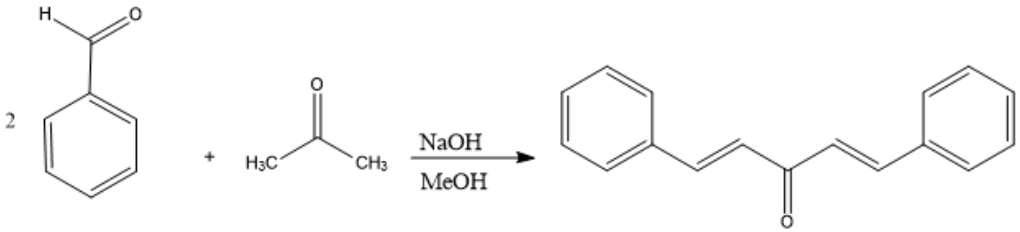
# CB102 : Experiment 9 : Synthesis and characterization of dibenzalacetone

## I- INTRODUCTION

Synthetic organic chemicals are widely used in many of the commercial products of our daily life such as detergents or pharmaceutics. One of these chemicals, dibenzalacetone, is use as a component for sunscreens due to its potential to absorb UV rays.

This molecule can be synthetize by a reaction called aldol condensation using two really common organic reagents, acetone and benzaldehyde.




Benzaldehyde Molecular weight: 106.1

Acetone Molecular weight: 58.1

Dibenzalacetone Molecular weight: 234.0

This procedure is a good example of “green chemistry”, which refers to exploring alternative synthetic route to minimize the impacts of chemistry on the environment.

**Objective: Synthetize dibenzalacetone, characterize it using IR spectroscopy, calculate the yield, and explain the mechanism of the reaction.**

What is the yield?

The yield of a reaction is the ration of the amount of product obtained experimentally to the expected amount of product theoretically, if all the limiting reagent was consumed.

The yield is a percentage given by the following formula:

$$\eta = \frac{amount\ of\ product\ obtained}{expected\ amount\ of\ product} \times 100$$

## II- PREMILINARY WORK

- Read carefully the procedure.

- What will be the limiting reagent in this procedure?

## III- PROCEDURE

**Chemicals required**:

- Acetone $C_3H_6O$ MW = 58.1 

- Benzaldehyde $C_6H_5CHO$ MW = 106.1 

- 10 % Sodium hydroxide NaOH solution 

- Methanol $CH_3OH$ 

**Apparatus and laboratory glassware required:** See appended.

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**IMPORTANT NOTICE:** To calculate the exact yield, write down cautiously the exact amount of reagents you weigh out rather than the one specified in the lab manuel.

Do not forget to write all your observations while performing the synthesis. It can partly be done in the form of diagrams.

**A) Characterization of benzaldehyde using IR spectrometry.**

Read carefully the manual of the IR-spectrometer provided

Obtain and draw the IR spectrum of benzaldehyde.

*Use the spectrometer in the presence of a professor.*

**B) Synthesis of dibenzalacetone**

Weigh out 1.8g of benzaldehyde in a 250mL conical flask. Then, add around 15 mL of methanol. Pipette 0.30 mL of acetone, add it to the flask(**Be quick as acetone is really volatile**) and close the flask with a cork.

Write down the exact amount of chemicals added.

Close the mouth of the conical flask with a cork and shake it for 2 minutes.

Add 15 mL of the provided 10% sodium hydroxide solution to the mixture and shake it vigorously for 10 minutes. Release the pressure 3 or 4 time during these 10 minutes to release the pressure.

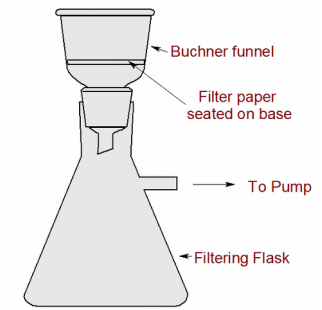
Put the conical flask into an ice-water mixture to cool it. Filter, wash and dry the solid formed using a vacuum pump.

***How to filter with a vacuum pump?***

*- Connect the filtering flask to the pump using the tube*

*- Put a filter paper inside the Buchner funnel (Make sure all the area of the Buchner funnel is covered by the filter paper)*

*- Take the mixture to be filtered inside the Buchner funnel and turn on the pump. the liquid will be sucked inside the filtering flask and the solid will remain inside the Buchner funnel*




*- Wash several time your product by adding distilled water to the Buchner funnel using the spatula to unstuck the product from the filter paper. Be careful to not make a hole in the filter paper.*

*- Important: Break the vacuum by removing the tube before turning off the pump.*

Weight a 25mL clean beaker, and transfer the solid in it. Dry your product with the desiccator for at least 15 minutes.

***How to use a desiccator?***

*- Connect the desiccator to the pump using the tube. Put your beaker inside the desiccator.*

*- Place the beaker with your product inside the desiccator.*

*- Close the tap.* ***Be careful, this is a three way tap, make sure that the pump and the desiccator are connected.***

*- Turn the pump on and let your product dry for at least 15 min.*

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Weight the dry product and calculate the yield of your procedure.

**C) Characterization of the dibenzalacetone using IR spectrometry**

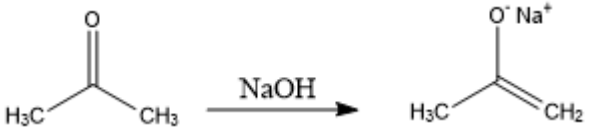
Obtain the IR spectrum of your product. Compare the two spectrums and explain its differences. Discuss the purity of your product.

**D) Mechanism of the reaction**

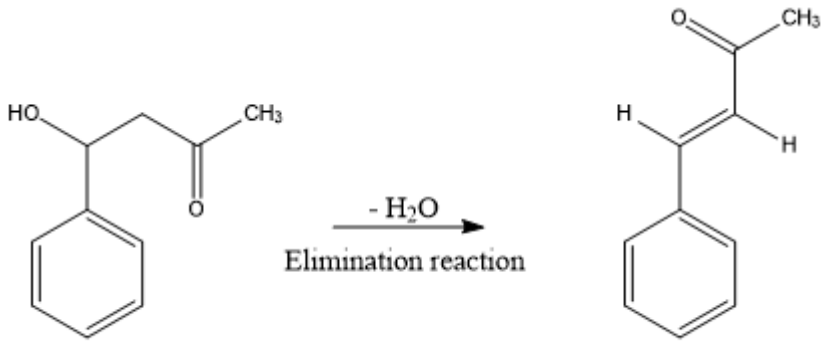
This reaction is known as an aldol condensation.

A base can abstract a proton from a methyl group of acetone to produce an enolate.

This enolate undergoes a nucleophilic attack on the aldehyde carbon of the benzaldehyde to produce an intermediate **1.** Then, a water molecule is eliminated to produce the next intermediate **2**.




Then another enolate is formed which will react with another molecule of benzaldehyde. A water molecule will be eliminated to give the product of the reaction.




**1** **2** (Trans isomer forms)

Suggest a mechanism for this reaction using the above information

**APPENDED:**

**Apparatus and glassware required:**

- 250mL conical flask
- cork for conical flask
- 50 mL beaker
- 25mL beaker
- spatula
- filtering flask
- Buchner funnel
- 50 mL measuring cylinder
- 500 mL beaker
- washbottle with distilled water
- 2mL graduated pipette
- pipette pump