Technical Information

Pressure Performance of NMR & EPR Sample Tubes

The application of NMR and EPR techniques to investigations in catalysis and the study of reactions involving gas and liquid phase components has renewed interest in the pressure performance of glass NMR and EPR sample vessels.

WILMAD glass sample devices should not be used in pressure applications before the hazards involved are fully understood. WILMAD does not guarantee the pressure performance of any of its glass sample devices for NMR and EPR spectroscopy. The nature of glass and the critical connection between the handling of sample tubes and their pressure performance makes it impossible for WILMAD to assure the performance of its tubes in these applications. Indeed, the application of glass vessels in pressure experiments is risky. WILMAD does not encourage the use of its glass sample devices in pressure experiments.

Glass Strength

However, there is an abundance of technical information from the literature and this technical bulletin was prepared to share some of this information with those preparing for such experiments.

Engineering formulas for cylinders not longer than 300mm predict the Pressure Maximum, P(max), of any rigid material is: P(max) = (Wall/O.D.) X K(t) where O.D. and Wall are the outside diameter and wall thickness of the tube, respectively, and K(t) is an empirical Tensile Strength of the material from which the cylinder is made.

Application Procedures

Thus, it is prudent in applying the engineering formula, above, to apply a safety factor to account for surface quality differences.

A Kt = 1,000 p.s.i. (70.3 kg/cm2) should be used for Wilmad Precision glass tube, and Kt = 2,500 p.s.i. (175.8 kg/cm2) for Wilmad Precision quartz tubing. While it has been reported that WILMAD glass sample tubes have been taken to pressures beyond those predicted by the formula, above, when using Kt = 10,000 p.s.i. (703 kg/cm2).

As many applications in NMR require the sample tube be rotated at frequencies approaching 25 Hz, it is important to test samples in a dynamic rather than just static mode outside the spectrometer prior to performing any NMR experiments. It is also important to protect yourself, those around you, and your equipment from injury or damage at all times when handling pressurized glass sample devices. If you suffer a catastrophic failure of a sample tube under pressure in a valuable spectrometer, you will not gain much research cooperation from others whose work relies upon the same NMR instrumentation. Proper shielding should be employed outside the spectrometer at all times when devices are pressurized.

Contact your safety officer or director prior to performing an experiments at elevated pressure to assure that all precautions prescribed by your organization are followed. WILMAD can't take responsibility for the results of failure of its glass sample devices at elevated pressure.

Although the Low Pressure/Vacuum (LPV) NMR Tube can be used at small to moderate pressures, WILMAD offers a tube with a more substantial closure in its Pressure Valve NMR Tube. This Pressure Valve (PV) NMR Tube, is made with a heavy-duty PTFE Angled Needle Valve Assembly atop a standard NMR tube. A modified flowmeter valve, the needle valve of these 'PV' tubes can be attached to a Swagelok 1/8" nut and ferrule or to accessories available for attachment to a vacuum rack. The valve can be closed by hand or using a small wrench. Samples must be limited to those which can pass through the 0.8mm orifice of the valve assembly. Axial symmetry is achieved through the design and preparation of the valve assembly. Although annealed, extreme caution should be applied when using PV NMR tubes, as noted above. Order the shortest tube possible to reduce the overall weight of the tube, which is substantial with the valve attached. Sample spinning or ejection can be difficult if your sample is too heavy.

Wilmad-LabGlass SP SCIENCEWARE™

1172 NW Blvd., Vineland, NJ 08360 USA
1-800-220-5171 • Email: cs@wilmad-labglass.com
www.wilmad-labglass.com