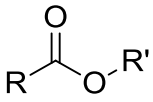
PW 03 : Synthesis of an Ester Used as a Food Flavoring

1. Introduction

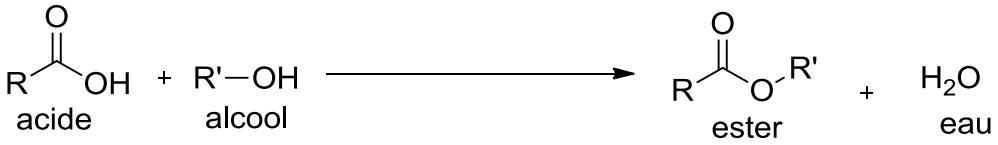
A food flavoring is a chemical compound that enhances the taste of food and beverages. It can be **natural**, extracted from plants by hydrodistillation, or **artificial**. It is not identified by any specific code on ingredient labels. Alkenes, alcohols, aldehydes, and ketones can be used as flavorings; however, the most commonly used substances are **esters**.

An ester has the following general formula:



With **R** = carbon chain or R = H, and R' = necessarily a carbon chain.

The esterification reaction: It is a reaction between a **carboxylic acid** and an **alcohol**.



2. Lab Equipment

Reflux setup, 20 mL and 1 mL pipettes, Propipette, 100 mL graduated cylinder, Separatory funnel, Erlenmeyer flask, Spatula

3. Chemicals

80% ethanoic acid, ethanol, concentrated sulfuric acid, saturated sodium chloride solution, 1 mol/L sodium hydrogen carbonate solution, anhydrous calcium chloride.

4. Synthesis of Ethyl Ethanoate

Ethyl ethanoate is an ester with a fruity apple-like smell. It is present in artificial flavors of apple, cherry, blackberry, pineapple, etc. It is a liquid that boils at 77°C. It can be synthesized through the reaction, in the presence of H^+ ions, between ethanoic acid and ethanol.

Procedure

- In a 250 mL round-bottom flask, introduce through the side opening:

- Using a graduated pipette and propipette, 20.0 mL of ethanoic acid (also called acetic acid)
- Using a graduated cylinder, 30 mL of ethanol
- Using a pipette with propipette, 1 mL of concentrated sulfuric acid
- A few boiling stones (boiling chips)

- Turn on the water in the condenser **slowly** and gently heat the mixture to a gentle boil for 30 minutes.
- Stop heating and lower the heating mantle.
- Remove the flask from the setup and allow the reaction mixture to cool, first at room temperature, then in a cold-water bath.

Extraction of the Ester

- Pour the contents of the flask into a separatory funnel containing approximately 50 mL of saturated sodium chloride solution. (*Note: retain the boiling chips*)
- Gently shake the funnel for a few moments (release gas pressure regularly), then discard the aqueous phase.
- Add to the organic phase 60 mL of 1 mol/L aqueous sodium hydrogen carbonate solution ($Na^+(aq) + HCO_3^-(aq)$). Let the gas release fully.
- Allow the mixture to separate and remove the aqueous phase.
- Collect the organic phase in a beaker. Dry it using anhydrous calcium chloride (or anhydrous copper sulfate), then filter the solution into a clean, dry Erlenmeyer flask.
- Determine the mass of the ester obtained.

Questions

1. Complete the labeling of the reflux setup.
2. Write the chemical equation for the esterification reaction.
3. Why is the reaction carried out under heat?
4. What is the role of concentrated sulfuric acid?
5. Using the data table:
   - Calculate the masses and initial mole quantities of each reactant.
   - Identify the excess reactant.
   - Why is one reactant added in excess, and why was that particular one chosen?
6. What is the theoretical maximum mass of ester that can be obtained?

Regarding the extraction:

7. Using the data:
    - Justify the presence of two phases.
    - Explain their position in the separatory funnel.
8. What is the nature of the gas formed when sodium hydrogen carbonate solution is added? Justify by writing the reaction equation. What is the purpose of this step?
9. What does it mean to “dry” the organic phase with anhydrous calcium chloride or anhydrous copper sulfate?
10. What is the appearance of the crude product? What impurities might still be present in the organic phase?

Reflux setup