

## Sample Handling Procedures for Bruker-Avance III-HD 600 MHz NMR

- Clean the outside of **Each-And-Every** NMR tube by wiping with **ethanol** or other solvent **BEFORE** bringing to the NMR lab. *This is critical to prevent contamination of the NMR probe.*

Tubes that have been handled in glove boxes often are contaminated with an oily residue.  
Never place NMR tubes in an oil bath.

- Maximum overall tube length is **8 inches**.

**J-Young** tubes in common use in the Department are **TOO LONG!!**  
See Mike Strain for specifications.

Beware of the 9" long tubes stocked by ScienceStores: the extra length is meant for flame-sealing.  
Do not use these for routine NMR. They will not fit the Bruker Sample Changer.

- Use only high grade NMR tubes made of **Pyrex** (ASTM Type 1 Class A), e.g.:

Wilmad "Precision" series: 507-PP-7, 528-PP-7, 535-PP-7  
Norell "Select" series: S-5-200-7 or S-5-600-7.

Defective tubes (cracks, chips, scoring, scratches, etching, warped, etc.) are **prohibited**.

- Recommended sample volumes:

600 MHz	0.5 to 0.6 ml	4 cm liquid height
500 MHz	0.6 to 0.7 ml	4.5 cm liquid height
300 MHz	0.7 ml	5 cm liquid height

- Excessive volume unnecessarily dilutes the sample, reducing sensitivity for low-abundance nuclides such as  $^{13}\text{C}$  and is a waste of expensive deuterated solvent.

Dilution by 1.4x doubles the number of scans required when signal averaging.

- Please, no NMR samples or chemicals on the console workstation desk!
- Lab gloves are NOT permitted in the NMR lab  
or, in general, when operating instruments, telephones, door knobs, etc
- Please take your samples with you when you leave the NMR lab.
- Don't use tippy scintillation vials or small flasks to hold NMR tubes.
- Use secondary containment (e.g. plastic bottle) for carrying tubes containing hazardous or toxic materials.
- **Slow down** and work carefully to avoid sample accidents. Don't drop samples on the floor... they break!  
*NMR sample holders ("spinners") are precision-machined (read EXPENSIVE) and will be damaged if dropped on the floor.*

### Sample Changer:

- Use sample changer in manual, push-button mode at magnet base.

*We will develop procedures for automated multiple samples later on.*

- Leave the magnet **empty** at the end of your session, i.e. don't re-insert the standard (*applies only to 600*)