

Experiment 13

What's That Smell? (Synthesis of Esters)

OUTCOMES

After completing this experiment, the student should be able to:

- Be able to identify the ester, carboxylic acid, and alcohol functional groups.
- Be able to systematically name esters.
- Predict the ester product to be made in each reaction.
- Use proper laboratory technique for identifying odors.

DISCUSSION

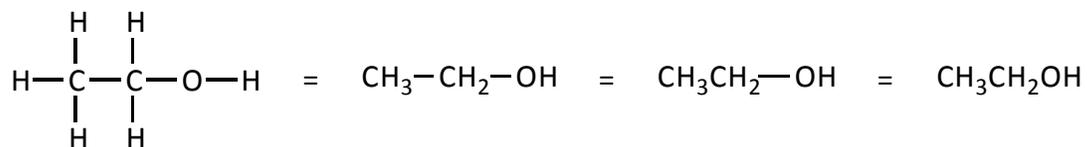
Organic chemistry is the study of carbon containing compounds. Carbon-containing compounds are found in many places, including in the food we eat and in our bodies as proteins, carbohydrates, and lipids. The focus of this experiment is on *esters*, which belong to a group of organic compounds known as carbonyl compounds. A carbonyl compound contains a carbon atom double bonded to an oxygen atom (C=O). Esters tend to have pleasant odors associated with flavoring and scents used in foods, perfumes, personal care products and many other commercial products. We will also be considering two other organic functional groups in this lab, carboxylic acids and alcohols. Carboxylic acids tend to have strong, foul odors.

A system of rules known as IUPAC is used to name organic molecules. The names and formulas of the normal alkanes containing 1 through 8 carbons are given below. Other organic molecules are named by modifying the end of the corresponding alkane name with a suffix based on the functional group the molecule contains:

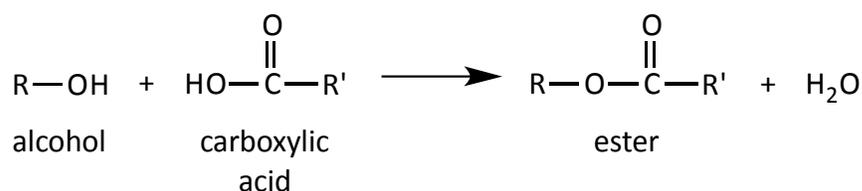
# of Carbons	Alkane name	Condensed Formula	Suffix	Functional Group	Formula
1	methane	CH ₄	-ol	alcohol	R—OH
2	ethane	CH ₃ CH ₃			
3	propane	CH ₃ CH ₂ CH ₃			
4	butane	CH ₃ (CH ₂) ₂ CH ₃	-oic acid	carboxylic acid	$\begin{array}{c} \text{O} \\ \parallel \\ \text{HO}-\text{C}-\text{R}' \end{array}$
5	pentane	CH ₃ (CH ₂) ₃ CH ₃			
6	hexane	CH ₃ (CH ₂) ₄ CH ₃			
7	heptane	CH ₃ (CH ₂) ₅ CH ₃	-yl -ate	ester	$\begin{array}{c} \text{O} \\ \parallel \\ \text{R}-\text{O}-\text{C}-\text{R}' \end{array}$
8	octane	CH ₃ (CH ₂) ₆ CH ₃			

In the general formulas in the rightmost column above, R and R' represent different hydrocarbon groups—the carbon-chain portion of the molecule derived from the alkane.

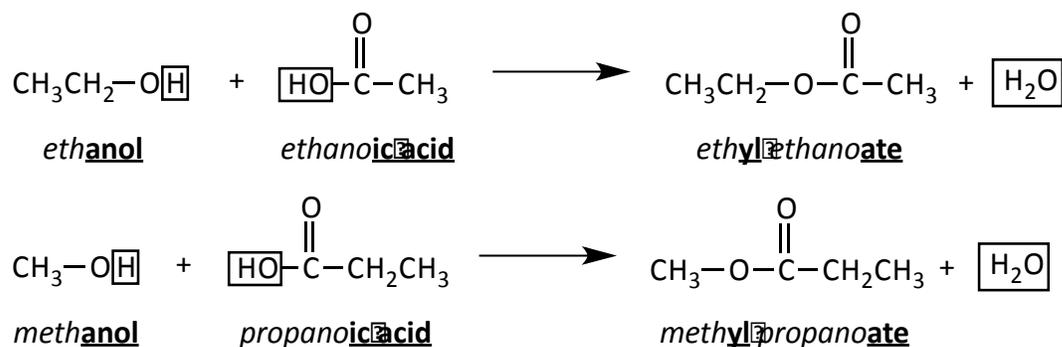
For example, a two-carbon alcohol would have a name based on ethane, the two-carbon alkane. The final e of ethane is dropped, and replaced with the suffix -ol to create ethanol. The structure may be written as a structural formula (with all bonds shown) or as a condensed formula, with each carbon listing the number of hydrogens attached.



In this lab, several different carboxylic acids and alcohols will be used to make several esters. Esters are readily prepared from the condensation reaction of a carboxylic acid and an alcohol, catalyzed by the addition of a strong acid such as sulfuric acid. This reaction is described as:



Two examples are shown below:



The reactions illustrate how the names of esters are derived. To name the ester, simply take the alcohol from which it is made, remove -anol from the end of the alcohol's name, and change it to a corresponding alkyl group by adding the suffix -yl. Take the name of the carboxylic acid, remove -ic acid from the end and replace it with the suffix -ate (similar to how nitric acid contains the nitrate ion). Therefore, the ester made by reacting ethanol with ethanoic acid is called ethyl ethanoate and the ester made by reacting methanol with propanoic acid is called methyl propanoate.

Sometimes common names are used rather than the systematic IUPAC names (shown above). The common name for ethyl ethanoate is ethyl acetate, since the common name of ethanoic acid is acetic acid. The common name for methyl propanoate is methyl propionate, since the common name of propanoic acid is propionic acid.

PROCEDURE

⚠ **Wear large chemical splash goggles at all times for this experiment.**

⚠ **Wear gloves while working with the chemicals in this experiment.**

1. Prepare a hot water bath by placing deionized water into a beaker and placing it on a hotplate. Heat the bath to a temperature of 80-95 °C. If the water in the beaker begins to boil, turn down the heat on the hot plate.
2. Label one test tube for each of the six esters you will synthesize. You may slide a piece of cut rubber tubing around the top of the test tube, just under the lip, so that it will be suspended when you place it in the hot water bath.

The following alcohols and carboxylic acids will be used to synthesize six different esters:

Ester	Alcohol (20 drops)	Carboxylic Acid (10 drops)	Catalyst (5 drops)
#1	ethanol (ethyl alcohol)	propanoic acid (propionic acid)	sulfuric acid
#2	3-methyl-1-butanol (isopentyl alcohol)	ethanoic acid (acetic acid)	sulfuric acid
#3	ethanol (ethyl alcohol)	butanoic acid (butyric acid)	sulfuric acid
#4	1-butanol (<i>n</i> -butyl alcohol)	ethanoic acid (acetic acid)	sulfuric acid
#5	1-octanol (<i>n</i> -octyl alcohol)	ethanoic acid (acetic acid)	sulfuric acid
#6	methanol (methyl alcohol)	salicylic acid	sulfuric acid

3. For the synthesis of each ester, add 20 drops of the alcohol and 10 drops of the carboxylic acid (or enough solid on the end of a spatula that is about the size of a large match head) to a small, labeled test tube.

⚠ **Use extra care when working with the concentrated sulfuric acid (H_2SO_4)! It causes severe chemical burns! Goggles and gloves are critical! Concentrated sulfuric acid is the most severely corrosive chemical you will use in this course. In the event of skin contact, immediately **rinse with cold water** for 15 minutes and report it to your instructor.**

4. Carefully add 5 drops of concentrated sulfuric acid to each test tube as a catalyst. Mix each of the solutions by holding the test tube at the top with one hand and use your other hand to "flick" the bottom of the test tube.

5. Place the labeled test tubes into the hot water bath (80-95 °C) for 10 minutes. Halfway through the heating, mix each of the solutions again by carefully removing each test tube one at a time, flicking the bottom, and replacing it. If the water in the beaker or any of the solutions in the test tubes begin to boil, turn down the heat on the hot plate. Do not allow the test tubes with the esters to boil dry.
6. After the reactions are finished, carefully note the odor of the ester from each test tube.

⚠ To safely smell chemicals, hold the test tube a safe distance from your face and gently waft the vapors toward your nose. Smelling chemicals directly can cause serious injury.

If the scent is not readily detected, try dipping a glass stir rod in the test tube and smell that. Alternately, you may pour some of the ester onto a watch glass and waft the aroma. Record the odor and any other chemical or physical changes you have noticed in the sensory table.

The odors for the esters in this lab include: banana oil, oil of wintergreen, artificial peach flavor, pineapple oil, orange oil, and an ester that is found naturally in apples, cherries, cheese, brandy and beer.

- ⚠ Turn off and unplug the hotplate when you are finished.**
- ⚠ Dispose of all chemicals in the proper waste container.**

PRELAB QUESTIONS

1. Consult the chapter in your text on organic chemistry. List five common substances that are considered "organic chemicals".

1.

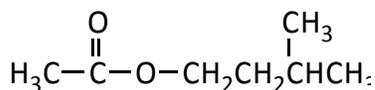
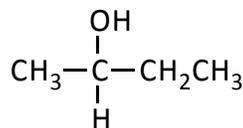
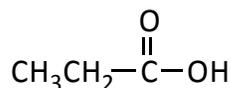
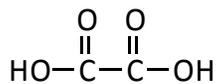
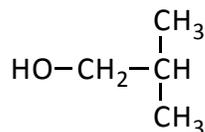
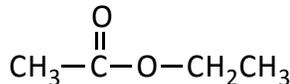
2.

3.

4.

5.

2. Identify each of the following compounds as an alcohol, ester, or carboxylic acid. Use the table on page 13.1 to help identify the functional groups. The direction of the functional groups may be flipped compared with the examples shown before.



Name _____

Lab Section _____

Partner's Name _____

DATA

Complete the sensory table below:

Compound	Odor
ester #1	
ester #2	
ester #3	
ester #4	
ester #5	
ester #6	

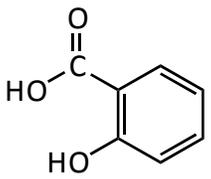
Draw the structural formula for each alcohol and acid. IUPAC names are given for each. (Note: Common names for some chemicals are included in parentheses.) For each ester, give the IUPAC name and draw its structural formula.

Alcohol Structure		Carboxylic Acid Structure		Ester Name and Structure
ethanol	+	propanoic acid (propionic acid)	=	(ester #1)
3-methyl-1-butanol + (isopentyl alcohol)		ethanoic acid (acetic acid)	=	(ester #2)
$\begin{array}{c} \text{CH}_3 \\ \\ \text{CH}_3\text{CHCH}_2\text{CH}_2\text{OH} \end{array}$				
ethanol	+	butanoic acid (butyric acid)	=	(ester #3)

Name _____

Lab Section _____

Partner's Name _____

Alcohol Structure	Carboxylic Acid Structure	Ester Name and Structure
1-butanol + (<i>n</i> -butyl alcohol)	ethanoic acid (acetic acid)	= (ester #4)
1-octanol + (<i>n</i> -octyl alcohol)	ethanoic acid (acetic acid)	= (ester #5)
methanol +	salicylic Acid 	= (ester #6)

