Use and Handling of NMR Solvents

CIL has implemented extensive quality control protocols for the evaluation of chemical and isotopic purities of our solvents. CIL understands that the increase in sensitivity and resolution of today’s high field NMR instruments requires solvents with the highest chemical purity as well as high isotopic enrichment. Each lot of our NMR solvents receives thorough quality control testing before being released for shipment. All ampoules and bottles are clearly marked with both a production and a packaging lot number for easy tracking in the unlikely event that a problem should occur.

Water Peaks

Water contamination is a common problem for deuterated NMR solvents. There are several things that can be done to minimize/eliminate water peaks.
- Consider using single-use ampoules. Many of our solvents are available in single-use breakseal ampoules ranging in size from 0.25 mL to 3 mL.
- Handle solvents in a dry atmosphere.
- Dry NMR tubes and pipettes used for sample preparation overnight in an oven and cool them in a dessicator prior to use.
- Precondition an NMR tube by rinsing it with D₂O. Remove residual D₂O by rinsing first with methanol-D₄ or acetone-D₆ and then with the solvent of choice. This process will not remove water, but it will exchange the protons for deuterium and minimize the water peak.

“100%” D₂O

To avoid loss of enrichment due to exchange with ambient moisture, “100%” D₂O stored in a serum bottle should be sampled with a syringe that has been preflushed with dry nitrogen. Additionally, a volume of dry nitrogen equal to the amount of D₂O being removed should be injected into the serum bottle prior to withdrawing D₂O.

TMS Evaporation

When stored at room temperature (unless noted below) and properly capped, solvents containing TMS should not suffer from TMS evaporation. However, upon extended storage of these solutions, some loss of TMS may occur.

Storage

All serum bottles should be stored upright in the refrigerator; we do not recommend freezing. We also recommend that chloroform, diethyl ether, diglyme, tetrahydrofuran, and TMS be stored in the refrigerator.

Deuterated Chloroform

The deuterated chloroform produced at CIL is of the highest chemical purity. Over time chloroform will decompose regardless of the storage container or conditions. Over many months of storage at room temperature (for example, in a stockroom), deuterated chloroform can become acidic. However, decomposition is minimized if bottles are stored refrigerated in the dark.

CIL takes several precautions during production and packaging of chloroform-d to further minimize decomposition. Exposure to oxygen is minimized by using an argon atmosphere during production and packaging. Amber bottles are used to protect the product from light. Finally, silver foil is added to the solvent to act as a radical scavenger, which helps to stabilize the material over time.

Quality Control of Deuterated Chloroform

To ensure the highest quality, CIL routinely tests each batch of solvent for chemical and isotopic purity. The chemical purity is monitored during production and packaged using ¹H NMR, GC, Karl-Fisher titration for total water content and other wet chemical methods for acidity and various impurities.

Proper Storage and Use of Deuterated Chloroform

Unopened bottles of chloroform-d should be refrigerated (-5°C to +5°C) to maximize shelf life. Moisture and oxygen will be introduced to the solvent following initial use through air entering the bottle upon opening. Decomposition can follow which results in the deuterated chloroform becoming acidic. The acidity can be easily tested using the following method: