In NMR, unlike other types of spectroscopy, 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 $^1$H spectra of organic compounds (except polymers) the quantity of material required is about 5 to 25mg. $^{13}$C is *six thousand times* less sensitive than $^1$H, and a good rule of thumb is to provide as much material as will give a saturated solution. If about 0.2 to 0.3 millimoles can be dissolved in 0.7ml, the spectrum will take no more than about half an hour to record. If the quantity of material is halved, the data accumulation time will be quadrupled.

**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. You should filter samples through a small plug of glass wool tightly packed into a Pasteur pipette. After filtration the sample should be as clear as water though, of course, not necessarily colorless.

**Make Samples to the Correct Depth**

The NMR sample depth must be between 4.5cm and 5.5cm. Shorter samples are very difficult to shim, and cause considerable delay in recording the spectrum. Samples that are too long are also difficult to shim and are a waste of costly solvent.

**Use Deuterated Solvents**

Samples must be prepared using solvents that contain deuterium in place of hydrogen. The NMR signal from the deuterium nuclei is called the NMR lock and is used by the spectrometer for stabilization.

**Use Clean Tubes and Caps**

After the use the NMR tubes should be rinsed with acetone or some other
suitable solvent, and then dried with a blast of dry air or nitrogen. 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.

**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.

**Use an internal reference**

TMS is commonly used as an internal standard. Small amount of TMS (less than a drop) added to a bottle of CDCl₃ would be enough. This provides a small TMS signal; you never want your reference signal to be taller than your solvent signal. Large amount of TMS could cause serious problems due to distorted baseline and exceeded dynamic range. Alternatively, the residual protons in the deuterated solvent may be used as a secondary reference.

**Degassing Samples**

Some samples need to be degassed or have oxygen removed. The only effective way of doing this is by using the Freeze-Pump-Thaw technique, at least three cycles. Do not bubble nitrogen through the solution in an NMR tube. This wastes costly solvent through evaporation, and is not an effective method of removing oxygen.