

EXAMPLE OF LABORATORY WRITE-UP – CHM 2201/2202

Experiment Title: Reduction of Benzophenone

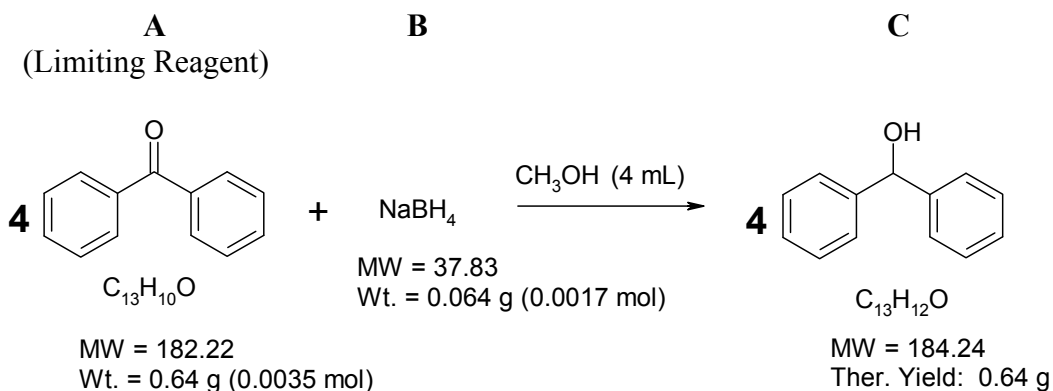
Date: September 23, 2007

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Statement of Purpose: To Reduce Benzophenone to Diphenylmethanol using Sodium Borohydride (*page 216 in Bell, Clark & Taber*)

Pre Lab

- Reaction Sequence/Theoretical Yield



(To ensure that you understand how to calculate the theoretical yield of product based on the above reaction sequence and stoichiometry, calculate the Theoretical Yield using quantities specified in text)

Calculation of Theoretical Yield:

$$0.64 \text{ g A} \times \frac{1 \text{ mole A}}{182.22 \text{ g A}} \times \frac{4 \text{ moles C}}{4 \text{ moles A}} \times \frac{184.24 \text{ g C}}{1 \text{ mole C}} = 0.64 \text{ g C}$$

- Physical/Chemical Properties

| Table of Physical Properties of Reagents and Solvents | | | | |
|--|-------------------|------------------|---------------------------|---------------|
| Reagent/Solvent | Molecular Formula | Molecular Weight | Melting/Boiling Point, °C | Density, g/mL |
| Benzophenone | $C_{13}H_{10}O$ | 182.22 | mp = 47 - 51 | |
| Methanol | CH_3OH | 32.04 | bp = 65 | 0.791 |
| Sodium Borohydride | $NaBH_4$ | 37.83 | mp > 300 | |
| Diphenylmethanol | $C_{13}H_{12}O$ | 184.24 | mp = 65 - 67 | |

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Pre Lab (continued)

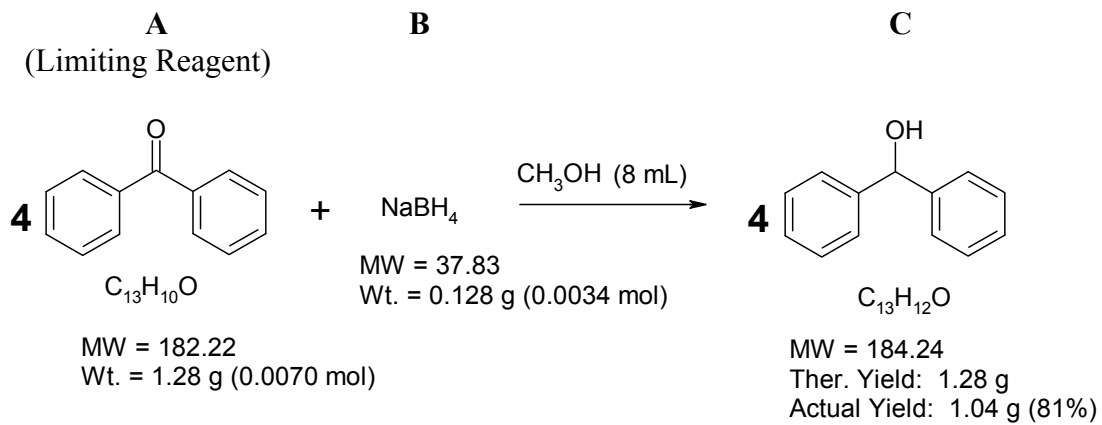
Note: Sodium Borohydride is moderately unstable to water, so protect from moisture while weighing. Sodium borohydride reacts vigorously with acid with the evolution of hydrogen gas, so do not expose to acid. Use the NaBH₄ immediately after weighing.

- Procedure Outline

1. Place ~0.64 g of benzophenone and ~4 mL of methanol in a 25 mL Erlenmeyer flask.
2. If necessary, warm the mixture to dissolve the benzophenone and then allow to cool back to room temperature.
3. To the benzophenone solution, add in one portion ~ 0.64 g of sodium borohydride.
4. Swirl the flask and allow the reaction mixture to stand at room temperature for 20 min.
5. Add 2 mL of cold water and warm on a steambath for 5 minutes.
6. Cool the resulting mixture in an ice bath to precipitate a solid.
7. Collect the solid using vacuum filtration and air dry.
8. Determine the weight and melting point of the solid.
9. Calculate the % yield of product and compare mp with literature mp of diphenylmethanol.
10. Obtain and interpret an infrared spectrum of product.

Laboratory Observations

(Redraw the reaction sequence and use actual quantities employed in the reaction to calculate the theoretical yield)



Laboratory Observations (continued)

Experimental Procedure:

To a 25 mL Erlenmeyer flask was added 1.28 g (0.0070 mole) of benzophenone and 8 mL of methanol. The solid did not completely dissolve, so the flask was swirled and gently warmed on a steam bath until a clear solution was obtained. The flask was then allowed to stand and to cool back down to room temperature. After cooling to room temperature, 0.128 g (0.0034 mole) of sodium borohydride was added in one portion to the flask and it was swirled briefly until the solids dissolved. The reaction mixture was allowed to stand at room temperature for 20 minutes with occasional swirling. The reaction mixture bubbled slightly when the solution was swirled.

After the 20 minute reaction period had elapsed, 4 mL of cold water was added to the reaction mixture and the resulting solution was boiled gently on a steam bath for 5 minutes. Care was taken to not overheat the flask so that the methanol was not boiled off. The resulting hot solution was then allowed to cool to room temperature and subsequently cooled in an ice bath. On cooling a small amount of white crystalline solid precipitated. After chilling in an ice bath for 10 minutes, there did not appear to be a significant amount of precipitated solid so 1 mL of cold water was added to the mixture to attempt to force more solid to precipitate. The resulting mixture was chilled in an ice bath for another 10 minutes and the flask's contents were stirred/agitated with a glass rod to facilitate crystallization. A significant amount of additional solid formed during this process and the solids were then collected using a Hirsch funnel and vacuum filtration. The collected solids were allowed to remain on the funnel and air was continued to be drawn through the solids to air dry the material. (During this drying process, an additional quantity of white solid precipitated in the filtrate in the filtration flask. This newly precipitated material was not pursued and was eventually discarded.) After the collected solids were dry and free flowing, they were weighed to provide 1.04 g of the product as a white crystalline solid, mp 64 – 66 °C.

An infrared spectrum (attach spectrum to the write-up that you hand in to be graded) of the product was obtained and the characteristic carbonyl absorption (1670 cm^{-1}) for benzophenone starting material was absent indicating that the reduction to the alcohol product (strong OH absorption bands present at $3300\text{-}3400\text{ cm}^{-1}$) was complete.

Conclusions

This reaction of benzophenone with sodium borohydride to form diphenylmethanol is an example of a sodium borohydride reduction of a ketone to an alcohol. The reaction was relatively easy to run and provided a fairly good yield of the desired product.

The reaction appeared to completely convert most, if not all, of the benzophenone to the desired diphenylmethanol. This conclusion is based on:

1. The absence of a carbonyl absorption and the appearance of strong OH absorption bands in the infrared spectrum of the purified product.

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Conclusions (continued)

2. The melting point (64 – 66° C) of the obtained product compares favorably with the literature mp value (65 - 67° C) of the expected diphenylmethanol.

According to the above equation for the borohydride reduction of benzophenone, there is an excess of borohydride in this reaction; consequently, benzophenone is the limiting reagent (LR) and it is used to calculate the theoretical and actual % yields as follows:

$$1.28 \text{ g A} \times \frac{1 \text{ mole A}}{182.22 \text{ g A}} \times \frac{4 \text{ moles C}}{4 \text{ moles A}} \times \frac{184.24 \text{ g C}}{1 \text{ mole C}} = 1.28 \text{ g C (Theoretical Yield)}$$

$$\text{Actual \% yield} = \frac{1.04 \text{ g product obtained}}{1.28 \text{ g (Ther. Yield)}} \times 100 = 81\% \text{ yield}$$

While the yield of desired product was relatively good in this experiment, the yield could have possibly been increased by the addition of slightly more water to force more of the product out of solution during the crystallization procedure; however, this may have caused more impurities to co-precipitate with the product and decreased its purity slightly. Overall, the yield and purity of product from this experiment is completely satisfactory for a reaction of this type.

INFRARED SPECTRA

