

NMR Sample Preparation ¹

To obtain high resolution NMR spectra it is necessary that your **NMR sample is free of suspended material** (precipitates, dust, etc.). Suspended material present in solution will increase the line width of the spectrum; broad spectral lines reduce spectral resolution and no amount of shimming can correct for this.

Suspended material can easily be removed from an NMR sample by constructing a filter using cotton wool as a filtering agent². Conversely, glass wool is a very poor filtering agent since glass wool does not filter out small particles, whereas cotton wool tends to filter out the smallest of particles. A filter can be made by placing a small amount of cotton wool inside a Pasteur pipette as shown in Figure 1. Note that the Pasteur pipette has been cut to facilitate handling of the cotton wool. Alternatively, the cotton wool can also be pushed down an uncut pipette with the help of a long³ second pipette (this method was used in Figure 2 to position the cotton).

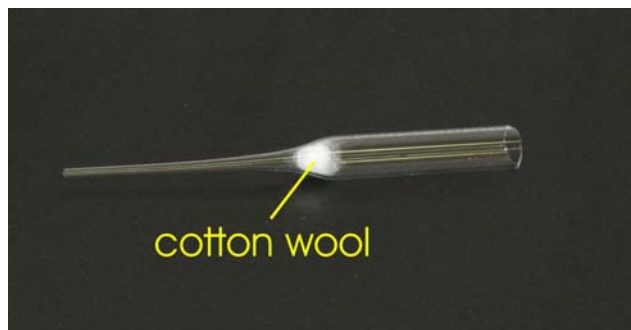


Figure 1: Pasteur pipette with cotton wool

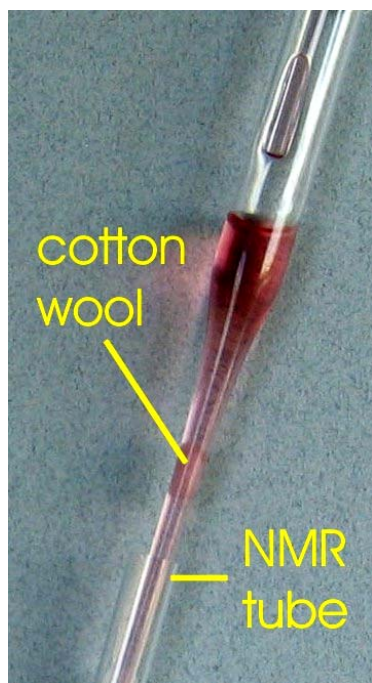


Figure 2: Filtration through cotton wool

The solute of interest can then be dissolved in a separate glass vial using less than the final volume of **deuterated** solvent (e.g. CDCl_3 , DMSO-D_8 , CD_3OD , etc.) required to make your NMR sample. After the solute has been dissolved it can be transferred directly to a NMR tube by passing the solution through the cotton filter as shown in Figure 2.

Finally, the sample volume can be adjusted by adding the remaining solvent to the NMR tube so that a final sample volume of $\sim 700 \mu\text{L}$ or a sample height of $\sim 55 \text{ mm}$ is reached (Figure 3), followed by vigorous shaking of the sample to effectively mix its content. The **suggested sample volume** for our instruments is **700 μL** . Trying to concentrate your sample by reducing the sample volume is not recommended. A short sample (i.e. volume less than $500 \mu\text{L}$) can only be shimmed through tremendous effort and this time can be much better spent acquiring your data.

The *Request for NMR Service* sheets contain a drawing to scale of a NMR tube and the suggested sample volume.

References

1. Adapted from the Chem 241/341 sample preparation guide by Dr. Jason Cooke, whose students consistently submit some of the best NMR samples seen by the NMR Service Lab.
2. Medical cotton is reported to be free of compounds that can be extracted by organic solvents (A.E. Derome, *Modern NMR Techniques for Chemistry Research*, Pergamon Press, 1987).
3. 9" pipettes are sufficient (no need to buy the much more expensive 10.5" type)

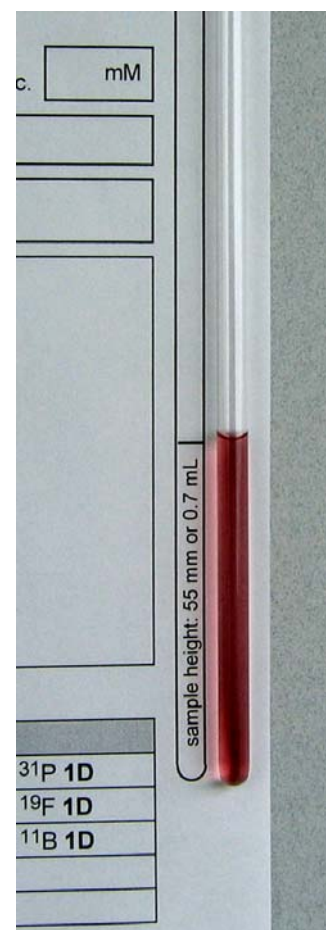


Figure 3: NMR request sheet depth gauge