

Warning: This very practical entry is rigorously for NMR spectroscopists.

I have met on the Net a little cute [set of instructions](#) on how to handle one's NMR tubes. I subscribe to everything the people at [Temple University](#) say, except that I would like to add three points. Today we mostly buy outrageously expensive sample tubes, thinking "I worked on this stuff 6 months so if I spend \$50 on the tube to put it in, what's the problem?".

Well the problem may well be how you store the tubes *before* you actually use them. Suppose you pick up a tube and, since you realize you have no immediate use for it, you put one of those plastic caps on it (to make sure no dust gets inside) and leave it on your desktop. And then a student comes around and drops down an NMR tome he/she was reading and does not notice there is a tube under it, leaving the tube in a situation like this:



Since glass is a meta-stable fluid solution (never mind how large is its viscosity), after a week in this position the tube will look like a Turkish scimitar (*this is an exaggeration ...*) and be ready for the dustbin (... *and this is not*). Even without the book, if you leave 10 packs, each with 10 capped crown diamond, laser-rectified NMR tubes lying horizontal in a closet for one year, you have *de-facto* thrown a few thousand bucks out of the window. The only really safe way to store your tubes, be they capped or not, is in a **vertical position** - the same way any glass workshop stores its sheet glass and its raw glass tubes!

The second point regards the removal of paramagnetic impurities. The document warns against washing the tubes by paramagnetic solutions like chromic acid. That is absolutely correct, of course. However, it does not say what to do when you actually measured a paramagnetic sample (such as a manganese or copper complex of a protein). Nor does it mention that when you go after the highest possible resolution, you should not consider even a virgin tube to be free of paramagnetics - at some level, the whole world is paramagnetic! In this case you will of course carefully degas your sample to remove the 0.25 - 0.30 Hz or so of the linewidth due to the paramagnetic oxygen, but the unavoidable paramagnetic impurities in the glass are still likely to contribute about 0.05 Hz.

A magic word in these cases might be **dithizone**. It is a cheap and universal metal-ions complexant which can scavenge metals out of everything (it is used in medicine in cases of metal poisoning). Just fill the tube with a suitable dithizone solution and let it stay still for a few days. It will strip metals (both paramagnetic ones like iron and diamagnetic ones like zinc) from even quite deep layers of the glass. The contents may change color which is a sure sign that metals were indeed present and got chelated. Finally, rinse it several times with iso-propyl alcohol or pure methanol before drying it. By the way, washing with iso-propylalcohol is in general better than washing with ethanol (methanol might be better in some cases, but it is too toxic for generic use).

Of course, I am no chemist so there may be many other chelating agents just as good as dithizone. It just so happens that I have used it (a very long time ago) with excellent results.

The third point may sound trivial, but it is not **when a sample is to be transported**. Once a sample tube has been filled and capped or sealed with parafilm, make sure that you never turn it over enough for the sample itself to get in contact with the seal. There is no tube cap, no plastic, and no parafilm (a polybutadiene-and-wax wafer) which is really inert and does not at least partially dissolve in common NMR solvents. Sealing the sample over flame is also problematic since the inner surface that gets scorched is never totally clean and uncontaminated by the sample or by solvent vapours. If you cannot avoid shipping a tubed sample, at least **seal it first with a good-quality teflon film** before putting on the cap and/or parafilm. It takes just a minute and, if you do it properly, it can reduce the risk of contamination during transport by an order of magnitude.