Experiment 3 - Reduction of a Ketone

OBJECTIVE

To learn a versatile reaction for the reduction of a ketone (or aldehyde) to an alcohol.

INTRODUCTION

The carbonyl group (C=O) found in aldehydes, ketones, carboxylic acids, esters, amides and other functional groups, plays a major role in determining the chemistry of these functional groups. This is due to the polar nature of the carbon-oxygen bond and to the presence of the relatively weak pi bond.

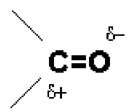

Figure 1

From the polarity of the bond, the direction of attack of many reagents can be predicted. Nucleophiles, such as hydride (H^-), cyanide (^-CN), and ammonia ($:NH_3$) attack the partially positive, *electrophilic carbon* of the carbonyl group. On the other hand, electrophiles, such as a proton (from the hydronium ion, H_3O^+) react with the partially negative, nucleophilic oxygen.

For the reduction of aldehydes and ketones, many different reducing agents are available. However, the most convenient and least expensive is probably sodium borohydride ($NaBH_4$). This reagent does not require very dry reagents and solvents like the very useful but much more reactive $LiAlH_4$ does. In fact, $NaBH_4$ may be used in aqueous solution.

In this experiment we will reduce fluorenone to fluorenol using $NaBH_4$ in methanol.

Equation

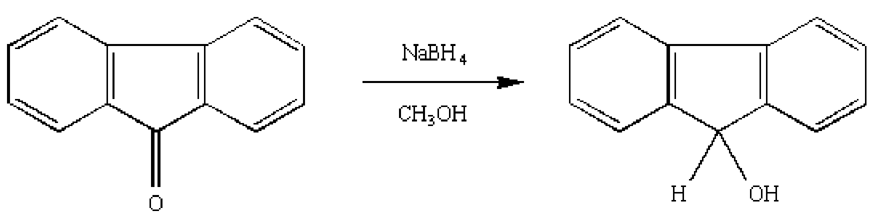

Figure 2

Stoichiometry Note

- Check your lecture textbook or online materials for a discussion of the reaction of sodium borohydride. Each of the four hydrides in $NaBH_4$ is "active" and capable of reducing the carbonyl group of a ketone.

- How many 'moles' of $NaBH_4$ will be required to reduce one mole of ketone?

PRE-LAB

Complete the pre-lab assignment in WebAssign.

PROCEDURE

In a 50 mL Erlenmeyer flask, dissolve about 0.500 g (note the actual amount used) of fluorenone in 8 - 10 mL of methanol. The contents may need to be warmed to completely dissolve the ketone. Quickly weigh between 0.040 g and 0.060 g of sodium borohydride. ($NaBH_4$ absorbs moisture from the atmosphere; therefore, weigh as rapidly (albeit accurately) as possible). Add the solid to the ketone solution in one portion. Swirl vigorously to dissolve. With intermittent swirling, let the reaction mixture stand at room temperature for a period of 15 minutes during which it will turn from yellow to colorless.

Add 5 mL of water (solid will form) and heat the reaction mixture to boiling (60°C - 65°C (hot plate)). Occasionally remove the mixture from the hot plate and swirl vigorously. After 5 minutes, remove the mixture from the hot plate and let cool to room temperature. Collect the crude 9-fluorenol via vacuum filtration. Wash the solid with ice-cold 50% aqueous methanol. Allow it to dry (spread the solid out on the tile). Determine the mass and melting point of the product. Also, determine purity of product as well as presence of unreacted starting material by TLC analysis in 7:3 hexane:acetone. The product can be recrystallized from a small amount of 75% aqueous methanol.

IN-LAB QUESTIONS

Download and print the worksheet. You will use this worksheet to record your answers to the In-Lab questions.

Questions

Record the following data.

Question 1: Amount of fluorenone ________________ g, ________________ mol

Question 2: Amount of sodium borohydride ________________ g, ________________ mol

Question 3: Amount of hydride ________________ mol

Question 4: Theoretical yield of 9-fluorenol ________________

Question 5: Show calculations for questions 1 - 4.

Question 6: Actual yield of 9-fluorenol ________________ g, ________________ mol

Question 7: Percentage yield ________________

Question 8: Melting point of 9-fluorenol ______________ (observed), ______________ (reported)

Question 9: R_f values: Fluorenone ________________ , 9-fluorenol ________________

