

1.2 Supporting Computers and Software

NMR Facility needs are supported by the computers and software indicated below.

- Sun Ultra 10 computers running the Solaris 8 (SunOS Release 5.8) UNIX operating system: one hosts the UI-500 spectrometer and the other is for off-line data processing
- Varian *VNMR* 6.1C software for UI-500 spectrometer control and Varian data processing
- Hewlett-Packard Z440 AV-400 host computer running the CentOS 7 Linux operating system
- Bruker TopSpin software for AV-400 spectrometer control and Bruker data processing
- Dell Optiplex 780 PC (Microsoft Windows 7) for independent data processing, archiving, and other tasks (Refer to [Table 2.1](#) on [page 24](#) for a list of software available on this computer.)
- Sun NFS protocol and Samba server provide connectivity between Microsoft Windows clients and NMR Facility data disk partitions
- HP LaserJet network-accessible printer

1.3 NMR Facility Policies

This section enumerates the current policies governing NMR Facility usage. These policies are subject to periodic review and revision; suggestions from the NMR user community for revisions are welcome. If you have comments or suggestions, please submit them to the NMR Facility Director. **Facility users are required to know and understand these policies**, and are encouraged to provide feedback for the benefit of the entire NMR user community.

1.3.1 Access and Use

The NMR laboratory is **a restricted-access facility for authorized users only**. Access is gained through direct authorization on an individual basis, obtained via user training and subsequent checkout procedures designed to ensure that all users are capable of safe and appropriate use of the NMR spectrometers and ancillary equipment. Authorized users will have individual (1) key-card access to the NMR Facility and (2) active computer accounts to operate the equipment for which they are granted access. Only those with authorized access are allowed to use the Facility equipment; **sharing of computer accounts and/or key access is explicitly prohibited**.

Taking or allowing *guests* into the laboratory is not permitted without prior approval by Facility staff. Approval can be arranged, in advance, for a variety of cases; contact the NMR Facility Director for more information. University security personnel patrol the School of Pharmacy, including the AIC, and users may be asked to show their UW identification and after-hours building permits. As a restricted-access facility, the NMR laboratory doors are to be closed and locked under normal circumstances, not left open or ajar; security personnel check this also.

1.3.2 User Training

Training is provided only by NMR Facility staff. If necessary, the standard training objectives may be expanded to better meet individual needs according to previous experience and anticipated research objectives. A variety of training events are offered periodically, usually on an *ad hoc* basis. Refer to the [NMR Training](#) web page for detailed information and a link to the on-line training registration form.

1.3.3 Fees and Services

The NMR Facility is primarily a user-operated laboratory in which individuals acquire, process and analyze their own data. Monthly accounting of and billing for spectrometer usage is in effect. A flat rate of \$4.00 per hour for both the AV-400 and UI-500 NMR spectrometers currently applies to internal (UW) customers who operate the instruments themselves. Direct access or NMR spectroscopy services are available to external (non-UW) customers at higher rates. Refer to the [AIC Fees](#) web page for more information. The fee structure is subject to periodic review and modification; the user community will be notified before rate changes take effect.

1.3.4 Reserving Instrument Time

An [Instrument Reservation System](#) (IRS) is available for authorized NMR Facility users to reserve time on the UI-500 spectrometer. (The IRS provides similar capabilities for MS Facility users.) Detailed Help Notes and Sign-Up Rules are available within the IRS after logging on via your user name and password.

1.3.5 Laboratory Safety and Health Issues

Access to the NMR laboratory (room 1411) is restricted to only those individuals who have either (1) successfully completed an NMR training course by Facility staff, which includes discussing and completing a safety checklist, or (2) have otherwise received training, from Facility staff, regarding the potential dangers inherent in a magnetic resonance facility. Requests for access authorization must be made through the NMR Facility Director. These restrictions apply to all personnel: NMR users, custodians, maintenance workers, etc.

The potential dangers inherent in a magnetic resonance facility involve the presence of strong magnetic and radio-frequency fields and cryogenic fluids (liquid nitrogen and helium), plus the general hazards of handling chemicals and glassware (primarily NMR tubes). Because only Facility personnel handle cryogenics in the NMR lab, related precautions are not discussed further in this document; common hazards regarding other topics are described below. Note, however, that the following examples are in no way all-inclusive; it is always the responsibility of each individual to ensure that safe practices are followed. When in doubt, consult with the NMR Facility Director before proceeding.

Refer to the [NMR Laboratory Mishaps](#) web page for select descriptions of real-life laboratory accidents, near misses, etc.

Preliminary Considerations

-  **Authorized Access Only** The NMR Facility (room 1411) is a restricted-access laboratory. Only those directly authorized are allowed into the lab, and the doors are to be shut and locked at all times except during entry and exit.
-  **Food and Drink** Neither food nor drink is allowed in the NMR laboratory. Period!

- ⊘ **Proper Attire** Loose-fitting or high-heeled shoes should not be worn in the NMR laboratory. Such footwear greatly increases the risk of losing balance or falling when using the step platforms to insert or remove sample tubes from the magnets. Open-toed shoes of any kind are prohibited in laboratories by campus policy. You are responsible: Be safe, not sorry.

Hazards Related to Super-Conducting Magnets

- ⊘ **WARNING:** Persons with implanted or attached medical devices such as pacemakers or prostheses are not allowed to enter the NMR Facility (room 1411) without authorization from a physician.
- ⊘ **WARNING:** High-field super-conducting magnets produce very strong, fringe magnetic fields that extend **in all directions** beyond the magnet canister, presenting invisible yet very real dangers related to the forceful attraction of ferromagnetic objects. These magnets are always on and cannot simply be turned off. The UI-500 magnet has its radial 5-Gauss perimeter marked out on the floor with red tape, and the AV-400 magnet's 5-Gauss perimeter falls within an imaginary circle circumscribing the magnet legs. Ferromagnetic objects must be kept outside these 5-Gauss perimeters at all times.
- ⊘ **WARNING:** Although fairly common during the initial energization of super-conducting magnets, the violent quench of a stable magnet does occasionally occur. Violent quenches can cause the liquid helium (e.g., 120 L in the UI-500's Oxford AS500 magnet when full) to boil off in a matter of seconds, venting spectacularly through safety check valves at the top of the magnet canister. If this happens, evacuate the lab immediately — after recovering from the initial scare. The very real danger associated with a violent quench lies in the risk of asphyxiation due to the displacement of oxygen in the room.⁴ Normal building ventilation will flush the helium gas out of the room after some time (about 15 minutes); there is no other danger and no real need to evacuate the building (although it would be okay to do so). Inform the NMR Facility Director of the news.
- ⊘ **CAUTION:** Magnetically encoded media (e.g., ATM cards), mechanical watches and some electronic devices may be damaged or destroyed if subjected to strong magnetic fields; keep such items outside the 5-Gauss perimeters.
- ☞ **Fire Extinguisher** A non-ferromagnetic, CO₂ fire extinguisher is located at the right-hand end of the laboratory bench in room 1411.

Chemical and Glassware Hazards

- ☞ **Chemical Hazards** NMR Facility users are responsible for knowing the chemical hazards of their compounds, and for taking proper steps to ensure their own and others' safety at all times, e.g., in the event of sample tube breakage and subsequent spill. It is the user's responsibility to completely clean up any spill, broken glass, etc., to the extent possible.
- ☞ **Radio-Nuclides** No. Samples containing enriched quantities of radio-nuclides are not permitted in the NMR Facility.

⁴The rapid boil-off of 120 L of liquid helium would produce approximately 90 m³ of gaseous helium, which is roughly 1/3 the total volume (at 310 m³) of the NMR lab.

- ☞ **Sample Preparation** Whenever possible, NMR samples should be prepared in advance in the user's laboratory. If sample preparation must be done in the NMR Facility (as is typically the case for kinetics studies, for example), it is to be done only on the laboratory bench in room 1411; the computer desks or spectrometer consoles are never to be used for such purposes.
- ☞ **Toxic or Unpleasant Substances** Such substances shall be addressed responsibly according to their nature. For example, flame sealing a sample within an NMR tube may be required to contain toxic vapors or an offensive smell.
- ☞ **Sample Disposal** Facility users must promptly remove their samples and related materials from the laboratory when their experiments are completed. Arrangements can be made for those with special needs to store samples/tubes in the lab to facilitate their work; however, unlabeled or unclaimed NMR sample tubes or related goods persisting in the laboratory will be discarded.
- ☞ **Gloves** If needed for extra protection, gloves (e.g., latex, nitrile) may be worn only while preparing or handling NMR samples. Gloves are **never** to be worn while operating computers or handling other community property.
- ☞ **Glassware Hazards** Routine precautions should be observed when handling glassware, especially when inserting and withdrawing NMR tubes into and from the spinner turbine. Some spinner turbines use rubber O-rings to grip the NMR tubes, and the fit can be quite snug, depending upon the condition of the O-ring and the specific NMR tube used. Grip the tube firmly near the spinner and use a twisting motion while inserting or withdrawing the tube. Carelessness has resulted in puncture wounds. *Ouch!* 😊

Miscellaneous Considerations

- ☞ **Be Careful!** Users must carefully insert and remove their NMR samples into/from the magnets, positioning themselves to maneuver the glass tube straight up or down — not at an angle — out of, or into, the upper barrel. Glass does not bend well at room temperature, and we have had far too many users snap a sample tube by catching it at an angle at the top of the upper barrel. These events are distracting and time-consuming to deal with, are potentially damaging and costly to the equipment, and **are easily and completely preventable**. If you think you're in a hurry in the NMR lab, go away and come back after you've adjusted your attitude; this is no place for reckless or irresponsible behavior!
- ☞ **Hands Off!** Please keep your hands off the magnet canisters. If you feel compelled to support yourself while inserting or removing samples from the magnets, then you are probably doing something else wrong.
- ☞ **Common Sense** It is apparently necessary to remind some users to wash their hands and wipe their feet. Come on folks, this is a research laboratory, not kindergarten! Winter in Wisconsin involves snow and ice and sand and salt; these all belong outside, not in the NMR lab, so please do not track this crap into the lab. Let's keep our laboratory space and community property — keyboards, mice, work desks, floor, etc. — clean.
- ☞ **Temperature Control** Variable-temperature (VT) work may be performed **only** after an individual has completed specific, on-site training by NMR Facility staff.⁵ Users are responsible for

⁵VT work may be performed only on the UI-500; sample temperature on the AV-400 is maintained at 25 °C at all times.

knowing and observing the temperature limitations of both their NMR samples and the Facility instrumentation, and must work safely within these limitations. Facility personnel are available for consultation and other assistance in these matters; refer to [Section 7.3](#) for further information.

- 🔔 **Eye Protection** Users must provide and use their own eye protection as needed.
- 🔔 **Consequences** Unsafe, irresponsible or otherwise inappropriate use of the NMR Facility may result in sanctions up to and including loss of access privileges.

1.3.6 AV-400 Sample-Management Policies

The sample workflow for an NMR spectrometer without an automated sample handler (e.g., the UI-500) is relatively uncomplicated: Individuals use the instrument serially, each person having one or more samples that they manually manage in sequence; consequently, the samples are essentially under control by their owner from start to finish. Because investigators are charged for instrument usage according to log-on time, individuals are usually motivated to log off from the instrument and remove their samples as soon as the experiments are completed.

Sample workflow and sample management are quite different for systems, such as the AV-400, with automated sample handlers. Here, the very core of the idea is to free the individual from the routine task of manually managing samples one after the other. This is great! (At least for many types of routine systems and experiments that lend themselves to this treatment.) It allows multiple users to each submit multiple samples in an *ad hoc* manner, with subsequent sample handling and other spectrometer functions executed under control of the automation software. Today's automation software is typically capable of sending users their spectral data via email. Wow! What is there to not love about this?

Automated sample handlers are available in a range of models accommodating from a few to hundreds of NMR samples; pick the model that best suits your goals and budget. An institution's particular needs and conditions determine its sample-management policy. The workflow in a Big Pharma research lab is obviously much different than that in an undergraduate teaching lab. In our NMR Facility, data show that a few tens of NMR samples are run during a typical 24-hour period.⁶

Considering that our SampleXpress automation robot has a capacity of 60 samples, and our currently typical throughput is approximately half of that (give or take), completed samples need to be removed from the sample handler on a daily basis to make room for incoming samples. It is the responsibility of individual users to remove their NMR samples from the SampleXpress unit in a timely manner. If necessary, individual users may remove others' completed samples to make holders available for incoming samples. Finally, in addition to all the other well-known reasons for properly labeling and documenting things in scientific research, this discussion should make clear the importance of legible and meaningful labeling of NMR sample tubes. Specific sample-management policy rules follow:

- If left in the NMR lab, all flasks or other containers for transporting NMR samples must be clearly and legibly labeled with the user's full name (not initials, etc.) so that ownership can be determined. Any such item that is not clearly identified will be discarded.
- The step unit has an integrated work table to facilitate adding and removing multiple samples from the SampleXpress sample changer. Spinner turbines, a depth gauge and tissues are kept on the table, which also provides a temporary place for a flask or other NMR sample-transport

⁶Roughly speaking, 30 ± 10 samples per day is a fairly typical throughput, although examples of more or fewer samples per day exist. A daily throughput of more than 60 samples is virtually unprecedented in our laboratory at the time this was written.

container during the work-flow process. When done adding or removing NMR samples at the sample changer, the container is to be removed from the work table — no exceptions.

- Users may, if desired, temporarily leave their clearly labeled container on the NMR lab bench if they have one or more samples in the sample changer. There is a designated area for this purpose.
- Each NMR facility user is responsible for removing his or her samples and related equipment from the lab in a reasonably timely manner. The term “reasonably timely” here means, for example, the next day for weekday sample submissions, and Monday for weekend sample submissions. This is not currently an absolute rule; instead, the goal is to foster responsible behavior.
- It is occasionally necessary for Facility staff to remove all the NMR samples from the SampleXpress cassette for system maintenance or repair. Users must therefore be able to identify their own samples from a collection of several.

1.3.7 Incident Report Form

This chapter ends with important comments and information about how and when to report problems — either real or perceived — related to the NMR Facility. Problems, issues and conditions appear in all manner of shape, size and significance; the term *incident* is used here in reference to such phenomena. It is the responsibility of the user community to promptly and properly report incidents they experience — or *cause*, as the case may be. During normal working days and hours, please contact directly either the NMR Facility Director or the Project Assistant (PA). For non-emergency incidents outside normal working hours (or if the Director or PA are otherwise unavailable), make a formal report via the on-line [Incident Report Form](#); this method ensures the most timely and meaningful reporting and response, no matter what time or day the incident occurs. Emergencies should, of course, be reported immediately via the proper channels, depending on the details; emergency contact information sheets are posted outside the laboratory doors and at the telephone within the NMR lab itself.

Many years of experience dealing with these kinds of issues prompts the following comments:

- If you experience an incident, report it. Some kinds of problems are real and universal, while others are imagined or isolated; therefore, unless a particular incident is reported, it may be unknown and remain unknown to facility staff.
- Do not assume that a particular incident you experience has already been reported by someone else; this is a corollary to the preceding point.
- Even if you know that a particular incident has been reported, report it again. Perhaps the problem is thought to have been fixed but has actually recurred, which is important to know. Intermittent problems can be exceedingly difficult to diagnose and repair, and it is important to know how frequently they occur; relatively minor issues that occur infrequently are assigned lower priority than if they occur frequently.
- Be responsible! In some circumstances, it may be necessary for you to personally take immediate action to prevent equipment damage or ensure the safety of others. For example, imagine what could happen if someone broke an NMR sample tube in the magnet, then simply walked off without taking measures to prevent another person from subsequently inserting a sample into the broken glass on top of the probe. (Yes, this really happened! What would **you** do?)