ELEMENTARY ORGANIC CHEMISTRY (LAB MANUAL)

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Triton College

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ELEMENTAL ORGANIC CHEMISTRY

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Licensing

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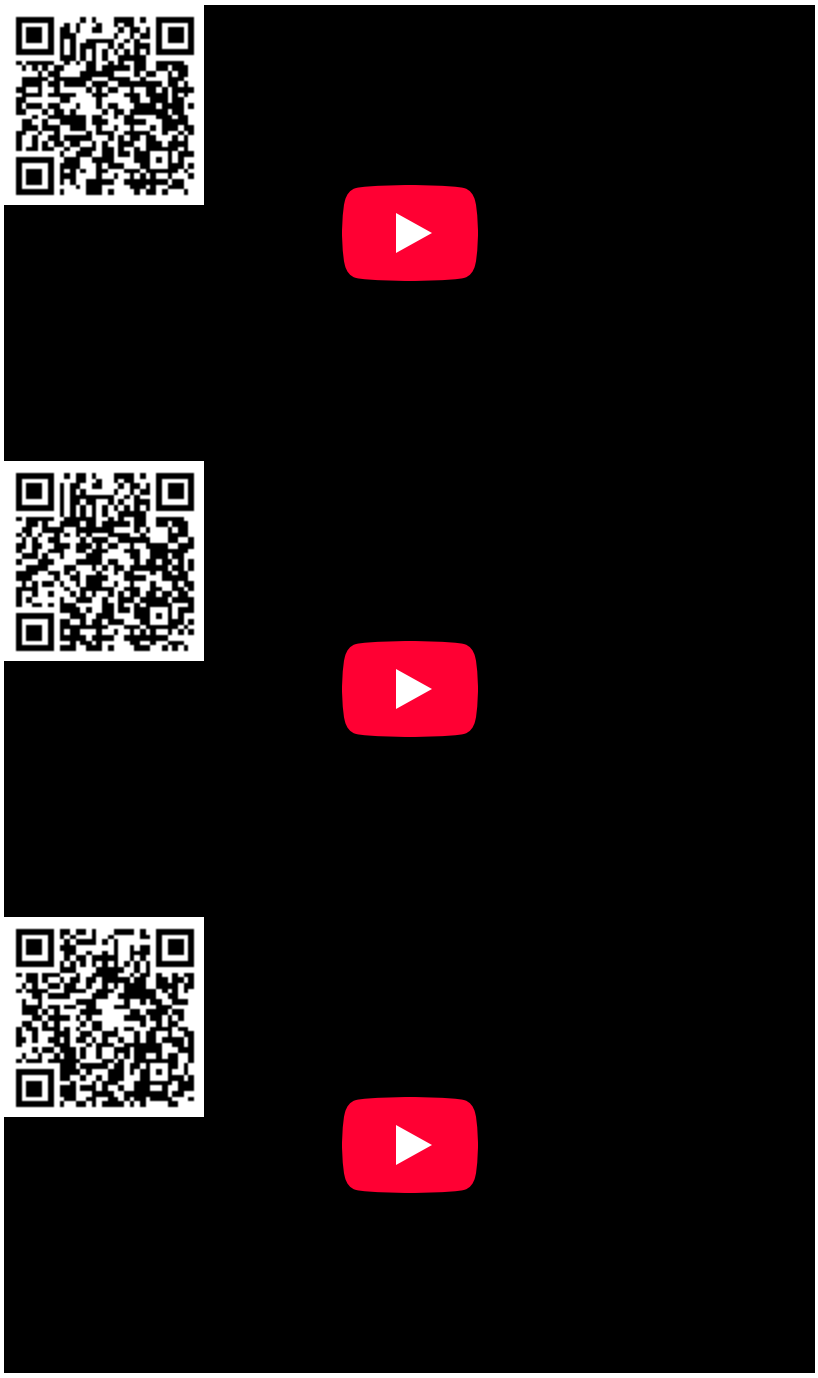
CHAPTER OVERVIEW

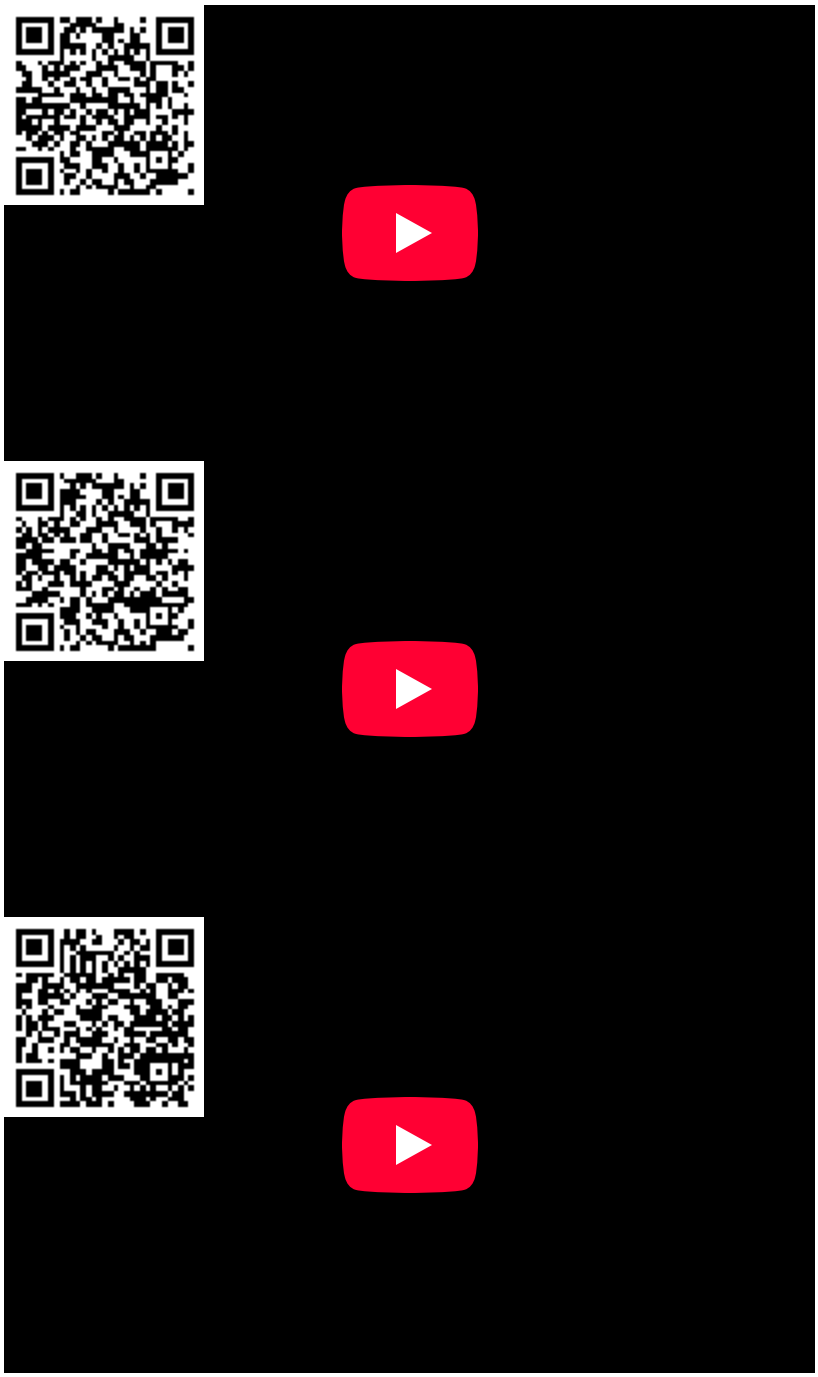
LAB 0 - LAB SAFETY ORIENTATION

0.1: LAB SAFETY VIDEOS

Students will find below the eight Lab safety orientation videos. The first six are part of the College Lab Safety Series by the American Chemical Society. Students should watch the first seven videos before signing the safety contract and attempting the safety quiz. The eighth video is an older version of the College Lab Safety video by the American Chemical Society, which should be watched, time permitting, as it provides detailed dos and don'ts in a college lab setting.

1) **ACS (American Chemical Society) College Lab Safety Series (ACS College Safety Video #1 - #6)**





2) Lab Safety and Techniques- Crash Course Safety

3) Safety Video by American Chemical Society (1991)

0.2: LAB SAFETY CONTRACT

Triton College Chemistry Division

Student Safety Contract

Purpose

At Triton College, safety is our number one priority. A list of rules has been developed and provided to you in this student safety contract to ensure a safe science laboratory experience. These rules must always be followed. Please be advised that you will be asked to leave the laboratory if you violate these rules.

No exceptions!!

This contract is available in both PDF and .doc formats. Download, sign, and date both contract pages before you participate in the laboratory sessions. Your signature indicates that you have read this contract and agree to abide by the rules. You will keep a copy for your records and upload or submit one to your instructor via your Blackboard or Learning Management System (LMS) as directed.

I, _____, dated _____, agree that I will comply with the following safety rules. I will:

1) Always wear safety goggles in the laboratory.

2) Always wear appropriate clothing in the laboratory. This includes long pants, shirts with sleeves, and closed shoes (i.e., no sandals). Also, I understand that long hair must be tied back, and loose or baggy clothing or jewelry must be secured.

*Note: Students violating rules one and/or two will be asked to leave the lab immediately. No second chances!! NO EXCEPTIONS!!

3) Read laboratory instructions beforehand and ask the instructor about any part of the lab I am unsure about.

4) Use proper equipment for laboratory procedures.

5) Never perform unauthorized experiments or work alone in the laboratory.

6) Never remove chemicals or other materials/equipment from the laboratory area.

7) Read the labels on reagent bottles carefully, remove only small amounts of reagent with the proper tools, and never return unused chemicals to the bottle.

8) Dispose of broken glass and waste chemicals in the appropriate waste container.

9) Immediately inform the instructor of a chemical spill or accident (even minor ones) in the laboratory.

10) Never taste or smell any chemicals.

11) Know the location of eyewash fountains, fire extinguishers, fire blankets, safety showers, first aid kits, and exits.

12) Do not bring food or drink into the lab; refrain from eating or drinking in the laboratory. This includes chewing gum and eating candy.

13) Keep aisles clear. Books, purses, backpacks, and other personal belongings will be kept outside or under the lab benches.

14) I will responsibly conduct myself at all times in the laboratory. Horseplay, practical jokes, and pranks are dangerous and prohibited.

15) Clean my work area before leaving the lab.

16) Wash my hands before I leave the lab.

Agreement

I, _____, have read and agree to follow all the safety rules outlined in this contract. I realize I must obey these rules to ensure my safety and that of my fellow students. I will fully cooperate with my instructor and fellow students to maintain a safe lab environment. I will also closely follow all oral and written instructions provided by the instructor, the science

department, and the college. I am aware that any violation of this safety contract, resulting in unsafe conditions in the laboratory or misbehavior on my part, may lead to my removal from the laboratory, a failing grade, and/or dismissal from the course.

_____ _____

Student Signature Date

_____

Course section and number

Questions

1) Are you color blind? Yes/No

2) Do you have any allergies? Yes/No

If yes, please list specific allergies.

3) Do you wear contact lenses? Yes/No

4) If you are pregnant, breastfeeding, or trying to conceive, avoid working with heavy metal compounds and organic solvents. Notify your instructor and request a list of chemicals used in this course. Use this list to consult with your physician before proceeding with the course. Under your physician's supervision, you may need to purchase a respirator, which a doctor must fit and adjust to your needs.

5) Please inform your instructor of any other medical conditions or situations you feel we should be aware of to ensure your safety and the safety of others while working in the laboratory. Please be aware that anything written in this contract will remain confidential.

0.3: LAB SAFETY QUIZ

Multiple-Choice Questions

1. What should you do if a chemical splashes into your eyes?
   - A) Rub your eyes vigorously
   - B) Rinse your eyes with water for at least 15 minutes
   - C) Close your eyes and wait for the pain to subside
   - D) Apply a cold compress
2. Which of the following is NOT appropriate lab attire?
   - A) Closed-toe shoes
   - B) Long pants
   - C) Loose, flowing clothing
   - D) Safety goggles
3. What is the first thing you should do if you spill a chemical on your skin?
   - A) Wipe it off with a cloth
   - B) Rinse the affected area with water immediately
   - C) Apply a bandage
   - D) Ignore it if it doesn't hurt
4. Where should you dispose of broken glassware?
   - A) Regular trash bin
   - B) Recycling bin
   - C) Designated glass disposal container
   - D) Sink
5. Which of the following statements about lab safety is TRUE?
   - A) Eating and drinking are allowed in the lab
   - B) You should always work alone in the lab
   - C) Always read labels and safety data sheets before using chemicals
   - D) It's okay to leave a Bunsen burner unattended

True/False Questions

6. Always wear safety goggles when handling chemicals.
   - True
   - False
7. It's safe to taste chemicals in the lab to identify them.
   - True
   - False
8. You should be familiar with the location of safety equipment, including fire extinguishers, eyewash stations, and safety showers.
   - True
   - False
9. When working in the lab, it is recommended to tie long hair back.
   - True
   - False
10. Using your phone in the lab is acceptable as long as you're careful.
    - True
    - False

Please click here for the Word file or PDF file of the quiz. Download one of these files complete and upload into your LMS as instructed by your

0.4: ADDITIONAL RESOURCES

LAB 1- ASSESSMENT OF PHYSICAL PROPERTIES OF ORGANIC COMPOUNDS

Determination of Melting and Boiling Points of Organic Compounds

PURPOSE

The purpose of this experiment is to:

- Determine the melting point of known and unknown organic solids.
- Set up and use a simple distillation apparatus to determine the boiling point of a known and an unknown organic liquid.
- Determine the identities of unknown substances based on their melting and boiling points.
- Identify the functional groups present in organic compounds.

INTRODUCTION

In general chemistry, you learned that each compound has a unique melting and boiling point. These physical properties, along with other methods, can aid in identifying organic compounds. The melting point is the temperature at which a converted into a liquid. The melting point of an organic solid can be measured using a melting point apparatus. In this lab, you will use the Vernier melt station to experimentally find the melting point of benzoic acid and an unknown organic compound.

The boiling point is the temperature at which a liquid is converted into a gas. The boiling points of organic liquids can be determined using simple distillation, as shown below.

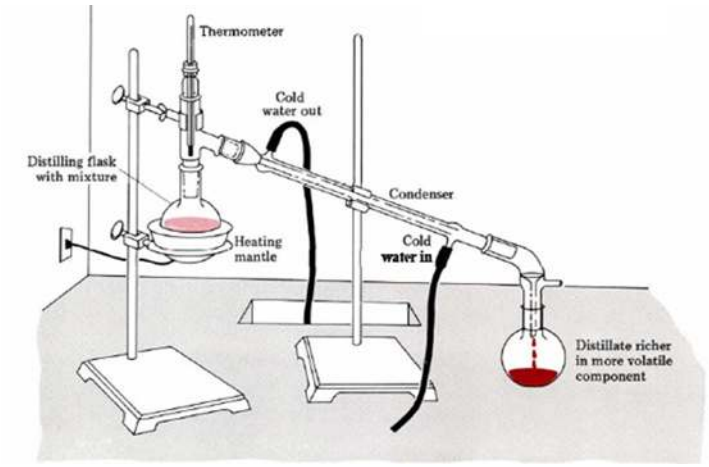


Figure 1: Distillation Process. (Copyright; 10.22: Distillation - Chemistry Libre Texts)

During this process, a liquid is heated in a round-bottom flask, and the temperature of its vapor is measured using a thermometer. Once the vapor hits the thermometer bulb, the temperature rises sharply and stabilizes at the liquid's boiling point. The vapor is then immediately condensed and collected into a separate container. Simple distillation can also be used to purify organic liquids. In this experiment, you will use simple distillation to determine the boiling point of acetone and an unknown organic liquid.

SAFETY PRECAUTIONS

1) Always wear chemical splash goggles while working on this experiment.

2) Work with distillation and all liquids in this experiment under a fume hood.

3) You are encouraged to wear gloves while handling chemicals.

4) Dispose of all waste in the proper containers, as your instructor indicates.

5) Thoroughly clean your work area when finished, and return all equipment and chemicals to the appropriate place.

CHEMICALS AND EQUIPMENT NEEDED

Table 1: Chemicals and Equipment

CHEMICALS	EQUIPMENT	EQUIPMENT
Benzoic acid	Watch glass	Simple distillation apparatus and equipment
Acetone	round-bottom flask	10 mL graduated cylinder
Unknown organic solid	Spatula	Vernier Melt Station (or similar melting point apparatus)
Unknown organic liquid	Weigh boat or Weighing paper	Capillary tubes - for melting point determination

EXPERIMENTAL PROCEDURE

Part A: Melting Points

1) Use a spatula to place a small amount of benzoic acid in a weigh boat or a watch glass.

2) Pack the benzoic acid into a capillary tube approximately 3 mm in diameter, ensuring it settles to the bottom of the tube.

3) Insert the capillary tube into the melting point apparatus and determine the melting range of benzoic acid. (Pure benzoic acid melts at approximately 121 - 123 °C.)

Note: Your instructor will demonstrate how to pack the capillary tube and operate the melting point apparatus.

4) Dispose of the capillary tube, as indicated by your instructor. Place the excess benzoic acid in the non-halogenated organic waste container.

5) Repeat steps 1 - 4 for the unknown solid. Based on the melting range, identify the unknown as one of the solids in the table for pre-lab question 2.

Part B: Boiling Points

1) Place 10.0 mL of acetone in a 50.0 mL round-bottom flask.

2) Set up a simple distillation apparatus under the fume hood, and determine the boiling range of acetone. (Acetone boils around 55 - 57 °C.)

Note: Your instructor will demonstrate how to set up a simple distillation apparatus based on available equipment.

3) Once you obtain the boiling range of acetone, turn off the heat but allow the water to continue running. (Note: Never distill to dryness.).

4) Once the glassware is cool, turn off the water, disconnect the round-bottom flasks from the distilling apparatus, and dispose of the acetone in the non-halogenated waste container.

5) Repeat steps 1 - 4 for the unknown liquid. Based on its boiling range, identify it as one of the liquids in the table for pre-lab question 3.

PRE-LAB QUESTIONS Name _____

1) Explain how melting and boiling points can infer the identity of unknowns.

2) Consider the following solids and their melting points:

Table 2: Melting Points of Known Organic Solids

	Melting Points
Acetaminophen	168 - 170 °C
Acetylsalicylic acid	135 - 136 °C
Anthracene	214 - 216 °C
Vanillin	81 - 82 °C
Resorcinol	109 - 110 °C
Ascorbic acid	190 -192 °C
Acetamide	79 - 81 °C
Urea	132 - 133 °C

Draw the structures for each compound and identify their non-alkane functional groups.

3) Consider the liquids listed below and their boiling points:

Table 3: Boiling Points of Known Organic Substances

Organic Liquids	Boiling Points
Toluene	108 - 110 °C
Cyclohexane	79 - 81 °C
Hexane	67 - 69 °C
Methylene chloride	38 - 40 °C
Benzaldehyde	177 - 179 °C
Ethyl acetate	76 - 78 °C
Ethanol	77 - 79 °C
Acetonitrile	81 - 82 °C

Draw the structure of each compound and identify its non-alkane functional groups.

Click here to download a Word or PDF document of this pre-lab.

DATA AND OBSERVATIONS

Name _____Lab Partner(s) _____

Table 4: Data Table - Melting and Boiling Points of Known and Unknown Organic Compounds

Measurement	Temperature
Melting range of benzoic acid	
The melting range of the unknown solid	
Possible identity of solid	
The boiling range of acetone	
Boiling range of an unknown liquid	
Possible identity of the unknown liquid	

POST-LAB QUESTIONS

1) What was the biggest takeaway from this activity?

2) What are some sources of error in this experiment?

3) How does your experimental melting point of benzoic acid compare to the actual melting point? What might this tell you about the purity of your benzoic acid sample?

4) Can you use melting and boiling points alone to identify unknowns positively? Why or why not?

5) In addition to melting and boiling points, what other properties can be used to support the identity of an unknown?

Please click here to access the Pre-Lab, Data Tables, and Post-Lab in Word or PDF format. Complete them and upload according to your instructor's instructions.

LAB 2 - PROPERTIES AND REACTIONS OF HYDROCARBONS

PURPOSE

The purpose of this experiment is to

- Draw structures of hydrocarbons.
- Name alkanes, alkenes, and alkynes.
- Explore the physical properties and reactions of hydrocarbons.
- Identify an unknown as saturated or unsaturated based on the results of chemical tests.

INTRODUCTION

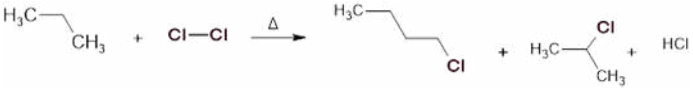
Hydrocarbons, composed of carbon and hydrogen, are the simplest organic compounds. Alkanes contain only single bonds and are classified as saturated hydrocarbons because they have the maximum amount of hydrogen possible. Open-chain alkanes can be represented by the formula C_nH_{2n+2}, where n indicates the number of carbon atoms. Other hydrocarbons have multiple bonds or rings and consequently possess fewer hydrogen atoms than predicted by this formula. For instance, cycloalkanes have two fewer hydrogens than alkanes and follow the general formula C_nH_{2n}. Alkenes and alkynes, which include carbon-carbon double and triple bonds, respectively, also do not match the hydrogen count of alkanes. These are known as unsaturated hydrocarbons. Aromatic hydrocarbons, which contain a benzene ring, are also unsaturated due to the presence of multiple double bonds.

In this experiment, you will first explore the nomenclature of hydrocarbons, structure, and physical properties. The pre-lab questions will provide a review of the material covered. Then, you will run combustion, halogenation, and oxidation reactions with select hydrocarbons.

All hydrocarbons undergo combustion in the presence of heat and oxygen. Assuming that the hydrocarbon is the limiting reactant, the products of this reaction will be carbon dioxide and water vapor, as demonstrated by the complete combustion of propane below:

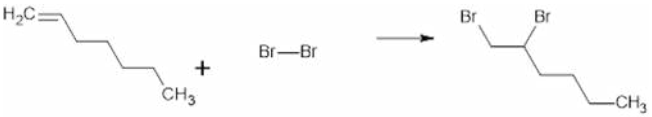
$$CH_3CH_2CH_3 + 5O_2 \xrightarrow{\Delta} 3CO_2 + 4H_2O$$

Hydrocarbons can also be halogenated under certain conditions. Alkanes can react with chlorine and bromine in the presence of heat or light to produce a mixture of alkyl halides. When predicting the products of these reactions, we assume mono-halogenation, although in reality, multiple halogenations can occur. For example, the chlorination of propane can give two mono-halogenated products:



The above chlorination reaction is a substitution reaction in which a chlorine atom replaces a hydrogen atom in a molecule.

Alkenes and alkynes are more reactive than alkanes and can react with halogens at room temperature. Consider the reaction of 1-hexene (hex-1-ene) with bromine:



The above reaction is an addition reaction, as both bromines add across the double bond. The reaction between an alkene or alkyne and bromine is also a chemical test for unsaturation. As you will see in this experiment, bromine is a dark red liquid. The solution will turn light yellow or colorless when added to an alkene or alkyne since no additional bromine is present, indicating a positive test result. If no reaction occurs, the dark red color of bromine will remain, resulting in a negative test result. Alkanes and aromatic hydrocarbons would yield a negative bromine test result.

The potassium permanganate test is another chemical test used to detect unsaturation. When potassium permanganate reacts with an alkene or alkyne, the purple permanganate solution disappears, and a brown solid is formed, as shown by the reaction with 1-hexene (hex-1-ene):

In the above reaction, the alkene is oxidized to form a diol. Manganese is reduced from the +7 oxidation state to the +4 oxidation state, indicating a positive test result for unsaturation. If potassium permanganate is added to an alkane or aromatic hydrocarbon, a negative test result would occur, in which the purple color of the permanganate solution remains unchanged.

SAFETY PRECAUTIONS

1) Always wear goggles while working with chemicals in this lab.

2) Wear gloves while working on this experiment.

3) Handle all chemicals under a working fume hood.

4) Dispose of all waste in the appropriate waste container, which should also be kept under a fume hood.

5) Thoroughly clean all glassware and your work area at the end of the experiment.

6) Before leaving the lab, wash your hands.

CHEMICALS AND EQUIPMENT NEEDED

Table 1: **Chemicals and Equipment**

CHEMICALS	CHEMICALS	EQUIPMENT
Pentane	Distilled or Deionized Water	6 small or medium-sized test tubes
Hexane	1% bromine in methylene chloride (or a solution of similar concentration)	Test Tube Rack
Heptane	1% aqueous potassium permanganate solution (or similar concentration)	10 mL graduated cylinder
Cyclohexane	Toluene	Watch glass
Cyclohexene	Halogenated and Non-halogenated organic waste containers	Evaporating dish

EXPERIMENTAL PROCEDURE

Part A: Structures of Hydrocarbons

i) Draw the structures (Lewis, condensed, or line-bond) for the following hydrocarbons used in this experiment, as listed in the data table. Pentane, Hexane, Heptane, Cyclohexane, Cyclohexene, Toluene

ii) Identify the functional groups present in each compound.

Part B: Solubility of Alkanes

1) Obtain six small or medium-sized test tubes.

2) Add the following reagents to each test tube:

Test tube 1: 1 mL of pentane and 1 mL of water

Test tube 2: 1 mL of hexane and 1 mL of water

Test tube 3: 1 mL of heptane and 1 mL of water

Test tube 4: 1 mL of pentane and 1 mL of toluene

Test tube 5: 1 mL of hexane and 1 mL of toluene

Test tube 6: 1 mL of heptane and 1 mL of toluene

3) Thoroughly mix each solution and record your observations.

4) Pour the contents of each test tube into the non-halogenated waste container. Thoroughly rinse and dry each test tube.

Part C: Density of Alkanes

1) Obtain the mass of an empty 10 mL graduated cylinder.

2) Add 2 mL of pentane and get the combined mass of the cylinder and pentane.

3) Calculate the density of pentane.

4) Pour the contents of the graduated cylinder into the non-halogenated waste container. Thoroughly clean and dry the graduated cylinder.

5) Repeat steps 1-4 for hexane and heptane.

Part D: Volatility of Alkanes

1) Obtain a watch glass. Under the hood, add 1 mL of pentane to the watch glass, and record the time it takes for the pentane to evaporate completely.

2) Repeat the procedure for hexane and heptane.

3) Thoroughly clean and dry the watch glass.

Part E: Combustion of Hexane

Under the hood, add 10 drops of hexane to an evaporating dish. CAREFULLY, with supervision from your instructor, light a match and touch it to the hexane. Close the hood door, and observe the reaction. (Note: This may be performed as an instructor demo.)

Part F: Halogenation of Hydrocarbons

1) Obtain four small or medium-sized test tubes. Under the hood, add the following reagents:

Test tube 1: 10 drops of cyclohexane and five drops of bromine solution.

Test tube 2: 10 drops of cyclohexene and five drops of bromine solution

Test tube 3: 10 drops of toluene and five drops of bromine solution

Test tube 4: 10 drops of unknown and five drops of bromine solution

2) Thoroughly mix each solution and record your observations.

3) Under the hood, pour the contents of each test tube into the halogenated organic waste container. Clean and rinse test tubes under the hood before removing them to dry.

Part G: Oxidation of Hydrocarbons

1) Gather four small or medium test tubes. Under the hood, add the following reagents:

Test tube 1: 10 drops of cyclohexane and five drops of potassium permanganate solution

Test tube 2: 10 drops of cyclohexene and five drops of potassium permanganate solution

Test tube 3: 10 drops of toluene and five drops of potassium permanganate

Test tube 4: 10 drops of unknown and five drops of potassium permanganate

2) Thoroughly mix each solution and record your observations.

3) Under the hood, pour the contents of each test tube into the non-halogenated organic waste container. Clean and rinse test tubes under the hood before removing them to dry.

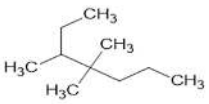
PRE-LAB QUESTIONS Name _____

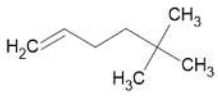
1) Using the molecular formulas, determine if the following compounds are saturated or unsaturated.

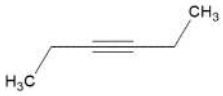
Part A: C_3H_8

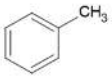
Part B: C_5H_8

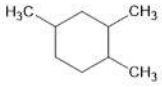
Part C: C_7H_8

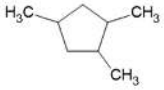
a) 

b) 

c) 

d) 

e) 

f) 

2) Provide an acceptable name for the following hydrocarbons.

3) Draw structures for the following hydrocarbons.

Part A: 2-methyl-1-pentene

Part B: hept-3-yne

Part C: 2,2,5-trimethylhexane

Part D: 1-ethyl-3-methylbenzene

Part E: 1-methylcyclopentene

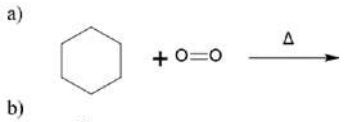
4) Are hydrocarbons polar or nonpolar? Explain.

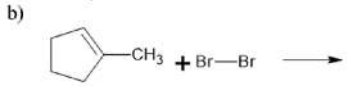
5) What would you predict will happen when water adds a hydrocarbon?

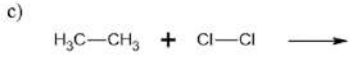
6) Rank the following compounds in order of increasing predicted boiling point.

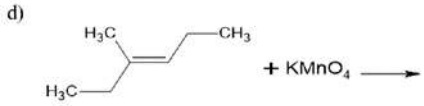
Pentane 2-methylbutane 2,2-dimethylpropane hexane

7) Predict the products of each reaction. If no reaction is predicted, write 'NR' for no response.

a) 

b) 

c) 

d) 

DATA AND OBSERVATIONS

Name _____Lab Partner(s) _____

Part A: Structures of Hydrocarbons

Table 1**: Identification of Hydrocarbons**

Compound	Structure	Functional group
Pentane		
Hexane		
Heptane		
Cyclohexane		
Cyclohexene		
Toluene		

Table 1**: Solubility Test**

Test Tube	Contents	Observation
1	Pentane and Water	
2	Hexane and Water	
3	Heptane and Water	
4	Pentane and Toluene	
5	Hexane and Toluene	
6	Heptane and Toluene	

Part C: Density of Alkanes

Table 4: **Calculation of Density**

	Pentane	Hexane	Heptane
Mass of the empty cylinder			
Mass of cylinder and compound			
Mass of compound			
Volume of compound			
Density of compound			

Show all the calculations for densities:

Part D: Volatility of Alkanes

Table 5: **Evaporation Tests**

Compound	Time for Complete Evaporation
Pentane	
Hexane	
Heptane	

Part E: Combustion of Hexane

Record your observations here:

Write the balanced equation for the complete combustion of hexane:

Parts F-G: Halogenation/Oxidation of Hydrocarbons

Unknown letter/number =

Table 6: **Bromine and Potassium Permanganate Test**

	Results of the Bromine Test	Results of the Potassium Permanganate Test
Cyclohexane		
Cyclohexene		
Toluene		
Unknown		

Based on these test results, is your unknown saturated or unsaturated? Explain.

POST-LAB QUESTIONS

1) Summarize the results of the solubility test of alkanes (part B). Why were certain solutions formed while others were not?

2) Explain any noted trends in part C (density of alkanes).

3) Part A: What is the relationship between volatility and boiling point?

Part B: Look up and record the boiling points of pentane, hexane, and heptane. Does the trend described in part A match the results of your experiment (see results from part D)? Explain.

4) Refer to part E (combustion of hexane). What other chemicals used in this experiment (pentane, heptane, cyclohexane, cyclohexene, toluene, and water) would you expect to react the same way as hexane?

5) Draw and name five constitutional isomers with molecular formula C_5H_{10}.

6) An unknown molecular formula C_5H_8 showed positive test results with the bromine and potassium permanganate reagents. Using only this information, draw three possible structures for the unknown

Please click here to access the Pre-Lab, Data Tables, and Post-Lab in Word or PDF format. Complete them and upload the lab report according to your instructor's instructions.

LAB 3 - ALCOHOLS AND AMINES

LPURPOSE

- The purpose of this experiment is to:
- Objective 2

PURPOSE

- Draw structures of alcohols and amines
- Name alcohols and amines
- Explore the physical properties and reactions of alcohols and amines
- Perform chemical tests on alcohols

INTRODUCTION

Alcohols are organic compounds with -OH bonded to carbon, and amines have a nitrogen bonded to a carbon chain. Both alcohols and amines are classified as primary, secondary, or tertiary according to the definitions provided below:

Definition: Alcohols and Amines

***Primary Alcohol*:** The carbon bonded to -OH is bonded to one carbon atom.

***Secondary Alcohol*:** The carbon bonded to -OH is bonded to two carbon atoms.

***Tertiary Alcohol*:** The carbon bonded to -OH is bonded to three carbon atoms.

***Primary amine*:** Nitrogen is bonded to one carbon and two hydrogen atoms.

***Secondary amine*:** Nitrogen is bonded to two carbons and one hydrogen atom.

***Tertiary amine*:** Nitrogen is bonded to three carbons and no hydrogen atoms.

Alcohols and amines exhibit similar physical properties, including water solubility and trends in boiling points. However, they widely differ in terms of their acid-base properties. Alcohols act as weak acids, and amines are weak bases. In this experiment, you will explore the structure, nomenclature, physical properties, and acid-base properties of alcohols and amines. The pre-lab questions will help you prepare for this part of the lab.

You will also run various reactions and chemical tests with compounds containing these functional groups. Amines react with HCl to produce amine salts, as shown by the reaction below:

$$CH_3NH_2 + HCl \rightarrow CH_3NH_3^+ + Cl^-$$

Amine salts are important in medicinal chemistry because they make pharmaceutical compounds water soluble and more bioavailable.

One of the most common reactions for alcohols is their oxidation to form aldehydes, ketones, and carboxylic acids. When alcohol is reacted with sodium dichromate in sulfuric acid (chromic acid reagent), the products of oxidation are as follows:

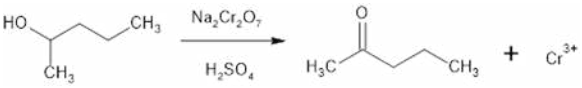
1) Primary alcohol is oxidized to form aldehydes, which are oxidized to carboxylic acids.

2) Secondary alcohols are oxidized to form ketones.

3) Phenols (which contain -OH bonded to a benzene ring) are oxidized to quinones.

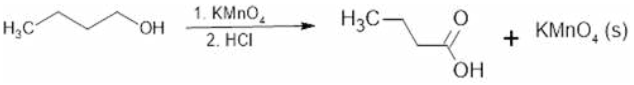
4) Tertiary alcohols do not undergo oxidation.

The chromic acid test is a chemical test used to identify certain alcohols. Consider the reaction between 2-pentanol (pentan-2-ol) and the chromic acid reagent:



The secondary alcohol is oxidized to form a ketone. In return, chromium is reduced from the +6 oxidation state (in which the solution is orange) to the +3 oxidation state (in which the solution turns blue-green). The formation of a blue-green solution is a positive test result. A negative test result would be the persistence of an orange solution.

Alcohols can also be oxidized under certain conditions with potassium permanganate, as shown by the reaction with 1-butanol.



When the primary alcohol is oxidized to carboxylic acid, the purple color of the permanganate solution disappears as manganese is reduced from +6 to +4, forming a brown solid. A positive test result for the oxidation of alcohols with potassium permanganate will be the formation of a brown solid in various colored solutions.

In addition to the chromic acid and potassium permanganate tests, you will also run the ferric chloride test, a chemical test for phenols. Phenols react with ferric chloride, forming a purple solution, a positive test result. A negative test result, indicating the absence of a phenol, would be the presence of a yellow solution, the original color of ferric chloride.

SAFETY PRECAUTIONS

1) Always wear goggles while working with chemicals in this lab.

2) Wear gloves while working on this experiment.

3) Always handle all chemicals under a working fume hood.

4) Dispose of all waste in the appropriate waste container, which should also be kept under a fume hood at all times.

5) Thoroughly clean all glassware and your work area at the end of the experiment.

6) Before leaving the lab, wash your hands.

EQUIPMENT AND CHEMICALS NEEDED

Chemicals	Chemicals	Equipment
Ethanol	Phenol	6 small or medium-sized test tubes
Isopropyl alcohol	unknown alcohol	Test tube rack

T-butyl alcohol	Aniline	Stirring rod
Cyclohexanol	N-methylaniline	pH paper
Triethylamine	1 M NaOH (or similar concentration)	Watch glass
2% Chromic acid reagent	1 M HCl (or similar concentration)	Red litmus paper
Acetone	Concentrated HCl	Blue litmus paper
1% aqueous $FeCl_3$	1% aqueous potassium permanganate solution (or similar concentration)	Halogenated and Non-halogenated waste containers

2% Chromic acid reagent (dissolve 2 g $K_2Cr_2O_7$ in 10 mL of 6 M H_2SO_4; carefully add H_2O to make 100 mL; must be freshly prepared) will be used by the whole class. Your instructor may have 2% Chromic acid ready for testing.

EXPERIMENTAL PROCEDURE

Part A: Structures of Alcohols and Amines

a) Draw structures (Lewis, condensed, or line-bond) for the following compounds that will be used in this experiment in the data table: Ethanol, 2-propanol, 2-methyl-2-propanol, Cyclohexanol, Phenol, Aniline, N-methylaniline, Triethylamine.

b) Classify them as primary, secondary, or tertiary.

Part B: Solubility and pH of alcohols

1) Obtain six small or medium-sized test tubes.

2) Add the following reagents to each test tube:

Test tube 1: 1 mL of ethanol and 1 mL of water.

Test tube 2: 1 mL of 2-propanol and 1 mL of water

Test tube 3: 1 mL of 2-methyl-2-propanol and 1 mL of water.

Test tube 4: 1 mL of cyclohexanol and 1 mL of water.

Test tube 5: 1 mL of phenol and 1 mL of water.

Test tube 6: 1 mL of the unknown alcohol and 1 mL of water.

3) Thoroughly mix each solution and record your observations.

4) Test the pH of each solution with pH paper. This can be done by dipping a stirring rod into the solution and touching the tip of the stirring rod to a piece of pH paper. Be sure to rinse and wipe dry the stirring rod between each solution and use only a minimal amount of pH paper.

5) To insoluble alcohols in #3 above, add drops of 1 M NaOH until the solution tests basic with red litmus paper. (Red litmus paper will turn blue.). This can be done similarly to pH paper (#4 above). Record your observations.

6) Pour the contents of each test tube into the non-halogenated organic waste container. Clean and dry the test tubes and save them for part C.

Part C: Solubility of pH of Amines

1) Obtain the three small or medium-sized test tubes.

2) Add the following reagents to each test tube:

Test tube 1: 1 mL of aniline and 1 mL of water.

Test tube 2: 1 mL of N-methylaniline and 1 mL of water.

Test tube 3: 1 mL of triethylamine and 1 mL of water.

3) Thoroughly mix each solution and record your observations.

4) Test the pH of each solution with pH paper This can be done by dipping a stirring rod into the solution and touching the tip of the stirring rod to a piece of pH paper Be sure to rinse and wipe dry the stirring rod between each solution and use only a minimal amount of pH paper.

5) To insoluble amines in #3 above, add drops of 1 M HCl until the solution tests acidic with blue litmus paper (Blue litmus paper will turn red). pH paper can be used in place of blue litmus paper (#4 above). Record your observations.

6.) Pour the contents of each test tube into the halogenated organic waste container. Clean and dry the test tubes, then store them for later use.

Part D: Volatility of Phenol and Aniline

1) Obtain a watch glass. Under the hood, add five drops of phenol and five drops of aniline to separate the ends of the watch glass.

2) Record the time it takes each substance to evaporate completely.

3) Clean and dry the watch glass, and save it for part E.

Part E: Reaction of Triethylamine with Concentrated HCl

Under the hood, add one drop of triethylamine and one drop of concentrated HCl to separate ends of a watch glass. Record your observations. (This can also be run as an instructor demo.)

Part F: Oxidation of Alcohols with the Chromic Acid Reagent

1) Obtain six small or medium-sized test tubes. Under the hood, add the following reagents:

Test tube 1: 1 mL of acetone, 10 drops of ethanol, three drops of chromic acid solution.

Test tube 2: 1 mL of acetone, 10 drops of isopropyl alcohol, three drops of chromic acid solution.

Test tube 3: 1 mL of acetone, 10 drops of 2-methyl-2-propanol, and three drops of chromic acid solution.

Test tube 4: 1 mL of acetone, 10 drops of cyclohexanol, and three drops of chromic acid solution.

Test tube 5: 1 mL of acetone, 10 drops of phenol, and three drops of chromic acid solution.

Test tube 6: 1 mL of acetone, 10 drops of the unknown alcohol, three drops of chromic acid solution.

2) Thoroughly mix each solution and record your observations.

3) Pour the contents of each test tube into the non-halogenated organic waste container. Clean and rinse the test tubes before removing them to dry. Save the test tubes for part G.

Part G: Oxidation of Alcohols with Potassium Permanganate

1) Obtain six small or medium-sized test tubes. Under the hood, add the following reagents:

Test tube 1: 10 drops of ethanol and three drops of potassium permanganate solution.

Test tube 2: 10 drops of isopropyl alcohol and three drops of potassium permanganate solution.

Test tube 3: 10 drops of 2-methyl-2-propanol and three drops of potassium permanganate solution.

Test tube 4: 10 drops of cyclohexanol, three drops of potassium permanganate solution.

Test tube 5: 10 drops of phenol and three drops of potassium permanganate solution.

Test tube 6: 10 drops of unknown alcohol and three drops of potassium permanganate solution.

2) Thoroughly mix each solution, and record your initial observations.

3) For the test tubes, after 10 minutes, add two drops of 6 M HCl to the purple permanganate solution. Record your observations.

5) Pour the contents of each test tube into the non-halogenated organic waste container. Clean and rinse the test tubes before removing them to dry. Save the test tubes for part H.

Part H. Ferric Chloride Test

1) Obtain six small or medium-sized test tubes. Under the hood, add the following reagents:

Test tube 1: 5 drops of ethanol and five drops of $FeCl_3$ solution.

Test tube 2: 5 drops of isopropyl alcohol and five drops of $FeCl_3$ solution.

Test tube 3: 5 drops of 2-methyl-2-propanol and five drops of $FeCl_3$ solution.

Test tube 4: 5 drops of cyclohexanol and five drops of $FeCl_3$ solution.

Test tube 5: and five drops of phenol and five drops of $FeCl_3$ solution.

Test tube 6: 5 drops of unknown alcohol and five drops of $FeCl_3$ solution.

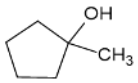
2) Thoroughly mix each solution and record your observations.

3) Pour the contents of each test tube into the halogenated organic waste container. Clean and rinse test tubes before removing them to dry.

PRE-LAB QUESTIONS Name _____

1. Provide an acceptable name for each of the following compounds.

a)



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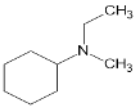

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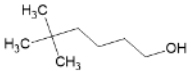

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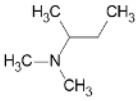
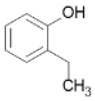


e)



2) Draw structures for the following compounds.

Part A: 3-pentanol

Part B: 1-methylcyclohexanol

Part C: hexan-1-ol

Part D: 3-methylphenol

Part E: 1-butanamine

Part F: N, N-dimethyl-2-pentanamine

Part G: N-ethyl-3-heptanamine

3) Identify the most important intermolecular force present in the following types of compounds:

Part A: Primary Amines

Part B: Tertiary Amines

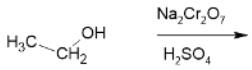
Part C: Secondary Alcohols

Part D: Primary Alcohols

4) What is litmus paper? Explain how it can determine whether a solution is acidic or basic.

5) Predict the products of each reaction.

a)



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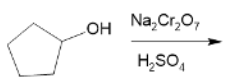

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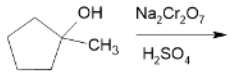

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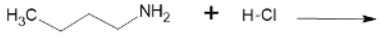


DATA AND OBSERVATIONS

Name _____Lab Partner(s) _____

Part A: Structures of Alcohols and Amines

Compound	Structure	Classificat
ethanol		
Isopropyl alcohol		
2-methyl-2-propanol		
Cyclohexanol		
Phenol		
Aniline		
N-methylaniline		
Triethylamine		

Part B: Solubility and pH of Alcohols

Test tube	Contents	Soluble or insoluble?	pH	Observations after adding NaOH (if applicable)
1	Ethanol and water			
2	Isopropyl alcohol and water			
3	2-methyl-2-propanol and water			
4	cyclohexane and toluene			
5	phenol and toluene			
6	Unknown and toluene			

Part C: Solubility and pH of Amines

Test tube	Contents	Soluble or insoluble?	pH	Observations after adding HCl (if applicable)
1	Aniline and water			
2	N-methylaniline and water			
3	Triethylamine and water			

Part D: Volatility of Phenol and Aniline

Compound	Time for complete evaporation
Phenol	
aniline	

Part E: Reaction with Triethylamine and Concentrated HCl

Record your observations here:

Write the balanced equation for the reaction:

Parts F-H: Chemical Tests for Alcohols

Compound	Results of the Chromic Acid Test	Results of the Potassium Permanganate Test	Results of the Ferric Chloride Test
Ethanol			

Isopropyl alcohol			
2-methyl-2-propanol			
Cyclohexanol			
Phenol			
Unknown			

Analysis of Unknown Alcohol:

Unknown number letter =

Solubility in water =

pH =

results of chromic acid test =

Results of the potassium permanganate test =

Results of the ferric chloride test =

Based on the above results, identify the unknown alcohol as one of the five knowns:

Is your result conclusive? Explain.

POST-LAB QUESTIONS

1) For parts B and C, explain what happens when NaOH is added to insoluble alcohols and when HCl is added to insoluble amines. What type of reaction is occurring here?

2) Part A: Look at the boiling points of phenol and aniline. How do the boiling points relate to the results from part D?

Part B: Look up the boiling points for the following pairs of compounds:

Ethanol and ethanamine

1-butanol and 1-butanamine

Cyclohexanol and cyclohexanamine

Part C: What do your answers for parts A and B of this question tell you about hydrogen bonding in alcohols and amines?

3) Draw structures for oxidation products of known alcohols that reacted with the chromic acid reagent in part F.

4) An unknown with molecular formula $C_4H_{10}O$ showed results with the following chemical tests:

Chromic acid test	Formation of orange solution
Potassium permanganate test	Formation of purple solution; after 10 min, two drops of 6 M HCl were added, and the purple solution.n remains
Ferric chloride test	Formation of yellow solution

Using only this information, draw two possible structures for the unknown.

LAB 4 - ALDRHYDE AND KETONE

LPURPOSE

- The purpose of this experiment is to:
- Objective 2

PURPOSE

· Draw structures of aldehydes and ketones

· Name aldehydes and ketones

· Explore the physical properties and reactions of aldehydes and ketones

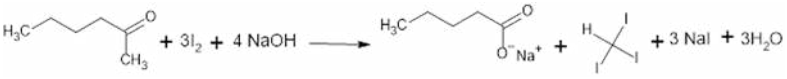
· Perform chemical tests on aldehydes and ketones

INTRODUCTION

Aldehydes are organic compounds that contain a carbonyl group (C=O) bonded to a hydrogen atom, whereas ketones have a carbonyl group located between two carbon atoms. As you will observe in this experiment, aldehydes and ketones exhibit similar physical and chemical properties. Like other functional groups you have previously studied, chemical tests can be used to identify aldehydes and ketones. Aldehydes react with Benedict's reagent, a solution containing copper(II) ions, to produce carboxylic acids, as illustrated by the equation below:

During this reaction, copper(II) ions are reduced to copper(I) ions, leading to the formation of a red-orange precipitate. This indicates a positive test result for Benedict's reagent. A negative test result is shown by the persistence of a blue solution, due to the presence of unreacted copper(II) ions. Since ketones do not undergo oxidation with common laboratory reagents, they will yield a negative test result with Benedict's reagent.

The iodoform test is a chemical procedure primarily used for methyl ketones, which have a methyl group attached to the carbonyl group. Consider the reaction between 2-hexanone and the iodoform reagent:



During the reaction, the red color of iodine disappears, forming a yellow solution or precipitate (due to the presence of iodoform). This indicates a positive test result. In contrast, if the red color of iodine remains, it signifies a negative test result.

SAFETY PRECAUTIONS

1) Wear chemical splash goggles while working on this experiment.

2) Handle all reagents and perform all parts of this experiment under a working fume hood.

3) Gloves are provided, and we encourage you to wear them.

4) Be cautious when handling hot glassware after performing the simple distillation.

5) Dispose of all waste in the appropriate container and clean all equipment and your work area when finished.

CHEMICALS AND EQUIPMENT NEEDED

CHEMICALS	CHEMICALS	CHEMICALS	EQUIPMENT	EQUIPMENT
Propanal	Cyclohexanone	Iodoform Reagent*	Styrofoam Cup	7 small or medium-sized Test Tubes
Butanal	Unknown aldehyde or ketone	Benedict's Reagent*	Thermometer	Test Tube Rack
Benzaldehyde	Solid NaBr	10% NaOH solution (or similar concentration)	Watch glass	400 mL Beaker
Acetone	Solid NaI	Non-halogenated organic waste container	Hot Plate	Stirring Rod
2-butanone	Solid NaCl	Halogenated organic waste container	600 mL Beaker	Spatula
Water	Isopropyl alcohol			

EXPERIMENTAL PROCEDURE

Part A: Structures and Solubility

1) Draw Lewis, condensed, or line-bond structures for the compounds used in this experiment.

Propanal, butanal, benzaldehyde, acetone, 2-butanone, cyclohexanone

2) Obtain 7 test tubes. Add the following reagents:

Test tube 1: 1 mL of Propanal and 1 mL of water.

Test tube 2: 1 mL of Butanal and 1 mL of water.

Test tube 3: 1 mL of Benzaldehyde and 1 mL of water.

Test tube 4: 1 mL of Acetone and 1 mL of water.

Test tube 5: 1 mL of 2-butanone and 1 mL of water.

Test tube 6: 1 mL of Cyclohexanone and 1 mL of water.

Test tube 7: 1 mL of Unknown and 1 mL of water.

3) Thoroughly mix the contents of each test tube and record your observations.

4) Pour the contents of each test tube into the non-halogenated organic waste container. Clean and dry the test tubes, then store them for later use.

Part B: Acetone as a Solvent

1) Obtain three small or medium-sized test tubes. Add the following reagents as indicated.

Test tube 1: a small scoop of solid NaCl and 1 mL of acetone.

Test tube 2: a small scoop of solid NaBr and 1 mL of acetone.

Test tube 3: a small scoop of solid NaI and 1 mL of acetone.

Note: Clean and dry the spatula between uses to avoid cross-contamination of the solid bottles.

2) Thoroughly mix the contents of each test tube and record your observations.

3) Pour the contents of each test tube into the halogenated organic waste container. Wash and dry the test tubes, then save them for future use.

4) Fill a 400 mL beaker with 10 mL of acetone. Add a Styrofoam cup containing acetone to the beaker and record your observations. (This can also be done as a demonstration by the instructor.)

5) Pour the contents of the beaker into the non-halogenated organic waste container. Clean and dry the beaker.

Part C: Volatility of Acetone

Get a watch glass. To separate the sides of the watch glass, add five drops of acetone and five drops of isopropyl alcohol. Record how long it takes for each solvent to evaporate completely.

Part D: Iodoform Test

1) Obtain seven small or medium-sized test tubes. Add the following reagents:

Test tube 1: 10 drops of Propanal, 2 mL of water, and 10 drops of 10% NaOH.

Test tube 2: 10 drops of Butanal, 2 mL of water, and 10 drops of 10% NaOH.

Test tube 3: 10 drops of Benzaldehyde, 2 mL of water, and 10 drops of 10% NaOH.

Test tube 4: 10 drops of Acetone, 2 mL of water, and 10 drops of 10% NaOH.

Test tube 5: 10 drops of 2-butanone, 2 mL of water, and 10 drops of 10% NaOH.

Test tube 6: Ten drops of cyclohexanone, 2 mL of water, and ten drops of 10% NaOH.

Test tube 7: 10 drops of Unknown, 2 mL of water, and 10 drops of 10% NaOH

2) Obtain a hot plate and a 600 mL beaker, filling it three-quarters with water. Place the test tubes in the beaker with water, and set the beaker on the hot plate. Heat the water bath to a temperature of no more than 55 °C. You can check the temperature with a thermometer.

3) Place the test tubes in the water bath and add 20 drops of the iodoform reagent. Mix using a glass stirring rod. After 2 minutes, document your observations.

4) Pour the contents of each test tube into the halogenated organic waste container. Rinse and dry the test tubes, then set them aside for later use.

5) Save the water bath for part E.

Part E: Benedict's Test

1) Obtain seven small or medium test tubes. Add the following reagents:

Test tube 1: 10 drops of Propanal and 2 mL of Benedict's reagent.

Test tube 2: 10 drops of butanal and 2 mL of Benedict's reagent.

Test tube 3: 10 drops of benzaldehyde and 2 mL of Benedict's reagent.

Test tube 4: 10 drops of acetone and 2 mL of Benedict's reagent.

Test tube 5: 10 drops of 2-butanone and 2 mL of Benedict's reagent.

Test tube 6: 10 drops of cyclohexanone and 2 mL of Benedict's reagent.

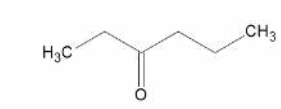
Test tube 7: 10 drops of unknown and 2 mL of Benedict's reagent.

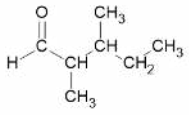
2) Mix the contents of each test tube using a glass stirring rod, and then place the test tubes in the water bath used for part D. Heat it to a boil. Once the water starts to boil, let the test tube sit in the water bath for 5 minutes. Record your observations after 5 minutes.

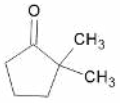
3) Pour the contents of each test tube into the non-halogenated organic waste container. Disassemble the water bath, then wash and dry the test tubes.

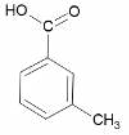
PRE-LAB QUESTIONS Name _____

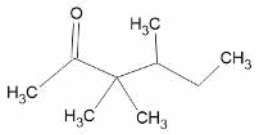
1) Provide a reasonable name for the following compounds.

a) 

b) 

c) 

d) 

e) 

2) Draw structures for the following compounds.

Part A: 2,2-dimethyl-3-hexanone

Part B: 2-bromocyclohexanone

Part C: 2-ethylpentanal

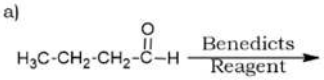
Part D: hexanal

Part E: 5-chloro-2,2-dimethylcyclopentanone

3) Are aldehydes and ketones polar or nonpolar? What is the primary intermolecular force present in these compounds?

4) Predict the major products from each of the following reactions.

a)



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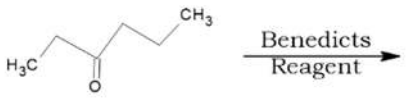

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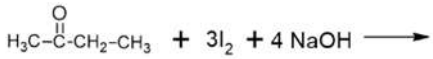

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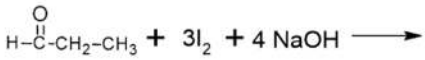


DATA AND OBSERVATIONS

Name _____Lab Partner(s) _____

Part A: Structures and Solubility

Compound	Structure	Soluble or Insolub
Propanal		
Butanal		
Benzaldehyde		
Acetone		
2-butanone		
Cyclohexanone		
Unknown	X	

Part B: Acetone as a Solvent

Compound	Soluble or Insoluble in Acetone?
NaCl	
NaBr	
NaI	

Describe what happens when the Styrofoam cup is added to acetone.

Part C: Volatility of Acetone

Compound	Primary Intermolecular Force	The Time it Takes to Evaporate
Isopropyl alcohol		
Acetone		

Parts D and E: Chemical Tests

Compound	Results of the Iodoform Test	Results of Benedict's Test
Propanal		
Butanal		
Benzaldehyde		
Acetone		
2-butanone		
Cyclohexanone		
Unknown		

Analysis of Unknown:

Unknown letter or number:

Based on the results of the solubility, iodoform, and Benedict's tests, identify your unknown as one of the six knows.

POST-LAB QUESTIONS

1) Explain how certain aldehydes and ketones can dissolve in water.

2) The halide test in acetone is a chemical test for certain alkyl halides. Based on the result from part B of this experiment, which halogen (Cl, Br, or I) would best serve this purpose? Explain.

3) Explain the results of part C (volatility of acetone) in relation to intermolecular forces.

4) Write the major product for any reaction occurring in part E (Benedict's test).

5) An unknown compound with the molecular formula C_4H_8O yielded the following results in four chemical tests.

Chemical test	**Result**
Bromine test	Formation of a colorless solution
Potassium permanganate test	Formation of a brown solid
Benedict's test	Formation of blue solution
Iodoform test	Formation of red solution

Using only the results of these chemical tests, draw two possible structures for the unknown.

*Preparation of Reagents: To prepare the Iodoform reagent, dissolve 10 g of I_2 and 20 g of KI in sufficient water to achieve a total volume of 100 mL. For Benedict's reagent, dissolve 86 g of sodium citrate and 50 g of anhydrous sodium carbonate in 400 mL of warm water. Next, dissolve 8.6 g of copper(II) sulfate pentahydrate in 50 mL of water, then add this solution to the citrate/carbonate mixture. Add sufficient water to obtain a final volume of 500 mL.

LAB 5 - CARBOXYLIC ACIDS AND DERIVATIVES

CARBOXYLIC ACIDS, ESTERS, AND AMIDES

PURPOSE

- The purpose of this experiment is to:
- Objective 2

PURPOSE

- Name and draw structures for carboxylic acids, esters, and amides
- Explore the physical properties and reactions of carboxylic acids, esters, and amides

INTRODUCTION

Carboxylic acids are organic compounds in which -OH is bonded to a carbonyl group. In the derivatives of carboxylic acids, the -OH group bonded to the carbonyl carbon is replaced by another group. Two common derivatives are esters and amides. In esters, the carbonyl group is bonded to an alkoxy group, -OR (where R is an alkyl group), while amides contain a nitrogen atom bonded to a carbonyl group.

Like alcohols and thiols, carboxylic acids behave as weak acids. They also react with alcohols to produce esters. Both esters and amides can be hydrolyzed in sodium hydroxide to yield carboxylic acid salts. In this experiment, you will investigate these reactions and analyze the products using various methods.

SAFETY PRECAUTIONS

1) Always wear goggles while working with chemicals in this lab.

2) Wear gloves while working on this experiment.

3) Always handle all chemicals under a working fume hood.

4) Dispose of all waste in the appropriate waste container, which should always be kept under a fume hood.

5) Thoroughly clean all glassware and your work area at the end of the experiment.

6) Before leaving the lab, wash your hands.

CHEMICALS AND EQUIPMENT NEEDED

Table 5.1: CHEMICALS AND EQUIPMENT

CHEMICALS	CHEMICALS	CHEMICALS	EQUIPMENT	EQUIPMENT
Acetic acid	Methyl salicylate	1-pentanol	Hot plate	Red litmus paper
Citric acid	Salicylic acid	Methanol	600 mL beaker	Test tube rack
Benzoic acid	10% NaOH	Ethanol	Thermometer	pH paper
Acetamide	10% HCl	1-propanol	Stirring rod	Spatula
Benzamide	Propanoic acid	1-butanol	Non-halogenated waste container	5 small or medium-sized test tubes
Water	Sodium bicarbonate	1-octanol	Halogenated organic waste container	-

EXPERIMENTAL PROCEDURE

Part A: Structure, Solubility, and pH of Carboxylic Acids and Amides

1) Draw the structures for the following compounds: acetic acid, citric acid, benzoic acid, acetamide, and benzamide. Identify the primary functional group in each compound.

2) Gather five small to medium-sized test tubes, and add the following reagents in each test tube:

Test tube 1: 2 mL of water and 1 mL of acetic acid

Test tube 2: 2 mL of water and a small amount of citric acid (enough to cover a spatula tip)

Test tube 3: 2 mL of water and a small amount of benzoic acid (enough to cover a spatula tip)

Test tube 4: 2 mL of water and a small amount of acetamide (enough to cover a spatula tip)

Test tube 5: 2 mL of water and a small amount of benzamide (enough to cover a spatula tip)

3) Use a stirring rod to mix each solution, ensuring that the rod is clean between each test tube. Record whether the substances dissolve in water.

4) Test the pH of each solution using pH paper. To do this, dip a stirring rod into the solution and touch its tip to pH paper. Ensure that you rinse and dry the stirring rod between each solution, using only a minimal amount of pH paper.

5) Save test tubes 1 to 3 for part B. Pour the contents of test tubes 4 and 5 into the non-halogenated waste container. Clean and dry the test tubes, and save them for future use.

Part B: Reactions of Carboxylic Acids with Sodium Bicarbonate

1) Place test tubes 1-3 from part A in a test tube rack.

2) Measure the room temperature with a thermometer. Keep the thermometer in the air until the temperature stabilizes.

3) To test tube 1, add a small scoop of sodium bicarbonate, and record your observations.

4) After about 10 to 15 seconds, measure the temperature of the solution in test tube 1. Wait for the thermometer reading to stabilize before recording the temperature.

5) Repeat steps 2-4 with test tubes 2 and 3.

6) Pour the contents of each test tube into the non-halogenated waste container. Clean and dry the test tubes, then save them for future use.

Part C: Formation of Esters

For this section of the experiment, you will create three esters. Select combinations of carboxylic acids and alcohols from the list below:

Table 5.2 List of Carboxylic Acids and Alcohols for the formation of Esters

Carboxylic Acids	Alcohols
Acetic acid	Methanol
Salicylic acid	Ethanol
Propanoic acid	1-propanol
	1-butanol
	1-pentanol
	1-octanol

1) Obtain three small or medium test tubes. Add 3 mL of alcohol, 2 mL of carboxylic acid, and 10 drops of concentrated sulfuric acid to each test tube while ensuring they are properly labeled.

2) Place the test tubes in a boiling water bath (a 600 mL beaker three-quarters filled with water on a hot plate) for 15 minutes. Do not start the timer until the water has come to a boil.

3) Carefully take out the test tubes from the hot water bath and detect the odor of each solution by gently wafting the vapors toward you.

4) Formulate an equation for the creation of each ester and include a name for each ester.

5) Pour the contents of each test tube into the non-halogenated waste container. Clean and dry the test tubes, then save them for future use.

Part D. Ester Hydrolysis

1) Carefully detect the odor of methyl salicylate.

2) Add 3 mL of water, five drops of methyl salicylate, and 1 mL of 10% NaOH to a medium-sized test tube. Note your initial observations.

3) Put the test tube in a boiling water bath for 30 minutes. Wait to start the timer until the water reaches a boil.

4) Remove the test tube from the water bath with care, and note your observations, including any changes in odor.

5) Compose an equation for the reaction that occurred.

6) Add drops of 10% HCl to the test tube contents until a solid forms.

7) Formulate an equation to clarify the observations noted in step 6.

8) Transfer the contents of the test tube into the halogenated organic waste container.

Part E. Amide Hydrolysis

1) Combine 2 mL of water, a small amount of acetamide (enough to cover the tip of the spatula), and 2 mL of 10% NaOH in a medium-sized test tube.

2) Place the test tube in a boiling water bath for 5 minutes. Do not start the timer until the water reaches a boil.

3) While the test tube is placed in the water bath, hold a piece of red litmus paper over its opening. Note any change in the litmus paper's color and any odors detected.

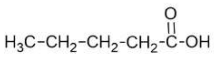
4) Write an equation to explain the observations noted in step 3.

5) Pour the contents of the test tube into the non-halogenated waste container.

PRE-LAB QUESTIONS Name _____

1) Provide a reasonable name for the following organic compounds.

a)



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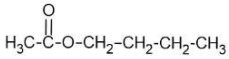

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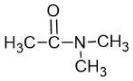

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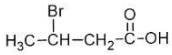

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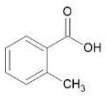


2) Draw structures for each compound.

Part A: 2,2-dimethyl butanoic acid

Part B: Butyl ethanoate

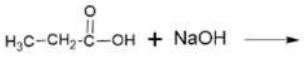
Part C: pentanamide

Part D: sodium propanoate

Part E: cyclopentyl hexanoate

3) Predict the products of the following reactions.

a)



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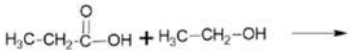

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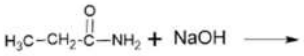


DATA AND OBSERVATIONS

Name _____Lab Partner(s) _____

Part A. Structure, Solubility, and pH of Carboxylic Acids and Amides

Table 5.3: Draw the Structure and Indicate pH and Solubility of the Compounds

Compound	Structure	Soluble or Insoluble in Water?	pH
Acetic Acid			
Citric Acid			
Benzoic Acid			
Acetamide			
Benzamide			

Part B: Reactions of Carboxylic Acids with Sodium Bicarbonate

Test Tube	Initial Temperature (air)	Observations upon the Addition of Sodium Bicarbonate	Final Temperatur (Temperature of Solution)
1 (Acetic Acid)			
2 (Citric Acid)			
3 (Benzoic Acid)			

Part C: Formation of Esters

Name of Alcohol	Name of Carboxylic Acid	Reaction	Name of Ester	Odor

Part D. Ester Hydrolysis

1) Odor of methyl salicylate:

2) Initial observations (before heating):

3) Final observations (after heating):

4) Final odor:

5) Equation for the hydrolysis of methyl salicylate:

6) Observations upon addition of HCl:

7) Equation to describe the formation of a solid upon the addition of HCl:

Part E. Amide Hydrolysis

1) Observations of reaction (change in litmus paper, odor, etc.)

2) Equation to describe the hydrolysis of acetamide:

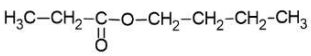
POST-LAB QUESTIONS

1) For part A, account for any differences in the solubility of citric acid and benzoic acid in water.

2) For part B, is the reaction between carboxylic acids and sodium bicarbonate exothermic or endothermic? Explain.

3) Name the following esters. Conduct a literature search to determine the odor (if any) associated with each ester.

a)



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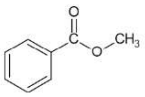

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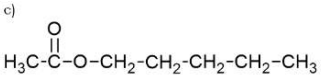

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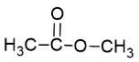

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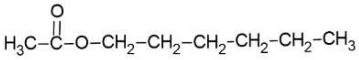


4) Soaps, which we will explore in future experiments, are formed from the reaction of a triglyceride with aqueous sodium hydroxide. Based on the results from part D of this experiment, predict the product(s) of the following response.

5) For part E, what is responsible for the odor produced during the saponification of acetamide?

LAB 6 - SYNTHESIS OF ASPRIN

Synthesis and Analysis of Aspirin

LPURPOSE

- The purpose of this experiment is to:
- Objective 2

PURPOSE

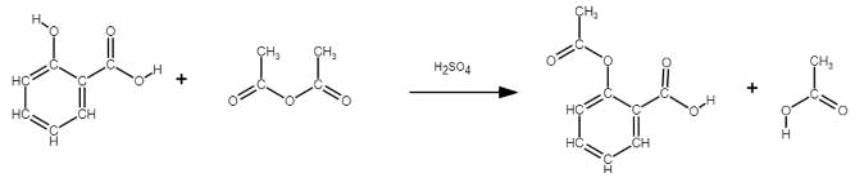
- Synthesize aspirin by the reaction of salicylic acid with acetic anhydride.
- Test the purity of the prepared sample and compare it to the commercial Aspirin.
- Analyze the product by determining its melting point, conducting thin-layer chromatography (TLC), and performing infrared spectroscopy.

INTRODUCTION

Acetylsalicylic acid, commonly known as Aspirin, reduces pain, fever, and inflammation while also lowering the risk of heart attacks and strokes. It is among the most widely used medications. Its effectiveness arises from its analgesic, antipyretic, and anti-inflammatory properties. In the small intestine, acetylsalicylic acid breaks down into salicylic acid, which is then absorbed into the bloodstream. Salicylic acid can irritate the lining of the mouth, esophagus, and stomach, potentially leading to hemorrhaging due to its phenolic and carboxylic acid groups. The Bayer company discovered that the ester of salicylic acid (acetylsalicylic acid) is less irritating than the original salicylic acid. However, it still presents side effects, such as stomach lining irritation and hemorrhaging; to reduce these effects, commercial Aspirin often includes coatings and buffering agents.

SYNTHESIS OF ASPIRIN (Acetylsalicylic Acid)

Aspirin is produced by combining salicylic acid with excess acetic anhydride (the o-acetylation of salicylic acid) and a small amount of a strong acid catalyst, which accelerates the reaction. A strong acid catalyst, such as 85% phosphoric acid or concentrated sulfuric acid, promotes the esterification reaction, resulting in the formation of white crystals. Water is then added to remove the surplus acetic anhydride. In summary, the reaction involves a carboxylic acid and an acid anhydride, which together form an ester.



Aspirin has very low solubility in cold water and is utilized for precipitation. Any unreacted salicylic acid is considered an impurity due to its similarly low solubility in cold water. Dissolved unreacted substances, such as acetic anhydride and acetic acid, remain in the water. Vacuum filtration effectively separates the crystalline aspirin from the reaction mixture, excluding unreacted salicylic acid. The synthesized aspirin should be evaluated for the presence of contaminating salicylic acid.

The purity of crude and recrystallized aspirin can be determined using iron (III) chloride ($FeCl_3$). $FeCl_3$ interacts with phenols (alcohol groups attached to aromatic rings) to produce violet-colored complexes. While salicylic acid contains the phenol functional group, aspirin does not; therefore, the greater the contamination of salicylic acid in your aspirin, the darker the color that will appear with $FeCl_3$. Pure or recrystallized aspirin should not form violet-colored complexes with iron (III) chloride ($FeCl_3$). Any contaminating salicylic acid in the product will react with $FeCl_3$. Additionally, you will measure the melting point of your product sample, perform Thin-Layer Chromatography (TLC), conduct melting point analysis, and take an infrared spectrum to analyze and test the relative purity of your sample.

SAFETY PRECAUTIONS

· Wear your safety goggles during the experiment.

· Wear gloves, particularly when working with concentrated acid or phosphoric acid.

· Exercise caution when handling phosphoric acid and acetic anhydride.

· Keep this compound concealed and avoid inhaling the vapors.

· Acetic anhydride irritates the nose and sinuses. Keep the acetic anhydride bottle capped after use.

· **The aspirin synthesized in this lab is not pure enough for internal use! Please do not ingest the aspirin!**

· Be especially careful when handling phosphoric acid and acetic anhydride.

· All waste must be placed in organic waste containers in the fume hood.

· Thoroughly clean your work area when finished and return all equipment and chemicals to their designated places.

· Make sure to wash your hands before leaving.

CHEMICALS AND EQUIPMENT NEEDED

CHEMICALS	EQUIPMENT	EQUIPMENT
Ethanol	Ring stand and ring clamp	Spatula
Salicylic acid	Thermometer	Stirring rod
Acetic anhydride	Watch glass	10-mL graduated cylinder
Iron (III) chloride solution 3%	Filter paper	600-mL beaker hot plate
85% phosphoric acid (H_3PO_4) or Concentrated Sulfuric Acid (Instructor's choice)	Büchner filtration apparatus	400-mL beaker
Ice	Dropper	125-mL Erlenmeyer flask
	Latex tubing/rubber band	Capillary tube

EXPERIMENTAL PROCEDURE

Part A: Synthesis of Aspirin

1. Measure 2.0 g of salicylic acid and place it in a 125.0 mL Erlenmeyer flask.

2. This reaction must be conducted in the fume hood. Move the flask to the fume hood and add acetic anhydride and concentrated acid inside the hood.

a) In the fume hood, measure 5.0 mL of acetic anhydride using a 10.0 mL graduated cylinder. Pour the contents of the cylinder into the flask and swirl the mixture (keep it under the hood, as it now contains acetic anhydride, which has very irritating vapors).

b) Add five drops of concentrated sulfuric acid or 85% phosphoric acid as a catalyst (your instructor will inform you which one; choose only one).

c) Utilize the provided dropper. Concentrated sulfuric acid and phosphoric acid are extremely corrosive. Add only one type of acid, as indicated by your instructor, which may differ.

3. Swirl the reactants to mix them, add a stir bar, and while stirring, gently bring the mixture to a boil on a hot plate inside a fume hood. Heat the reactant mixture for 8 to 10 minutes, stirring until all the solids have dissolved.

4. Avoid vigorous boiling or temperatures that are too high or too low. Alternatively, heat the mixture for 15.0 minutes by placing the flask in a boiling water bath. When using a boiling water bath, securely clamp the flask to keep its opening above the water line. Ensure your instructor approves your setup.

5. While the reaction heats up, take a 100.0 mL beaker, fill it with approximately 75.0 to 80.0 mL of distilled water, and place it in an ice bath.

6. Remove the flask from the hot plate or water bath, and swirl in 20.0 mL of deionized ice water. This water will react with any excess acetic anhydride, turning it into acetic acid.

7. As the flask cools, aspirin crystals will start to form. When you notice the formation of crystals, place the flask in an ice bath for 10-15 minutes. If acetylsalicylic acid does not begin to crystallize, gently scratch the walls of the flask with a glass rod.

8. Collect the crude solid aspirin product by vacuum filtration using a Buchner funnel. Rinse the product with three to four 10.0 mL portions of cold distilled water to remove any acetic acid from the crystals. Allow the crystals to air dry, and then weigh the dry crude aspirin.

9. Reserve a small quantity of crude aspirin for melting point, IR, and TLC analysis, then recrystallize the crude aspirin using ethanol. Dry the purified crystals.

10. Store the dried, purified crystals according to the instructor's guidelines for subsequent analysis and testing of the synthesized Aspirin sample's purity in part B of the experiment.

Part B: Tests

- To evaluate the purity of the prepared sample, assess the product using the tests listed below, and compare the results.
- Your instructor may require you to evaluate your product using all the tests, or they may choose a few from those available.
- i) Melting point range of the aspirin sample (crude and recrystallized); ii) pH test; iii) iron (III) chloride (FeCl3) test; iv) thin-layer chromatography (TLC); v) IR spectrum of the product (crude and recrystallized) to confirm its structure. (Follow your instructor's directions; they may ask you to perform all or some of these tests.)

i) Melting point of the Aspirin sample

a) Use a spatula to transfer a small amount of recrystallized aspirin to a weighing boat or watch glass. If the aspirin is not finely powdered, grind it with a mortar and pestle until it becomes a fine powder.

b) Place the recrystallized aspirin in a capillary tube approximately 3 mm in diameter, ensuring it settles at the bottom of the tube.

c) Insert the capillary tube into the melting point apparatus and record the melting range of the recrystallized aspirin.

d) Note: Your instructor may offer extra guidance or show how to fill the capillary tube and use the melting point apparatus.

e) The melting point range is the temperature at which the liquid first appears until the solid has fully disappeared. Document your melting point range.

f) As your instructor indicated, dispose of the capillary tube and place the excess acid into the container designated for nonhalogenated organic waste.

g) Repeat steps 1 to 5 using the crude aspirin (synthesized aspirin).

pH Test and Iron (III) chloride ($FeCl_3$) Test

a) Label five small test tubes A through E.

b) In test tube A, add a few crystals of salicylic acid.

c) In test tube B, add a few crystals of crude aspirin; in test tube C, add a few crystals of recrystallized aspirin, and in test tube D, add commercial aspirin (non-enteric coated). Please do not add any crystals to the fifth tube, as it will serve as your blank.

d) Add 1 mL of ethanol and 4.0 mL of distilled water to test tubes A-E, then mix the solution with a stirring rod. Be sure to clean the stir rod before mixing the next test tube.

ii) pH Test

(a) Touch the pH paper to the stir rod after dipping it into each solution. Ensure that you clean the stir rod before dipping it into the next test tube.

(b) Compare the color of the pH paper to the chart, then record the pH values of the solutions in the data table.

(c) Do not discard the solutions; we will use them to test with iron (III) chloride (FeCl3).

iii) Iron (III) chloride ($FeCl_3$) Test

a) Add five drops of 1% $FeCl_3$ solution to test tubes A through E.

b) Mix the solution with a stirring rod. Ensure that you clean the stir rod before mixing the next test tube.

c) Document your observations in the data table and respond to the questions.

iv) Thin Layer Chromatography (TLC)

Procedure to Analyze Aspirin Using TLC

Preparation of TLC Plate:

-Obtain a TLC plate coated with a thin layer of silica gel.

-Draw a light pencil line about 1 cm from the bottom of the plate. This will be the baseline where you will spot your samples.

Preparation of Samples:

-To create a concentrated solution, dissolve a small amount of aspirin in an appropriate solvent, such as ethanol.

-Prepare a reference solution of pure salicylic acid and acetylsalicylic acid (aspirin) in the same solvent.

Spotting the TLC Plate:

Using a capillary tube or a TLC spotter, apply small, well-separated spots of the aspirin salicylic acid solution and pure aspirin solution along the baseline of the TLC solution plate.

Developing the TLC Plate:

-Place the TLC plate in a developing chamber containing a small amount of solvent, such as a mixture of ethyl acetate and hexane. Keep the solvent level below the baseline.

-Cover the chamber and allow the solvent to rise on the plate through capillary action. This process is referred to as developing the plate.

-Once the solvent front is approximately 1 cm from the top of the plate, remove the plate and mark the solvent front with a pencil.

Visualization:

-Let the plate dry.

-Visualize the spots with UV light or in an iodine chamber. The spots will appear as dark marks against a fluorescent background when seen under UV light.

Analysis of TLC Results

Calculate Rf Values:

-Determine the distance each spot moves from the baseline. Also, determine the distance the solvent front moves from the baseline.

-Take the image/picture of your chromatograph paper once you have developed it to include with your report.

-Calculate the R_f value for each spot using the formula:

R_f = Distance traveled by the compound/Distance traveled by the solvent front

Compare R_f Values:

Compare the R_f values of the spots from the aspirin sample with those of the reference compounds (salicylic acid and pure aspirin). Pure aspirin should display a specific Rf value, and any additional spots with different R_f values may indicate impurities or unreacted starting materials.

Interpretation:

If the aspirin sample shows a single spot with the same R_f value as the pure aspirin reference, it indicates high purity. Multiple spots or spots with different R_f values suggest the presence of impurities or incomplete reactions. By following this procedure, you can effectively analyze the purity and composition of aspirin using thin-layer chromatography (TLC).

v) IR Spectrum

Procedure for obtaining an IR spectrum of aspirin to confirm its structure.

a) Turn on the FT-IR spectrometer and allow it to warm up.

b) Ensure the instrument is calibrated using standard reference material.

c) Grind a small amount of recrystallized aspirin into a fine powder. Mix it with potassium bromide (KBr) in a 1:100 ratio (aspirin: KBr). Press the mixture into a thin, transparent pellet using a pellet press.

d) Place the prepared sample pellet in the sample holder of the IR spectrometer.

e) Set the spectrometer to scan the sample over the desired wavelength range (typically 4000-400 cm^{-1}).

f) Start the scan and record the spectrum. The instrument will measure the sample's transmittance at each wavelength.

g) Analyze the obtained IR spectrum. Identify the characteristic peaks corresponding to different functional groups in the aspirin molecule.

h) Confirming the Structure of Aspirin

i) To confirm the structure of aspirin using its IR spectrum, look for the characteristic peaks and record them in the data table.

PRE-LAB QUESTIONS Name _____

1. Write the balanced equation for the reaction.

2. Why is an acid catalyst used in this reaction?

3. What is the theoretical yield of aspirin for this reaction?

DATA AND OBSERVATIONS

Part A: Synthesis of Aspirin

Mass of Salicylic Acid:

Volume of Acetic Anhydride:

Theoretical yield of aspirin (show your calculations):

Mass of Crude Aspirin:

Mass of Recrystallized Aspirin:

Percent Yield (show your work):

Part B: Tests

i) Melting Point of Samples (Crude and recrystallized):

Melting Point of a Crude Aspirin Sample:

Melting Point of the Recrystallized Aspirin Sample:

ii) pH Test

Test Tube No.	Sample	pH Test
Test Tube A	Containing Salicylic Acid Crystals	
Test Tube B	Containing Crude Aspirin	
Test Tube C	Containing Recrystallized Aspirin Crystals	
Test Tube D	Containing Commercial Aspirin	
Test Tube E	Blank	

iii) Test with Iron (III) chloride ($FeCl_3$):

Test Tube No.	Sample	Observations with $FeCl_3$
Test Tube A	Containing Salicylic Acid Crystals	
Test Tube B	Containing Crude Aspirin	
Test Tube C	Containing Recrystallized Aspirin Crystals	

Test Tube D	Containing Commercial Aspirin	
Test Tube E	Blank	

iv) Thin Layer Chromatography (TLC):

Test Tube No.	Sample	Observation and R_f Value (show your Calculation)
Test Tube A	Containing Salicylic Acid Crystals	
Test Tube B	Containing Crude Aspirin	
Test Tube C	Containing Recrystallized Aspirin Crystals	
Test Tube D	Containing Commercial Aspirin	
Test Tube E	Blank	

Calculations:

v) IR Spectrum:

Obtain an IR spectrum of the product. Attach an image of your IR spectrum along with your remote and identify the distinguishing peaks (both crude and recrystallized) to confirm its structure. Your instructor might perform this, but it's not guaranteed. Analyze the IR spectrum: what are the key absorption bands that confirm aspirin's structure?

POST-LAB QUESTIONS

1) Discuss any possible sources of experimental error in the synthesis and suggest potential ways to increase the percent yield.

2) Explain the melting point data. Does it align with the expected range for pure aspirin?

3. Why is the aspirin washed with cold water?

4. According to your results from the ferric chloride test, what can you say about the purity of your aspirin? Be as specific as possible.

References:

1.) **Figure 1** adapted from https://chem.libretexts.org/Courses/Westfield_State_University/Z_GenChem2_lab/GenChem2_lab_Spring_2023/Synthesis_and_Recrystallization_of_Aspirin

LAB 7 - IDENTIFICATION OF AN UNKNOWN COMPOUND

PURPOSE

- The purpose of this experiment is to:
- Objective 2

PURPOSE

• Determine the boiling point and density of an unknown organic compound.

• Test the pH and solubility of an unknown organic compound.

• Perform chemical tests on an unknown organic compound.

• Identify an unknown organic compound based on chemical tests and physical properties.

INTRODUCTION

Over the past several experiments, you explored physical properties, structure, reactions, and chemical tests of organic compounds containing various functional groups. Now, you will use these methods to identify an unknown compound. Each lab group will be given a unique unknown. You will first test the pH of the unknown and then determine whether it is soluble in various solvents. Next, you will determine the boiling point and density of your compound. Finally, you will perform six chemical tests on the unknown. Using your results and various molecular formulas, you will propose a structure for your unknown compound.

SAFETY PRECAUTIONS

1) Wear chemical splash goggles while working on this experiment.

2) Handle all reagents and perform all parts of this experiment under a working fume hood.

3) Gloves are provided, and we encourage you to wear them.

4) Be careful while handling hot glassware after running the simple distillation.

5) Dispose of all waste in the designated container and clean all equipment and your work area when you are finished.

CHEMICALS AND EQUIPMENT NEEDED

CHEMICALS	CHEMICALS		
Unknown	Bromine test solution	10 small or medium test tubes	Hot plate
Hexane	Potassium permanganate test solution	Test tube holder	400. mL beaker
Ethanol	Chromic acid test solution	Simple distillation apparatus and equipment	Thermometer
Methylene chloride	Ferric chloride test solution	10 mL graduated cylinder	pH paper
Iodoform test solution	Benedict's test solution	Disposable droppers	Stirring rod
Water	10% NaOH	Halogenated organic waste containers	Non-halogenated organic waste containers

Note: Refer to previous labs for concentration and preparation of test solutions. (The whole lab shares test solutions)

EXPERIMENTAL PROCEDURE

Part A: pH and Solubility of the Unknown

1) Gather four test tubes and a test tube rack. Add 10 drops of your unknown to each test. Dip a stirring rod into **one** test tube and touch the tip of the rod to a strip of pH paper. Record the pH of your unknown.

2) Under the fume hood, add 10 drops of water, hexane, ethanol, and methylene chloride (using only one type of solvent in each test tube) to the four test tubes with the unknown from step one. Briefly shake each test tube and assess the solubility of the unknown substance in the provided solvent. Dispose of the contents of the test tubes in the halogenated waste container. Clean all equipment before proceeding to the next step.

Part B: Boiling Point of the Unknown

1) Add 10 mL of your unknown to a round-bottom flask and set up a simple distillation apparatus. After your instructor approves the setup, use simple distillation to determine the boiling point of your unknown. Remember, never distill to dryness.

2) Disassemble the simple distillation apparatus and clean all equipment before proceeding to the next step.

3) Dispose of the unknown, as directed by your instructor.

Part C: Density of the Unknown.

1) Determine the mass of an empty 10.0 mL graduated cylinder.

2) Add 5.0 mL to 10.0 mL of the unknown to the graduated cylinder and record the correct volume.

3) Record the combined mass of the unknown and graduated cylinder.

4) Subtract the mass of the empty graduated cylinder from the combined mass of the unknown and cylinder to obtain the mass of the unknown.

5) Using the unknown's mass and the unknown's volume (from step 2), calculate the density of the unknown, rounding your answer to the appropriate number of significant digits.

6) Repeat steps 1-5 two more times using a different amount of the unknown.

7) Calculate the average density of the unknown by adding up the three density values and dividing the sum by three.

8) Dispose of the unknown substance as directed by your instructor and clean all equipment before proceeding to the next step.

Part D: Chemical Tests (Remember to work under the fume hood)

1) Obtain six test tubes and add 10 drops of your unknown to each.

2) To test tube 1, add 3-5 drops of the bromine test reagent. Gently swirl the contents of the test tube and record your observations.

3) To test tube 2, add 3-5 drops of the potassium permanganate test reagent. Gently swirl the contents of the test tube and record your observations.

4) To test tube 3, add 3-5 drops of the chromic acid test reagent. Gently swirl the contents of the test tube and record your observations.

5) To test tube 4, add 3-5 drops of the ferric chloride test reagent. Gently swirl the contents of the test tube and record your observations.

6) To test tube 5, add 2 mL of water and 10 drops of 10% NaOH. Obtain a hot plate and a 400 mL beaker, half-filled with water. Place the test tube in the beaker with water and set the beaker on a hot plate. Warm the water bath to 55 °C. While keeping the test tube in the water bath, add 20 drops of the iodoform reagent. Mix using a glass stirring rod. After 2 minutes, record your observations.

7) To test tube 6, add 2 mL of Benedict's reagent. Mix the contents using a glass stirring rod, and then place the test tubes in a 400 mL beaker that is half-filled with water. Heat it to a boil. Once the water starts to boil, let the test tube sit in the water bath for 5 minutes. Record your observations.

8) Discard the contents of each test tube as directed by your instructor.

PRE-LAB QUESTIONS Name _____

1) Briefly describe the purpose of the following chemical tests. Explain what both positive and negative test results would look like.

Bromine Test:

Potassium Permanganate Test:

Chromic Acid Test:

Ferric Chloride Test:

Benedict’s Test:

Iodoform Test:

2) Which of the above chemical tests can distinguish between the following pairs of compounds?

Part A: 2-pentanone and 3-pentanone

Part B: Cyclohexane and Cyclohexane

Part C: 2-butanol and 2-methyl-2-butanol

Part D: Toluene and Phenol

Part E: Hexanal and 2-Hexanone

3) Draw three constitutional isomers with molecular formula C_3H_6O. Identify any non-alkane functional groups present in the compounds drawn. Explain which chemical tests can be used to identify the compounds drawn.

DATA AND OBSERVATIONS

Name _____Lab Partner(s) _____

Unknown letter or number =

Part A: pH and Solubility of the Unknown

pH of unknown =

Solubility of the Unknown

Solvent	Yes/No
Water	
Hexane	
Ethanol	
Methylene chloride	

What do the results of pH and solubility tests tell you about your unknown?

Part B: Boiling Point of the Unknown

Boiling point of unknown (from simple distillation) =

Part C: Density of the Unknown

	Trial 1	Trial 2	Trial 3
Mass of an empty 10 mL graduated cylinder			
Mass of the graduated cylinder and the unknown			
Mass of unknown			
Volume of unknown			
Density of unknown			

Show calculations for the density of each trial and the average density.

Part D: Chemical Tests (Remember to Work Under the Fume Hood)

Test	Functional Group Tested for	Observations	Positive or Negative Test
Bromine Test			
Potassium Permanganate Test			
Chromic Acid Test			
Ferric Chloride Test			
Benedict's Test			
Iodoform Test			

Briefly summarize what the chemical tests infer about your unknown.

POST-LAB QUESTIONS

1) Your unknown has one of the following molecular formulas:

C_3H_8O, C_4H_8O, C_6H_6O, $C_3H_6O_2$, $C_4H_{10}O$

Based on the results from part D (chemical tests), propose a structure for your unknown. Be sure to explain your decision in detail.

2) Consider the structure drawn in post-lab question 1. Perform a literature search to determine the compound's actual density and boiling point. Does this information agree with your unknown choice? Explain.

3) Does the result of the pH test support your unknown decision? Explain.

4) Based on answers provided for post lab questions 1-3, how confident are you in your decision about the unknown's identity? Explain.

5) Can the unknown positively be identified based solely on the results of this experiment? If not, what other tests would need to be performed?

LAB 8 - MOLECULAR MODELS (Stereochemistry)

Stereochemistry – 3D Printing

PURPOSE

- The purpose of this experiment is to:
- Objective 2

PURPOSE

- Use drawings to try to determine if pairs of isomers are constitutional isomers, geometric isomers, or stereoisomers.
- Also, use the drawings to determine if the stereoisomers are enantiomers or diastereomers.
- Use printed 3D models to confirm your predictions.

INTRODUCTION

The structure of molecules can differ, even if they share the same molecular formulas. Molecules with identical formulas but different arrangements of atoms are called isomers. The way atoms are connected and their spatial positions in three dimensions often cause isomers to have very different physical, chemical, and biological properties. In this lab, you will investigate various types of isomers by creating computer models to visualize their structures. Additionally, you will build and print 3D models to help you compare the molecular structures of some isomers.

PRE-LAB QUESTIONS Name _____

1) Define the following terms.

Constitutional isomers

Geometric isomers

Stereoisomers

Enantiomers

Diastereomers

Meso compound

SAFETY PRECAUTIONS

Be sure to follow all your instructor's instructions regarding properly using your 3D printer. Parts of the printer can be hot, and the mechanical moving parts can cause injury unless proper care is taken.

EQUIPMENT NEEDED

- 3D printer
- Computer with the 3D chemical structure drawing and 3D printing software installed (described below)

EXPERIMENTAL PROCEDURE

The following programs will be used for the instructions for this lab. These are all available free of charge.

- Jmol[1] to create a 3D computer model of a molecule and export it as an STL file.
- Ultimaker CURA[2] to slice the STL file and create the G-code needed by the 3D printer.
- More details on how to attain and use these programs are given in the appendices at the end of this document.

Part I – Constitutional Isomers

1. For each of the following molecular formulas, determine two constitutional isomers.

2. Sketch each of the isomers and give the correct name for the compound.

3. Use Jmol to make a 3D computer model of each of the isomers. Use File→Get MOL, enter the name of the molecule, and press OK. The window should display a 3D model of the molecule. See the appendix for more details.

4. Select Tools→2-D Editor to open a window showing a drawing of your molecule. Compare the program's drawing with your drawing. How are they different?

5. Repeat the above procedure for each of the chemical formulas.

6. Choose one of the pairs of constitutional isomers. Export each isomer as an STL file and print it. Use the 3D printed models to compare the structures and explain why these are constitutional isomers.

Molecular Formula	Constitutional Isomer #1	Constitutional Isomer #2	Observations and comments
C_4H_{10}	Name: Sketch:	Name: Sketch:	
C_3H_8O	Name: Sketch:	Name: Sketch:	

C_2H_6O	Name: Sketch:	Name: Sketch:	

Part II –Geometric Isomers

1. What are the complete names of the two isomers of 2-butene?

2. Sketch each of these isomers.

3. Use the complete name of each of the isomers to get the MOL files into Jmol and observe the 3D models. Briefly describe the differences between the two isomers.

4. Transfer the 3D models to the 2D window and compare the drawings with yours.

5. Repeat the above exercise with 1,2-dichlorocyclohexane.

6. Choose either the pair of 2-butene isomers or 1,2-dichlorocyclohexane isomers, export the STL files, and make 3D prints of the pair. What are your observations after looking at the 3D printed models?

Molecule and Formula	Geometric Isomer #1	Geometric Isomer #2	Observations and comments
2-butene Molecular Formula:	Complete Name: Sketch:	Complete Name: Sketch:	
1,2-dichlorocyclohexane Molecular Formula:	Complete Name: Sketch:	Complete Name: Sketch:	

Part III –Stereoisomers

A – Molecules with one chiral center.

Alanine is one of the amino acids used to build proteins, which are essential for biological organisms.

1. The molecular structure of alanine has a central carbon connected to hydrogen and three different groups, an amine group, a carboxylic acid group, and a methyl group. Sketch the two isomers of alanine indicating the 3D structure and give the complete name for each of the isomers.

2. Use the complete name for each to load the structures into Jmol using Get MOL.

3. Transfer each of the 3D models to the 2D drawing window and compare the drawing with your sketches.

4. Export each of the isomers as an STL file and 3D print them. Compare the models of the two isomers and record your observations.

Molecule and Formula	Stereoisomer #1	Stereoisomer #2	Observations and comments
alanine Molecular Formula:	Complete Name: Sketch:	Complete Name: Sketch:	

B – Molecules with more than one chiral center.

1. 2,3-butanediol has two chiral centers and will have three isomers. Why aren't there four isomers?

2. Sketch each of the three isomers and give their complete names.

3. Use the complete name for each to load the structures into Jmol using Get MOL.

4. Transfer each of the 3D models to the 2D drawing window and compare the drawing with your sketches.

5. Export each of the isomers as an STL file and 3D print them. Compare the models of the three isomers and record your observations.

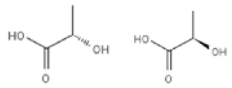
2,3-butanediol Molecular Formula:

	Complete Name	Sketch	Observations and comments
Stereoisomer #1			
Stereoisomer #2			
Stereoisomer #3			

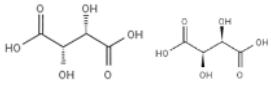
POST-LAB QUESTIONS

Determine whether the following pairs of compounds are the same, different, resonance structures, constitutional isomers, enantiomers, or diastereomers

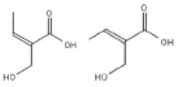
1.



2.



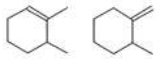
3.



4.

5.

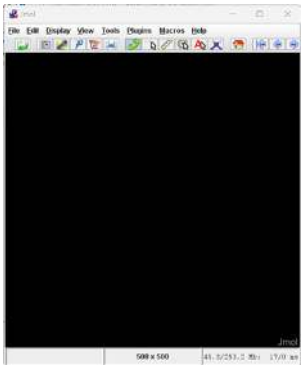
6.



7.

Appendix A – Creating a 3D computer model.

1. Open Jmol by clicking on the Jmol.jar file. This will open the 3D window of the application.



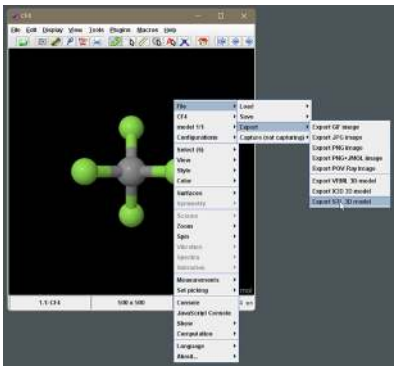
2. Select Get MOL from the File menu. You can enter each of the molecules in the Data Table as a formula (e.g. CF4) or by name (e.g. tetrafluoromethane). Sometimes the formula will not work. If you have difficulties, ask your instructor for assistance.

3. When you press enter or click on OK, a 3D model of the molecule will be displayed in the window.

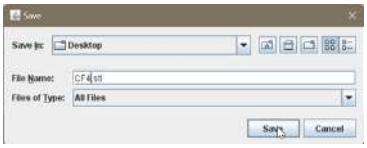
Appendix B – Exporting and printing the 3D model

1. Export an STL file.

a. Right click on the Jmol display window and open successive menus to select File►Export►Export STL 3D model.



b. Choose a folder and give your file a name with the .stl extension.

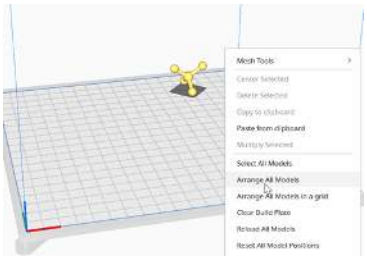


2. Slice the STL file in CURA.

a. Open CURA then click on the open file icon.

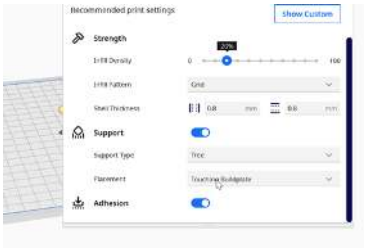
b. Browse to where you saved the STL file and open it.

c. The model will be displayed on the printing surface. Right-click on the printing surface and select “Arrange all models” to move your model to the center of the surface.

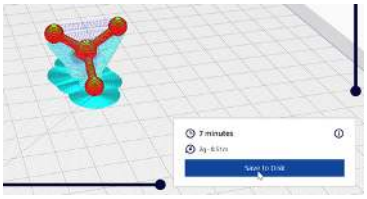


d. You can do various manipulations to the 3D model, such as scaling it and tilting it, but usually, the default will work well. Ask your instructor for help if you are unsure.

e. Make sure that you include supports for your print. The Tree type supports work well with these models and usually selecting “Touching Buildplate” will be sufficient. If you have trouble with the print, try “Everywhere” for your support placement and as your instructor for assistance.



f. Click on the SLICE button. When the slicing is done, the program will display the amount of time needed to print the model. Click on the “Preview” button to see what the print and supports will look like. Get your instructor's approval before continuing.



g. Export the G-code by clicking on the Save to Disk button.

h. Copy the .G-code file that you just saved to an appropriate removable card as per your instructor.

3. Print the model.

a. Insert the memory card in your printer and use the printer’s control knobs to select your model. The printer will start warming up the nozzle and should begin printing within a few minutes.

b. Once the printing is complete, carefully remove the model and its supports from the printing surface.

c. The supports should be carefully removed using needle-nose pliers or other tools provided by your instructor.

Appendix C – Required Programs

· A program to create and save a stereolithography file, known as an STL file. This is a 3D solid model that can be “sliced” and printed.

· Jmol (requires java): https://sourceforge.net/projects/jmol/files/Jmol/. The download consists of a zip file containing all the files needed to run the program. Simply extract the files to a folder of your choice. The program is started by clicking the Jmol.jar file.

· Note: If Java is not available on your computer, an online version can be accessed on the Hack-a-mol website (https://chemapps.stolaf.edu/jmol/jsmol/hackamol.htm).

· A slicer program to prepare the STL file for printing and generate the G-code that is needed by the printer to make the print.

· Your printer may come with a slicer program, in which case it should be used.

· One of the best is Ultimaker CURA (https://ultimaker.com/software/ultimaker-cura/) The program must be installed on your computer, so some administrative rights are required.

· There are also several free sites that can generate G-code from an STL file online. An example is Creality Cloud https://www.crealitycloud.com/blog/tutorials/how-to-convert-stl-to-gcode. Many others can be found by searching the web.

[1] Jmol: an open-source Java viewer for chemical structures in 3D. http://www.jmol.org/

[2] https://ultimaker.com/software/ultimaker-cura/

CHAPTER OVERVIEW

LAB 9 - TESTS FOR CARBOHYDRATES

Learning Objectives

- The purpose of this experiment is to:
- Objective 2

LAB 10 - SYNTHESIS OF SOAP

PURPOSEearning Objectives

The purpose of this experiment is to:

- Objective 1
- Objective 2

PURPOSE

• Prepare soap via the saponification reaction.

• Observe the properties of soap

INTRODUCTION

Soap, a salt of fatty acids, is the most common cleaning agent in modern society. Its rich history, dating back thousands of years, can be traced to ancient Babylon around 2800 BCE. This long-standing history underscores the enduring significance of soap in human civilization, connecting us to our ancestors, who also appreciated its cleansing properties.

Soap is a salt derived from a fatty acid. More specifically, it is a sodium or potassium salt of a long-chain carboxylic acid. Soaps exhibit a characteristic amphipathic or amphiphilic structure, which means they possess both hydrophilic (water-loving) and hydrophobic (water-repelling) components:

- The hydrophilic part (head) has an ionic charge and typically features a carboxylate group (carboxylate anion; $-COO^-$), which is soluble in water.
- The hydrophobic part (tail) consists of a long hydrocarbon chain and is soluble in oil.

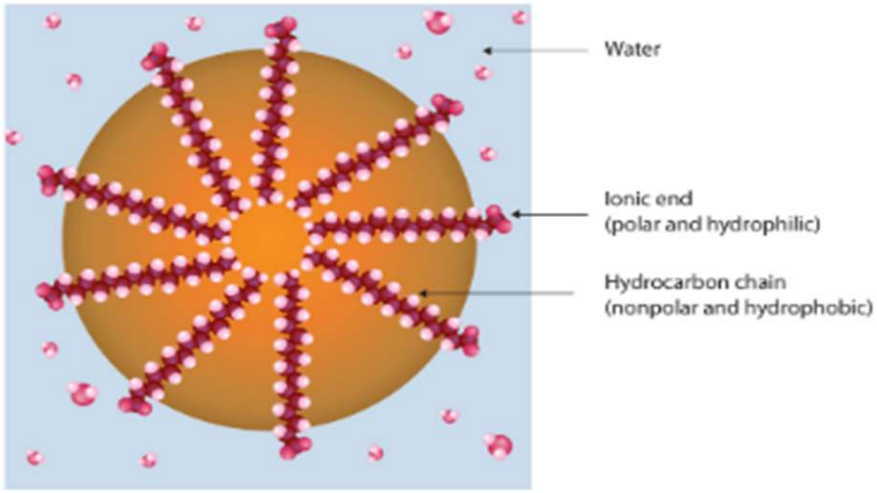


Figure 1: Spherical clusters of soap-like molecules aggregate in aqueous solution. The ionic heads of these molecules are positioned on the outside, where they are solvated by water, while the organic tails cluster together on the inside of the micelle.

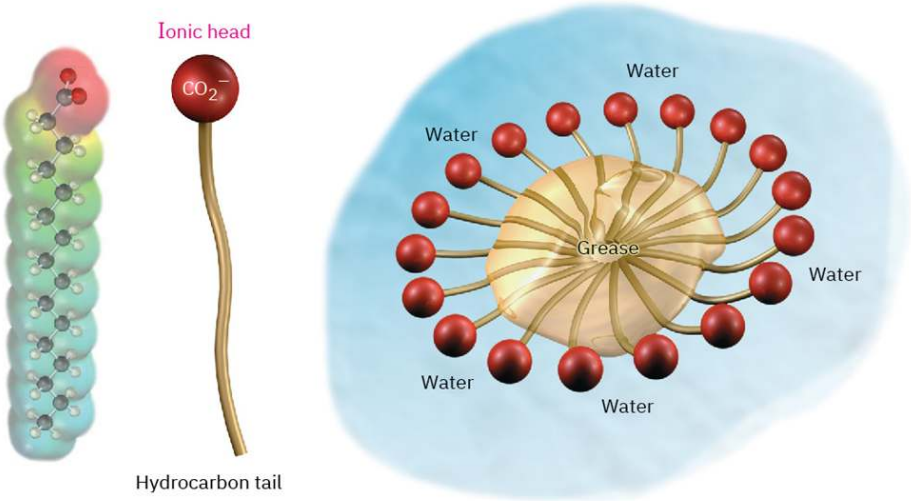


Figure 2: A soap micelle solubilizing a grease particle in water. An electrostatic potential map of a fatty acid carboxylate illustrates the location of the negative charge in the head group.

A long-chain soap molecule has an ionic carboxylate end (hydrophilic) that attracts water, while its long hydrocarbon part is nonpolar and hydrophobic. This structure enables soap to form micelles, which emulsify fats and oils for effective cleaning.

Soap molecules surround an oil droplet or grease with their nonpolar tails embedded in the oil and their charged "head" groups facing outward toward the water. If the oil droplets are small enough and there are sufficient soap molecules to cover them, they become dispersed in the water and can be easily washed away. Therefore, using plenty of soap, hot water, and agitation can help clean greasy dishes. Hot water dissolves solid fats, and agitation helps break them into smaller droplets.

Soap production is one of the oldest chemical reactions practiced by humans. It occurs through saponification, a process in which fats or oils (triglycerides) react with a strong base, typically sodium hydroxide (lye) or potassium hydroxide. This reaction produces glycerol and fatty acid salts (soap).

Saponification Reaction

The general equation for saponification is: typical **Fat/Oil + Base → Glycerol + Soap.**

More specifically:

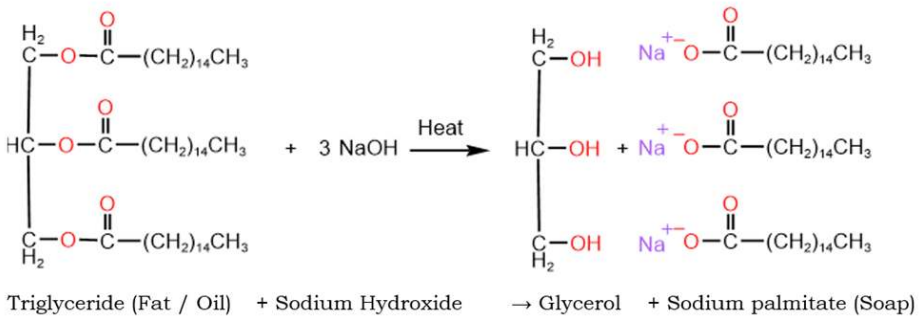


Figure 3: Saponification Reaction[3]

Types of Soap

The choice of base and fatty acid influences the soap's properties:

• Sodium salts of fatty acids produce hard soaps

• Potassium salts yield soft soaps

• Common types of soap include sodium stearate, oleate, and linoleate.

• Saturated fatty acid salts produce rigid soaps, whereas polyunsaturated fatty acids lead to softer soaps.

The additional ingredients suggested below are often incorporated to enhance the properties of soap.

• Perfumes for scent

• Dyes for color

• Sand or Pumice in scouring soaps for abrasive action

• Air bubbles to create floating soaps

In this experiment, you will engage in the time-honored process of soap-making, testing, and comparative analysis with commercial soap and detergent. This comprehensive experiment will provide hands-on experience with soap chemistry, from production to performance evaluation, deepening your understanding of this essential cleaning agent.

Properties of Soap and Detergent:

- Amphipathic structure: one end is hydrophilic (water-loving) and the other is hydrophobic (water-repelling)
- Emulsifying ability: can mix oil and water
- Surfactant properties: reduces the surface tension of water
- Cleansing action: lifts dirt and oil from surfaces
- Lathering ability: forms foam in water
- pH: Typically, alkaline (pH > 7)

SAFETY PRECAUTIONS

1) Always wear chemical splash goggles, closed-toe shoes

2) You are encouraged to wear gloves while working on this experiment.

3) Work in a well-ventilated area or under the hood

4) Exercise caution when dispensing the 9.0 M NaOH. It is highly corrosive.

5) Never add water to NaOH; always add NaOH to water to avoid violent reactions.

6) If skin comes into contact with NaOH or other chemicals, rinse immediately with plenty of water for at least fifteen minutes and inform your instructor.

7) Dispose of all waste in the designated containers, as instructed by your instructor.

8) Thoroughly clean your workspace after finishing and return all equipment and chemicals to their proper locations.

EQUIPMENT AND CHEMICALS NEEDED

EQUIPMENT	EQUIPMENT	EQUIPMENT	CHEMICALS	CHEMICALS
Safety equipment (goggles, gloves)	Stirring rod	Test tubes	Coconut oil (or other vegetable oil)	stearic acid
Thermometer	Beakers (250 mL, 100 mL)	Test tube rack	Sodium hydroxide (NaOH)	0.5 M calcium chloride solution
Hot plate with stirrer	Mold for soap	Glass pipets and pipette bulbs	Distilled water	0.5 M Iron (III) chloride solution
Magnetic stir bar	pH paper	10 mL graduate cylinder	Assorted fragrances (optional)	0.5 M Magnesium chloride solution

EXPERIMENTAL PROCEDURE

A. Synthesis of Soap

1. **Soap Synthesis**: Measure out 10.0 mL of the provided oil using a 10.0 mL transfer pipette. Transfer 25.0 mL from a graduated cylinder into a 150.0 mL beaker. This is typically one of the following: olive oil, vegetable oil, or coconut oil.

2. Use the glass stirring rod to mix the ingredients. Pour 3.0 mL of 20% sodium hydroxide solution into the beaker. Stir for 20 to 45 minutes; you may alternate with your lab partner. The mixture will gradually become smoother and more opaque, eventually thickening to a pudding-like consistency.

3. After receiving approval from your instructor, add 2-3 drops of your preferred food coloring. Stir thoroughly.

4. Add a dash (about 1/8 teaspoon or the tip of a spatula) of stearic acid. This will help solidify the liquid soap. Stir it well. Pour the mixture into the selected mold shape. Clearly label it with your name and lab section number.

5. The process will continue after the soap is poured into the mold (small beaker). It will heat up and liquefy again, then cool slowly, harden, and dry. Therefore, the soap must remain undisturbed for at least 12 hours.

B. Properties of Soap and Comparative Analysis

a) pH Test:

- Dissolve a small amount of soap in water and test with pH paper.
- Compare with commercial soap and detergent, repeating the above step.

b) Foam Test:

- Shake a soap solution in a test tube and observe the formation of foam.
- Compare with commercial soap and detergent, repeating the above step.

c) Reaction with Oil:

- Add a drop of oil to water in one test tube and soap solution in another.
- Shake both and observe the difference in mixing.
- Compare with commercial soap and detergent, repeating the above steps.

d) Hard Water Test:

- Prepare a soap solution in distilled and hard water (containing Ca^{2+} or Mg^{2+} ions).
- Compare lather formation in both.
- Compare with commercial soap and detergent, repeating the above steps.

e) Emulsifying Properties:

- Mix equal parts of oil and water in a test tube.
- Add soap and shake vigorously.
- Observe the formation of an emulsion.
- Compare with commercial soap and detergent, repeating the above steps.

PRE-LAB QUESTIONS Name _____

1. What are soaps? What is saponification?

2. What is the difference between soap and detergent? What determines whether the final soap product is solid or liquid?

3. Why is NaOH added to water and not vice versa?

4. Identify the primary safety hazard in this experiment.

5. Why is sodium chloride solution used after the reaction?

DATA AND OBSERVATIONS

Name _____Lab Partner(s) _____

Part A: Synthesis of Soap

1. Identity of oil/ fat used:

2. Volume of oil/ fat used:

3. Volume of NaOH used:

4. Fragrance (if any) used:

5. Coloring (if any) used:

Describe the color, texture, and appearance of synthesized soap. Does it smell like any soap you've used?

Part B: Properties of Soaps and Detergents

Tests	Soap	Commercial Soap	Commercial Detergent
pH Test			
Foam Test			
Reaction with Oil			
Hard Water Test			
Emulsifying Properties			

POST-LAB QUESTIONS

1. What role does the amphipathic structure of soap play in its cleaning action? Why does soap produce less lather in hard water?

2. Write the complete ionic equation for the saponification reaction. In this chemical process used to synthesize soap, what is the ester, and what is the base? Which bases are commonly used for this reaction?

3. Describe what happens when soap can emulsify fats and oils. How does the emulsifying property of soap help in cleaning?

4. Suggest ways to modify this experiment to make different types of soap.

5. During the lab section, why did the saponification reaction require a long period of stirring? Why do we age the soap after it has been made?

6. Do you think the type of fat will make a difference in the product? Why or why not?

References:

1. https://saylordotorg.github.io/text_...-and-oils.html

2. 27.2 Soap - Organic Chemistry | OpenStax (https://openstax.org/books/organic-chemistry/pages/27-2-soap)

3. 6.3: Glycerolipids - Chemistry LibreTexts (https://chem.libretexts.org/Bookshelves/Introductory_Chemistry/Introduction_to_Organic_and_Biochemistry_(Malik)/06%3A_Lipids/6.03%3A_Glycerolipids)

LAB 11 - SYNTHESIS OF HAND LOTION

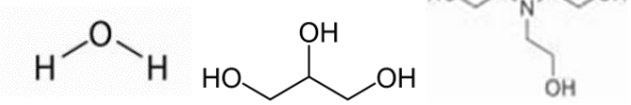
PURPOSE

Synthesize a simple hand lotion and understand the chemistry behind emulsions and their applications in cosmetic products.

INTRODUCTION

Hand lotion is an emulsion, which is a mixture of two immiscible liquids (like oil and water) stabilized by an emulsifying agent. The oil component provides moisturizing benefits, while the water component allows for easy spreading and absorption. Emulsifying agents, such as stearic acid and triethanolamine, help to keep the oil and water phases combined.

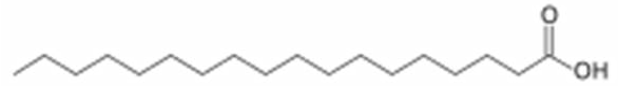
The hand lotion formulated in this lab contains three polar ingredients: water, triethanolamine, and glycerol.



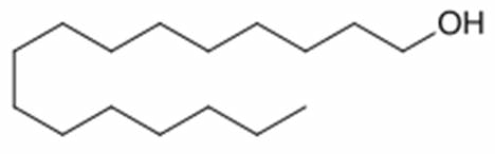
Figures: I. Water **II.** Glycerol **III.** Triethanolamine

And four non-polar ingredients: stearic acid, Cetyl alcohol, lanolin, and mineral oil.

Stearic Acid: CH_3-$(CH_2)_{16}$-COOH:



Cetyl alcohol: CH_3-$(CH_2)_{15}$-OH:



Lanolin is a complex mixture of waxes. It primarily consists of long-chain waxy esters (approximately 97% by weight), with the remainder comprising lanolin alcohols, lanolin acids, and lanolin hydrocarbons.

Mineral oil: Mixture of higher alkanes from a mineral source, particularly a distillate of petroleum, distinct from edible vegetable oils.

Understanding Hand Lotion Formulation

Before we delve into the procedure, it's essential to understand the basic components of a hand lotion:

			Common Examples
1	Emulsifiers	Help to combine oil and water	**Cetyl Alcohol:** While primarily an emollient, it can also act as an emulsifier under certain conditions. **Stearic Acid:** When neutralized with an alkali (like triethanolamine), it forms a soap-based emulsifier.
2	Humectants	Attract and retain moisture.	Glycerin, hyaluronic acid, sorbitol.
3	Emollients	Soften and smooth the skin.	Mineral oil, lanolin, shea butter, and cocoa butter.
4	Thickening agents	Increase the viscosity of the lotion.	Carbomer, xanthan gum, cetearyl alcohol.
5	**Preservatives**	Prevent microbial growth.	Phenoxyethanol, benzyl alcohol, parabens (controversial), sorbic acid.
6	**Fragrance and color**	Aesthetic purposes	

In this lab, we will make a hand lotion using polar and nonpolar ingredients. We will examine the concept of emulsions, the function of each ingredient, and the process of creating a stable cosmetic product. This is a basic formulation. It is important to perform thorough testing and stability studies before applying any product to the skin.

SAFETY PRECAUTIONS

1) Always wear goggles that protect against chemical splashes when conducting this experiment.

2) It's advised to wear gloves during the experiment.

3) Exercise caution when dealing with hot equipment and liquids.

4) Avoid inhaling vapors from essential oils or preservatives. Perform this experiment under a fume hood.

5) If chemicals come into contact with skin or eyes, rinse thoroughly with water.

6) Dispose of all waste in the designated containers, as instructed by your instructor.

7) When you're finished, thoroughly clean your glassware and workspace, then return all equipment and chemicals to their proper places.

8) Be sure to wash your hands before you leave.

EQUIPMENT AND CHEMICALS NEEDED

EQUIPMENT	EQUIPMENT	EQUIPMENT	CHEMICALS
250 mL beaker	Magnetic stir bar	Triethanolamine (2.0 mL)	Distilled water (50.0 mL)
100 mL beaker	Glass stirring rod	Cetyl alcohol (1.0 g)	Essential oil for fragrance (optional, 5-10 drops)
Hot plate with magnetic stirrer	Digital scale	Lanolin (anhydrous) (2.0 g)	Preservative (optional, e.g., phenoxyethanol, 0.5 mL)
Thermometer	Graduated cylinder	Glycerol (Glycerin) (2.0 mL)	Students bring their favorite lotion for comparison during tests.
Pipettes	Small containers for the finished product	Stearic acid (3.0 g)	-
pH strips	-	Mineral oil (10.0 mL)	-

EXPERIMENTAL PROCEDURE

Part A: Synthesis of Lotion

1. **Water Phase:** In a clean 150.0 mL beaker, combine 50.0 mL of distilled water and 3.0 mL of glycerol, also called glycerin. Place the beaker on a hot plate with a magnetic stirrer and gently stir the water phase, heating the mixture to 70.0°C.

2. **Oil Phase:** In a separate, clean 100.0 mL beaker, combine 10.0 mL of mineral oil, 3.0 g of stearic acid, 1.0 g of cetyl alcohol, and 2.0 g of anhydrous lanolin.

3. Heat the second beaker to approximately 70.0°C on a hot plate, stirring gently using a magnetic stirrer, until all the compounds have melted and formed a homogeneous liquid—oil *Phase*.

4. **Emulsification:** Once both mixtures have reached 70.0°C and have formed a homogeneous phase, slowly pour the oil phase (beaker 2) into the water phase (beaker 1) while stirring constantly.

5. Continue heating and stirring the mixture vigorously for about 5 minutes (for a finer mixture).

6. Turn off the hot plate and continue stirring until the mixture cools to approximately 45.0°C or to room temperature, at which point a smooth emulsion forms (for a fine mixture, continue stirring using a magnetic stirrer or a homogenizer).

7. For a thicker texture, remove the beaker from the heat and allow it to cool to room temperature or about 45.0°C, stirring occasionally.

8. **Neutralization and Thickening:** i) Add 1.0 mL of triethanolamine to the mixture and stir vigorously for 2-3 minutes.

ii) Check the pH; if it is not around pH 6, add more triethanolamine dropwise.

iii) Allow the mixture to cool to room temperature while stirring occasionally. Cool the emulsion gradually to avoid phase separation. This step neutralizes the stearic acid, which contributes to the emulsion's stabilization.

9. **Additional Ingredients:** Once cooled, add 0.5 mL of preservative and 5 -10 drops of essential oil (if desired) for fragrance. Stir to combine.

10. **Cooling and Packaging:** Allow the lotion to cool completely before transferring it to clean, airtight containers. Transfer the lotion to small containers for storage.

Remember: This is the basic formulation for a hand cream. Commercial hand lotion production involves more complex ingredients, precise measurements, strict quality control, and thorough testing and stability studies before any product is used on the skin.

If time or the instructor permits, students can experiment with different combinations of ingredients to achieve the desired texture and feel, comparing the results with and without the addition of triethanolamine or stearic acid. Each group can try additional combinations and compare them with the

rest of the class.

For a richer lotion, increase the amount of emollients (such as lanolin and mineral oil).

-For a lighter lotion, reduce the number of emulsifiers and thickeners.

Part B: **Tests**

1. **pH Test:**

Utilize pH paper to measure the pH of your lotion and document the result in your data table. Assess the pH of a provided commercial lotion, as well as that of your favorite lotion.

2. **Stability Test:**

Leave the samples of the lotion you prepared, along with a commercial lotion, at room temperature for 24 hours or until the next class to check for separation.

3. **Spreadability Test:**

Apply a small amount of the lotion you prepared, along with a commercial lotion, on a watch glass to assess consistency and spreadability.

PRE-LAB QUESTIONS Name _____

1. What is an emulsion? Give three examples of emulsions in everyday life.
2. Why is it important to use a preservative in cosmetic products?
3. What role does stearic acid play in this formulation?
4. Why is triethanolamine added to the mixture?
5. How is the water and oil phase mixed into a stable homogeneous phase while preparing the hand lotion?

DATA AND OBSERVATIONS

Name _____Lab Partner(s) _____

Ingredients	Amounts
Volume of distilled water	
Glycerol	
Mineral oil	
Stearic acid	
Cetyl alcohol	
Anhydrous lanolin	
Triethanolamine	
Preservative if used	Name: Volume:
Essential oil, if used	Name:
	Number of Drops:
Tests	
pH Test	
pH of prepared hand lotion	
pH of commercial hand lotion	
Stability Test	
Prepared hand lotion	
Commercial hand lotion	
Spreadability Test	
Prepared hand lotion	
Commercial hand lotion	

POST-LAB QUESTIONS

1. Describe the appearance and texture of your hand lotion. How does it compare to commercial products?
2. What was the pH of your lotion? Why is pH important in skincare products?
3. Did you notice any separation or instability in your lotion after 24 hours? If so, what could have caused it?
4. How might you modify this formulation to create a thicker or thinner lotion?
5. Discuss the significance of each ingredient in the lotion and how it contributes to the final product.
6. What challenges did you face during the synthesis process, and how did you resolve them?
7. How can you scale this process for commercial production? What additional factors need to be considered?
8. After preparation, the hand cream appeared smooth and uniform, but most of the water and oil had separated after a week of storage. What do you think went wrong with the preparation?

LAB 12 - ISOLATION OF MILK PROTEIN (CASEIN)

PURPOSE

The purpose of this experiment is to:

- Objective 2

- Casein, the primary protein in milk, is isolated by acid precipitation at its isoelectric point.
- Understand the chemistry behind protein denaturation and separation.

INTRODUCTION

Milk is a complex mixture of water, proteins, fats, carbohydrates, and minerals. The primary protein in milk is casein, which accounts for approximately 80% of the total protein content. Casein is essential for the nutritional value of milk and plays an important role in cheese making.

Casein exists in milk as a colloidal suspension of casein micelles. These micelles are stable at the natural pH of milk (approximately pH 6.6) due to the presence of κ-casein on their surface, which provides both steric and electrostatic stabilization. When the pH is lowered to the casein's isoelectric point (approximately pH 4.6), these micelles become destabilized, leading to the precipitation of casein from the solution.

In this lab, we will use acetic acid to lower the pH of milk, causing the casein to precipitate at its isoelectric point. We will then isolate and analyze the precipitated casein.

Chemical Equations: While there isn't a specific chemical reaction occurring, the process can be represented as:

Casein (soluble) + H^+ → Casein (insoluble precipitate)

The acetic acid provides the H^+ ions: $CH_3COOH \rightleftharpoons CH_3COO^- + H^+$

SAFETY PRECAUTIONS

1) Always wear chemical splash goggles while working on this experiment.

2) Wear gloves while handling chemicals.

3) Use caution when handling hot liquids and equipment.

4) Even in dilute form, Acetic acid can cause skin irritation. Rinse immediately if contact occurs.

5) Ensure proper ventilation when working with acetic acid. All liquids used in this experiment should be handled under a fume hood.

6) Dispose of all waste in the designated containers, as instructed by your instructor.

7) When finished, thoroughly clean your work area and return all equipment and chemicals to their appropriate places.

8) Wash your hands before you leave

CHEMICALS AND EQUIPMENT NEEDED

EQUIPMENT	EQUIPMENT	EQUIPMENT	CHEMICALS
1000 mL beaker	pH meter or pH paper	Stirring rod	Skim milk (500 mL)
250 mL beaker	Buchner funnel and filter paper	Graduated cylinder	Acetic acid (5% solution, 50 mL)
Hot plate with magnetic stirrer	Vacuum filtration setup	Digital scale	Distilled water
Thermometer	Ice bath		0.1 M NaOH solution (for pH adjustment if needed)

EXPERIMENTAL PROCEDURE

Part A: Isolation of Casein

1) Measure 100.0 mL of skim milk into a 400.0/600.0 mL beaker. If you are not using skim milk, centrifuge the milk using centrifuge tubes to remove the fat. When centrifuging non-skim milk, the fat collects on top and should be removed before heating the milk in the next step.

2) Heat the milk to 40.0°C while stirring gently on a hot plate with a magnetic stirrer.

3) While the milk warms, obtain a pH electrode, rinse it thoroughly with distilled water, and place it in a 250.0 mL beaker containing approximately 100.0 to 150.0 mL of distilled water.

4) Gradually add 5.0 mL of 5% acetic acid solution to the warm milk while stirring continuously. Keep stirring and monitor the pH using a pH electrode.

5) The milk should start to curdle. If necessary, add more acetic acid drop by drop until the pH reaches 4.6, which is the isoelectric point of casein.

6) Your instructor may use 0.2 M HCl for steps 4 and 5. If using 0.2 M HCl, add it dropwise to the milk until a steady pH of 4.6 is reached.

5) Remove the mixture from the heat and let it stand for 5 minutes.

7) Prepare an ice bath and cool the mixture for 10 minutes, stirring occasionally.

8) Set up the vacuum filtration apparatus with a Buchner funnel and filter paper.

9) Filter the cooled mixture to separate the precipitated casein from the whey.

9) Rinse the precipitate with cold distilled water to remove any remaining acid.

10) Transfer the casein to a pre-weighed filter paper and let it air dry overnight.

11) Weigh the dried casein and calculate the yield.

Part B: Tests

1. **Biuret test:** Mix a small amount of isolated casein with the Biuret reagent. A positive result (purple color) confirms the presence of protein.
2. **Solubility test**: Test the solubility of isolated casein in water and dilute NaOH solution.
3. **pH test**: Check the pH of the whey (filtrate) after casein precipitation.

PRE-LAB QUESTIONS Name _____

1. Why do we use skim milk instead of whole milk for this procedure? Can whole milk be used for this experiment? If so, how can we use it?
2. What is the isoelectric point, and why is it important in this experiment?
3. How does acetic acid cause the precipitation of casein?
4. What other methods could isolate proteins from a solution?
5. What is the theoretical yield of casein for skim milk?

DATA AND OBSERVATIONS

Name _____Lab Partner(s) _____

Measurement	Value
Initial volume of milk (mL)	
Volume of acetic acid used (mL)	
Final pH of the mixture	
Mass of empty filter paper (g)	
Mass of filter paper + dried casein (g)	
Mass of isolated casein (g)	

1. Calculate the concentration of casein in the original milk sample (g / 100 mL).

Note: Skim milk typically contains about 3.4% protein, of which about 80% is casein.

2. Calculate the percentage yield of casein:

% Yield = (Mass of isolated casein / Expected mass of casein in milk) × 100

% Yield =

POST-LAB QUESTIONS

1. How does the yield of casein you obtained compare to the theoretical yield? Explain any discrepancies.
2. Describe the appearance and texture of the isolated casein. How does it differ from its appearance in milk?
3. What factors might affect the efficiency of casein isolation using this method?
4. How might the pH of precipitation affect the purity and yield of the isolated casein?
5. Discuss potential applications of isolated casein in the food industry or other fields.
6. What other milk components remain in the whey after casein precipitation? How might these be isolated?

7. How would you modify this procedure to isolate other milk proteins, such as whey proteins?

8. Describe how this lab demonstrates the relationship between protein structure and solubility.

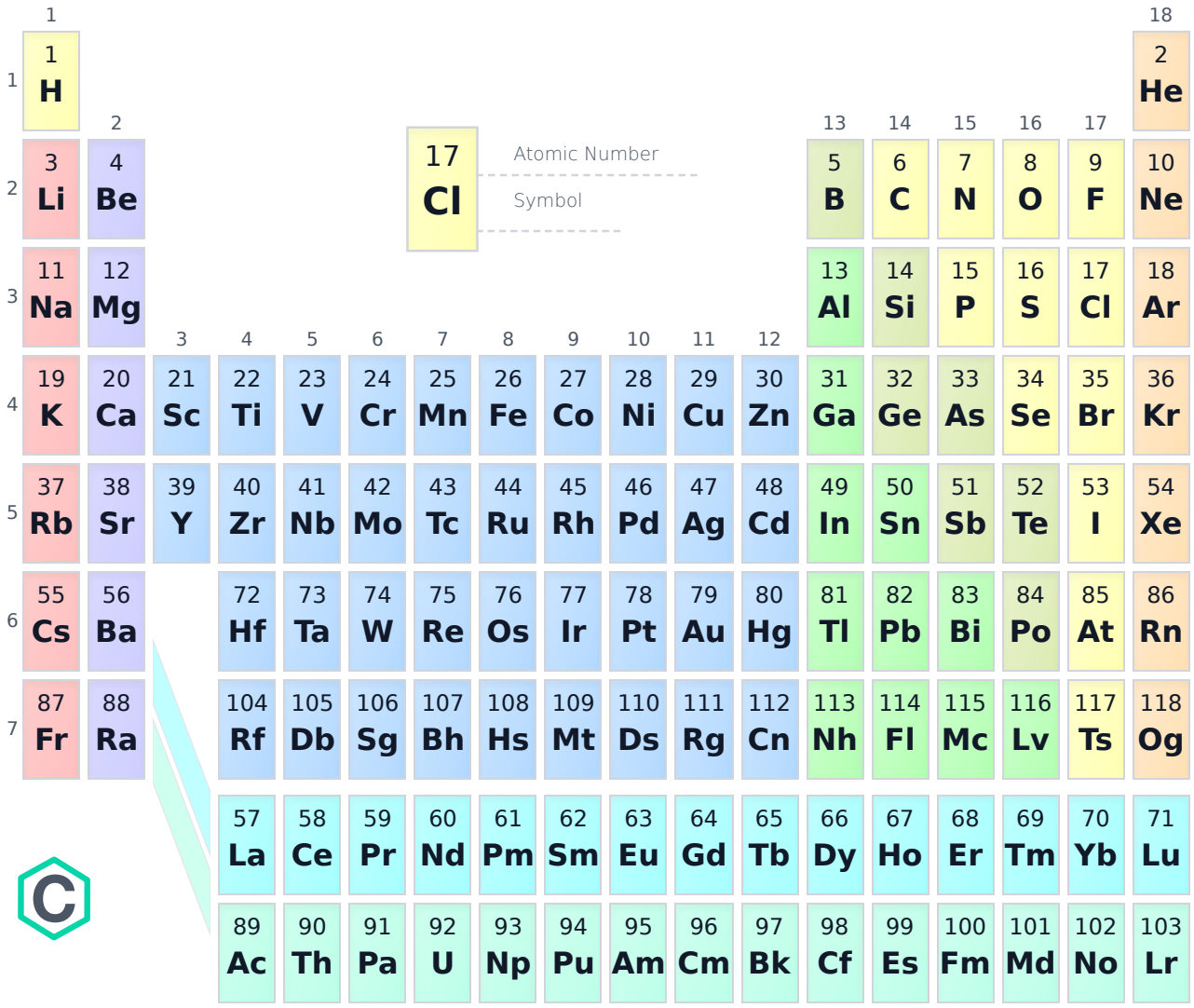
CHAPTER OVERVIEW

13: APPENDIX

13.1: PERIODIC TABLE

PUBCHEM > PERIODIC TABLE

Periodic Table of Elements



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