Exp 6 - Aldol Reactions

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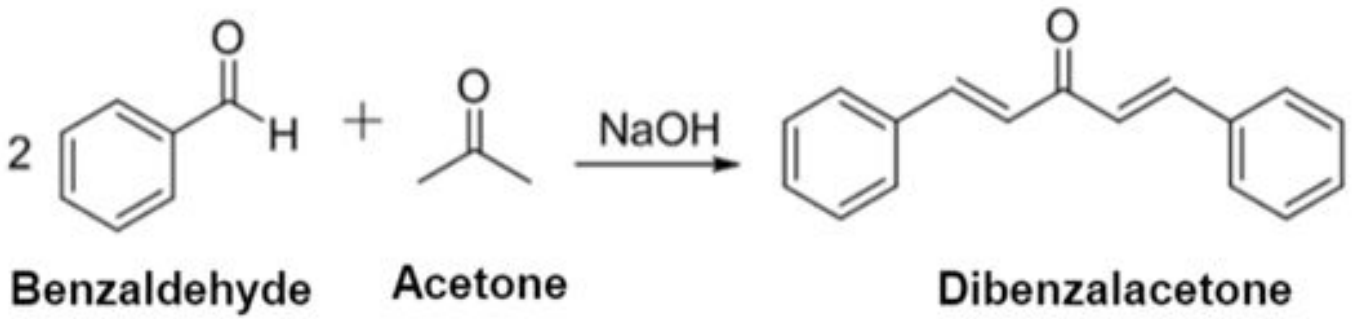
I pledge my honor that I have abided by the Stevens Honor System.

Purpose and Reaction

The purpose for this experiment was the Preparation of Dibenzalacetone from acetone and benzaldehyde via Aldol condensation reaction.



Reagents

Name	**M.W.** (0.5 pts)	**Density** (0.5 pts)	**Amount (grams/mL)** (0.5 pts)	**Moles** (0.5 pts)	**Hazards/Precautions** **(MSDS data) and melting point or boiling point** (2 pts)	**Role of the reagent** (1 pts)*
Benzaldehyde	106.12 g/mol	1.04 g/cm³	0.82 g/ 0.8 mL	0.008 mole	Combustible liquid. Harmful if swallowed or in contact with skin. Causes skin irritation. May cause an allergic skin reaction BP: 352.6°F (178.1°C) @760 mmHg	Reactant
acetone	58.08 g/mol	784 kg/m³	0.24 g/ 0.3 mL	0.004 mole	Highly flammable liquid and vapor. Causes serious eye irritation. May cause drowsiness or dizziness. BP: 132.8°F (56°C) @760 mmHg	Solvent
Dibenzalacetone	234.29 g/mol	1.1±0.1 g/cm3			May cause eye, skin, and respiratory tract irritation. The toxicological properties of this material have not been fully investigated. BP: 130 °C @760 mmHg	Product

Reagents

sodium hydroxide	39.997 g/mol	2.13 g/cm³	0.4 g	0.001 mole	Corrosive. May cause skin burn and eye damage. **Melting Point:** 604.4°F (318° C)	Catalyst
ethanol	46.07 g/mol	789 kg/m³	2 mL	1.578	Flammable liquid and vapor. Toxic, both by ingestion and inhalation. May be fatal or cause blindness if swallowed. BP: 173.1°F @760 mmHg	Solvent
ethyl acetate	88.11 g/mol	902 kg/m³			**Flammable liquid and vapor.** Causes eye irritation. Breathing vapors may cause drowsiness and dizziness. May cause respiratory tract irritation. Prolonged or repeated contact causes defatting of the skin with irritation, dryness, and cracking. BP: 170.8°F @760 mmHg	Solvent for recrystallization
Chloroform	119.38 g/mol	1.49 g/cm³			Harmful if swallowed. Causes skin irritation. Causes serious eye irritation. Toxic if inhaled. May cause drowsiness or dizziness. Suspected of causing cancer. BP: 142.2°F (61.2°C) @760 mmHg	use only if run IR

Procedure

1. In a 50 mL Erlenmeyer flask prepare solution of 0.4 g (0.001 mole) of sodium hydroxide in 2 mL of water and 2 mL of 95% ethanol (5% is water).

2. After the solution has cooled, add 0.3 mL (0.24 g, 0.004 mole) of acetone and then 0.8 mL (0.82 g, 0.008 mole) of benzaldehyde.

3. A yellow turbidity will appear almost immediately, which quickly turns into a flocculent precipitate.

4. Swirl the flask from time to time (stir) over a 15-min period.

5. Collect the mushy reaction product on a Buchner funnel and wash it first with water and then little chilled 95% Ethanol.

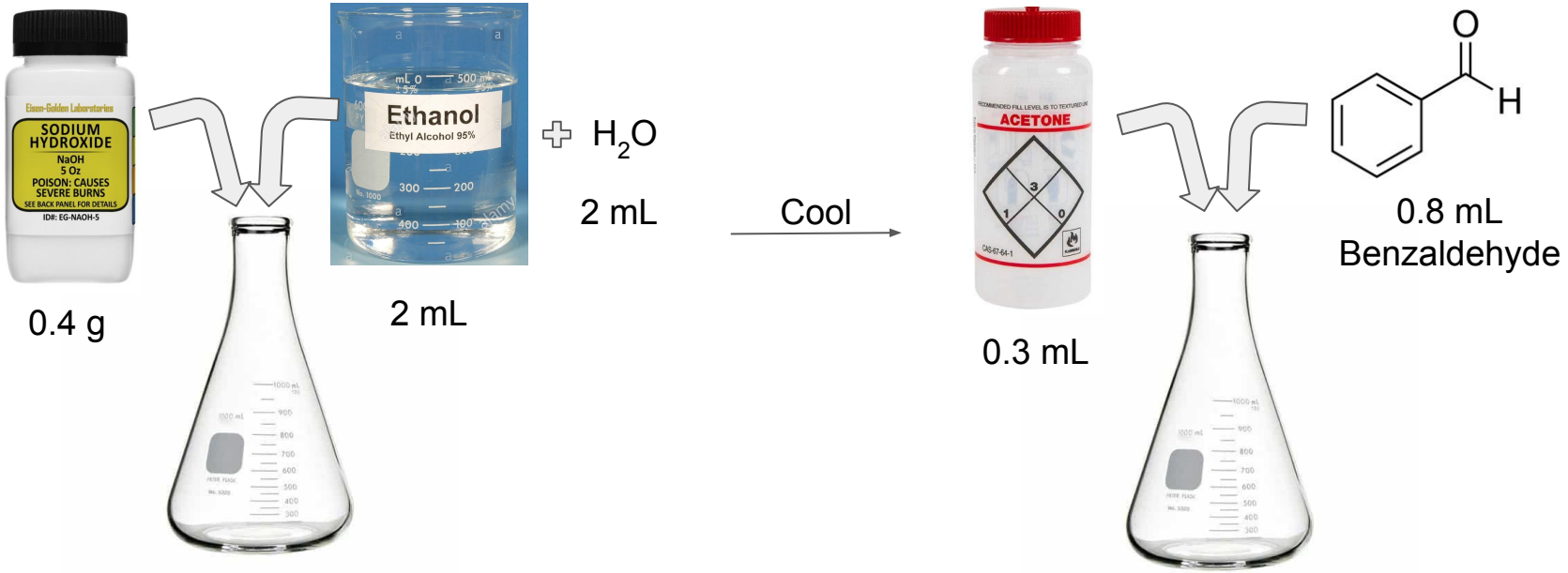
6. Dry the product.

7. Recrystallize it from ethyl acetate using about 2.5 g of the solvent per gram of the product.

8. Determine the melting point of the recrystallized product.

Stepwise Procedure

1. In a 50 mL Erlenmeyer flask prepare a solution of 0.4g of sodium hydroxide in 2 mL of water and 2 mL of 95% ethanol
2. After the solution has cooled, add 0.3 mL of acetone and then 0.8 mL of benzaldehyde



Stepwise Procedure

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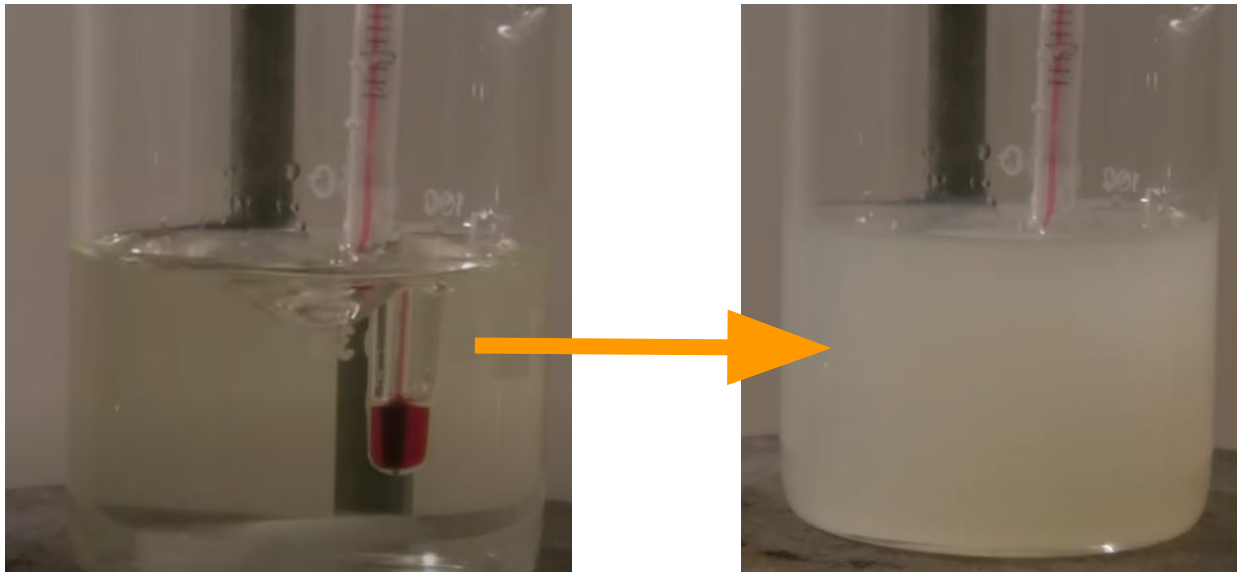
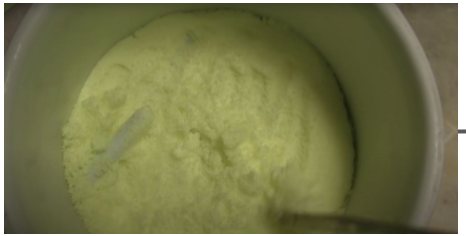


Stepwise Procedure

5. Collect the mushy reaction product on a Buchner funnel and wash it first with water and then little chilled 95% Ethanol.

6. Dry the product.

Collect Product

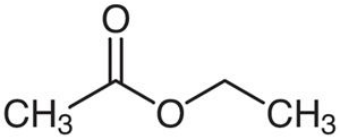


Wash with Water & 95% Ethanol

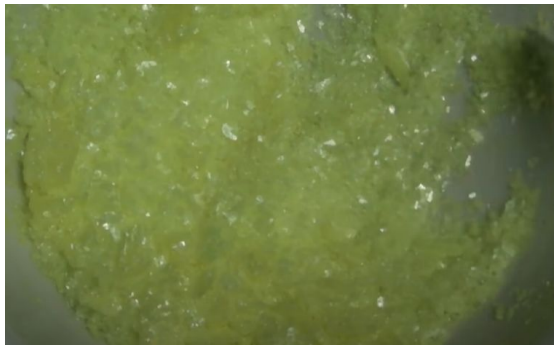
Dry

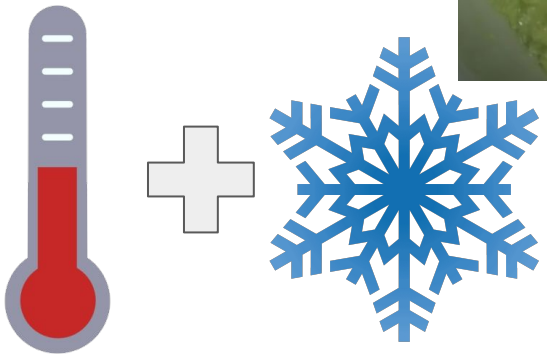
Stepwise Procedure

7. Recrystallize it from ethyl acetate using about 2.5 g of the solvent per gram of the product.
8. Determine the melting point of the recrystallized product.



Recrystallize
Product





Determine
Melting Point

Results

Expected Yield:

$$0.8\ \text{mL benzaldehyde} * \frac{1.0415\ \text{g benzaldehyde}}{1\ \text{mL}} * \frac{1\ \text{mol benzaldehyde}}{106.041\ \text{g}} * \frac{1\ \text{mol dibenzalacetone}}{2\ \text{mol benzaldehyde}} * \frac{234.30\ \text{g dibenzalacetone}}{1\ \text{mol dibenzalacetone}} = 0.920\ \text{g dibenzalacetone}$$

Actual Yield: 0.17g

Percent yield: 0.17g / 0.920g = 18.48%

Melting point determination: 109-110℃

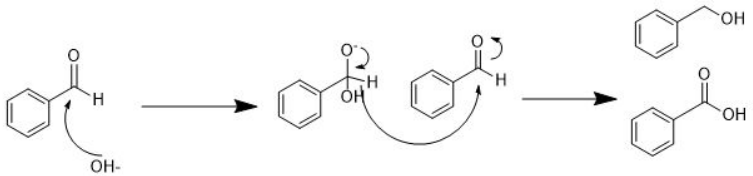
Conclusion

In this experiment, we learned how to conduct an base-catalyzed aldol condensation reaction by using acetone and benzaldehyde to form dibenzalacetone using sodium hydroxide as the catalyst. In isolating the product, we practiced techniques such as vacuum filtration and recrystallization, as well as melting point determination to confirm the identity of the product. The measured melting point of 109-110℃ is in the range of the expected melting point of dibenzalacetone, which is 107-114℃. There were no issues or further recommendations for this experiment, as it was conducted remotely. Applications of the dibenzalacetone formed in this experiment is as an ingredient in sunscreens and a ligand in organometallic chemistry.

Post Lab Questions

1. **What will happen if do not add any acetone to the reaction mixture and only benzaldehyde and NaOH were present in the 95% ethanol solvent. Will there be any reaction? If yes, draw the reaction. What is the name of such reactions? (2 points)**

The Cannizzaro reaction will happen between the OH^- ion and the benzaldehyde, creating benzyl alcohol and benzoic acid (or sodium benzoate).



2. **Instead of just doing filtration at the end of the reaction if you have to do the work-up using extraction (using dichloromethane and water) then briefly describe how would perform the work-up and isolate the product. (2 points)**

The product would be soluble in dichloromethane, and not the water. Therefore, I would use a separation funnel and mix the two solvents together in the funnel. Because DCM is denser than water, I would remove the bottom layer and repeat a couple more times by washing with more DCM. Then, I could distill off the extracted DCM as well as other leftover reactant to isolate the solid product.