UVM Department of Chemistry NMR Facility Policies

Incoming graduate students, undergraduates and all first-time users should visit the Department of Chemistry Instrumentation Facility website: http://www.uvm.edu/~chem/?Page=instruments.html for information on the facility and training.

General policies

All users must reserve slots online and sign the instrument log book each time.

If there is a problem with the NMR instrumentation or software, please give a detailed description of the problem to help us resolve the problem in an efficient manner. If you encounter a problem during business hours, please report it to the NMR manager right away, in person or by email or phone. In case of a hardware or software issue, immediately put a note on the NMR instrument to prevent others from potentially worsening the situation until the problem is resolved.

Remember: Prompt reporting of problems may help prevent a minor issue from becoming a major (and very expensive!) one. Don’t wait for the problem to magically “disappear”: during nights and weekends, call the manager’s cell phone immediately for assistance.

Failure to report problems may result in suspension of your user privileges.

To minimize the possibility of tube breakage in the NMR and to ensure good spectra, only high-quality tubes rated for a 500 MHz instrument are permitted.

The department highly recommends quality Wilmad 528-PP-7 tubes for use on both NMR instruments. These tubes are also available for purchase individually from the chemistry stockroom. While other high-quality NMR tubes rated at 500+ MHz may also be acceptable for use, they may not produce the same results as Wilmad tubes. Please contact the NMR manager beforehand if you wish to use other and/or specialty tubes.

NMR tubes unacceptable for use in the department NMR facilities include: “disposable” tubes; NMR tubes rated below 500 MHz; broken, chipped or cracked tubes; or tubes that have previously been repaired. Researchers will be held responsible for any damages caused by the use of substandard tubes.

Note: Do not spin top-heavy screw-capped or J. Young tubes.

Training and instrument access

NMR instruments are accessible to fully-trained individuals with a current user account. All training must be arranged through the facility manager. Upon successful completion of the training on a specific instrument, the facility manager will create an instrument account, which will also be tied to account billing. Separate training must be completed and a user account created for each of the NMR instruments.

Use of the instruments without manager training is not permitted. Never allow another user to “piggyback” on your online scheduling or instrument accounts.

If you feel at all uncertain about proper operation of the instruments, schedule some refresher training with the manager, or ensure that the manager or an experienced user will be available to help you.
The NMR instruments will be unavailable for use during and immediately after liquid nitrogen and liquid helium fills. While liquid helium fills happen infrequently (every few months), liquid nitrogen fills are done weekly. LN2 fills are typically done on Wednesday afternoons, and will be blocked off on the online reservation system one week in advance (typically 2 pm for the Varian and 4 pm for the Bruker), and require 1 – 2 h for filling and equilibration of the magnetic field.

While rare, the instruments are subject to interruption in access at any time due to facility or instrument issues. Unfortunately, staff may need immediate access to an instrument due to unforeseen circumstances which may impact your experiment or reservation times. Please be understanding of these situations, and the manager will keep you informed of developments in a timely manner.

Reserving time on the instruments

All reservations for time on either instrument should be made in advance using the Faces scheduling system at faces.cccrc.uga.edu. See our instrumentation website for instructions on creating an account and for the link to log into Faces: http://www.uvm.edu/~chem/?Page=instruments.html

1) Signup rules for the Bruker:
   a. “Daytime” reservation blocks are available in 10 min increments between 8:00 am and 5:00 pm on weekdays, up to 30 min maximum consecutive time.
   b. Reservations may only be made the day of intended usage.
   c. Between 5 pm and 8 am, longer “overnight” blocks may be reserved.

2) Signup rules for the Varian:
   a. “Daytime” reservation blocks are available in 15 min increments between 8:00 am and 5:00 pm on weekdays, for a maximum of 4 hours.
   b. Reservations may be made up to 2 days in advance without manager approval.
   c. Between 5 pm and 8 am, longer “overnight” blocks may be reserved.

3) Spectroscopist-assisted experiments (e.g. specialty experiments, such as variable-temperature NMR), new user training, and cryogen fills may be scheduled as needed with the NMR manager at least 24 h ahead of time on either instrument.

4) Booking the “full overnight” slot for either instrument from 5pm – 8am requires prior NMR manager approval, and should be scheduled at least 24-48 h in advance.
   a. To have at least one instrument available at any time, both instruments cannot be booked for “full overnight” reservations concurrently.

5) All users are accountable for the time that they reserve. If you find that you cannot make your reserved time for any reason, please delete your reserved time slot so that another user may take your time.
   a. Past time cannot be deleted (i.e. you cannot delete a 12:00 reservation at 12:02 pm).
   b. Tip: If you do not know if you will need the full 30 min, consider signing up for a block of 20 min, and then 10 min, and cancelling the latter block as time permits.
   c. You can contact the user of a reserved block through the online scheduling utility by clicking on their block of time and typing a message; it will be sent by email.

6) All users are required to sign in at the logbook at each workstation, indicating times, name, solvent, and nuclei. Notify the NMR manager promptly in case of a problem.
Note: Failure to comply with signup and usage rules will lead to suspension or termination of user privileges.

If you have any questions, contact the NMR manager.

Instrument configuration and use

Two complementary probes are installed by default on the NMR instruments to best highlight the strengths of the two NMRs. Your choice of instrument should reflect the experiment needed:

- For high-sensitivity heteronuclear experiments such as $^{13}$C and $^{31}$P, as well as routine $^1$H acquisition, the Bruker AVANCE III 500 Hz NMR with broadband direct detection probe should be used.
- For high-sensitivity $^1$H, as well as advanced 2D experiments and chiller-cooled variable temperature NMR (down to -15 °C), the Varian Unity/Inova equipped with a PFG (pulsed field gradient) inverse detection probe should be used.
- Both the Bruker and Varian are equipped to handle elevated-temperature and liquid nitrogen-cooled low-temperature NMR experiments.

Looking to run a specific experiment? Contact the manager for training on heteronuclear or multidimensional NMR experiments, or to have a new experiment configured to fit your needs.

One weekend per month upon reservation at least one week in advance, the Bruker Avance NMR may be dedicated to solid-state NMR. Installation of the solids probe for these weekends will necessitate that the facility manager have access to the instrument typically starting at noon that Friday and until noon the following Monday to reconfigure the instrument.

Data processing and archiving

Currently, data is not archived on an external server. You, the researcher, are responsible for regularly downloading your files from the spectrometers remotely using FTP software, or locally via USB memory key. It is highly recommended that you keep hard copies of your spectra and back up your digital data frequently.

NMR data will be purged periodically. It is the user’s responsibility to archive their own data.