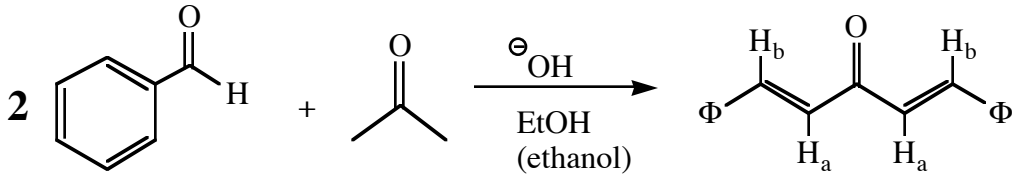
DIBENZALACETONE BY THE ALDOL CONDENSATION



1,5-diphenyl-1E,4E-pentadien-3-one

(dibenzalacetone)

The reaction of an aldehyde with a ketone employing sodium hydroxide as the base is an example of a mixed aldol condensation reaction (a 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 give a β-hydroxyketone, which easily undergoes base-catalyzed dehydration. Note the relative quantities of the reactants; this reaction provides dibenzalacetone.

Procedure

In a 5 mL round bottom flask, place a magnetic stir bar and 1.6 mL of 95% ethanol. Then, using a 1-mL syringe, add 0.204 mL of benzaldehyde. Stir this solution thoroughly to dissolve all of the benzaldehyde. Then add 2 mL of 10% sodium hydroxide solution, followed by 0.073 mL of acetone. (The storeroom may give us a solution of ethanol with 0.073 mL of acetone in every 1.6 mL of ethanol solution). Place a condenser on the flask, and keep stirring the reaction mixture. The solution should quickly become a pale yellow color. After a couple of minutes, the solution will become cloudy and a yellow precipitate of the product forms. Continue to stir the reaction for 30 minutes. If the product fails to crystallize, scratch the inside of the flask with a glass rod. After crystallization, allow the solid to settle and carefully remove the liquid from the flask using a Pasteur pipette. Press the tip of the pipette against the bottom of the flask and suction the liquid into the pipette, leaving the crystals behind. Add 3 mL of water, stopper, and stir vigorously. Remove the wash liquid as before and wash the crystals twice more with water. Remove as much water as possible. It is important to remove as much of the water as possible since water causes the "oiling out" problem during crystallization. You can also do this filtration and all the washes with water on the Hirsch Funnel. The suction will be able to remove the water more effectively than a pipet.

Transfer the crystals to a small Erlenmeyer flask and recrystallize them using 70/30 ethanol/water. Begin with about 3 mL of solvent. Remember the steps of a recrystallization. Dissolve in the minimum amount of boiling solvent (use a boiling stick), let it cool to room temp., then cool in an ice/water bath. Filter and wash with ice cold solvent. If your crude product has too much water, it may "oil out" when you're doing the recrystallization. If this happens, add a bit more pure ethanol to try to dissolve the organic product when you are boiling the solvent.

Be sure to get the mp, IR, and percent yield of your product.

Cleaning Up.

All organic solutions should go in the organic solvent waste container.

Questions: (these answers can be included in your discussion)

1. Why do we get only the *E* (trans) alkenes? Is this aldol reaction under kinetic or thermodynamic control?

2. How do we know that we obtained only the trans alkenes?

3. Almost all of the hydrogens are at about 7 ppm in the ^{1}H NMR. I have indicated where the two alkene hydrogens (H_a and H_b). Which is which? Explain your answer.

4. Why doesn't acetone attack itself?

5. Why doesn't benzaldehyde attack itself?