NMR Sample Preparation

Under any circumstances, it is not allowed to prepare the sample at the NMR room.

In NMR, the quality of the sample has a profound effect on the quality of the resulting spectrum. So that the sample you prepare gives a spectrum in which useful information is not lost or obscured, you must follow a few simple rules:

Use the Correct Quantity of Material

For 1H spectra of organic compounds the quantity of material required is about 5 to 25mg. It is possible to obtain spectra from smaller quantities, but at very low concentrations, the peaks from common contaminants such as water and grease tend to dominate the spectrum. 13C is six thousand times less sensitive than 1H, and if about 0.2 to 0.3 millimoles can be dissolved in 0.6ml, the spectrum will take approximately half an hour to record. If the quantity of material is halved, the data accumulation time will be quadrupled. You should be aware that if you make up a sample at high concentration for 13C, and then record a 1H spectrum from it, the increased solution viscosity may result in a spectrum that has broader lines than you would get from a more dilute solution.

Remove All Solid Particles

Solid particles distort the magnetic field homogeneity because the magnetic susceptibility of a particle is different from that of the solution. This causes broad lines and indistinct spectra that cannot be corrected. So that there are no solid particles in your samples, you must filter the samples into the NMR tube.

Make Samples to the Correct Depth

In the magnet, the main field direction is vertical, along the length of the sample. Each end of the sample causes a major distortion of the field homogeneity, which is corrected using the spectrometer's shim controls. Achieving optimal line shapes in the NMR experiment depends critically on the amount of solvent used. In general, a solvent column needs to extend the rf coils-length both above and below the coil (i.e., total height = 3 times the coil length). Different probes have different coil lengths, so there is no universal guideline to apply. The recommendation is:

Bruker 5 mm probes, coil length = 12 mm => 36 mm solvent length => 0.5 mL solvent

Use Deuterated Solvents

Samples must be prepared using at least 5-10% deuterated solvents (solvents that contain deuterium). The NMR signal from the deuterium nuclei is called the NMR lock and is used by the spectrometer for stabilization. In case you are not interested in observing protons that exchange with the solvent, use a “100%” deuterated solvent.

Use Clean Tubes and Caps

NMR tubes are available from the stores, and after use they should be cleaned and dried. There are several vendors that sell similar products, but according to our experience, Wilmad tubes give consistently good results. Tubes must be capped, and caps should be treated the same way as tubes. You must not use NMR tubes with a chipped or broken top because they are dangerous, and very likely to splinter lengthwise.

Wilmad NMR tubes for high-resolution NMR:

For 300/400/500 MHz work:

507-PP-7 routine work

528-PP-7 recommended

535-PP-7 best

Label Your Samples

This is best done with a permanent marker directly on the top of the tube, or on the cap. If you use a sticker or a piece of tape, your label must stick smoothly on the tube. Do not leave a flap. Remember that the tube has to spin at 20Hz (1200rpm) while it is in the magnet