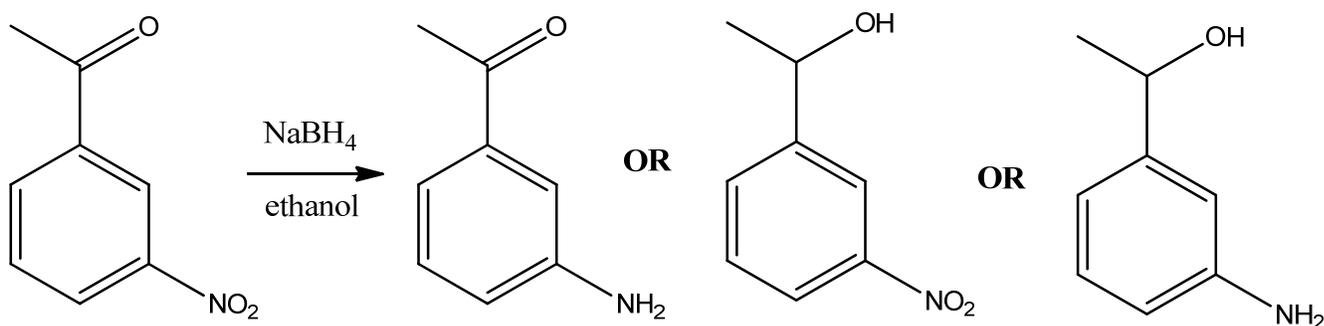


Reduction of 3-Nitroacetophenone

Question: Which group is more susceptible to reduction with sodium borohydride, a ketone or a nitro group?

Reaction:



Procedure: Dissolve/suspend 200 mg of 3-nitroacetophenone in 2.5 ml of ethanol. (This may require heating at the lowest heat setting of the hotplate. If all the solid doesn't dissolve, that is ok, but having the smallest possible particles in suspension will make the reaction go more quickly and completely.) Allow the reaction to cool to room temperature, then stir the reaction while adding 70 mg of sodium borohydride in three portions over 2-4 minutes. Add 2.0 mL of water to the reaction and heat the reaction to a gentle boil for one minute. Cool the reaction. If it is still bubbling when cool (due to decomposition of sodium borohydride) add 3M HCl dropwise until bubbling stops. Cool the reaction for 10 minutes in an icebath.

Transfer the reaction mixture to a 15 mL centrifuge tube to perform an extraction on miniscale. (Proper procedure will be demonstrated in class, but it follows the same principles as extraction with a separatory funnel.) Wash the reaction vessel with 2 mL of dichloromethane and add it to the centrifuge tube for the first extraction. Cork the centrifuge tube and shake gently but thoroughly. Allow layers to separate, and remove the dichloromethane from the bottom layer of the centrifuge tube with a pipet and place the solvent in a second centrifuge tube. Extract the aqueous layer with a second 2 mL portion of dichloromethane, then remove the dichloromethane and combine it with the first portion. Wash the combined dichloromethane with 2 mL of water. Separate the dichloromethane into a small Erlenmeyer flask and dry with sodium sulfate for at least 5 minutes. Decant the dichloromethane into a small, tared beaker and evaporate the solvent under air at low heat. If the product solidifies, obtain a melting point.

Results and observations: Collect thorough observations of the reaction and all reactants and products. Determine for yourself what results are necessary to write the comments as outlined below.

Discussion:

1. Which compound or compounds were formed in this reaction? How does the following information help (or not help) in this identification?
 - a. Yield
 - b. Melting point (if obtained)
 - c. IR spectrum OR ^1H NMR spectrum
2. Refer to a standard organic chemistry text to find three other reducing agents that could be used in complementary fashion (i.e. selectively reduce the carbonyl, selectively reduce the nitro group, or do both.)

Lab 6 assignment: Turn in a hardcopy of your lab notebook and a formal Abstract

- Due at the beginning of lab next week
- 25 pts based on in-lab performance, notebook, and formal abstract
- The abstract MUST contain this information in as succinct a manner as possible: main purpose of experiment, major experiment and techniques, major results (data), and major conclusions. It should also have an Abstract Scheme (using ChemDraw).
- See “Writing a Formal Report” for more information
- No electronic submission required.