



## 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  $^1\text{H}$  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 ( $-5^\circ\text{C}$  to  $+5^\circ\text{C}$ ) 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:

### Testing Deuterated Chloroform Acidity

A 1 mL aliquot of the solvent is added to a test tube containing 1 mL of distilled water (pH 5.0-7.0) and 2 drops of Bromothymol Blue (0.04% W/V). The color is compared to a 2 mL blank of distilled water (pH 5.0 – 7.0). If the sample solution is discolored (yellow) relative to the blank (blue-green), the deuterated chloroform is acidic.

Samples of deuterated chloroform which have become acidic can be easily neutralized using the following procedure:

- Place 3-5 grams of a  $5\text{\AA}$  molecular sieves into a 50 or 100 g bottle of the solvent.
- Swirl slightly and allow to stand overnight. Excess water and traces of acidity will be removed. This is also the preferred way to store chloroform-d bottles once they have been opened, as it will keep the solvent dry and stable over time.
- Maintain an inert atmosphere (argon or nitrogen) in the bottle.
- Small "dust or powder" particles may break off from the molecular sieves. However, these particles can be removed simply by filtering the quantity of deuterated chloroform needed for an NMR sample through a small plug of glass wool or cotton in a glass pipette.

### Special Applications Which Require Ultra Dry and Acid-Free Deuterated Chloroform

For applications involving highly acid-sensitive or moisture-sensitive compounds, deuterated chloroform can be purified further prior to use. Solvents treated in the following manner will be exceptionally dry and acid free.

- Place a glass wool plug into a disposable glass pipette (~7mm diameter).
- Add dry alumina powder into the pipette to a height of 3-4 cm.
- Pass the solvent through the small alumina bed into the sample container containing the product to be analyzed.
- Analyze the sample as soon as possible.

This procedure will ensure that the deuterated chloroform is dry and free of trace amounts of acid prior to contact with the sample. Note that the chloroform will react with basic compounds, such as alkaloids or amines. If the product is to be recovered, this should take place as soon as possible to minimize possible reaction.