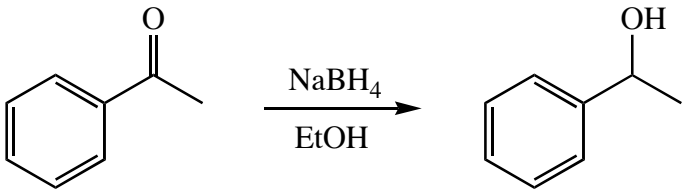
Sodium Borohydride Reduction of Acetophenone

Organic Lab – revised 10/4/07

For this experiment, you will do a simple hydride reduction of a ketone and follow the progress of the reaction by thin layer chromatography (TLC). You will also characterize your product by IR and/or NMR spectroscopy. Here is the overall reaction:



Before lab, be sure to read the sections from your lab text that describe the theory and practice of TLC. You may need to review your understanding of hydride reductions, and IR and NMR spectroscopy, too. With that background, consider whether the R_f value of your product will be higher or lower than the R_f of your starting material? What would be the principal IR and NMR absorption frequencies for your starting material and the product? That is, what would you look for to confirm the conversion of the staring material to your product?

Preparing to Monitor the Reaction by TLC.

A commercial TLC plate will be cut for you to be a square with each side measuring 6 -7 cm. Prepare this plate for monitoring the progress of the reaction by dotting four PENCIL spots evenly spaced across one edge and one cm from that edge (at the "bottom" of the plate) and 1 cm from each of the sides. We will spot the acetophenone onto this plate at the first spot and the reaction mixture at two times during the reaction process. A final spot of your product will also be run on the plate. All spots will be developed at the same time. Please make sure that you have enough material on each spot by visualizing the spots with a UV light while you spot them! Be careful to NEVER look directly at the light of this UV lamp, or your eyes could be severely damaged. If you can see your sample on the plate using the UV light, you have enough.

Reduction of Acetophenone

Dissolve ~0.25 g acetophenone (know the exact amount) in 5 mL of 95% ethanol in a 10 mL Erlenmeyer flask. Use a TLC spotter to put a spot of this solution at spot #1 on your TLC plate; you will probably need to use 10-12 "dots". Add ~0.08 g sodium borohydride (again, know the

exact amount) and your stir bar and clamp the flask above your stirring hotplate. Carefully begin to stir the mixture at RT – do NOT heat. After 5 minutes, stop the stirring (so you don't break your spotter in the solution) and use a fresh TLC spotter to spot the reaction mixture on spot #2. Then begine stirring again. After 5 more minutes (10 minutes total), stop stirring and use a fresh TLC spotter to spot reaction mixture onto spot #3. Begin stirring again and allow the reaction to proceed for 15 more minutes.

The reaction should be complete now, so slowly add 3 mL of ice-cold distilled water by pipette to the reaction mixture in the flask. Transfer the mixture to a centrifuge tube and extract with methylene chloride (CH_2Cl_2; 3 x 3 mL) by mixing the mixture with your pipet. Combine the CH_2Cl_2 extracts in a 25 or 50 mL Erlenmeyer and dry them over sodium sulfate (Na_2SO_4). Gravity filter the solution directly into a 25 mL round bottom flask and use a new TLC spotter to spot this mixture as your final spot of the TLC plate (spot #4). Remove the solvent with the rotary evaporator; you should expect no more than about 0.25 mL of an oily product in the RB flask. Weigh the amount of your product and calculate the theoretical and percent yield. Characterize by IR and/or NMR, as directed by your instructor.

Develop the TLC Plate

Develop the TLC plate in your solvent chamber using an appropriate solvent system. Each table in the lab will use a slightly different solvent mixture for this purpose; you will need to prepare about 40 mL of your mixture. The options are given below and all students will report to the whole class their R_f values for both acetophenone and for 1-phenylethan-1-ol. When you remove the plate, mark the solvent front with a pencil (never a pen). Locate the four spots with a UV lamp (again, NEVER look directly at the lamp itself). Calculate the R_f values for the largest spot(s) of your samples and report them to the class on the white board. Draw a picture of this plate in your notebook, and attach the original on a permanent page. Use the class data and your own to determine which solvent system would work best to follow the course of this reaction.

Solvent options for mixtures of hexane and ethyl acetate: 70:30; 60:40; 50:50; 40:60; and 30:70.

Clean Up

The TLC solvent mixtures should be placed in the organic waste bottles. Check the pH of the EtOH/aqueous mixture; neutralize as needed and flush down the drain.