NOTES

Apparatus for the Preparation of Sealed Samples for NMR

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Many times when measuring the NMR spectra of certain compounds it is necessary to protect the sample from atmospheric oxygen or water. The effect of dissolved oxygen on the NMR spectrum is well known.[1] It may also be necessary or desirable to obtain the NMR spectrum with the sample compound in an appropriate solvent together with a reference standard. These considerations are particularly important when obtaining reference spectra of high-purity standard-sample compounds.

This laboratory is engaged in obtaining reference NMR spectra of API–USBM Standard sulfur compounds and nitrogen compounds. To prepare these samples properly, it is necessary to seal a measured volume of each compound together with a measured amount of solvent and reference compound in an NMR sample tube while excluding air. It is also convenient to prepare and store a number of samples in advance, such that the standard compound is isolated from the solvent and reference compound until just prior to obtaining the spectrum.

The literature did not reveal any convenient method for meeting our sampling requirements. The simple all-glass apparatus described in this paper fulfills all of the requirements and yet is easy to construct and use.

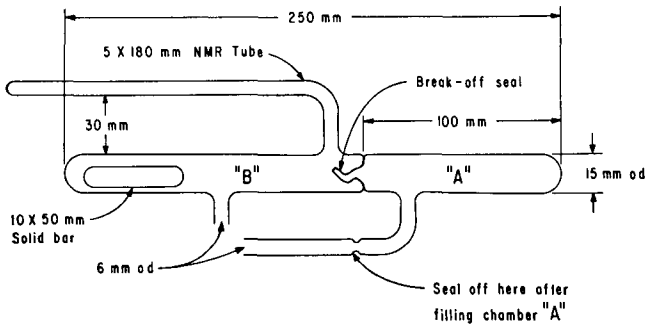

FIG. 1. Apparatus for preparation of sealed NMR tubes.

Description and Use of Apparatus: Figure 1 is a diagram of the apparatus. The all-glass unit consists of two chambers (A and B) separated by a break-off seal. A solid glass bar, for breaking the seal, is enclosed in chamber B and the NMR sample tube is sealed onto a side arm on chamber B.

Chamber A, the sample chamber, is sealed onto a vacuum manifold by means of the side arm. A measured amount of sample is vapor transferred into the chamber and sealed off. The apparatus is then carefully inverted (care must be exercised to avoid damaging the break-off seal) and resealed onto the vacuum manifold by means of the remaining side arm on Chamber B. Measured quantities of solvent and NMR reference standard are then vapor transferred into chamber B and the apparatus is again sealed off. The sample, solvent, and reference standard are isolated from each other as well as from the atmosphere and may be stored for future use.

To prepare the sample for use in the spectrometer the break-off seal separating chambers A and B is broken, using the solid glass bar in chamber B. Mixing the sample, solvent, and reference standard is best accomplished by cooling chamber A in liquid nitrogen and drawing the solvent and reference into chamber A. The solution may then be thawed and poured into the NMR tube which is then sealed off after being cooled in liquid nitrogen.

This apparatus provides the spectroscopist with a method of preparing a sample that has been isolated from the atmosphere, thus reducing the deleterious effect that may be brought about by exposure to oxygen, moisture, or other atmospheric agents. Samples subject to actinic decomposition could be protected by use of the proper type glass ampoule or storage in the dark.

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1. See, for example, J. W. Emsley, J. Feeney, and L. H. Sutcliffe, *High Resolution Nuclear Magnetic Resonance Spectroscopy* (Pergamon Press, Ltd., London, 1965), Vol. I, p. 260.