Lab Instructions

Chemistry 337
Unit 5 Synthesis of E,E-dibenzalacetone (E,E-DBA)

INTRODUCTION

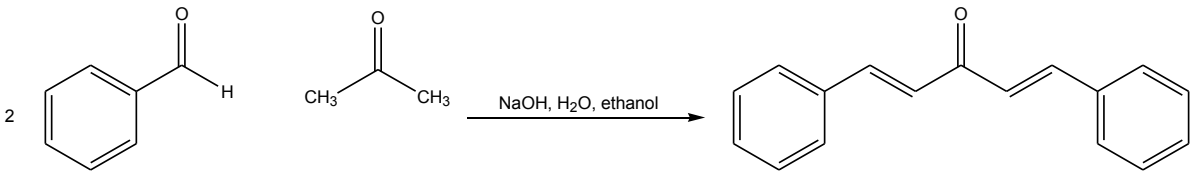
The centerpiece of this unit is the synthesis of E,E-dibenzalacetone (E,E-DBA) for which we will employ a base-catalyzed aldol condensation. The crude product will be purified by recrystallization. The crude and purified products will be characterized by melting point determination.

PURPOSE

To employ a based-catalyzed aldol condensation in the synthesize of E,E-dibenzalacetone

To characterize the crude and purified samples of E,E-dibenzalacetone by melting point determination

REACTION SCHEME



Benzaldehyde Acetone E,E-dibenzalacetone

GENERAL COMMENTS

This comments segment need not appear in your prelab assignment.

Students will work individually. Other than the recrystallization all worked described below will be completed at your lab bench. The recrystallization will be performed in one of the fumehoods under the supervision of a TA.

The **Unit 5 Fumehood** is equipped with dispensing pipettes, for delivering benzaldehyde and **reagent grade acetone**, a burette for dispensing the catalyst solution, several bottles of 95% ethanol, a squeeze bottle of **wash grade acetone**, a **solid waste container** (typically a glass tray), a **waste pipette container,** an **aqueous waste container** (typically a large, wide-mouthed plastic bottle), and an **organic waste container** (typically a large plastic bottle with a red flip-lid).

Disposable gloves, sample vials, Pasteur pipettes, black threaded caps, O-rings, full plastic septa, creased weighing papers, boiling stones and Hirsch funnel filter papers are available on the front benches.

Metal clamps and thermometer clamps are available in bins located on the front bench nearest the lab door.

PRELAB ASSIGNMENT TO BE COMPLETED BEFORE YOUR SCHEDULED UNIT 5 LAB SESSION

Please follow the format used for the Unit 2 prelab assignment.

It is not necessary to repeat steps involving equipment used in a prior Unit. Just note the number of the page in your lab notebook where this information is provided.

It is not necessary to repeat "Physical constants and properties" for reagents, reactants and products encountered in a prior Unit. Just note the number of the page in your lab notebook where this information is provided.

It is not necessary to repeat "Chemical hazards and precautionary measures" for reagents, reactants and products encountered in a prior Unit. Just note the number of the page in your lab notebook where this information is provided.

LIST OF CHEMICALS/CHEMICAL HAZARDS AND PRECAUTIONARY MEASURES/SUPPLEMENTARY LAB EQUIPMENT

List of chemicals

Acetone
Benzaldehyde
E,E-dibenzalacetone
Ethanol
Ethanolic sodium hydroxide (1.4 M) (as a solution in water and ethanol)

Chemical hazards/precautionary measures

Acetone, ethanol and benzaldehyde are irritants - handle with care. Avoid inhalation and skin contact (wear gloves). Avoid eye contact (wear safety goggles). Notify the instructor immediately should you spill either of these materials on yourself or the lab bench. Ethanolic sodium hydroxide (1.4 M) is corrosive and causes burns – handle with care. Avoid inhalation and skin contact (wear gloves). Avoid eye contact (wear safety goggles). Notify the instructor immediately should you spill any of this material on yourself or the lab bench.

Supplementary lab equipment from the Issue Room

None required.

CLEANING GUIDELINES

These guidelines need not appear in your prelab assignment.

You will be using wash acetone to clean lab glassware and equipment. Since acetone is such a good cleaning solvent only minimal amounts are required. It is provided in plastic bottles fitted with red caps having curved tips. The bottles are located in the fumehoods next to the organic waster containers.

Cleaning locker items

For container type items (e.g. beakers, conical vials, flasks, graduated cylinders, jars, round bottom flasks and test tubes) add 2 or 3 short squirts of wash acetone then swirl to dislodge/dissolve any noticeable organics. Finally pour the contents into an organic waste container. For all other items direct 2 or 3 short squirts of wash acetone through/over the item directly into an organic waste container. If noticeable organics still remain please speak with your lab instructor.

Cleaning items signed out from the Issue Room

Since these are generally larger than locker items you will need to use a bit more wash acetone. For container type items use 6 or 8 short squirts of wash acetone. For all other items direct 6 or 8 short squirts of wash acetone through/over the item directly into an organic waste container. If noticeable organics still remain please speak with your lab instructor.

TABLE OF REACTANTS AND PRODUCTS

Include the following table in your prelab assignment. **Complete the first three rows as part of your prelab assignment.**

reactant/product	molar mass	volume, mass, moles	yield	melting point
benzaldehyde (density 1.043 g/mL)	_____ g/mole	_____ mL _____ g _____ moles		
acetone (density 0.788 g/mL)	_____ g/mole	_____ mL _____ g _____ moles		
E,E-DBA (a yellow solid)	_____ g/mole		Theoretical _____ g _____ moles	Literature value 110-111°C
E,E-DBA (a yellow solid)	_____ g/mole		Actual crude* _____ g _____ % purified** _____ g _____ %	Actual crude _____ purified _____

* appearance ** appearance

EXPERIMENTAL PROCEDURES

STEP 1 Preparation of E,E-dibenzalacetone (performed at your lab bench)

a. Fit the bottom end of a microscale condenser first with a black threaded cap followed by an O-ring. The O-ring needs to be positioned just above the ground-glass joint. Affix a black threaded cap, fitted with a full plastic septum, to the top end of the condenser.

b. After obtaining 0.250 mL of benzaldehyde from the instructor use the dispensing pipettes/burettes in the **Unit 5 Fumehood** to transfer 0.100 mL of **reagent grade acetone**, 0.5 mL of 95% ethanol and 1 mL of the catalyst solution to a 10 mL RB flask. The technical staff prepared the catalyst solution by dissolving 10 g of sodium hydroxide in 100 mL of water and 75 mL of 95% ethanol.

c. Fit the RB flask with the condenser. Secure the connection by firmly threading the black cap onto the RB flask in the manner employed in Unit 2.

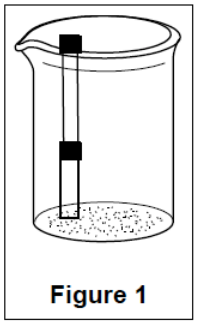


Figure 1

d. Hold the apparatus by the RB flask and gently swirl its contents for 15 minutes. Set the apparatus in a large beaker (as shown in Figure 1) while setting up the equipment depicted in Figure 2.

STEP 2 Collection of the crude E,E-dibenzalacetone by vacuum filtration (performed at your lab bench)

a. Using a metal clamp secure a 250 mL filter flask to the left most vertical support bar. Fit the filter flask with a Hirsch funnel equipped with a Hirsch funnel filter paper. Connect the quick-fit adapters of the vacuum trap and filter flask. Your apparatus should now resemble Figure 2.

b. Prepare to collect the crystals by wetting the filter paper on the funnel with a little de-ionized water and applying full suction (using the house vacuum line).

c. Transfer 60 mL of de-ionized water to a 100 mL graduated cylinder.

d. Transfer approximately 5 mL of de-ionized water from the graduated cylinder to the RB flask through the air condenser then gently swirl to suspend the crystalline material. Remove the air condenser and pour the suspension into the Hirsch funnel. Re-attach the air condenser.

e. Repeat Step 2d three times.

f. Gently score the crystalline material with a microspatula.

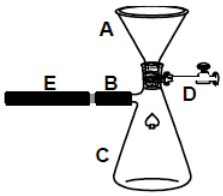


Figure 2

A Hirsch funnel
B quick-fit adapter on filter flask
C filter flask
D clamp
E quick-fit adapter from vacuum trap

g. Pour approximately 20 mL of the remaining de-ionized water from the graduated cylinder into the Hirsch funnel. Gently score the crystalline material with a microspatula.

h. Pour the remainder of the de-ionized water (~20 mL) from the graduated cylinder into the Hirsch funnel. Gently score the crystalline material with a microspatula.

i. To facilitate the recrystallization to follow it is necessary to dry the solid by continued suction for 5 minutes while proceeding with other work.

j. While the drying under suction is underway transfer 6 mL of **95% ethanol** to a large test tube. Place the test tube in a beaker of ice and water (replenish ice and water as needed). This chilled **95% ethanol** will be used in Step 4.

k. Clean the 10 mL RB flask, condenser and microspatula with **wash grade acetone**.

l. Carefully disconnect the quick-fit adapters of the vacuum trap and the filter flask then turn off the house vacuum line.

m. At your lab bench place a **pre-weighed** 10 mL Erlenmeyer flask on a piece of creased weighing paper. Using a microspatula carefully transfer the crude E,E-dibenzalacetone from the Hirsch funnel directly into the Erlenmeyer flask (the creased weighing paper is used solely to contain any spilled material). Transfer any spilled material to the flask. Reweigh the flask and determine the mass of the crude E,E-dibenzalacetone. Retain a small sample of the crude E,E-dibenzalacetone (about the size of a grain of uncooked rice) in a small test tube for a melting point determination next week.

n. Discard the filter paper and weighing paper in the appropriate **solid waste container**.

o. Discard the filtrate in the appropriate **aqueous waste container**. Clean the filter flask, microspatula and Hirsch funnel with **wash grade acetone.**

STEP 3 Purification of the E,E-dibenzalacetone by recrystallization (performed in the designated fumehood)

a. Transfer an appropriate volume of **95% ethanol** to the 10 mL Erlenmeyer flask containing your crude *E,E*-dibenzalacetone (solubility of *E,E*-dibenzalacetone in **95% ethanol** at 78°C is 0.4 g/1 mL). Add two boiling stones. Label your Erlenmeyer flask with your name (Sharpies® are available at the Issue Room window).

b. Heat the Erlenmeyer flask on a hotplate stirrer in the **Unit 5 recrystallization fumehood** (do not leave unattended). Once **the entire sample has dissolved** remove the flask from the hotplate stirrer (exercise caution as the flask will be **very hot**) and allow it to cool to room temperature, undisturbed on the bench top, in the **Unit 5 recrystallization fumehood**. To complete crystallization cool the flask in a medium sized beaker containing a 3/4" layer of ice and water (this may be performed at your lab bench).

STEP 4 Collection of the purified E,E-dibenzalacetone by vacuum filtration (performed at your lab bench)

a. Using a metal clamp secure a 250 mL filter flask to the left most vertical support bar. Fit the filter flask with a Hirsch funnel equipped with a Hirsch funnel filter paper. Connect the quick-fit adapters of the vacuum trap and filter flask. Your apparatus should now resemble Figure 2.

b. Prepare to collect the crystals of purified E,E-dibenzalacetone by wetting the filter paper on the funnel with a little **95% ethanol** and applying full suction (using the house vacuum line). Gently swirl the contents of the 10 mL Erlenmeyer flask to suspend the crystalline material (use a microspatula to break up any solid chunks) then pour this suspension into the Hirsch funnel. You may use small quantities of the chilled **95% ethanol** prepared earlier to aid in the transfer of your crude product to the Hirsch funnel (2 mL at a time; but no more than a total of 6 mL).

c. Dry the solid by continued suction for 5 minutes while proceeding with other work.

d. Transfer the Hirsch funnel to a beaker, leaving the boiling stones and filter-cake of crystals intact. Leave this assembly in your locker until next week. This will provide an opportunity for the crystals to dry thoroughly.

e. Discard the filtrate in the appropriate **organic waste container**. Clean the filter flask and microspatula with **wash grade acetone.**

EXPERIMENTAL PROCEDURES NEXT LAB SESSION

STEP 5 Weighing the purified E,E-debenzalacetone (sample vials are available at the front bench)

a. Place a **pre-weighed** sample vial on a piece of creased weighing paper. Using a microspatula carefully transfer the purified E,E-dibenzalacetone (though not the boiling stones) from the Hirsch funnel directly into the sample vial (the creased weighing paper is used solely to contain any spilled material). Transfer any spilled material to the sample vial. Reweigh the sample vial and determine the mass of purified E,E-dibenzalacetone.

b. Discard the boiling stones, filter paper and weighing paper into the appropriate **solid waste container**.

c. Clean the Hirsch funnel and microspatula with **wash grade acetone**.

STEP 6 Characterization of the crude and purified E,E-dibenzalacetone (melting point capillary tubes are available at the front bench)

a. Retrieve two melting point capillary tubes. Using a Sharpie® label the top of one with a single line. Label the top of the other with two lines.
b. Place a **few crystals** of crude E,E-dibenzalacetone on the ground-glass portion of a clean microscope slide (working over a creased weighing paper will contain any spilled material). Repeatedly crush the crystals into a smear using a clean microspatula (TA will demonstrate).
c. Clean the microspatula with **wash grade acetone**.
d. Place a **few crystals** of purified E,E-dibenzalacetone acid on the ground-glass portion of a separate clean microscope slide (working over a creased weighing paper will contain any spilled material). Repeatedly crush the crystals into a smear using a clean microspatula.
e. Fill the first capillary tube with crude E,E-dibenzalacetone in the manner employed in Units 1 and 2.
f. Fill the second capillary tube with purified E,E-dibenzalacetone in the manner employed in Units 1 and 2.
g. Discard the weighing papers in the appropriate **solid waste container**.
h. Clean the microspatula and microscope slides with **wash grade acetone.**
i. Using the method employed in Units 1 and 2 carryout simultaneous melting point determinations of your crude and purified samples of E,E-dibenzalacetone (using the same MelTemp device). Don't forget to record the voltage setting.
j. Discard the used melting point capillary tubes in the appropriate **waste glass container**.

STEP 7 Submission of the purified E,E-dibenzalacetone

Affix a "E,E-DBA" label (available at the front bench) to your sample vial. Submit vial as directed by instructor.