NMR Instrumentation

University College Dublin
School of Chemistry

General information:

Magnetic Fields

The NMR spectrometers incorporate superconducting magnets which exert a very high magnetic field. This field is so strong that it can pull metal objects to the magnet (eg. take a spanner from your grasp) if they are not within the safe operating distance, otherwise known as the 5 Gauss Line. The closer to the magnet, the larger is the force, which can come as a surprise. Therefore, *all metal objects must be kept as far away from the magnet as possible.*

A metal object becoming accidentally attached to the magnet **must be avoided** as it would disrupt the shimming and therefore sample lineshape radically; to remove it would require bringing the magnet off-field at great expense. There is another possible consequence: the disruption of the field around a localised region of the superconducting coil can lead to localised heating of that area of coil, causing the superconducting wire in the coil to lose superconductivity. The increased resistivity of the now normal wire will heat the helium, causing a very rapid chain reaction resulting in all the helium in the magnet being heated almost at once, changing it from liquid to gas. When this happens, the gas expands dramatically in volume, causing risk of asphyxiation to anyone present, as it leaves the magnet and fills the room very quickly. This whole process is known as a **quench**.

In the event of a quench:

Leave the room immediately and ensure nobody else enters the room.

Sound the fire alarm and leave the building.

DO NOT RE-ENTER THE ROOM until the NMR staff and/or safety officers declare it safe.

YOU MUST CONTACT an NMR staff member and/or call UCD emergency number (01 716 7999).

If you can see somebody who is incapacitated in the room, open the door/window to allow oxygen into the room, but DO NOT RE-ENTER THE ROOM as you must ensure your own safety. Helium is inert, but it is an asphyxiating gas and large quantities can displace oxygen in a confined space. Please read carefully the risks and safety procedures associated with helium and other cryogens available at the NMR laboratories.

How would I spot a quench?

A quench warranting evacuation would be obvious by the noise of the escaping gas and white cloud developing at the ceiling. Oxygen depletion alarms and helium detectors are fitted in the room, but even if these are not sounding, leave the room IMMEDIATELY following the above procedures if a quench occurs.

Pacemakers, Medical Implants and Prosthetic Devices:

Cardiac pacemaker wearers must not enter an NMR laboratory until permission has been given by NMR staff. An NMR superconducting magnet generates strong magnetic and electromagnetic fields that can inhibit operation of some cardiac pacemakers, resulting in death or serious injury to the user. Consult the pacemaker user's manual, contact the manufacturer, or confer with a physician to determine the effect on a specific pacemaker. Overall, it is advisable that those with pacemakers, medical implants (clips, stents) or other prosthetic devices do not enter the room at all without prior consultation with NMR staff.

Bank Cards, Watches, Mobile Phones:

One would advise a common sense approach towards approaching a superconducting magnet with items which can be influenced by magnetic fields, such as bank cards, phones, watches (digital, battery-operated and wound), calculators, laptops and PDAs. These can be permanently damaged by the magnetic fields. Therefore, do not approach the magnets with these items. The School of Chemistry and their staff accept no responsibility or liability caused by approaching the magnets with such items. If you do, it is your own responsibility.

Care with Samples:

Do not exceed the boiling or freezing points of your sample. A sample subjected to a temperature change can build up excessive pressure which can break the tube. Broken glass, projectiles and hot or toxic chemicals can cause injury. To avoid this hazard, establish the freezing and boiling points of a sample before carrying out a variable temperature experiment. Never rapidly heat or cool a sample. Always wear safety glasses near the magnet when adding your sample to the sample changer.

Please take care while putting a sample in a spinner. Always insert your NMR tube with your fingers close to the spinner to avoid any excessive constrain on the glass tube.

Sample tubes must be stored carefully and not subjected to long heating as it will cause it to warp it. Always replace periodically (every 6 months or so) as tubes deteriorate with time.

To submit a sample:

- Do wear protective glasses when you are close to the sample tray.
- Do always follow the "*Sample Submitting Procedure*".
- Always submit a clear homogeneous solution sample as it will affect the shims.
- The solution (0.6 mL to 0.7 mL) in the tube should measure between 4 cm to 5 cm as it will affect the shims. Heavy solution may cause issue during inserting/ejecting sample from magnet.
- DO NOT SUBMIT any sample which does not hold properly to the spinner.
- DO NOT SUBMIT any sample which has been broken or fractured as the robot will likely break it.
- DO CHECK the sample length on the tray before submitting a sample as it will block the air flow inside the probe and potentially destroy it.

Care with Spinners:

Please take care while putting a sample in a spinner. Be very careful with sample tubes as they are fragile and break easily. If you break the tube, you may injure yourself and possibly contaminate your wound with chemicals. Please seek help from a first-aider if this happens. Please take care with the spinners, as they are precision-milled and are very expensive. If you accidentally drop one, inform one of the NMR staff, otherwise further use of this spinner may affect your or a colleague's data.

If a Sample Tube Breaks inside or outside the Magnet:

If a sample tube breaks in the NMR magnet, please stop the robot by pressing the red emergency bottom and **inform the NMR staff immediately**. The tube must be removed as soon as possible by trained staff in a safe manner to avoid damage to the probe or the robot, which are now irreplaceable (the Agilent NMR section closed in October 2014). The NMR staff understand that accidents can happen but **at least 1 week suspension** will apply to users for each incident.

If a sample breaks while putting it in a spinner, do not clean the spinner yourself but give it to the NMR staff. Each spinner costs around 100 euros each and solvents will deteriorate them. Please take any necessary precautions in case of contamination.

Sample Tube Storage:

Sample tubes must be stored carefully, and not subjected to long heating/drying as this will warp the tube (glass is a super-cooled liquid!) potentially causing damage to the NMR spectrometer probe. NMR tubes must be replaced periodically (every six months or so) as they do deteriorate with usage. The quality of the spectrum is dependent on the condition of the tube.

Cryogens:

Liquid nitrogen and liquid helium are routinely used within the NMR laboratories to keep the magnets at superconducting temperatures.

When the magnets are being filled with helium, no-one other than trained NMR staffs or technical staff may enter the room (*2 yellow signs* at the front door), as there are risks of cryogenic burns and/or asphyxiation.

When the magnets are being filled with nitrogen, NMR users may enter the room (*1 yellow sign* at the front door) but they must evacuate the room immediately in case of any unexpected events.

Please read the SOP associated with the risks of cryogens available at the NMR laboratories.

NMR software:

You need to register to the LAN network of your laboratory before asking NMR staff for access to the NMR server or to install MestreNova software.

MestreNova:

The NMR Centre has a server licence allowing access to this software to all PIs, PhDs, Postdocs and Stage 4 undergrad project students from the School of Chemistry.

Access to the NMR server:

The NMR data is transferred to a server regularly over the day. Data can be accessed by any NMR users from the School of Chemistry.

Data Storage and Retention:

By default, the NMR data can be viewed and downloaded non-encrypted from the NMR server by any users from the same research group except otherwise advised by the PI. The collected data is guaranteed on the NMR server for 1 month only so user/ PI should make their own copy of NMR research data. The retention of the NMR data on the servers has no expiry date except advised by the PI.

According to UCD policy, the Principal Investigator is the custodian of the research data and is responsible for its management, including security, storage and retention. The Principal Investigator, who must determine and control access rights to research data, is also responsible for informing research participants of their obligations in relation to the data.