

DCIF FAQ: FREQUENTLY ASKED QUESTIONS

BRUKER

Last edit 20061210

How do I get help?

Please review the material here and in the training guide.

Open NMR GUIDE in TOPSPIN: [HELP] > [NMR GUIDE]

For night and weekend support to fix instrument acquisition problems email: DCIFhelp@mit.edu

In an Emergency DIAL 100 for Campus Police

Major Problems: Fire, Smoke, Flood, Water leaks

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

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. Spare spinners are available near the shop door.

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 to let the DCIF staff 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.)

Locking and Shimming

Q: Trouble Locking.

A: 1) Type **lock off**, and relock **lock**

- 1) Make sure the sample is properly centered in the magnet.
- 2) Reload the bestshim file.
- 3) Lock on the solvent
- 4) Maximize the lock signal by adjusting the lock phase.

- Do you have deuterated 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, **rsh best** 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. 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.

BSMS will not respond

Q: The BSMS will not execute commands. how do I reset the BSMS?

A: 1) Are there error messages in the terminal window? Yes, go to 2
2) Close the BSMS panel and reopen the panel.

3) If #2 does not work, close Topspin and reopen the program.

Q: The Spin button on the BSMS will not respond, is there a fix?

A: 1) type **ro off** in the command line with the BSMS panel open.

USB ICON does not respond

Q: How do I use a Memory Stick (USB drive) on the Linux OS

A: 1) Insert your Memory Stick
2) Right-Click the Memstick icon and choose **mount**
3) Drag your files to the icon
4) Right-Click the Memstick icon and choose **unmount**

Reprint from Bruker's TOPSPIN Installation Manual for Linux:

©Bruker 2005

12.2 TopSpin troubleshooting

TOPSPIN should not get into any trouble in general! Nevertheless, if your current **TOPSPIN** session has a problem, you can find in this chapter some instructions how to get rid of this situation.

In order to stop TopSpin in case of any problems, the first thing to know is, that TopSpin is working with a client / server structure. The server is the program itself and the client is the graphical interface. It is possible to exit each of them separately. It is also possible to start more than one client (graphical interface). This feature is used for the remote control but can also be very useful for troubleshooting (see below)!

- The client (=graphical user interface of **TOPSPIN**) can be stopped without touching a running acquisition.
- Stopping the program itself will also stop a running acquisition.

12.2.1 Troubleshooting while no acquisition is running

The most convenient way to get rid of a problem inside TOPSPIN is, to stop the complete program. For this you can do the following steps. These steps are increasingly drastic.

1. Enter **exit** in the TOPSPIN command line or press the X in the upper right corner of the program window.

Is the TOPSPIN window closed?

- Yes? Start TOPSPIN once again, in case there will be still some problems go ahead with step 2
- No? go ahead with step 2

2. Stop the current TOPSPIN window and start a new one

a) Enter in the TOPSPIN command line:

- **logoff**

b) Open another Linux shell and start a second TOPSPIN:

- **topspin -client**

Has the first TOPSPIN window been closed and does the second one work fine?

- Yes? The problem is solved you can go ahead with TOPSPIN
- No? go ahead with step 3

3. Stop all TOPSPIN windows and end the program itself

a) Try to stop all TOPSPIN windows by enter the command:

- **logoff**

in the TOPSPIN command line

b) To close all the TOPSPIN windows that could not be closed in step a), open a Linux shell and enter

- **ps -ef | grep <TOPSPIN_HOME>/jre/bin/java**

You have to replace the <TOPSPIN_HOME> with the path of your **TOPSPIN** installation. In a default installation this is /opt/topspin, in this case the command would be:

- **ps -ef | grep /opt/topspin/jre/bin/java**

The answer is something like this:

```
<USER> <xxx> <yyy> 0 <date/time> pts/<z> 00:00:00
/opt/topspin/jre/bin/java ...
```

c) Kill all those sessions by entering the command in a Linux shell:

- **kill <xxx>**

d) In case the ,normal‘ kill does not work use the kill -9 command:

- **kill -9 <xxx>**

e) Enter the command in a Linux shell:

- **shrm**

12.2.2 Troubleshooting while an acquisition is running

The difference to the situation that no acquisition is running is, that you can try to stop only the graphical user interface without stopping the acquisition. Steps 1-3 do not stop the acquisition, step 3-5 will stop also the acquisition.

1. Stop the current TOPSPIN window and start a new one

a) Enter in the TOPSPIN command line:

- **logoff**

b) Open another Linux shell and start a second TOPSPIN:

- **topspin -client**

Has the first TOPSPIN window been closed and does the second one work fine?

- Yes? The problem is solved you can go ahead with TOPSPIN
- No? go ahead with step 2

2. Stop all TOPSPIN windows and end the program itself

a) Try to stop all TOPSPIN windows by enter the command:

- **logoff**

in the command line of the TOPSPIN windows

b) Open a Linux shell and enter

- **ps -ef | grep <TOPSPIN_HOME>/jre/bin/java**

You have to replace the <TOPSPIN_HOME> with the path of your **TOPSPIN** installation. In a default installation this is /opt/topspin, in this case the command would be:

- **ps -ef | grep /opt/topspin/jre/bin/java**

The answer is something like this:

```
<USER> <xxx> <yyy> 0 <date/time> pts/<z> 00:00:00
/opt/topspin/jre/bin/java .....
```

c) Kill all those sessions by entering the command in a Linux shell:

- **kill <xxx>**

d) In case the ,normal‘ kill does not work use the kill -9 command:

- **kill -9 <xxx>**

Has the first TOPSPIN window been closed and does the second one work fine?

- Yes? The problem is solved you can go ahead with TOPSPIN
- No? go ahead with step 4

3. Enter the command in a Linux shell:

- **shrm**

4. To get a list of all TOPSPIN processes open a Unix shell and enter

- **uxproc**

5. Kill any hanging **TOPSPIN** processes with the command:

- **kill -9 <xxx>**

and the respective ID of the process you got in step 4.