

NMR Sample Preparation

NMR tubes are thin-walled glass tubes and are fragile. Although they are used daily, personal injury can result when fixing the cap to the tube and inserting the tube into the spinner or turbine.

When handling your NMR tube we recommend:

- DO use your fingers, instead your palm when inserting an NMR tube into a spinner and/or depth gauge.
- DO NOT use excessive force to fix a cap to a NMR tube.
- DO dispose of 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 put an empty spinner into the spectrometer. They are impossible to retrieve.**
- DO remember to TURN ON (press the eject button) the lift air before inserting your sample in the magnet. Neglecting to do so can break your sample and damage the instrument.

Be aware of the chemical hazard of your sample prior to entering the NMR Facility. **If a sample is broken within the magnet, the NMR staff must be informed immediately!!!** This should be done both by phone and email. It is imperative that no one uses the instrument until after Dr. Lee has assessed the situation. The student should place a “broken sample” note on the keyboard and lock the computer screen.

Good NMR tubes generally give better spectra, especially at higher field. A good NMR tube costs about 2 hours worth of NMR time, but you can easily recover the investment from better signal to noise and faster shimming. We recommend the precision grade NMR tubes that are rated for 600 MHz NMR, such as the Wilmad (535-PP-7), Norell (509-UP-7 or S-5-600-7), or NewEra (NE-UP5-7). For limited sample quantity, the Shigemi NMR tubes are preferred.

Type	Diameter	Volume
Standard	5 mm	700 μ L
Standard	3 mm	175 μ L
Shigemi	5 mm	260 μ L (300 μ L for the inexperience user)
Shigemi	4 mm	155 μ L
Shigemi	4 mm	220 μ L (no plunger, for viscous solution)
Shigemi	3 mm	100 μ L

For highest spectrum quality with the least effort: make samples full length (700 μ L) in a high quality tube.

For highest mass sensitivity: use a Shigemi tube.