisy)***

This problem could be solved simply by checking the autotune button in the "Start>Standard" panel. Also you can reload the shim files by entering in the command line, *rts('last') su* followed by a carriage return.

- ***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 on m300.***

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=tpwr10 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 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**]** button in this parameter panel. If the temperature still doesn't go down, reset the communication with the console with *su acqproc*.

- ***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. Please let me 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 are done with spinning turned off.

- ***I broke a sample***

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, bring the spinner to me so I can clean the o-ring and the spinner. If the sample broke inside the magnet or some of the solution or glass pieces went into the magnet, notify me immediately. Failure to do so may result in costly repairs and termination of your user privileges.

- ***I dropped an empty spinner in the magnet and now it does not eject it.***
- ***I dropped a sample tube without spinner in the magnet***

Notify Paul Ralifo immediately. Place a “Do not use...” message describing the problem next to the computer.

- ***I cannot find the lock signal or lock the sample.***
- ***The lock signal is unstable.***
- ***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. 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. 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 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. If none of the above works, then new gradient shims needs to be retouched and in that case, please email me.

- ***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% if you are using the m300. If you are using the v300 or the v400 please enter in the command line, *rts('last') su* followed by a carriage return to reload the shim files. Also send me an email to let me know so I can retouch the shims.

- ***All my peaks show a shoulder or tail on one side or my spectrum has broad peaks and poor resolution***

This is probably due to poor shimming. Reload the shims and contact me to touch up on the shims again.

- ***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.
You can fix this by using 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 by typing in the command line *gain?* followed by a carriage return. If it is set to any value other than 'n', set it to 'n' (enter in command line *gain='n'*) and take a new spectrum. Otherwise, type *gain=6* in the command line and take a new spectrum. If none of these work, take your sample back to the lab and dilute your sample.

- ***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.

- ***loc=50 change command gives an error 'seq:file cannot be found'***

This error appears because the software is looking for a sequence called file which doesn't exist. Load a previously run spectrum and then reneter *loc=50 change* in the command line again. Make sure that there is a sample tube in spinner 50.