

NMR Sample Preparation

Sample Quality:

To get best possible quality NMR samples needs to be free of suspended material (e.g. precipitates, dust). Suspensions led to increased signal linewidth and reduced spectral resolution.

Avoid solvent gradients inside the NMR tube (which can easily be seen with colored samples)

Tube handling:

Make sure that the NMR tube is clean inside AND outside. Tubes that are dirty on the outside cannot be put into the NMR instrument and will also result in bad spectra (and in a penalty of 2 weeks!). The tube should be in good condition with no cracks or chips. If the top of the NMR tube breaks take it to the glass blower to grind it down (minimum length: 17cm).

Avoid heating NMR tubes in ovens. Tubes may warp, bent and flatten, destroying the tube's quality. It is best to dry the tubes with a blast of dry air or nitrogen. If you decide to dry tubes in the oven, Wilmad recommends placing tubes on a flat surface in the oven at 125°C for 30-45minutes. Again: store them in a flat surface.

How to get rid of suspended material?

Suspended materials can easily be removed from an NMR sample by using cotton wool as a filtering agent. Cotton wool tends to filter out the smallest particles (whereas glass wool does not filter out small particles). The wool can be placed inside a pipette for filtering. (Or use a plug of cotton wool on the tip of a pipette. Leave the wool in the vial/flask).

How to prepare a good NMR sample?

Use a flask or a glass vial. Put your sample inside the vial/flask and use a balance to determine the sample amount. Add an adequate amount of deuterated solvent (e.g. CDCl_3 , DMSO-d_6 , CD_3OD , Acetone- d_6 , CD_3CN , etc.) into the vial/flask to dissolve your sample, followed by shaking of the sample for mixing (or use a Vortex-System). If necessary, filtrate your sample through cotton wool (or membrane filters). Transfer the solution into the NMR tube by using a pipette and put the cap on the tube (avoid contact of sample and cap, or use new caps). You may use some Parafilm around the cap.

Clean your sample tube with a Kleenex/Kimtech wipe.

Adequate amounts of solvents and filling heights in NMR tubes for our NMR lab are:

5mm tube (400MHz): 0.5-0.6ml Filling height: 4-5cm

3mm tube (600MHz): 120-150 μl Filling height: 2.5-3.5cm

If you make a mark on the NMR tube for measuring the correct solvent height, clean it off before you submit the sample for analysis.

DO NOT:

Do not fill the solid compound directly into the NMR tube and try to dilute by adding deuterated solvent. This led to sediments or compound gradients inside the NMR tube.

You can be absolutely sure that the spectral quality will not be good!