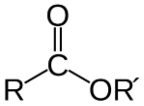
Lab N °05: Synthesis of an ester used as a food flavoring

Introduction

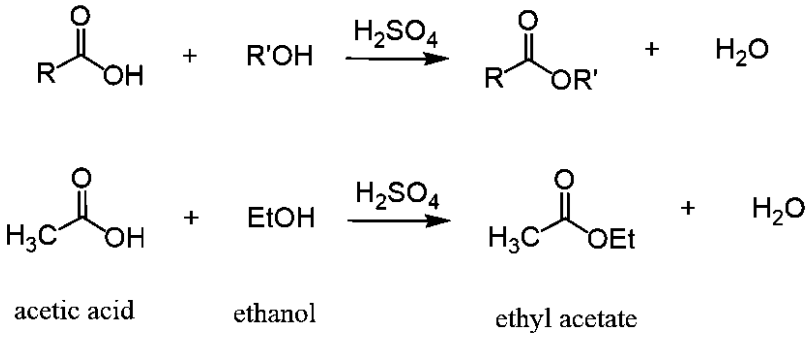
A food flavoring is a chemical species that enhances the taste of food and beverages; it can be naturally extracted by hydro distillation from plants or artificial. It is not identified by a specific code on labels. Alkenes, alcohols, aldehydes, and ketones are used as flavors; however, the most commonly used substances are esters."

Esters are organic compounds that are derived from carboxylic acids. The carboxyl group –COOH is contained in carboxylic acid. The hydrogen in this group is being replaced by a hydrocarbon group in esters. These hydrocarbon groups could be a methyl, ethyl, or phenyl group. Esters have a characteristic pleasant, fruity odor and are common in organic and biological materials. They are used mostly in the flavor and fragrance industry.

Esters are formed from the reaction of two reactants which are alcohols and acids in an esterification reaction. Fischer esterification is the classic synthesis of producing esters. This involves the treatment of carboxylic acid with an alcohol and a dehydrating agent. Sulfuric acid serves as catalyst to hasten this esterification process.



Where R may be a hydrogen atom, an alkyl group, or an aryl group, and R′ may be an alkyl group or an aryl group but *not* a hydrogen atom.



	*E*thanoic acid (*Acetic acid)*	*Ethanol*	*Ethyl ethanoate (Ethyl acetate)*
M (g/mol)	= 60,052	46g/mol	88
d	1.05	0,789	0.902
$T_b(°C)$	= 118	79	77

Materials & Equipment

Materials	Equipments
80% Ethanoic acid Ethanol Conc sulfuric acid H_2SO_4 Sodium chloride NaCl Sodium hydrogen carbonate Anhydrous $NaHCO_3$ calcium chloride	Reflux setup 20 mL and 1 mL pipette rubber bulb 100 mL graduated cylinder Separatory funnel Erlenmeyer flask Spatula

Synthesis of ethyl ethanoate

Ethyl ethanoate is an ester with an apple odor; it is present in artificial flavors of apple, cherry, blackberry, pineapple, etc. It is a liquid that boils at 77°C. It can be obtained by reaction, in the presence of H+ ions, of ethanoic acid with ethanol.

Procedure

Step 1: Ester synthesis

1. In a 250 mL double necked flask, introduce:
    - 20.0 mL of ethanoic acid (or acetic acid), using a volumetric pipette.
    - 30 mL of ethanol, using a graduated cylinder
    - 1 mL of concentrated sulfuric acid, using a pipette equipped with a micropipette.
2. Add a few grains of pumice stone.
3. Supply water to the condenser (CAUTION: open the valve very gradually).
4. Gently boil the mixture for 30 minutes.
5. Stop heating and lower the heating mantle.
6. Remove the flask from the setup and allow the reaction mixture to cool to room temperature and then put it in a cold-water bath.

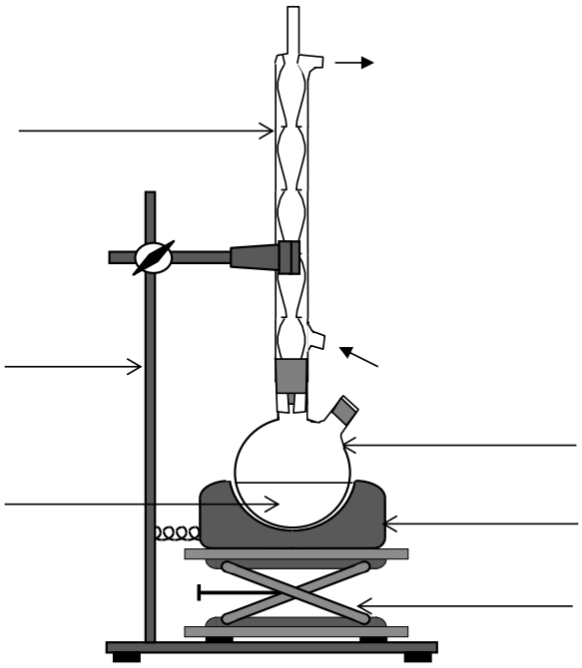
Step 2: Extraction of the ester

1. Pour the contents of the flask into a separatory funnel containing approximately 50 mL of a saturated solution of sodium chloride. ($NaCl$ solubility in water: $36g/100g$)
2. Gently agitate for a few moments (while regularly venting) and then remove the aqueous phase.
3. Then add 60 mL of a 1 mol/L aqueous solution of sodium hydrogen carbonate (Na^+_{aq} + $HCO_{3_{aq}}$) to the organic phase. Allow to vent until complete.
4. Let it settle, then remove the aqueous phase.

5. Collect the organic phase in a beaker. Dry this phase with anhydrous calcium chloride (or anhydrous copper sulfate) then filter, collecting the filtrate in a clean, dry Erlenmeyer flask.
6. Determine the mass of ester obtained

Questions

1. Label the parts of the reflux setup in the Figure below.
2. What is the role of the reflux apparatus?
3. Why use reflux setup and not placing a cap on the flasck?
4. Write the equation for the esterification reaction.
5. Why conduct the experiment at high temperature?
6. What is the role of concentrated sulfuric acid?
7. Using the data table, calculate the masses and initial quantities of each reactant.
8. Identify the reactant in excess.
9. Explain why one reactant is introduced in excess and why this one was chosen.
10. What is the maximum theoretical mass of ester that can be obtained?



1- Help not to loos volatile substances without building up pressure
2- The acetic acid could now be neutralized and removed by addition of 5 mL of 5% aqueous sodium bicarbonate solution. Carefully add the bicarbonate solution and shake the resulting mixture until no carbon dioxide gas is evolved. Separate and discard the lower aqueous layer. Repeat the extraction of acetic acid with two more 5 mL portion of sodium bicarbonate.

This process is called salting out. It is essentially a washing, which enhances the difference between the polarities of the two organic and aqueous layers by making the aqueous layer more polar. This forces any residual ionic (or polar) substance out of the organic layer, thus increasing its purity

The addition of saturated bicarbonate of soda will react with any unreacted acid The ester will be retained by organic phase; the carboxylate salt will cross to the AQUEOUS phase

Sodium bicarbonate is used to neutralize the unreacted carboxylic acid and the catalyst (concentrated sulfuric acid) that are dissolved in the organic layer.

$$RCOOH + HCO_3^- \quad \text{------>} \quad RCOO^- + CO_2 \uparrow + H_2O \text{ and}$$

Brine works to remove water from an organic layer because it is highly concentrated (since NaClNaCl is so highly water soluble). A saturated NaCl(aq) solution is highly ordered, causing a large motivation for water to draw into the solution from the organic layer to increase the entropy of the salt solution (to dilute the solution).

The purpose of this wash is to **remove large amounts of water** than may be dissolved in the organic layer. Although the organic layer should always be later exposed to a drying agent (e.g. anhydrous sodium sulfate, magnesium sulfate, or calcium chloride), these reagents at best remove only small amounts of water

An organic layer is always treated with a drying agent after having been exposed to water in a separatory funnel (step c) in Table 4.4). Drying agents are anhydrous inorganic materials that favorably form "hydrates", which incorporate water molecules into their solid lattice structure (for example, $Na_2SO_4 \cdot 7H_2ONa2SO4 \cdot 7H2O$). A drying agent is swirled with an organic solution to **remove trace amounts of water**.