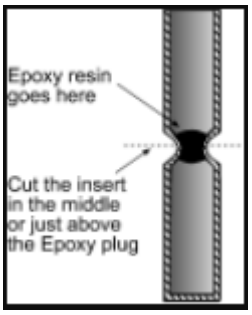
Technical Information

Sealable Pyrex Rotor Inserts for CP-MASS

Keeping samples sensitive to air or moisture from making contact with atmospheric oxygen or Investigations of solid-state samples using CP-MASS NMR techniques have become routine in many of today's NMR laboratories. Recently, much time and effort have been expended in enlarging the variety of samples which can be studied using these techniques. Included in this growing list are studies of air-sensitive samples, such as catalysts or organometallic compounds, and semi-solid samples, like gels or highly viscous liquids.

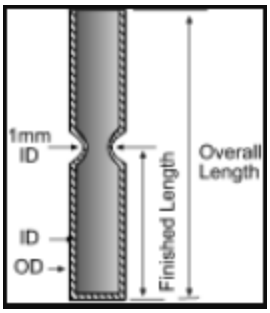
In the past, methods for preparing these types of samples for CP-MASS NMR study have required heat-sealing of the sample in a glass rotor or rotor insert 1,2. These methods, however, were not used without difficulty. Some of the problems involved avoiding axial dissymmetry in sealing the rotor or insert . . . the prevention of sample destruction . . . and the minimizing of solvent loss.



More recently, special rotors with O-ring seals have been made available by CP-MASS probe manufacturers for the study of air-sensitive or gelatinous samples. However convenient these special rotors are, they often come with a substantial price tag. Only if you are studying large numbers of such samples is this likely to be a cost-effective method.

A Simpler Alternative

An alternative, developed by scientists at Texaco, Inc. in close cooperation with WILMAD, makes it possible to avoid the problems in heat-sealing inserts and the high costs of special rotors. As described originally 3, this approach uses a small drop of epoxy applied to the constriction of a Pyrex rotor insert to seal the sample. One advantage of this sealing method is that it can be performed in the inert atmosphere of a glove box, so it's ideal for air-sensitive solids. It creates a reproducibly symmetrical seal, too.using this table. Powdered samples can be funneled and packed into the insert and gelatinous samples can be warmed and transferred into the insert as a fluid via syringe. After the epoxy is set and dry (10 minutes with quick setting epoxy), the sealed insert is cut through the constriction with a glass saw. In the original work, the sealed insert was placed in the lower portion of a Otsuka/Chemagnetics Kel-F rotor for 13C CP-MASS studies in an M-100S probe. Spinning rates in excess of 3KHz were reported with gelatinous polysaccharide samples.



Rotor inserts are now available for all major MASS probe types, including those from Bruker, Chemagnetics, Doty, and Varian. Product numbers and a description of the rotor into which each fits is provided in Rotor Insert Listings in WILMAD's NMR96 catalog online. Can't find your rotor in the list? Let us know. Chances are good that an insert can be prepared for it, too. Contact WILMAD with details of the rotor dimensions.

1 J. D. Gay, J. Mag. Res. 58, 413 (1984).
2 D. Geschke and E. Quillfeldt, J. Mag. Res. 65, 326 (1985)
3 P. J. Giammatteo, J. Mag. Res. 71, 147 (1987).

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Sample Solvents in NMR Spectroscopy

When you open the container in which a solvent is delivered, the solvent will rapidly absorb moisture from the atmosphere, compromising deuterium purity and adding unwanted contaminant to the solvent. TMS bottles should be warmed to room temperature before opening to avoid condensing water onto the cold inner surfaces of the bottle and cap. Always recap TMS tightly.

We recommend that deuterated solvents with >99.8% isotopic purity be opened only in a glovebox or glovebag under a dry atmosphere. Most solvents can be dried over molecular sieves; Linde 3A and 4A, 1.6mm are acceptable. Avoid bead-shaped sieves; they don't do as well in drying solvents.

Make sure that all the glassware you use is dry, too. Since water chemisorbs to glass and even vigorous oven drying doesn't remove the last traces, we recommend you equilibrate residual water through D2O exchange. It's important to recognize that oven drying NMR tubes can irreparably distort precision NMR tubes. Rather, in a dry atmosphere, soak all glassware with a quantity of D2O that covers the working area of the glassware, rinse with sieve dried deuterated Acetone, and dry in vacuo or under a stream of dry nitrogen. Cap all NMR tubes and store all glass in a very dry atmosphere or in a D2O saturated atmosphere (e.g. in a desiccator). WILMAD long-tip NMR pipettes help minimize the area of glass that must be equilibrated with D2O, because they allow you to place the sample directly into the bottom portion of a preconditioned NMR tube. Large bottles of D2O can be kept isotopically pure even with a septum affixed on top. Use PTFE-sheathed Silicone septa (good resealability) and always flush a carefully dried syringe with dry nitrogen before using. Remember to inject the bottle with a volume of dry nitrogen equal to the volume of solvent to be withdrawn. Careful, firm application of a septum cap under dry atmosphere in a glovebox or glovebag will help avoid contamination when you first open a capped bottle of deuterated NMR solvent. For the study of nuclei other than 1H and 13C, most any solvent that dissolves the sample can be used. The addition of a sufficient quantity of a miscible deuterated solvent to provide a lock signal is necessary to assure spectrometer stability. Combinations of protic and deuterated solvent are frequently used to control solvent costs while providing a sufficient source of deuterium lock signal.

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