UNIVERSITY OF ALBERTA

NMR Laboratory General Safety Information

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College of Natural and Applied Sciences
Faculty of Science
Department of Chemistry NMR Facility
uab.ca/nmr
780.492.2573

Overview

The use of the Nuclear Magnetic Resonance (NMR) instrumentation within the Department of Chemistry is a privilege not a right. It is of utmost importance that each user demonstrates an understanding of all the safety information and hazards by complying with correct standard operating procedures. Failure to do so can result in instrument damage and more seriously personal injury and even death. Due to the high cost of the NMR equipment and related repairs **all users must inform NMR personnel of any issue(s) with equipment and/or supporting infrastructure. Do not attempt any repairs yourself**. Failure to do so will result in the suspension of privileges.

Staff Contacts

The NMR staff can be found in the West Chemistry Bld., room WB-13, and the laboratory phone number is **780.492.2573**. Contact information is provided on the desk beside each phone in the NMR facility, on the website, and on the instrument room doors.

General Guidelines and Procedures

The Magnetic Field

The NMR spectrometers contain powerful superconducting (*i.e.* almost no resistance) electromagnets requiring no external power source. The materials comprising the core wiring of the magnet remain superconducting as long as the extremely low temperatures are maintained[1]. Because of the nature of the superconducting permanent magnet, it cannot be turned off without trained personnel, special equipment, and substantial risk of damage to the magnet.

So please be aware: **The magnets are ALWAYS ON!**

To obtain NMR spectra with high resolution and sensitivity strong static magnetic fields are required. Magnetic field strengths from 9.39 Tesla to 16.5 Tesla[2] (400-700 ^{1}H MHz) can be found within the Department of Chemistry. An 18.8 Tesla magnet (800 ^{1}H MHz) is expected in 2023. There are large attractive forces associated with these magnets and every magnet has a stray magnetic field that extends out past the physical structure of the magnet. The magnetic field increases exponentially the closer one approaches, and there are large and increasing attractive forces closer to the magnet. Also, the larger the mass of the equipment, the larger the attractive force. The safety zone or 5 Gauss, i.e. 5G line is indicated by signs, plastic chains, and yellow lines on the floor around each NMR magnet.

[1] Liquid helium (minus 269°C) maintains the magnet coil, and liquid nitrogen (minus 196°C) shields the helium. The liquid nitrogen is sacrificed to reduce the most costly liquid helium boil off.

[2] Compared with the Earth's magnetic field of ~1x10^{-4} T; 1 Tesla = 10000 Gauss

Please note - **Only the Department of Chemistry uses yellow lines to denote the 5G line.** When using instrumentation in other departments, check with their staff for designated signage.

Example of 5G line

Figure 1 - 700 MHz NMR spectrometer with 5G point indicated by the painted yellow line on the floor.

Inside the 5 G line, near the magnet

- Damage, injury and even death can occur to users who have medical implants and/or pacemakers.
- Damage can occur to personal items such as mechanical watches, cards with magnetic strips, magnetic media, *etc.*
- Common metal items such as tools, pressurized gas cylinder tops, nails, chairs, mop buckets, etc. can be drawn quickly and unexpectedly to the magnet and can even become a projectile, potentially causing personal injury and equipment damage.

- Large equipment such as gas cylinders can be particularly dangerous due to their mass. These items can cause crushing amputation injuries and death.
- Small objects drawn to the magnet can become lodged inside the central bore of the magnet while larger objects will likely remain on the side of the magnet. These items can be difficult or even impossible to remove, impeding the function of the spectrometer.
- **Objects drawn quickly to the magnet can puncture the structure of the magnet or distort the magnet's internal structure causing the magnet to quench.**

The spectrometer host computer is situated outside the 5 G line, and it is recommended that users remove all metallic objects and valuables (*i.e.* coins, tools, cell phones, bank cards, watches, *etc.*) and leave them beside the spectrometer host computer before approaching the magnet.

Persons with medical implants/devices or medical concerns are recommended to stay outside of the 5G lines in labs that house the NMR spectrometers. Warning signs are posted at the entrance of all NMR labs, as shown below.

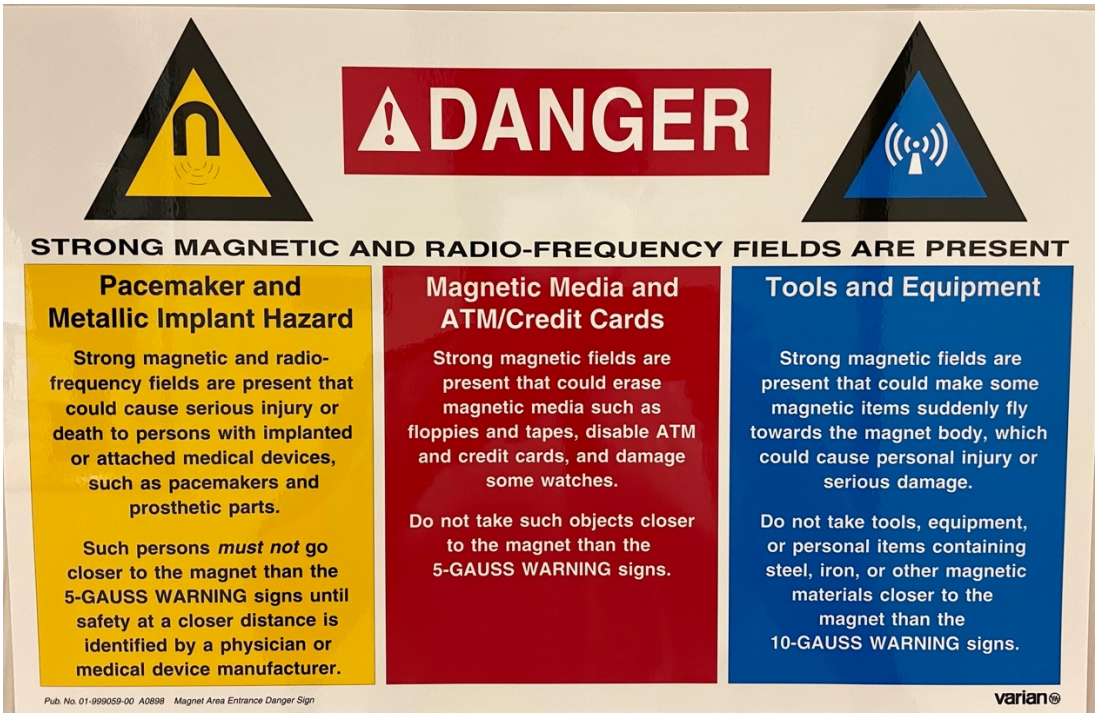

Figure 2 – Magnetic field warning signs posted outside of every NMR lab.

Metal objects such as compressed gas cylinders or other equipment should be moved in NMR magnet rooms only by the authorized personnel (*i.e.* the NMR staff).

Magnet Quench

Superconducting NMR magnets contain a large solenoid or coil made up of several kilometers of superconducting wire. This coil normally resides in a bath of liquid helium, however if even a small section of the solenoid wiring warms or is impacted sufficiently that section of wire may become partially resistive causing heat generation. This heat will cause a cascading heating event quickly spreading to neighbouring wire sections which in turn cease being superconducting and generating more heat. This cascade event is called a magnet quench. Safety components in the magnet are designed to convert the entire magnet energy to heat and direct the energy into the surrounding cryogens. During a magnet quench, there will be a sudden, large, rapid, and noisy expulsion of helium gas from the magnet (Fig. 3) as liquid helium vaporizes into a gas (1L of helium turns in to 757L of gas@STP). Magnet quenches are very rare events but if a quench occurs:

- There will be a sudden and loud release of helium gas from the top of the magnet. Clouds of water vapor will appear as the air cools, and frost will appear on top of the magnet and anything along the path of release (see Fig. 3)
- The rapidly expanding helium gas emanating from the magnet can displace the air in the room and there is a possibility of asphyxiation in a confined space.

Figure 3 - April 10, 2003 a relatively small 200 MHz NMR magnet quench during decommissioning of the instrument.

What to do in case of a Quench

- Do not panic!
- Notify everyone in the vicinity to evacuate.
- Leave the room immediately.
- When everyone is out, close the door behind you.
- Warn others not to enter the room.
- Send someone or notify the NMR personnel yourself immediately.

Oxygen Sensors

Figure 4 – Example of an oxygen sensor located (room EB-44).

Figure 5 – Example of oxygen visual alarm.

Due to the number of NMR magnets, users, and/or room size, rooms SB-3E, and EB-44 have oxygen sensors with audible and visual alarms (examples shown in Fig. 4 and 5). These alarms will sound when oxygen levels have dropped (potentially due to a magnet quench) to unsafe levels. If these alarms, lights, or sounds activate warn everyone and leave the room immediately. DO NOT re-enter the room.

NMR Tubes and Samples

NMR tubes are thin-walled glass tubes and are quite fragile. Although they are used daily, personal injury can (and has) resulted when fixing the cap to the tube and/or inserting the tube into the NMR spinner.

When handling your NMR tube we recommend:

- Use fingers not the palm when inserting an NMR tube into a spinner and/or depth gauge.
- Use care and no excessive force when inserting the sample into the spinner, very little effort should be required. Examine the NMR tube and spinner for foreign material if force seems necessary and seek assistance from the NMR staff.
- Be careful when securing the cap to an NMR tube.
- Dispose of any NMR tubes that are chipped, cracked, scratched, or damaged in any manner. Damaged NMR tubes greatly increase the likelihood of personal injury and equipment damage. **Do not use damaged tubes.**
- Do not dry NMR tubes in an oven. NMR tubes are precision pieces of glassware and the heat of an oven will distort the shape of the NMR tube. Distorted oven dried NMR tubes will likely break in the spinner and/or may damage the spectrometer.
- Always TURN ON ejector air before inserting your sample in the magnet. Neglecting to do so can break your sample and damage the instrument.

With enough time a broken sample tube is likely inevitable, however care must be taken by the end user to ensure the health and safety of the other users of the NMR facility. Samples may be harmful, especially if they are an unknown. Items and surfaces at the spectrometer host computer are communal items that everyone uses, *e.g.* depth gauge, spinner, mouse, keyboard, desk, *etc.* and therefore one must be courteous and careful. Some of these items do need to be disassembled to be cleaned properly.

If a sample breaks, the end user is required to:

- Inform the NMR staff and ask for assistance.
- Do not attempt to clean the spinner, get Staff help.
- Clean up all broken glass and place in a labeled glass waste container.
- Make sure all equipment is free of solvent/sample by cleaning up all chemicals.
- If a sample is broken within the magnet, the NMR staff must be informed immediately! Stop all NMR and place Out of Order sign in front of keyboard.

Miscellaneous

NMR magnets tend to be quite large. Steps and/or ladders are provided to facilitate inserting and retrieving samples from the magnet. Take caution to avoid losing your balance, falling, dropping samples, *etc.*

Due to the multiuser environment and high throughput of the NMR facility within the Department of Chemistry eating is not permitted in the lab. While sealed beverages are technically permitted, they are strongly discouraged whenever possible.

Please refrain from having guests in the facility, but if absolutely necessary never leave them unescorted.

Specific Hazards and Procedures

1. Cryogenic Liquids

Liquid nitrogen and liquid helium used in the NMR magnets (and for variable temperature NMR studies) is extremely cold (boiling points: -196 and -269 °C, respectively), and must be handled with the appropriate equipment and attention.

- Containers of cryogenic liquids must not be completely sealed as a pressure build-up (due to increased volume from vaporization) presents an explosion hazard. Although kept in insulated dewars, the liquid cryogens will gradually evaporate.
- Non-magnetic dewars must be used for the transport and storage of cryogenic liquids to prevent attraction to the magnets. The use of magnetic dewars are accessories could result in injury or even death.
- Cryogenic liquids can easily cause freezing damage if they come in contact with exposed spin. These liquids should always be handled with insulated gloves, eye protection, closed-toed shoes, pants, and long-sleeved clothing. If any of the cryogenic liquids come into contact with eyes or skin, flood the affected area with cool water and seek medical attention.
- Inserting a warm object, such as a transfer line, into the cold liquid will result in rapid boiling and splashing. The same will occur when transferring the cryogen into a warm container.
- In the case of a large spill, the expanding cryogens may displace oxygen from the room, leading to asphyxiation. Vacate the area immediately.

2. Gas Cylinders and Other Equipment

Compressed gas cylinders must be stored and transported according to standard operating procedures:

- Gas cylinders should **never** be brought into close proximity of the magnet as the large attractive forces of the magnet may act on the cylinder.
- Gas cylinders should be transported on non-magnetic 4-wheel hand carts with restraints for the cylinder.
- Gas cylinders should always be stored and transported with the protection cap on.
- Gas cylinders must be secured upright and individually chained to a strong, stable structure such as a wall or bench.
- Regulators used for the dispensing of the compressed gas should be checked regularly for leaks.

When working near the magnet be mindful of the 5 gauss line and ensure that all unnecessary equipment is kept outside of this area.

If work is to be performed on or near the magnet, ensure that all tools are non-magnetic.