

جامعة الانبار

كلية : الصيدلة

قسم : الكيمياء الصيدلانية

اسم المادة باللغة العربية: الكيمياء العضوية

اسم المدة باللغة الإنكليزية **Organic Chemistry lab**

المرحلة: الثالثة

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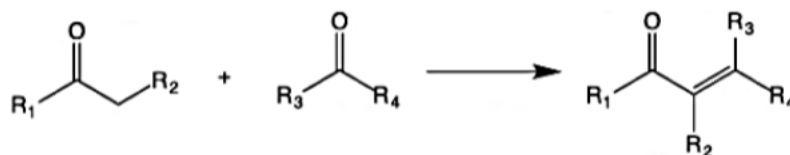
عنوان المحاضرة باللغة العربية: تجربة تحضير الداى بنزل اسيتون

عنوان المحاضرة باللغة الإنكليزية: **Synthesis of Dibenzalacetone**

Synthesis of Dibenzalacetone

Introduction:

An aldol condensation is an organic reaction in which an enol or an enolate ion reacts with a carbonyl compound to form a β hydroxyaldehyde or β -hydroxyketone, followed by a dehydration to give a conjugated enone (Mahrwald, 2004). The following equation is an example of an Aldol condensation:



Aldol condensations are important in organic synthesis, providing a good way to form carbon – carbon bonds. The Robinson annulation reaction sequence features an aldol condensation; the Wieland Miescher ketone product is an important starting material for many organic syntheses.

Aldol condensations are also commonly discussed in university level organic chemistry classes as a good bond-forming reaction that demonstrates important reaction mechanisms. In its usual form, it involves the nucleophilic addition of a ketone enolate to an aldehyde to form a β -hydroxy ketone, or "aldol" (aldehyde and alcohol), a structural unit found in many naturally occurring molecules and pharmaceuticals. The name aldol condensation is also commonly

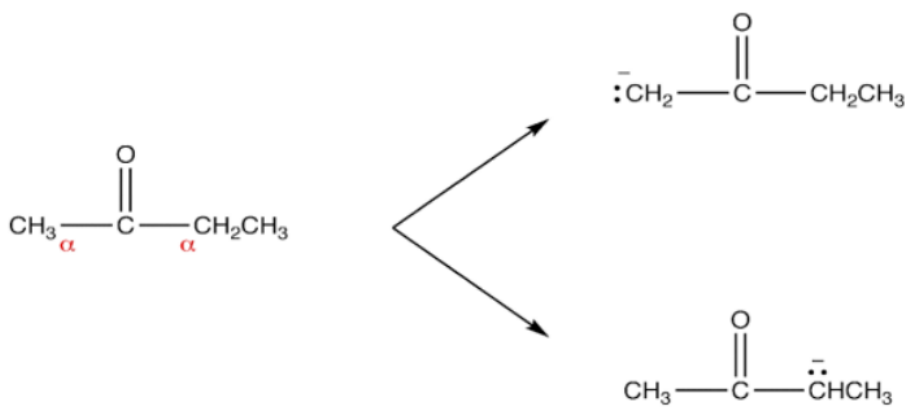
used, especially in biochemistry, to refer to just the first (addition) stage of the process — the aldol reaction itself — as catalyzed by aldolases. However, the aldol reaction is not formally a condensation reaction because it does not involve the loss of a small molecule.

The reaction of an aldehyde with a ketone employing sodium hydroxide as the base is an example of a mixed aldol condensation reaction, the Claisen Schmidt reaction. The double mixed aldol condensation reaction between acetone and benzaldehyde was carried out. Acetone has α hydrogens (on both sides) and thus can be deprotonated to give a nucleophilic enolate anion. The alkoxide produced is protonated by solvent, giving a β -hydroxyketone, which undergoes base catalyzed dehydration. The elimination process is particularly fast in this case because the alkene is stabilized by conjugation to not only the carbonyl but also the benzene. In this experiment, excess benzaldehyde such that the aldol condensation can occur on both sides of the ketone.

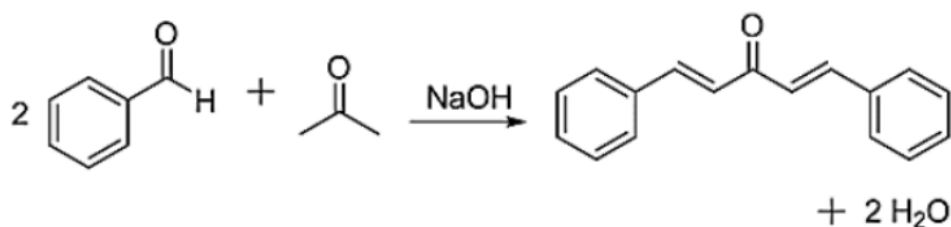
Dibenzalacetone is readily prepared by condensation of acetone with two equivalent of benzaldehyde. The aldehyde carbonyl is more reactive than that of the ketone and therefore reacts rapidly with the anion of the ketone to give a β hydroxyketone, which easily undergoes base catalyzed dehydration. Depending on the

relative quantities of the reactants, the reaction can give either mono- or dibenzalacetone.

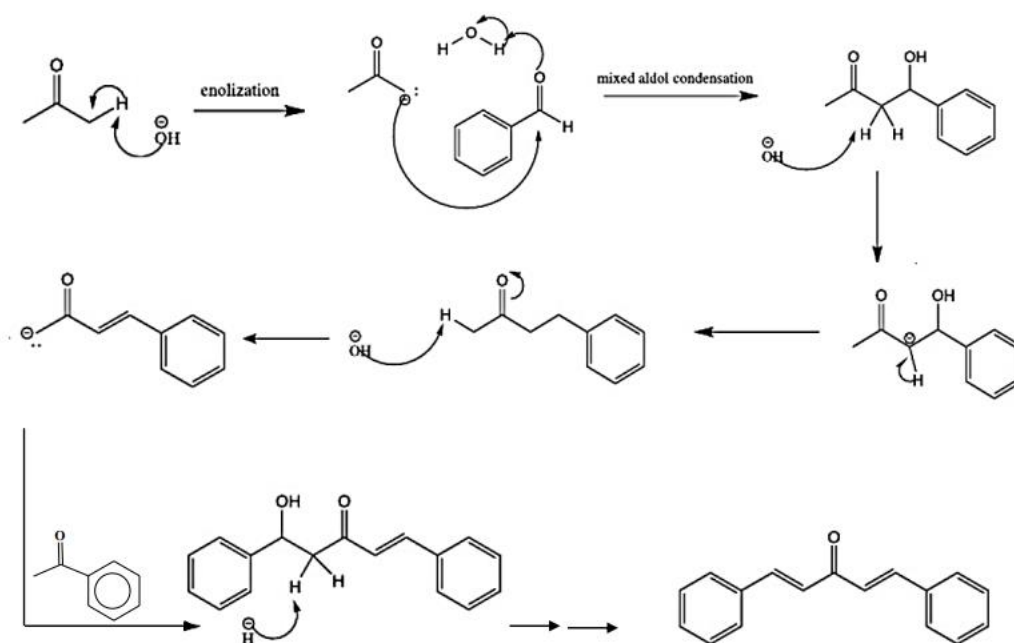
Dibenzalacetone is a fairly innocuous substance in which its spectral properties indicate why it is used in sun-protection preparations. In the present experiment, sufficient ethanol is present as solvent to readily dissolve the starting material, benzaldehyde and also the intermediate, benzalacetone. The benzalacetone once formed, can then easily react with another mole of benzaldehyde to give the desired product in this experiment, dibenzalacetone. An enolate ion is the anion formed when an alpha hydrogen in the molecule of an aldehyde or a ketone is removed as a hydrogen ion.



The General Equation of the Reaction



Mechanism of Dibenzalacetone Step 1: Deprotonation (Make nucleophilic enolate) Step 2: Attack by nucleophilic by electrophilic Step 3: Protonate to give natural hydroxy-ketone Step 4: Deprotonation again (Make nucleophilic enolate) Step 5: Eliminate hydroxide to generate alkene bi bond.



Application of Dibenzalacetone Dibenzalacetone, or DBA, is often used as an ingredient in sunscreen lotions and sprays because it has spectral properties that make it capable of absorbing UV light. It is also known to possess anti-inflammatory properties and can therefore be used to treat pain, swelling and inflammation. Sunscreens utilize dibenzalacetone due to the fact that it absorbs UV rays, preventing them from absorbing into the skin itself. Other chemicals within sunscreen include those that actually reflect UV

rays, pairing up with absorbers like dibenzalacetone to prevent as much skin absorption as possible. Although some concerns have been raised about the potentially toxic nature of the chemical, the benefits of UV protection have led to its continued use in sun protection products.

Procedure:

1. In a small beaker, dissolve (2.5 g) of NaOH in (25 mL) distilled water and (20 mL) of ethanol. Stir the mixture using a magnetic bar, and then cover it by a watch glass and finally let it cool down at room tem. (20 C is perfect).
2. In a clean test tube mix (2.6 mL) of freshly benzaldehyde and (1 mL) acetone and cover the test tube with parafilm.
- 3- Due to a practical reason, mark the level of the half of the mixture with a marker. 3.
- 4- Place a thermometer into the beaker while keeping it stirring and add to it the first half of the mixture in test tube. Stir the entire mixture for 15 min while maintaining the temperature between (20-25 °C). Within a few minutes the solution turns yellow and then a precipitate is formed.
- 5- When the 15 min is finished, add the second half of the mixture in the test tube and stir the mix for 30 min. (Watch the temperature all the time and if it exceeds 25 °C, cool the solution using a water

bath. After 30 min, the reaction should be finished and filter out the precipitate. The precipitate should be yellow.

6- Wash the precipitate with water to make the solution natural. Finally, wash the resulting solid with ethanol to get rid of the remaining water that makes the solid wet.