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NMR-010: Proper Cleaning Procedures for NMR Sample Tubes

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Introduction

WILMAD NMR tubes are not 'analytically clean' when delivered to you. So if your NMR samples require scrupulously clean glass, follow the procedures below for Difficult Cleaning Problems to assure your sample purity is never jeopardized. Since NMR tubes are formed ove a metal mandrel and certain organic lubricants are used, these cleaning steps will assure that any trace organic or inorganic residues from these procedures is removed.

When you invest in high quality precision NMR Sample Tubes, you expect high resolution and sensitivity. Proper cleaning procedures can help you preserve the quality of your investment. Since the purpose of an NMR Sample Tube is to confine a liquid sample in a perfectly cylindrical volume within the spectrometer probe, the degree to which the tube accomplishes this determines the quality of the sample tube. Improper cleaning can damage NMR tubes and reduce your apparent spectrometer performance.

You should never use a brush or other abrasive materials to clean NMR tubes. Scratches on the inside surface of the tube allow a portion of the sample to extend beyond the perfect cylinder defined b the NMR tube. Because the portion of your sample which fills a scratch on the inner surface of a tube experiences a different magnetic field than the rest of the sample, lines will broaden and resolution will deteriorate when you use scratched tubes. And you'll see a reduction in apparent spectrometer performance, unless you reshim your spectrometer for each sample. That's a tedious procedure your investment in high quality tubes was designed to eliminate to begin with.

Proper cleaning of NMR tubes can be easy or difficult, depending on your sample. We'll start with simple cleaning situations and move to the harder cleaning problems. Because even difficult cleaning procedures end with a proper rinsing, explained under Simples Cleaning of NMR Tubes, you should be familiar with both cleaning procedures.

Simple Cleaning of NMR Tubes

When cleaning your NMR tubes is as simple as rinsing the tube with water or an organic solvent, you can rinse them one at a time. Your main concerns, then, are what to do with the rinsate. And, if you're using Acetone, also preventing dermatitis that results when oils are

removed from your skin by this potent solvent.

If you rinse a lot of tubes, there are apparatuses available that will make your job much simpler. Tube washers, listed in the WILMAD NMR Catalog as Solvent Jet Cleaners, provide an easy way to clean either one or five tubes at a time. Using a vacuum flask and aspirator solvent recovery is simple. And your hands won't be so easily dried out by solvents, either. A final rinse with Acetone is frequently used to remove the last organic contents from the tube. When your sample is to be dissolved in water or D_2O, a final rinse with distilled water is usually adequate. You may want to take steps to remove traces of water from the surface of the tube. Follow the procedures for deuterium exchange, below.

Difficult Cleaning Problems

Tubes left with samples in them for a period of time frequently preser a more challenging cleaning problem. Sample degradation or precipitation can cause material to adhere to the inner walls of the tube. Rinsing the tube doesn't always remove this adhered material. So WILMAD recommends using strong mineral acids such a concentrated or, in severe cases, fuming Nitric Acid soaks of 1-3 days, as needed. Nitric Acid can oxidize many organic chemicals and dissolves most inorganic materials, as well. WILMAD doesn't recommend using Chromic Acid, since residual Chromium can often adversely affect NMR experiments. Chromic Acid, while a stronger oxidizer, can leave paramagnetic Chromium VI behind, which can be removed only with repeated soaks with Nitric Acid. Copious rinsing of NMR tubes washed in acids is required to assure removal of residual acids. A final rinse with distilled water or Acetone is also appropriate.

Tubes which contained polymeric samples can be even more difficult to clean. When the polymers are natural products, like proteins and polysaccharides, strong acid soaks will usually be sufficient. However when dealing with synthetic polymers, the challenge is more severe, since many polymers are inert to acids or insoluble in organic solvents by design.

Although polymers may not readily dissolve in solvents, it may be possible to soften them by soaking the tubes in a solvent that swells the polymer. Then a pipe cleaner might be sufficient to remove the softened material. It may take some experimentation to find the solvent combination that works best with your polymer system.

Agitation in an Ultrasonic bath with an appropriate solvent can also help dislodge stubborn sample residues. However, you should take precautions to assure that NMR tubes don't touch, since contact and vibrations can fracture delicate thin wall tubes. WILMAD offers a special tube rack for use in its Ultrasonic bath that prevents such destructive contact between tubes.

Removing Water from NMR Tubes

Drying tubes at elevated temperatures can reshape and ruin precisio

NMR tubes. If you dry tubes in an oven, WILMAD recommends placing tubes on a perfectly flat tray at 125° C for only 30-45 minutes Better is the use of a vacuum oven that will remove water at lower temperatures. In a flat position, tubes that do reshape could be out-of round and may not fit the spinner turbine as well. But they'll not affect the spectrometer probe adversely. Tubes placed in an oven in a beaker, flask, or tube rack can bend, increasing Camber (lack of straightness)[1]. Bent tubes may still fit the spinner turbine, but can damage or break the NMR probe insert, a costly repair with many probes.

But even drying at high temperatures doesn't remove water chemisorbed to the surface of the tube. Thus, the preferred method o water removal is chemical, not physical, treatment. In most cases, it i the protic content of water that must be avoided. So WILMAD recommends exchanging the protons of chemisorbed water with a deuterated solvent such as D_2O prior to a short drying period in the oven. A bottle of D_2O that isn't being used any longer is perfect for this purpose.

When water chemically degrades your samples, then removal of water is essential. Here, reaction of the water with a hydride solution can be used, with caution. After rinsing the hydride solution, a final rinse with very dry Acetone can be used to remove rinse solvent prior to oven drying. Cap tubes promptly to avoid absorption of moisture when removing dry tubes from the oven.

[1] See Resonance Report NMR-001 for an explanation of NMR tube specifications, like camber.