Aldol Condensation: Synthesis of Dibenzalacetone

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Organic Chemistry Lab II

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Abstract:

The purpose of this experiment was to synthesize dibenzalacetone via an aldol condensation reaction between acetone and benzaldehyde. This was done by mixing the two reactants with NaOH and ethanol, then allowing the reaction to sit for thirty minutes. The crystals were then washed with water three times and recrystallized using ethanol. It was then characterized using melting point analysis. The percent yield for this reaction was 59.84%. This was due to loss of crystals during recrystallization and during solvent removal from the reaction mixture. The observed melting point was 104 – 107.5°C, compared to a literature value of 110°C. The lower and broader observed melting point may have been due to the product still being wet. It may also be due to unevaporated ethanol or other impurities in the product. However, the observed melting point was close to the literature value, and it can thus be concluded that the product was dibenzalacetone. Thus, the aldol condensation reaction was successful.

Aldol Condensation: Synthesis of Dibenzalacetone

Introduction: The purpose of this experiment is to synthesize dibenzalacetone via an aldol condensation reaction between acetone and benzaldehyde. The product will be recrystallized using ethanol, then characterized using melting point analysis.

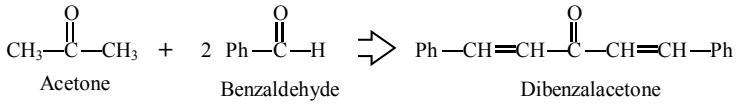
Materials Used:

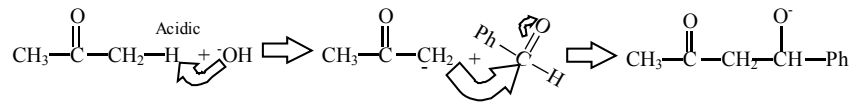
2 beakers, 100-mL	melting point capillary tubes
2 vials, 5-mL	100 µL micropipet
10-mL Erlenmeyer flask	Pasteur pipettes, plastic
25-mL Erlenmeyer flask	glass Pasteur pipettes
filter paper	glass stirring rod
rubber bulb	Hirsch funnel
support stand	clamp
sand bath	watch glass
ice (for ice bath)	

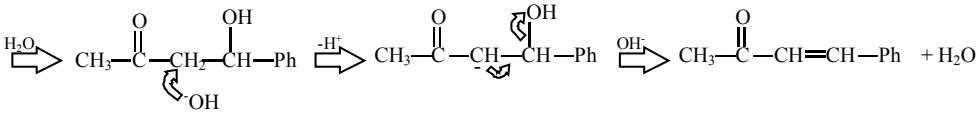
Reagents and Properties:

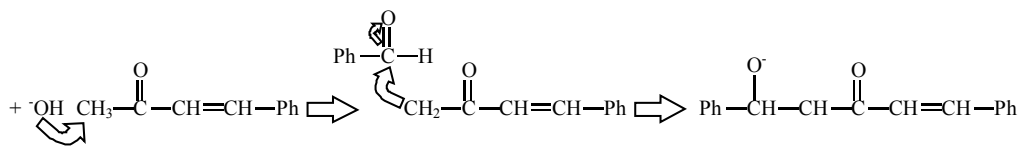
Substances	Formula Weight, g/mol	Amount Used	Moles Used	Mole Ratio	Melting Point °C	Boiling Point °C	Density g/mL
Acetone	58.08	38 µL	5.17×10^{-4}	1 to 1	N/A	56	0.731
Benzaldehyde	106.12	100 µL	9.84×10^{-4}	1 to 1	N/A	178	1.044
Dibenzalacetone	224	Product	N/A	N/A	110	N/A	N/A
ethanol	46.07	2.8 mL	0.048	N/A	N/A	78.5	0.791
10 % Sodium Hydroxide	40.00	1.0 mL	0.05325	N/A	N/A	N/A	2.130

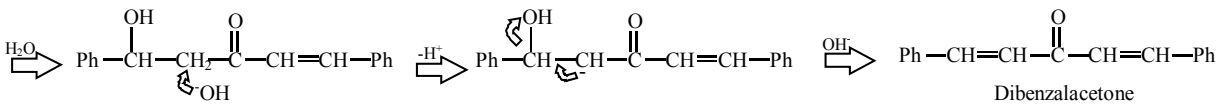
Reaction and its Mechanism:











Aldol Condensation: Synthesis of Dibenzalacetone

Procedure:

Part 1 – Condensing Acetone with Benzaldehyde

Prepare an ice-water bath in a 100-mL beaker. Place 0.75 mL ethanol and 1.0 mL 10% NaOH in a 10-mL test tube. Place this tube in the ice-water bath to cool to approximately 20 °C. Remove the tube from the ice bath. Prepare a mixture of 100 µL fresh benzaldehyde and 38 µL acetone. Add this mixture to the ethanol-NaOH solution in two separate portions, approximately 5 minutes apart. Then, let the solution sit for 30 minutes, stirring occasionally.

Once the thirty minutes is up, cool the mixture in the ice-water bath. Then, use a glass Pasteur pipet and rubber bulb to remove the solvent from the tube. Add 2 mL distilled water to the tube in order to wash the crystals. Remove the water using the Pasteur pipet. Rinse the crystals two more times with 1 mL portions of distilled water each time. Make sure you remove all the solvent from the crystals.

Part 2 – Recrystallization

Prepare a hot water bath by placing approximately 50 mL of water into a 100-mL beaker. Heat the water to boiling using a sand bath. Add 0.5 mL ethanol to the reaction tube. Place the tube in the hot water bath and stir continually for awhile. After several minutes, if the solid in the tube has still not dissolved, add another 0.5 mL portion of ethanol to the tube. Continue stirring the solution as the tube sits in the hot water bath. After several more minutes, if the solid still has not gone into solution, add 0.1 mL portions of ethanol to the test tube until all the solid has dissolved. Do NOT add more than 2.0 mL ethanol to the test tube. If solid impurities remain, use a Pasteur pipet to transfer the solution to another test tube. If everything in the test tube goes into solution, remove the tube from the hot water bath and allow to cool to room temperature. If necessary, scratch the inside of the tube with a glass stirring rod to induce crystallization. Once the tube has cooled to room temperature, cool the tube in an ice-water bath for 5 – 10 minutes, allowing crystallization to complete. Collect the crystals via vacuum filtration with a Hirsch funnel. Then, spread the crystals on a watch glass and allow them to dry for a couple days.

Part 3 – Characterization and Clean-Up

Prepare a melting point capillary tube of the product and measure the melting point of the product. Place all waste materials in their appropriate containers.

Data and Calculations:

Mass product crystals: 0.069 g

Theoretical Yield: 0.116 g
Percent Yield: 59.48%

Observed melting point: 104 – 107.5 °C
Literature value: 110 °C

Finding Limiting Reagent

$$\text{Acetone} \quad \frac{(38 \times 10^{-6}\ \text{L A})}{} \left| \frac{(1000\ \text{mL})}{(1\ \text{L})} \right| \frac{(0.791\ \text{g})}{(1\ \text{mL})} \left| \frac{(1\ \text{mol A})}{(58.08\ \text{g A})} \right| \frac{(1\ \text{mol pdt})}{(1\ \text{mol A})} \left| \frac{(224\ \text{g pdt})}{(1\ \text{mol pdt})} \right.$$

$$= 0.116\ \text{g dibenzalacetone}$$

$$\text{Benzaldehyde} \quad \frac{(100 \times 10^{-6}\ \text{L B})}{} \left| \frac{(1000\ \text{mL})}{(1\ \text{L})} \right| \frac{(1.044\ \text{g B})}{(1\ \text{mL B})} \left| \frac{(1\ \text{mol B})}{(106.12\ \text{g B})} \right| \frac{(1\ \text{mol pdt})}{(1\ \text{mol B})} \left| \frac{(224\ \text{g pdt})}{(1\ \text{mol pdt})} \right.$$

$$= 0.220\ \text{g dibenzalacetone}$$

The limiting reagent for this reaction is acetone. It yields the least amount of dibenzalacetone in this reaction, and therefore is the limiting reagent.

Aldol Condensation: Synthesis of Dibenzalacetone

Theoretical Yield

$$\text{Acetone} \quad \frac{(38 \times 10^{-6}\ \text{L A})}{(1\ \text{L})} \left| \frac{(1000\ \text{mL})}{(1\ \text{mL})} \right| \frac{(0.791\ \text{g})}{(58.08\ \text{g A})} \left| \frac{(1\ \text{mol A})}{(1\ \text{mol pdt})} \right| \frac{(224\ \text{g pdt})}{(1\ \text{mol pdt})}$$

$$= 0.116\ \text{g dibenzalacetone}$$

Percent Yield

$$\frac{(\text{mass of dibenzalacetone crystals})}{(\text{theoretical yield})} \rightarrow \frac{(0.069\ \text{g dibenzalacetone})}{(0.116\ \text{g dibenzalacetone})} \times (100) =$$

$$= 59.48\%\ \text{yield}$$

Results and Conclusions:

The aldol condensation reaction between acetone and benzaldehyde was successful and yielded dibenzalacetone. The percent yield for this reaction was 59.48%. This was low due to the fact that many crystals were lost during the removal of the solvent from the reaction mixture in Part 1. Some crystals were also lost during the washing of the crystals with water. When the water was removed, some crystals got stuck in the Pasteur pipet with the water, and thus did not contribute to the percent yield. Also, not all of the crystals remained on the filter paper. Some filtered right through the paper with the solvent, and ended up in the flask. Thus, these crystals were not included in the percent yield either.

The observed melting point of the product was 104 – 107.5°C. This was relatively close to the literature melting point value of 110°C. The observed melting point may have been lower because the product may still have been wet or there may have been impurities in the product, which might have included some ethanol that was not completely evaporated from the crystals during recrystallization. However, the melting point was close to the literature value, thus indicating that the obtained product was dibenzalacetone and the reaction was successful.

Reference:

Chemistry Lab Experiments CHEM 224 SYNT 720 pgs. 85 - 95
By Wigal/Manion/LeFevre/Wade, Jr./Rapp/Lee/Wikholm

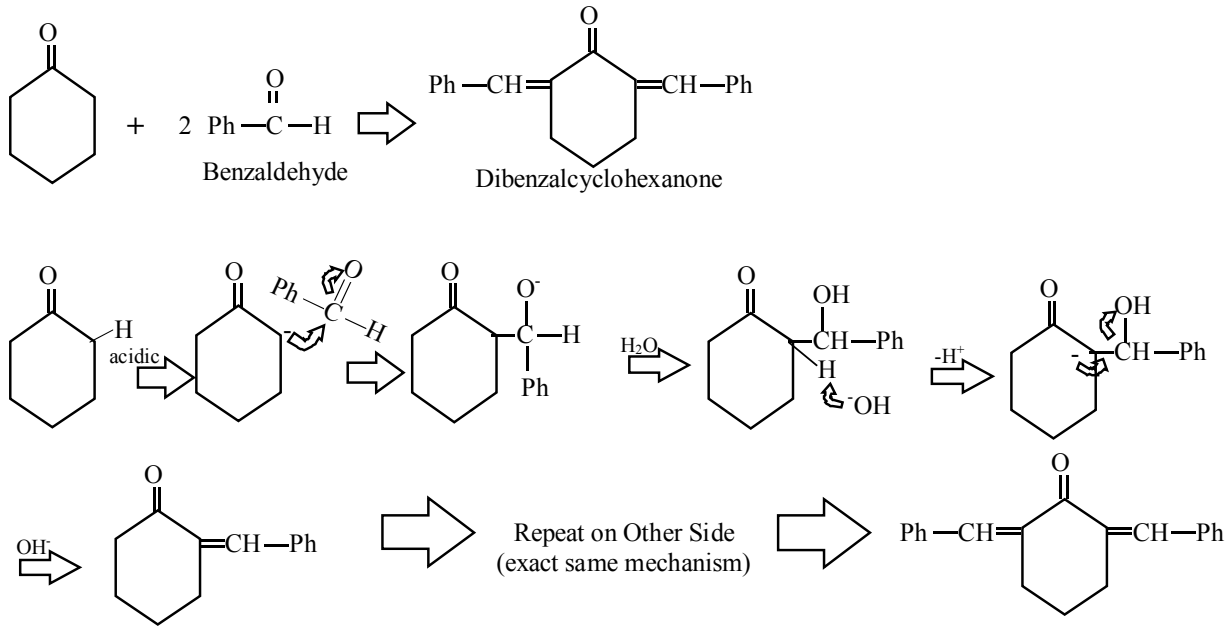
Weast, Robert C., ed. CRC Handbook of Chemistry and Physics. 70th ed. Boca Raton, FL: CRC Press, Inc., 1990.

Aldol Condensation: Synthesis of Dibenzalacetone

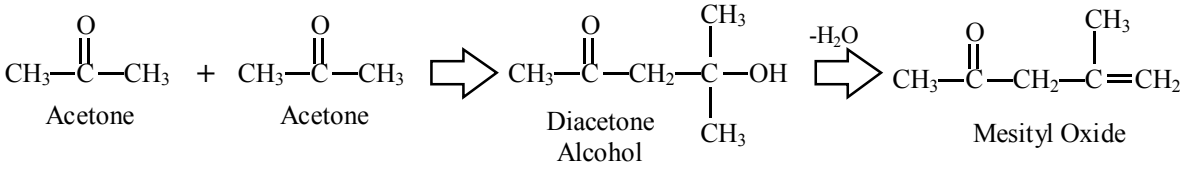
Post-Lab Questions

1. The percent yield for this experiment was 59.48%. Please see calculations section for explanation.

2. The mechanism of the base-catalyzed condensation of cyclohexanone with two molecules of benzaldehyde to yield dibenzalcyclohexanone is as follows:



3. If the student added a two-fold excess of acetone, the acetone would react with itself and the product that would be isolated would be diacetone alcohol or mesityl oxide. The first product formed is diacetone alcohol, but it is dehydrated to mesityl oxide.



4. Dibenzalacetone is a conjugated molecule. This conjugation allows the molecule to absorb sunlight and UV rays. Thus, when dibenzalacetone is used in sunscreen, the conjugation in the molecule allows the dibenzalacetone, and thus the sunscreen, to absorb the sunlight before it reaches the skin, thus preventing sun burns from occurring.