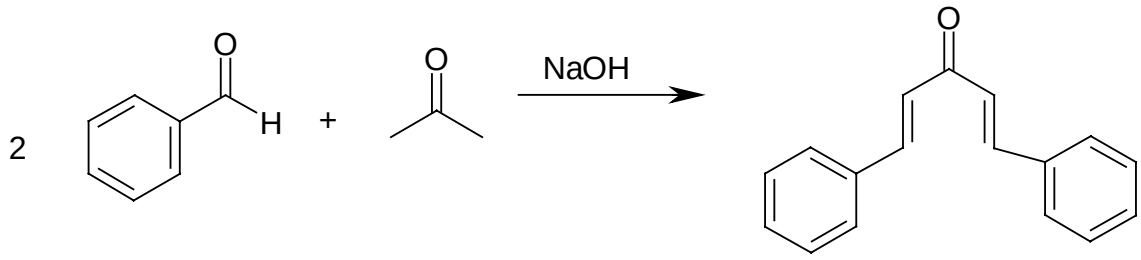
A Double Aldol Condensation
The Synthesis Of Dibenzalacetone

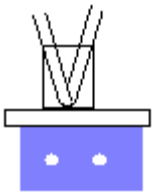


Procedure:

1. In a 10x100mm reaction tube, place 2 ml of 3 M NaOH solution, 0.212g of benzaldehyde (you might want to find the density of the benzaldehyde as it is easier to measure volumes of this liquid then mass) and 1.6 ml of a 3.63 % solution of acetone in ethanol. (0.058g of acetone in the 1.6 ml of solution.)[1]
2. Shake the solution vigorously for 30 minutes. A solid yellow precipitate should form. This is your product.
3. Using a glass Pasteur pipette, carefully remove the solution leaving the solid behind. This is best accomplished by aspirating the liquid while the pipette is at the bottom of the tube. Do not leave enough room between the pipette and the test tube for the solid to pass by.
4. Wash the crystals by adding about 3 ml of water, shaking vigorously and then removing the water as before with the Pasteur pipette. Wash the solid twice more with water.
5. Using 3 ml of water, isolate the solid using a Hirsh funnel.

Purification procedure:

The Aldol product will be purified by recrystallization from a 70:30 (v:v) ethanol water mixture. Remember, the idea is to get all of the product to dissolve at a high temperature and then to cool



1. Start a hot water bath in a 250 ml beaker. Obtain two test tubes, put the 70 % ethanol in one and the solid in the other. Slowly add the ethanol to the solid keeping both solutions hot. Make sure to stir/swirl the mixture well after each addition of the ethanol.
2. When the solid has dissolved, allow the tube to cool slowly to room temperature by leaving it in an empty beaker. Crystals should form. If crystals do not form, try adding a seed crystal, scratch the side of the tube or, if it oiling out, add a little more ethanol.
3. To recover as much product as possible, cool the tube in an ice bath.
4. Collect the product with a Hirsh funnel using ice-cold 70% ethanol to wash the crystals.
5. Let the product dry over the week and check melting point before class Tuesday.

Post Lab Questions

1. Why is it important to maintain equivelent proportions of reagents in this experiment?
2. What side products do you expect in this experiment? How are they removed?
3. The geometric possibilities for products are cis/cis, trans/trans or trans/cis. Which isomer do you think is the major product and why?

[1] Someone will have to make this solution by mixing 0.870 g of acetone with enough ethanol to make 24 ml.

4. What evidence do you have that the product is a single geometric isomer? (melting point?) Explain.
5. Please write a complete mechanism for this reaction.