

Aldol Condensation - Synthesis of Dibenzalacetone

March 19 –23

Background

1. Organic Chemistry Textbook (Smith):
 - Carbonyl Condensation Reaction: Section 24.1 - 24.2

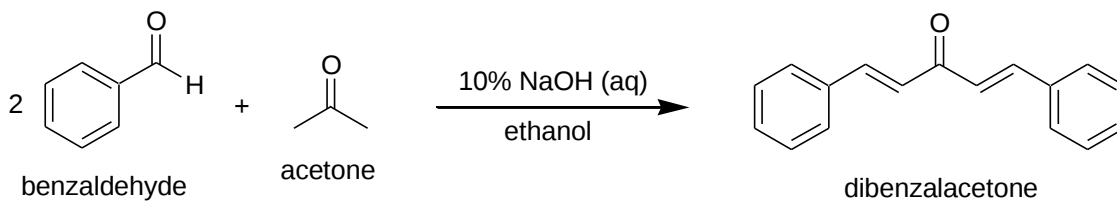
Major Techniques

1. Melting point
2. IR Spectroscopy

Experiment

You will carry out the Aldol Condensation of benzaldehyde with acetone to give dibenzalacetone. The product will be recrystallized and analyzed by melting point and IR spectroscopy.

Principle Reaction



Prelab

Prepare a common purpose, reagent data table, safety precautions list, and procedure for the synthesis, purification and analysis of dibenzalacetone. Calculate the amount of benzaldehyde required for the reaction (see procedure) and the amount of solid NaOH to prepare a 50 mL aqueous solution of 10% NaOH. Show calculations.

Procedure

- Preparation of Dibenzalacetone
 1. In a 125-mL Erlenmeyer flask add 20 mL of 95% ethanol and 25 mL of the 10% NaOH solution. Cool to 20°C. (If you don't have a 10% NaOH solution in your lab drawer, prepare 50 mL of 10% NaOH solution per hood).
 2. In a test tube, add 1.0 mL of acetone and 2.2 equivalents of benzaldehyde. Swirl the contents to mix the two organics.
 3. Pour half the acetone-benzaldehyde solution into the solution from step 2. Swirl periodically over the next 15 minutes. Then add the remaining portion of the acetone-benzaldehyde solution. Rinse the test tube with two 1 mL aliquots of the 95% ethanol to complete the transfer of the benzaldehyde-acetone solution. Swirl periodically over the next 20 minutes. A yellow flocculent precipitate should form.
 4. Cool in an ice-bath for 5 minutes.
 5. Collect the solid using suction filtration. Wash the solid with 50 mL of deionize water (to dissolve any sodium hydroxide). Allow air to be pulled through the solid for 5 minutes to

- help dry it. Remove the solid from the Büchner funnel and press between two sheets of filter paper to further dry the solid.
- Recrystallize your product from 95% ethanol (need about 2.5 mL of ethanol per gram of compound).
 - Collect the solid using suction filtration. Rinse the solid with 1 mL of cold 95% ethanol.
 - Do a melting point of the product and IR spectroscopic analysis your dibenzalacetone product.

Waste Disposal

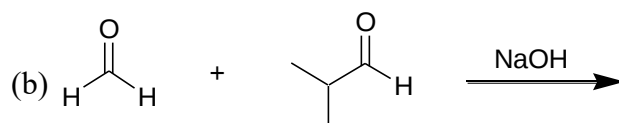
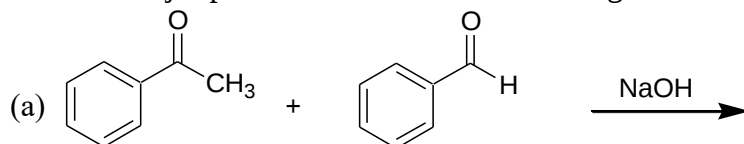
- The filtrates may be discarded down the drain in the hood with copious amounts of water.
- The dibenzalacetone may be placed in the appropriately labeled container.

Results

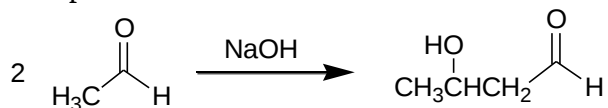
- Report all yields/calculations, recoveries from recrystallization, m.p. and other data. Tabulate the important IR stretches for acetone, benzaldehyde and dibenzalacetone.

Questions

- Give the reaction equation and product expected if cyclohexanone was used in place of acetone in the reaction.
- Given that heat is typically required to give the dehydration product, explain why this wasn't necessary for your reaction.
- Draw the enolate formed in the reaction of acetone with hydroxide.
- Give the major product of each of the following reactions.



- Give the mechanism for the following reaction. Use electron-pushing arrows to show electron movement. Show pertinent resonance structures.



- From your mechanism, what role does NaOH play? Explain.