Preparation of Esters

15B

Esters are a group of organic compounds best known for their interesting odors and flavors. Many natural odors and flavors were discovered to be esters and therefore, many synthesized esters are used in perfumes and foods.

An ester has the functional group $-\overset{\overset{O}{\|}}{C}-O-$, which is also known as an ester link. In the laboratory, an ester is usually formed from the reaction of a carboxylic (organic) acid and an alcohol, giving an ester and water as the products. This is an example of a condensation reaction, in which two molecules link up by the elimination of a small molecule between them; in this case, water.

We can write a general equation for the formation of esters as follows:

$$\underset{\text{acid}}{R-\overset{\overset{O}{\|}}{C}-O-H} + \underset{\text{alcohol}}{H-O-R'} \rightarrow \underset{\text{ester}}{R-\overset{\overset{O}{\|}}{C}-O-R'} + \underset{\text{water}}{H-O-H}$$

Here, R and R′ represent any alkyl group, of general formula C_nH_{2n+1}. Examples would be methyl (CH_3–), ethyl (C_2H_5–), or propyl (C_3H_7–). R and R′ can also represent any aryl group (one that contains a benzene ring) such as phenyl (C_6H_5–). The first part of the name of an ester is derived from the alkyl or aryl group of the alcohol used and the second part is from the carboxylic acid, using the ending -oate. As an example, if ethyl alcohol (ethanol) combines with propanoic acid, the resulting ester is named ethyl propanoate. The aroma of oranges is attributed to octyl ethanoate (formed from octanol and ethanoic acid) and apricots have an aroma because of the presence of pentyl butanoate (formed from pentanol and butanoic acid).

The reaction between the alcohol and acid is rather slow at room temperature. In order to speed it up and get an appreciable yield in the time available, you will use a temperature of about 60°C and add sulfuric acid to act as a catalyst in the reaction. In this experiment, you will prepare four esters and carefully smell them to see if there are any odors you recognize.

OBJECTIVES

1. to synthesize several esters and to try to identify the odor of each
2. to write the chemical equations for the formation of each ester using structural formulas

SUPPLIES

Equipment

4 test tubes (18 mm × 150 mm)
water-soluble marker
test-tube rack
dropping pipet
centigram balance
hot plate
2 beakers (250 mL)
2 graduated cylinders (10 mL)
thermometer
lab apron
safety goggles
plastic gloves

Chemical Reagents

methanol
ethanol
1-propanol
glacial acetic acid (ethanoic acid)
butanoic acid
salicylic acid
concentrated sulfuric acid, H_2SO_4

PROCEDURE

Concentrated sulfuric acid is a powerful oxidizing agent and dehydrating agent. If mixed incorrectly with the other chemicals used in this experiment a fire can result. If added to a small amount of water, a large temperature rise can occur, which can result in severe burns. Follow the directions exactly.

All of the acids used in this experiment are corrosive to skin, eyes, and clothing. Wear a lab apron, safety goggles, and plastic gloves while performing this experiment. Any spills or splashes must be washed off your skin and clothing immediately, using plenty of water. Report any spills to your instructor.

1. Put on your lab apron, safety goggles, and gloves.
2. Label the four test tubes 1 to 4 with your water-soluble marker and place them in the test-tube rack.
3. Into the appropriate test tube, pour the correct amount of the alcohol and add the corresponding carboxylic acid as indicated in Table 1 below. Your instructor may suggest alternative or additional combinations depending on the chemicals available. (Use the centigram balance to measure the solid salicylic acid). Add 4 drops of concentrated sulfuric acid to each test tube.

Table 1 Reagents for Preparation of Esters

Test Tube	Carboxylic Acid	Alcohol
1	1 mL acetic acid	1 mL ethanol
2	1 mL acetic acid	1 mL 1-propanol
3	1 g salicylic acid	1 mL methanol
4	1 mL butanoic acid	1 mL ethanol

4. Put about 150 mL of water in a 250 mL beaker. Place the test tubes in the water and heat the water on a hot plate to a temperature of about 60°C. Leave the test tubes in the hot water bath for 15 min. (Do not use a bunsen burner as the alcohol vapors are flammable.)
5. Cool the test tubes by immersing them in cold water in another beaker.
6. Add 5 mL of water to each of the test tubes.
7. Carefully note the odor of the contents of each of the test tubes in your copy of Table 2 in your notebook. Hold the test tube about 30 cm away from your nose and gently waft the vapors toward your nose without

inhaling deeply. Each of the odors should be somewhat familiar to you. Alternatively, the contents of the test tube may be poured into a beaker half full of water and the odor above it detected carefully.

8. Dispose of all materials following the reagent disposal instructions.
9. Before leaving the laboratory, wash your hands thoroughly with soap and water.

The alcohols and organic acids used in this experiment are all flammable. Do not use a bunsen burner to heat the water bath and make sure all burners or other sources of ignition in the laboratory are extinguished before the experiment begins.

Care must be taken when detecting odors. The vapors present can cause dizziness, drowsiness, sore throat, or headache if inhaled too deeply or in too great a quantity. Waft the odor toward your nose cautiously until the odor is detectable.

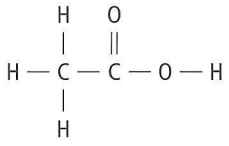
REAGENT DISPOSAL

Any remaining concentrated H_2SO_4 must first be diluted before disposal. To dilute, slowly add the acid to at least ten times its volume of water in a beaker. If requested by your instructor, place the diluted acid in the designated waste container. All other liquids can be rinsed down the sink with copious amounts of water.

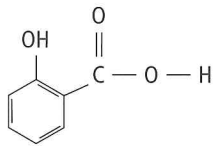
POST LAB CONSIDERATIONS

The reason for adding water to the contents of the test tube is to separate the esters from the reactants used. Esters are soluble in alcohol, but insoluble in water, and they generally have a density less than that of water, enabling them to separate and float to the top of the liquid mixture. This makes the detection of the odor more reliable.

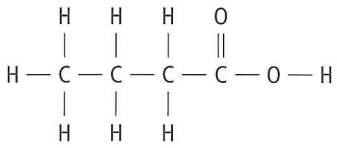
To obtain a better understanding of the chemical changes that occur, you must write the balanced equations for these reactions using structural formulas. The structures of the alcohols and carboxylic acids used in this experiment are as follows:



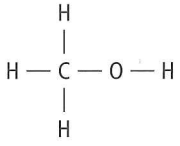
Acetic Acid (Ethanoic Acid)
CH_3COOH



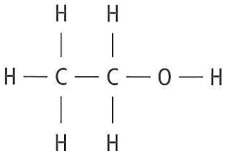
Salicylic Acid (2-hydroxybenzoic acid)
HOC_6H_4COOH



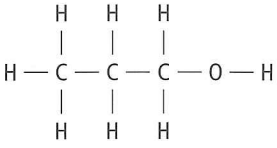
Butanoic Acid
C_3H_7COOH



Methanol
CH_3OH



Ethanol
C_2H_5OH



1–propanol
C_3H_7OH

EXPERIMENTAL RESULTS

Table 2 Odors of Esters

Test Tube	Name of Ester Formed	Odor
1		
2	COMPLETE IN YOUR NOTEBOOK	COMPLETE IN YOUR NOTEBOOK
3		
4		

ANALYSIS OF RESULTS

1. Using structural formulas, write the equations for the reactions tha occurred in each of the test tubes.
2. Name the ester formed in each of the test tubes.

FOLLOW-UP QUESTIONS

1. What is the name of the ester formed from each of the following combinations?
   a. ethanol and hexanoic acid
   b. methanol and pentanoic acid
2. What combination of alcohol and acid will form the following esters?
   a. hexyl octanoate
   b. methyl butanoate
3. Use a reference source to find two other esters that are used as flavorings Cite the reference source you used.
4. The ester methyl salicylate is also known as "oil of wintergreen." Name some commercial products that contain this substance.
5. Salicylic acid is both an acid and an alcohol, and can also form an ester with its –OH functional group and an acid. Draw the structural formula for the ester formed when the –OH group of salicylic acid links up with acetic acid (ethanoic acid). It should be called salicyl acetate, but usually goes by the name acetyl salicylic acid (sometimes abbreviated as ASA). What is it used for?
6. Use a reference source to identify the chemicals involved in the manufacture of the synthetic fiber, polyester. Explain how these chemicals permit the formation of a long chain of molecules joined by ester links. Cite the reference source you used.

7. Acid was used in this experiment to promote the formation of the ester, but if the base sodium hydroxide is added to an ester, the reverse process occurs and the ester undergoes hydrolysis (reaction with water) to reform its original alcohol and acid. Write the equation for the hydrolysis of the ester n-propyl butanoate, $CH_3CH_2CH_2COOCH_2CH_2CH_3$.

CONCLUSION

Make a list of the odors you were able to detect and the ester responsible for that odor.