**Friedel-Crafts Acylation of Toluene**



**Experimental Procedure:** (Modified from Pavia, et. al., Introduction to Organic Laboratory Techniques: A Microscale Approach, 4th ed., Thompson, Brooks/Cole, 2007)

Add 2 ml of CH2Cl2 and 0.8 g of AlCl3 to a 10 ml round-bottom flask. Add stir bar and a Claisen adaptor with a septum and a vent going to a small beaker of water. (The instructor will explain in more detail). Over two minutes, add 0.6 ml of acetyl chloride through the septum using a 1 ml syringe. Weigh out 0.5 ml of your aromatic compound (see additional document for specific compound assignment) into a small vial then add it to the reaction slowly (over 5 minutes) using a syringe. Use 1 ml of CH2Cl2 to complete the transfer. Let the reaction continue for 30 minutes.

Remove the adaptor and slowly add 4 ml of ice-cold water with a syringe followed by 4 ml of concentrated HCl with a pipet. Stir to dissolve all solids; then stop stirring and let the mixture separate into two layers. Add another 1-2 ml of CH2Cl2 if necessary to achieve clear separation. Pour the mixture into a centrifuge tube then use a pipet to transfer the lower, organic layer to a 5 ml conical vial. Try not to get any of the top, acid layer in the vial. Add 1 ml of 5% sodium bicarbonate to the vial and mix gently until the bubbling has stopped. Transfer the organic layer to another conical vial and dry it with sodium sulfate. Decolorize the solution using a pipet column containing ~ 3 cm of alumina. Collect this solution in a dry, pre-weighed flask. Remove the solvent using a gently stream of N2.  Be careful not to evaporate away your product. Get the final weight then seal up your flask so that the product does not evaporate over the week.

Characterize the product using IR spectroscopy and 1 H NMR as directed by your instructor.

**Report Format:**

The grade for this experiment will be based on the laboratory notebook and your word processed portion which will include experimental, results and discussion. The results and discussion will include yield, IR and NMR. The format for this will be discussed in lab.

**Relevant Sections in Lab Text:**

Chapter 2: Infrared Spectroscopy and NMR

Chapter 3: Extraction and Drying Solvents

Notes on the pre-lab:

* The primary goals for this lab are to successfully complete the reaction and to determine which isomer is formed in the reaction.
* You will be assigned a specific aromatic compound at the beginning of lab, so you should leave space to fill in safety data and physical constants. Since you do not know the specific aromatic, the reaction in the pre-lab will have to be generic.