

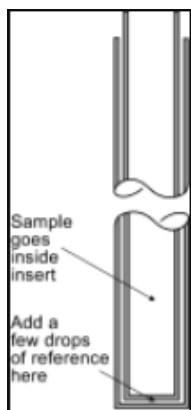
# Technical Information

## Coaxial Inserts in NMR Studies

NMR experiments that involve external reference/lock or that compare two solutions require two well defined sample volumes within the probe. WILMAD makes a variety of unique sampling systems that create such distinct sample volumes. This report will help you select the right insert or coaxial system for experiments where two volumes are needed. Refer to the table on the last page for a summary of the inserts WILMAD makes and what experiments each can be used for.

### Challenges in Coaxial NMR Sampling

In superconducting NMR spectrometers, cylindrical geometries work best in most applications requiring inserts. While spherical sample devices like WILMAD's 529-A series can be used for some comparisons, it can be difficult to maintain magnetic field homogeneity over the sample volume. It's easier to make highly precise cylindrical shapes in Pyrex than to blow precise spherical shapes, except for small sizes (spherical bulbs <5mm OD). Consider saving spherical sample inserts for microsampling applications. When determining magnetic susceptibilities, modulation sidebands caused by imperfect sample tubes can make precise measurements of chemical shifts impossible. Only highly skilled manufacturers can offer precision inserts of this type, so the number of suppliers of coaxial inserts is severely limited.



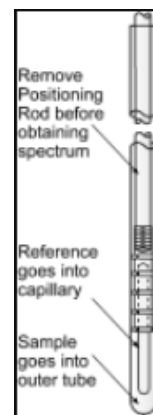
Generally, coaxial systems made only of Pyrex will provide a higher degree of precision than systems incorporating PTFE components. Because Pyrex is rigid while PTFE is soft and cold-flows, Pyrex can be made to tighter tolerances than PTFE (see precautions in Resonance Report NMR-010 about preserving NMR tube precision). If you're performing Variable Temperature (VT) experiments, all-Pyrex coaxial systems are preferred. With these systems, components have the same coefficient of expansion and you'll avoid breakage that can sometimes occur when you mix plastic and Pyrex under VT conditions.

### Inserts in External Referencing

External referencing has been replaced in recent years by assignment of chemical shifts calibrated to the deuterium signal of the spectrometer's lock channel. But when you have a sample that you shouldn't dissolve in a deuterated solvent, you'll need an external lock and a reference (internal is pre-ferred). However, when your samples are too precious to mix with a reference chemical that is difficult to remove or reacts with your sample, then external referencing can be employed.

In these rare circumstances, the external reference must be present only in sufficient quantity to produce a discernable signal in your spectrum. Generally, small amounts of the reference are needed if the signal produced by the reference lies away from the critical regions of the NMR spectrum of your sample. WILMAD Product Number 516 is a coaxial system that provides a tiny 50µm space between the insert and outer tube, into which a few drops of concentrated reference can be placed. Sets are available in sizes from 3 to 15mm OD. Filling factor is reduced with this system, so use it only when you aren't sample or solubility limited. If you need a larger reference volume, e.g. to combine an external reference with an external lock, follow the recommendations for external locking, below.

The 529 microsample system can also be used with samples studied in 5mm sample tubes. Place or seal a small volume (~16µl) of reference material into a 529-D capillary and carefully position in a 5mm tube using the PTFE Holder (529-B). Make certain the capillary is straight in the holder. The 529-D is ideal for sample or solubility-limited situations, since you get spectra with only a small reduction in filling factor.



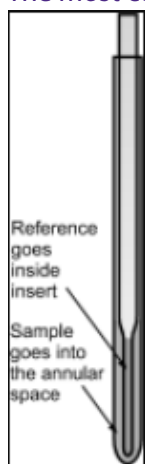
# Technical Information

## Coaxial Inserts in NMR Studies

### External Locking

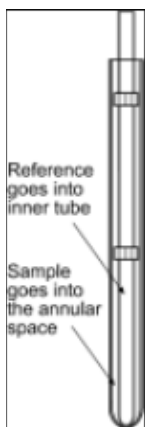
The lock channel of an NMR Spectrometer requires a significant signal to maintain Rf stability. Thus, coaxial systems like the 516 series should not be used for external locking. The spacing between the walls of these inserts and outer tubes (50 $\mu$ ) is not sufficient to support a spectrometer lock, even if 100% deuterated solvents are used.

The most commonly used coaxial insert, which will give the best lock signal, is WILMAD's WGS-XBL Series inserts (X = 4, 5, 8, 10, 12, 13, 15, 16, 18, or 20). These fit standard WILMAD outer tubes snugly, just like the 516, but each tapers to a smaller diameter stem in the lower 50mm. For the most common 5mm size, the stem is 2.5mm OD. The capacity of the stem, where reference material goes, varies in relative capacity from 5% (for large diameter outer tubes) to 16% of the sample volume. You place your sample in the annular space. Add just the amount of sample that will fill the annular space below the taper of the insert. For approximate volumes of the annular space of the outer tube (see WILMAD's NMR Catalog).



If you seal your lock/reference solution in the insert, you can switch the insert from one tube to another simply by rinsing and drying the outside of the insert. If you're performing VT studies of two solutions, then this is the ideal coaxial system. Special insert sizes can be made if the standard sizes aren't right for you.

WILMAD Product Numbers 517-519 can also be used for external referencing and magnetic susceptibility comparisons, too. Although confined to the 5mm OD tube size, these coaxial systems offer a way of varying the ratio between the reference and sample volumes without buying specially prepared products. And they're ideal for VT experiments, because the components are all made from Pyrex. It may be necessary to use a wire hook to position or remove the Pyrex spacers, which fit snugly into the outer 5mm tube. Avoid scratching the tubes or positioning the spacers in the Rf coil of the spectrometer probe.



The least costly approach to external referencing is WILMAD's PTFE Sleeve Adapter Sets. Useful for probes that take tubes between 8 and 20mm OD, these permit you to combine standard NMR tubes to make coaxial sets. Because of the machinability of PTFE, which is soft and easily cold-flows, these sets are of moderate precision. PTFE Adapters should not be used in VT studies. The lower adapter is easily positioned. It has notches on the outer edge that permit you to position it with a wire hook. Avoid scratching the tubes or positioning the spacers within or near the Rf coil of the probe. PTFE Sleeve Adapters for 5mm tubes are not available. Use WILMAD product numbers 517-519, above, instead.

### Measuring Magnetic Susceptibility

Because of the high degree of precision of WGS-XBL inserts, you'll get the best resolution in susceptibility comparisons using these coaxial inserts. The 517-519 Series of inserts also offers the degree of precision and filling-factor needed for these critical experiments, but WGS-XBL is easier to use. We recommend that WGS-XBL inserts be used for all magnetic susceptibility studies, unless there is an overwhelming reason to avoid them. A reference solution can be sealed into a WGS-XBL insert that's ordered 50mm longer with a constriction. Inquire about prices.

Product Number	Description	Sizes	Experiments
516	Coaxial Insert	3-15mm OD	External Reference
517-519	Coaxial Insert	5mm OD <sup>1</sup>	Microsamples, External Reference, External Lock, Susceptibility Measurements
529-A Systems	Spherical Bulbs	5 - 20mm OD	Microsamples
529-D Systems	Capillary Tube	5mm OD	Microsamples, External Reference
529-E Systems	Cylindrical Bulbs	5 - 20mm	Microsamples
WG-1132-1 to WG-1132-32	PTFE Sleeve Adapters	8 - 20mm OD	External Reference, External Lock
WGS-XBL	Stem Coaxial Inserts	4 - 20mm OD	Microsamples, External Reference, External Lock, Susceptibility Measurements