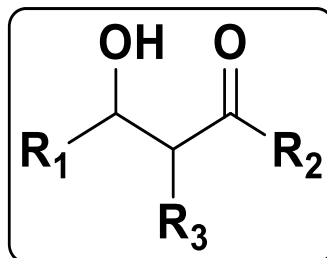


EXPERIMENT 9

Aldol Condensation **Synthesis of dibenzalacetone**

Aldol Condensation : Synthesis of dibenzalacetone

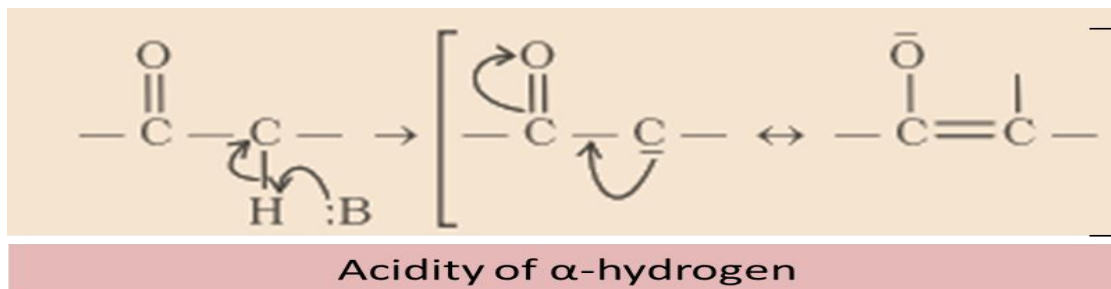


Generalized structure of the aldol moiety

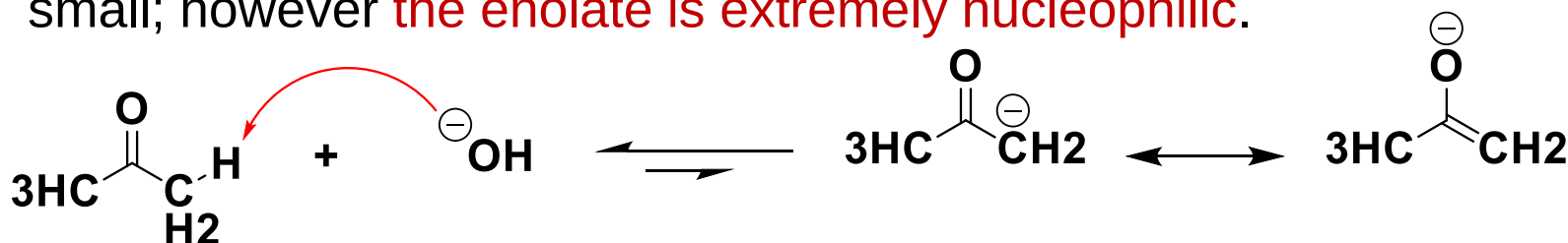
- **Aldol: Aldehyde and alcohol**
- A class of compounds containing both an alcohol and an [aldehyde](#) functional group, formed by a condensation reaction between aldehyde or ketone molecules.
- The purpose of this experiment is synthesis of dibenzalacetone (trans, trans-1,5-diphenyl-1,4-pentadien-3-one) through the aldol condensation of acetone with benzaldehyde. **(Claisen-Schmidt condensation).**

Aldol Condensation : Synthesis of dibenzalacetone

- The synthesis begins by using strong base to generate the acetone enolate ion. Water (not shown) is formed as a byproduct.



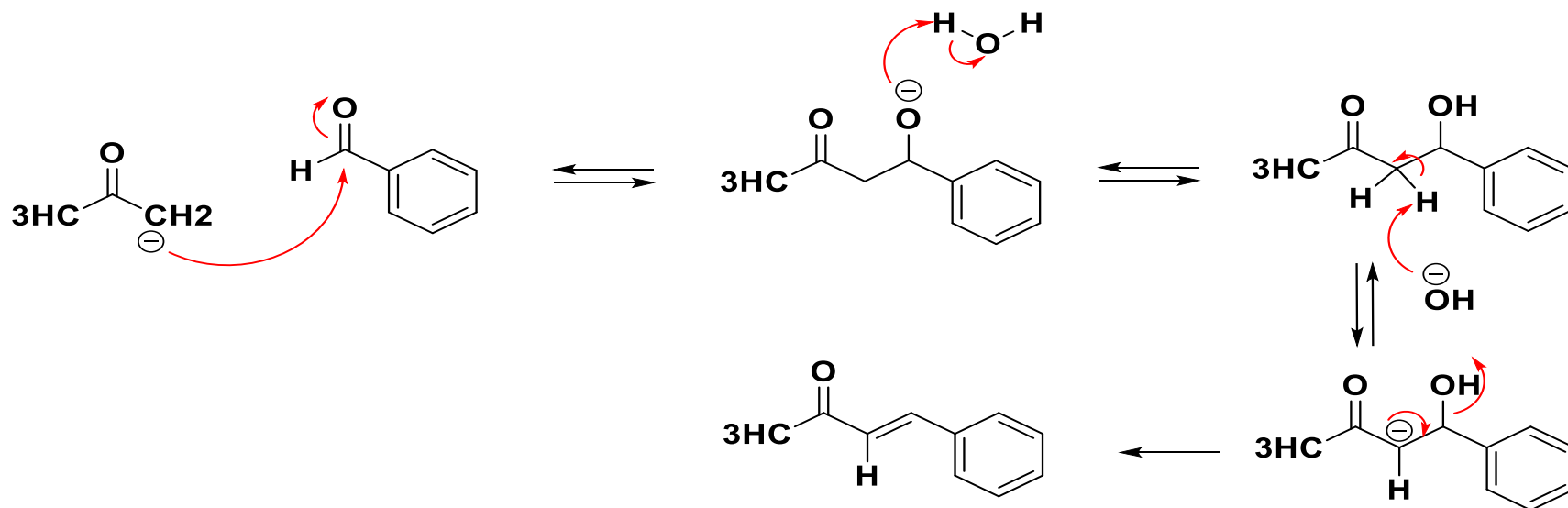
- The equilibrium position of this reaction strongly favors the starting acetone, and the amount of acetone enolate formed is quite small; however the enolate is extremely nucleophilic.



- Being a very strong nucleophile, this enolate attacks the carbonyl of benzaldehyde and forms a β -carbonyl alkoxide ion.

Aldol Condensation : Synthesis of dibenzalacetone

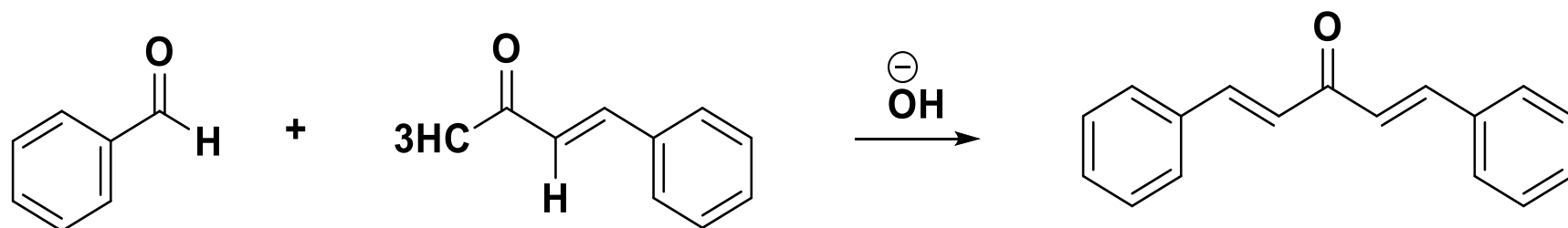
- This alkoxide ion abstracts a proton from water to form a **beta hydroxy ketone**.
- Sodium hydroxide abstracts another acidic alpha H to form a stabilized carbanion. The electron pair on carbon is used to eliminate the hydroxide ion, forming an alpha-beta unsaturated ketone in an irreversible step.



- This is an example of an **E1c** (Elimination Unimolecular conjugate Base) mechanism.

Aldol Condensation : Synthesis of dibenzalacetone

- **Note:** In this reaction, the intermediate alcohol is **dehydrated under basic conditions**, unlike most alcohol dehydrations, which are generally **E1 mechanisms under acid conditions**. The E1cb mechanism is made possible by the presence of the carbonyl, which stabilizes the intermediate carbanion.
- Since this newly formed ketone still possess **alpha hydrogens**, it too can undergo the **same enolate condensation** reaction with a second mole of benzaldehyde to form the **final product**:



Aldol Condensation : Synthesis of dibenzalacetone

- Experimental Procedure:

1. Place into a 500-mL Erlenmeyer flask the following quantities: **1.6g acetone** (2.0 ml, 0.028 mole) and **6.3g benzaldehyde** (6.0 ml, 0.059 mole) and 50 ml. of ethanol.

- **Note:** it is important to maintain a 1:2 molar ratio of acetone to benzaldehyde.

2. Add **60 ml. of 10% sodium hydroxide** and **shake the flask for 15-20 minutes**.

- **Note any color changes that may occur.**

3. The reaction mixture should be **first clear**, then it becomes **milky** and a **precipitate** forms a bit later.

4. **Isolate the yellow** precipitate by **suction filtration** using water to transfer and wash the product.

5. **Press the solid** onto the filter paper to **remove as much water** as possible, **then turn off the suction** and break up lumps of crystals with a spatula.

Aldol Condensation : Synthesis of dibenzalacetone

6. Add to the solid on the filter paper an ice-cold solution of ethanol and acetic acid (1.0 ml. of acetic acid in 25 ml of ethanol). Let it stand for 1 minute, then apply the suction filtration for an additional 5-10 minutes to allow the product to air dry. (The acid treatment removes traces of the remaining base)

7. Recrystallize the crude product:

- a) Place it in an Erlenmeyer flask of appropriate size and add enough ethanol to make a thick slurry of the crystals.
- b) Place a boiling chip in the flask (to prevent “bumping”) and warm the mixture on a hot plate.
- c) While it is boiling gently, slowly add ethanol until the crystals just dissolve.
- d) Allow the solution to cool until crystals have formed and it is no longer hot. (You may cool the mixture in ice at this point.)
- e) After crystals have stopped forming, collect the recrystallized dibenzalacetone in a Buchner funnel
- f) Wash the crystals with a **little cold ethanol**.
- g) Determine the melting point ,the weight of the crystals and the percentage yield.

Aldol Condensation : Synthesis of dibenzalacetone

Physical properties of reactants and products

Compound	Molar mass	M.p.	B.p.	Density
Benzaldehyde	106.13		178°C	1.04
Acetone	58.08		56°C	0.79
Dibenzalacetone	234.30	113°C		