

## Sample preparation and positioning

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The sample quality can have a significant impact on quality of the NMR spectrum. The following is a brief list of suggestions to ensure high sample quality.

### Sample preparation

- Always use deuterated solvents (except if there is any valid reason not to use).
- For  $^1\text{H}$ , **0.5 - 5.0 mg** of the sample is sufficient. For high molecular weight samples, more concentrated solutions are sometimes recommended. However, too concentrated solution leads to lower resolution due to saturation and/or increased viscosity. For  $^{13}\text{C}$  and other nuclei ca. five times the concentration of  $^1\text{H}$  is recommended (**20-100 mg**).
- For 2D measurements, the sample should be concentrated enough to achieve an acceptable S/N ratio within reasonable time period. As a rule of thumb, **15-25 mg** of the sample is sufficient for homo- and heteronuclear 2D NMR measurement. However, with small sample quantity  $^1\text{H}$ - $^1\text{H}$  homonuclear experiments (e.g. COSY, TOCSY, etc.) are still feasible, but those involving X nuclei (e.g.  $^{13}\text{C}$ ,  $^{15}\text{N}$ ,  $^{29}\text{Si}$ , etc.) may take overnight to complete.
- Always use the same sample volume or solution height. This minimizes the shimming that needs to be done between sample changes. The recommended volume is (at least) **0.6 mL or 4 cm solution for 5 mm sample tube** and **4 mL or 4 cm solution for 10 mm sample tube**.
- Always use clean and dry sample tubes to avoid contamination of the sample. **Brand new NMR tubes straight from the packet are not usually very clean!**
- Always use high quality sample tubes to avoid difficulties with shimming and spinning during both room and variable temperature measurement. It also damages the inside coil as well as glass insert inside the probe.

Some of the recommended tubes to be used in NMR Facility are as follows:

NMR Field Strength	Wilmad	Norell	New Era
600 MHz	535-PP/528-PP	509-UP	NE-UP5
500 MHz	528-PP/527-PP	508-UP	NE-HP5
300/360/400 MHz	527-PP/507-PP	507-HP/506-P	NE-HL5/NE-HP5

\*all tubes should be preferably of **7 inch** length

**Note:** You will lose your access to NMR spectrometer if found using cheap quality NMR tubes.

### THE CONSEQUENCES with poor quality tubes (adopted from New Era Catalog)

A poor quality tube precesses within the magnetic field presenting an inhomogeneous, unstable sample to your NMR probe.

- Shimming will be time consuming, if not impossible.
- Time averaged data collection will be inefficient resulting in lost sensitivity and productivity.
- Line broadening may obscure important minor spectral components.
- Early time points in kinetic studies may be missed with loss of meaningful peaks.
- Spinning side bands may produce spectral artifacts leading to wrong conclusions.
- Delayed research and missed instrument time slots.
- Poor spinning can cause costly damage to probe and solenoids.
- Possible loss of valuable samples.

- Always filter the sample solution. A small plug of fresh medical cotton wool at the neck of a Pasteur pipette will do the trick. However, it is necessary to pre-rinse it with a little amount of the solvent to be used to flush out any loose fibers.

### Sample positioning before inserting into the magnet

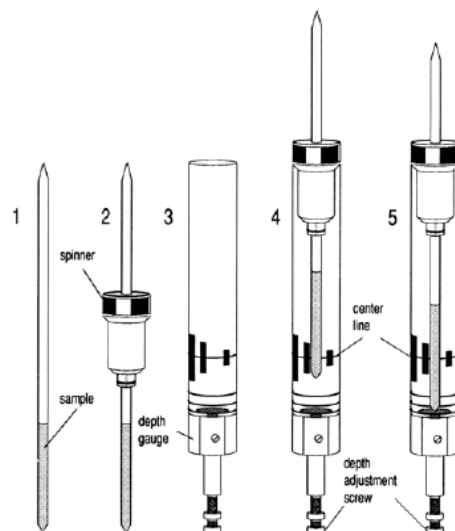
- Use the sample depth gauge to position the sample tube in the spinner. Be careful with the depth gauge. **Do not change the depth!** The slider should be in position 5-15 mm.
- Always wipe the NMR tube with Kimwipe before inserting it into the probe.



base plate). **Remove the depth gauge before inserting the sample and spinner into the magnet!**

- Sample tube depth adjustment: First, seat the spinner on top of the depth gauge. Second, carefully push the sample tube through the spinner holding the tube from the middle (not from top) until the bottom just touches the top of the slider (white

- For experiments using sample spinning, be sure that the spinner, especially the reflectors, is clean. This is important for maintaining the correct spinning rate. **Do not touch the reflector part of the spinner with your bare hands!**
- Check that the sample tube is tightly held in the spinner so that it does not slip during inserting the sample into the magnet.



### 5.3 Sample Tube Maintenance

NMR tubes should not be treated as standard laboratory glassware and it is necessary to handle these tubes carefully to maintain their quality for repetitive uses.

In general, empty NMR tubes may be soaked in a mild cleaning solution and rinsed with deionized water (never use any acid solutions to clean NMR tubes). Follow this with a rinse of clean methanol or acetone and air dry afterwards\*.

\*If moisture and undried methanol/acetone presents problem, the tubes may be placed in a vacuum oven on a very flat surface (never standing and only in one layer). Heating should not exceed 100 °C and one hour duration (never leave tubes overnight in an oven).

Dried tubes should be capped and stored in a desiccator.

**Note: If tubes cannot be clean by above procedures, then it should be discarded.**