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# REVIEW ON SYNTHESIS OF DIBENZALACETONE FROM BENZALDEHYDE BY CLAISEN-SCHMIDT REACTION AND THEIR BIOLOGICAL ACTIVITIES

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## ABSTRACT

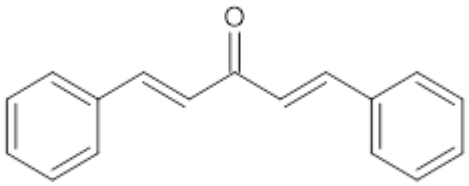
Aldol condensation are essential to organic synthesis because they are able to produce carbon-carbon bonds. An enolate anion is formed starting aldol condensation, the enolate anion comes from an aldehyde or a ketone. Enolizable aldehydes and ketones undergo a reaction when either an acid or base catalyst is present. The product of the reaction gives α,β-unsaturated aldehyde or ketone. The reaction of an aldehyde with a ketone employing sodium hydroxide as the base is an example of a mixed aldol condensation reaction. Condensation of benzaldehyde with acetone in ethanolic alkaline solution leads to the formation of dibenzalacetone, which when treated with selected nucleophiles undergoes Michael addition to give a variety of heterocyclic compounds, these compounds have been characterized by physical method and also they have been screened for their anti bacterial activities. The dibenzalacetone exhibits a wide range of biological activities such as antifungal, antioxidant, antimalarial, analgesic and antitumor characteristics.

**KEYWORDS:** Benzaldehyde, Dibenzalacetone, Claisen-Schmidt reaction, Biological Activities.

## INTRODUCTION

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. Dibenzalacetone is readily prepared by condensation of acetone with two equivalents of

benzaldehyde. The aldehyde carbonyl is more reactive than that of the ketone and therefore reacts rapidly with the anion of the ketone to gives β-hydroxyketone, which easily undergoes base catalyzeed dehydration. Depending on the relative quantities of the reactants, the reaction can give either mono-or dibenzalacetone.[1]



(1*E*,4*E*)-1,5-diphenylpenta-1,4-dien-3-one

When an ethanolic solution containing acetone and its two equivalents of benzaldehyde is made alkaline with sodium hydroxide, rapid condensation occurs with the formation of dibenzal-acetone, or dibenzylidene-acetone. This is a particular example of Claisen Reaction as Claisen showed that aldehyde under the influence of sodium hydroxide condenses with (i) another aldehyde, or (ii) a ketone, with the elimination of water. Thus, benzaldehyde condenses with (i) acetaldehyde to give cinnamic aldehyde, and with (ii) one equivalent of acetone to give (mono) benzal-acetone.[2]

Dibenzalacetone is a fairly innocuous substance; 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 gives the product, dibenzalacetone.[3]

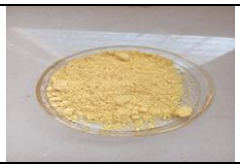
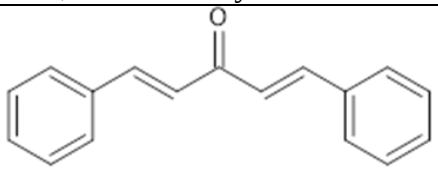
Aromatic aldehyde undergoes condensation reaction with aldehyde or ketone which contain alpha hydrogen atoms in the presence of an alkali. This reaction is called Claisen-Schmidt reaction. The double mixed-aldol condensation reaction between acetone and benzaldehyde was carried out. Acetone has a-hydrogens (on both sides) and thus can be deprotonated to give a nucleophilic enolate anion. The alkoxide produced is protonated by solvent, giving a B-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 synthesized by condensation of acetone with two equivalents 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 B-hydroxyketone, which easily undergoes base catalyzed dehydration. Depending on the relative quantities of the reactants, the reaction can give either mono- or dibenzalacetone.[4-8]

Synthesis of Dibenzalacetone occurs via an aldol condensation reaction shows the reaction of an aldehyde with a ketone employing sodium hydroxide as the base which is an example of an aldol condensation mixed reaction. It is a double mixed-aldol condensation reaction between acetone and benzaldehyde. Acetone has α-hydrogens (on both sides) and thus can be deprotonated to give a nucleophilic enolate anion. The aldehyde carbonyl is much more electrophilic than ketone, therefore it reacts rapidly with the enolate. Dibenzalacetone is a compound that has been recognized as the basic ingredient for making sunblock cream that can counteract UV radiation. The presence of various UV sunblock cream products in the market will make students more familiar with the functions of Dibenzalacetone compounds. Dibenzalacetone compound is a cyclic aromatic compound that has two known pi bonds found in double bonds. The existence of the double bond makes a structural variation available.[9-11]

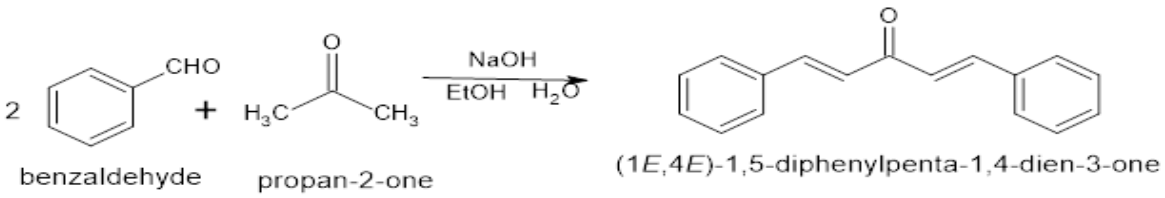
**Drug profile**

**Table-Properties**

| | |
|---|---|
| Dibenzalacetone |  |
| Synonym | Dibenzylideneacetone,1,5-Diphenylpenta-1,4-dien-3-one,trans-Dibenzylideneacetone |
| Structure |  |
| Molecular Formula | C17H14O |
| Molecular weight | 234.29gm/mol |
| Colour | Yellow Solid |
| Nature | Crystalline |
| Odour | Vanilla Like |
| Melting point | 112°C |
| Boiling point | 130°C |
| Solubility | Soluble in Organic Solvent such as ethanol, Diethylether |
| Use | In some sunscreens it absorbs the harmful UV light, also used as ligand in preparing organometallic complexes |

**Various conventional methods for synthesis of dibenzalacetone**

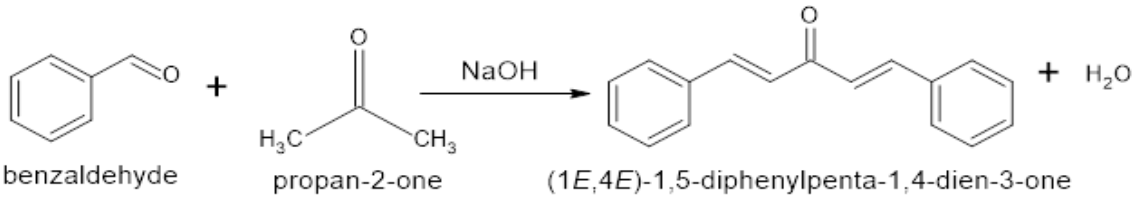
**1) Reaction of dibenzalacetone**




**Preparation of dibenzalacetone**

Place 5.0 gm of sodium hydroxide pellets in conical flask. Add 50 ml of water and 40 ml of ethanol. Cool the flask in ice cold water. Add 5.0 ml of benzaldehyde and 2.0 ml of acetone into it with swirling of the contents. Shake frequently and maintain the temperature at 20-25°C for 15 minutes by immersion of the flask in ice cold water. Filter off the precipitated dibenzalacetone at the pump. Wash it with cold water to eliminate the alkali. Recrystallize from hot rectified spirit. The yield of pure dibenzalacetone is 4.4 gm. M.P. = 112°C.[12]

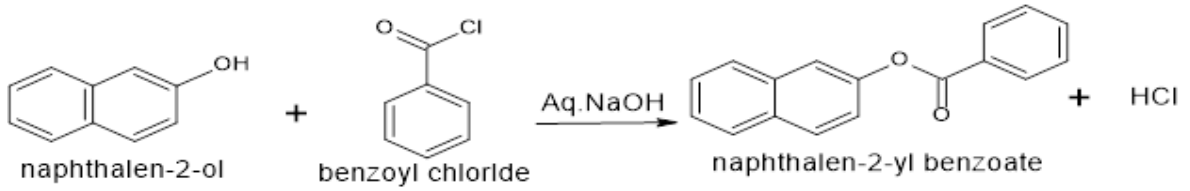
**2) Reaction of dibenzalacetone**




**Preparation of dibenzalacetone**

In 250ml Erlenmeyer flask dissolve 7.5 gm of sodium hydroxide in 75 ml DI water and 60 ml of ethanol. Maintain the temperature of the solution at 20-25 º C. Add previously prepared mixture of 7.5 ml of Benzaldehyde and 3 ml Acetone shake well. A precipitate forms in 2-3 min. after 15 min add the remaining portion of the Benzaldehyde –Acetone mixture, shake for further 30 min now cool the Erlenmeyer flask and separate the solid precipitated was filtered, washed and recrystallized from hot Ethanol.[13-18]

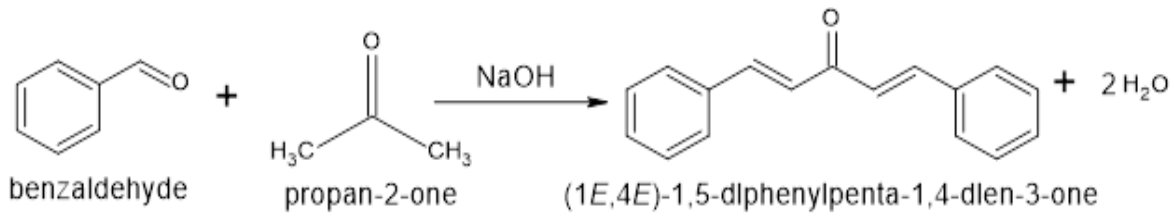
**3) Reaction of dibenzalacetone**



**Preparation of dibenzalacetone**

Dissolve 3.5g of B-napthal in 20ml of 5% NaOH son contained in 250ml Iodine Flask. If the solution is Highly Colored so add 0.5gm charcoal warm if filter. Add 2.5 ml (3.59m) of benzoyl Chloride 2gml Cork the flask securely and shake the Mixture vigorously for lo minutes until the- odour of benzoyl Chloride disappears (caution). Filter the solid obtained on Buckner funnel & washed it with cold water. Recrystallize the crude product from ethanol. Filter the crystals and dried them upon filter Paper. Report the yield and melting Point. Yield of pure compound is 3.3gm and melting point is 110°C.[19]

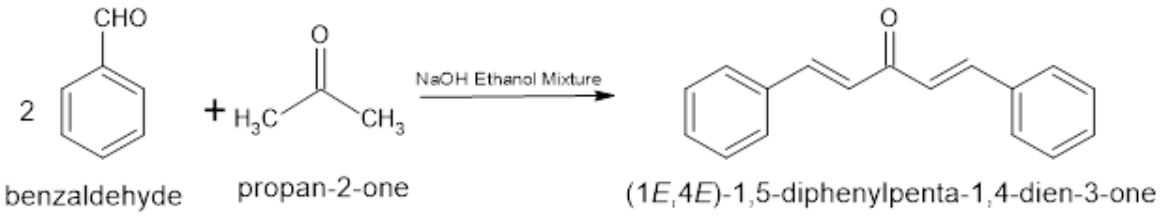
**4) Reaction of dibenzalacetone**




**Preparation of dibenzalacetone**

Place a 100ml of conical flask add 10ml (10.4 gm) freshly distilled benzaldehyde and 4 ml of pure acetone. Place the flask in cold water bath and then add 2 ml of 10% hydroxide solution dropwise with constant stirring. The reaction mixture temperature should not be raised beyond Should be maintain the temperature at 30°C. After completing the addition of sodium hydroxide stir the mixture and allow to stand for 30 minutes. Occasionally shaking reaction mixture and finally cooled the mixture in ice water. During the shaking Dibenzalacetone separates initially as a fine emulsion and then forms yellow crystals. The reaction mixture is filtered with at the pump, washed well with a water to eliminate traces of alkali. Filter out the pale-yellow crystals with cold water, dried the crude product and recrystallize with using of 30ml of ethanol. Crude Dibenzal acetone is obtained, yield 10 gm and m.p. 112°C.[20-21]

**5) Reaction of dibenalacetone**



**Preparation of dibenzalacetone**

Prepare an ice-water bath in a 250ml beaker, place 15ml of ethanol and 20ml of aqueous 10% NaOH into a 100 ml beaker & add a stirbar. Place the 100ml beaker into the ice-water bath. Set then the entire assembly on to a magnetic stirrer. While stirring, cool the solution to 20°c. After the solution reaches to 20°c, remove the ice-water bath, continue to stir the solution. Prepare the mixture of 2.1 ml (2 m mol) of fresh Benzaldehyde and 758 µl (1 m mol) of Acetone in a test tube over a period of 5-10 mins, add the Benzaldehyde-acetone mixture to the ethanol-NaOH solution in a small portions, then stir the reaction for another 30 mins. Cool the mixture using the ice water bath for one hour. collect the crystals by vaccum filtration. Wash the crystals by suspending them in 50ml of distil water. Again collect the crystals by vaccum filtration. Finally recrystalised with ethyl acetate. Check the filtrate by testing the last few drops of water using red-litmus paper. If the litmus changes to blue, wash the crystals again until red-litmus does not changes colour. Keep a small sample aside to dry for a crude melting point measurement.[22]

**Biological activities of dibenzalacetone**

**1) Antibacterial and Antifungal activity**

The antibacterial activity of synthesized compounds were determined by screening them against the E. coli and S. aureus. The antifungal activity of synthesized compound were determined against the Aspergillus niger, Trichophytum rubrum and Candida ablicans. The basic principle of antibacterial and antifungal assay lies in the comparison of inhibition of growth of microorganism produced by the known concentration of antibacterial and antifungal agents to be tested with that produced by known concentration of standard antifungal agent having known activity.[23]

**2) Anti parasitic activity**

The antileishmanial activity of DBA was tested initially against promastigotes of L. donovani. Promastigotes which resulted in the loss of parasite viability in a concentration-dependent manner. The DMSO solvent employed had no effect on the parasite viability.[24-25]

**Applications of dibenzalacetone**

Dibenzalacetone used as a component in some sunscreens as it absorbs the harmful UV light. also used in some industrial organometallic compounds because it bonds to metals and helps form a stable chemical structure. Used in preparing organometallic complexes used as catalysts in coupling reactions.[26]

## CONCLUSION

The experiment utilized aldol condensation mechanism to form the most stable isomer trans, trans (E, E) dibenzalacetone. The conformation was first indicated by the observation of the yellow product precipitate formed after the reaction It is found that this dibenzalacetone nucleus is yet to fully explore for its biological potential. The compounds had been synthesized with an attempt to potentiate them by fulfilling the structural requirement for special biological activities. Dibenzalacetone were screened for their possible in vitro antibacterial and antifungal activity by well diffusion method with the help of different bacterial strain like E. coli and S. aureus and fungal strain like Aspergillus niger, Trichophytum rubrum and Candida albicans.

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