

## Basic NMR Training at UO NMR Facility

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This is a brief outline of good practices you should follow while using the UO NMR facility. We strongly encourage you to review this "cheat sheet" before using the NMR facility for the first time. This document includes information for the most basic NMR experiments that can be run at UO NMR facility. More advanced users can sign up for training or find application guides on the NMR website: [nmr.uoregon.edu](http://nmr.uoregon.edu). There are 6 categories covered: Safety, Facility Access and Instrumentation, Sample Handling, Basic Data Acquisition, Data Processing, and Basic Troubleshooting.

### SAFETY

- 1) The NMR Facility is a laboratory space. This means it is a no food, no drink zone. Proper laboratory attire (long pants or equivalent and closed toed shoes) are required in order to enter the NMR Facility.
- 2) The defining feature of NMR labs is the presence of high magnetic fields. For your own safety, if you have a pacemaker or surgical implant that could possibly be adversely affected by strong magnetic fields, do NOT enter the NMR labs.
- 3) Users must never bring ferromagnetic materials (e.g., keys, paper clips, hair pins, AirPods, etc.) within the 5 Gauss line or close enough to the magnets to experience a force. Before entering the lab, think about what you are bringing in!
- 4) If the fire alarm goes off, calmly and safely leave the facility and evacuate the building.
- 5) First aid kits and spill/clean-up kits are located by the main entrances.

### FACILITY ACCESS & INSTRUMENTATION

The NMR facility at UO is available 24/7 to trained users via key code access, granted after initial training. You can make an online reservation by opening the web calendar @ <http://hotwax.uoregon.edu> and logging in with your NMR username and password. You must be on the campus network or VPN connected.

The UO NMR Facility is equipped with 4 high field NMR spectrometers: 2 Varian and 2 Bruker. Each instrument has additional hardware, but these are the probes typically installed:

Instrument	Probe	Suggested Usage
Varian Inova-500 (Kla-344)	5 mm AutoX DB probe with z-gradient	Routine 1D NMR: ( $^1\text{H}$ , $^{19}\text{F}$ , and $^{15}\text{N}$ - $^{31}\text{P}$ ), DOSY, 2D NMR, VT $-80^\circ\text{C} \rightarrow +130^\circ\text{C}$
Bruker AV-500 (Kla-344)	5 mm Prodigy CryoProbe with z-gradient	Routine 1D NMR ( $^1\text{H}$ , $^{19}\text{F}$ , $^{15}\text{N}$ - $^{31}\text{P}$ ), 2D NMR, DOSY, limited VT $0^\circ\text{C} \rightarrow +80^\circ\text{C}$
Varian VNMRS-500 (Kla-60)	1.6 mm HXY Phoenix probe	Solid-state NMR, limited solution state NMR (1D, 2D, etc)
Bruker AV-600 (Kla-60)	5 mm Prodigy CryoProbe with z-gradient	Routine 1D NMR ( $^1\text{H}$ , $^{19}\text{F}$ , $^{15}\text{N}$ - $^{31}\text{P}$ ), 2D NMR, DOSY, limited VT $-40^\circ\text{C} \rightarrow +80^\circ\text{C}$

The Varian VNMRS-500 and Bruker AV-600 are capable of solid-state NMR experiments. Please contact the NMR Facility Director (Dr. Nanette Jarenwattananon [njaren@uoregon.edu](mailto:njaren@uoregon.edu)) for more information.

### SAMPLE PREPARATION AND HANDLING

- 1) All sample handling should be done in the bench areas. No samples on the workstation desk!
- 2) EH&S requires all chemicals being transported in public areas to be in a secondary containment with a lid. Use an approved secondary container with a lid.
- 3) Use clean and dry NMR tubes. DO NOT write on NMR sample tubes.
- 4) Use only high-grade NMR tubes made out of Pyrex (ASTM Type 1 Class A). You can buy these from ScienceStores or directly from Wilmad (507-PP-7, 528-pp-7, 535-PP-7), Norell (S-5-200-7 or S-5-600-7), or New Era (NE-HP5-7, NE-UP5-7). Standard NMR tubes are 7" long. The maximum tube length is 8".
- 5) When possible, we recommend you use a sample volume of ~0.5 mL. When shorter samples are required, make sure to center the sample around the line marking the center of the *rf*-coils when using the depth gauge.
- 6) Use deuterated solvent.
- 7) Filter your sample to remove any particles from the solution.
- 8) Before you insert your NMR tube into the spinner, make sure to wipe thoroughly with a KimWipe.

- 9) Avoid handling spinners with your bare hands. Try to use a Kimwipe if possible.
- 10) If a tube is loose in the spinner, do NOT use it.
- 11) Before placing your sample in the instrument, make sure the eject air is ON!
- 12) All solution NMR samples are run **non-spinning** in this facility.
- 13) If your sample breaks outside of the instrument, you are responsible for cleaning it up. Cleaning tools are located at the main entrance to the lab. Dispose of any broken glassware and chemically-contaminated material in your own lab. Notify NMR facility staff by filing a problem report.
- 14) **If your sample breaks inside an instrument, contact NMR facility staff immediately.** Clearly mark the instrument "Out of Service" by leaving a note.

## BASIC DATA ACQUISITION

The general data acquisition procedure is the same, whether or not you use a Bruker or Varian spectrometer: insert your sample, read in acquisition parameters, lock to deuterated solvent, tune the probe, shim the magnet, edit acquisition parameters, set the receiver gain, acquire data. However, the details and commands for acquisition will differ. Consult either the Bruker or Varian handouts for more information.

## DATA PROCESSING

MestreNova is free of charge for University of Oregon students and researchers. You will need to be connected to the UO network when activating the Mnova license. You will also need the license file.

The MestreNova License should not be shared with outside parties as the license is only available to UO students and researchers. If a data processing software is required and you are not affiliated with the University of Oregon, Bruker offers the TopSpin data processing software free to academic, government and non-profit institutions. If you utilize the Varian spectrometers, there is a free open-source version of VnmrJ available.

There are two work station computers in the center of the Klamath 344 NMR lab. VnmrJ, TopSpin and Mestrenova are all installed on these computers. If you need assistance accessing your data on these computers, please contact Nanette.

You can also remotely access your data from a computer connected to the campus network or VPN connected. Use an SSH or SFTP program to connect to the NMR data server **hotwax.uoregon.edu** using your **NMR username and password**. Examples of SSH/SFTP software for Windows are Putty, CuteFTP, WinSCP, Cyberduck and FileZilla. Mac software includes Cyberduck and Fetch.

## BASIC TROUBLESHOOTING

- 1) Double-check your sample prep if something isn't working correctly. Problems can be caused by the incorrect solvent, undissolved solids, or bubbles.
- 2) If you have difficulty locking try:
  - a. Loading the default shims and then attempting to lock
  - b. Manually locking
- 3) Approximately 90% of the problems you encounter are software related. The first thing to try is to restart the NMR software. In both TopSpin and VnmrJ, you can click the X in the upper right. Relaunch the software from the desktop icon.
- 4) If that doesn't resolve your issue, it's time to get help from the NMR facility staff.
  - a. If a staff member is in the facility or in their office and available, flag them down.
  - b. If it's a simple problem, you can describe in a sentence or two, file a Problem Report on the Facility website or email Nanette.
  - c. If it's a more complicated problem or you get any error messages, please file a Problem Report, making sure to note down the error message.
- 5) If your problem would prevent someone else from acquiring even a simple <sup>1</sup>H experiment, you should let other users of the instrument know so they can plan their experiments accordingly.
  - a. Leave a note at the instrument computer, e.g., "Sample Stuck Inside-- Out of Service".
  - b. Email the NMR mailing list: [nmr\\_internal@lists.uoregon.edu](mailto:nmr_internal@lists.uoregon.edu)