CH 106 LAB MANUAL - CASCADE CAMPUS

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Caution

The experiments in this manual are intended to be performed under the supervision of a qualified instructor and with access to proper personal protective equipment and ventilation.

CH 106: Allied Health Chemistry III - Lab Manual

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CHAPTER OVERVIEW

1: Exploring Organic Structures

1.1: Organic Structures Procedure and Report Sheets

Organic Structures

It is very important to **build** the models as described in order to gain the visual experience with the molecules and their geometry. In Chemistry 106 it is expected that the student will have the basic understanding of Lewis Structures and VSEPR Theory from Chemistry 104 and be able to relate the physical structures and shapes to physical properties by applying intermolecular force theory between molecules from Chemistry 105. This lab is designed to allow you to review these concepts.

Because there is no prelab during week 1, the lab is worth 25 points total, 0.5 point for each numbered question unless noted.

Part A. Alkenes

Cis-Trans (Geometric) Isomerism

Alkenes are hydrocarbons containing one or more double bonds. They are referred to as *unsaturated* hydrocarbons. They are named according to the longest continuous chain of carbon atoms, adding –ene to the end, and using a number to indicate the position of the double bonds, if necessary. The simplest alkene is ethene, CH_2=CH_2, also known by the common name, ethylene.

First, we will review alkanes.

Make a model of butane, C_4H_{10}. The structure is shown below in Figure 1.1.1 using the line drawing and the structural formula.

Figure 1.1.1: Skeletal and structural formula for butane

Rotate the ***single*** carbon-carbon bonds such that the end methyl (CH_3) groups are on opposite sides of the center carbons.

Now, rotate the ***single*** carbon-carbon bonds such that the end methyl groups are on the same side of the center carbons.

1. What are the bond angles and molecular geometry around each of the carbon atoms?
2. Would the structures you have produced in parts a and b be considered isomers of one another? Explain your answer. (use a complete sentence)

Now **make a model** of cis-2-butene (in which both methyl groups are on the same side of the double bond). The structural formula for cis-2-butene is shown below:

Figure 1.1.2: Skeletal structure of cis-2-butene.

3. What is the approximate bond angle **and** molecular geometry around the 2 middle carbon atoms? (You do not need to memorize each specific bond angle in individual molecules. Think about the general geometry of C=C double bonds.)

4. Try to rotate or “spin” the middle carbon atoms. Is this possible with the double bond? Describe what you see. (1 point)

Make a model of trans-2-butene (the end methyl groups are on the opposite sides of the carbon-carbon double bond).

5. Draw the structural formula for trans-2-butene.

6. What are the approximate bond angles and molecular geometry around the double bonded carbons?

7. Can your trans-2-butene model be converted back into the cis-2-butene model without breaking bonds? Describe what happens when you try. (1 point)

The cis- and trans-2-butenes are called **geometric isomers.**

For numbers 8-11, you will indicate whether each of the following compound pairs are geometric isomers or structural isomers or not isomers at all.

Write the names for the compounds under each formula.

If they have the same names, then they **are the same molecule and are not isomers**. (**2 point each**)

8. For the following 2 structures:

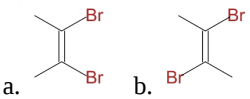
   a. CH_3 - CH = CH - CH_3 b. CH_3 - CH_2 - CH = CH_2

   Write the names:

   Are these structures isomers?

   If so what type?

9. For the following molecules:

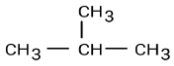


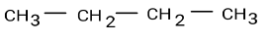
   Write the names:

Are these structures isomers?

If so what type?

10. For the following 2 molecules:



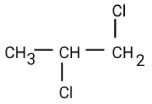


Write the names:

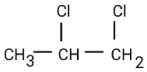
Are these structures isomers?

If so what type?

11. For the following 2 molecules:



and



Write the names:

Are these structures isomers?

If so what type?

Part B. Aromatic Hydrocarbons and Resonance

Construct a model of the benzene molecule from the structural formula.

It can be a struggle with the longer bonds to get them to fit the 3 double bonds in between the 3 single bonds.

Benzene is probably the most recognizable example of an aromatic hydrocarbon, it appears to have alternating double and single bonds, in a “ring or cyclic” structure, and is a specialized alkene. However, it has been determined that the structure is not the alternating double/single bonds (which is the Lewis Structure you constructed), but it is actually a series of 6 equal covalent bonds that are about longer than the length of a single bond, and shorter than a double bond (shown in the center structure below).

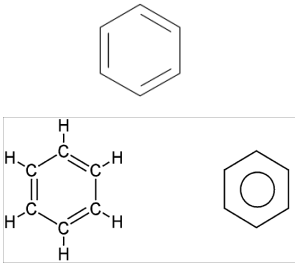


Figure 1.1.3: The three depictions of benzene.

Three different representations of benzene are shown above in figure 1.1.3, the stick model, the complete Lewis structure and the "ring" version. The circle in the ring reflects this and distinguishes the "aromatic ring" from a cyclohexane ring. It is possible to draw the Lewis Structure with the double bonds between the other carbons, this is called "resonance". (Review: Drawing Lewis Structures from Chemistry 104) Benzene compounds are the basis of a field of Organic Chemistry called "Aromatic Compounds".

The following questions are designed to guide you through understanding the structure and the related physical properties. Review Intermolecular forces (IMFs).

Study the structure you built and the structures drawn above.

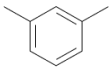
12. What are the bond angles and the geometry of each of the carbons in benzene?

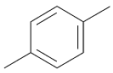
    (Hint: They are all the same.)

13. Draw the structural formula for methylbenzene.

14. What is the common name of this molecule?

Name the following structures use the **IUPAC** name.

15. 

16. 

17. Look up the boiling point of benzene and the boiling point of water.

Explain the difference in boiling points of these two compounds using the concept of intermolecular forces. (It is helpful to list the IMFs for each.)

    Use your own words for full credit. (2 points total).

Part C. **Alkynes**

Alkynes are hydrocarbons containing one or more **triple** bonds. They are also referred to as unsaturated. They are named according to the longest continuous chain of carbon atoms using a number to indicate the position of the triple bonds, if necessary. The simplest alkyne is ethyne, CHCH, also known by the common name, acetylene.

18. Construct a model of 1-propyne and draw its structural formula here.

19. What is the molecular geometry **around all 3 of** the carbon atoms

Part D. **Functional Groups**

The behavior of organic molecules is governed by the presence of ***functional groups***, which are combinations of atoms that act as chemically reactive sites on the molecule. Compounds with the same functional group will have many similar physical properties and undergo similar chemical reactions. Double and triple bonds are also considered to be ***functional groups***.

Build the following molecules and answer the questions based on the structures.

Alcohols

Alcohols have the general formula R-OH, where R is any alkyl group and the OH is called a **hydroxyl group**. Note: since the OH is bonded covalently to a carbon, it is not an ionic compound, therefore it is not a hydroxide ion.

Construct a model for 1-propanol, C_3H_7OH.

20. Draw the structural formula for 1-propanol, C_3H_7OH. (The common name is propanol, or propyl alcohol)

21. What is the molecular structure (molecular geometry) on each of the carbon's and on the oxygen?

Construct a model for 2-propanol, C_3H_7OH.

22. Draw the structural formula for 2-propanol, C_3H_7OH. (The common name is isopropanol, or isopropyl alcohol).

23. Write the definition of “structural isomers” here.

24. Are 1-propanol and 2 propanol structural isomers of each other?

Ethers

Ethers have the general formula R-O-R', where R,R' = alkyl groups.

25. Draw the structural formula of dimethyl ether, CH_3OCH_3.

26. Construct a model of ethanol CH_3CH_2OH, then draw the structural formula here:

27. Look up and list the boiling points of dimethyl ether and ethanol.

28. a. List the intermolecular forces between the dimethyl ether molecules.

(Hint: draw 2 molecules to see the IMFs.)

b. List the intermolecular forces between the ethanol molecules.

(Hint: draw 2 molecules to see the IMFs)

29. Explain the difference in the boiling points using the concept of intermolecular forces. Use your own words for full credit.

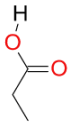
There are many types of ***Functional Groups*** that are listed in your text, most of which we will study during this course. Some are oxygen containing compounds: aldehydes, ketones, carboxylic acids and esters. Some are nitrogen containing compounds called amines. Some have both oxygen and nitrogen and are called amides.

Look up the general formula of aldehydes and ketones, carboxylic acids, ethers and esters.

Look up the general formula of amine and amides.

Name the **functional group only** in the following compounds. (You do not need to name the compound)

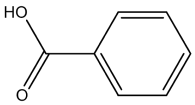
30. 

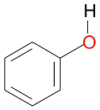
31. 

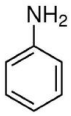
32. 

Match the following compounds with their common names:

Choices: Trinitrotoluene(TNT), para-Xylene, aniline, phenol, benzoic acid, benzaldehyde, toluene, ethylbenzene.

33. 

34. 

35. 

CHAPTER OVERVIEW

2: Reactions of Alkanes and Alkenes

2.1: Alkanes and Alkenes Prelab

Reactions of Alkanes and Alkenes Prelab (5 points)

1. What are the products of combustion of a hydrocarbon?

2. Write the balanced combustion reaction for 1-butene.

3. What is the color change that you are looking for in the Bromine Test to determine a substance is an alkane? An alkene?

4. What is the color change that you are looking for in the $KMnO_4$ Test to determine a substance is an alkane? An alkene?

5. Why is the reaction of ethene with bromine called an addition reaction?

2.2: Alkanes and Alkenes Procedure

Learning Objectives

- Observe the reactions of hydrocarbons with oxygen, bromine, and potassium permanganate.
- Use chemical tests to distinguish alkanes from alkenes.
- Draw the products of combustion, addition and/or substitution reactions of alkanes and alkenes.

Safety

- The organic compounds in the lab should be handled with care. If they are spilled on the skin, flood with water for 15 minutes. They are highly flammable and may cause dizziness if too much is inhaled. Wear gloves when working with this substance.
- $KMnO_4$ can cause skin and eye irritation. It also stains your skin and clothing with direct contact. If $KMnO_4$ is spilled on the skin, flood with water for 15 minutes. Wear gloves when working with this substance.
- Bromine is dangerous and can burn your skin with contact. If bromine is spilled on the skin, flood with water for 15 minutes while using soap. Wear gloves when working with this substance.
- **ALL WASTE FROM THIS LAB MUST GO IN THE WASTE CONTAINER PROVIDED.** Glass can be rinsed after the majority is in the waste container.
- **This experiment must be done in the hood.**

Background:

Alkanes are saturated hydrocarbons, as they only contain single bonds between carbon atoms. The alkenes and alkynes are unsaturated hydrocarbons, containing at least one double or triple bond respectively. The alkenes and alkynes are more reactive than alkanes due to the double or triple bond present. Alkanes are typically inert under normal circumstances. This means that they only participate in reactions that involve other more reactive substances. Most reactions involving alkenes are called addition reactions as they involve the addition of atoms or groups of atoms to the carbons involved in the double bond. This breaks the double bond leaving only a single bond. The reason these bonds are so reactive is that an addition reaction results in a net gain of bonds. Consider the addition of hydrogen to ethene.

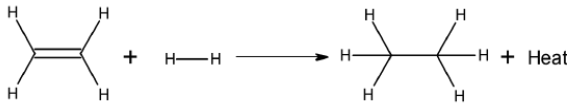


Figure 2.2.1: Reaction of ethene with hydrogen gas results in ethane and heat.

The double bond of the ethane and the single bond between the hydrogens are replaced with 3 new single bonds. Two of the bonds are C-H bonds and one is a C-C bond. This is an exothermic reaction. As energy is released in this reaction the products are more stable than the reactants. This move from a lower stability to higher stability is the reason the reaction occurs. The reaction still needs the proper activation energy to occur.

This lab will have you explore three types of reactions that alkanes and alkenes experience. You will look for the differences or similarities between these types of molecules in these reactions. The three reactions are: Combustion, Halogenation and oxidation.

Definition: Combustion

Combustion is the when a compound burns in the presence of oxygen. The products of this reaction are carbon dioxide and water. Both alkanes and alkenes combust in the presence of oxygen with enough activation energy.

$$CH_4(g) + O_2(g) \rightarrow CO_2(g) + 2\,H_2O(g) \tag{2.2.1}$$

Definition: Halogenation

Halogenation is the addition of a halogen atom(s) to an alkane or alkene with the addition of heat or light. For alkanes one atom of the halogen is added to a carbon of the alkane. For alkenes both atoms are added. One to each of the carbons involved in the double bond. Halogenation of alkenes will be a fairly fast process due to the higher reactivity of alkenes. Halogenation of alkanes will be a **slower** process as alkanes are not as reactive.

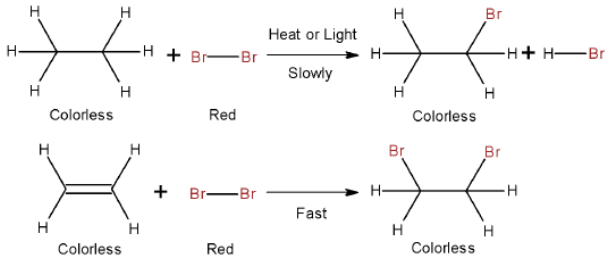


Figure 2.2.2: The reaction of bromine gas with ethane and ethene.

You will be using a bromine test in this lab. When bromine reacts with an alkene, the dark red color of the bromine disappears quickly as the atoms of the bromine bond with the carbon atoms in the double bond. If the red color disappears **rapidly**, we know the compound contains a double bond. If the color disappears **slowly** the compound does not contain a double bond.

Definition: Oxidation

Oxidation is the addition of oxygen atoms to the carbons of an organic compound. The oxidation of an alkene occurs at the carbons of the double bond. As alkanes have no double bonds the reaction does not occur.

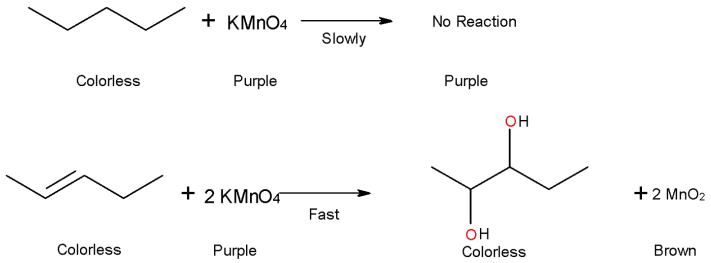


Figure 2.2.3: The reaction of $KMnO_4$ with ethane and ethene.

You will use a potassium permanganate test in this lab. When $KMnO_4$ reacts with alkenes it adds an OH to each carbon of the double bond. This causes a change from $KMnO_4$ which is purple to MnO_2 which is brown. No reaction would be seen with alkanes.

Materials

- 3 evaporation dishes
- Matches
- Pipet pump
- Dropper bottle of unknown
- Test tube rack with 6 test tubes
- Dropper bottle of 1% bromine solution
- Model kit
- Wooden splint
- Dropper bottle of cyclohexane
- Dropper bottle of cyclohexene
- Dropper bottle of 1% $KMnO_4$

Procedure

> **Note**
>
> There are not enough bottles of each reagent for each lab group. You will have to share reagents.

Combustion

1. **Working in the hood**, place 5 drops of cyclohexane in an evaporation dish. Using a lighted splint **carefully** ignite the sample.
2. Record your observations in the data table.
3. Repeat step 1 with 5 drops of cyclohexene
4. Repeat step 1 with one of the unknowns.
5. Rinse the split with water and place in the trash. Clean the evaporation dishes and set them to dry

Halogenation – Bromine Test

1. **Working in the hood**, place 15 drops of each hydrocarbon in a separate dry test tube. Carefully add 3-4 drops of the bromine solution to each. Observe whether the red color disappears immediately or not.
2. Record your observations in the data table.
3. Empty the contents of your test tubes into the waste container. Clean the test tubes in the sink and set them to dry on the rack you found them on.

Oxidation – Potassium Permangnate ($KMnO_4$) Test

1. **Working in the hood**, place 5 drops of each hydrocarbon in a separate test tube. Add 15 drops of 1% KMnO4 solution. Observe whether a change from purple to brown occurs.
2. Record your observations in the data table.
3. Empty the contents of your test tubes into the waste container. Clean the test tubes in the sink and set them to dry on the rack you found them on.

Identification of Unknown ($KMnO_4$) Test

Complete the questions as directed.

Modeling and Naming

Complete the questions and make the models as directed.

2.3: Alkanes and Alkenes Report Sheets

Name: _____

Lab Partner's Name: _____

Date of Lab: _____

Data Table (2 points)

Record your careful observations for the tests you perform.

Compound	Combustion	Bromine Test	$KMnO_4$ Test
Cyclohexane			
Cyclohexene			
Unknown			

Identification of Unknown

1. Is your unknown and alkane or an alkene? Write at least one paragraph in complete sentences describing the reasoning behind your choice (2 points)

2. Write a balanced reaction for each of the tests you did with the cyclohexane and cyclohexene. If no reaction occurred write "no reaction". (1 points each)
    - Combustion of Cyclohexane:

    - Bromination of Cyclohexane:

- Oxidation of Cyclohexane:

- Combustion of Cyclohexene:

- Bromination of Cyclohexene:

- Oxidation of Cyclohexene:

Modeling and Naming

1. Make models of the following and then draw the structure: (0.5 points each)
    - butane

    - pentane

    - ethene

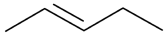
    - propene

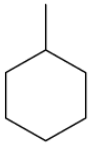
- 1-butene
- cis-2-butene
- Cyclohexane
- Cyclohexene
- 3-methylcyclohexene
- 1-methylcyclopentene
- 2-methyl-2-pentene
- chloromethane

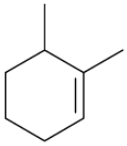
- 1,2 dibromoethane

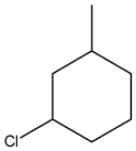
- 1,1-dibromoethane

2. Make models of the following and then name the structure: (0.5 points each)

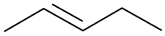
a. 

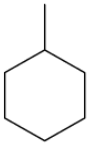
b. 

c. 

d. 

3. (0.5 points each) Write a balanced reaction of the following reactants. If there is no reaction write "no reaction".

a.  with $KMnO_4$

b.  and $KMnO_4$

CHAPTER OVERVIEW

3: Exploring Chirality

3.1: Chirality Prelab

Chirality Prelab (5 points)

Define the following terms:

1. Chirality

2. Isomer

3. Stereocenter

4. Enatiomer

5. Diasteromer

3.2: Chirality Procedure and Report Sheets

Learning Objectives

- To learn the isomer family tree.
- To use models to distinguish between chiral and achiral systems.
- To define and illustrate enantiomers and diasteromers.
- To learn how to represent these systems in two-dimensional space using a chemistry drawing program.

Materials:

Molecular Model Kit

Helpful Hints and Resources

- You will build the molecules, and use the structures to answer the questions in this lab report. You are asked to draw structures on this form or you may be directed to draw structures on your scratch sheet, be sure to read each question carefully.
- You will need to recall the concepts of Lewis Structures and the VSEPR (Valence Shell Electron Pair Repulsion Theory)

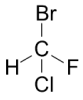
Procedure: (0.5 points per number question unless noted)

Part I. Stereocenters

1. Carbon atoms form four bonds in organic chemistry. Draw a picture of methane.

   (Do not use wedges or dashes in this compound in your drawing. We will introduce them later in this lab.)

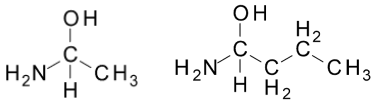
2. Stereocenters are carbon atoms that are bound to 4 different groups. Build the following molecule.



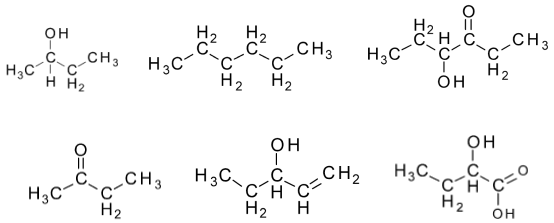
3. Now build the following molecule as well. Is the carbon in the following molecule a stereocenter?

4. Draw a chiral carbon, that is a carbon attached to four different single atoms. Be sure your compound is a complete and correct Lewis structure.
5. The carbon you just drew is a stereocenter. Carbons that have double bonds are not stereocenters. Why is this statement true?

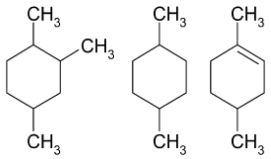
6. List the four different groups around the following carbon stereocenters in each molecule in the space below each molecule.



6. Number all the carbons from left to right starting with #1. List which carbons in the following molecules are stereocenters. For instance, in the molecule above there are two carbons, C1 and C2. C1, the carbon connected to the hydroxyl, is a stereocenter and C2, the other carbon, is not. If there are none write none below the molecule. (3 points)



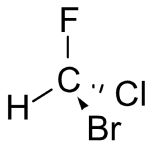
7. Build and then draw 1-bromocyclohexane and build and draw 1-bromo-2-chlorocyclohexane.
8. Notice 1-bromocyclohexane does not have a stereocenter because it is symmetrical. Whereas, 1-bromo-2-chlorocyclohexane is does have two stereocenters. Which carbons are stereocenters? (describe using words which C you mean)
9. Number the carbons from the top of the hexagon moving clockwise. Do not include the methyl groups in your numbering. List which carbons that are stereocenters. If there are none, write none below the molecule. (1.5 points)



10. Chiral molecules are optically active. In one paragraph of complete sentences explain what this means? (You may use the internet or your book for help.) (1 point)

Part II. Enatiomers

1. Build a molecule of methane with the models. What is the VSEPR geometry of methane?
2. Does the molecule below have a stereocenter?



3. Place the molecule in front of a mirror. Using the models build the image you see in the mirror. Draw a picture here.
4. Place one model on top of the other as if one was riding piggy-back on the other. When you do this keep the white, black, and red atoms as close as physically possible to the other white, black, and red atoms respectively. Are they superimposable? What is different about these two molecules?

5. These molecules are enantiomers. They have the same molecular formula, the some connectivity, but are spatially arranged about the tetrahedral atom differently. Enatiomers – stereoisomers that are nonsuperimposable mirror images. Switch the

positions of the orange and green atoms in one of these molecules. How are these two molecules now related? Are they still mirror images of each other?

6. Build a model of ethanol. Draw a picture of it below. (1.5 points)

   Using the models build the mirror image of this molecule. Piggy-back the two molecules in the same fashion as you did before.

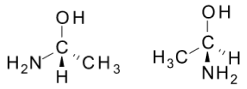
   - Do they overlap completely?
   - Does this molecule have one or more stereocenters (chiral atoms)?
   - Are these molecules enantiomers? Why or why not?
   - Does having a mirror image mean you have an enantiomer?

7. Build the following molecule: 1-chloroethanol. Draw a picture of it below.
8. Using the models create the mirror image of this molecule. Piggy-back the two molecules in the same fashion as above. (1 point)

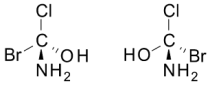
   - Do they overlap completely?
   - Does this molecule have one or more stereocenters (chiral atoms)?
   - Are these molecules enantiomers? Why or why not?

- Use what you see to explain what enantiomers are?

9. Make the following molecules exactly as shown. Be sure that the wedges and dashed bonds are in the right order. Are they enantiomers or the same molecule?

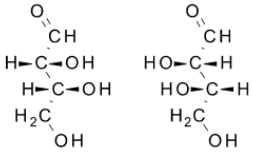


10. Make the following molecules exactly as shown. Be sure that the wedges and dashed bonds are in the right order. Are they enantiomers or the same molecule?



Part III. Molecules with Two or More Stereocenters

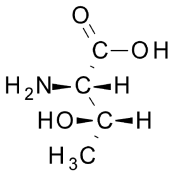
1. Make the models below. Remember that solid wedges are pointing at you and dashed ones are pointing away from you. These will look very different from the 2D picture. (It is sometimes easier to see if you leave the H's off the hydroxyl groups.)



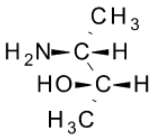
> Be sure to hold one up to a mirror and check to see if the other one looks identical to the reflection.

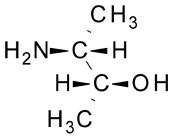
Do you have mirror images? If not be sure to check with your instructor.

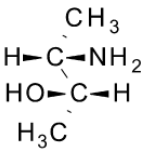
2. How many chiral centers does each molecule have? By number, which carbons are chiral?
3. Are these two molecules enantiomers?
4. At this point you might be asking yourself, why are these molecules different. To prove that they are unique molecules try to piggy-back them. Are they superimposable?
5. Can you spin them in any manner to make them superimposable, without breaking any bonds?
6. Go back to the positions you had at the end of step 1. Switch hydroxyl and hydrogen on carbon #3 on one of the molecules. (You are breaking bonds in this step.) (0 points)
7. Now you do not have mirror images. Therefore, these molecules are no longer enantiomers. However, you do still have two chiral centers. These molecules are now examples of diastereomers. Diastereomers are stereoisomers that are not mirror images. (0 point)
8. Here is the amino acid Theronine. Build this molecule.

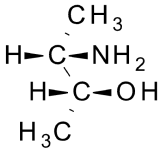


9. Determine which of the following 3-amino-2-butanol isomers are pairs of enantiomers and which are pairs of diastereomers. Be sure to compare all 4 structures with all the other structures. (1 point)

1. 

2. 

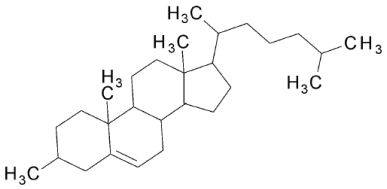
3. 

4. 

List your answer below.

Part IV. Chirality in Biochemistry

1. There are many examples of chiral molecules in biochemistry. Below is a picture of cholesterol. How many stereocenters does this molecule have? Circle them on the molecule below.(1 points)



An amazing fact about cholesterol is that our bodies only have one of its isomers.

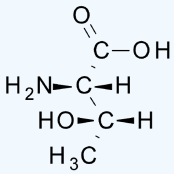
The formula for determining total number of isomers is as follows.

$$2^{x} = \text{number of possible isomers} \tag{3.2.1}$$

where x = Number of stereocenters.

Example 3.2.1

How many isomers does Theronine have?



Solution

Theronine has 2 stereocenters.

$$2^2 = 4 \text{ possible isomers} \tag{3.2.2}$$

2. How many isomers of cholesterol exists?

3. Ibuprofen is created as a racemic mixture. In one paragraph of complete sentences explain what this means? (You may use the internet or your book for help.) (1.5 point)

CHAPTER OVERVIEW

4: Gas Chromatography

4.1: Gas Chromatography Prelab

Using a Gas Chromatograph Prelab (5 points)

1. Complete the table below. Look up the boiling points online for the ketones listed. (1 point)
2. Predict the order in which these compounds will exit the GC column (known as *elution order*). Rate the compound you think will exit the column first with the number 1; the last compound to exit the column is given the number 3. (1 point)

Compound	Boiling point (°C)	Predicted elution order (1–3)
2-butanone		
cyclohexanone		
acetone		

3. Draw the structure of each compound. (3 points)

Compound	Structure
2-butanone	
cyclohexanone	
acetone	

4.2: Gas Chromatography Procedure

Using a Gas Chromatograph: Identifying Unknown Compounds

Learning Objectives

- Become familiar with the concept of using chromatography to separate substances in a mixture.
- Become familiar with using chromatography to identify unknown substances.
- Learn to operate and use the Vernier Mini GC
- Measure the retention time of three different ketones and analyze each peak as they pass through a Vernier Mini GC.
- Analyze a known mixture of the ketones as they pass through a Vernier Mini GC.
- Identify the ketones in a mixture (previous knowns) based on retention times.

Safety

- Obtain and wear goggles.
- Handle the organic chemicals with care. Avoid spilling it on your skin or clothing. Do not directly smell these chemicals.
- Properly handle and store the syringe and Mini GC.
- Ensure all organic chemical containers are closed when not in use.

Materials

- Vernier Mini GC
- computer or LabQuest
- Logger *Pro or* LabQuest App
- 1 µL glass syringe
- imwipes® or paper towel
- acetone
- 2-butanone (ethyl methyl ketone)
- cyclohexanone
- ketone mixture of all 3 ketones
- unknown ketone mixture

Discussion & Review

There are many different types of chromatography: paper, thin layer, liquid, high-pressure liquid (HPLC) and gas (GC). Chromatography is applied in many fields. Biochemists use liquid chromatography to separate proteins; chemists use GC and HPLC to identify organic compounds. Technicians use GC for drug tests, toxin screens and environmental analysis. Many forensic tests involve the use of chromatography.

All chromatography approaches operate under the same basic principles. There is a stationary phase and a mobile phase. The mobile phase (a gas or a liquid) travels along the stationary phase (the column or on a plate) from a start point to an end point. Compounds can travel from the start to the end at different rates, depending on whether they tend to “stick” to the stationary phase or “float” in the mobile phase. Compounds stick to the stationary phase through intermolecular attractions such as dipole interactions, dispersion forces or ionic interactions. When a substance has properties similar to the stationary phase it will tend to stick to it along the way and move through the mobile phase more slowly.

The Vernier Mini GC uses a metal column with the inside of the column coated with the stationary phase. A sample, consisting of one or more compounds, is injected onto the column and is pushed through by air, which acts as the mobile phase. Organic compounds flowing out of the chromatography column are seen as a *peak* on a chromatograph, as seen in Figure 1. The amount of time it takes for a compound to exit the column after it is injected is called the *retention time*. With a GC, a compound can be identified from a mixture of chemicals by its retention time.

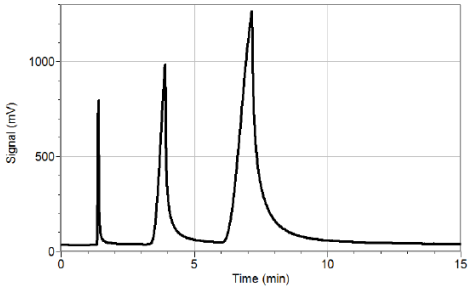


Figure 4.2.1. Sample Gas Chromatogram

Several factors can affect the interaction of a compound with the GC. More volatile compounds (i.e., compounds with a lower boiling point and weak Intermolecular forces) tend to move through the column faster because they are flowing in the mobile phase and interacting or “sticking" very little with the stationary phase. The functional groups present on the compound are also a factor. For example, alcohols may interact with a polar stationary phase more than esters because alcohols can form stronger hydrogen bonds. The molecular weight of a compound can also play a role, although it is not a simple matter of saying that the heavier the molecule, the slower it will travel through a GC column.

Part A: Procedure

> **Important**: The glass syringe is fragile and can be easily damaged. Be careful not to bend the needle or bend the plunger. If the plunger is accidentally pulled out of the glass barrel, reinserting it is extremely difficult, and sometimes impossible.

Figure 4.2.2. Syringe with liquid drawn to the 0.2 µL mark.

1. Obtain and wear goggles.
2. Obtain a glass syringe and a set of vials containing the three ketones, a mixture of all 3 ketones, and an unknown mixture of ketones to be tested. You will not only test acetone but use it to clean the syringe needle.
3. Prepare the Vernier Mini GC for data collection.
    a. Turn on the Mini GC.
    b. **Important:** Connect the USB cable of the Mini GC to the **USB port on your computer**.
    c. Start the data-collection program, and then choose New from the File menu.
    d. Click Collect in Logger *Pro*, or tap ► in LabQuest, to bring up the Temperature-Pressure profile.
    e. Set the Temperature-Pressure values to:

Start temperature	35 °C
Hold time	2 min
Ramp rate	5 °C/min
Final temperature	55 °C
Hold time	9 min
Total length	15.0 min
Pressure	5.0 kPa

    f. Select Done to initiate the Mini GC warm up. Note: A new message will appear, “Do not inject until GC is ready”, and the LED on the Mini GC is red. The Mini GC will take a few minutes to warm up and stabilize. When the Mini GC is ready for injection in Step 7, the message will read, “Inject and select Collect simultaneously”, and the LED will turn to green. Continue with Step 4 during warm up.

4. Follow the steps below to clean and flush the syringe with acetone. **Important**: The glass syringe is fragile. Be careful not to bend the needle or bend the plunger. Never pull the plunger back more than 50% of its total volume. Be careful not to bend the plunger as you press it down.
    a. Depress the plunger fully.
    b. Submerge the tip of the syringe needle into the vial of acetone.
    c. Pull back the plunger to fill the barrel about 1/3 full of acetone. Examine the barrel of the syringe and estimate the amount of acetone in the barrel.
    d. Expel the liquid onto a Kimwipe or a paper towel.
    e. Repeat Steps a–d at least two times, until you are comfortable pulling up a liquid into the syringe and measuring the volume in the syringe barrel. Use a Kimwipe or a paper towel to carefully pat around the tip of the syringe needle.
5. Collect a volume of acetone for injection.
    a. Submerge the needle into the vial of acetone one last time.
    b. Draw up approximately 0.2 µL of liquid. See Figure 4.2.2. It is not critical that the volume be exactly 0.2 µL; a tiny bit more or less volume is all right.

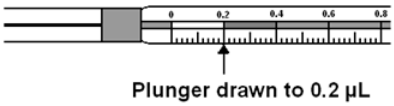


Figure 4.2.2. Syringe with liquid drawn to the 0.2 µL mark.

    c. After collecting your sample, use a Kimwipe to gently wipe the needle from barrel to tip.
6. Prepare for injection and the start of data collection. It is important for you and your lab partner to divide the tasks in this step. One person will operate the syringe and the other person will operate the computer controls.
    a. When the Mini GC has reached the correct start temperature and pressure, the message reads, “Inject and select Collect simultaneously,” and the LED on the Mini GC is green.
    b. To insert the needle of the syringe into the injection port of the Mini GC, hold the syringe with one hand and steady the needle with your other hand. Insert the needle into the injection port until the needle stop is fully seated, as shown in Figure 3. If the needle sticks, rotate it slightly while inserting. Do not move the plunger yet.

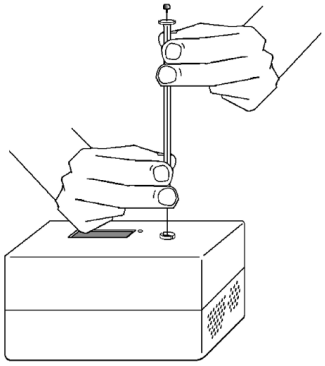


Figure 4.2.3. Injecting the liquid into the Mini GC.

    c. Simultaneously, depress the syringe plunger and select Collect to begin data collection. Pull the needle out of the injection port immediately.
7. While the data collection proceeds, repeat Step 4 with Acetone to thoroughly clean the syringe and needle. It may take more than three flushes to feel the syringe plunger move smoothly again, which is your indicator that the syringe and needle are both suitably clean.Choose Peak Integration from the Analyze menu.
8. Data collection will end after fifteen minutes.
9. Analyze your chromatogram.

a. Select and integrate the left-most peak. To do this, drag from a little before the peak to a point far enough to the right that includes all of the peak. Then choose Add.
b. Record the retention time in your data table.
c. Enter the name of the compound, if known.
d. To analyze another peak on the same graph, repeat Steps b and c.
e. When you are finished with all of the peaks, select OK.

10. (optional) You can choose to save this chromatogram and peak analysis for later use, with a unique file name, by choosing Save from the File menu.
11. Prepare another sample.
    a. Click Collect in Logger *Pro*, or tap ► in LabQuest, to bring up the Temperature-Pressure profile. This profile will be the same as for your previous run. If you are satisfied with these values, select Done to initiate the Mini GC profile.
    b. While the Mini GC adjusts to its Temperature-Pressure profile prepare the next sample.
    c. Follow the process in Step 4 using 2-butanone rather than acetone to clean and flush the syringe with 2-butanone.
    d. Repeat Steps 5–6 with a sample of 2-butanone.
    e. After the Mini GC is ready, repeat Steps 7–11 using your new sample.
12. Repeat Step 11 for the other 2 samples, cyclohexanone, mixture of the 3 ketones and the unknown ketone mixture. In total you should run 5 total samples.
13. When you have completed your final data collection run, turn off the Mini GC. Carefully put away the Mini GC and Hamilton syringe.

4.3: Gas Chromatography Report Sheets

Name (first and last):

Lab Partner (first and last):

Date of experiment:

Observations **(2 points)**

Write all observations below while performing the experiment as you work through each step. This will be your basis for answering questions and for validating your conclusion statement. You will refer to these observations for the answers to the questions and for the conclusion.

Data Tables (3 points)

Table 4.3.1. Boiling Points and Retentions Times for individual ketones.

Compounds	Boiling Point (°C)	Retention Time (min)
acetone		
2-butanone		
cyclohexanone		

Table 4.3.2. Retention Times for the 3 Peaks in the known mixture.

Elution Order	Compound	Retention Time (min)
1		
2		
3		

Analysis of the Unknown Mixture (2 points)

Record the retention times of the peaks present in the unknown.

Identify the compounds that relate to the retention time.

> **Note**
>
> Staple the chromatograms for your group to the lab report of one team member or upload these to the D2L assignment box and note this in each group members report sheets.

Table 4.3.3. Retention Times for the peaks in the unknown mixture. All spaces may not be filled in.

Elution order	Retention Time (min)	Proposed Compound
1		
2		
3		

Post Lab data analysis and questions

(2 point each unless noted)

1. Draw 2 molecules of acetone and show with labels all of the intermolecular forces that are present in a sample of pure acetone? (1 point)

2. Based on what you learned about intermolecular forces in CH105, explain the trend in ketone boiling points you reported in the pre-lab exercise.

3. In the Pre-Lab table, you predicted the elution order of some of the ketones. Did the elution times of the ketones support your prediction? Explain why or why not use your data to support your answer.

4. 2-hexanone and 4-methyl-2-pentanone are isomers, thus their molecular weights are equal. Suggest reasons for their differing boiling points. Predict their relative GC retention times. (hint: draw each structure)

5. Draw the structure of diethyl ketone (3-pentanone) below. Based on the results of your testing, predict the retention time of diethyl ketone. Explain your prediction.

6. If you ran the GC at higher temperatures, how would this change the retention time of the ketones you examined in this lab? Why?

7. Draw the structure of isopropyl alcohol (2-propanol) below. Assuming that the stationary phase in the column can act as both a hydrogen bond donor and a hydrogen bond acceptor, how would you expect the retention time of isopropyl alcohol to compare to that of acetone? Why?

CHAPTER OVERVIEW

5: Synthesis of Aspirin

5.1: Aspirin Prelab

Synthesis of Aspirin Prelab (5 points)

Read the lab completely prior to attending lab.

1. The reaction to form an ester is classified as a _____ reaction. (0.5 point)
2. Calculate the molar mass of salicylic acid and acetylsalicylic acid. (0.5 point)

3. Calculate the theoretical yield of acetylsalicylic acid from 2.01 g of salicylic acid assuming excess acetic anhydride. (2 points)

4. Assume that 1.93 g of acetylsalicylic acid is produced from the reaction in the previous question. Calculate the percent yield. (2 points)

5.2: Aspirin Procedure

Learning Objectives

- Understanding the chemical reaction involved (esterification)
- Accurately performing laboratory techniques like weighing, heating, and filtration.
- Calculating the percentage yield of the product
- Practicing safe lab techniques in a chemistry lab under the supervision of a trained instructor.

Safety

- Keep acetic anhydride under the hood and avoid breathing its vapors.
- Use the hood when using acetic anhydride or phosphoric acid.
- Place all waste in organic waste containers in the fume hood.
- If you spill acetic anhydride, notify your instructor.
- If you spill phosphoric acid, notify your instructor.
- Do not ingest the aspirin.
- Take care not to directly touch the chemicals.

Introduction

In CH 106, you have been introduced to organic chemistry nomenclature and functional groups. The active ingredient in commercial aspirin is the organic compound, acetyl salicylic acid which is classified as an ester. The general formula for esters is RCOOR', where R and R' may be different alkyl (carbon) groups.

Esters are widespread in nature and are often responsible for pleasant flavors in fruits and floral aromas. Esters are most often prepared from an esterification reaction between a carboxylic acid (R-COOH) and alcohol (R-OH) in the presence of a catalytic acid. The reverse reaction, a hydrolysis, also occurs.

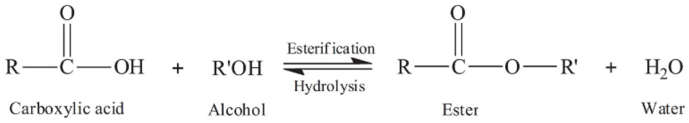


Figure 5.2.1. General format of an esterification reaction.

In today's lab, we will react salicylic acid, a naturally occurring pain reliever found in the bark of willow trees, with acetic anhydride. The mechanism of this reaction is similar to an esterification forming acetyl salicylic acid (aspirin), and acetic acid as a byproduct.

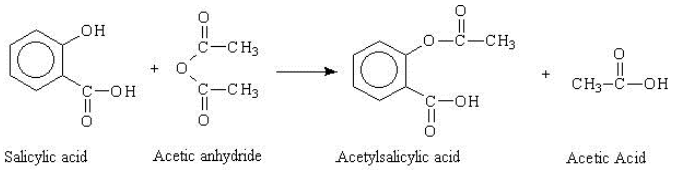


Figure 5.2.2. The synthesis of aspirin is an esterification reaction.

Organic chemistry reactions seldom result in pure products. A desired reaction may have unreacted starting materials or competing reactions resulting in additional products. There are ways to purify the sample including recrystallization, which is based on chemical solubilities within a mixture. Impurities will dissolve within a hot solution and upon cooling, the desired pure substance may form crystals that may be collected.

To test the purity of the aspirin we created we will determine the melting point of our sample. The melting point is the temperature at which the solid coexists in equilibrium with the liquid at atmospheric pressure. Most pure organic compounds have sharp melting points; that is the process of melting occurs over a very narrow range of temperature. Impurities have a dramatic influence on the melting points of organic compounds. They typically lower the melting point and widen the melting point range. For example, while pure acetyl salicylic acid (aspirin) melts sharply at 138 – 140 °C, a sample of wet acetyl salicylic acid (water is the

impurity) may melt in the range 126 – 130 °C. In contrast the melting point of salicylic acid is 158 – 160 °C. A melting point range larger than 3°C is an indication that the sample is either impure or wet.

Procedure

Part 1. MSDS and GHS

An MSDS (Material Safety Data Sheet) is designed to give information about the risks involved with using a particular chemical. For every chemical PCC purchases, it is required that the appropriate MSDS is loaded into our online database. You will read the MSDS's for the chemicals in today's lab. First focus on the GHS pictograms that appear at the beginning of the MSDS. These are an international system intended to give a quick overview of the risks associated with a particular reagent. Here is a table of what the pictograms mean:

Hazard Class Pictograms ☝

Read the hazard statements that directly follow the pictograms. They will give a more complete picture of what you need to be mindful of using a particular reagent.

Category	Pictogram	Information
Explosives		Explosives, Self-Reactives, Organic Peroxides
Flammables		Flammables, Pyrophorics, Self-Heating, Emits Flammable Gas, Self-Reactives, Organic Peroxides
Oxidizers		Substances that can cause or enhance the combustion of other materials.
Compressed Gases		Compressed Gases, Liquified Gases, Dissolved Gases
Corrosives		Skin Corrosion/Burns, Eye Damage, Corrosive to Metals
Acute Toxicity		Acute Toxicity (fatal or toxic)
Irritant		Irritant (skin and eye), Skin Sensitizer, Acute Toxicity (harmful), Narcotic Effects, Respiratory Tract Irritant
Health Hazard		Carcinogen, Mutagenicity, Reproductive Toxicity, Respiratory Sensitizer, Target Organ Toxicity, Aspiration Toxicity
Environment		Aquatic Toxicity (Non-Mandatory)

To identify the hazards associated with the chemicals in this lab, complete the table in your post lab. The first line is completed as an example. The MSDS documents you need can be found posted with the labs online.

Part 2: Synthesize the Aspirin

1. Place a heating/stir plate in the fume hood. Place approximately 350 mL of tap water in a 500 mL beaker. Place a stir bar in the beaker, and place the beaker on the hot plate. Set the stirring at 200 rpm and turn the heater ¾ of the way to full. Place a portable ring stand on one side of the stir plate and attach a utility clamp to the ring stand.
2. Tare a scale with a 125 mL Erlenmeyer flask on top. Place about 2 g of salicylic acid in the flask and record the mass.
3. In the hood, add 5.0 mL of acetic anhydride to the Erlenmeyer flask. Your instructor will move the bottle of acetic anhydride from one hood to another. Use the pipette attached to the side of the bottle to dispense the anhydride into a 10 mL graduated cylinder.
4. Add 5 drops of 85% phosphoric acid to the Erlenmeyer flask. Use the pipette attached to the side of the phosphoric acid bottle. Your instructor will move the bottle of phosphoric acid from one hood to another.
5. Attach the Erlenmeyer flask to the utility clamp. After it is secure, lower the flask into the beaker of tap water.
6. Take the glass stirring rod and place it in the Erlenmeyer flask. Every few minutes, stir the contents of the flask until you see that the salicylic acid has completely dissolved. Leave the glass stir rod in the flask.
7. Let the flask sit in the water bath until the water reaches boiling. This will probably take about 20 minutes.
8. Once the water reaches boiling, turn off the heat on the hot plate but keep it stirring. Let the Erlenmeyer flask remain in the water for 10 minutes.

Complete steps 9 and 10 while you are waiting.

9. While you are waiting, place about 50 mL of distilled water in a 100-mL beaker and place it into an ice-water bath. The ice-water bath is made by filling a 500-mL beaker half-full with ice, then just covering the ice with tap water.
10. Also while you are waiting, weigh a watch glass with a piece of filter paper.
11. After your reaction has sat for 10 minutes, raise the clamp on the ring stand so the Erlenmeyer flask is no longer in the water bath. Add 2 mL of room temperature distilled water. Stir the mixture and then allow it to sit for a minute. This water decomposes any unreacted acetic anhydride to acetic acid.
12. After a minute, add 20 mL of room temperature distilled water. Stir it a couple of times and then leave it alone. Let it cool to room temperature undisturbed. Be patient. After time has passed and the flask no longer feels warm to the touch, take it out of the hood and place it in your ice+water bath. Leave it alone and let it cool further. Let it sit in the ice for at least ten minutes. You can do steps 13 and 14 while you wait.
13. While you are waiting, clean up the stir plate, ring stand, beaker, and clamp that were in the hood.
14. Set up a Buchner funnel on a vacuum flask. Use a ring stand and clamp to stabilize the Buchner funnel. (See Figure A below) You will need to place a rubber cuff between the funnel and the flask. Attach tubing from the flask to the vacuum line in the hood. Place the paper filter you weighed into the top of the funnel (do not place the watch glass in the funnel).

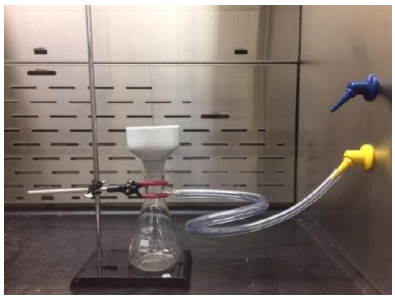


Figure 5.2.3. Buchner Funnel set up in a hood.

15. When crystals have fully formed in your flask, filter them. First, use distilled water from a squirt bottle to moisten the paper and make sure there is a good seal between the paper and the funnel. Turn on the vacuum line and then pour the contents of the flask onto the paper. Use the stir rod to remove as many crystals as you can. Rinse the flask several times using the 50 mL of distilled water that you cooled.
16. Turn off the vacuum!

> **Note**
>
> The oven must be set low for the next step. The aspirin could melt if the oven is set too high or in the oven too long.

17. Transfer the paper filter onto the watch glass you weighed. Use a metal spatula to help remove the paper. Be sure to label your sample so you know which one is yours. Place the watch glass and paper in the oven for 10 minutes. Do not use the bottom rack of the oven. While you are waiting, place the filtrate in the vacuum flask into the waste container in the hood.
18. Weigh the filter paper with the dried crystals.
19. You need to save your aspirin for Part 3.

Part 3: Melting Temperature of an Aspirin Sample

A melting point can be obtained by visually watching the substance melt. The picture below shows a Melting Point Apparatus commonly used in the laboratory. The substance to be measured is introduced into the apparatus by a narrow glass capillary tube as shown below left, just as you will use in this experiment. The tube is observed through a magnifying lens built into the apparatus during the measurement.

Figure 5.2.4. Melting Temperature Apparatus

1. Obtain a small amount of your synthesized aspirin from Part 2. The solid should be in a powdered form. If it is not, use a mortar and pestle to carefully grind the solid to a powder.
2. Place a small amount of the aspirin crystals into the open end of a glass capillary tube, but be very careful to not break the thin tube. This is best accomplished by gently pushing and tapping the capillary tube directly into the powdered aspirin crystals as shown to the right.
3. Move the powder toward the closed end of the capillary tube by gently tapping the closed end onto a hard flat surface. Repeat this process until the aspirin has settled into the closed end of the tube and occupies 1 to 2 mm of tube space.

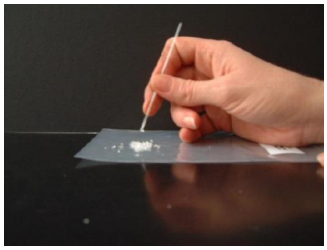


Figure 5.2.5. Loading a capillary tube with aspirin

Note that only a very small amount of material is used. If too much material is used, the temperature can vary from one end of the sample to the other, and the resultant melting point may be erroneous.

4. Check the control dial on the Melt Station to confirm that it is in the Off position. Connect the Melt Station power supply to a powered electrical outlet.
5. Connect the Melt Station to a computer. Choose New from the File menu of the data-collection program.
6. Carefully insert the capillary tube of solid into one of the sample holders of the Melt Station.
7. Begin collecting melting temperature data. In the first trial, you will want to observe the melting process and make a *rough estimate* of the melting temperature of your unknown sample.
8. When you have determined the approximate melting temperature range for the sample, stop data collection and turn the dial to the Fan/Cooling setting. Record the melting temperature range in your data table.
9. Now that you have a rough idea of the melting temperature, a more accurate determination of the melting temperature can be made. Prepare 2 samples in a capillary tube and determine the melting temperature of the samples one at a time, recording the melting range in your post-lab.

10. When finished, stop data collection and turn the dial to the Fan/Cooling setting. Record the melting temperature range in your data table.
11. At the end of the experiment turn the control dial on the Melt Station to Off. Dispose of the capillary tubes as directed by your instructor.
12. Complete the Data Analysis section before exiting Logger Pro. Print a copy of your graph for your lab group and/or save your data, as directed by your instructor.

Waste Disposal

Place the capillary tubes in the broken glass container. The aspirin and the contaminated filter paper should be placed in the solid waste container.

5.3: Aspirin Report Sheets

Name (first and last):

Lab Partner (first and last):

Date of experiment:

Part 1: Synthesis of Aspirin

MSDS and GHS

To identify the hazards associated with the chemicals in this lab, complete the following table. The hazard statements in the first line is completed as an example. The MSDS documents you need can be found posted with the labs online.

Materials	GHS Pictograms (Circle all that apply)	Hazard Statements
acetone		Highly flammable liquid and vapor. Causes serious eye irritation. Toxic to aquatic life.
phosphoric acid, 85%		
acetic anhydride		
salicylic acid		

Data and Calculations

Show dimensional analysis for all calculations.

	Mass (g)
Mass of salicylic acid used	
Mass of filter paper and watch glass	
Mass of aspirin + filter paper + watch glass	
Mass of aspirin	

1. Initial moles of salicylic acid ($C_7H_6O_3$):

2. Ideal mass of aspirin ($C_9H_8O_4$):

3. Use the following equation to calculate percent yield.

$$\% \text{ yield} = \frac{\text{actual moles of aspirin}}{\text{ideal moles of aspirin}} \tag{5.3.1}$$

Discussion of Experimental Error

1. Explain two errors that may cause the yield to be below 100%.

2. Explain two errors that may cause the yield to be above 100%.

Part 2: Melting Point of Aspirin

Data

Rough Melting Point Range: _____ °C to _____ °C

Melting Point Range: _____ °C to _____ °C

Melting Point Range (Repeated): _____ °C to _____ °C

Questions

1. Consider your melting point ranges. Is your aspirin sample pure? How do you know?

CHAPTER OVERVIEW

6: Synthesis of Fragrant Esters

6.1: Esters Prelab

Synthesis of Fragrant Esters Prelab (5 points)

1. The reaction to form an ester is classified as a _____ reaction. (1 point)
2. Esters in this lab will be formed by combining what 2 types of molecules? (1 point)
3. Fill in the following table before coming to lab. (6 points)

Alcohol	Acid	Ester Common Name	Ester IUPAC Name	What fragrance should you smell?
Isopentyl (isoamyl)	acetic			
Methanol	acetic			
Methanol	salicylic			
Ethanol	salicylic			

6.2: Esters Procedure

Learning Objectives

- Gain a deeper understanding of the esterification process
- Practice naming esters
- Practice appropriate labratory safety techniques.

Safety

- Handle concentrated acids like sulfuric acid with extreme care. Use appropriate tools like pipettes and avoid direct contact.
- Work in a well-ventilated area or under a fume hood to avoid inhaling fumes from volatile chemicals.
- Always wear safety goggles
- Be cautious when using the Bunsen burner and hot water bath. Always use heat-resistant gloves when handling hot equipment or containers.
- Never leave the Bunsen burner unattended while it is lit.

Introduction

An ester is an organic compound that is formed when a carboxylic acid reacts with an alcohol while in the presence of a strong mineral acid such as sulfuric acid acting as a catalyst. When carboxylic acids are esterified by being combined with an alcohol to form an ester, many of the resulting esters have a very pleasant smell. That is why esters are often found in perfumes and essential oils. Synthetic esters produced in the laboratory are nearly the same molecules that give fruits their characteristic fragrance.

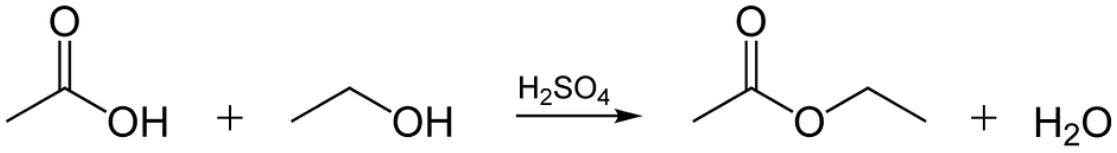


Figure 6.2.1. An Esterification reaction is a dehydration reaction

A carboxylic acid contains the -COOH functional group. In an ester, the hydrogen in this group is replaced by a hydrocarbon group of some kind. This could be an alkyl group like methyl or ethyl, or one containing a benzene ring like phenyl. A common ester is ethyl ethanoate. In this case, the hydrogen in the -COOH group has been replaced by an ethyl group. The formula for ethyl ethanoate is shown below:

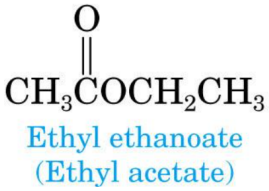


Figure 6.2.2. Ethyl ethanoate

Notice that the ester is named the opposite way from the order in which its formula is written. The “ethanoate” formula shown in blue comes from ethanoic acid. The “ethyl” comes from the ethyl group on the end.

Naming Esters: The common names for esters are derived from the organic acid and the alcohol from which they are derived. For example, when acetic acid reacts with ethyl alcohol, the resulting ester is commonly called ethyl acetate.

However, the International Union of Pure and Applied Chemistry (IUPAC) is responsible for formally naming chemical compounds, and per the IUPAC rules, acetic acid is called ethanoic acid. Thus, the ester formed is called ethyl ethanoate. IUPAC names for esters come from two words: first from the prefix of the alcohol and second from the name of the acid. Drop the ” –ic acid” from the IUPAC acid name and add “-ate.”

An ester from methanol or methyl alcohol and ethanoic acid (acetic acid) is named as follows:

1. From the alcohol comes the prefix “methyl”
2. From the ethanoic acid, drop “–ic acid” and add “–ate,” making it ethanoate
3. Thus, the full IUPAC name is: methyl ethanoate
4. The common name is: methyl acetate since acetic acid becomes acetate

Procedure

Part I

1. Use the Data Table on the report sheet for recording your observations for both Parts I and II.
2. Mark three small test tubes with the numbers 1 and 2. Record which number will relate to which ester as reflected in the Data Table.
3. Place the test tubes in a rack or well plate to securely hold them while you perform the experiment
4. Place 4 drops of acetic acid into test tubes #1 and #2.
5. Place 5 drops of isopentyl alcohol (also known as isoamyl alcohol) into test tube #1.
6. Place 5 drops of butanol into test tube #2.
7. Add 2 drops of sulfuric acid to each test tube.
8. Gently swirl, shake, and agitate each test tube to mix the acids and alcohols
9. Set up the burner stand and Bunsen burner as demonstrated by your instructor.
10. Half-fill the 100 mL glass beaker with very hot tap water and place it on the burner stand and heat until the water come to a rolling boil.
11. After the water has begun to vigorously boil, extinguish the Bunsen burner. The water will remain hot long enough for the esterification reaction to take place.
12. Use a hot-glove to transfer the beaker of hot water from the burner stand to your work area.
13. Place the three test tubes with the acid-alcohol mixtures into the beaker of hot water and let them stand in hot water for 5 minutes.
14. Use a clamp to remove the test tubes, one at a time, from the hot water bath. Suck water up into an empty pipet and then drop 20 drops of water from the pipet into each test tube’s contents. Agitate to mix. Then replace the test tubes into the test tube stand
15. Cautiously try to detect the odor of the products in each test tube by using your hand to waft the scent from the test tube toward your nose. Do this until you can detect and record an odor. Do not worry about having the “correct” odor for a particular ester. Descriptions of odors often vary with people, and some odors hard to describe.
16. Pour the contents of the test tubes into the labeled waste container. Wash the test tubes with the test tube brush, rinse with distilled water, and set to dry.

Part II

1. Mark 2 small test tubes with the numbers 3 and 4. Record which number will relate to which ester as reflected in the Data Table.
2. Place the test tubes in a rack or well plate to securely hold them while you perform the experiment
3. Using weigh-boat or weigh-paper on the digital scale, weigh 0.2 grams of salicylic acid and then transfer it into test tube #3. Repeat this for test tube #4.
4. Place 5 drops of methanol into test tube #3.
5. Place 5 drops of ethanol into test tube #4.
6. Add 2 drops of sulfuric acid to each test tube.
7. Repeat steps 8-17 of Part I for test tube #3 and #4.

6.3: Esters Report Sheets

Name (first and last):

Lab Partner (first and last):

Date of experiment:

Data Table: (6 points)

Tube#	Alcohol	Acid	Common Name	IUPAC Name	Describe the Fragrance
1	Isopentyl (isoamyl)	acetic			
2	Methanol	acetic			
3	Methanol	salicylic			
4	Ethanol	salicylic			

Questions for analysis: (14 points total)

1. For each reaction you completed in this experiment, draw a reaction with full structures of all reactants and products. Use the reaction show in the introduction as a guide. (1 point for each reaction)

    a. Test tube 1:

    b. Test tube 2:

    c. Test tube 3:

d. Test tube 4:

2. What role does sulfuric acid play in the reaction for this in experiment? (1 point)

3. Name two commercial products that contain oil of wintergreen. (2 point)

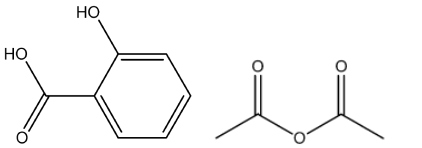
(You can look at the ingredient list on your household products.)

4. Explain why the esters you made in your experiment could not be used as flavoring agents. (1 point)

5. What would be the IUPAC name of the ester produced from isopentyl alcohol and isopentanoic acid? Draw the ester below.(2 points)

6. Raspberry oil can be prepared from the reaction of formic acid with 2-methyl-1-propanol. What is the IUPAC name for the ester formed from these 2 compounds and draw the product? (2 points)

7. When salicylic acid is reacted with acetic anhydride the products are aspirin and acetic acid. Name and draw the structures of the products. (2 points)



CHAPTER OVERVIEW

7: Determining % Sugar Content in Beverages

7.1: Percent Sugar Prelab

Determining % Sugar Content in Beverages Prelab (5 points)

Read the lab completely prior to attending lab.

1. How does the density of a 10.0% (w/v) sugar solution compare to a 15.0% (w/v) solution? (1 points)

2. How much sucrose (sugar) is needed to make 100.0mL of a 25.0% (w/v) solution. (2 points)

3. Describe in detail how you would make 75.0mL of a 30.0% (w/v) sugar solution starting with 100.0mL of a 50.0% (w/v) sugar solution. (2 points)

7.2: Percent Sugar Procedure

Learning Objectives

- Gain hands-on experience in constructing a homemade hydrometer and calibrating it using solutions of known sugar concentrations.
- Develop skills in accurately measuring the density of various beverages and using this data to determine their sugar content.
- Understand how density relates to the concentration of sugar in a solution.

Introduction

The word carbohydrate comes from “hydrate of carbon”, which is derived from the formula $C_n(H2O)_m$. Carbohydrates are the body’s most important and readily available source of energy. An adult human consumes between 1200 and 2400 calories just to keep alive. The bulk of this energy comes from burning carbohydrates and fats. Your brain runs on nothing but glucose. The two major forms of carbohydrates are:

- Simple Sugars (Monosaccharides), such as glucose, fructose, galactose, mannose and others found in nutritious whole fruits.
- Complex Sugars and Starches (Disaccharides), such as sucrose, lactose. (Polysaccharides), such as glycogen and cellulose.

The simple carbohydrates in many refined foods (white flour, white rice) as well as fruits are easily broken down and cause your blood sugar level to rise quickly. Foods that are high in added simple carbohydrates (soda and fruit drinks) also tend to be high in calories and low in other valuable nutrients. Today in the United States high-fructose corn syrup (HFCS), a 55% fructose and 45% glucose syrup from maize (corn), is used nearly exclusively as a sweetener because of its lower cost. As a result, a high-sugar diet is often linked with obesity.

The complex carbohydrates found in whole-grain products such; as brown rice, breads, and cereals, as well as, starchy vegetables are broken down more slowly, allowing blood sugar to rise more gradually. Therefore, eating a diet that's high in complex carbohydrates can minimize the risk for developing health problems like diabetes and heart disease.

Good sources of carbohydrates include:

- whole-grain cereals
- brown rice
- whole-grain breads
- fruits
- vegetables
- low-fat dairy

The key is to make sure that the majority of carbohydrates we eat are from sources such as those listed above and to limit the amount of added sugar in your diet.

From a chemical perspective, fruit juices and sodas are basically sugar-water solutions. Sugar added to the water makes the solution denser than pure water. In other words, a cup of pure water weighs less than a cup of sugar-water. Increasing the amount of sugar in the sugar-water solution results in an increase in the density of the solution. Therefore, by comparing the densities of sugar-water solutions you can determine the amount of sugar present in fruit drinks and sodas.

Normally a chemist would use a commercially available hydrometer to compare specific gravities of solutions. The hydrometer measures the relative density of its resting free-floating position in the solution. For example, when it is dropped into a low density solution, the device will sink and come to rest at a low position without touching the bottom.

In this lab you will prepare a home-made hydrometer to determine the densities of various fruit drinks and sodas. To accomplish this you will record the resting positions of your home-made hydrometer (a free-floating pipette) in a set of reference solutions that you will prepare. These will be use to create a calibration curve by plotting the resting positions versus sugar content. You will then match the resting position in the fruit drinks or sodas to the sugar content using the calibration curve.

Procedure

> **Note**
>
> Fruit juices and soda need to be left out overnight so that they are flat before you begin this lab. Be sure all fruit juices and soda are at room temperature before you begin. Record the temperatures in the tables provided.

Part I. Preparing a Free Floating Pipette

1. Obtain a plastic pipette, a 25 mL graduated cylinder, a 100 mL beaker and a few paper towels.
2. Pour 40 mL of distilled water into your 100 mL beaker. Record the temperatures in the tables provided.
3. Fill the graduated cylinder to just under the 25 mL mark with distilled water. Tap out the air bubbles. Draw up some water in an empty pipette. Use this to add the few necessary drops to bring the water level to the 25 mL mark. Be sure to look at this at eye level.
4. Fill the pipette completely with distilled water. This can be accomplished by the following steps. Squeeze out as much air as you can from the pipette and place the tip of the pipette into the distilled water in the beaker. Let go and water will flow into the pipette. Turn the pipette upward and squeeze out any remaining air until water begins to leave the tip. (Paper towels come in handy here.) Quickly insert the tip back into the distilled water and allow it to completely fill with distilled water. At this point the pipette should be completely full.
5. Gently drop the pipette into the graduated cylinder. It should sink to the bottom. If it does, carefully retrieve the pipette and squeeze out 2 or 3 drops.
6. Gently drop the pipette into the graduated cylinder again. It should now be floating just off the bottom. Be sure the bottom of the pipette is floating somewhere between the 1 and 5 mL mark. To ensure the pipette is not getting hung up on the sides of the graduated cylinder tap the pipette toward the center of the cylinder to see if the pipette will sink more. Repeat this until the pipette stabilizes.
7. Once the pipette has stabilized, record the level of the bottom end of the bulb at eye level. Record the resting position of the pipette in Table 1 under 0% sugar solution.
8. Remove the pipette and repeat steps 6 and 7 two more times. Find the average of these three readings and record in Table 1. This is known as the relative density of water in this experiment.
9. Remove the pipette and discard the water in the cylinder. Carefully set the pipette aside. A 24-well tray works great as a pipette stand.

Part II. Preparing Reference Sugar Solutions and Determining their Resting Positions.

If your instructors were trapped on a deserted island with only a 50 mL graduated cylinder and a 200 mL beaker, we would prepare 100.0 mL of a 20.0% (w/v) solution as follows. (To recall information from CH 105, a 20.0% (w/v) solution means 20.0g of solute per 100.0 mL of solution.)

- Weigh 20.0 grams of table sugar and place in a clean dry beaker.
- Add about 30-40 mL of distilled water and stir until dissolved.
- Transfer this solution to a 50 mL graduated cylinder and carefully bring to the 50.0 mL mark with distilled water.
- Pour the contents of the graduated cylinder into a clean dry 200 mL beaker.
- Add another 50.0 mL of water to the graduated cylinder.
- Add this 50.0 mL to the beaker and swirl. Now you have 100.0 mL of a 20.0% (w/v) solution.

Your challenge is to make 50 mL of a 20.0% (w/v) reference sugar solution.

1. Describe how you made the 20.0% (w/v) solution. This should be about one paragraph reported in your post-lab.
2. Make 25 mL of a 15.0% (w/v). (Hint: $C_1V_1 = C_2V_2$ using your 20.0% solution)
3. Fill your well rinsed 25 mL graduated cylinder with 25.0 mL of the 15.0% (w/v) sugar solution.
4. Gently drop the pipette filled with distilled water you created in Part 1 into the graduated cylinder. To ensure the pipette is not getting hung up on the rim of the graduated cylinder tap the pipette toward the center of the cylinder to see if the pipette will sink more. Repeat this until the pipette stabilizes.
5. Once the pipette has stabilized record the level of the bottom end of the bulb at eye level. Record the resting position of the pipette in Table 1 under 15.0% sugar solution.
6. Remove the pipette and repeat steps 5 and 6 two more times. Find the average of these three readings and record in Table 1.
7. Be sure to rinse your pipette well with water and wipe dry before going on.

8. Make 25 mL of a 10.0% (w/v). (Hint: $C_1V_1 = C_2V_2$ using your 20.0% solution)
9. Repeat steps 5-9 using the 10.0% (w/v) solution.
10. Make 25 mL of a 5.0% (w/v). (Hint: $C_1V_1 = C_2V_2$ using your 20.0% solution)
11. Repeat steps 5-9 using the 5.0% (w/v) solution.

Part III. Establishing a Calibration Curve of Sugar Solutions

Plot your data using excel or sheets with average resting position on the y-axis and Percentage of sugar on the x-axis. Add a linear trendline and the equation of the line to the graph. This is your calibration curve.

Part IV. Determining Resting Position of pipette in Fruit Drinks or Soda

1. Be sure all fruit juices and soda are at room temperature before you begin. Record the temperatures in the tables provided.
2. Record the 3 types of drinks you will be testing in Table 2.
3. Using the pipette filled with distilled water you created in Part 1 determine the resting position for each drink. Do this 3 times and find the average of these three readings and record in Table 2.

Part V. Using your Calibration Curve

1. Print out the graph you made in Excel. Copy this graph into your lab report form at the appropriate place.
2. Use the equation of the trendline to determine the % sugar in the 3 types of drink you chose. Show your calculations in the post-lab.
3. Record the percentage sugar reading in Table 3. This in the sugar content of your drink.

7.3: Percent Sugar Report Sheets

Name (first and last):

Lab Partner (first and last):

Date of experiment:

Lab Notes and Observations:

Respond to the prompts below. (0.5 points each)

1. Describe how you prepared each of standard sugar solutions solution including any calculations.
    a. 20.0% (w/v)

    b. 15.0% (w/v)

    c. 10.0% (w/v)

    d. 5.0% (w/v)

Data Table 1: (2.5 points)

All volume data should be +/-0.1 mL, be sure to read your graduated cylinder correctly.

Solution	Trial 1 Resting Position	Trial 2 Resting Position	Trial 3 Resting Position	Average Resting Position
0.0% (w/v) sugar solution				
5.0% (w/v) sugar solution				
10.0% (w/v) sugar solution				

15.0% (w/v) sugar solution				
20.0% (w/v) sugar solution				

Data Table 2: (1.5 points)

All measurements should be +/-0.1 mL.

Solution List your drinks in the boxes below	Temp °C	Trial 1 Resting Position	Trial 2 Resting Position	Trial 3 Resting Position	Average Resting Position
Drink 1					
Drink 2					
Drink 3					

Graph: (3 points)

Use excel to plot the **% sugar solutions on the y axis** and the resting position of each in **milliliters on the x axis.**

Include the following components.

- Include axis labels
- Include the equation of the line (the **trendline)** and include and r2 value.
- Look at the **correlation coefficient, the r2** value of your line, it should be at least 0.9. If it is less than that you may want to redo the solutions that are not in line.
- You will use this graph and/or the line equation to determine the % sugar in your "unknown" solutions, the soft drink products.

Include your graph when you submit this lab.

Results: (1.5 points)

Use the equation of your trendline to determine the average % sugar in your soft drinks. Show your calculations here

Drink 1:

Drink 2:

Drink 3:

Table 3: (1.5 points)

Copy the result you obtained for each unknown sugar here.

List which drink is drink 1, drink 2, etc.

Solution List the drink type in the boxes below	Average Resting Position (from table 2)	% Sugar Content
Drink 1		
Drink 1		
Drink 1		

Questions for analysis:

Always answer using your own words. You should cite any resources used, but resist copying exactly. Use complete sentences.

1. How does this resting position of the pipette and the % sugar in the solutions relate to the density of your sugar solutions? (Hint: Your graph will be very helpful for this question.) (1.5 points)
2. Common table sugar is sucrose. What two monosaccharides make up sucrose? Describe what type of glycosidic linkage between the two sugars. (2 points)
3. List 3 source of healthy carbohydrates and explain why they are healthy. (1.5 points)

Reflection: (3 points)

Look at the ingredient list of several common types of processed food items that may be in your home or wander through a grocery store (or even look up on the internet).

Do some research on HFCS (high fructose corn syrup). Use two resources to address the following issues in an essay below.

- What is high-fructose corn syrup (HFCS)?
- Where is it often used?
- What recommendations you would make to someone if you were their health professional about consuming foods containing HFCS?

Use at least ***2 different*** sources. Always cite your sources.

If you use Wikipedia, ***go to at least two of the listed resources at the bottom of the article*** to get further support for your claims about HFCS.

You can attach your reflection to your report sheets.

CHAPTER OVERVIEW

8: Saponification- Making Soap

Thumbnail: Soap Film. (CC BY-SA 2.0 Generic; Umberto Salvagnin via Wikipedia)

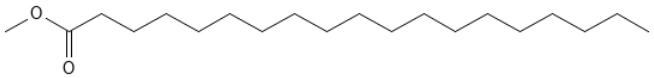
8.1: Saponification Prelab

Saponification: Making Soap Prelab (5 points)

(1 point each unless noted)

Read the lab completely prior to attending lab.

1. Determine the products of reacting the following molecule with NaOH. (2 points)



2. What type of molecule is the largest product (by mass) from question 1?

3. Circle the hydrophobic portion of the largest product (by mass) from question 1.
4. Classify the reaction from question 1.

8.2: Saponification Procedure

Learning Objectives

- Understanding the chemical process of saponification
- Identifying the key components of soap
- Evaluating the properties of different soaps based on their ingredients
- Gaining practical laboratory skills

Safety

- Oil and ethanol will be hot, and may splatter or possible catch fire.
- Keep a watch glass nearby to smother any flames.
- NaOH is caustic and can cause burns and eye damage.
- Wear goggles at all times.
- Wear disposable gloves.

Introduction

Saponification of an ester occurs when a hydroxide attacks the carboxyl carbon breaking the ester linkage to produce a carboxylate and an alcohol:

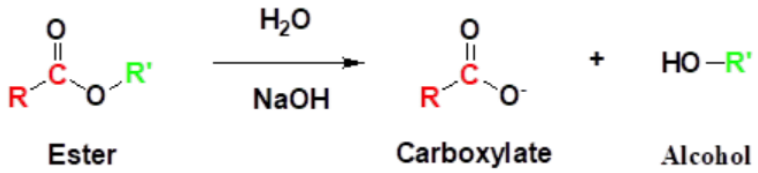


Figure 8.2.1. Saponification. Reaction of an ester with NaOH.

Using long chain acids in which the R group can have 14 to 24 carbon atoms attached to a glycerol molecule, is an example of a triglyceride called a fat as in the example below, to react with a strong base or "lye" to produce a soap through this saponification process.

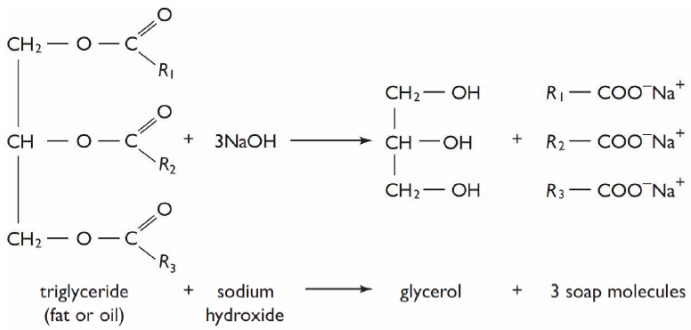


Figure 8.2.2. Saponification of a triglyceride.

Soap is the salt of a fatty acid. Sodium salts are known as "hard" salts found in most cake soaps. Potassium salts are the "soft" soaps used in shaving creams and liquid soaps.

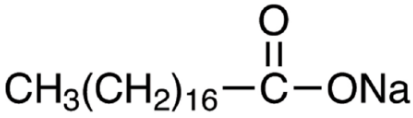


Figure 8.2.3. Sodium stearate is a typical soap.

Historically the preparation of soap began by using the extract of wood ashes as the base, and animal fats. The art of soap making was lost during the Middle Ages after the fall of the Roman Empire.

As you study the long chain soap molecule you can see that there is a nonpolar “hydrophobic” end and, the other end is the carboxylate ion which is ionic and “hydrophilic” or water loving. The nonpolar ends dissolve the grease or oils that accompany dirt, the water loving ends extend outside where they dissolve in water. The clusters that form from this action are called “micelles” .The small globules of oil and fat that are coated with soap molecules are pulled into the water layer and rinsed away.

One problem of using soaps is that the carboxylate end reacts with ions in the water such as Ca^{2+} or Mg^{2+}, to form insoluble substances. (Recall those solubility rules?)

$$\underbrace{2\,C_{17}H_{35}COO^{-}Na^{+}}_{\text{soap}} + M^{2+} \rightarrow \underbrace{[C_{17}H_{35}COO^{-}]_2M^{2+}}_{\text{scum}} + 2\,Na^{+} \tag{8.2.1}$$

$M = Ca^{2+} \text{or } Mg^{2+}$

Another important test for soaps is the acidity. If a soap is too alkaline it can cause damage to skin and clothes. It the solution is too acidic the sodium salt is reverted back to the fatty acid, thus losing the cleansing action.

$$C_{17}H_{35}COO^{-}Na^{+} + H^{+} \rightleftharpoons C_{17}H_{35}COOH + Na^{+} \tag{8.2.2}$$

Procedure:

Part A: Making the Soap

Read All the safety warnings above.

1. Prepare a hot water bath using a 250 mL beaker.
2. Weigh *approximately* 5-10 g of fat or vegetable oil into a clean 150 mL beaker.
3. Using a graduated cylinder, add 15.0 mL of ethanol, CH_3CH_2OH, and stir using a glass rod.
4. Warm the beaker in the hot water bath until the lard dissolves, remove from heat.
5. **Carefully** add 15.0 mL of 30**%** NaOH, stirring constantly. ***Use care when pouring NaOH!***
6. Place the beaker in the water bath on a hot plate and heat to a gentle boil and stir continuously. Heat the mixture for about 30 minutes, or until saponification is complete and the solution is almost a solid. **Be careful of splattering, the mixture contains a strong base. Wear disposable gloves. Do not let the mixture overheat or char.**
7. Allow the mixture to cool for about 10 minutes, until it is slightly warm to touch.
8. Obtain 50 mL of a saturated NaCl solution in a 400-mL beaker. Pour the soap solution into this salt solution and stir. This process, known as "salting out," increases the density of the solution, which causes the solid soap curds to float on the surface.
9. Place the beaker in an ice bath until the solution equilibrates to the ice bath temperature.
10. Collecting the soap. Collect the solid soap using a Buchner funnel and filter paper. Wash the soap with two 10-mL portions of cold water. Pull air through the product to dry it further. Place the soap curds on a watch glass or in a small beaker and dry the soap until the next lab session. Use disposable, plastic gloves to handle the soap. **Handle with care: The soap may still contain NaOH, which can irritate the skin. Save the soap you prepare for the next part of this experiment**

Part B: Testing Soap and Detergent:

> **Materials:** Test tubes, stoppers to fit, droppers, small beakers, 50- or 100-mL graduated cylinder, stirring rod, laboratory-prepared soap (from part A), commercial soap product, detergent, pH paper, oil, 1% $CaCl_2$, 1% $MgCl_2$, and 1% $FeCl_3$

Prepare solutions of the soap you made in part A, a commercial soap, and a detergent by dissolving about 1 gram of each in 50 mL of warm distilled water. If the soap is a liquid, use 20 drops instead of 1 gram.

Record your observations in the Data Table below for steps 1-4.

1. **pH test**: Place 10 mL of each soap solution into a separate test tube. Use 10 mL of water as a comparison. Label. Stir each solution with a stirring rod. Touch the stirring rod to pH paper. Determine the pH. Save tubes for part step .2
2. **Foam test**: Stopper each of the tubes from step 1 and shake for 10 seconds. The soap should form a layer of suds or foam. Record your observations. Save tubes for step 3.
3. **Reaction with oil**: Add 5 drops of oil to each test tube from step 2. Stopper and shake each one for 10 seconds. What happens to the oil layer? Record your observations. Compare the sudsy layer in each test tube to the sudsy layers in part step 2. Which substance dispersed (emulsified) the oil?

4. **Hard water test**: Place 5 mL of each soap solution in three separate test tubes. Add 20 drops of 1% $CaCl_2$ to the first sample, 20 drops of 1% $MgCl_2$ to the second tube, and 20 drops of 1% $FeCl_3$ to the third tube. Stopper each test tube and shake 10 seconds. Compare the foamy layer in each of the test tubes.

8.3: Saponification Report Sheets

Name (first and last):

Lab Partner (first and last):

Date of experiment:

Table 1 (6 points)

TEST	Water	Lab Soap	Commercial Soap	Detergent
pH				
Foam				
Rxn w/ Oil				
Hard Water A. $CaCl_2$				
Hard Water B. $MgCl_2$				
Hard Water C. $FeCl_3$				

Questions: (1 point each)

1. Describe the soap that you made.

2. Which of the soaps were basic? Why?

3. What is the effect of soap and detergents on oil? How do they work?

4. How could you determine if a water sample is soft or hard water?

5. What happens to the glycerol produced during the saponification?

6. Why is the product of saponification called a salt?

7. Sodium palmitate is: $CH_3(CH_2)_{14}COONa$

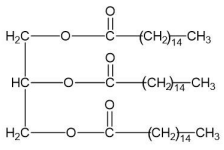
Identify the hydrophobic end and the hydrophilic end of this molecule.

8. Which has the greater solubility in water? palmitic acid or sodium palmitate

9. The following molecule is an example of a **nonionic detergent**. Circle the end that is the grease soluble component.

$$CH_3(CH_2)_{10}CH_2O(CH_2CH_2O)_7CH_2CH_2OH$$

10. Write the product/s for the following reaction



+ 3 NaOH →

11. If “lauryl” is the common name for dodecyl (12 carbons), what is the formula for the detergent, sodium lauryl sulfate?

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D

Glossary

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