NMR SAMPLE PREP 101

NMR TUBES

"Crap in; Crap out" - A Wise NMR Manager

Quality spectra start at your NMR tubes. Not all companies will sell quality NMR tubes. The top 3 companies are as follows: **Wilmad Glass**, **Shigemi Inc**, and **Norell**.

Wilmad glass is the preferred company among NMR staff and faculty. They are sold by Fisher Scientific (its on your P.I.'s EZ-Buy) which makes ordering easier.

BUT WAIT! THERE'S MORE!

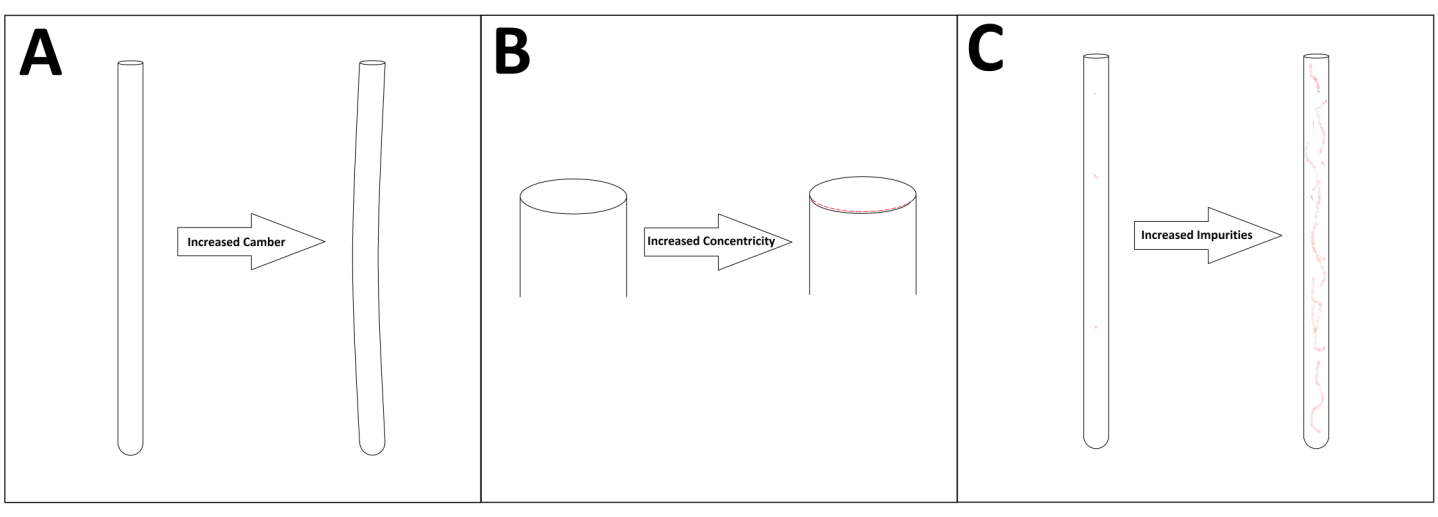
Each supplier has a plethora of tubes that you can purchase. They are usually broken down into 3 categories: High Throughput, Economy, and Precision. On each tube you also have a selection of length: 7", 8", or 9". For our auto sampler stick to 7" or 8" tubes (unless you need to seal a tube). A high quality tube not only gives better spectra but avoids damaging the probe.

Tube Type (Item Number)	Box	Application	Price per Unit (7" tube price shown)*
High Throughput (WG-1000-7-SJ)		300 MHz or Trash	Don't Buy
Wilmad Economy (WG-1235-7)		Basic 500 MHz Work Checking ^{1}H, ^{13}C No variable temp	$3.78
Wilmad Precision (528-PP-7)		High Resolution Work 2D NMR Variable Temperature Solvent Suppression	$8.58

*From Fisher Chemical

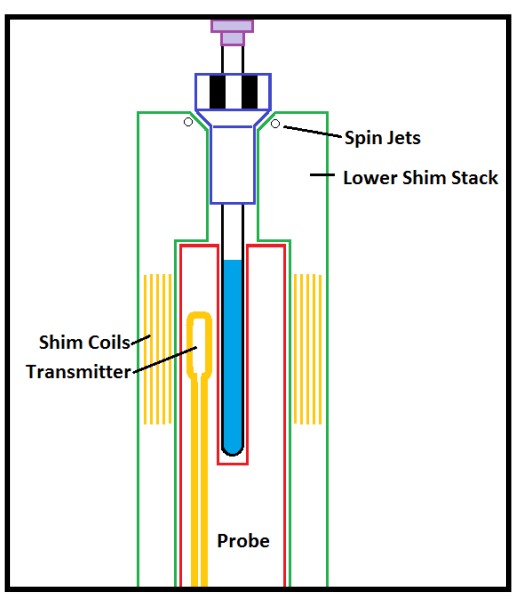
What's the Difference?

Three main things affect tube quality: Camber, Concentricity, and glass rating. Camber refers to the bend in a tube (A). Concentricity refers to deviation in the radius (B). Glass rating refers to the amount of impurities (C). All of these factors can lead to poor shim quality which leads to low resolution and possible probe damage.



Solvent Amounts

In general, for the best line shape your solvent fill needs to be 3x the rf coil length. On newer Bruker probes this ends up being ~54 mm of solvent length with a standard thin walled tube. (~0.7 mL). This is so the shim coils do not see an abrupt change in the magnetic susceptibility field that they would have to compensate for. You are able to get a signal without this volume, however shimming can take longer, give broader peaks, or completely fail.



Not Enough Sample?

When only low amounts of sample are available and ~0.7 mL would make too dilute of a sample for reasonable acquisition, specialty tubes and items can be used to obtain quality spectra without large amounts of instrument time.

Recommended specialty tubes and times include the following:

A. Heavy Walled Precision Tubes (Wilmad: 522-PP-7)

B. Micro Bottom Tubes (Shigemi: SP501—SP505)

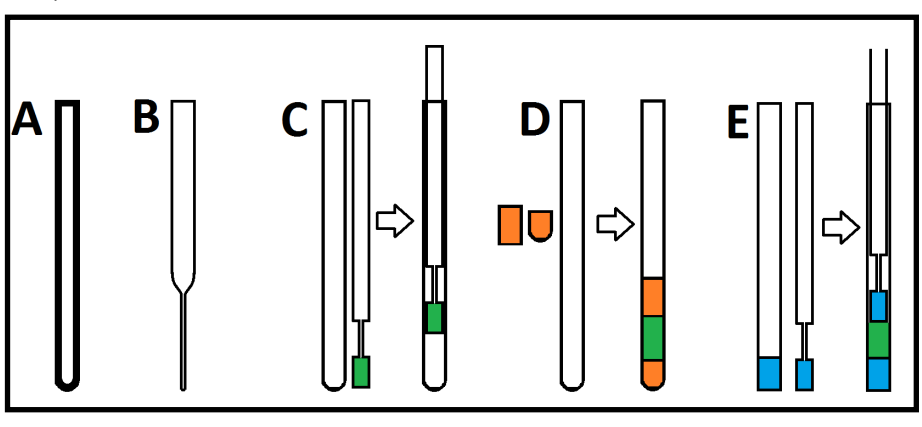
C. Microcell Inserts (Wilmad or Shigemi)

D. Doty Plugs (Wilmad or Shigemi)

E. Shigemi Tubes (Wilmad or Shigemi)

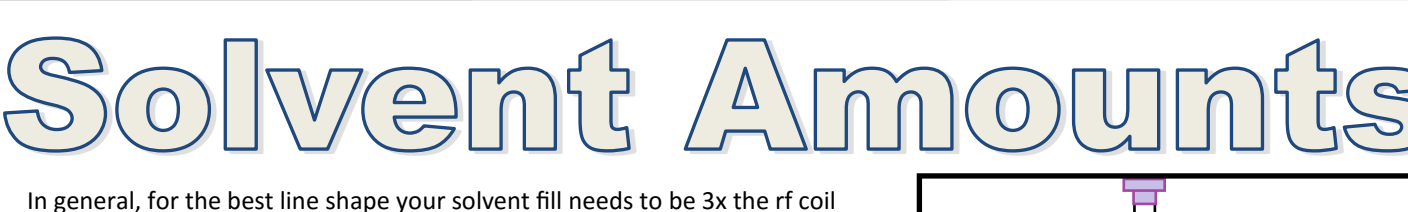
Each tube has variable options to choose from for the best application including volume/mm, magnetic susceptibility matching properties, and overall volume.

Consult Dr. Simons for more information.



Green—Sample; Orange—Doty plugs; Blue—Magnetic susceptibility matched glass

NMR CAPS



Things to know about traditional caps:

THEY ARE DISPOSABLE

Any kind of wear, stretching, chemical damage seen on or in cap should call for its immediate toss. Cap = $0.10, your sample = more.

THEY ARE NOT COMATABLE WITH ACETONE OR CHLOROFORM

Do not have the tubes come into contact with either of these solvents unless you want small peaks of the caps in your NMR. A good habit to get into if you must use the disposable caps (better caps are sold) is to use a small piece of Teflon tape on the top of your tube then place the cap over. Even with this technique do not store acetone or chloroform samples with disposable tubes.

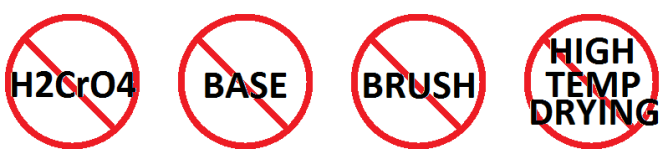
DO NOT INVERT YOUR TUBE TO MIX IT

Use small mixers or sonicators to attempt any mixing of sample.

ALTERNATIVES TO STANDARD DISPOSABLES

- Rubber NMR Septa (WG-3891 or WG-3892)
  - Can be used with air sensitive compounds
- PTFE NMR Caps (WG-1264)
  - Use with precision tubes only
  - Compatible with chloroform and acetone
- Norloc™ Security Caps
  - Chloroform compatible

Cleaning & Storing NMR Tubes



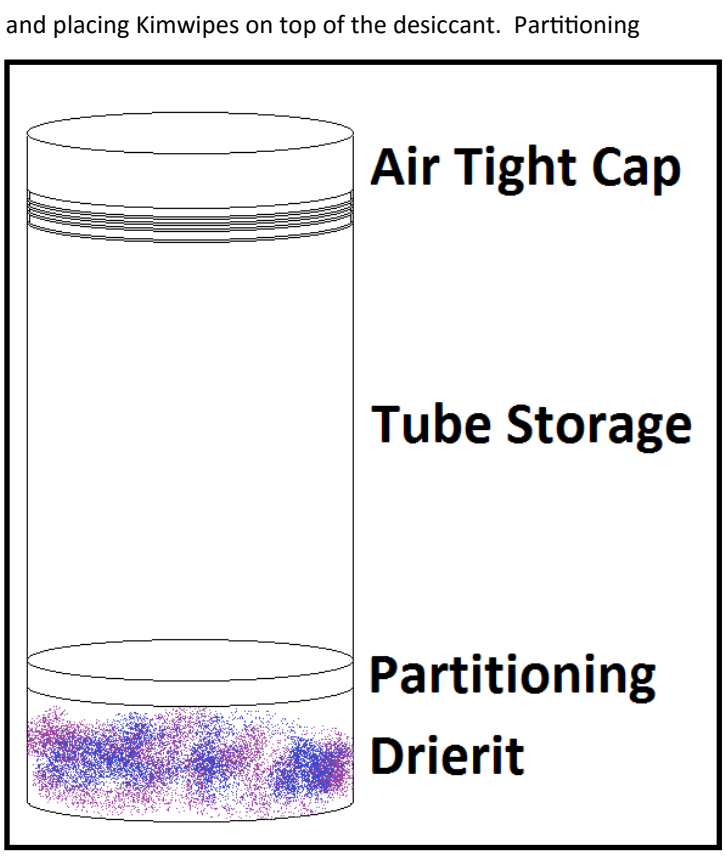
After running a sample it is imperative you clean your NMR tube and not let the tube sit for extended periods of time. Recover your sample and perform a rinse of the tube with the non deuterated solvent that was contained. Combine this rinse with your sample.

Rinse several times with acetone or methanol. Avoid using water if possible. If you do use water, follow the rinse with several of acetone Do not use mini glassware brushes. These can scratch the glass and ruin the concentricity of the tube.

For tough to clean tubes, soak them in HNO_3 being sure to remove any air bubbles. If cleaning out synthetic polymers try using a solvent that will swell the polymer and follow with a pipe cleaner to try and remove the material.

Dry using a dry nitrogen line or by using a low temperature oven if available (~60 °C). High temperatures can cause slight tube distortions (not seen to the naked eye) which will effect camber.

Tube storage should be done in a non heated environment, preferably with a desiccant to keep tubes dry. I recommend buying spaghetti storage jars, placing a desiccant at the bottom and placing Kimwipes on top of the desiccant. Partitioning



Miscellaneous

Specialty Tubes

Various specialty tubes are sold for a variety of applications:

Boron NMR & Photochemistry	528-PP-7QTZ
Silicon NMR & Corrosive Samples	PTFE-5MM-Kit
UV sensitive NMR	528-PP-7AMB
Reactive Internal Standard	WGS-5BL

Tube Transport

Be sure to transport your samples in a secondary container to and from the NMR laboratory.

Z567078 ALDRICH

Z286095 ALDRICH

Solid Particulates

For best shimming and signal, filter out all solids before inserting sample into the NMR tube. Use a Pasteur pipet with a small bit of cotton to filter the dissolved compound.

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