

## DCIF Lab Policies

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Incoming graduate students, undergraduates, and all first time users should visit the DCIF website: [web.mit.edu/speclab/www/newuser.htm](http://web.mit.edu/speclab/www/newuser.htm) for information on the facility and training.

### Training & Instrument Access

Instruments, with the exception of the Bruker FTMS, are accessible to anyone who has been trained by the DCIF staff and has a current account. No exceptions. If you wish to use a particular instrument, please visit the DCIF Instrument Training webpage and submit the training request web-form.

NMR trainings progress by field strength, i.e., for Varian training you must start with the 300 MHz instrument and for Bruker training your must start with 400 MHz instruments. New NMR users must take a quiz and pass it before NMR training.

If you are planning nonstandard experiments (VT work, exotic nuclei, etc.) make an appointment with the DCIF staff at least two business days in advance.

Instrument usage is tracked for billing purposes. Each authorized user is given a 6-character User ID (UID) for reserving time and for using the instruments. Please use a secure password and DO NOT let anyone else use your account. Violators may lose access privileges.

If you have been trained but have not used the instrumentation in over six months your account(s) may have been deactivated. If so, please see a staff member to have your account(s) reactivated.

### Reservations

Reservations should be made for all instrument time except for "walk on" time periods. See the reservations webpage for instructions: [web.mit.edu/speclab/www/reservations.html](http://web.mit.edu/speclab/www/reservations.html)

If you are 5 minutes late for your reservation, you automatically lose that reservation; remaining time is then treated as "walk on" with first come first serve. Some special cases can be treated individually by DCIF staff based on user's request.

A 'walk on' policy has been implemented for both the Varian Mercury-300 and Bruker Avance 401 systems between the hours of 1 – 6 pm Monday through Saturday. During these periods, please be considerate of your fellow researchers and limit yourself to **10 minutes** if others are waiting. Additionally, the Varian Inova 500 has 'walk on' time between the hours of 1:00pm and 3:00pm Monday through Friday. Users are encouraged to process data offline, if others are waiting to acquire data.

## **Safety**

In an **Emergency, DIAL 100** and contact MIT Campus Police.

Secondary containers **labeled with name and research group** are required for all NMR tubes and other chemicals.

### **Measurement of radioactive samples requires staff approval.**

Users are responsible chemical spills. If you break a sample tube, do not leave without first notifying a DCIF staff member. If it happens after hours, email to [DCIF@mit.edu](mailto:DCIF@mit.edu) with detailed information such as the name of chemicals, toxicity, solvents, suggested solvents for clean up. Put a note on the monitor/key board "Broken sample tube in the magnet, do not use this instrument until further notice", record the incident on the log sheet and lock the computer screen.

No food or beverages are allowed in the DCIF. This is not our rule, but rather this comes from OSHA and MIT EHS. If you have a problem with this rule please talk to Jim Doughty, EHS Coordinator for the Chemistry Department.

## **MS**

FTMS is performed by Li Li. The policy is first come first serve during the operating period of each ion source (EI/CI, ESI or MALDI). Special requests will be considered.

GC/MS and MALDI-TOF instrument time must be reserved on Cruella. Offline data processing currently is not available.

Do not inject sample with appropriate NMR concentrations into the GC/MS. The sample must be diluted down to 10 -1000 ppm (w/v).

Do not walk away with the camera and light on when you are finished using MALDI-TOF mass spectrometer.

## **Getting Help**

- Review the FAQ sheets next to the spectrometer computer.
- Review the training guide and 'tips and tricks' page located on the DCIF website.
- Review the Varian and Bruker manuals in the computer room. (do not remove manuals from 18-0090)

**NIGHT & WEEKENDS:** [DCIFhelp@mit.edu](mailto:DCIFhelp@mit.edu)

A group of graduate students and postdocs have volunteered to troubleshoot NMR hardware and acquisition issues. The volunteers have been trained to fix minor problems with stuck samples, tuning, locking, shimming and data acquisition. The goal is to prevent minor problems from keeping an instrument down for an extended period of time.

The volunteers are not responsible for helping individuals setup an experiment or correct problems immediately. Software and processing problems are also not covered. If there is an NMR hardware or acquisition problem, note the issue in the instrument log and email: [DCIFhelp@mit.edu](mailto:DCIFhelp@mit.edu) a detailed description. The email will be forwarded to the DCIF staff and volunteers. Volunteers will note if an instrument has been fixed in the instrument log.

## **General Policies**

The DCIF is closed every Thursday morning from 8:00 am until 10:00 am for routine instrument maintenance, please plan accordingly.

At all times a DCIF staff may need to access an instrument immediately due to special situations, in which your experiment may be interrupted. Please be cooperative.

All users must sign the instrument log books. Please do not be vague in your description of the problem. A detailed description of the problem will help us better resolve the problem. If you encounter a problem between 8am and 5pm (Monday- Friday) please notify a staff member directly.

Please try to leave the facility neat. Over 400 users within the MIT community use the DCIF, so please clean up after yourself.

Be vigilant. Report suspicious individuals or activities to a staff member.

## **Data Processing and Archiving**

- The DCIF only supports Bruker and Varian software located within the DCIF.
- **DCIF researchers are responsible for archiving their own data.**
- The DCIF is not responsible for data loss in any circumstances.

Researchers are encouraged to download fid files from the spectrometers on a weekly basis, critical data as soon as possible. The MIT software distribution site (<http://web.mit.edu/software/>) provides the following icon based, secure file transfer programs: SecureFX (PC) Fetch (MAC). Each instrument's IP address and data file path is posted in the DCIF Computer Room.

The DCIF will continue to backup new data daily and archive data on a weekly basis. This will allow a researcher to recover a few corrupt or missing files with the filename, date and spectrometer name. Bulk recovery of data is not available.

NMR Data will remain on the spectrometer computer for 1 year and will be available on the off-line processing computer for two years.

All data collected at Varian NMR instruments should be saved using the svfz command to simultaneously copy the data to the spectrometer hard drive and the off-line processing computer zippy.

Users can perform Varian off-line data processing using PCs in DCIF computer room using XWin32. XWin32 is a desktop simulation program (available from [web.mit.edu/software/](http://web.mit.edu/software/)) that connects to Zippy. If research groups have a local printer connected to the internet, ask a staff member to configure zippy to print locally.

Users can perform Bruker off-line data processing using PCs in DCIF computer room. Due to Bruker's license structure, offline processing is not available on computers outside of the DCIF.

Data collected at Bruker NMR instruments can be transferred to PCs within the DCIF computer room for off line data processing using SecureFX (available from [web.mit.edu/software/](http://web.mit.edu/software/)). Users are responsible for data on the offline processing computers.

## **NMR Sample Preparation**

Do not use chipped or cracked NMR tubes.

Wipe off your tube prior to inserting your sample into the spectrometer.

Warped and/or dirty tubes often do not spin well. NMR tube testers can be used to evaluate empty NMR tubes for their 'spinability'. Note that tubes should not be left in a drying oven for more than 20 minutes, and do not dry them at an angle. This will lead to an elliptical cross-section and result in spinning problems.

We recommend the use of Wilmad 528-PP (or better) NMR tubes with the Varian instruments to prevent damage to the probes.

J. Young tubes are not to be used in the Bruker 400.

The NMR lab does not provide solvents for your use.

The standard shims (or 'bestshim' file) are determined using a 5mm tube containing 700ul (~55mm in height) of deuteriochloroform. Shorter samples will require additional time to shim. Longer samples waste solvent, dilute the sample, also require additional time to shim, and occasionally spin poorly.

## **Variable Temperature Work**

No variable temperature (VT) experiments are to be performed without prior training by a DCIF staff member.

Do not do VT experiment at near and above boiling point and near or below freezing point of the solvents.

Be aware of the temperature limitations on the probe and the carrier gas preconditioning unit (if present). The temperature limitations for each of the available probes are posted on the respective magnet leg. If you are unsure, please ask a staff member!!

Verify you are using the correct carrier gas for the temperature range in which you wish to work. You must use nitrogen for temperatures outside the range of 10 to 40 °C. Again, if you are unsure, please ask a staff member!!

Always use new Wilmad 528-pp NMR tubes for VT work. Old tubes may be scratched or otherwise flawed and as a result are more likely to break.

For variable temperature work, you must schedule enough instrument time to bring the probe back to room temperature when you are finished. This means between 20-25 °C.

If you have difficulty ejecting a sample, you may turn up the eject air pressure at the control regulator on the wall. When you are finished, please turn the air pressure back down.

Users must supply their own liquid nitrogen for low temperature experiments.

# **!\$! THINGS THAT CAUSE MAJOR DAMAGE !\$!**

**DON'T GUESS, ALWAYS ASK!!!!**

If you don't know how, ask a staff member.

## **NEVER POWER OFF : SUN, O2 OR LINUX COMPUTERS**

### DANGERS DURING SAMPLE INSERTION

- Never place a sample & spinner in the magnet bore without the eject air turned on.
- Never put a spinner in the magnet bore without a sample tube.
- Never position the sample tube below the probe bottom (always use depth gauge).
- Never take a spinner from another magnet.

### LOCKING

- Setting the lock power too high causes probe damage and lock saturation.
- Learn how to check for lock saturation.

### TUNING THE PROBE

- Never tune the Varian 300 or Bruker 401 probe.
- Varian 500/501: Never move cables when the tuning screen is illuminated.
- Never apply excessive force to the tuning rods.

### BROKEN TUBE

If you break a sample tube, do not leave without first notifying a DCIF staff member. If it happens after hours, email to [DCIF@mit.edu](mailto:DCIF@mit.edu) with detailed information such as the name of chemicals, toxicity, solvents, suggested solvents for clean up. Put a note on the monitor/key board "Broken sample tube in the magnet, do not use this instrument until further notice", record the incident on the log sheet and lock the computer screen.

### VT WORK

- Never attempt VT work without first being trained by a DCIF staff member.
- Reserve enough time to complete your work: experiment time plus at least one minute per degree C (0-100 °C range requires experiment time plus 100 minutes).
- Never change the temperature (up or down) by more than 10° / 5 minutes
- Use nitrogen carrier gas for temperatures lower than 10 °C or higher than 40 °C.