

NMR Sample Preparation

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NMR Tubes

Tube Length

- **The sample changer uses only 8-inch long tubes.** Shorter tubes, such as 7-inch tubes are not allowed and can break.
- Do not use tubes with chipped or cracked tops.
- Tubes with stopcocks cannot be used on the sample changer and must be approved before using on the other spectrometers.

Care of tube and cleaning

- Do not heat NMR tubes in ovens. The tubes will warp, bend and flatten. This destroys the value of the tube.
- **Bent tubes may cause severe probe damage.**
- Clean with detergent, wash with acetone or ethanol, air dry. Blow nitrogen or air through the tube.
- To remove moisture, leave tubes under vacuum.
- Replace tube caps; do not use split or broken caps. Dirty caps will contaminate your sample.
- Have the top of chipped tubes cut off by the glassblower and flamed.

Preparing your sample

- Use about 700 μL of deuterated solvent (for a 5-mm tube).
- If necessary, filter your sample using glass wool or a kimwipe in the end of a pipette. Wash glass wool with a small amount of deuterated solvent to remove glass particles.
- The height of liquid should be about 4.5 cm.
- Dilute samples do not benefit from a short liquid length.
- Label your tube (on the cap or topmost 2 cm of the tube).
- NMR tube caps must be on tightly to prevent evaporation of solvent while waiting in the queue.
- Do not attach paper or plastic labels. They may interfere with the gripping fingers or fall off in the magnet.
- Paramagnetic compounds have broad peaks or missing signals and NMR spectra may not be obtainable.
- Typical concentrations are: 10-mg ($\text{H}1$)/50 mg ($\text{C}13$) for 300 MHz, 5 mg ($\text{H}1$) 20 mg ($\text{C}13$) for 400 MHz.

Magnetic field cautions

NMR uses high field magnets. Although most of the magnetic field is contained inside the magnet, there is still significant stray field.

- Uses with pacemakers, prosthetics, insulin injectors or other medical devices must **consult a physician before entering the NMR labs.**
- Magnetic field warnings are marked on the floors, keep all metal objects outside the 10G lines.
- The magnetic field increases exponentially and can cause unexpected or sudden pulling of ferrous objects. Personal injury or magnet quench or damage may result.
- Credit cards and mechanical watches may be damaged by the magnetic fields at the 5 G line.

Preparing your NMR tube

- All NMR tubes must be cleaned with isopropanol and a kimwipe **prior** to being inserted in spinner.
- Before inserting the NMR tube into the spinner, clean the tube from the bottom to 2 cm from the top.
- Hold the spinner loosely with two fingers on the black stripe.
- Insert the tube while rotating the tube to make the insertion easier.
- Insert the tube about 2 cm.
- Do not force the tube; breaking the tube may cause severe cuts to your fingers.
- Do not put the spinner into the depth gauge before inserting the tube into the spinner. It is very easy to break off the bottom of the tube if the spinner is held too rigidly.

- Put the tube and spinner into the depth gauge and push the tube to the bottom.

Broken tube in spinner

If you break a tube while inserting into the spinner:

- Clean the spinner with isopropanol. *Use no other solvent!*
- Wear gloves if necessary
- Do not wipe the black strip or white dots
- Use a cotton swab to clean the inside of the spinner. Check that there is no glass inside the spinner!
- Check that the o-ring is not damaged.
- Leave the spinner in the white bin. Do not re-use it on the sample changer or put it where it will be used again.
- Fill out a broken sample report and return it to staff as soon as possible.

Walkup or Sample Changer

If you are using a system with a sample changer, follow the procedure below, otherwise skip to **Walkup NMR**.

Sample changer rack

When you are ready to put your sample on the sample changer rack:

- Look at the login screen for the next available location.
- The location numbers are shown to the **right** of the location.
- Put your sample at the desired location.
- There are two parts to the rack: Locations 1-50 are on the right and locations 51-100 are on the left.

Sample rack cautions

- The sample changer is running in automation all the time. The robot fingers have force feedback but may cause personal injury or break samples.
- The robot arm may move quickly and unexpectedly—watch out when samples are being changed.
- Never put anything under the robot fingers; there is always a sample in the magnet and that sample is returned to the rack under the robot fingers.
- Do not put empty spinner on the rack. Put them in the empty spinner tray.
- Do not remove spinners from the lab for move them between the spectrometers.
- There is an emergency stop button that stops the robot immediately. Press emergency stop if:
 - There is a broken sample on the sample changer rack.
 - Samples have collided or about to collide.
 - The robot is about to hit a person.

Walkup NMR

When using the NMR without a sample changer:

- Log into the Sun computer.
- Turn on the eject air. By clicking the Eject button on the Start tab.
- Do not proceed if the eject air is not on.
- The sample should be in a spinner (see above).
- Walk up the ladder and put the spinner and sample put it on the top of the magnet.
- Do not lean on or touch the magnet.
- The sample must be floating on the eject air at the point.
- On the computer, click the Insert button.
- Never put an empty spinner in the magnet.

Booking schedule

- Reserve time on the booking schedule. Do this even at nights and on weekends.
- Always cancel time that will not be used.
- Walk-on times are for quick spectra - a maximum of 10 minutes or 2 samples if others are waiting.
- The NMR schedule refreshes every 10 seconds so all users can easily see when the spectrometers are free.
- The URL for the NMR booking schedule is <<http://nmrweb.chem.utoronto.ca/>>.
- To access the booking schedule on the spectrometers or NMR workstations, type in “sched”.

Dirty spinner

Sometimes the spinner is dirty due to mishandling. This affects the spinning of the sample.

- Clean the lower part of the spinner with isopropanol.
- Do not wipe the white dots or black stripe.

Samples left in the NMR labs

- Samples left in the NMR lab will be moved into the fumehood in LM43.
- On Mondays, samples will be moved to the “OVERFLOW NMR TUBES” bin.
- After one week these tubes will be removed and given away.

NMR computers

- All authorized NMR users will have valid usernames and passwords.
- You must keep your password confidential.
- Unauthorized use of computers is a federal crime under section 342.1 of the Canadian Criminal Code and against the University Student Code of Conduct. Sanctions, fines or charges may result from unauthorized use or mischief.
- NMR spectrometers are not to be used for private consulting or NMR time sold to others.

NMR doors

- Doors are to be kept locked after hours, on weekends and holidays.
- Keys are available, *per group*, for LM49 and LM51.
- LM43 is unlocked by any key for the building.

Spectrometer problems

Always inform staff of problems with spectrometers, such as:

- Samples not locking or shimming.
- Spectra blank (no signal).
- Automatic tuning not working.
- Broken samples or unknown material on the spinners .