

# **DCIF FAQ: FREQUENTLY ASKED QUESTIONS**

## **VARIAN**

Last Update (2/21/2005)

### **How do I get help?**

**Please review the material here and in the training guide.**

**For night and weekend support to fix instrument acquisition problems email: [DCIFhelp@mit.edu](mailto:DCIFhelp@mit.edu)**

### **In an Emergency DIAL 100 for Campus Police**

### **Major Problems**

When appropriate, contact facilities or/and campus police and ask that they contact the DCIF staff at home.

### **Locking and Shimming**

**Q:** Trouble Locking.

**A:**

- 1) Make sure the sample is properly centered in the magnet.
- 2) Reduce the lock power/ increase lock gain.
- 3) Review lock saturation causes and cures
- 4) Adjust the lock phase.
- 5) Reload the bestshim file.
- 6) Do you have deuterated solvent?
- 7) Where is the Z0 usually for this solvent?

**Q:** The sample is properly locked, but when I activate the shim window the lock level drops dramatically. Why is this happening?

**A:** The lock phase is probably not adjusted properly. Re-open the lock window and (with lock turned on) adjust the lock phase to maximize the lock level.

**Q:** My peaks aren't symmetric. They all have a long decaying tail, a hump on the side, or are all split into doublets. What's wrong?

**A:** Start by re-loading the bestshim file. If the peaks have long decaying tails the problem is most likely an even order shim (Z2, Z4, etc.). Humps and splittings are most often resolved with the odd ordered shims (Z1, Z3, etc.). Remember, proper sample height and a quality NMR tubes are also imperative to sharp symmetric peaks. Concentration also matters. Keep shimming.

## Spinning and Ejecting

Q: My sample won't spin, what's the problem?

A: These are the most likely suspects:

- 1) What type of tube are you using? Many spinning problems are the result of cheap NMR tubes. We recommend a Wilmad 528 or better.
- 2) Check the base of the spinner. If the base is dirty, clean the surface with a Kimwipe and try it again.
- 3) Are you drying your tube in oven? If so, the tubes must be placed on a flat surface. Leaning in a beaker and excess heat can lead to distortion and spinning problems. See our website for proper tube cleaning procedures.
- 4) See next answer also.

Q: I can't eject. Why?

- A:
- 1) Is the eject gas pressure correct (about 70 psi—see individual gauge mark)?
  - 2) Sometimes, J-Young tubes, e.g., are too heavy. Covering the capacitor stick hole at the bottom of the probe with a finger sometimes increases the pressure enough to eject a heavy tube (this takes two people). Increasing the eject gas pressure temporarily may be used with great caution (tube sometimes EJECTS into the air and then breaks back into the probe). Be sure to return the pressure back to the normal set point.

## Broken Tube

Q: What do I do if a tube is broken?

A1: If the tube is broken outside the magnet, clean up the chemical spill. There is a hood, a sink and dust pan under the sink. Place all the broken glass in the sharps container near the hood. Be sure the broken glass is removed from the spinner. Check the o-ring carefully.

A2: If the tube is broken inside the magnet, the probe is likely contaminated. Amazingly, the probe, itself, often survives broken tubes if we can clean them out before the next user spins broken glass and shreds the probe's innards. No one should use the instrument until a DCIF staff member can look at the probe/spectrometer. It is not worth risking damage if we can remove the broken glass and clean the contaminated probe (free). If we have to send the probe back to the manufacturer, it costs a lot. Probe repairs in the past few years range from almost \$2,000 to well over \$10,000, with the majority being in the \$4000-\$5000 range. It's simply not worth risking if you think there may be broken glass in the probe.

- 1) Turn off the spin to avoid further damage.
- 2) Put a sign on the spectrometer saying: **BROKEN TUBE, DO NOT USE.**
- 3) Put the spinner with a note in the hood (we need to look at the o-ring).
- 4) We prefer that you lock the screen so others can't log in (terminal window, xlock) (You will be credited for billing—be sure to email us to remind us to remove the billing charges).
- 5) Email the [DCIF@mit.edu](mailto:DCIF@mit.edu) to let everyone know what happened (include all details about the sample—what it is or might be, whether it is poisonous—double-glove-poisonous, what dissolves it, what the solvent was, etc. If the sample is precious, let us know, we'll try to save it if we can.)

## Spectrometer/Software Problems:

- Q: Locked experiment or no experiment number at top of VNMR window.
- A: This is usually caused by someone in your group logging out without first exiting VNMR. Within VNMR, you can type the macro, **restart** which is the same as typing: unlock(1) jexp1 exit
- A: Detailed) There are other macros in the exit routine that fix other minor problems. If that doesn't fix the problem, open a new terminal window, type "cd vnmrsys", check if there is any "lock\_x.primary" (where x is the experiment numbers) files, and delete them. If there are no such files, then exit the VNMR and restart it. Sometimes, experiments become corrupt. You can delete and recreate experiments in VNMR: e.g., delexp(2) and cexp(2). You can't delete exp 1.
- Q: The [acqi] button does not respond or is missing.
- A: In VNMR, type the macro, **reset**
- A: Open a terminal window by right clicking on the desktop and selecting terminal in the pull down menu.
- 1) At the Unix prompt type su acqproc then a <return>. You will see the message "Killing Expproc"
  - 2) Again type su acqproc at the Unix prompt followed by a return. You will see the message "Starting Expproc"
  - 3) Type acqi in the VNMR command window (if the [acqi] button is not present.
- A: Detailed) su acqproc breaks/makes (toggles) the communication between the computer that you see on the table and the computer inside the NMR console. Usually breaking and remaking the communication will fix minor problems. Like a reboot fixes PCs. Rebooting the unix computer must be done by a superuser however. Hard drives crash when power cycling is done without properly unmounting the devices.
- Q: There are queued experiments listed in the AcqStat Window, which I did not queue. What should I do?
- A: See the previous answer.
- Q: The acquisition status says 'Inactive'. How do I make the status active?
- A: See the previous answer.
- Q: The spectrum doesn't automatically appear after the acquisition completes. Where did it go?
- A: 1) Type **wft** (for a quick-fix) or 2) See the previous answer.

- Q: The instrument won't acquire and I get the following error message: "Autogain failure, gain driven to zero, reduce pulsewidth."
- A: This typically happens when the sample is very concentrated. Type **pw=pw/2 ga** <return>. If the message reappears, repeat as necessary until the error message no longer appears.
- Q: The autogain routine takes too long. I think it may have crashed.
- A: Type **sa** or **aa** if **sa** doesn't work. The autogain occasionally gets itself in an endless loop (this most often happens with F-19 on the Mercury). If this happens, type "**gain?**" or "**gain=xx**", here xx is number from 0 to 60 and run the experiment with nt=1, type "df" to see FID, if the FID is too big (clipped) or too small, type in another number, repeat this until an appropriate gain is found, then set nt and type "ga"
- Q: Someone has locked the screen and they're eating into my reservation time.
- A: (First, double-check cruella.) (If a staff member is present, you may ask them to help—our users have been known to throw hissy fits with one another—all MIT staff members are adept at dealing with persons exhibiting this type of behavior.) This requires the user to type a password. You must finger the user on cruella and call the person's lab. Feel free to bother them at home.
- Q: I'm having trouble tuning.
- A: If you are uncomfortable tuning, DON'T TUNE! More money is spent on damage from tuning probes than any other problem. Get help from someone with more experience (preferably a staff member).
- A: Be sure you have:
- 1) loaded parameters
  - 2) typed su
  - 3) closed the acqi window
  - 4) INOVA: checked the connections (probe to probe, output to output)
  - 5) INOVA: tn on channel 1. dn on channel 2.
  - 6) Correct ¼ wave cable?
  - 7) Capacitor stick required for this nucleus?
  - 8) Proper Bandpass filter?
- A: If all that is fine but the tuning just won't minimize, be sure you know how to play the tune off the match to find a global minimum reflected power. Ask for a staff member to help you learn how to do this.

## Integration

Q: I messed up while integrating my spectrum. How do I start over?

A: Type **cz** (clear zeros) to clear all of the integral resets and then start over.

Q: My integration values are all too small (or zero). How do I fix this?

A: Type **ins=100**

Q: Can I trust my integration values?

A: No, but steps may be taken to minimize the experimental uncertainty. For quantitative measurements, it is critical that the nuclei have sufficient time to relax following each pulse. This time is 5 times the T1 of the nuclei of interest. A T1 experiment can be performed to determine the proper relaxation time. If you decide not to determine the proper T1 time, try setting **d1=30** and then reacquire your spectrum. Note that this will increase your acquisition time by about 15 times. Other factors to consider:

- 1) There should be no peaks within ~10% of the base wings. Regions of spectral congestion increase experimental uncertainty.
- 2) The S/N should be at least 250:1 for the smallest peak to be integrated.
- 3) The baseline should be flat. (See next question)

## Displaying

Q: My baseline isn't flat. How do I fix it?

A: Integrate all of the peaks (this includes solvent peaks) and then type **bc** for baseline correction.

Q: I can't see the entire graphics window because the parameter window is in the way. How do I get the window to the front?

A: Click on the [**Flip**] menu button. You may have to click this button twice.

## Plotting

Q: I cannot plot my spectrum. What's the problem?

A: Is the proper plotter selected? Type **plotter?** to view which plotter is currently selected. The plotter names are clearly marked on the front of the plotter. To select a specific plotter, type **plotter='XXX'** where XXX is the name of the plotter from which you wish to print. If you type **setlj**, this will select the default plotter.

Q: The spectrum is not plotting in landscape mode.

A: **plotter='ps\_landscape' printer='ps\_landscape'**

Q: The printer is out of paper. Where is the paper?

A: The paper and other lab supplies (such as Kimwipes) are stored in the cabinet next to the INOVA 501.

Q: How do I print an inset or expanded region on the same page as a full spectrum?

A: Process your spectrum just as you would like to be in the figure.

1) When you are satisfied type **pl pscale pirn** (or whatever you want plotted), but DO NOT type page.

2) Place the cursors around the region you want printed as an inset.

3) Type **inset**. The selected region will now appear on the screen.

4) Use the mouse buttons to move the spectrum to the region where you would like it printed.

a) The left mouse button will move the inset back and forth.

b) The middle mouse button adjusts the inset intensity.

c) The right mouse button adjusts the width of the inset region.

5) Plot the inset: **pl pscale pirn** (or whatever you want plotted), then type page. 6) To get back to the original spectrum, type **vp=12 f full**.

Q: I need to make a postscript or pdf or other odd plot. How do I do this?

A: Easy. Log onto zippy and type **man('mkfig')** in the VNMR window. Follow the directions.

Q: I need to make an ASCII text file because I would like to import my spectrum into a spreadsheet such as SigmaPlot or Excel. How do I do this?

A: Follow these steps: 1) Load the FID you wish to convert into an ASCII file. 2) Query the Fourier number by typing **fn?** If the Fourier number is large (>16,000) type **fn=16k**. 3) Reprocess the spectra (**wft**) and verify that you have not lost too much resolution. If the spectrum looks digitized increase **fn** until you are satisfied. 4) Type **makeascii('your\_file.txt')**, where **your\_file.txt** is the name you choose for the text file. 5) Note: If you expand a portion of the spectrum in the graphics window, only that region will be written to the text file!

Q: I've made a postscript/ text file on zippy. How do I get the files to my Mac/ pc in my lab?

A: From a networked computer in your lab use the ftp program of your choice (Mac: Fetch, PC: SecureFX) and ftp to [zippy.lms.mit.edu](ftp://zippy.lms.mit.edu).

Q: I'm using the X-terminal software in my lab to work up my data on zippy. Why can't I print my spectrum to my own printer?

A: You need a network printer. If you have a network printer in your lab let us know and we can add your printer to the devicenames on zippy.

## Saving and retrieving data

Q: What's the difference between **svf** and **svfz**?

A: **svfz** is an amazing MIT macro which saves the data on the spectrometer computer and also saves a copy of the data files on zippy. **svf** is (now) an unadulterated Varian macro that saves data only on the spectrometer. When zippy is down, **svfz** takes A VERY LONG TIME and often users become crazy enough to cause other problems. So normally use **svfz**, but if zippy should happen to crash, use **svf** instead until zippy is restored. Of course the data files will have to be remotely copied by hand (see note in the data processing room).

Q: Am I limited as to the amount of disk space I can use to save my spectra?

A: No. Use as much disk space as you need.

Q: Do I need to back up my data?

A: YES you need to back up your data. The DCIF is not responsible for lost data. From a networked computer in your lab use the ftp program of your choice (Mac: Fetch, PC: SecureFX) and ftp to zippy.lms.mit.edu.

Q: How long does my data remain on the spectrometer?

A: Data on the spectrometers is backed up weekly and burned to a DVD. Data that has not been accessed for more than 365 days is removed from the spectrometers but is still available from CD. If you cannot find a data set, please ask we will have it on CD.

Q: How do I get my files from the spectrometers over to the workstation zippy?

A: Log onto zippy as usual and open a terminal window. To open a terminal window right click the mouse on the desktop and select terminal under the programs option.  
1) Create or change to a subdirectory where you would like your files to be stored.

Example: `cd directory_name` (changing to an existing directory)

Example: `mkdir new_directory_name` (making a new directory)

2) Remote log into rocky (or one of the other spectrometers) using the following command line: `ssh UID@rocky.lms.mit.edu`

3) Locate the files you wish to transfer (use `ls` to list files and `cd` to change directories). The files can be identified by the `.fid` extension.

Example: `your_file.fid`

4) Copy the desired files to zippy into your newly created subdirectory using the remote copy command.

Example: `scp -r your_file.fid zippy:~/new_directory_name/`.

Q: Do you have any free software?

A: No. but MIT does. X-Win32, SecureCRT and SecureFX are great!  
[web.mit.edu/software](http://web.mit.edu/software)