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7.1. 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 page 295 is relatively useful, but you should follow the specifics in this handout. Also, keep in mind that 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 ($\text{CD}_3\text{C}(\text{O})\text{CD}_3$), acetonitrile (CD_3CN), and tetrahydrofuran ($\text{C}_4\text{D}_8\text{O}$). Chloroform is by far the most popular and will be used exclusively in 5.301. The bottle of CDCl_3 that you will use for the course will be prepared by the TA. 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 Å 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: 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.
- 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 your sample. Capillary action will draw approximately 10 mg of the compound into the pipet.
- 5) Insert this pipet into the pipet filter and rinse the sample into the NMR tube with 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. (Writing on the colored cap is 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) Perform steps 1–3 above.
- 2) Place approximately 10 mg of your sample into a vial.
- 3) Dissolve your compound in about 1 mL of deuterated solvent.
- 4) Using a pipet, transfer the liquid through the pipet filter into the NMR tube.
- 5) Perform 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.