Chemistry 212 Laboratory

Dibenzalacetone via Crossed Aldol Condensation

Prelab: Calculate the amounts of all chemicals needed in measurable amounts (i.e. grams or milliliters rather than moles.)

Introduction: Aldol condensations are important in organic synthesis, providing a good way to form carbon–carbon bonds. The "**aldol**" (**ald**ehyde + alcoh**ol**) product is a structural unit found in many naturally occurring molecules and pharmaceuticals, and is therefore important. In an Aldol condensation an enolate ion reacts with a carbonyl compound to form a β-hydroxyaldehyde or β-hydroxyketone, followed by dehydration to give a conjugated enone. The general equation is shown in Figure 1.

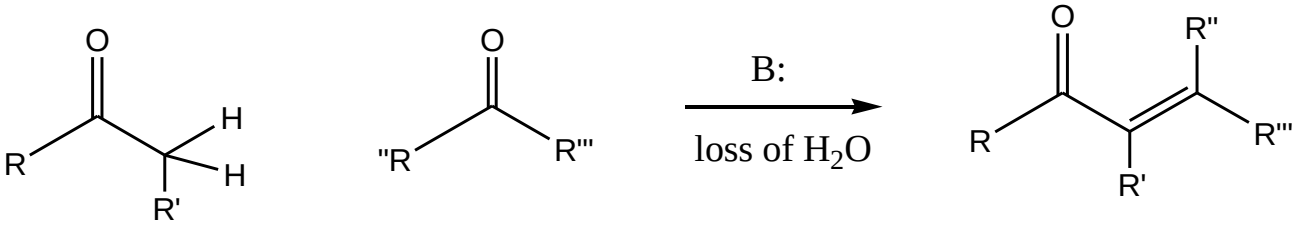

Figure 1. The equation for the Aldol Condensation.

The reaction involves the nucleophilic addition of an enolate to an aldehyde to form a β-hydroxy carbonyl. The β-hydroxy carbonyl is readily dehydrated under mild conditions. The aldol reaction occurs under both acidic and basic conditions as seen in Figure 2.

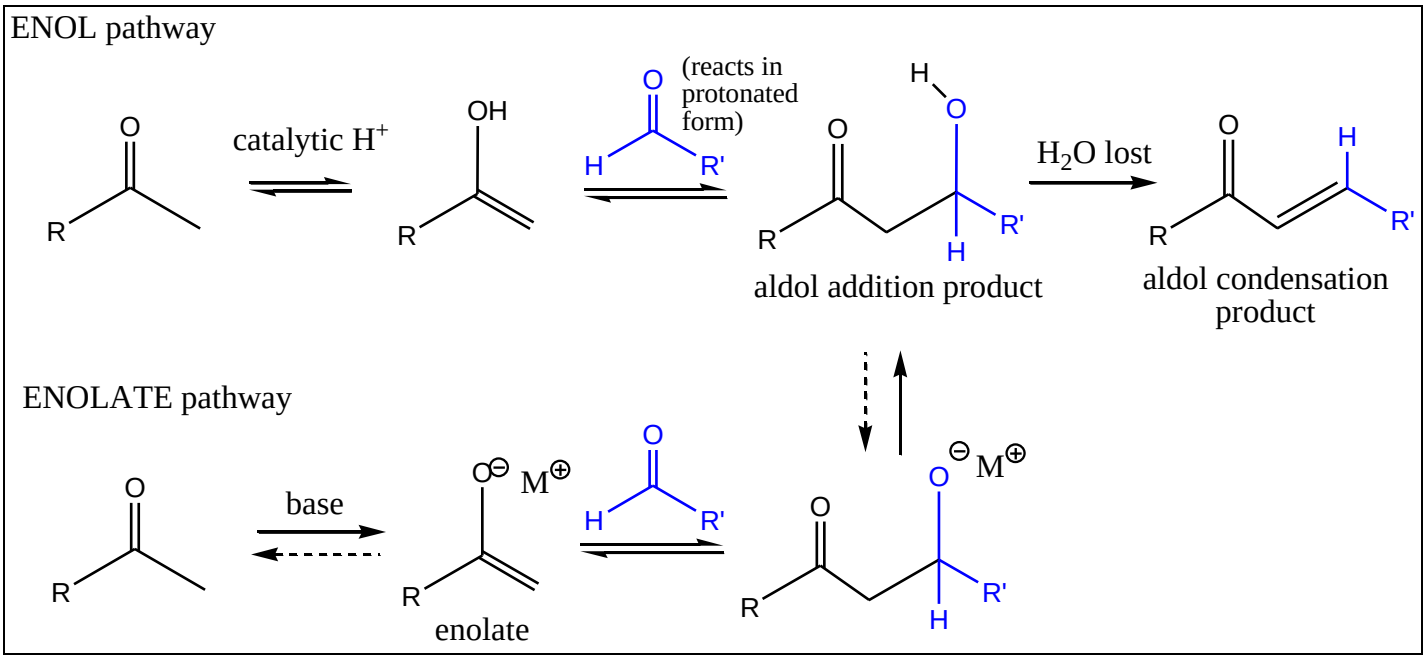

Figure 2. The Aldol reaction and subsequent dehydration under acidic and basic conditions.

The reaction we will be doing this week involves the reaction between benzaldehyde and acetone to do a double Aldol Condensation. The overall equation is shown in Figure 3.

The first part of this reaction is an aldol reaction, the second part a dehydration—an elimination reaction. Dehydration may be accompanied by decarboxylation when an activated carboxyl group is present. The aldol addition product can be dehydrated via two mechanisms; a strong base like potassium t-butoxide, potassium hydroxide or sodium hydride in an enolate mechanism, or in an acid-catalyzed enol mechanism. Which mechanism is the reaction in this experiment going to follow?

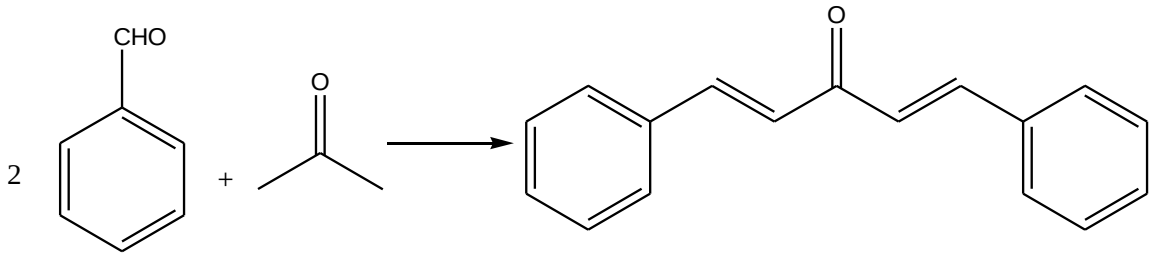

Figure 3. The equation for the Aldol Condensation between benzaldehyde and acetone.

Procedure:

1. Place 0.006 moles of benzaldehyde, 0.003 moles of acetone, and 3 mL of 95% ethanol (as a solvent) in a conical test tube. Mix until completely dissolved.
2. Add 1 mL of 10% sodium hydroxide solution. Mix the contents until precipitation is observed.
3. Let the mixture stand for another 20 minutes with occasional shaking.
4. After the 20-minute period is completed, cool the mixture in an ice bath for 10 minutes.
5. Place the conical test tube in the centrifuge (remember to cork and counterbalance). Centrifuge for a few minutes. Decant to remove the liquid from the tube. If it is difficult to separate, use a cotton-plugged Pasteur Pipette. (In order to assure that no solid product is transferred out of the test tube, you could take a very small piece of cotton, and plug up the tip of the pipette from the outside, such that only liquids are transferred into the pipette, leaving the solids behind in the conical test tube.)
6. Wash the crystals with 2 mL ice-cold water. Centrifuge and decant. Wash with the second portion of 2 mL ice-cold water. Centrifuge and decant.
7. Recrystallize the solid product in the same conical test tube, using 95% ethanol as the solvent. Place the test tube in a water bath on the hot plate. Preheat the ethanol in the water bath then add *drop wise* into the test tube until all the crystals dissolve. Heating (keep temperature lower than the boiling point of the ethanol), stirring, and adding more ethanol will help with the dissolving. Let the solution cool slowly and without agitation. Once crystals are formed, place the test tube in the ice bath to maximize the amount of the crystals.
8. Collect the product via vacuum filtration (rince with ice-cold ethanol) and allow it to dry. Note that you may need to tare your filter paper if you have very little product.
9. Weigh your product. Measure the melting point and the IR spectrum of your obtained product.

For your Report:

Calculate and discuss your percent yield.
Discuss the melting point and the IR spectrum of your obtained product.
Predict the ^{13}C-NMR spectrum for the product. Draw the mechanism of this reaction.