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Synthesis, Isolation, and Purification of an Ester

AP Chemistry Laboratory #22

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Introduction

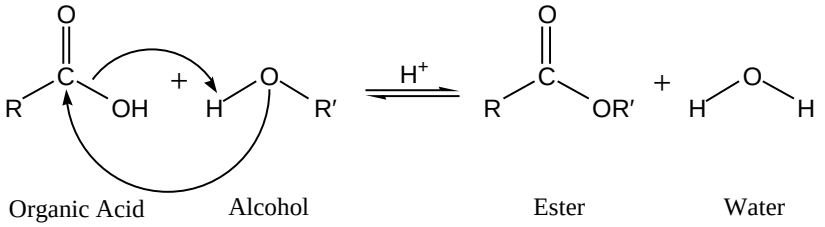
An ester is a chemical compound that is formed when an organic acid reacts with an alcohol. Esters frequently have distinctive odors and are naturally occurring flavor and fragrance chemicals in many fruits and plants. In this experiment, the ester ethyl acetate (ethyl ethanoate) is prepared and purified by distillation.

Concepts

- Esters
- Distillation
- Reflux
- Theoretical yield
- Percent yield
- Solvent extraction

Background

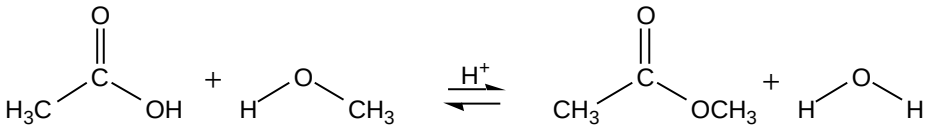
The reaction between an organic acid and an alcohol in the presence of an acid (H^+) catalyst is called esterification (Equation 1).



Equation 1

In the diagram, R and R′ represent organic groups such as hydrocarbons. The —OH group from the acid combines with the —H atom from the alcohol to form a water molecule. The R′—O— group from the alcohol attaches to the carbonyl carbon on the acid to produce the *ester*. The reaction is catalyzed by the addition of concentrated sulfuric acid, H_2SO_4, and the reaction is reversible. Adding concentrated sulfuric acid, a strong dehydrating agent, shifts the equilibrium to the products side by removing the water as it is formed.

Equation 2 illustrates a specific example of an esterification reaction, that of methyl alcohol and acetic acid, to form methyl acetate. The systematic name for acetic acid is ethanoic acid, and the systematic name for the ester product is methyl ethanoate.



Acetic Acid Methyl Alcohol Methyl Acetate Water

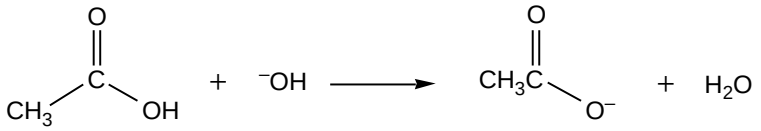
Equation 2

Experiment Overview

In this experiment, a quantitative esterification reaction is performed. The process has three parts—reaction, isolation, and purification.

In the reaction step, known amounts of acetic acid, ethyl alcohol, and sulfuric acid are combined and heated under *reflux. Refluxing* a reaction mixture involves heating the mixture to its boiling point in a flask equipped with a reflux condenser that allows a continuous return of the volatile materials to the flask. Using a reflux condenser, the reaction is conducted at a higher temperature without losing reactants or products.

Once the reaction is complete and the flask is cool, the ester is isolated and collected by the technique of *solvent extraction*. A solution of saturated sodium carbonate is added to a separatory funnel along with the contents of the reaction flask. These contents include any unreacted acetic acid, ethyl alcohol, and sulfuric acid along with the ester and water. The saturated sodium carbonate solution is strongly basic (pH ≈ 10). This basic solution will convert any undissociated acetic acid to its salt (Equation 3).



Acetic Acid Acetate Ion

Equation 3

By vigorously mixing this basic solution with the flask contents, the acetate ion along with the acidic and polar components are extracted from the flask mixture and two immisible layers result. The top, or less dense layer, contains the ester, while the bottom water layer contains sodium carbonate along with the water-soluble components acetate ion, ethyl alcohol, and sulfuric acid.

The ester layer is transferred back to the flask and purified by the technique called *distillation.* Distillation is the process of heating a compound to its boiling point and then removing the vapors by cooling them with a condenser. The purified ester product is identified by its boiling point and its odor. The condensed vapor, or distillate, is weighed, and the *percent yield* of ethyl acetate calculated.

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Pre-Lab Questions

1. Explain what an ester is.
2. Draw the structural formula for acetic acid.
3. Draw the structural formula for ethyl alcohol.
4. Write an equation using structural formulas for the esterification reaction of acetic acid with ethyl alcohol. Name the product.
5. Why are the reflux and distillation setups always open to the atmosphere?
6. How is distillation used to purify an ester?
7. The theoretical yield of a product is the maximum amount of product that can be formed from a given amount of reactants. In this esterification reaction, 12 mL of 17.4 M acetic acid are combined with 10 mL of absolute ethyl alcohol. The density of ethyl alcohol is 0.79 g/mL.
   a. Calculate the number of moles of each reactant.
   b. Compare the ratio of reactant moles used in this experiment to the ratio of reactant moles in the balanced equation. Are both reactants completely consumed? If not, which one is consumed? How much of the excess reactant is left over?
   c. The reactant that is completely consumed in the reaction is called the limiting reactant, because its amount limits the amount of product that can be formed. Once the limiting reactant is consumed, the reaction stops. Determine the limiting reactant for this experiment and calculate the theoretical yield of ethyl acetate, in moles and grams.

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Materials

Acetic acid, CH_3COOH, 17.4 M, 12 mL
Distilled or deionized water, 5 mL
Ethyl alcohol, CH_3CH_2OH, 10 mL
Sodium carbonate, $Na_2CO_3 \cdot 10H_2O$, 4.5 g
Sulfuric acid, H_2SO_4, 18 M (conc.), 15 drops
Beaker, 600-mL
Beaker, 50-mL
Boiling stones
Capillary dropper
Clamps, 2
Condenser and plastic tubing
Condenser outlet adapter
Erlenmeyer flask, 125-mL
Ground glass joint grease
Hot plate
Ring stands, 2
Round-bottom flask, 125-mL
Rubberbands
Option 1—Separatory funnel or test tube, 18 × 125-mm, and test tube stopper
Option 2—Test tube, 15 × 150-mm, and stopper
Thermometer, 0–100 °C, and adapter
Three-way adapter

Safety Precautions

Concentrated sulfuric acid is severely corrosive to eyes, skin, and other tissue; use extreme caution when handling. Ethyl alcohol is a flammable liquid and a dangerous fire risk; the addition of denaturants makes ethyl alcohol poisonous. Acetic acid is corrosive to skin and tissue; it is a moderate fire risk. The ester produced in this experiment, ethyl acetate, is a dangerous fire hazard; it is irritating to skin and eyes and slightly toxic by inhalation, ingestion, and skin absorption. Use extreme caution when distilling mixtures containing flammable liquids. Never smell chemicals directly by putting them under the nose. Instead, hold the compound at least eight inches from the face with one hand, and use the other hand to gently waft the vapors toward the nose. This lab should be performed in a fume hood or well-ventilated area. Wear chemical splash goggles, chemical-resistant gloves, and a chemical-resistant apron. Wash hands thoroughly with soap and water before leaving the laboratory.

Procedure

Preparation of ethyl acetate

1. Place 10 mL ethyl alcohol, 12 mL glacial acetic acid, 15 drops of concentrated sulfuric acid, and a boiling stone in a 125-mL round bottom flask.
2. Place a 600-mL beaker, filled with approximately 450 mL of water and a few boiling stones, on a hot plate.
3. Place the round bottom flask in the beaker of water so that the reaction mixture is below the water line. Clamp the flask to a ring stand (Figure 1).
4. Attach a length of tubing to the inlet (bottom) and another to the outlet (top) of the condenser.

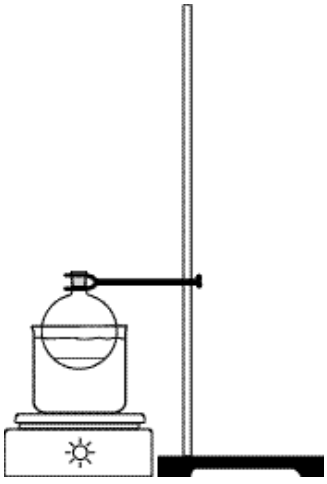

Figure 1.

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5. Attach the condenser to the round bottom flask, making sure the fitting between the two is secure and the condenser is vertical. If the connection between the two is a ground glass tapered joint, lightly grease the inner (male) joint to create a good seal. Clamp the condenser to the ring stand (Figure 2).

6. Attach the condenser inlet tubing to the water source and place the outlet tubing in the drain. *Slowly* run cold water through the condenser.

7. Turn the hot plate on and heat the round-bottom flask in the hot water bath. Raise the temperature of the hot water until the mixture in the round-bottom flask is gently boiling.

8. Continue the gentle boil of the reaction mixture for about 15 minutes. Turn off the hot plate and cool the mixture by removing the hot water bath.

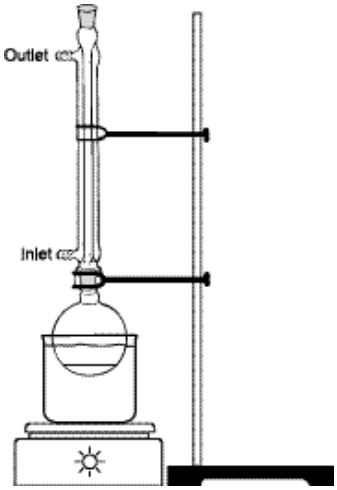

Figure 2.

Figure 3.

Isolation of Ethyl Acetate

1. Prepare a saturated solution of sodium carbonate by combining 4.5 g of sodium carbonate with 15 mL distilled water in a 15 × 150-mm test tube.

2. Stopper the test tube with a cork, shake well, and allow any undissolved solid to settle.

3. In the hood, pour the clear Na_2CO_3 solution into a separatory funnel (Figure 3) or, if none is available, into a 18 × 125-mm test tube.

4. Add the reaction mixture to the separatory funnel (or test tube), stopper and mix the solution. If using a separatory funnel, turn it upside down and open the stopcock occasionally to vent the system. If using a test tube, remove the stopper with caution—some pressure may have built up. Invert at least 15–20 times.

5. Allow the two layers to separate. Ethyl acetate (density 0.90 g/mL) is less dense than water, therefore the top layer is ethyl acetate.

6. If using the separatory funnel, remove the stopper, open the stopcock and slowly drain off the waste aqueous layer into a 50-mL waste beaker, then close the stopcock.

7. Transfer the remaining layer (ethyl acetate) to a clean, dry, round-bottom flask.

8. If using the test tube, remove the top layer (ethyl acetate) with a glass capillary dropper and transfer the ethyl acetate to a clean, dry, round bottom flask.

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Purification of Ethyl Acetate

1. Add a boiling stone to the round-bottom flask.
2. Set up the distillation as shown in Figure 4. For all ground glass connections, lightly grease the inner (male) joint to create a good seal.

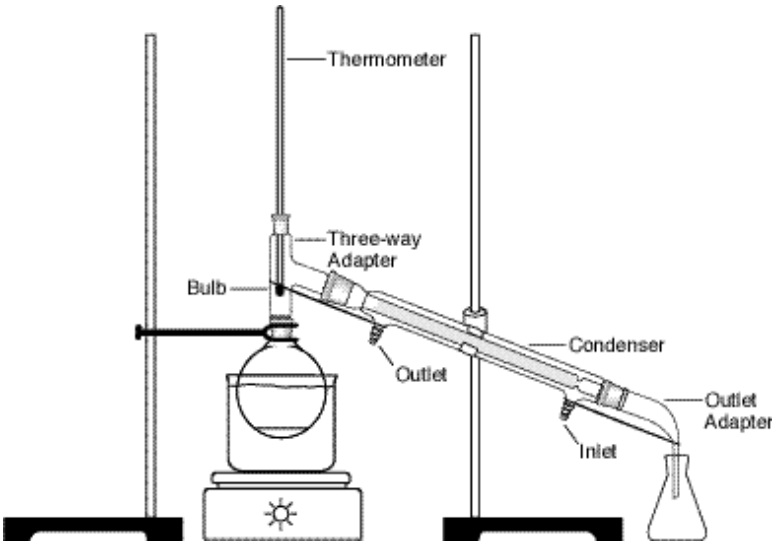

Figure 4.

3. Place the distilling flask in the 600-mL beaker and clamp, as in the preparation step 3.
4. Place the three-way adapter vertically in the neck of the distilling flask.
5. Insert the thermometer into its adapter.
6. Place the thermometer and its adapter in the top of the three-way adapter so that the thermometer bulb is just below the side arm.
7. Connect the condenser, with attached tubing, to the side arm of the three-way adapter and clamp it to a ring stand.
8. Connect the outlet adapter to the condenser. Add rubber bands to secure the condenser connections.
9. Weigh a clean, dry 125-mL Erlenmeyer flask on an analytical balance. Record the mass in the Data Table.
10. Place the Erlenmeyer flask under the outlet condenser. Check all fittings to make sure all connections are secure.
11. Attach the condenser inlet tubing to the water source and place the outlet tubing in the drain.
12. Slowly run cold water through the condenser.
13. Turn the hot plate on and heat the round-bottom flask in the hot water bath. Heat until the ethyl acetate is gently boiling.

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14. As the ethyl acetate vapors start to carry over and condense, record the temperature of the vapors in the Data Table. Record this temperature at the beginning and end of the distillation in the Data Table.

15. Distill the ethyl acetate until no more distillate comes over. There should be some liquid remaining in the round-bottom flask. *Never* distill to dryness!

16. Turn off the hot plate.

Identification of Ethyl Acetate

1. Remove the 125-mL Erlenmeyer collection flask and weigh the flask plus ethyl acetate on an analytical balance. Record the mass in the Data Table.

2. In the hood, add about 2 mL of the distilled ethyl acetate to a beaker with about 200 mL of distilled water.

3. Swirl the mixture and carefully smell the ester by wafting some of the vapors toward the nose. Record the odor and fragrance in the Data Table.

Disposal

Dispose of the ethyl acetate as directed by your instructor.

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Data Table

Boiling point range — ethyl acetate	_____ °C
Boiling point — beginning of distillation	_____ °C
Boiling point — end of distillation	_____ °C
Mass of Erlenmeyer flask	_____ g
Mass of Erlenmeyer flask plus ethyl acetate	_____ g
Mass of ethyl acetate	_____ g
Theoretical yield of ethyl acetate	_____ g
Percent yield of ethyl acetate	_____ %
Fragrance	_____

Post-Lab Calculations and Analysis

1. In Pre-Lab Question #6, part c, the theoretical yield of ethyl acetate, in grams, was calculated. Enter this value in the Data Table.
2. Use the mass of the ethyl acetate collected from the distillation and the theoretical yield to calculate the percent yield of ethyl acetate. Record this value in the Data Table.
3. Look up the literature value of the boiling point of ethyl acetate in a reference book, such as the *Merck Index* or the *CRC Handbook of Chemistry and Physics*. Compare the experimental and literature values of the boiling point.

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