8.1 - FT-NMR Sample Preparation Guide

Overview:

A good ^1H NMR sample contains about 10 mg of compound. The solution should contain no solids or paramagnetic impurities. Your deuterated NMR solvent should be free of water, and your NMR spectrum should contain no solvent peaks.

Reference:

Zubrick CH 35 and Mohrig CH 21 are relatively useful, but you should follow the specifics in this handout. Also, keep in mind, we won't be running continuous wave spectrometers, so you should disregard any discussion of them.

NMR Solvents:

Typical deuterated solvents include chloroform ($CDCl_3$), water (D_2O), benzene (C_6D_6), acetone (CD_3COCD_3), acetonitrile (CD_3CN), and tetrahydrofuran (C_4D_8O). Chloroform is by far the most popular and will be used exclusively in 5.301. The TA will prepare the bottle of $CDCl_3$ that you will use for the course. In the future, when you purchase bottles of $CDCl_3$, you will have to prepare them for use. There are typically three things that must be done before your deuterated chloroform is ready for the NMR. First, a few drops of a standard (tetramethylsilane (TMS)) are usually added. Second, any residual water in the solvent is removed by the addition of activated 4 Ångstrom molecular sieves. Third, the acidic nature of the $CDCl_3$ (and the molecular sieves) is sometimes neutralized by the addition of anhydrous, granular K_2CO_3 (a weak base). The chloroform that we will use in 5.301 has been treated with molecular sieves and TMS has been added, but, since we won't use any acid sensitive compounds, K_2CO_3 has not been added. (Note: Remember that you do not want water getting into your chloroform, so keep the bottle open to the atmosphere as little as possible. As long as it's open, water from the air will dissolve in your NMR solvent.)

Before Preparing the Sample:

1) Determine the minimum height of a sample by checking the depth gauge in the NMR room.

2) Make a measuring standard to ensure that your samples will always have enough solvent. (Hint for making a standard: use a 10-mL graduated cylinder to hold your NMR tube

when filling it. Mark the outside of the graduated cylinder with a Sharpie at the minimum height level.)

Preparing NMR Samples of Liquids:

1) Dry and remove all solvent from your compound.

2) Take a clean, dry NMR tube and place it in a 10-mL graduated cylinder (or other holder).

3) Place a Kimwipe pipet filter on top of the NMR tube. This is constructed by taking a small piece of a Kimwipe and stuffing it into a small Pasteur pipet. It can be tamped into place using the tip of a large Pasteur pipet. (This filter will remove any insoluble impurities.)

4) Dip the tip of a different pipet into the sample. Capillary action will draw approximately 10 mg into the pipet.

5) Place this on top of the pipet filter and rinse it into the NMR tube with your deuterated solvent.

6) Check to see that you have enough solvent.

7) Cap your NMR tube and record the sample number if running more than one spectrum. (The colored caps are the easiest way to do this.)

8) After running the NMR, rinse the sample back into the flask containing your compound and concentrate it to remove the solvent.

Preparing NMR Samples of Solids:

1) Do steps 1+3 above.

2) Place approximately 10 mg of your sample into a vial.

3) Dissolve your compound in about 1 mL of your NMR solvent.

4) Using a pipet, transfer the liquid through the pipet filter into the NMR tube.

5) See steps 6-8 above.

Cleaning NMR Tubes:

1) Rinse the tube thoroughly with acetone.

2) Place the tube in a drying oven for about one hour.

3) Store the tube in a desiccator at room temperature.

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