ORGANIC CHEMISTRY I LAB MANUAL [CHM 234]

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Triton College

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Organic Chemistry I Lab Manual [CHM 234]

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TABLE OF CONTENTS

Licensing

0: Lab Safety Orientation

- 0.1: Lab Safety Videos
- 0.2: Lab Safety Contract
- 0.3: Lab Safety Quiz
- 0.4: Additional Resources

1: Recrystallization

- 1.1: Recrystallization - Experiment
- 1.2: Recrystallization - Pre-lab
- 1.3: Recrystallization - Data and Report

2: Identification of an Unknown Using Simple Distillation

- 2.1: Identification of an Unknown - Experiment
- 2.2: Identification of an Unknown - Pre-lab
- 2.3: Identification of an Unknown - Data and Report

3: Fractional Distillation

- 3.1: Fractional Distillation - Experiment
- 3.2: Fractional Distillation - Pre-lab
- 3.3: Fractional Distillation - Data and Report

4: Extraction

- 4.1: Extraction - Experiment
- 4.2: Extraction - Pre-lab
- 4.3: Extraction - Data and Report

5: Thin-Layer Chromatography (TLC)

- 5.1: Thin-Layer Chromatography - Experiment
- 5.2: Thin-Layer Chromatography - Pre-lab
- 5.3: Thin-Layer Chromatography - Data and Report

6: Column Chromatography

- 6.1: Column Chromatography - Experiment
- 6.2: Column Chromatography - Pre-lab
- 6.3: Column Chromatography - Data and Report

7: Stereochemistry

- 7.1: Stereochemistry - Experiment
- 7.2: Stereochemistry - Pre-lab
- 7.3: Stereochemistry - Data and Report

8: Gas Chromatography

- 8.1: Gas Chromatography - Experiment
- 8.2: Gas Chromatography - Pre-lab
- 8.3: Gas Chromatography - Data and Report

9: Polarimetry

- 9.1: Polarimetry - Experiment
- 9.2: Polarimetry - Pre-lab
- 9.3: Polarimetry - Data and Report

10: Synthesis of 1-bromobutane

- 10.1: Synthesis of 1-bromobutane - Experiment
- 10.2: Synthesis of 1-bromobutane - Pre-lab
- 10.3: Synthesis of 1-bromobutane - Data and Report

11: Synthesis of t-Butyl Chloride

- 11.1: Synthesis of t-Butyl Chloride - Experiment
- 11.2: Synthesis of t-Butyl Chloride - Pre-lab
- 11.3: Synthesis of t-Butyl Chloride - Data and Report

12: Synthesis of Cyclohexene

- 12.1: Synthesis of Cyclohexene - Experiment
- 12.2: Synthesis of Cyclohexene - Pre-lab
- 12.3: Synthesis of Cyclohexene - Data and Report

13: Appendix

Index

Glossary

Detailed Licensing

Detailed Licensing

Licensing

*A detailed breakdown of this resource's licensing can be found in **Back Matter/Detailed Licensing**.*

CHAPTER OVERVIEW

0: Lab Safety Orientation

0.1: Lab Safety Videos
0.2: Lab Safety Contract
0.3: Lab Safety Quiz
0.4: Additional Resources

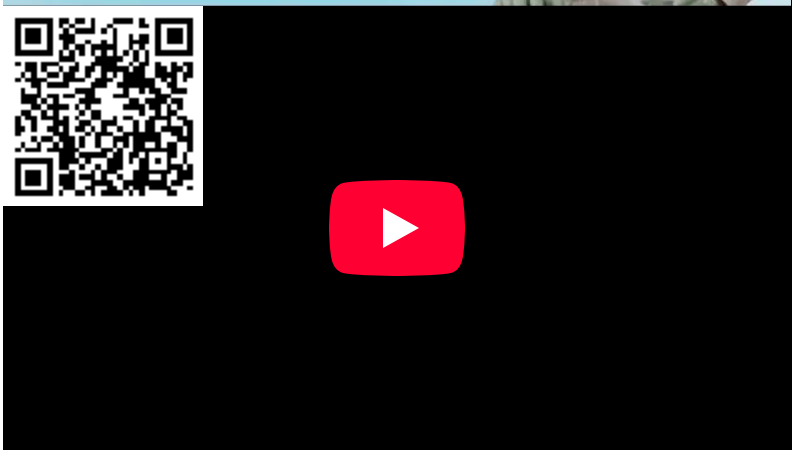
Figure **0.1**: The four principles of safety (Copyright; ACS)

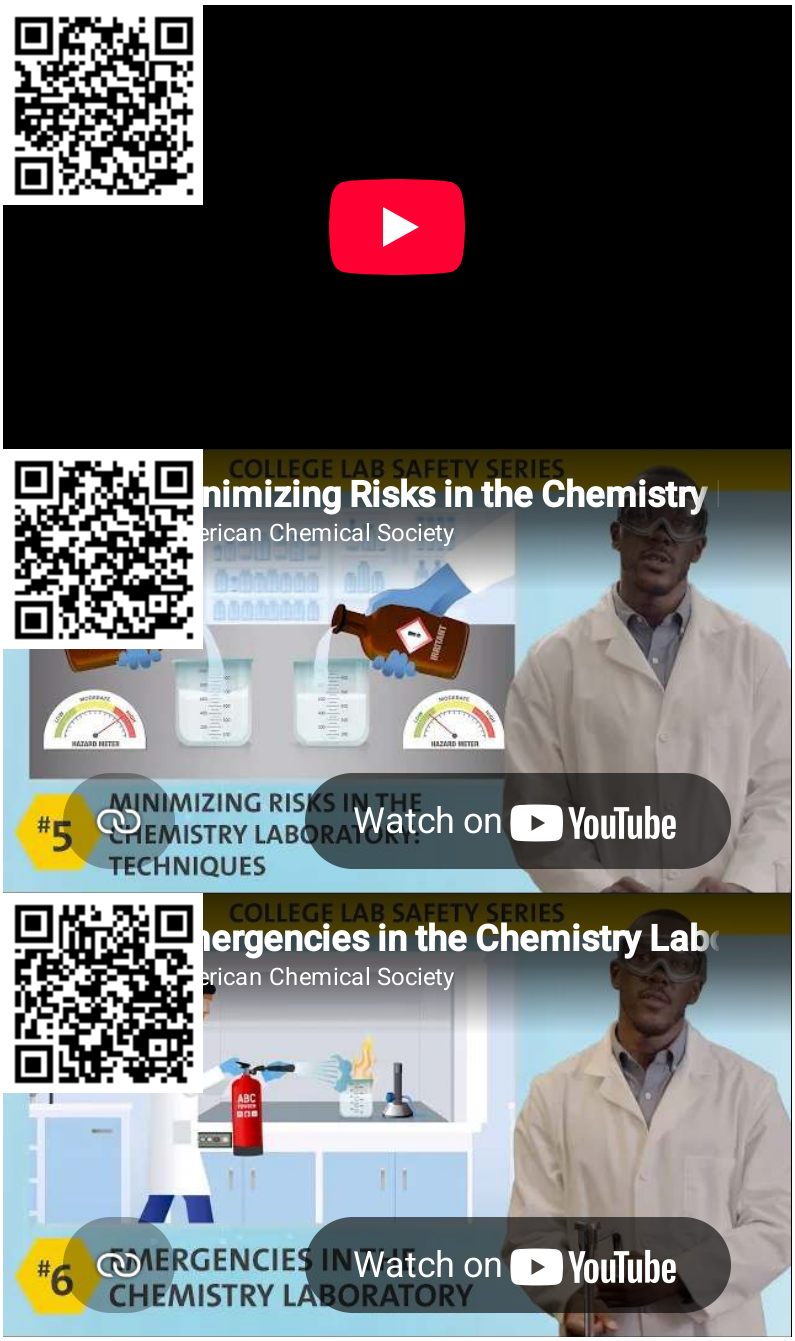
0.1: Lab Safety Videos

Safety Orientation 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**





2. Lab Safety and Techniques- Crash course safety

3. Safety Video by American Chemical Society (1991)

License

0.2: Lab Safety Contract

Triton College Chemistry Division

Student Safety Contract

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!!**

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. **You should always wear safety goggles when handling chemicals.**
   - True
   - False
6. **It's safe to taste chemicals in the lab to identify them.**
   - True
   - False
6. **You should know the location of safety equipment such as fire extinguishers, eyewash stations, and safety showers.**
   - True
   - False
6. **Long hair should be tied back when working in the lab.**
   - True
   - False
6. **It's acceptable to use your phone in the lab as long as you're careful.**
   - True
   - False

0.4: Additional Resources

OSHA® QUICK CARD™

Hazard Communication Standard Pictogram

The Hazard Communication Standard (HCS) requires pictograms on labels to caution users of the chemical hazards that they may be exposed to. A pictogram consists of a symbol on a white background framed within a red border and represents a distinct hazard(s). The pictogram on the label is determined by the chemical hazard classification. Each pictogram may only appear once on a label. If multiple hazards require the use of the same pictogram, it may not appear a second time on the label.

HCS Pictograms and Hazards

Health Hazard	Flame	Exclamation Mark
		
• Carcinogen • Mutagenicity • Reproductive Toxicity • Respiratory Sensitizer • Target Organ Toxicity • Aspiration Toxicity	• Flammables • Pyrophorics • Self-Heating • Emits Flammable Gas • Self-Reactives • Organic Peroxides • Desensitized Explosives	• Irritant (skin and eye) • Skin Sensitizer • Acute Toxicity (harmful) • Narcotic Effects • Respiratory Tract Irritant • Hazard Not Otherwise Classified (non-mandatory) • Hazardous to Ozone Layer (non-mandatory)
Gas Cylinder	**Corrosion**	**Exploding Bomb**

Figure 0.4.1: OSHA Hazard Communication Standard Pictogram QuickCard

CHAPTER OVERVIEW

1: Recrystallization

PURPOSE

The purpose of this experiment is to:

- Recrystallize benzoic acid from water.
- Determine the melting point of recrystallized benzoic acid.
- Set up and use a vacuum filtration apparatus.

INTRODUCTION

Products synthesized from organic reactions often contain impurities. Recrystallization is a technique used to purify an organic solid. The process occurs by dissolving the solid in a solvent in which it is insoluble at room temperature but soluble near the boiling point. The solution is then cooled to room temperature, resulting in the precipitation of the pure solid in its crystalline form. The impurities remain dissolved in solution.

One way to test the purity of a solid is by taking its melting point. A pure solid will sharply melt very close to the compound's actual melting point. However, impurities will cause a solid to melt over a lower and wider temperature range.

In this experiment, an impure sample of benzoic acid will be recrystallized using water as the solvent. Once the solution is cooled to room temperature and crystals form, they will be isolated using vacuum filtration. The purity of the recrystallized benzoic acid will then be tested by measuring its melting point and comparing it with the literature value.

1.1: Recrystallization - Experiment
1.2: Recrystallization - Pre-lab
1.3: Recrystallization - Data and Report

1.1: Recrystallization - Experiment

SAFETY PRECAUTIONS

- Wear chemical splash goggles throughout the experiment.
- Gloves are provided if you wish to wear them.
- Work with all chemicals under the fume hood.
- Be careful when handling hot glassware.
- Dispose of all waste in the appropriate waste containers.
- Clean your work area (hood and lab bench) and return all glassware and equipment to the appropriate place once you finish the experiment.
- Always wash your hands immediately upon leaving the lab.

EQUIPMENT AND CHEMICALS NEEDED

Table **1.1.1**: Materials Needed

Equipment	Equipment	Chemicals
Spatula	Milligram balance	Impure benzoic acid
Stirring rod	Fume hood	Deionized water
50 mL Erlenmeyer flask	Watch glass	Ice
100 mL graduated cylinder	Melting point apparatus	Non-halogenated organic waste container
250 mL beaker	Capillary tube with one open end (for melting point determination)	
Hot plate	Vacuum filtration set-up (Buchner funnel, rubber hose, flask with side-arm, open rubber stopper, filter paper)	

EXPERIMENTAL PROCEDURE

Part I: Recrystallization of Benzoic Acid

1. Add 1.000 g of benzoic acid to a 50 mL Erlenmeyer flask. Record the exact mass in line 1 of the data table.
2. Using a 100 mL graduated cylinder, add 15.0 mL of room temperature deionized water to the flask containing the benzoic acid. Record the exact volume of water used in line 2 of the data table. Be sure there is no solid remaining on the sides of the flask.
3. Place the flask containing the benzoic acid and water on a hot plate under the hood, and gently heat the mixture until all of the benzoic acid dissolves. You may occasionally stir the solution to promote dissolution of the solid. Note: To minimize water evaporation, keep the hot plate on a lower heat setting.
4. Once all the benzoic acid has dissolved, carefully remove the flask from the hot plate and allow it to cool to room temperature.
5. Once cooled, place the flask in a 250 mL beaker containing water and ice for approximately 15 minutes. During this time, the maximum amount of recrystallized benzoic acid should form.

Part II. Collection of Recrystallized Benzoic Acid

1. Weigh an empty watch glass and a sheet of filter paper. Record the exact value in line 3 of the data table.
2. Set up a vacuum filtration apparatus* under the hood. This includes connecting a rubber hose to the vacuum valve and the side arm of a flask. An open rubber stopper should be placed between the flask and the Buchner funnel. The filter paper will then be placed in the Buchner funnel. Your instructor will demonstrate this setup and any variations (such as the use of a vacuum trap) specific to your lab room.
3. Collect the recrystallized benzoic acid using the vacuum filtration apparatus. Rinse any remaining crystals in the flask with ice-cold water.

4. Allow the crystals to completely dry. This may take up to 30 minutes. To promote drying, gently break up the solid with a glass stirring rod. The solid will be considered dried when it no longer clings to the glass stirring rod.
5. Weigh the watch glass, filter paper, and dried crystals. Record the exact mass in line 4 of the data table.
6. Calculate the mass of recrystallized benzoic acid, and record this in line 5 of the data table.
7. Calculate the percent recovery, as follows:

$$\text{Percent recovery} = \frac{mass\ of\ recrstallized\ benzoic\ acid}{mass\ of\ original\ sample} \text{ x } 100$$

Part III. Determining the Melting Point of the Recrystallized Benzoic Acid

1. Pack a capillary tube approximately 5 mm with the recrystallized benzoic acid.
2. Place the capillary tube into the melting point apparatus, and record the melting point* of the recrystallized product. (**Note:** Your instructor will demonstrate steps 1 and 2, as different processes and instruments may be used in various lab rooms.)
3. Dispose of all waste in the non-halogenated organic waste container, and clean your work area as directed by your instructor.

1.2: Recrystallization - Pre-lab

Name: _____ Date: _____

1) What is recrystallization, and how can it be used to purify an organic solid?

2) An unknown solid needs to be recrystallized. Solubility data using three different solvents is shown below:

Solvent	Solubility at Room Temperature	Solubility near Boiling Point
A	Solid is soluble	Solid is soluble
B	Solid is insoluble	Solid is soluble
C	Solid is insoluble	Solid is insoluble

Based on this information, which solvent would be the best to use for the recrystallization of the unknown solid? Explain.

3) The solubility of benzoic acid in water is approximately 2 g/L at room temperature and 68 g/L near the boiling point. How much of a 1.000 g sample would you expect to dissolve in 15.0 mL of room-temperature water?

1.3: Recrystallization - Data and Report

Name: _____ Partner(s): _____ Date: _____

DATA AND OBSERVATIONS

Table 1.3.1: Data Table

Procedure	Data
Mass of benzoic acid	
Volume of water used	
Mass of empty watch glass and filter paper	
Mass of watch glass, filter paper, and recrystallized benzoic acid	
Mass of recrystallized benzoic acid	

POST-LAB QUESTIONS

1. Calculate the percent recovery of recrystallized benzoic acid. Show your work.

2. What would a percent recovery over 100 indicate? If your percent recovery was over 100, how can you prevent this from occurring in the future?

3. Explain the importance of recrystallization in relation to characterizing organic compounds.

CHAPTER OVERVIEW

2: Identification of an Unknown Using Simple Distillation

PURPOSE

The purpose of this experiment is to:

- Set up a simple distillation apparatus, and use it to determine the boiling point of an unknown.
- Calculate the density of an unknown substance.
- Determine the solubility of an unknown in water.

Physical properties (such as density, solubility, and boiling point) can be used to identify an unknown. For example, we can infer that an unknown liquid with a density of 1.0 g/mL at 25 °C and a boiling point of 100.0 °C at 1 atm is water. However, we would have to run more conclusive tests to either prove or disprove this.

In general chemistry, you learned how to determine the density of an unknown. In this experiment, you will use density and solubility data, along with the boiling point, to identify an unknown organic liquid from a list of known solutions. One technique used to determine the boiling point of an organic liquid is simple distillation. This process involves heating a liquid to its boiling point and then condensing and collecting the resulting distillate. Since only the liquid, and not any impurities present, will collect at the boiling point, simple distillation can also be used to purify an organic liquid.

2.1: Identification of an Unknown - Experiment

2.2: Identification of an Unknown - Pre-lab

2.3: Identification of an Unknown - Data and Report

2.1: Identification of an Unknown - Experiment

SAFETY PRECAUTIONS

1. Wear chemical splash goggles throughout the experiment.
2. Gloves are provided if you wish to wear them.
3. Work with all chemicals under the fume hood.
4. Be careful when handling hot glassware.
5. Dispose of all waste in the appropriate waste containers.
6. Clean your work area (hood and lab bench) and return all glassware and equipment to the appropriate place once you finish the experiment.
7. Always wash your hands immediately upon leaving the lab.

EQUIPMENT AND CHEMICALS NEEDED

Table **2.1.1**: Materials Needed

Equipment	Chemicals
Medium Size Test Tube	Unknown
Glass Stirring rod	Deionized water
10.0 mL graduated cylinder	Non-halogenated organic waste container
Simple Distillation Apparatus	
Milligram balance	

EXPERIMENTAL PROCEDURE

PART I: SOLUBILITY OF THE UNKNOWN

1. Add 1.0 mL of water and 1.0 mL of the unknown to a medium-sized test tube. Stir the liquids to determine whether or not a solution forms.

2. Record your observations in the data and observations section.

3. Pour the contents of the test tube into the non-halogenated organic waste container. Clean the test tube with soap and water, and return it to its appropriate place.

PART II: DETERMINATION OF THE DENSITY OF THE UNKNOWN

1. Weigh an empty 10.0 mL graduated cylinder. Record the value under trials 1-3 in the data table.

2. Add anywhere between 1.0 and 10.0 mL of the unknown to the graduated cylinder. Record the exact volume used under trial 1 in the data table.

3. Obtain the combined mass of the graduated cylinder and the unknown liquid. Record the exact value under trial 1 in the data table.

4. Find the mass of the unknown liquid, and then calculate its density. Record this information under trial 1 in the data table.

5. Pour the contents of the graduated cylinder into the non-halogenated organic waste container.

6. Repeat steps 2-4 for trials 2 and 3, using a different volume of the unknown each time.

7. Calculate the average density of the unknown liquid.

PART III: DETERMINATION OF THE BOILING POINT OF THE UNKNOWN

1. Set up a simple distillation apparatus under the hood, and determine the boiling point of 10.0 mL of the unknown liquid. Your instructor will demonstrate the setup based on the equipment and conditions for your lab.

2. Once approximately 2.0 mL of liquid remains in the distilling flask, turn off the heat but leave the water running.

3. Once the glassware is cool, disassemble the apparatus, clean all glassware, and pour the unknown liquid into the non-halogenated waste container.

PART IV: IDENTIFICATION OF THE UNKNOWN LIQUID

Using the results of the solubility test, density, and boiling point, identify the unknown liquid from the list of known solutions listed in pre-lab question 3.

2.2: Identification of an Unknown - Pre-lab

Name: _____ Date: _____

PRE-LAB QUESTIONS

1.) Briefly describe how to set up a simple distillation apparatus.

2.) Explain why simple distillation is a safe method for determining the boiling point of an organic liquid (as opposed to heating the liquid in a beaker on a hot plate and using a thermometer to determine the boiling point).

3.) The unknown liquid analyzed in this experiment will be one of the knowns listed in the following table. Complete the table by supplying solubility, density, and boiling point information for each substance.

List of Known Liquids

Compound	Soluble in water?	Density (g/mL)	Boiling point (^{o}C)
cyclohexane			
Acetone			
Isopropyl alcohol			
Toluene			

2.3: Identification of an Unknown - Data and Report

Name: _____ Partner(s): _____ Date: _____

DATA AND OBSERVATIONS

Unknown letter/number =

PART 1: SOLUBILITY OF THE UNKNOWN

Is the unknown soluble in water?

If the unknown is not soluble in water, which layer did it form (top or bottom) when added to water?

PART II: DETERMINATION OF THE DENSITY OF THE UNKNOWN

	Trial 1	**Trial 2**	**Trial 3**
Mass of an empty graduated cylinder			
Mass of graduated cylinder and unknown			
Mass of unknown			
Volume of unknown			
Density of unknown			

Show the calculation of the density of the unknown for each trial:

Trial 1:

Trial 2:

Trial 3:

Show the calculation for the average density of the unknown:

PART III: DETERMINATION OF THE BOILING POINT OF THE UNKNOWN

Boiling point of unknown =

PART IV: IDENTIFICATION OF THE UNKNOWN LIQUID

Based on the solubility test, density, and boiling point, identify the unknown liquid as one of the knowns from the table in prelab question 3. Provide evidence to support your decision.

POST-LAB QUESTIONS

1.) What was the biggest takeaway from performing this experiment?

2.) Imagine that the unknown liquid was not pure. What would you observe during simple distillation?

3.) How conclusive are the results of this experiment? What are some additional tests you could run to solidify your identification of the unknown?

CHAPTER OVERVIEW

3: Fractional Distillation

PURPOSE

The purpose of this experiment is to:

- To perform fractional distillation of a mixture of ethyl acetate and butyl acetate.
- To collect fractions and determine purity using Gas Chromatography (GC).
- To analyze retention times and peak areas for ethyl acetate, butyl acetate, and collected fractions.

INTRODUCTION

Fractional distillation separates miscible liquids with different boiling points. Ethyl acetate (bp ≈ 77.1°C) and butyl acetate (bp ≈ 126.1°C) differ by ~49°C, allowing efficient separation. The fractionating column provides multiple theoretical plates for repeated condensation and vaporization, enriching the vapor in the more volatile component (ethyl acetate).

3.1: Fractional Distillation - Experiment
3.2: Fractional Distillation - Pre-lab
3.3: Fractional Distillation - Data and Report

3.1: Fractional Distillation - Experiment

SAFETY PRECAUTIONS

- Wear safety goggles and gloves.
- Work in a fume hood to avoid inhalation of vapors.
- **CAUTION:** Ethyl acetate and butyl acetate are both hazardous in case of ingestion or inhalation.
- Keep flammable liquids away from flames.
- Dispose of waste in designated organic waste containers.

EQUIPMENT AND CHEMICALS NEEDED

Table **3.1.1**: Materials Needed

Equipment	Chemicals
Fractional distillation apparatus (flask, column, condenser)	Ethyl acetate
Heating mantle	Butyl acetate
Thermometer (Wide-Range Temperature Probe)	50:50 ethyl acetate and butyl acetate
Collection flasks or 10 ml graduated cylinders	Parafilm
GC (Vernier Mini GC and LabQuest interface)	Boiling stone

EXPERIMENTAL PROCEDURE

1. Assemble the fractional distillation apparatus with a 100 mL round-bottom flask on a heating mantle.
2. Connect the fractionating column with a Thermometer. If not using any interface, monitor the thermometer carefully for any temperature changes and fluctuations. (We will use a wide-range temperature probe connected to the LabQuest interface. Start the data-collection program, and then choose New from the File menu. Change the data-collection duration to 90 minutes to ensure all data is collected.)
3. Attach the condenser with water inlet and outlet connected.
4. Add 20.0 mL of a 50:50 or unknown (as directed by your instructor) mixture of ethyl acetate and butyl acetate to the flask. Add a boiling stone to the flask, and reattach it to the apparatus. **CAUTION**: *Ethyl acetate and butyl acetate are both hazardous in case of ingestion or inhalation*
5. Heat gradually and use 10.0ml graduated cylinders to collect fractions, as the temperature changes from ~77.0°C to ~126.0°C. **Important**: Do not allow the round-bottom distilling flask to go dry.
6. Record observations and temperatures for each fraction.

GC ANALYSIS STEPS

1. Prepare the GC instrument. (We use Vernier GC and LabQuest Interface. The steps for the sample analysis using this instrumentation and software are listed as provided by Vernier. If your lab uses a different GC and platform, your instructor will provide you with the necessary steps.)
2. Inject samples and record retention times.
3. Compare with standards and calculate purity.

3.2: Fractional Distillation - Pre-lab

Name: _____ Date: _____

1. Mention different types of distillation techniques. Which technique provides better separation and why?
2. Taking into account the boiling points of ethyl acetate and butyl acetate, which component will be collected first?
3. Explain the advantages and disadvantages of fractional distillation.

3.3: Fractional Distillation - Data and Report

Name: _____ Partner(s): _____ Date: _____

DATA AND OBSERVATIONS

Fractional Distillation

Fraction Number	Temperature Range (°C)	Observations
1		
2		
3		
4		
5		

GC ANALYSIS

GC ANALYSIS

Sample	Peak Area of Ethyl Acetate and Butyl Acetate	Observations
Ethyl Acetate (Pure)		
Butyl Acetate (Pure)		
50:50 mixture of Ethyl Acetate and Butyl Acetate		
Fraction 1		
Fraction 2		
Fraction 3		
Fraction 4		
Fraction 5		

POST-LAB ANALYSIS

1. To what extent did the fractionating column effectively separate the components in your experiment? What modifications could be implemented to enhance the efficiency of the separation process?
2. Which fraction was purest and why?
3. Calculate the peak area ratio of ethyl acetate: butyl acetate for each fraction, based on your distillation and GC data?

CHAPTER OVERVIEW

4: Extraction

Learning Objectives

The Purpose of this Experiment is to:

- **Separate** a three-component organic mixture (benzoic acid, 3-nitroaniline, and naphthalene) using **sequential acid–base extractions** in a separatory funnel.
- **Characterize purity** by measuring the **melting temperature ranges** of the isolated solids with a melting-point apparatus

INTRODUCTION

Liquid–liquid extraction exploits differences in solubility between immiscible liquids to transfer a solute from one phase to another. In organic chemistry, **acid–base extractions** convert acids or bases into **water-soluble ionic forms**, enabling phase separation from neutral organics. For example, carboxylic acids react with base to form **carboxylate salts** that partition preferentially to aqueous solution, and amines react with acid to form **ammonium salts** that do the same.

In this experiment, a mixture of **benzoic acid** (carboxylic acid), **3-nitroaniline** (amine), and **naphthalene** (neutral aromatic) is dissolved in an organic solvent immiscible with water (**diethyl ether**). Selective extractions with **dilute HCl** (to protonate the amine) and **dilute NaOH** (to deprotonate the carboxylic acid) shuttle the ionic species into the **aqueous phase** while leaving the neutral compound in the **organic phase**. Subsequent **back-neutralization** regenerates each neutral compound as a precipitate for isolation and analysis.

Key acid–base transformations:

Carboxylic Acid De-protonation:

$$\mathrm{RCO_2H\ (solv) + OH^-\ (aq)\ \rightarrow\ RCO_2^-\ (aq) + H_2O\ (l)}$$

Re-protonation to recover the acid:

$$\mathrm{RCO_2^-\ (aq) + H^+\ (aq)\ \rightarrow\ RCO_2H\ (s)}$$

Amine protonation:

$$\mathrm{RNH_2\ (solv) + H^+\ (aq)\ \rightarrow\ RNH_3^+\ (aq)}$$

Basification to recover the free amine:

$$\mathrm{RNH_3^+\ (aq) + OH^-\ (aq) \rightarrow\ RNH_2\ (s) + H_2O\ (l)}$$

As a purity check, you will determine the **melting temperature ranges** of the isolated solids. Pure crystalline organics melt over a narrow range; impurities broaden and depress the melting range.

4.1: Extraction - Experiment
4.2: Extraction - Pre-lab
4.3: Extraction - Data and Report

4.1: Extraction - Experiment

SAFETY PRECAUTIONS

- Wear safety goggles and gloves.
- Work in a fume hood to avoid inhalation of vapors.
- **CAUTION:**
- - **Diethyl ether** – extremely **flammable**, highly volatile; peroxide former; use **no open flames** and work in a **fume hood**.
  - **Hydrochloric acid (HCl)** – **corrosive**; causes burns; strong fumes.
  - **Sodium hydroxide (NaOH)** – **corrosive**; causes severe skin/eye burns.
- **3-Nitroaniline** – **toxic/irritant**; avoid inhalation and skin contact; consult SDS.
  Use GHS labels/pictograms and SDSs to confirm hazards before use.
- Ether pressure builds readily in a closed sepratory funnel during shaking; acid/base neutralizations are exothermic; potential splashes during transfers; inhalation of vapors
- Handle ether and acid/base solutions in a **fume hood**; **vent** the separatory funnel frequently, pointing away from people; use secondary containment for waste.
- Collect **organic ether solutions** and rinses in a capped **non-halogenated organic waste** container;
- Collect **acidic/caustic aqueous extracts** separately.

Equipment and Chemicals

Glassware & hardware

- 125-mL separatory funnel; ring stand + ring/clamp; two 100-mL beakers; four 50–125-mL Erlenmeyer flasks; Pasteur pipets + bulbs; vacuum and gravity filtration setups; watch glasses; 10-mL graduated cylinder; pH paper; mortar & pestle; ice bath; **Melting-point apparatus,** compressed air (optional),closed-end capillary tubes, lint-free tissues.

Reagents

- **Mixture** of benzoic acid, 3-nitroaniline, naphthalene (~3 g total).
- **Diethyl ether** (anhydrous).
- **HCl**: 5% (w/w) for extraction; **6.0 M** for neutralization.
- **NaOH**: 5% (w/w) for extraction; **6.0 M** for neutralization.
- **Saturated NaCl** (“brine”) for washing; **anhydrous Na_2SO_4** for drying; cold distilled water.

Experimental Procedure

Conduct all ether handling and acid/base neutralizations in a **fume hood**. Vent the separatory funnel frequently and point away from yourself/others.

Part I — Sequential Acid–Base Extraction

1. **Set up & PPE.** Put on splash **goggles** and gloves. Inspect the **separatory funnel** for leaks with a brief water test; drain and dry.
2. **Dissolve the mixture.** Accurately weigh **~3.000 g** of the solid mixture (±0.001 g). Transfer to a 100-mL beaker and dissolve in **50 mL** diethyl ether with gentle swirling. *Caution: extremely flammable—no flames or hot plates; use a steam bath only when instructed.*
3. **Load the sep funnel.** Transfer the ether solution to the 125-mL separatory funnel. Add **50 mL of 5% HCl**. Cap, invert, and **vent immediately**. Gently shake for ~30 s, **venting every few seconds**. Place the funnel in the ring and allow full separation; **uncap while standing**.
4. **Split layers.** Drain the **lower aqueous (acidic)** layer (contains **protonated 3-nitroaniline, $\mathrm{RNH_3^+}$**) into a labeled Erlenmeyer (“Aqueous-Acidic”). Return the **upper ether** layer to the funnel.
5. **Recover the amine.** Cool the acidic extract in an **ice bath**. With stirring, **basify** dropwise using **6.0 M NaOH** to **pH > 9** (pH paper). Collect the precipitated **3-nitroaniline** by **vacuum filtration** on pre-weighed filter paper; rinse with **cold water**; dry on the funnel, then on a watch glass. *Record masses for percent recovery.*

6. **Extract the acid.** To the saved ether layer, add **50 mL of 5% NaOH**. Shake/vent as before. Drain the **lower basic aqueous** layer (contains **benzoate, $\mathrm{RCO_2^-}$**) into a labeled Erlenmeyer (“Aqueous-Basic”). Keep the ether layer in the funnel.
7. **Recover carboxylic acid.** Ice-cool the basic extract; **acidify** carefully with **6.0 M HCl** to **pH < 2**. Collect the precipitated **benzoic acid** by **vacuum filtration** on pre-weighed filter paper; rinse with **cold water**; dry thoroughly. *Record masses.*
8. **Wash the neutral fraction.** To the ether layer (now enriched in **naphthalene**), add **50 mL saturated NaCl (brine)**; shake/vent; allow separation; **discard** the lower aqueous brine appropriately. Dry the organic phase over **~1 g anhydrous Na_2SO_4** (swirl ~10 min). Gravity filter the dried ether solution.
9. **Remove solvent safely.** In the hood, **evaporate ether** on a **steam bath** or with gentle **air flow**—**never** near an ignition source; do not leave unattended. Dry the **naphthalene** residue to constant mass and record.

Part II — Melting Temperature Determination

10. **Sample prep.** Finely powder each isolate; fill a **closed-end capillary** to ~3–4 mm.
11. **Instrument setup.** Confirm the **Melt Station** control dial is **set to Off; connect power and the** interface (LabQuest/Graphical Analysis). Insert the capillary into a slot. **clear-point** to define the melting range. Cool between runs using the **Fan/Cooling** setting. Record ranges for each compound.
12. Dispose of used capillaries in **sharps** as directed.

4.2: Extraction - Pre-lab

Name: _____ Date: _____

Pre-Lab Questions

1. Using equations, explain how benzoic acid and 3-nitroaniline are transferred into the **aqueous** layer under **basic** and **acidic** conditions, respectively. Write the **two neutralization steps** used to recover each as solids.
2. With **diethyl ether** and water, which layer is typically on top? How would you **confirm** the layer identity if unsure?
3. List the **GHS pictograms** for ether, HCl, and NaOH, and propose **engineering/administrative/PPE** controls for the highest-risk step in this procedure.
4. What is the purpose of a **brine wash** and **anhydrous Na_2SO_4**, and how do they improve recovery/purity? *(Short paragraph.)*
5. Why do we heat rapidly at first, then slow to ~1–2 °C min^{-1} near the expected melting point, and why does **impurity** broaden the range?

4.3: Extraction - Data and Report

Name: _____ Partner(s): _____ Date: _____

Data & Observations

Record **raw data in ink** in a **bound notebook** at the time of observation; correct mistakes with a single strike-through so the original remains legible. Include **units** and **uncertainties**.

Part I — Extraction

Masses

- Mass of mixture / g: _____
- **Benzoic acid:** filter paper / g _____; filter+product / g _____; product / g _____
- **3-Nitroaniline:** filter paper / g _____; filter+product / g _____; product / g _____
- **Naphthalene:** product / g _____

Process notes (layer positions, pH values at neutralizations, observations of emulsions/precipitation, drying time, odors/volatility handled in hood).

Part II — Melting Temperatures

Compound	Measured melting range / °C
Benzoic acid	
3-Nitroaniline	
Naphthalene	

Post-Lab Calculations & Analysis

1. **Percent recovery**
   For each component i: latex

$$\%\ \text{recovery}_i = \frac{m_{\text{recovered},\,i}}{m_{\text{initial mixture}}/3} \times 100\% \tag{4.3.1}$$

   (Assuming equal initial masses unless your instructor provides a different composition.)
2. **Melting ranges & purity**
   - Compare each measured range to the literature/known values provided by your instructor. Discuss **range breadth** and **depression** as indicators of impurities (residual solvent, salts).
3. **Error analysis**
   - Identify **systematic** sources (layer misidentification, incomplete neutralization, wet solids, solvent losses) vs. **random** (mass balance fluctuations). Propose **improvements** (e.g., confirm layer identity via density drop test, allow full crystallization on ice, ensure complete drying over Na_2SO_4, gentle evaporation).
4. **Safety reflection**

Note any **near-misses** (pressure in separatory funnel, splashes, ether vapors) and how controls can be strengthened in future runs (hood sash height, slower additions, more frequent venting).

References:

CHAPTER OVERVIEW

5: Thin-Layer Chromatography (TLC)

Learning Objectives

The Purpose of this Experiment is to:

- Separate analgesics with TLC and compute accurate Rf values.
- Optimize eluent polarity (e.g., hexanes/ethyl acetate) to obtain informative Rf values (≈0.2–0.8).
- Visualize spots using UV first; apply iodine/chemical stains only if needed (stains are typically destructive).

INTRODUCTION

Chromatography techniques operate on the principle that different compounds distribute themselves differently between two phases: a **stationary phase** and a **mobile phase**. In TLC, the stationary phase is a thin layer of a solid adsorbent—most commonly **silica gel** or **alumina**—coated onto a glass, plastic, or aluminum plate. These materials are **polar** and interact strongly with polar functional groups in organic molecules.

Thin-layer chromatography (TLC) is a fast, simple, and widely used analytical technique in organic chemistry that allows chemists to **separate**, **identify**, and **analyze** the components of a mixture. Because TLC requires only very small sample amounts and minimal equipment, it is commonly used in instructional laboratories to study the behavior of organic compounds and to introduce fundamental principles of chromatography.

The mobile phase in TLC is a **liquid solvent or solvent mixture**, called the *eluent*. When a TLC plate is placed upright in a developing chamber containing a shallow pool of eluent, the solvent rises up the plate by **capillary action**. As the solvent travels upward, it carries the sample components with it. However, because each compound interacts differently with the stationary and mobile phases, it moves at **different rates**, leading to separation.

In normal-phase TLC (the most common mode used in teaching laboratories), **less polar compounds** interact weakly with the polar stationary phase and spend more time dissolved in the mobile phase. As a result, they travel farther up the TLC plate. **More polar compounds**, by contrast, interact more strongly with the stationary phase and move more slowly. This difference in polarity is the primary factor governing separation in TLC.

Once development is complete, the solvent is allowed to evaporate, and the separated compounds appear as distinct **spots** on the plate. Because many organic compounds are colorless, the spots are typically visualized using a **short-wave ultraviolet (UV) lamp** or chemical visualization methods such as **iodine vapor** or staining solutions. Under UV light, compounds that absorb UV radiation appear as dark spots against a fluorescent background.

The results of a TLC analysis are expressed quantitatively using the **retention factor (R_f), or R_f** value. The R_f value is defined as the ratio of the distance traveled by a compound to the distance traveled by the solvent front:

$$R_f = \frac{distance\text{-}traveled\text{-}by\text{-}substance}{distance\text{-}traveled\text{-}by\text{-}solvent}$$

The R_f value is always between 0 and 1 and is characteristic of a compound **only under a specific set of conditions**, including the stationary phase, solvent system, and temperature. For this reason, comparisons between unknown and known compounds must be made using TLC plates developed **simultaneously** under identical conditions.

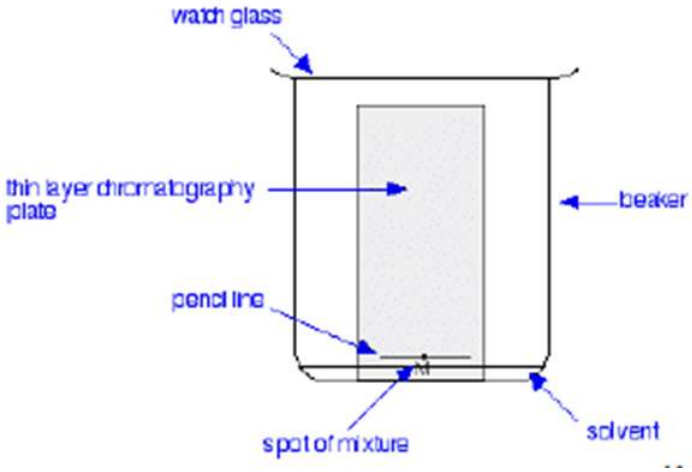
Thin-layer chromatography has many practical applications in the chemistry laboratory. It is commonly used to monitor the progress of chemical reactions, assess the purity of compounds, select appropriate solvent systems for column chromatography, and

identify unknown substances by comparison with known standards. In this experiment, TLC will be used in conjunction with melting-point measurements to identify the active ingredient in an unknown analgesic sample.

General Procedure for TLC

1. Spotting.

A pencil line is drawn parallel to the short side of the TLC plate 1.0 cm from the edge. Two or three points evenly spaced are marked on the line. The sample (~1mg) to be analyzed is placed in a 0.1mL conical vial and a few drops of solvent are added. A capillary tube is used to apply a small fraction of the solution from the vial to the TLC plate.



2. Developing

The chromatogram is performed by placing the spotted thin-layer plate in a wide-mouth jar or beaker with a watch glass cover, then adding a small amount of developing solvent. **The material spot on the TLC plate initially must be positioned above the solvent line.** The jar is quickly recapped or the watch glass replaced in order to maintain an atmosphere saturated with the developing solvent. The elution solvent rapidly ascends the plate by capillary action. The spotted material becomes eluted vertically up the plate. Development is interrupted when the solvent line reaches the top of the plate. The position of the solvent front should be quickly marked on the plate when the chromatogram is terminated.

3. Visualizing

If the compounds being chromatographed are colorless, visualization can be achieved by placing the plate in an iodine vapor chamber after the thin-layer plate has been removed from the developing chamber and allowed to stand for a few minutes. Organic materials readily absorb the iodine vapor to produce brown spots. On removal from the iodine chamber, the spots are marked by pencil because they will rapidly fade. The second widely used method is by placing the plate under an ultraviolet light. Since spots disappear when the light is removed, it is necessary to circle the spots with a pencil in order to have a permanent record of the chromatogram.

Once the separation of the components of the mixture is complete and the individual spots have been detected, the retention factor (Rf) of each compound may be calculated as shown below:

The Rf value for a compound is a physical constant for a given set of chromatographic conditions, so the adsorbent and the eluting solvent should be recorded along with the experimentally determined Rf values.

In this experiment, you will determine the Rf values of some commercially available analgesics or other compounds (identified by your instructor when conducting the experiment) and identify an unknown analgesic.

5.1: Thin-Layer Chromatography - Experiment
5.2: Thin-Layer Chromatography - Pre-lab
5.3: Thin-Layer Chromatography - Data and Report

5.1: Thin-Layer Chromatography - Experiment

SAFETY PRECAUTIONS

- Always wear safety goggles and gloves, and work in the fume hood.
- keep chambers covered; UV exposure, corrosive/oxidizing stains,
- Flammable/volatile eluents (hexanes, ethyl acetate, acetone),
- Collect spent eluents in appropriate hazardous waste (halogenated vs non-halogenated); discard stains/iodine as hazardous liquid waste; place used TLC plates in solid chemical waste; keep containers closed and labeled.

Equipment & Chemicals

TLC plates (silica), capillary spotters, covered chambers with filter-paper wick, UV lamp (254 nm), forceps, ruler, pencil; melting point apparatus and closed-end capillaries; standards (acetaminophen, aspirin, caffeine, ibuprofen); unknown solution; eluents (hexanes, ethyl acetate, 1:1 hexanes:EtOAc); iodine or approved stains as assigned.

Procedure

A. TLC (Find the Best Eluent)

1. Prepare three covered chambers (hexanes; ethyl acetate; 1:1 hexanes:EtOAc). Line each chamber with filter paper; ensure the solvent level is below the pencil baseline on the plate.

2. Draw a baseline ~1.0 cm from the bottom and a top mark ~1.0 cm from the top. Label lanes clearly in pencil.

3. Spot small (1–2 mm) dots of standards and the unknown on the baseline; allow to dry; re-spot to concentrate without spreading (keep ≥0.5 cm between spots and edges).

4. Develop each plate; remove when the solvent is ~5–10 mm below the top mark. Immediately mark the solvent front and dry the plate.

5. Visualize under 254 nm UV; circle spots. Apply iodine/chemical stain only if needed, then circle spots again.

6. Select the eluent that produced the clearest resolution and compute Rf for all analytes on that plate.

B. Melting Temperature (Standards and Unknown)

1. Finely powder the sample and load 3–4 mm into a closed-end capillary.

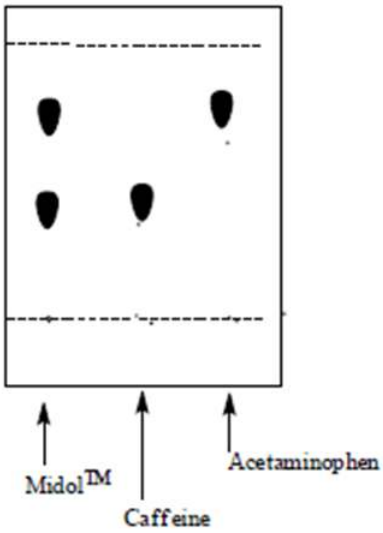
2. Heat rapidly to ~10 °C below the expected mp, then at 1–2 °C/min; record onset and clear-point (melting range). Cool before the next run.

3. Run acetaminophen, aspirin, caffeine, and ibuprofen; then run the unknown (quick estimate followed by a precise run).

5.2: Thin-Layer Chromatography - Pre-lab

Name: _____ Date: _____

1. A student conducted an experiment using the silica gel TLC plate shown below by spotting a sample of MidolTM, caffeine, and acetaminophen on the plate and eluting with petroleum ether: chloroform (9:1, v/v).



a. What are the Rf value of acetaminophen and of caffeine, respectively?

b. Based on this TLC analysis, what are the ingredients in a tablet of MidolTM?

c. Which one is more polar, caffeine or acetaminophen?

d. Another student accidentally used MidolPM in his experiment and observed only one spot. Speculate as to which spot was absent and explain.

2. Why must the spot be applied to the TLC plate above the level of the development solvent?

3. What will be the result of adding too much sample to the TLC plate?

4. Why must a pencil be used for drawing the lines and spotting?

5. Describe what an iodine vapor chamber is and explain how it works?

5.3: Thin-Layer Chromatography - Data and Report

Name: _____ Partner(s): _____ Date: _____

Data Tables

TLC (Best Eluent):

Sample	d_spot (cm)	d_front (cm)	Rf
Acetaminophen			
Aspirin			
Caffeine			
Ibuprofen			
Unknown			

Sample Calculations

Retention factor (Rf): Rf = distance(spot center from baseline) / distance(solvent front from baseline)

Example: If d_spot = 3.4 cm and d_front = 6.8 cm, then Rf = 3.4 / 6.8 = 0.50.

Melting Temperature Ranges (°C)

Sample	Trial 1	Trial 2
Acetaminophen		
Aspirin		
Caffeine		
Ibuprofen		
Unknown		

Post-Lab Analysis

• Identify the unknown using TLC Rf (co-run with standards) and melting range; justify both lines of evidence.

• Explain how changing the hexanes: EtOAc ratio would shift Rf values and affect separation.

• Discuss the main error sources (baseline submerged, oversized spots, unsaturated chamber, overrun solvent front, early staining) and how you mitigated them.

• Document safety controls and waste streams (solvents, stains/iodine, solid plates).

CHAPTER OVERVIEW

6: Column Chromatography

Learning Objectives

The purpose of this experiment is to:

- Objective 1
- Objective 2

6.1: Column Chromatography - Experiment
6.2: Column Chromatography - Pre-lab
6.3: Column Chromatography - Data and Report

6.1: Column Chromatography - Experiment

6.2: Column Chromatography - Pre-lab

Name: _____ Date: _____

6.3: Column Chromatography - Data and Report

Name: _____ Partner(s): _____ Date: _____

CHAPTER OVERVIEW

7: Stereochemistry

PURPOSE

The purpose of this experiment is to:

- Construct models in order to observe three-dimensional shapes of molecules.
- Draw structures of molecules, showing stereochemistry when appropriate.
- Assess stereochemical properties of molecules.

INTRODUCTION

In general chemistry, you learned that molecules adopt various three-dimensional shapes based on the electron geometry around the central atom. Once the shape of a molecule is known, its polarity can be determined. Based on whether a molecule is polar or nonpolar and how its atoms are bonded, one can identify the most important intermolecular forces present, which further influence the substance's physical properties.

Structure and shape also play a key role in organic molecules, leading to the formation of various types of isomers. Compounds with the same molecular formula but different arrangements of atoms (different bonding) are known as constitutional isomers. Constitutional isomers can have different physical and chemical properties, depending on the types of functional groups present. Another type of isomerism commonly observed in organic molecules is based on how groups are placed on specific carbon atoms. These are known as stereoisomers. In stereoisomers, the bonding is the same, but the three-dimensional shapes are different. In this experiment, you will explore the three-dimensional properties of organic molecules by constructing various models.

7.1: Stereochemistry - Experiment
7.2: Stereochemistry - Pre-lab
7.3: Stereochemistry - Data and Report

7.1: Stereochemistry - Experiment

SAFETY PRECAUTIONS

This will be a dry lab using molecular models. Be sure to neatly put all parts of the models back when finished, and clean your work area before leaving.

EQUIPMENT AND CHEMICALS NEEDED

Molecular model kit (will vary based on equipment availability and the type of model kits used; a 3D printer can also be substituted for a model kit or added in addition to one). In this case, your instructor will provide you with a procedure on how to operate the printer.)

EXPERIMENT

Students will draw, make models, and answer questions for the following molecules listed below based on the instructions given in the data and observation section

Part I: 1,2-dichlorocyclopropane:

Part II: 1,2-dichlorocyclopentane:

Part III: 2-bromo-3-chlorobutane:

Part IV: 2,3,4-tribromopentane:

Part V: Exploring the chirality of allenes (molecule with two consecutive C=C bonds)

7.2: Stereochemistry - Pre-lab

Name: _____ Date: _____

PRE-LAB QUESTIONS

1.) Describe the three important intermolecular forces present in substances.

2.) For each of the following molecules: H_2O, CH_2Cl_2, CCl_4, NH_3

Part A: Draw the Lewis structure.

Part B: Predict the three-dimensional shape.

Part C: Determine whether the molecule is polar or nonpolar.

Part D: Identify the most important intermolecular force present in the liquid phase of the molecule.

3.) Define the following terms:

Part A: Constitutional isomers

Part B: Conformational isomers

Part C: Stereoisomers

Part D: Enantiomers

Part E: Diastereomers

Part F: Meso compound

Part G: Chiral carbon atom

Part H: Chiral molecule

Part I: Achiral molecule

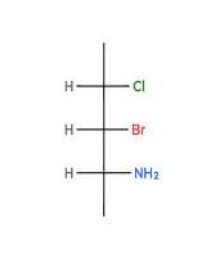
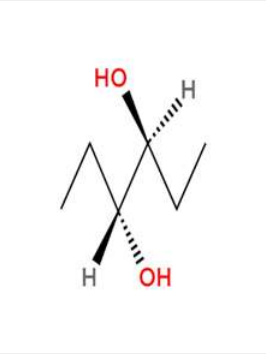
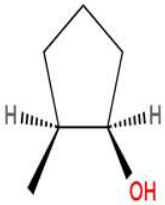
Part J: Resonance structures

4.) What is the 2^n rule, and how can it be used to determine the maximum number of stereoisomers possible for a compound?

5.) For each of the following molecules:

Part A: Identify any chiral carbon atoms as R or S.

Part B: Determine the maximum number of stereoisomers that can exist.

Molecule	Identify any chiral carbon atoms as R or S	Maximum number of stereoisomers
		
		
		
		

6.) Is the maximum number of stereoisomers always the same as the actual number of stereoisomers that can exist for a compound? Explain.

7.) Draw five constitutional isomers with molecular formula C_3H_6O.

7.3: Stereochemistry - Data and Report

Name: _____ Partner(s): _____ Date: _____

Part I: 1,2-dichlorocyclopropane

1) Draw the structure of 1,2-dichlorocyclopropane.

2) What is the maximum number of stereoisomers possible for this compound?

3) Construct a model for 1,2-dichlorocyclopropane in which both chlorine atoms are on the same side of the ring. Using dashes and wedges, draw the structure of this compound, and provide a name.

4) Consider the model constructed in step 3. Can you construct another model that would be a nonsuperimposable mirror image?

5) Based on the results of steps 3-4, would you expect this molecule to be chiral or achiral?

6) Construct a model for 1,2-dichlorocyclopropane in which the chlorine atoms are on opposite sides of the ring. Using dashes and wedges, draw the structure, and provide a name.

7) Consider the model constructed in step 6. Can you construct another model that would be a nonsuperimposable mirror image?

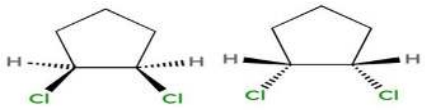
8) Based on the results of steps 6-7, would you expect this molecule to be chiral or achiral?

9) Based on the results of steps 1-8, draw all possible stereoisomers for 1,2-dichlorocyclopropane, and determine the relationship between each.

10) Draw and name a constitutional isomer of 1,2-dichlorocyclopropane.

Part II: 1,2-dichlorocyclopentane

1) Construct models for the following representations of 1,2-dichlorocyclopentane.



2) What is the relationship between these two structures?

3) How many chiral carbons are present in each structure?

4) Which of these structures (if any) are chiral?

5) True or false: Based on the results of this exercise, molecules with chiral carbon atoms can be achiral.

Part III: 2-bromo-3-chlorobutane

1) Draw the structure for 2-bromo-3-chlorobutane.

2) What is the maximum number of stereoisomers possible for this compound?

3) Using Fischer projections, draw all possible stereoisomers, and determine the relationship between each. (Construct models to assist you in drawing the stereoisomers.) Be sure to label all chiral carbon atoms as R or S.

4) Draw a constitutional isomer of 2-bromo-3-chlorobutane.

Part IV: 2,3,4-tribromopentane

1) Draw the structure for 2,3,4-tribromopentane.

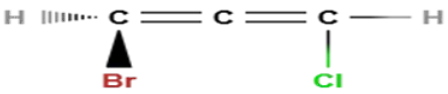
2) What is the maximum number of stereoisomers possible for this compound?

3) Using Fischer projections, draw all possible stereoisomers, and determine the relationship between each. (Construct models to assist you in drawing the stereoisomers.) Be sure to label all chiral carbon atoms as R or S.

4) Draw a constitutional isomer of 2,3,4-tribromopentane.

Part V: Exploring the chirality of allenes (molecule with two consecutive C=C bonds)

1.) Construct a model for the following compound:



2) Now, try constructing a model that is the nonsuperimposable mirror image. Were you able to do so?

3) How many chiral carbon atoms does this compound have?

4) Based on the results from steps 1-3, would this molecule be chiral or achiral? Explain.

5) True or false: Based on the results from this exercise, molecules may not have chiral carbon atoms but can still be chiral.

POST-LAB QUESTIONS

1.) Based on the results of this experiment, what is the sole criterion for a molecule to be labeled as chiral?

2) Refer to part I of the experiment. Was the actual number of stereoisomers the same as the number predicted using 2^n rule? Explain.

3) Refer to part II of the experiment. Which terms in prelab question 3 can be used to classify this compound?

4) Refer to part III of the experiment. Enter the name of each stereoisomer into the ChemSpider link provided. For example, you would enter (2S,3S)-2-bromo-3-chlorobutane into the search box. Once the structure appears, click on properties. Do this for each stereoisomer and determine which properties are the same and which are different. What does this tell you about properties of stereoisomers? https://www.chemspider.com/

5) Refer to part IV of the experiment. How does changing the position of groups on carbon 3 affect the stereochemistry of the compound?

6) Refer to part V of the experiment. Imagine the chlorine atom is replaced by another hydrogen atom. Would this affect the molecule's chirality? Explain.

7) What is the difference between R/S and +/- notations for organic molecules?

CHAPTER OVERVIEW

8: Gas Chromatography

Learning Objectives

The purpose of this experiment is to:

- Objective 1
- Objective 2

8.1: Gas Chromatography - Experiment
8.2: Gas Chromatography - Pre-lab
8.3: Gas Chromatography - Data and Report

8.1: Gas Chromatography - Experiment

8.2: Gas Chromatography - Pre-lab

Name: _____ Date: _____

8.3: Gas Chromatography - Data and Report

Name: _____ Partner(s): _____ Date: _____

CHAPTER OVERVIEW

9: Polarimetry

Learning Objectives

The purpose of this experiment is to:

- Objective 1
- Objective 2

9.1: Polarimetry - Experiment
9.2: Polarimetry - Pre-lab
9.3: Polarimetry - Data and Report

9.1: Polarimetry - Experiment

9.2: Polarimetry - Pre-lab

Name: _____ Date: _____

9.3: Polarimetry - Data and Report

Name: _____ Partner(s): _____ Date: _____

CHAPTER OVERVIEW

10: Synthesis of 1-bromobutane

PURPOSE

The purpose of this experiment is to:

- Set up a reflux apparatus.
- Convert 1-butanol to 1-bromobutane.
- Analyze the product using chemical tests.

INTRODUCTION

Under certain conditions, primary alcohols can be converted into primary alkyl halides. In this experiment, 1-bromobutane will be synthesized by the S_N2 reaction between 1-butanol and sodium bromide. In the first step of the mechanism, the alcohol is protonated to form a water molecule, which serves as a good leaving group. Then the iodide ion will attack the carbon atom as the water molecule leaves.

The reaction will be run under reflux conditions, a safe method for carrying out organic syntheses. Reflux involves attaching a round-bottom flask to a condenser, leaving it open to avoid pressure buildup. Cold water circulates from the bottom to the top of the condenser, allowing the reactants to be heated without evaporating.

Once the product is synthesized, it will be characterized using the sodium iodide reagent, a chemical test for primary or secondary alkyl halides. When the reagent is added to an unknown, the formation of a precipitate indicates the presence of a halogen. The Beilstein test will also be used to test for the presence of an alkyl halide. This chemical test involves placing a copper wire coated with the sample into a flame. A green flame indicates the presence of a halogen.

10.1: Synthesis of 1-bromobutane - Experiment
10.2: Synthesis of 1-bromobutane - Pre-lab
10.3: Synthesis of 1-bromobutane - Data and Report

10.1: Synthesis of 1-bromobutane - Experiment

SAFETY PRECAUTIONS

1. Wear chemical splash goggles throughout the experiment.
2. Gloves are provided if you wish to wear them.
3. Work with all chemicals under the fume hood.
4. Be careful when handling hot glassware.
5. Dispose of all waste in the appropriate waste containers.
6. Clean your work area (hood and lab bench) and return all glassware and equipment to the appropriate place once you finish the experiment.
7. Always wash your hands immediately upon leaving the lab.

EQUIPMENT AND CHEMICALS NEEDED

• Reflux apparatus (100 mL round-bottom flask, round-bottom flask holder, condenser, two rubber tubes, heating mantle)

• Deionized water

• 10 mL graduated cylinder

• 50 mL beaker

• 250 mL beaker

• Separatory funnel

• Ring stand and ring clamp

• Ice

• Milligram balance

• Fume hood

• Saturated sodium bicarbonate solution

• Three test tubes

• 15% sodium iodide in acetone solution

• Copper wire

• Bunsen burner

• Halogenated organic waste container

• 1-butanol

• Sodium bromide

• Concentrated sulfuric acid

EXPERIMENTAL PROCEDURE

1) Add around 17.0 g of sodium bromide, 10.0 mL of 1-butanol, and 17.0 mL of deionized water to a 100 mL round-bottom flask. Be sure the flask is securely held so it does not tip over.

2) Place the round-bottom flask in a 250 mL beaker containing ice, and slowly add 14.0 mL of concentrated sulfuric acid to the mixture. Be sure this step takes place under a working fume hood.

3) Assemble a reflux apparatus under the hood. Your instructor will demonstrate how to set this up based on the availability of equipment specific to your lab. Once your instructor approves the setup, reflux the mixture for 1 hour.

4) After the reflux is complete, turn off the heat, but leave the water running. Once the glassware is cool, turn off the water, disassemble the reflux apparatus, and pour the contents of the round-bottom flask into a separatory funnel. (Note: If any solid is remaining in the round-bottom flask, avoid transferring it to the separatory funnel.) Attach the separatory funnel to a ring clamp on a ring stand and allow the solution layers to separate.

5) Drain the lower layer into a 250 mL beaker. This layer can then be discarded into the halogenated organic waste container. **Be sure to save the top layer still remaining in the separator funnel.**

6) Add 10 mL of saturated sodium bicarbonate solution to the mixture remaining in the separatory funnel. Shake and vent the funnel (or stir the layers for a few minutes). Drain the bottom layer into a pre-weighed 50 mL beaker. **This layer should be saved, as it contains the product.** The layer remaining in the separatory funnel can be discarded into the halogenated organic waste container.

7) Determine the mass of the product, and calculate the theoretical and percent yield.

8) Obtain three test tubes. Place approximately 1 mL of the following into the test tubes:

Test tube 1 = 1-butanol

Test tube 2 = 1-bromobutane

Test tube 3 = product

To each test tube, add 1 mL of 15% sodium iodide in acetone solution. Record your observations.

9) Set up a Bunsen burner under a fume hood. **Be sure all organic reagents have been removed from the hood before lighting the burner.** Place the tip of an approximately 25 cm strip of copper wire into the flame for 10 seconds. Then, place the tip of the wire into the solution containing the product. Hold the tip of the wire in the flame and record your observations. For comparison, repeat the test on 1-butanol and 1-bromobutane. (Note: You can use the same wire.)

10.2: Synthesis of 1-bromobutane - Pre-lab

Name: _____ Date: _____

10.3: Synthesis of 1-bromobutane - Data and Report

Name: _____ Partner(s): _____ Date: _____

CHAPTER OVERVIEW

11: Synthesis of t-Butyl Chloride

PURPOSE

The purpose of this experiment is to:

- Convert t-butyl alcohol to t-butyl chloride
- Analyze the product using chemical tests.

INTRODUCTION

Under certain conditions, tertiary alcohols can be converted into tertiary alkyl halides. In this experiment, t-butyl chloride will be synthesized by the S_N1 reaction between t-butyl alcohol and hydrochloric acid. In the first step of the mechanism, the alcohol will be protonated, generating a water molecule, which serves as a good leaving group. Once water leaves, the chloride ion will attack the resulting tertiary carbocation, forming the product.

The reactants will be mixed in a separatory funnel, and once two layers form, the product will be isolated. The silver nitrate reagent will then be used to test for the presence of a tertiary alkyl halide. If such a halogen is present in an unknown, it will react with silver nitrate to form a precipitate. The Beilstein test will also be used to identify an alkyl halide. This chemical test involves placing a copper wire coated with the sample into a flame. A green flame indicates the presence of a halogen.

11.1: Synthesis of t-Butyl Chloride - Experiment
11.2: Synthesis of t-Butyl Chloride - Pre-lab
11.3: Synthesis of t-Butyl Chloride - Data and Report

11.1: Synthesis of t-Butyl Chloride - Experiment

SAFETY PRECAUTIONS

1. Wear chemical splash goggles throughout the experiment.
2. Gloves are provided if you wish to wear them.
3. Work with all chemicals under the fume hood.
4. Pay special attention while working with concentrated hydrochloric acid, as it fumes. Do not open the bottle of hydrochloric acid unless it is under a working fume hood.
5. Dispose of all waste in the appropriate waste containers.
6. Clean your work area (hood and lab bench) and return all glassware and equipment to the appropriate place once you finish the experiment.
7. Always wash your hands immediately upon leaving the lab.

EQUIPMENT AND CHEMICALS NEEDED

- t-butyl alcohol
- t-butyl chloride
- Concentrated hydrochloric acid
- Saturated sodium bicarbonate solution
- Saturated sodium chloride solution
- 1% ethanol silver nitrate solution
- Ice
- 10 mL graduated cylinder
- 100 mL graduated cylinder
- Separatory funnel
- Ring stand and ring clamp
- Milligram balance
- Fume hood
- 50 mL beaker
- 250 mL beaker
- 50 mL Erlenmeyer flask
- 3 test tubes
- Glass stirring rod
- Copper wire
- Bunsen burner
- Halogenated organic waste container

EXPERIMENTAL PROCEDURE

1) Under the hood, add 15 mL of concentrated hydrochloric acid to a 50 mL Erlenmeyer flask. Nestle the flask containing the acid in a 250 mL beaker filled with ice.

2) Place an empty 50 mL beaker on a milligram balance, and press the tare button to zero out the balance. Add approximately 4 g of t-butyl alcohol to the beaker. Record the exact mass in the data table.

3.) Place a separatory funnel into a ring clamp on a ring stand under the hood. Make sure the valve on the funnel is closed. Carefully add the cooled hydrochloric acid, followed by the t-butyl alcohol, to the separatory funnel. Using a glass stirring rod, stir the mixture for 20 minutes.

4) Allow the layers to separate. Once two layers are visible, drain the bottom layer into a 250 mL beaker and discard it in the halogenated organic waste container. Save the layer remaining in the separatory funnel, as it contains the product.

5) Slowly add 30.0 mL of saturated sodium bicarbonate solution to the separatory funnel. Stir the resulting solution for 5 minutes, noting any gas evolution. Allow the layers to separate. Once two layers are visible, drain the bottom layer into a 250 mL beaker and discard it in the halogenated organic waste container. Save the layer remaining in the separatory funnel, as it contains the product.

6) Repeat step 5 using 30 mL of saturated sodium chloride solution. Again, save the upper layer, as it contains the final product.

7) Pour the remaining layer in the separatory funnel into a pre-weighed 50 mL beaker. Determine the mass of the product, and calculate the theoretical and percent yield.

8) Obtain three test tubes. Place approximately 1 mL of the following into the test tubes:

Test tube 1 = t-butyl alcohol

Test tube 2 = t-butyl chloride

Test tube 3 = product

To each test tube, add 1 mL of 1% ethanolic silver nitrate solution. Record your observations.

9) Set up a Bunsen burner under a fume hood. **Be sure all organic reagents have been removed from the hood before lighting the burner.** Place the tip of an approximately 25.0 cm strip of copper wire into the flame for 10 seconds. Then, place the tip of the wire into the solution containing the product. Hold the tip of the wire in the flame and record your observations. For comparison, repeat the test on t-butyl alcohol and t-butyl chloride. (Note: You can use the same wire.)

11.2: Synthesis of t-Butyl Chloride - Pre-lab

Name: _____ Date: _____

1) Write the reaction and full mechanism for the synthesis occurring in this experiment.

2) Which of the following compounds should show a positive test result with 1% ethanolic silver nitrate solution?

t-butyl alcohol:

t-butyl chloride:

Deionized water:

2-chloropropane:

1-chloro-1-methylcyclopentane:

3) What is the Beilstein test? How can it be used to infer the presence of an alkyl halide?

4) Why is the reaction in this experiment run in a separatory funnel?

11.3: Synthesis of t-Butyl Chloride - Data and Report

Name: _____ Partner(s): _____ Date: _____

DATA AND OBSERVATIONS

Synthesis of t-butyl chloride

Mass of t-butyl alcohol =

Volume of concentrated hydrochloric acid =

Mass of an empty 50.0 mL beaker =

Mass of beaker and product =

Mass of product =

Theoretical yield of product (show work):

Percent yield of product (show work):

Silver Nitrate Test

Reagent	Observations
t-butyl alcohol	
t-butyl chloride	
Product	

Beilstein Test

Reagent	Observations
t-butyl alcohol	
t-butyl chloride	
Product	

POST-LAB QUESTIONS

1) Summarize the results of this experiment. Based on the results, did you successfully synthesize the correct product?

2) What are some sources of error associated with this synthesis? How can they have been minimized?

3) Explain why this reaction takes place by the S_N1 mechanism as opposed to the S_N2 mechanism.

4) What is the purpose of adding saturated solutions of sodium bicarbonate and sodium chloride to the product?

5) What other tests or procedures can be used to positively confirm the identity of the product?

CHAPTER OVERVIEW

12: Synthesis of Cyclohexene

This page is a draft and is under active development.

PURPOSE

The purpose of this experiment is to:

- Perform an alcohol dehydration reaction in order to convert cyclohexanol to cyclohexene.
- Use chemical tests to differentiate between the reactant and product.

INTRODUCTION

Alcohols can be dehydrated in the presence of an acid catalyst to form alkenes. This reaction follows the E1 mechanism. If multiple products form, the major product will be the one in which the double bond is more highly substituted, as explained by Zaitsev's rule. In this experiment, cyclohexanol will be dehydrated to form cyclohexene. The reaction will be run using fractional distillation. Once the product is collected, its mass and percent yield will be determined.

The product will be further analyzed using chemical tests, which can often detect the presence of a specific functional group. The first chemical test involves adding the bromine reagent to the product. This test can be used to identify an alkene or alkyne. 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. 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 forms. A negative test would be indicated by the persistence of the purple solution.

The chromic acid test, which detects primary and secondary alcohols, will also be used to infer the product's identity. In the presence of the chromic acid reagent, primary alcohols are oxidized to carboxylic acids, and secondary alcohols are oxidized to ketones. 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. For comparison, the three chemical tests will be run on both the reactant and product.

12.1: Synthesis of Cyclohexene - Experiment
12.2: Synthesis of Cyclohexene - Pre-lab
12.3: Synthesis of Cyclohexene - Data and Report

12.1: Synthesis of Cyclohexene - Experiment

SAFETY PRECAUTIONS

1. Wear chemical splash goggles throughout the experiment.
2. Gloves are provided if you wish to wear them.
3. Work with all chemicals under the fume hood.
4. Cyclohexene has a very irritating odor. Only work with this compound under a working fume hood.
5. When transferring cyclohexene from the fume hood to the lab bench or balance, be sure it is in a sealed vial, beaker/test tube covered with Parafilm.
6. Perform the bromine test only under the fume hood, as bromine is toxic. Do not open the bottle of bromine or work with it outside the fume hood.
7. Be careful when handling hot glassware.
8. Dispose of all waste in the appropriate waste containers.
9. Clean your work area (hood and lab bench) and return all glassware and equipment to the appropriate place once you finish the experiment.
10. Always wash your hands immediately upon leaving the lab.

EQUIPMENT AND CHEMICALS NEEDED

- Fractional distillation (with column packing) apparatus
- 50 mL beaker
- 10 mL graduated cylinder
- 3 test tubes
- Boiling chips
- Cyclohexanol
- Cyclohexene
- 85% phosphoric acid
- Anhydrous sodium sulfate
- 1% bromine solution (in methylene chloride)
- 1% potassium permanganate solution
- 2% chromic acid reagent
- Ice
- Halogenated organic waste container
- Non-halogenated organic waste container

EXPERIMENTAL PROCEDURE

1. Place 15.0 mL of cyclohexanol, 5.0 mL of 85% phosphoric acid, and a boiling chip in a 100 mL round-bottom flask. Under the fume hood, set up a fractional distillation apparatus. Make sure to use a packed distilling column. Also, place the receiver flask in a beaker of ice to prevent the collected cyclohexene from evaporating. Have your instructor check your final setup. Once your setup is approved, start the distillation process.

2. The cyclohexene should collect around 80-85 °C. Record the boiling range in the data table. Do not distill to dryness.

3. After the reaction is complete, turn off the heat, keep the water in the condenser running, and allow the setup to cool. Add a scoop of anhydrous sodium sulfate to your collected product, and decant the product into a pre-weighed 50 mL beaker. Find the

mass of your product, and record it in the data table. From there, calculate the theoretical and percent yields.

4. Obtain 3 test tubes. Add 10 drops of cyclohexanol to test tube 1, 10 drops of cyclohexene to test tube 2, and 10 drops of your product to test tube 3. To each test tube, add 3 drops of 1% bromine solution, and record your observations in the data table. Pour the contents of the test tubes into the halogenated organic waste container. Clean and dry the test tubes and save them for step 5.

5. Repeat step 4 with 3 clean test tubes, adding 3 drops of 1% potassium permanganate solution (instead of 1% bromine solution). Record your observations in the data table. Pour the contents of the test tubes into the non-halogenated waste container. Clean and dry the test tubes and save them for step 6.

6. Repeat step 4 with 3 clean test tubes, adding 3 drops of the chromic acid solution. Record your observations in the data table. Pour the contents of the test tubes into the non-halogenated organic waste container.

7. Clean and dry all glassware/equipment, and return everything to its appropriate place.

12.2: Synthesis of Cyclohexene - Pre-lab

Name: _____ Date: _____

1) Write the overall reaction and mechanism for the conversion of cyclohexanol to cyclohexene.

2) Describe what one would observe for both a positive and a negative test result for each of the following:

Part A: Bromine Test

Part B: Potassium Permanganate Test

Part C: Chromic acid test

3) Determine whether the following compounds would have a positive or negative result with each of the following chemical tests.

Chemical Tests

Compound	Bromine test	Potassium Permanganate Test	Chromic acid test
1-hexene			
Pentane			
2-hexanol			
Methylcyclopentanol			

12.3: Synthesis of Cyclohexene - Data and Report

Name: _____ Partner(s): _____ Date: _____

DATA AND OBSERVATIONS

Volume of cyclohexanol =

Volume of phosphoric acid =

Boiling range of collected product =

Mass of empty 50 mL beaker =

Mass of 50 mL beaker and product =

Mass of product =

Theoretical yield of product (show calculation):

Percent yield of product (show calculation):

Chemical Tests

Chemical Test	Cyclohexanol	Cyclohexene	Product
Bromine Test			
Potassium Permanganate Test			
Chromic Acid Test			

POST-LAB QUESTIONS

1) How does the boiling range of your collected product compare to the actual boiling point of cyclohexane?

2) Based on the results of the experiments, did you synthesize the correct product? What evidence supports (or does not support) this?

3) Explain why fractional distillation (as opposed to reflux) was used to carry out this synthesis.

4) What was the purpose of adding anhydrous sodium sulfate to the collected product?

CHAPTER OVERVIEW

13: Appendix

Index

A

American Chemical Society
- 0.1: Lab Safety Videos

C

chemical disposal
- 0.3: Lab Safety Quiz

chemical exposure
- 0: Lab Safety Orientation

chemical spills
- 0.3: Lab Safety Quiz

Chemistry Division
- 0.2: Lab Safety Contract

crash course safety
- 0.1: Lab Safety Videos

E

education
- 0.1: Lab Safety Videos

emergency equipment
- 0: Lab Safety Orientation

emergency procedures
- 0.2: Lab Safety Contract
- 0.3: Lab Safety Quiz

F

first aid
- 0.3: Lab Safety Quiz

H

hazardous materials
- 0.2: Lab Safety Contract

L

lab attire
- 0.3: Lab Safety Quiz

lab conduct
- 0.2: Lab Safety Contract

lab safety
- 0: Lab Safety Orientation
- 0.1: Lab Safety Videos
- 0.2: Lab Safety Contract
- 0.3: Lab Safety Quiz

laboratory procedures
- 0.2: Lab Safety Contract

laboratory regulations
- 0.3: Lab Safety Quiz

laboratory safety
- 0: Lab Safety Orientation
- 0.1: Lab Safety Videos

P

personal protective equipment
- 0.3: Lab Safety Quiz

R

responsible behavior
- 0.2: Lab Safety Contract

S

safety attire
- 0: Lab Safety Orientation

safety contract
- 0: Lab Safety Orientation
- 0.1: Lab Safety Videos

safety equipment
- 0.2: Lab Safety Contract
- 0.3: Lab Safety Quiz

safety goggles
- 0: Lab Safety Orientation
- 0.3: Lab Safety Quiz

safety guidelines
- 0.2: Lab Safety Contract

safety orientation
- 0.1: Lab Safety Videos

safety quiz
- 0: Lab Safety Orientation
- 0.1: Lab Safety Videos

safety rules
- 0: Lab Safety Orientation

safety techniques
- 0.1: Lab Safety Videos

student safety
- 0: Lab Safety Orientation

T

Triton College
- 0.2: Lab Safety Contract

Glossary

Sample Word 1 | Sample Definition 1

Detailed Licensing

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Webpages: 65

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    - InfoPage - *CC BY 4.0*
    - Table of Contents - *CC BY 4.0*
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  - 0: Lab Safety Orientation - *CC BY 4.0*
    - 0.1: Lab Safety Videos - *CC BY 4.0*
    - 0.2: Lab Safety Contract - *CC BY 4.0*
    - 0.3: Lab Safety Quiz - *CC BY 4.0*
    - 0.4: Additional Resources - *CC BY 4.0*
  - 1: Recrystallization - *CC BY 4.0*
    - 1.1: Recrystallization - Experiment - *CC BY 4.0*
    - 1.2: Recrystallization - Pre-lab - *CC BY 4.0*
    - 1.3: Recrystallization - Data and Report - *CC BY 4.0*
  - 2: Identification of an Unknown Using Simple Distillation - *CC BY 4.0*
    - 2.1: Identification of an Unknown - Experiment - *CC BY 4.0*
    - 2.2: Identification of an Unknown - Pre-lab - *CC BY 4.0*
    - 2.3: Identification of an Unknown - Data and Report - *CC BY 4.0*
  - 3: Fractional Distillation - *CC BY 4.0*
    - 3.1: Fractional Distillation - Experiment - *CC BY 4.0*
    - 3.2: Fractional Distillation - Pre-lab - *CC BY 4.0*
    - 3.3: Fractional Distillation - Data and Report - *CC BY 4.0*
  - 4: Extraction - *CC BY 4.0*
    - 4.1: Extraction - Experiment - *CC BY 4.0*
    - 4.2: Extraction - Pre-lab - *CC BY 4.0*
    - 4.3: Extraction - Data and Report - *CC BY 4.0*
  - 5: Thin-Layer Chromatography (TLC) - *CC BY 4.0*
    - 5.1: Thin-Layer Chromatography - Experiment - *CC BY 4.0*
    - 5.2: Thin-Layer Chromatography - Pre-lab - *CC BY 4.0*
    - 5.3: Thin-Layer Chromatography - Data and Report - *CC BY 4.0*
  - 6: Column Chromatography - *CC BY 4.0*
    - 6.1: Column Chromatography - Experiment - *CC BY 4.0*
    - 6.2: Column Chromatography - Pre-lab - *CC BY 4.0*
    - 6.3: Column Chromatography - Data and Report - *CC BY 4.0*
  - 7: Stereochemistry - *CC BY 4.0*
    - 7.1: Stereochemistry - Experiment - *CC BY 4.0*
    - 7.2: Stereochemistry - Pre-lab - *CC BY 4.0*
    - 7.3: Stereochemistry - Data and Report - *CC BY 4.0*
  - 8: Gas Chromatography - *CC BY 4.0*
    - 8.1: Gas Chromatography - Experiment - *CC BY 4.0*
    - 8.2: Gas Chromatography - Pre-lab - *CC BY 4.0*
    - 8.3: Gas Chromatography - Data and Report - *CC BY 4.0*
  - 9: Polarimetry - *CC BY 4.0*
    - 9.1: Polarimetry - Experiment - *CC BY 4.0*
    - 9.2: Polarimetry - Pre-lab - *CC BY 4.0*
    - 9.3: Polarimetry - Data and Report - *CC BY 4.0*
  - 10: Synthesis of 1-bromobutane - *CC BY 4.0*
    - 10.1: Synthesis of 1-bromobutane - Experiment - *CC BY 4.0*
    - 10.2: Synthesis of 1-bromobutane - Pre-lab - *CC BY 4.0*
    - 10.3: Synthesis of 1-bromobutane - Data and Report - *CC BY 4.0*
  - 11: Synthesis of t-Butyl Chloride - *CC BY 4.0*
    - 11.1: Synthesis of t-Butyl Chloride - Experiment - *CC BY 4.0*
    - 11.2: Synthesis of t-Butyl Chloride - Pre-lab - *CC BY 4.0*

- 11.3: Synthesis of t-Butyl Chloride - Data and Report - *CC BY 4.0*
- 12: Synthesis of Cyclohexene - *CC BY 4.0*
  - 12.1: Synthesis of Cyclohexene - Experiment - *CC BY 4.0*
  - 12.2: Synthesis of Cyclohexene - Pre-lab - *CC BY 4.0*
  - 12.3: Synthesis of Cyclohexene - Data and Report - *CC BY 4.0*
- 13: Appendix - *CC BY 4.0*
- Back Matter - *CC BY 4.0*
  - Index - *CC BY 4.0*
  - Glossary - *CC BY 4.0*
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    - InfoPage - *CC BY 4.0*
    - Table of Contents - *CC BY 4.0*
    - Licensing - *CC BY 4.0*
  - 0: Lab Safety Orientation - *CC BY 4.0*
    - 0.1: Lab Safety Videos - *CC BY 4.0*
    - 0.2: Lab Safety Contract - *CC BY 4.0*
    - 0.3: Lab Safety Quiz - *CC BY 4.0*
    - 0.4: Additional Resources - *CC BY 4.0*
  - 1: Recrystallization - *CC BY 4.0*
    - 1.1: Recrystallization - Experiment - *CC BY 4.0*
    - 1.2: Recrystallization - Pre-lab - *CC BY 4.0*
    - 1.3: Recrystallization - Data and Report - *CC BY 4.0*
  - 2: Identification of an Unknown Using Simple Distillation - *CC BY 4.0*
    - 2.1: Identification of an Unknown - Experiment - *CC BY 4.0*
    - 2.2: Identification of an Unknown - Pre-lab - *CC BY 4.0*
    - 2.3: Identification of an Unknown - Data and Report - *CC BY 4.0*
  - 3: Fractional Distillation - *CC BY 4.0*
    - 3.1: Fractional Distillation - Experiment - *CC BY 4.0*
    - 3.2: Fractional Distillation - Pre-lab - *CC BY 4.0*
    - 3.3: Fractional Distillation - Data and Report - *CC BY 4.0*
  - 4: Extraction - *CC BY 4.0*
    - 4.1: Extraction - Experiment - *CC BY 4.0*
    - 4.2: Extraction - Pre-lab - *CC BY 4.0*
    - 4.3: Extraction - Data and Report - *CC BY 4.0*
  - 5: Thin-Layer Chromatography (TLC) - *CC BY 4.0*
    - 5.1: Thin-Layer Chromatography - Experiment - *CC BY 4.0*
    - 5.2: Thin-Layer Chromatography - Pre-lab - *CC BY 4.0*
    - 5.3: Thin-Layer Chromatography - Data and Report - *CC BY 4.0*
  - 6: Column Chromatography - *CC BY 4.0*
    - 6.1: Column Chromatography - Experiment - *CC BY 4.0*
    - 6.2: Column Chromatography - Pre-lab - *CC BY 4.0*
    - 6.3: Column Chromatography - Data and Report - *CC BY 4.0*
  - 7: Stereochemistry - *CC BY 4.0*
    - 7.1: Stereochemistry - Experiment - *CC BY 4.0*
    - 7.2: Stereochemistry - Pre-lab - *CC BY 4.0*
    - 7.3: Stereochemistry - Data and Report - *CC BY 4.0*
  - 8: Gas Chromatography - *CC BY 4.0*
    - 8.1: Gas Chromatography - Experiment - *CC BY 4.0*
    - 8.2: Gas Chromatography - Pre-lab - *CC BY 4.0*
    - 8.3: Gas Chromatography - Data and Report - *CC BY 4.0*
  - 9: Polarimetry - *CC BY 4.0*
    - 9.1: Polarimetry - Experiment - *CC BY 4.0*
    - 9.2: Polarimetry - Pre-lab - *CC BY 4.0*
    - 9.3: Polarimetry - Data and Report - *CC BY 4.0*
  - 10: Synthesis of 1-bromobutane - *CC BY 4.0*
    - 10.1: Synthesis of 1-bromobutane - Experiment - *CC BY 4.0*
    - 10.2: Synthesis of 1-bromobutane - Pre-lab - *CC BY 4.0*
    - 10.3: Synthesis of 1-bromobutane - Data and Report - *CC BY 4.0*
  - 11: Synthesis of t-Butyl Chloride - *CC BY 4.0*
    - 11.1: Synthesis of t-Butyl Chloride - Experiment - *CC BY 4.0*
    - 11.2: Synthesis of t-Butyl Chloride - Pre-lab - *CC BY 4.0*

- 11.3: Synthesis of t-Butyl Chloride - Data and Report - *CC BY 4.0*
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  - 12.1: Synthesis of Cyclohexene - Experiment - *CC BY 4.0*
  - 12.2: Synthesis of Cyclohexene - Pre-lab - *CC BY 4.0*
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