NMR Solvents

Use and Handling of NMR Solvents

Most deuterated NMR solvents readily absorb moisture. To minimize the chance of water contamination, use carefully dried NMR tubes and handle NMR solvents in a dry atmosphere.

How to Obtain a Nearly Moisture-free Surface

1. Dry glassware at ~150 °C for 24 hours and cool under an inert atmosphere.
2. Rinse the NMR tube with the deuterated solvent prior to preparing the sample. This allows for a complete exchange of protons from any residual moisture on the glass surface.
3. For less demanding applications, a nitrogen blanket over the sample preparation setup may be adequate.

How to Avoid Sources of Impurities and Chemical Residues

1. Use clean, dry glassware and PTFE accessories.
2. Use a vortex mixer instead of shaking the tube contents. The latter action can introduce contaminants from the NMR tube cap.
3. Residual chemical vapor from equipment can be a source of impurities; residual acetone in pipette bulbs is a common example.

How to Remove Solvent Residue

1. Protonated solvent residue can be removed by co-evaporation.
2. Use a small quantity of the desired deuterated solvent, a brief high-vacuum drying (5-10 min), and then prepare the NMR sample.
3. Solvents such as chloroform-d, benzene-d_6, and toluene-d_8, also remove residual water azeotropically.

How to Avoid TMS Evaporation

1. Extended storage of TMS-containing solvents can lead to some loss of TMS. Storing these solvents in *Sure/Seal*™ bottles virtually eliminates such a loss.*
2. Purchase TMS-containing solvents in single-use ampules.

* To dispense the product from *Sure/Seal*™ bottle or septum vials, use standard syringe needle techniques. For details and recommended procedures, please refer to Aldrich Technical Bulletin AL-134 or visit our Web site at **sigma-aldrich.com**.

Sample Preparation

Most deuterated products are hygroscopic, readily absorbing moisture from the surface of glass bottles, pipettes, NMR tubes, etc. Avoiding water contamination requires extra care in sample preparation. All glassware that will be in contact with the solvent should be dried at 150 °C for 24 hours, and then allowed to cool under a dry inert atmosphere. The capped NMR tube can then be transferred to a glove bag or box where the sample can be prepared under a dry inert atmosphere. An easy way to ensure this for samples in which water contamination is not as important is to use an inverted plastic bag or funnel and flow dry nitrogen over the bottle and NMR tube when transferring the solvent. Using a vortex mixer, rather than shaking, to get a homogeneous solution can avert leaching from cap liners and using PTFE caps eliminates the problem.

Packaging

To meet individual needs, deuterated solvents are available in a variety of sizes and package types. We offer solvents in gram units, as well as in ampules. We recommend ampules for those customers who are concerned with maintaining high product integrity.