Preparation of Esters & Soap Lab

Objectives:
- Prepare several fragrant esters
- Prepare a soap & attempt to purify it.

Materials:
- 4 Test tubes
- Test tube rack
- Plastic pipettes
- Hot Plate
- 400-mL beaker
- 250-mL beaker
- Thermometer
- pH paper
- Filter paper
- Funnel

Chemicals:
- Acetic acid
- Ethanol
- Methanol
- Salicylic acid
- Benzoic acid
- Sulfuric acid

DO NOT CONSUME ANY OF THE PRODUCTS IN THIS EXPERIMENT! Wear safety glasses and gloves at all times.

Introduction:

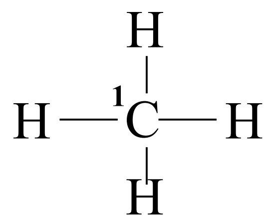
Hydrocarbons

Organic compounds are defined as those that contain carbon atoms. Hydrocarbons are a specific class of organic compounds that only contain carbon and hydrogen. Alkanes are saturated hydrocarbons that contain only single covalent bonds between carbon and hydrogen atoms. Their prefixes are determined based on the number of carbon atoms present in their hydrocarbon backbone. Hydrocarbons are named by the longest chain of carbon atoms in the molecule followed by the suffix *-ane.* For example, a hydrocarbon containing only one carbon is named *methane*.

Alkyl groups are substituents that contain one less hydrogen than the corresponding alkane. These are named by changing the -ane suffix of the corresponding alkane to –yl. For example, an alkyl group with one carbon and three hydrogens is considered a methyl group. These groups can be attached to an alkane.

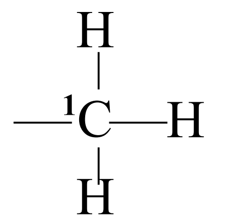
Prefix	# of Carbons
meth-	1
eth-	2
prop-	3
but-	4
pent-	5

ALKANE



Methane

ALKYL GROUP

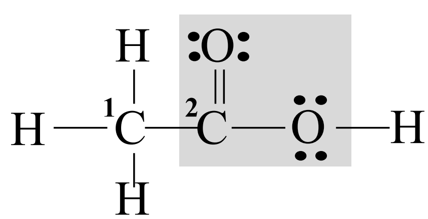


Methyl group

Carboxylic Acids

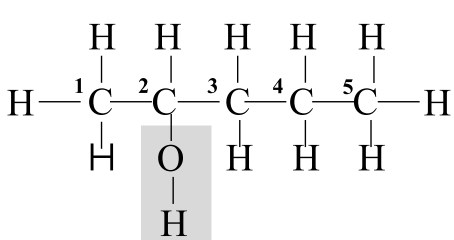
Carboxylic acids are weak organic acids that have the general formula R_COOH (R represents any carbon group). The hydrogen bonded to the oxygen is the only acidic proton present within the acid. While these carboxylic acids are classified as weak, they often sufficiently dissociate to create acidic solutions and, therefore, should always be treated with appropriate caution.

The nomenclature of carboxylic acids is dependent on the hydrocarbon structure as discussed above. The *-e* at the end of the alkane name is dropped and *-oic acid* is added.
For example the molecule below contains a 2 carbon chain. Therefore, its hydrocarbon base is ethane but, because it contains a carboxyl group, it is a carboxylic acid. *Ethane* is transformed into ***ethanoic acid***.

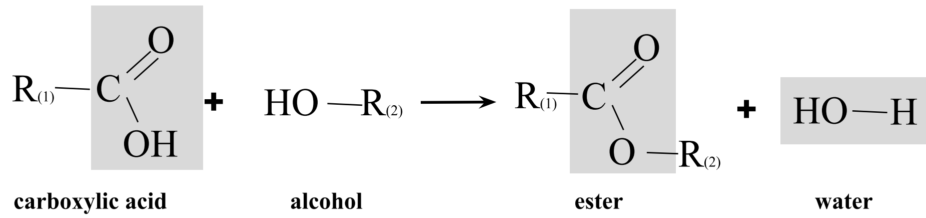


Alcohols

Alcohols are derivatives of hydrocarbons in which a hydroxyl group (-OH) has replaced a hydrogen atom. Alcohols are covalent molecules. Once again, the nomenclature of an alcohol is based on its hydrocarbon structure. First, identify the hydrocarbon base. Then, change the *-e* suffix to *-ol*. Finally, identify the number of the carbon in which the hydroxyl group is attached. This number is placed before the name. For example, the alcohol below is named ***2-pentanol.***

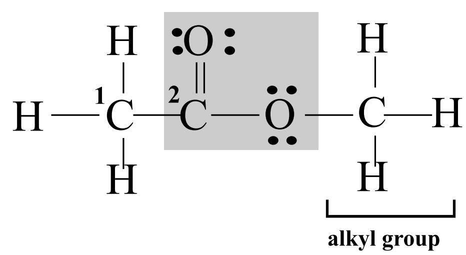


Esters



Carboxylic acids undergo a particularly useful reaction with alcohol to form esters (the reaction is shown above). The other product is water. The reaction is catalyzed by the addition of an acid. A distinct property of esters is their pleasant aromas. Since smell is a powerful component of the sense of taste, esters exhibit many odors which correspond to food flavors. Some examples include bananas, raspberries, pineapples, and wintergreen.

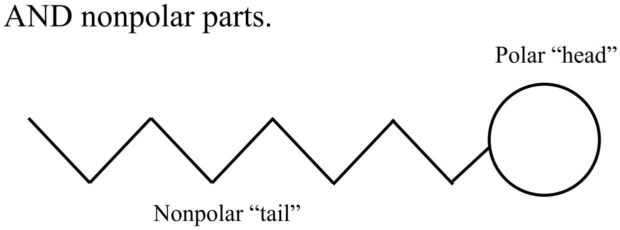
Esters are named by first naming the alkyl part of the alcohol (based on the number of carbons within the R group), then the carboxylic acid part is named by dropping the *–ic acid* ending and adding *–ate*. For example, the ester below contains two carbons as well as a single carbon alkyl group. Thus, the name of the ester is ***methyl ethanoate.***



Soaps

An interesting application of the esterification reaction is the preparation of soap. The soap making process is relatively simple: a source of fat and a strong base are heated together to produce a fatty acid salt. In primitive times, people made their own soap using a form of animal fat. The strong base, commonly *lye*, was prepared from fire ash, which could be dissolved in water to form sodium hydroxide (NaOH). Unfortunately, sodium hydroxide is quite harsh on the skin, causing drying and peeling at high concentrations. *Lye soap*, as it was called, was not particularly likable because of this. After you have made your own soap, you will attempt to purify it by removing as much of the remaining NaOH as possible to create more pleasant soap.

How does soap work? The cleaning action of soap works by removing dirt from skin or clothes. "Dirt" is a general term for any complex mixture of soil and debris. Usually this mixture is composed of both polar and nonpolar parts. Thinking back to our in-class discussion of polarity, we know that "like dissolves like." Therefore, water (which is polar) can dissolve and wash away the polar parts, but it is necessary to remove an oil surface layer with a nonpolar material first. Thus, a good soap has to have both polar and nonpolar parts in order to effectively remove dirt. Recall that soap is the salt of a long-chain fatty acid. The long hydrocarbon "tail" is nonpolar, while the "head" is polar. These fatty acid salts, therefore, make effective soaps because of their polar AND nonpolar parts.



In this experiment you will synthesize several fragrant esters, and prepare and purify your own soap. Tests on your soap will reveal the purity of your preparation as well as general properties of soap.

Procedure:

Part A: Synthesis of Esters

1. Prepare a hot water bath by filling a 400-mL beaker about one-third full of DI water. Place it on the hotplate on medium heat. You want the water bath to be about 70-80°C (do not boil the water).

2. Label three large test tubes #1, #2, and #3. Into each you will place an alcohol and a carboxylic acid. In every case use 1 mL of a liquid or a match-head sized sample of solid. The reaction requires a strong acid as a catalyst, so you will also need to add 6–8 drops of concentrated sulfuric acid.
   CAUTION: Sulfuric acid is a powerful reagent. It can cause severe skin burns. Handle it very carefully. If you spill any on your skin, wash immediately with soap and water and inform your instructor.

   The contents of each test tube is as follows:

#1	1 mL Butyl Alcohol	1 mL Acetic Acid	6–8 drops Sulfuric Acid
#2	1 mL Ethanol	solid Benzoic Acid	6–8 drops Sulfuric Acid
#3	1 mL Methanol	solid Salicylic Acid	6–8 drops Sulfuric Acid

3. Mix each well with a *clean* stirring rod. Check the temperature of the hot water bath. It should be below 90°C. If it gets too hot, simply turn off the hot plate or remove the beaker. Place the test tubes in the hot water bath.

4. Remove the test tubes after 10 minutes in the hot water bath.

5. Sample the odors by gently wafting the fumes from the test tubes to your nose. Try to identify each odor: apple, butter rum, & wintergreen.

6. Record your observations in your Report Form.

7. Dispose of the esters as directed by your instructor in the appropriately labeled waste container. DO NOT dispose of them by pouring down the sink.

Part B: Synthesis & Purification of Soap

1. Place 3 mL of liquid fat in a 250-mL beaker. Add 10 mL of ethanol and stir. To this mixture add 5 mL of 50% sodium hydroxide. Stir well.
   CAUTION: 50% NaOH is very concentrated. It can cause serious caustic burns, especially when heated. Be certain to wear your safety glasses and gloves. Wash immediately if any touches your skin. Inform your instructor.

2. Place the mixture in the beaker on a hotplate set to low heat.

3. Stir continuously as you heat. Be careful to avoid spattering. The mixture will turn into a thick paste as the soap forms and as the ethanol slowly evaporates. Remove the beaker when it looks like most of the liquid is gone. Allow the mixture in the beaker to cool.

4. Wet the tip of a *clean* stirring rod with distilled water and touch it to the mass of crude soap. Test the small sample on the stirring rod on a piece of pH paper. Record your observations in your Lab Report form.

5. Next, we will attempt to purify the crude soap by removing as much NaOH as possible. To the paste in the beaker, add about 30-35 mL of distilled water. Stir to dissolve. You may need to return the beaker to the hotplate to dissolve all of the solid. Remove once all the materials are in solution and allow to cool.

6. Obtain a small dish of salt (NaCl). Begin adding small amounts of solid sodium chloride. Stir. Then add more and stir. After several additions, you will note that the NaCl no longer dissolves in the solutions. At this point, the mixture is said to be *saturated*.

7. Ideally the soap should form in the beaker as a floating, foamy mass on the surface. It can then be isolated by skimming it off with a spatula onto a piece of filter paper. If the soap is not easily isolated, you will have to perform a filtration using a funnel. Seek help from your TA if you cannot isolate your soap product.

8. As done in Step 4, use a clean stirring rod to test the pH of the purified product. Have you removed some or all of the base?

9. Finally, place a small sample of the product in a test tube. Add about 1 mL of water and shake. Does your soap form bubbles.

10. **Dispose of your materials as directed by your instructor.** Clean your glassware thoroughly to ensure no soap/oil residue remains.

Esters & Soap Lab　　　　Lab Report

Name:___________________　　　　Date: _______________

Part A: Synthesis of Esters

Test Tube	Name of Ester	Fragrance
#1		
#2		
#3		

Part B: Preparation & Purification of Soap

Appearance of crude soap:

Appearance of purified soap:

Approximate pH of crude soap: ___________________

Approximate pH of purified soap: _________________

Were you able to remove some or all of the strong base (NaOH)? Use your knowledge of pH to explain.

Did your soap form a foam or bubbles?

Post-Lab Questions:

1.