NMR Sample Preparation

Proper sample preparation is the key to obtaining a high quality NMR spectrum. The follow points should be considered.

1. A good quality sample tube free from cracks, scratches or other defects should be used. So-called "throw-away" tubes should ***never*** be used in a modern NMR instrument. The minimum grade of tube required is Wilmad 507-PP, with 528-PP or better recommended for the 500 MHz and 600 MHz instruments. NMR sample tubes are not laboratory glassware, and should not be treated as such. Sample tubes should not generally be placed in a drying oven, but if drying is necessary the oven must not exceed 100°C, and the drying time should not exceed 1 hour. The tubes must lie flat on a horizontal surface while in the oven. ***Distorted or cheap sample tubes can cause expensive damage to the instrument.*** NMR sample tubes must ***never*** be cleaned with chromic acid. Sample tubes that ***have*** been cleaned with chromic acid should be thrown in the trash.
2. Particulate matter in a sample can result in a very poor spectrum. Sample tubes should be stored where dust cannot collect in the tube, and can be cleaned with a jet of dry filtered nitrogen (not compressed air) if necessary. Samples for routine organic work do not usually need to be filtered unless there are obvious particles. Samples for high resolution or for more critical 2D NMR work will benefit from filtering. A convenient filter is a small wad of KimWipe stuffed tightly in a Pasteur pipette and cleaned with chloroform. The chloroform can be removed by passing a stream of nitrogen through the pipette.
3. Samples must be the proper length for the instrument. (That is, the length of sample in the tube, not the length of the tube itself.) Modern Bruker probes require a sample depth of 4 cm, which is 0.55 ml in a standard 5 mm tube. Shorter samples do not "shim" well, and longer samples waste solvent. Note that there are special tubes available for running shorter samples. These may be desirable when the sample quantity is very limited and you want to maximize the amount of sample in the active region of the probe (which is typically 14 to 20 mm in length). Varian NMR instruments require a slightly longer sample.
4. Good laboratory practice dictates that all sample tubes must be labeled. A marking pen works fine, but Scotch "Magic Tape" has a good writing surface and makes a more permanent label. Labels must be firmly and symmetrically attached to the tube.
5. The typical plastic NMR tube caps should be considered a disposable item. Re-use is acceptable if you are careful to avoid contact of the sample with the cap. Common problems resulting from re-use of the caps are cross contamination of one sample to the next, and contamination of the sample with plasticizers and oligimers from the caps themselves. This is much less of a problem with aqueous samples. Cleaning the caps with a solvent such as acetone further aggravates the problem, as solvent will absorb into the plastic and then contaminate subsequent samples. Note also that the cost of solvent used to clean the cap will probably exceed the cost of the cap itself. Replacement caps can be ordered from Wilmad Glass in bags of 1000 caps, at a cost of about $ 0.05 each.