

# An Aldol Condensation

Unlike most of the experiments in this course, this experiment is not done on a microscale. Macroscale reactions use 5 g or more of reagents. Miniscale procedures use 0.5 to 5 grams of reagents, and microscale use 0.5 g or less of reagents.<sup>i</sup> Today's reaction is still a considerably smaller-scale reaction than those that were commonly done in organic chemistry laboratories in the past.

In this reaction two molar equivalents of benzaldehyde react with one molar equivalent of acetone to form a substituted pentadieneone product. Two dehydrations occur which form two double bond. The crude product is isolated by vacuum filtration. The crude product is recrystallized. The purity of the crude and recrystallized product are assessed by melting point and compared.

## PRE-EXPERIMENT ASSIGNMENT

Study this chapter in the manual, the procedure and notes relating to recrystallization on the web site and the lecture notes on Aldols on the Chemistry Department web site. You will also find it helpful to review sections 22.1 and 22.3 of Klein. Do the first seven parts of your notebook writeup.

**A student who has prepared for the Aldol Condensation experiment should be able to:**

1. Define: condensation reaction
2. Give a drawing of, and identify the components from a drawing of, the apparatus used in the various steps of the reaction and its components.
3. Calculate the theoretical yield and the percent yield for this and similar experiments given the necessary data, and perform any of the intermediate calculations required by this process. For this reaction, "necessary data" may include the volume of a given starting material and its density rather than simply the mass.
4. Draw the structure given the name, or give the name from the structure, of the compounds used in the day's experiment, and give the role of each (reactant, solvent, catalyst, etc.).
5. Understand the mechanism of the condensation of benzaldehyde and acetone.
6. Identify and explain safety considerations for this experiment.
7. Perform the day's experiments safely and successfully.

Quizzes given after the experiment has been performed may also include:

7. Predict the products of aldol reactions and condensations, and give and explain the mechanism of such reactions given the balanced equation.

Important topics include enolate anions, electrophiles, and nucleophiles. For more details see your CHEM 2220 lecture materials.

### **Safety Precautions**

Acetone, benzaldehyde, and ethyl acetate are flammable organic materials. Do not have any open flames or sparking devices in the vicinity of the reaction. Swirl Erlenmeyer flasks in hood to keep vapors to a minimum.

Sodium Hydroxide is a strong base and very corrosive. Keep sodium hydroxide solution away from skin and eyes. If contact is made flush with copious amounts of water.

### **The Reaction<sup>ii</sup>**

Obtain approximately 4 mL of 5 M aqueous sodium hydroxide solution and approximately 4 mL of 95% ethanol. (Read volumes accurately, record measurements directly in notebook.) Add these to a 50 mL Erlenmeyer flask and swirl to mix. Into a separate 50mL beaker add approximately 2.0 mL of benzaldehyde . Using your small syringe, measure 0.60 mL of acetone and add to the benzaldehyde. Swirl to mix. Add half of the acetone/benzaldehyde mixture to the sodium hydroxide/ethanol mixture. Swirl the result.

The mixture should quickly change from yellow to orange and warm up a bit. A yellow precipitate should begin to appear almost immediately, though the exact colors may vary slightly. Record observations. As soon as the reaction starts, add the rest of the acetone/benzaldehyde mixture to the flask. You may rinse the beaker with a small amount (< 1mL) of ethanol and add to Erlenmeyer. Allow the reaction to proceed for about fifteen minutes, swirling occasionally.

### **The Workup**

After fifteen minutes, suction-filter the mixture using your large Büchner funnel and filter flask. Disconnect the vacuum, wash the product with a few milliliters of cold ethanol, and reconnect the vacuum. Allow the suction to run for at least five minutes longer to help dry the crude product. Note the texture and color of the product. Weigh the crude product and put some in a capillary so you can determine its melting point *later*.

### **Recrystallization**

Place a 450 mL beaker approximately half full of water and one boiling stone and a thermometer on a hot plate. Heat water. The water should **NOT** be allowed to rise higher than 70°C since the ethylacetate boils at 77°C and can shoot out of test tube. IF the water temperature rises above 70, add some tap water to cool and turn down the hot plate. Place about 5 mL of ethylacetate and one boiling stone in a test tube. Place the test tube in the warm water

beaker to heat. Place the solid in a 50 mL clean E. flask. Hold E. flask in the hot water. Pipette warm ethylacetate into the E. flask containing solid. Shake to mix and dissolve the solid. Add just enough ethylacetate to dissolve all of the solid. You will need approximately 2.5 mL of solvent for each gram of crude product<sup>iii</sup>. Any additional solvent will only decrease yield. As soon as all of the solid has dissolved, remove small beaker from water bath and place on benchtop to cool. Do not disturb. Observe. After about 5 minutes crystals should form. When no more crystals are forming, place in 250 mL beaker with a few inches of ice and water. Leave in ice bath at least 5 minutes. Scrape side of small beaker if no crystals are observed.

Collect the crystals by suction filtration. Spread the crystals out on the filter paper as much as possible. To help the crystals dry faster, allow the vacuum to run for at least five minutes after the last of the liquid has been pulled through the funnel. Note the texture and color of the product. Determine the mass of these crystals and the melting point of both these crystals and the crude product. Calculate the percentage yield.

## **CLEANUP**

Used Pasteur pipettes and melting point capillaries are placed in the broken glass box. Leftover ethanol and ethyl acetate are placed in the non-halogenated liquid waste bottle in the hood; your product goes in the non-halogenated solid waste bottle if your instructor does not collect it. Leftover NaOH solution in small amounts is washed down the drain with plenty of water.

## **POST-EXPERIMENT ASSIGNMENT**

Write the lab report and have it ready to turn in by the beginning of the next lab. Your writeup of this experiment (as for every synthesis experiment) should include a calculation of the percentage yield for both the crude product and the recrystallized product. Your conclusions section should include a comparison of the melting point of your crude and recrystallized product with the literature value. Provide a reasonable explanation for any differences. Explain the success or failure of the condensation reaction. Explain the success or failure of the recrystallization. If your percent yield was not 100%, you should explain where the missing material might have gone (or where extra material might have come from). Prepare for the aldol condensation portion of the next quiz.

---

<sup>i</sup> Schoffstall, A. M, Gaddis, B. A., Druelinger, M.L., *Microscale and Miniscale Organic Chemistry Laboratory Experiments*, McGraw Hill, 2000, pg 2

<sup>ii</sup> Dibenzylacetone Synthesis <http://www.orgsyn.org/orgsyn/pdfs/CV2P0167.pdf> (March 4, 2011)

<sup>iii</sup> Conrad, C., Dolliver, M, *Organic Syntheses, Coll. Vol. 2, p.167 (1943); Vol. 12, p.22 (1932)* available online <http://www.orgsyn.org/orgsyn/orgsyn/prepcontent.asp?prep=cv2p0167>

