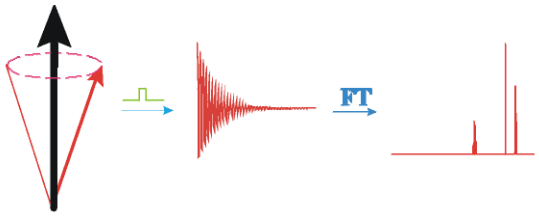

ATLANTIC REGION MAGNETIC RESONANCE CENTRE

Sample Tubes in NMR Spectroscopy

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September, 2002

Those of you who have asked me to run high-resolution NMR spectra on the AMX-400 will be aware that I insist on an NMR tube of an appropriate quality before I'll put it in the magnet. When ARMRC becomes host to a state-of-the-art 500 MHz NMR instrument this winter, an even higher quality tube requirement will be put in place and strictly enforced. Many of the hands-on users of the NMR spectrometers in ARMRC have told me that they were not even aware NMR tubes came in different quality flavours and considered them simply another piece of lab glassware. The purpose of this note is to dispel this popular misconception and explain exactly what you get when you purchase a high quality NMR tube.

Before I can do that, however, I need to first introduce the term magnetic susceptibility (MS). In very general terms, the MS of an NMR sample describes how it becomes "magnetized" when placed within an external magnetic field. Even if the external magnetic field is perfectly homogeneous, placing a body (such as an NMR sample) with a finite MS within the field can destroy the homogeneity and therefore the resolution in your NMR spectrum. As it turns out, the shape of the sample plays a crucial role in how the external field is modified by MS effects. An ellipsoid is the only shape, which will produce a homogeneous field internally in your NMR sample when the external field is homogeneous.[1] A sphere and a cylinder of infinite length are two types of ellipsoids, known as ellipsoids of rotation. Thus, the task of an NMR tube is to hold the liquid sample in the shape of a perfect cylinder; the more expensive tubes do a better job of this. Your job when preparing the sample is firstly to completely dissolve the material in the appropriate solvent so that the MS is homogeneous throughout and then secondly to add enough solvent so that the cylinder of liquid looks to be infinitely long to the receiver coil.

That being said, the specifications on NMR tubes relate to how closely the internal volume of the tube approximates a perfect cylinder. These specifications are typically:

Camber - A measure of the lack of straightness of an NMR tube. It is measured as a deflection at the middle of the tube when it is held at both ends and rotated. Poor camber in an NMR tube can lead to "wobbly" spinning, producing spinning sidebands. **Extremely poor camber can lead to probe damage**!

Concentricity – The maximum variation in the wall thickness of an NMR tube. Many documents describe this parameter as the degree to which the two cylinders defined by the inner diameter and outer diameter run parallel to one another. Poor concentricity causes a portion of the sample to lie outside the "perfect cylinder" region, leading to difficulties in shimming.

Roundness – Symmetry around the centre axis of the tube. NMR tube companies often characterize this as a tolerance by measuring the inner and outer diameters for a large number of points along the length of the tube.

Specifications for three types of common Wilmad tubes (in stock at ARMRC) are provided in the following table:

Tube Type	~ Price (CA $)	Wall Thickness (mm)	Camber (inches)	Concentricity (inches)
507-PP (300 MHz)	$14 each	0.38	0.001	0.002
528-PP (400 MHz)	$21 each	0.38	0.0005	0.001
535-PP (500 MHz)	$29 each	0.38	0.00025	0.0005

Stronger magnetic field strengths enhance the distortions/flaws in NMR tubes. In other words, a sample in a cheap tube might be acceptable with a bit of shimming on the AC-250. However, the same tube placed in the new 500 MHz instrument could produce drastically poor results, which can't be compensated for with shimming. This is why a 500 MHz quality tube (535-PP) requires stricter tolerances than a 400 MHz tube (528-PP) and so on.

As ARMRC users begin using high quality tubes routinely, proper care and cleaning will be of utmost importance! For example, a definite "no-no" is to wash a 500 MHz tube and then stand it vertically in a hot oven to dry until its next use. High quality NMR tubes should be washed appropriately and then laid flat to air dry. Wilmad Glass has an excellent article on their website regarding NMR tube care and I encourage everyone to read it. (http://www.wilmad.com/NMR010.html)

In closing, I realize for a very long time now researchers have been using whatever NMR tubes they could find to run samples in the AC-250 and the older CW instruments. The take-home point here is that this trend **must** change once the 500 arrives; only 500 MHz quality tubes will be permitted. I encourage everyone to start thinking about taking an inventory of the NMR tubes in their labs and sorting them in terms of quality. Please come talk to me should you need any assistance with this.

[1] VanderHart, D.L., "Magnetic Susceptibility & High Resolution NMR of Liquids and Solids", in Encyclopedia of NMR, Vol. 5, Wiley, New York, 2938-2945.