

NMR Sample Preparation & Handling

- 1) Use clean and dry NMR tubes. Please clean the outside of *EACH and EVERY* NMR tube by wiping with ethanol or other solvent BEFORE you bring it into the NMR lab. This is critical to prevent contamination of the probes.
 - a. NMR tubes that have been in glove boxes are frequently contaminated with an oily residue. Please be extra thorough in cleaning these.
 - b. Never place NMR tubes in an oil bath.
 - c. **DO NOT** write on NMR sample tubes. The ink from markers accumulates in/on spinners, and eventually into the probe. When this happens, the probe needs to be removed and cleaned, leading to instrument down time. If you want to label your samples, please use tube labels or write on the caps.
- 2) Use only high-grade NMR tubes. Those made out of Pyrex are usually quite good (ASTM Type 1 Class A). You can buy these from ScienceStores or directly from Wilmad, Norell, or New Era. Some examples include:
 - a. Wilmad "Precision" Series: 507-PP-7, 528-pp-7, 535-PP-7
 - b. Norell "Select" Series: S-5-200-7 or S-5-600-7
 - c. New Era: NE-HP5-7, NE-UP5-7
- 3) Standard NMR tubes are 7 inches long. The maximum allowable tube length is 8 inches.
 - a. J. Young tubes that are commonly used in the department are too long!!!
 - b. Be aware that ScienceStores stocks a 9" NMR tube. These are designed for flame-sealing. Please **DO NOT** use these on the Bruker systems, as they will not fill in the sample changer.
- 4) When possible, we recommend you use make the column of liquid in the NMR tube comparable to the size of the radiofrequency (*rf*) coil of the probe. Excessive volume dilutes the sample, reducing the sensitivity. Using 1 mL of solvent when you could use 0.5 mL results in 4x the number of scans during signal averaging!

<u>Instrument</u>	<u>Recommended Sample Volume</u>
Bruker AV-600 or AV-600	0.5 mL (40 mm liquid height)
Varian Inova-500, VNMRS-500	0.6 mL (45 mm liquid height)

When shorter samples are required, make sure to center the sample around the line marking the center of the *rf*-coils when using the depth gauge.

- 5) Use deuterated solvent. If you need to use protonated solvent, please see your group operator or one of the NMR facility staff for assistance in setting up the experiment.
- 6) Filter your sample to remove any particles from the solution. This can make substantial differences to the resolution obtainable.
- 7) Always transport your samples in secondary containment with a top and bottom half! This is an EH&S policy, please follow it! We provide suitable containers if you do not have one. Don't use tippy scintillation vials or small flasks.
- 8) Slow down and work carefully to avoid sample accidents. NMR sample holders ("spinners") are *very* expensive and easily damaged if dropped.
- 9) **DO NOT SPIN** samples on any of the spectrometers in the NMR facility.

Contact Information

Nanette Jarenwattananon, NMR Facility Director
Onyx 386 | njaren@uoregon.edu | (541) 346-4605
nmr.uoregon.edu