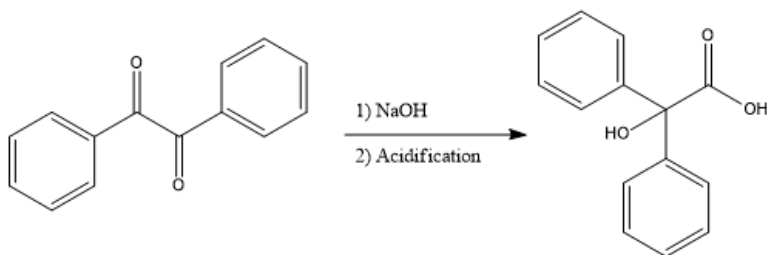


CB102 : Experiment 8 : Synthesis of benzoic acid via benzil in solid phase – Benzil-benzoic acid rearrangement

I- INTRODUCTION

Synthetic organic chemicals are widely used in many of the commercial products of our daily life such as detergents or pharmaceuticals. One of these chemicals, benzoic acid is used in organic synthesis as a base point for the preparation of pharmaceuticals. This kind of molecule is called precursors in organic synthesis.

The benzoic acid can be prepared in solid phase by a reaction called benzoic acid rearrangement.



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: Synthesize benzoic acid in solid phase, characterize it using a melting point apparatus, and calculate the yield.

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:

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

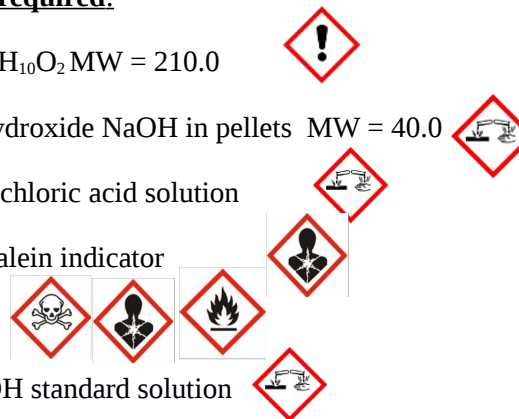
II- PRELIMINARY WORK

- Read carefully the procedure.
- What will be the limiting reagent in this procedure?
- Given V_E the volume at the endpoint obtained in the titration (last procedure). Find the expression of the purity in terms of V_E .

III- PROCEDURE

Chemicals required:

- Benzil $C_{14}H_{10}O_2$ MW = 210.0
- Sodium hydroxide NaOH in pellets MW = 40.0
- 2M Hydrochloric acid solution
- Phenolphthalein indicator
- Methanol
- 0.1M NaOH standard solution



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 weighed rather than the one specified in the lab manual.

1) Synthesis

Wear gloves for every steps of the synthesis procedure.

Make sure mortar, pestle, test tube and spatulas are dry.

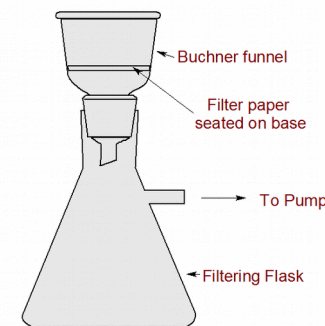
- Weight 1.0 g of benzil and take it into a dry mortar.
 - Weight around 1 g of NaOH pellets and add it to the benzil into the mortar.
 - Grind the mixture thoroughly using a pestle to make it a fine powder.
- Without using water, transfer the powder into a test tube and close the mouth of the test tube using a piece of polythene and tape. Put the test tube into a water bath at 100 °C and heat it for 30 minutes. *In the meantime measure the melting point of benzil (see end of procedure) and start writing the report.*
- After 30 minutes, take the test tube out of the water bath and cool it down to room temperature by placing the test tube in cold water.
 - Then take the content of the test tube in a beaker, and add about 50 mL of water. Mix well: the unreacted benzil will not be dissolved in water and can be removed by filtration. Filter the solution in a 100 mL conical flask, using filter paper (any type) and funnel.
 - Take the conical flask containing the filtrate (i.e. the benzoic acid in water) and cool it down by taking the flask into ice-water mixture. Acidify

the mixture under cold condition by dropwise addition of 2M HCl. A white precipitate will appear when the pH will be low enough. The pH should be around 2. (To control the pH, place a drop of the mixture on a single pH indicator paper with a glass rod.)

- Filter, wash and dry the precipitated benzoic acid using a vacuum pump:

How to use the vacuum pump to filter?

- 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) You will have to cut the filter paper to put it inside the Buchner funnel. To do so, put the Buchner funnel upside down and draw it from the filter paper using a pencil. Then cut it following the mark.
- Turn on the pump. Important: never switch off the pump if the tube is still connected to the flask.
- Wet the filter paper
- Carefully 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 times your product by adding distilled water to the Buchner funnel, and unsticking the product from the filter paper with a spatula. Be careful not to make a hole in the filter paper.
- Break the vacuum by removing the tube before turning off the pump



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2) Product characterization

- a) Yield: Weight the dry product, and calculate the yield of your procedure.
- b) Melting point: Check the purity of your product measuring its melting point following the procedure hereafter. Melting point of pure benzoic acid: 150-153 °C.

How to use a melting point apparatus

- Place a thermometer at the back of the device (larger hole)- Turn on the light- Insert a little bit of the substance to be analysed inside a capillarity tube
- Place the capillary tube inside the device (smaller hole)
- Increase the temperature slowly until you observe the product melting inside the capillary tube, and write down the exact melting point.
- If possible, repeat the experiment increasing the temperature more slowly when approaching the melting point. You would measure the melting point with better accuracy.

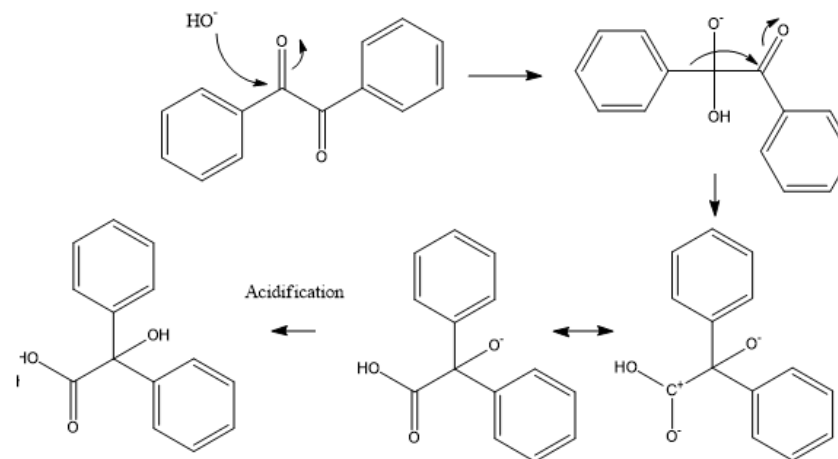
c) Determination of purity by titration:

- Weigh out around 0.5 g of your previously formed benzoic acid. Write down the exact mass of product you actually weighed.
- Pour it into a 50.0 mL volumetric flask
- Add around 10 mL of methanol to the flask. Shake to dissolve, and complete with water.
- Titrate 20.0 mL of this solution with the provided 0.1M NaOH standard solution, and 2 drops of phenolphthalein as indicator.

Compare the molarity of benzoic acid expected when preparing the solution, to the molarity obtained by titration and determine the purity of your product.

Calculate the actual yield, taking into account the purity.

Mechanism of the reaction. This reaction is a benzilic acid rearrangement:



APPENDED: Apparatus and glassware required:

- | | |
|------------------------------------|-------------------------|
| - wash bottle with distilled water | - glass stirrer |
| - large test tube | - two 250 mL beaker |
| - 100 mL conical flask | - 500 mL beaker |
| - funnel | - burette on stand |
| - filtering flask | - 50mL volumetric flask |
| - buchner funnel | - dropper |
| - spatula | - watch glass |
| - polythene and tape | - pH indicator paper |
| - dry mortar and pestle | |