NATIONAL OPEN UNIVERSITY OF NIGERIA

SCHOOL OF SCIENCE AND TECHNOLOGY

COURSE CODE: CHM 192

COURSE TITLE: PRACTICAL CHEMISTRY II

CHM 192

INTRODUCTORY PRACTICAL CHEMISTRY II

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COURSE GUIDE

Introduction

Practical organic chemistry instruction has a purpose beyond the apparent one of providing practical training essential to the prospective chemist. In affording an opportunity for the student to become acquainted with a number of representative organic compounds, to observe their special
properties and characteristic behaviours, and to have some experience with the methods of handling them, such work forms a supplement to a lecture course (CHM 102 – Introductory Organc Chemistry I) which is essential to a full understanding and appreciation of the subject. With thought and study, much information regarding the general theory of the carbon compounds can be gained in the organic laboratory, and there is in addition ample opportunity for acquiring manipulative skill and dexterity. Organic compounds present such an interesting array of properties and reactions, and the methods of manipulation are so ingenious, that work in the organic laboratory is usually found to be a stimulating experience.

Organic chemistry can be fun, and hopefully this will be proved to you in this course. The work in this laboratory course will teach you a lot. The personal satisfaction that comes with performing an experiment skilfully and successfully will be great. To get the most out of this laboratory course, you should strive to do several things.
First, you must read carefully the safety tips at the beginning of the material. Secondly, you need to understand the organization of the course material and how to use it effectively. Thirdly, you must try to understand both the purpose and the principles behind each experiment you do. Finally, you must try to organize your time effectively during the lab period.

Course description

Experiments in this course are coordinated with the lecture topics in CHM 102. This course will introduce you to various techniques used in the organic laboratory as well as synthetic procedures and some methods of identification and purification of organic compounds.

The contents of this course material illustrate important concepts and principles in general organic chemistry. Three basic goals guided the development of all experiments in this material: (1) the experiments illustrate the concepts learned in the classroom; (2) the experiments are clearly and concisely written so that students will easily understand the task at hand, will work with minimal supervision because the manual provides enough information on experimental procedures, and will be able to perform the experiments in a 2-1/2 hour laboratory period; and (3) the experiments are not only simple demonstrations, but also contain a sense of discovery. In addition, laboratory safety tips as well as common laboratory techniques are explored.

Module 1 consists of four units that introduce you to laboratory safety rules and a series of detailed instructions and explanations dealing with the basic laboratory techniques of organic chemistry.

Module 2 contains experiments that introduce you to the techniques used in the isolation and purification of organic compounds

Module 3 consists of experimental methods for the determination of physical constants (e.g., melting point) of organic compounds.

Module 4 is devoted to experiments that illustrate important organic reactions.

What you will learn in this course

The main purpose of an organic laboratory course is to teach you the techniques necessary for a person dealing with organic chemicals. You will also learn the techniques needed for separating and purifying organic compounds. You will learn how to identify unknown compounds; the experiments are only the vehicles learning these techniques.

Besides good laboratory techniques and methods of carrying out basic laboratory procedures, other things you will learn from this laboratory course are:

- How to take data carefully
- How to record relevant observations
- How to plan for the isolation and purification of substances
- How to work safely
- How to solve problems and think like a chemist.

Course Aims

The aim of this course is to introduce you to the majority of techniques used within an organic laboratory and to demonstrate how these techniques are used when carrying out organic experiments.
The course is also aimed at familiarizing students with the main techniques encountered in organic chemistry lab such as extraction, recrystallization, simple and fractional distillation, identification of functional groups, conduct chemical reactions. Emphasis is placed on the theory of these techniques.

Objectives

Specific objectives are stated at the beginning of respective units in the course material. However, after going through the modules, you should be able to:

- Apply the basic safety rules in the organic chemistry laboratory
- Identify and use the common laboratory apparatuses and equipment
- Demonstrate the basic organic laboratory techniques
- Design and perform experiments for the isolation and purification of organic compounds
- Design and perform experiments for the determination of physical constants of organic compounds
- Carry out simple experiments illustrating the identities and reactions of organic compounds

Working Through This Course

The course is written in four modules subdivided into various units. It is required that the learner studies the units in details and attends laboratory sessions as the appointed time and venue.

Course Materials

Items to be made available to each learner are:

1. Course Guide
2. Study Units in three modules
   Lists of reference materials and texts for further reading have also been given at the end of each unit.

Study Units

The followings are the units contained in this course:

- Module 1
  Unit 1: Safety in the laboratory
  Unit 2: Common laboratory apparatus
  Unit 3: Basic laboratory techniques
  Unit 4: Weighing in the laboratory

- Module 2
  Unit 1: Distillation – Separation of a mixture
  Unit 2: Purification – Recrystallization of benzoic acid
  Unit 3: Extraction – Determination of distribution coefficient

- Module 3
  Unit 1: Measurement and density
  Unit 2: Micro Method Determination of Boiling Point of Hydrocarbons
  Unit 3: Melting point determination

- **Module 4**
  Unit 1: Structures of Hydrocarbons - Experiment with Models
  Unit 2: Reactions of Hydrocarbons - Properties and Identification of Hydrocarbons
  Unit 3: Alkene Synthesis from Alcohol - Preparation of Cyclohexene From Cyclohexanol

Sources of Information

- http://swc2.hccs.edu/pahlavan/
- Arthur Vogel, "Vogel's Practical Organic Chemistry", 4th ed., Longman Inc., New York, 1978.
- Fessenden, R.J; Fessenden, J.S and Landgrebe, J.A, Organic Laboratory Techniques. 2nd ed. Brooks/Cole Publishing Company, 1984.
- Steere, N.V., *Handbook of Laboratory Safety.* 2nd ed. CRC Press, Cleveland, Ohio, 1971.

- Green, M.E. and Turk, A, *Safety in Working with Chemicals.* Macmillan, New York, 1978.
- Williamson, K.L, *Macroscale and Microscale Organic Experiments,* 3rd ed. Houghton Mifflin Company, Boston, 1999.
- Website: http://blogsciencestuff.blogspot.com

MODULE 1

MODULE 1

UNIT 1 - SAFETY IN THE LABORATORY

7.0. INTRODUCTION

Organic chemistry is an experimental science. Our understanding of organic chemistry is mainly the result of laboratory observation and testing. For this reason, the laboratory is an important part of a student's education in organic chemistry.

Because of the nature of organic compounds, the organic chemistry laboratory is generally more dangerous than the inorganic chemistry laboratory. Many organic compounds are volatile and flammable. Some can cause chemical burns; many are toxic. Some can cause lung damage, some can lead to cirrhosis of the liver, and some are carcinogenic (causing cancer). Yet organic chemists generally live as long as the rest of the population because they have learned to be careful. When working in an organic laboratory, you must always think in terms of safety.

8.0. OBJECTIVES

After studying this unit, you should be able to:

- See the laboratory as a new and potentially hazardous environment and understand safe practice.
- Discuss the need for specialized apparatus for the handling of chemicals and materials.
- learn laboratory safety rules and the necessity of practicing these rules in the laboratory

9.0. MAIN CONTENT

9.1. Personal safety

9.1.1. Using Common Sense

Most laboratory precautions are nothing more than common sense. The laboratory is not a place for horseplay. Do not work alone in the laboratory. Do not perform unauthorized experiments. Do not sniff, inhale, touch or taste organic compounds, and do not pipette them by mouth. Wipe acids and bases. Neutralize residual spilled acid with sodium bicarbonate and spilled base with dilute acetic acid. Do not put dangerous chemicals in the waste crock – the janitor may become injured. Do not pour chemicals down the sink – the environment will be injured. Instead, use the containers provided for chemical disposal.

When working in the laboratory, wear suitable clothing. Jeans and a shirt will rolled-up sleeves, plus a rubber lab apron or cloth lab coat, are ideal. Do not wear your best clothing –laboratory attire usually acquires many small holes from acid splatters and may also develop a distinctive aroma. Loose sleeves can sweep flasks from the laboratory bench, and they present the added hazard of easily catching on fire. Long hair should be tied back. Broken glass sometimes litters the floor of a laboratory; therefore, always wear shoes. Sandals are inadequate because they do not protect the feet from spills. Wash your hands frequently, and always wash them before leaving the laboratory, even to go to the rest room.

Because of the danger of fires, smoking is prohibited in laboratories. Because of the danger of chemical contamination, food and drink also have no place in the laboratory. On the first day of class, familiarize yourself with the locations of the fire extinguishers, fire blanket, eyewash fountain and shower.

9.1.2. Safety Glasses

Chemicals splashed in the eyes can lead to blindness; therefore it is imperative that you wear safety glasses, or better yet, safety goggles. Wear them at all times, even if you are merely adding notes to your laboratory notebook or washing dishes. You could be an innocent victim of your lab partner's mistake, who might inadvertently splash a corrosive chemical in your direction. In the case of particularly hazardous manipulations, you should wear a full-face shield (similar to a welder's face shield).Your instructor will tell you when this is necessary.

Contact lenses should not be worn, even under safety glasses. The reason for this rule is that contact lenses cannot always be removed quickly if a chemical gets into your eye. A person administering first aid by washing your eye might not even realize that you are wearing contact lenses. In addition, "soft" contact lenses can absorb harmful vapours. If contact lenses are absolutely necessary, properly fitted goggles must be worn. Also inform your laboratory instructor and neighbours that you are wearing contact lenses.

9.2. Laboratory Accidents

9.2.1. Chemicals in the Eyes

If a chemical does get into your eye, flush it with gently flowing water for fifteen minutes. Do not try to neutralize an acid or base in the eye. Because of the natural tendency of the eyelids to shut when something is in the eye, *they must be held upon during the washing*. If there is no eyewash fountain in the laboratory, a piece of rubber tubing attached to a tap is a good substitute. Do not take time to put together a fountain if you have something in your eye, however! Either splash you eye (held open) with water from the tap immediately or lie down on the floor and have someone pour a gentle stream of water into your eye. *Time* is important. The sooner you can wash a chemical out of your eye, the less the damage will be.

After the eye has been flushed, medical treatment is strongly advised. For any corrosive chemical, such as sodium hydroxide, prompt medical attention is imperative.

9.2.2. Chemical Burns

Any chemical (whether water-soluble or not) spilled unto the skin should be washed off immediately with soap and water. The detergent action of the soap and the mechanical action of washing remove most substances, even insoluble ones. If the chemical is a strong acid or base, rinse the splashed area of the skin with *lots and lots of cool water*. Strong acids on the skin usually cause a painful stinging. Strong bases usually do not cause pain, but they are extremely harmful to tissue. Always wash carefully after using a strong base.

If chemicals are spilled on a large area of the body, wash them off in the safety shower. If the chemicals are corrosive or can be adsorbed through the skin, remove contaminated clothing so that the skin can be flushed thoroughly. If chemical burns result, the victim should seek medical attention.

9.2.3. Heat Burns

Minor burns from hot flasks, hot tubing, and the like are nt uncommon occurrences in the laboratory. The only treatment needed for a very minor burn is holding it under cold water for 5 – 10 minutes. A painkilling lotion may then be applied. To prevent minor burns, keep a pair of inexpensive loose-fitting cotton gloves in your laboratory locker to use when you must handle hot beakers, tubing or flasks.

A person with a serious burn, as from burned clothing, is likely to go into shock. He or she should be made to lie down on the floor and kept warm with the fire blanket or with a coat. Then, an ambulance should be called. Except to extinguish flames or to remove harmful chemicals, do not wash a serious burn and do not apply any ointment. However, cold compresses on a burned area will help dissipate heat.

9.2.4. Cuts

Minor cuts from broken glassware are another common occurrence in the laboratory. These cuts should be flushed thoroughly with cold water to remove any chemicals or sliver of glass. A pressure bandage can be used to stop any bleeding.

Major cuts and heavy bleeding are a more serious matter. The injured person should lie down and be kept warm in case of shock. A pressure bandage (such as folded, clean dish towel) should be applied over the wound and the injured area elevated slightly, if possible. An ambulance should be called immediately.

9.2.5. Inhalation of Toxic Substances

A person who has inhaled vapours of an irritating or toxic substance should be removed immediately to fresh air. If breathing stops, artificial respiration should be administered and an emergency medical vehicle called.

9.3. Laboratory Fires

9.3.1. Avoiding Fires

Most fires in the laboratory can be prevented by the use of common sense. Before lighting a match or burner, check the area for flammable solvents. Solvent fumes are heavier than air and can travel along a bench top or a drainage trough in the bench. These heavy flammable fumes can remain in sinks or waste baskets for days. While it is indeed true that a flammable solvent should not have been discarded in the sink or waste basket, it is always possible that some inconsiderate fool has done so. Therefore, do not discard hot matches, even if extinguished, or any other hot substance in sinks or waste baskets.

Whenever you use a flammable solvent, extinguish all flames in the vicinity beforehand. Always cap solvent bottles when not actually in use. Do not boil away flammable solvents from a mixture except in the fume hood. Place solvent-soaked filter paper in the fume hood to dry before discarding it in a waste container. Spilled solvent should not be allowed simply to evaporate. If a solvent is spilled, clean it up immediately with paper towels which should be placed in the hood to dry.

Solvents should never be poured into a drainage trough (which is for water only). Because of environmental concerns, solvents should be disposed of only in containers provided for solvent disposal. In general, these disposal containers are located in the fume hood in the laboratory.

9.3.2. Extinguishing Fires

In case of even a small fire, tell your neighbours to leave the area and notify the instructor. A fire confined to a flask or beaker can be smothered with a watch glass of large beaker placed over the flaming vessel. (Try not to drop a flaming flask – this will splatter burning liquid and glass over the area). All burners in the vicinity of a fire should be extinguished, and all containers of flammable materials should be removed to a safe place in case the fire spreads.

For all but the smallest fire, the laboratory should be cleared of people. It is better to say loudly, “Clear the room,” than to scream “Fire!” in a panicky voice. If you hear such a shout, do not stand around to see what is happening, but stop whatever you are doing an d walk immediately and purposefully toward the nearest clear exit.

Many organic solvents float in water; therefore water may serve only to spread a chemical fire. Some substances, like sodium metal, explode on contact with water. For these reasons, water should not be out to extinguish a laboratory fire; instead, a carbon dioxide or powder fire extinguisher should be used.

If a fire extinguisher is needed, it is best to clear the laboratory and allow the instructor to handle the extinguisher. Even so, you should acquaint yourself with the location, classification and operation of the fire extinguish the first day of class. Inspect the fire extinguishers. Find the sealing wire (indicating that the extinguisher is fully charged) and the pin that is used to break this sealing wire when the extinguisher is needed.

Fire extinguishers usually spray their contents with great force. To avoid blowing flaming liquid and broken glass around the room, aim toward the base and to the side of any burning equipment, not directly toward the fire. Once a fire extinguisher has been used, it will need recharging before it is again operable. Therefore, any use of a fire extinguisher must be reported to the instructor.

9.3.3. Extinguishing Burning Clothing

If your clothing catches fire, walk (do not run) to the shower if it is close by. If the shower is not near, lie down, roll to extinguish the flames, and call for help.

A clothing fire may be extinguished by having the person roll in a fire blanket. The rolling motion is important because fire can still burn under the blanket. Wet towels can be use to extinguish burning clothing. A burned person should be treated for shock (kept quiet and warm). Medical attention should be sought.

9.4. Handling Chemicals

9.4.1. Acids and Bases

To prevent acid splatters, *always add concentrated acids to water* (never add water to acids). Concentrated sulphuric acid (H_2SO_4) should be added to ice water or crushed ice because of the heat generated by the mixing. Do not pour acids down the drain without first diluting them (by adding them to large amounts of water) and then neutralizing them. Strong bases should also be diluted and neutralized before discarding. If you splash an acid or strong base on your skin, wash with plentiful amounts of water, as described in the section on chemical burns. Concentrated hydrochloric acid (HCl) and glacial acetic acid (CH_3COOH) present the added hazard of extremely irritating vapours. These two acids should be used only in the fume hood.

Sodium hydroxide (NaOH) is caustic and can eat away living tissues. As a solid (usually pellets), it is deliquescent; a pellet that is dropped and ignored will form a dangerous pool of concentrated NaOH. Pick up spilled pellets while wearing plastic gloves or by using a piece of paper, neutralize them, and then flush the neutralized mixture down the drain with large amounts of water.

Aqueous ammonia ("ammonium hydroxide") emits ammonia (NH_3) vapours and thus should be used only in the fume hood.

9.4.2. Solvents

Organic solvents present the double hazard of flammability and toxicity (both short-term and cumulative).

- Diethyl ether ($C_2H_5OC_2H_5$) and petroleum ether (a mixture of alkanes) are both very volatile (have low boiling points) and extremely flammable. These two solvents should never be used in the vicinity of a flame, and they should be boiled only in the hood.
- Carbon disulphide (CS_2), which is now rarely used in the organic laboratory, is uniquely hazardous. Its ignition temperature is under 100 ^oC, the boiling point of water; therefore, fires can result even from its contact with a steam pipe.
- Benzene (C_6H_6) is flammable and also extremely toxic. It can be absorbed through the skin, and long-term exposure is thought to cause cancer. Benzene should be used as a solvent only when absolutely necessary (and then handled with great care to avoid inhalation, splashes on the skin, or fire). In most cases toluene can be substituted for benzene. Although toluene is flammable, it is less toxic than benzene.
- Most halogenated hydrocarbons, such as carbon tetrachloride (CCl_4) and chloroform ($CHCl_3$), are toxic, and some are carcinogenic. Halogenated hydrocarbons tend to accumulate in the fatty tissues of living systems, instead of being detoxified and excreted, as most poisons are. In repeated small doses, they are

associated with chronic poisoning and damage to the liver and kidneys. If either carbon tetrachloride or chloroform must be used, it should be handled in the fume hood.

Because of the dangers inherent with all organic solvents, they should always be handled with respect. Solvent vapours should not be inhaled, and solvents should never be tasted or poured on the skin. Wash any splashes on your skin immediately with soap and water. Keep solvent bottles tightly capped. Always heed precautions to avoid fires.

9.4.3. Symbols used to convey information about chemicals:

Flammable

Corrosive

Explosive

Oxidising (helps other things to burn)

Toxic

Harmful or irritant

Environmental hazard

10.0. SUMMARY

Standard safety practices are an essential part of all laboratory operations. The chemicals employed in the laboratory can be flammable or irritating, and many possess known or as yet undetermined toxic characteristics.

Accidents in the chemistry laboratory can be avoided if you enter the laboratory properly prepared for the experiment, if you use common sense in reacting to unexpected situations, and if you rigidly follow basic safety rules that are enforced to ensure your personal safety.

11.0. TUTOR-MARKED ASSIGNMENTS

1. Give reasons for the following safety rules:
   a. Contact lenses should not be worn in the laboratory
   b. A chemical spill on the skin should be washed off with water, not with solvent
   c. Solvents are not to be poured into the drainage trough
   d. Water should not be used to extinguish laboratory fires
   e. To dilute concentrated sulphuric acid, we pour it into ice instead of simply mixing it with water.

2. What should you do in each of the following circumstances?
   a. Your neighbor splashes a chemical into his or her eye
   b. A strong acid spills onto your hands

c. Your neighbor's clothing catches fire
d. Your neighbor's flask catches fire.

3. What are the principal hazards of each of the following solvents?
   a. Carbon disulphide
   b. Diethyl ether
   c. Benzene
   d. Carbon tetrachloride.

4. According to the dress code, what should you wear in the laboratory?

12.0. REFERENCE AND OTHER RESOURCES

- Fessenden, R.J; Fessenden, J.S and Landgrebe, J.A, Organic Laboratory Techniques. 2nd ed. Brooks/Cole Publishing Company, 1984.
- Steere, N.V., *Handbook of Laboratory Safety.* 2nd ed. CRC Press, Cleveland, Ohio, 1971.
- Green, M.E. and Turk, A, *Safety in Working with Chemicals.* Macmillan, New York, 1978.

UNIT 2 - COMMON LABORATORY APPARATUS

1.0. INTRODUCTION

This section of this course provides an introduction to various pieces of equipment - glassware and non-glassware - that are used in the laboratory.

Laboratory glassware refers to a variety of equipment, traditionally made of glass, used for scientific experiments and other work in science, especially in chemistry and biology laboratories. Some of the equipment is now made of plastic for cost, ruggedness, and convenience reasons, but glass is still used for some applications because it is relatively inert, transparent, more heat-resistant than some plastics up to a point, and relatively easy to customize. Borosilicate glasses are often used because they are less subject to thermal stress and are common for reagent bottles. For some applications quartz glass is used for its ability to withstand high temperatures or its transparency in certain parts of the electromagnetic spectrum. In other applications, especially some storage bottles, darkened brown or amber (actinic) glass is used to keep out much of the UV and IR radiation so that the effect of light on the contents is minimized. Special-purpose materials are also used; for example, hydrofluoric acid is stored and used in polyethylene containers because it reacts with glass. For pressurized reaction, heavy-wall glass is used for pressure reactor.

There are many different kinds of laboratory glassware items, the majority are covered in separate articles of their own; see the list further below. Such glassware is used for a wide variety of functions which include volumetric measuring, holding or storing chemicals or samples, mixing or preparing solutions or other mixtures, containing lab processes like chemical reactions, heating, cooling, distillation, separations including chromatography, synthesis, growing biological organisms, spectrophotometry, and containing a full or partial vacuum, and pressure, like pressure reactor. When in use, laboratory glassware is often held in place with clamps made for that purpose, which are likewise attached and held in place by stands or racks. Aspects of laboratory glassware which may be common to several kinds of glassware are described in this unit.

2.0. OBJECTIVES

After studying this unit, you should be able to:

- Be familiar with the apparatus and equipment used in the study of chemistry, and their correct use
- Classify each piece as apparatus for measuring, containing, supporting, delivering liquid, heating, testing, etc.

3.0. MAIN CONTENT

3.1 Glassware

Glass equipment can be divided into those with ground-glass joints and those without. New techniques of glassworking and the employment of glasses with a very low expansion have allowed the mass production of really interchangeable, standard-taper, ground-glass joints. Therefore standard-taper, ground-glass joint equipment is strongly recommended. In fact, apparatus for a range of experiments can be assembled quickly and easily from relatively few basic items and there is no need to utilize corks or rubber bungs. The advantages of this are:

- corrosive liquids and solids are easily manipulated;
- no impurity is introduced from reaction with corks or rubber bungs (this prevents contaminating the reaction products with impurities);
- all joints are interchangeable and a good “fit” is assured (this is very advantageous in assembling apparatus that have to work under reducing pressure);

- Apparatus may be rapidly assembled.

In the use of ground-glass joints some precautions need to be observed, such as cleaning the joints and the use of a little grease or a thin Teflon sleeve. The dimension of standard taper joints are designated by a number that refer to the external diameter of the smaller end of the cone (in millimeters) and the length of the ground zone (also in millimeters).
Common glassware without ground-glass joints can generally be used in the chemical laboratory. The main glassware without glass joints are illustrated in Figure 1.

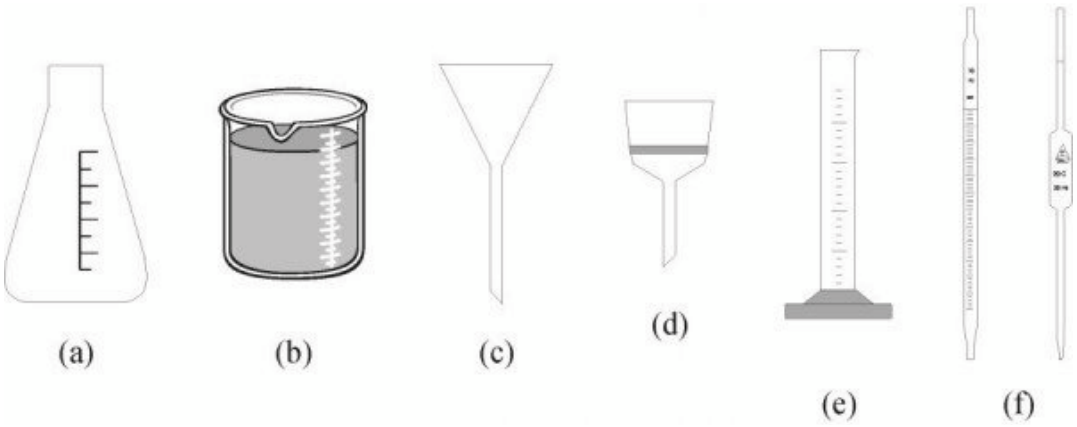
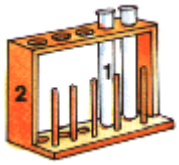


Figure 1: Common glassware without ground-glass joints

Notes: (a) Erlenmeyer flask and (b) beaker, used for temporary storage or transfer of materials; (c) funnel, for transfer of liquids and solids, and for filtration; (d) Buchner funnel, which is made of porcelain and has a perforated porcelain plate to support a filter paper, and is used in conjunction with a suction flask; (e) graduated cylinder, for measuring liquids by volume; (f) graduated pipette, for accurate measurement.
At last vacuum desiccators are available to remove water or solvents of relatively low boiling point adhering to solids, under reduced pressure at room temperature, or to keep anhydrous compounds. The reagent in the bottom of the desiccators naturally depends on the exact nature of the substance to be adsorbed—water or organic solvent—or the acid or basic vapor generated during the drying process.



1.Test Tube
2.Test Tube Rack

Test Tube Holder

Reagent Bottle

Beaker

Bunsen Burner

Stand and Clamp

Crucible

Measuring Cylinder

Dropper

Electronic Balance

Evaporating Dish

Filter Funnel

Flat Bottomed Flask

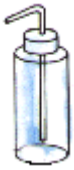
Safety Glasses

Mortar and Pestle (used for grinding paste or powder)

Round Bottomed Flask

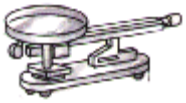
Spatula

Tripod



Plastic Wash Bottle

Wire Gauze



Triple Beam Balance

Figure 2: Some laboratory apparatus

3.1.1. Flasks

The **erlenmeyer flask** is the favourite for general use. The tapered top allows you to fit other pieces of glassware as needed and makes it harder to slop out the contents. The flat bottom allows it to stand up on the desk top, and straight sides make it easy to scrape out a solid product

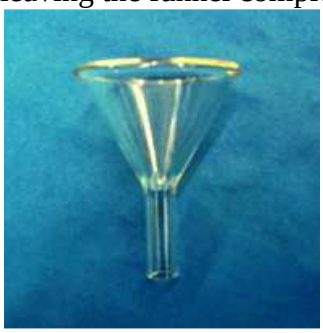
Filter flasks are thick-walled erlenmeyer type flasks used in vacuum or suction filtration setups.

The **round-bottom flask** is preferred if you are planning to heat a solution to boiling, as in a distillation. Its geometry favors uniform heating and helps minimize the splashing associated with boiling. It may be hard for a student to discern the benefit of using the proper flask in a distillation, but it is always considered bad form to use an Erlenmeyer for such a purpose.

3.1.2. Funnels

The **Buchner funnel** is used in vacuum or suction filtration in order to separate solids from liquids. This is used with a piece of filter paper covering the holes. Wet the filter paper with the solvent you are using to create a seal before filling it with the solution to be filtered. Sometimes you must press the funnel down onto the top of the vacuum flask to create a good seal. Other times, when you have a mushy, damp product, it can help to leave the funnel in place and continue to pull air through it until the product dries slightly.

The **long stem funnel** can help you pour anything neatly, but it is of particular convenience when working with volatile solvents. You will find that volatile solvents, because of their low surface tension, are quite easy to splash and spill. It is very convenient to use a funnel when measuring out these solvents because there is no glassware washing cost associated with using it. Just wave the funnel in the air and the solvent will evaporate, leaving the funnel completely clean.



The **separatory funnel** is used to separate two immiscible liquids. Typically, you will shake the two liquids together to allow a solute to pass from one to the other. The liquids are then allowed to separate and the lower liquid layer can be carefully drained out the bottom. Always close the stopcock on the funnel before filling it, and always hold on to the stopper when you shake the funnel.

3.1.3. Condensers

The **air condenser** is mainly used as a fractionating column in distillation setups. It can be left open or packed with material which could serve as plates for condensation of the vapour in fractional distillation

The **lie-big condenser** cools the vapour causing it to reliquify and directs this condensate to the receiving flask. The most common type of condenser is the water-jacketed type shown. The water supply is connected to the condenser with rubber hoses. The water flows in the lower hose connection (most remote from the distillation flask) and out the upper hose connection. During the distillation of very high boiling liquids, it is common practice to cool the condenser with air instead of water. The thermal shock to glassware from a large temperature difference between cold water and hot vapour can crack the glassware

3.1.4. Other glassware

A **drying tube** is used to exclude moisture from an experimental setup during an operation such as refluxing or distillation. It is packed with a suitable drying agent (or dessicant) such as granular alumina, silical gel, calcium sulfate (Drierite) and calcium chloride. The drying tube is filled by tamping a plug of dry cotton or glass wool into the bottom opening, adding the dessicant and inserting another plug of cotton or glass wool to prevent spillage

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The **watch glass** is used to hold solids when being weighed or transported. They should never be heated.

3.2. Other Apparatus and Equipment

3.2.1. Clamp and clamp holders

There are several styles of clamp and clamp holders that can be used to support glassware on ring stands. Usually these can be adjusted to accommodate any angle. Take a little time to see how they work. One obvious indicator of whether a student has everything under control is the appearance of the apparatus. If it is clamped skew to the ring stands and benchtop, it not only looks bad but it may not work well. You should adapt the clamps to your work, not vice-versa.

3.2.2. Bunsen burner

The Bunsen burner is an important heat source for the laboratory. The burner has a **needle valve** located in its base, and this valve controls the amount of gas that will enter the **chimney** via the **gas inlet** to be mixed with oxygen from the atmosphere. Air enters at the bottom of the chimney through the **air vents**, which can be enlarged by turning the chimney.

Bunsen burners are used for heating and exposing items to flame. They have many more uses than a hot plate, but do not replace a hot plate.

3.2.3. Others

Crucible

Crucibles are used to heat small quantities to very high temperatures.

Evaporating dish
The evaporating dish is used to heat and evaporate liquids

Mortar and Pestle
The Mortar and Pestle are used to crush solids into powders for experiments, usually to better dissolve the solids.

Tongs
Tongs are used to hold many different things such as flasks, crucibles, and evaporating dishes when they are hot.

4.0. SUMMARY

A study on common laboratory apparatus is very useful for both scientists and up-and-coming scientists. Up-and-coming scientists will find this useful when it comes to getting to know their way around the lab. The study is also very useful when it comes to passing exams as chemistry exams; particularly at an earlier stage tend to examine the student to ensure they know how and when to use the apparatus, in a safe and sound manner. For the experienced scientist, the study serves as a good way to refresh one's mind on the uses of the different apparatus.

5.0. TUTOR-MARKED ASSIGNMENTS

1. What is the most common laboratory apparatus give me twenty-five common laboratory apparatus with their uses.
2. What are the classifications of laboratory apparatus??
3. What do you use to measure mass?
   (a) Graduated cylinder (b) Ruler (c) Balance
6. In what unit do we measure length?

(a) inches (b) feet (c) metres

7. When measuring a liquid you use a _____, you also should put it on a flat surface and get at _____ level to read it properly.
   (a) Beaker, desk (b) Triple Beam Balance, elevated (c) Graduated cylinder, eye

6.0. REFERENCES AND FURTHER READING

- Williamson, K.L, *Macroscale and Microscale Organic Experiments*, 3rd ed. Houghton Mifflin Company, Boston, 1999.
- Website: http://blogsciencestuff.blogspot.com

UNIT 3 - BASIC LABORATORY TECHNIQUES

7.0. INTRODUCTION

Basic laboratory techniques in chemistry have an essential place in the training of a chemist. They provide a good background for experimental skills and for subsequent independent research.

By assembling suitable apparatus, you can carry out reactions from starting materials to pure products. Sometimes these operations require particular laboratory reactors that work in well-defined operation conditions. At the end of the reaction, different techniques are needed in order to separate the products from the reaction mixture, and then the final compound will need to be purified by means of specific operations. The separation of reaction products is obtained by techniques such as extraction with solvents, crystallization, and distillation. These methods, which are almost standardized, are often able to purify organic compounds by separating the impurities. Chromatographic methods are very effective in separating more complex mixtures.

8.0. OBJECTIVES

After studying this unit, you should be able to:

- Identify the importance of studying the basic chemistry laboratory techniques
- Identify 10 basic laboratory techniques in organic chemistry
- Explain the significance of the basic laboratory techniques

9.0. MAIN CONTENT

Basic Laboratory Techniques

9.1. Measuring Volume

9.1.1. Medicine Droppers.

A satisfactory, but often rough, method of estimating volumes, the dropper is calibrated by counting the number of drops it produces to make up a milliliter.

9.1.2. Beakers and Flasks.

The volume on the side of the beaker or flask is only a rough approximation at best. Use this method only for crude estimates of volume.

9.1.3. Volumetric Glassware.

This is the most precise and accurate method of transferring and delivering liquids. It is important that the volumetric glassware be extremely clean before use, as dirt and other chemicals will not only reduce precision due to improper draining but also can contaminate the experiment. The best time to clean glassware is immediately after its use. Special cleaning solutions are available, but soap, warm water, and a brush, followed by thorough rinsing-first with tap water and then with small amounts of distilled water-is often satisfactory. No drops of distilled water should adhere to the surface of clean glassware. To dry, the glassware is inverted onto a paper towel. Do not wipe or air-blow dry due to possible contamination.

In determining the volume of all volumetric glassware, it is important to understand how to read the *meniscus*. The meniscus is the apparent downward curvature of the liquid mainly due to surface tension. It is necessary to read the bottom of the meniscus with the eye horizontal to this surface. If it is not read at eye level, error in the

reading will result. Proper lighting is important to see the meniscus clearly. To enhance the meniscus, a small white card with a 1 x 1.5 black rectangle in the lower one-third of the card is placed behind the glassware. The card is slowly raised until the reflection of the black rectangle on the meniscus is seen. The bottom of the meniscus is now readily visible against the white background.

a. *Graduated cylinders* are the most commonplace measuring instruments in the laboratory. A tall cylinder with a small diameter will be more accurate than a short one with a large diameter.

b. *Burettes* are constructed so that it is possible to measure and deliver accurate volumes. To clean a burette, clean with a soap solution. If a burette brush is used be sure not to scrape the sides of the burette wall with the metal handle. Rinse the soap solution from the burette several times through the stopcock, first with tap water and then with several small portions of distilled water. Be sure to roll the burette in a near horizontal

position to thoroughly wet the entire surface of the glass.

To *activate* the burette, close the stopcock and add a small portion of titrant (3-5 mL). Tilt the burette to a nearly horizontal position and roll the burette so that the rinse comes in contact with the entire inner surface. Drain this material through the burette tip into a waste beaker. Repeat this procedure three times.

To fill and operate a burette, close the stopcock and, with a funnel, fill the burette to just above the zero mark. Be sure the burette is vertical and does not slant. Open the stopcock briefly to remove any air bubbles and drain the titrant to some point below the zero mark. Record the starting volume and perform the titration. Be sure to record the final volume. When the titration is finished, drain the excess titrant and rinse the burette with a small amount of distilled water two to three times. Clean it if necessary.

c. *Volumetric pipettes*, properly manipulated, can deliver volumes reliable to one part per thousand. *Graduated pipettes* are not capable of such precision. **In pipetting a liquid, oral suction must never be used.**

Keeping the pipette tip under the surface of the liquid and, using a pipette bulb, draw the liquid above the graduation mark. Slowly squeeze the release button to level off the amount. Any drops adhering to the bottom of the tip should be removed before delivering the volume. Hold the pipette vertically and let the liquid drain for 20 seconds after the liquid has been delivered; the tip is then touched to the wall of the receiver. The liquid in the tip of the pipette must not be removed; calibration of the pipette has allowed for this.

d. *Volumetric flasks* are used to prepare solutions of a specified concentration. In making a solution the solute is placed in the flask first. Then a small amount of the solvent is added and the flask is vigorously shaken to dissolve or mix the solutions. Care should be taken as the flask and solution may get very warm. **Never** use your fingers to cover the opening at the top. There will be an appropriate sized stopper to use. Add more solvent to just below the fiducial mark, stopper, and shake again. Let the flask come to room temperature and then fill the flask to the fiducial mark and stopper. Invert the stoppered flask several times to thoroughly mix contents. Clean the glassware when finished.

9.2. Glass working

9.2.1. Cutting Glass Tubing

Make a scratch across the glass tubing at the desired location with a single stroke of a triangular file. Place a

drop of water on the scratch with your finger. The tubing must always be held in a towel while pressure is being applied to prevent injury to the hands. Place both thumbs close together on the side of the tubing opposite the scratch, and snap the tubing in a direction away from you and others against the pressure of the thumbs.

9.2.2. Fire Polishing Glass Tubing

All edges of glass tubing must be fire-polished to round off the sharp edges. Hold the sharp edges of the tubing at an angle in the flame and rotate the tubing until a bright yellow color is imparted to the flame. Be sure to avoid over heating the glass tubing.

9.2.3. Bending Glass Tubing

Attach a wing top to the burner to spread the flame. Hold a piece of tubing horizontally in the upper portion of the flame and slowly rotate to ensure uniform heating. Continue rotation of the tubing until it becomes soft. Smoothly bend the glass tubing and allow it to cool on a fire resistant surface.

9.3. Separating Liquids and Solids

9.3.1. Decanting

Allow the solid to settle in the beaker or test tube. Then transfer the liquid, or supernatant, with the aid of a stirring rod. Hold the stirring rod against the lip of the beaker and pour the liquid down the rod, which is touching the inner wall of the receiving vessel. Do this slowly so as not to disturb the settled solid.

9.3.2. Gravity Filtration

Fold a piece of filter paper in half; refold to within 10^{O} of a 90^{O} fold; tear off the corner unequally, and open. Place the folded filter paper snuggly into the funnel. Moisten the filter paper with the solvent being used and press the filter paper against the funnel s top wall to form a seal. Hold a stirring rod against the lip of the beaker and pour the liquid down the rod, which is touching the inside of the funnel. The funnels tip should touch the inside wall of the receiving vessel to reduce splashing. **Never** fill the funnel more than two-thirds full.

9.3.3. Vacuum Filtration

A Buchner funnel fitted with a rubber stopper is inserted into a suction flask. The side arm of the flask is connected to a safety trap, which in turn is connected to the water aspirator by a short piece of pressure tubing. Filter paper, slightly smaller than the funnel diameter, is placed over the holes and moistened with the solvent. Turn the aspirator on full.

9.3.4. Centrifugation

Whenever the centrifuge is used it must be balanced or it may become damaged. To do this fill, an identical tube with the same level of water in the position opposite the mixture to be separated. Close the cover and set the machine in motion. **Keep the cover closed and your hands away from the top of the centrifuge while it is rotating.**

9.4. Transferring Liquids

When the reagent is being transferred from a reagent bottle, remove the glass stopper and hold it between the fingers of the hand used to grasp the reagent bottle. **Never** lay the stopper on the desktop; impurities may be picked up and contaminate the solution. Hold the stirring rod against the lip of the reagent bottle and pour the

liquid down the rod, which is touching the inner wall of the receiving vessel. This prevents splashing and losses of reagent down the side of the bottle. **Never** transfer more liquid than is necessary and **never** return unused portions to the reagent bottle.

9.5. Testing Gases for Odour

An educated nose is an important and useful asset to have in the laboratory. It must be used with caution because some gases are toxic or just irritant. **Never** hold your nose directly over the vessel from which the gas is coming from. Rather, fan some of the vapors toward your nose.

9.6. Testing with Litmus

To test the acidity/basicity of a solution with litmus paper, insert a stirring rod into the solution, withdraw it, and touch it to a litmus paper that is resting on a clean, inverted watch glass. **Never** place the litmus paper directly into the solution.

9.7. Keeping Samples Dry

Materials may absorb water if left exposed to the air. This is to be avoided, especially if the sample of material is to be weighed precisely. The desiccator is the container to keep samples dry. It contains Drierite as a drying agent; blue Drierite turns pink when it is no longer effective.

9.8. Heating

Many of the operations carried out in a laboratory experiment require heating. Although there are numerous methods available, reaction conditions and chemical and physical properties of the materials make certain heating methods preferable to others. Since most organic compounds, especially the commonly used solvents such as hexane and ether, are flammable, a flameless method is generally preferred.

A steam bath is often used to heat solutions that boil below about 90 ^{o}C, or to heat a mixture to approximately 100 ^{o}C. The laboratories are supplied with steam from a central boiler and in this case use of the steam bath eliminates the hazards of a flame. Connect the steam line to the top inlet on the bath. The bottom inlet is attached to a hose, which drains into the sink. Any water that condenses in the bath while you're using it will drain out. Usually, steam baths have concentric rings as covers. You can control the "size" of the bath by adding or removing rings.

9.8.1. Steam bath

A steam bath is often used to heat solutions that boil below about 90 ^{o}C, or to heat a mixture to approximately 100 ^{o}C. The laboratories are supplied with steam from a central boiler and in this case use of the steam bath eliminates the hazards of a flame. Connect the steam line to the top inlet on the bath. The bottom inlet is attached to a hose, which drains into the sink. Any water that condenses in the bath while you're using it will drain out. Usually, steam baths have concentric rings as covers. You can control the "size" of the bath by adding or removing rings.

9.8.2. Hot water bath

The **hot water bath** is a useful device for heating at temperatures below 100 degrees Celsius - simply a beaker or steam bath full of hot water, sometimes from the faucet will be hot enough or it might be necessary to heat the water on a hot plate. A steam bath or hot water bath should be used with flammable substances whenever possible

9.8.3. Sand bath

A sand bath is a convenient source of heat for micro-scale reactions. A sand bath warms up much more slowly than a steam bath. Unlike the hot water and steam baths, a sand bath can provide temperatures that range from near room temperature at the surface up to 200 deg C and above, deep in the sand.

9.8.4. Oil bath

An oil bath provides an even source of heat whose temperature can be closely controlled. A simple oil bath can be made by heating a dish of oil on a hotplate. The temperature of the oil can be monitored by a thermometer. It is most suitable, in combination with a hotplate, for heating something at a high temperature (depending on the oil used) or at constant temperature for an extended period of time. The disadvantage of an oil bath is the long time required to bring it to the desired temperature, splattering if it gets contaminated with water and the possibility of spilling the hot oil

9.8.5. Hot plate

The hot plate can be used as a source of heat. It tends to be rather inefficient due to poor contact between the top of the hotplate and the bottom of the flask, and it takes a while to warm up. A hotplate is most useful when something must be heated for a long time, and when it used with an oil bath.

9.8.6. Heating mantle

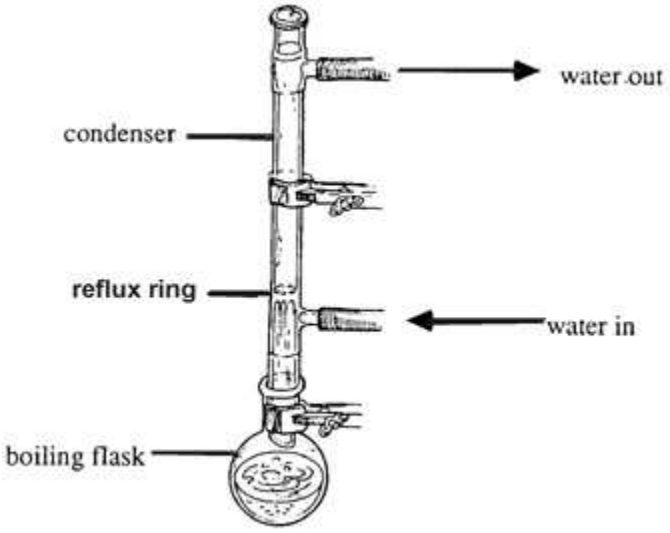
A useful source of heat for reactions, especially where temperatures over 100 ^{o}C are required, is the **heating mantle**. The heating mantle used in these laboratories consists of a ceramic shell with embedded electric heating coils. The ceramic bowl will accommodate flasks with volume capacities up to 250 mL. With flasks smaller than 250 mL, the mantle can be used as an air bath or a sand bath, usually the latter. A small layer of sand in the bowl will serve as a medium for conducting heat to the reaction flask. The heating mantle is safe, because it does not produce flames. It is rapid. One must be careful, however, not to overheat the heating mantles. Heating mantles must be used with variable transformers.

9.9. Refluxing

Reflux is the process of boiling reactants while continually cooling the vapor returning it back to the flask as a liquid. It is used to heat a mixture for extended periods and at certain temperatures. The reflux apparatus is shown below. A condenser is attached to the boiling flask, and cooling water is circulated to condense escaping

vapors. One should always use a boiling stone or a magnetic stirrer to keep the boiling solution from "bumping."

If the heating rate has been correctly adjusted, the liquid being heated under reflux will travel only partly up the condenser tube before condensing. Below the condensation point, solvent will be seen running back into the flask; above it, the condenser will appear dry. The boundary between the two zones will be clearly demarcated, and a reflux ring or a ring of liquid will appear there. In heating under reflux, the rate of heating should be adjusted so that the reflux ring is no higher than a third to a half the distance to the top of the condenser. The temperature of a reaction in a refluxing mixture will be approximately the boiling point of the solvent used for the reaction



9.10. Filtration

Filtration involves the separation of insoluble solid materials from a liquid. In this operation, the liquid passes through a porous barrier (sintered glass or filter paper) and the solid is retained by the barrier. The liquid can be made to pass through the barrier by gravity alone, in which case the procedure is called a gravity filtration. Alternatively, the liquid can be caused to pass through by a combination of gravity and air pressure. Such an operation is called a vacuum or suction filtration.

A piece of filter paper and a conical glass funnel to support it are all that are required for gravity filtration. In order to maximize the rate at which the liquid flows through the filter paper, the paper should be folded as indicated by the steps shown below:

- The folded paper is then dropped into the funnel.
- The funnel is best supported in an iron ring, as shown in the figure.
- The material to be filtered is poured into the filter paper cone, in portions if necessary.

For smaller amounts, as in microscale work, a Pasteur dropping pipette can be used instead of the glass funnel, and a tiny piece of cotton or glass wool pushed down into the narrow part of the tip can serve as the filter.

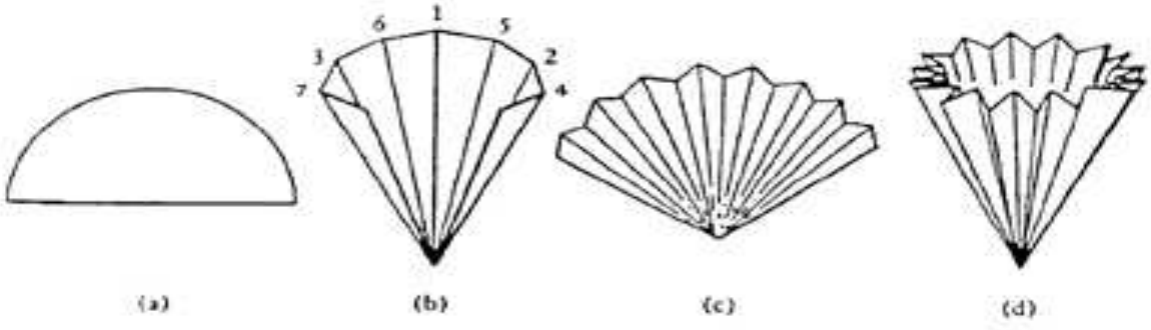
Folding of filter paper for gravity filtration:

(a) Fold the filter paper circle (11 cm diameter) in half

(b) Crease the half to divide it into eight pie-shaped sections; it is easiest to make the creases in the numerical order shown below.

(c) Turn the piece over and pleat it into a fan by folding each pie-shaped section in half in the direction opposite to the previous creases

(d) Pull the two sides apart



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9.11. Heating of the Reaction Mixture

Several methods of heating are commonly encountered in the laboratory, but the ready flammability of a wide range of reagents, coupled with their volatility, always requires vigilance when heating. For these reasons open flames represent an obvious hazard, but hot metal surfaces can also give rise to dangerous situations.

These can normally only be used for heating aqueous solution in open vessels. When heating beakers, flasks, or flat-bottom vessels with a burner, wire gauze should be placed between the vessel and the flame. This serves both as a support and as a mean of dispersing heat. Burners can also be used for distillation and reflux procedures involving high-boiling-point materials, but in these cases care must be taken to ensure that no flammable vapors come into contact with the flame.

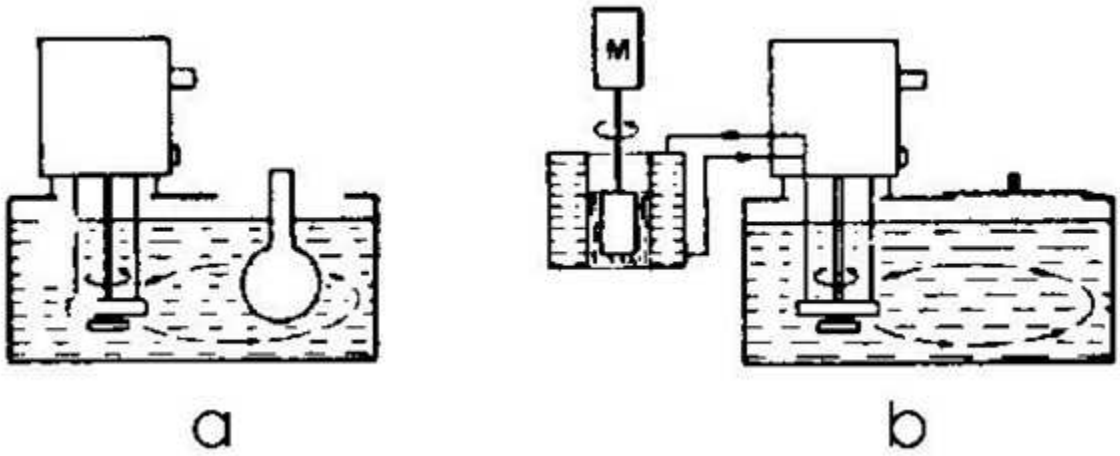
9.11.1. Heating Baths

For temperatures up to 100 °C a water bath or steam bath is generally employed, although water condensing can be a problem if it is necessary to ensure anhydrous conditions within the reaction. Water is placed in the vessel, which is heated by means of the flame. For this reason it may be used for non-inflammable liquids or for refluxing of low-boiling-point products, but in this case the presence of a naked flame introduces considerable risks of fire. These baths are normally equipped with a series of

overlapping concentric rings, which can be removed to give the right size of support for the particular vessel being heated.
If the laboratory is equipped with a steam service, it is convenient to have a series of steam baths. Alternatively, if the laboratory has no external steam service, to avoid naked flames, an electrically heated bath may be utilized and fitted with a constant level device. A resistance connected to temperature regulator heats water in the bath.
For temperatures above 100 °C, oil baths are generally used. The bath can be heated with a heating element or on a hotplate. Medical paraffin, glycerol, silicon oil, and cottonseed oil may be employed; it depends on the work temperature. Silicon oils carry a low risk of inflammation, do not give off unpleasant odors, and have a long service life. Synthetic thermal liquids are mainly produced on a hydrocarbon basis and exhibit a low viscosity within the recommended working temperature range. Mineral oil is mainly used for the high temperature range. Unpleasant odors are kept to a minimum.
The silicon fluids are probably the best liquids for oil baths, but they are very expensive for general use. On the other hand, these fluids can be heated to up to 250 °C without loss or discoloration.

In modern equipment, an immersion heating circulator is mounted onto the rear panel of the bath vessel (see Figure a below). This combines a heater, a temperature control, and a circulating pump for temperature uniformity throughout the bath, which is of great advantage in temperature control. It can be moved from one vessel to another and can be used with any tank. The heater and control sensors are sometimes located underneath the bath, thus guaranteeing easy cleaning.

Heated baths feature external parts to circulate the fluid to an external system. Heating circulators (Figure b) are mainly used for temperature controlling external systems, such as densitymeters, reaction vessels, autoclaves, and viscosimeters. Powerful pumps provide good heat exchange and optimum temperature accuracy.



Schematic (a) heated bath and (b) heated circulator bath

Higher temperatures may be obtained with the support of a bath of fusible metal alloys (e.g., Rose's metal: 2 parts of Bi, 1 part of Pb, and 1 part of Sn, melting point 94 °C; woods metal: 5 parts of Bi, 2 parts of Pb, 1 part of Sn, and 1 part of Cu, melting point
71 °C; and a mixture of Pb (37%) and Sn (63%), melting point 183 °C). These metal baths should not be used at temperatures in excess of 350 °C owing to the rapid oxidation of the alloy. Metal baths are solid at ordinary temperatures, and for this reason the flask and the thermometer should be removed from the bath before the latter solidifies.

9.11.2. Electric Hot Plates and Electric Heating Mantles

These items of equipment may also be employed for heating, although their use for several tasks (e.g., distillation) is to be discouraged.

The hot plate/magnetic stirrer is a single device that can heat liquids and stir them with a magnetic stirring bar. One knob controls the rate of stirring and another controls heating. A stirrer hot plate keeps the solution at a constant temperature while stirring. The built- in magnetic stirrer permits efficient agitation of non-viscous solutions by adding an appropriately sized magnetic stirrer bar to the liquid in the container. It is designed for heating flat-bottomed vessels, such as flasks and beakers, in a temperature range from 40 °C to 200 °C. Round-bottomed flasks may be heated using a stirrer hotplate by immersing the flask in a flat-bottomed oil bath. The flat, exposed surface of the hotplate, designed for transferring heat rapidly, makes it extremely dangerous when hot.

The electric heating mantel is a very convenient method of heating, especially for temperature above 100 °C. It consist of an electric resistance embedded within a hemispherical knitted mantle, so that the heat supply is as close to the flask to be heated as possible. Electric mantels are designed only for heating round-bottomed flasks and can accept a flask of a particular size.

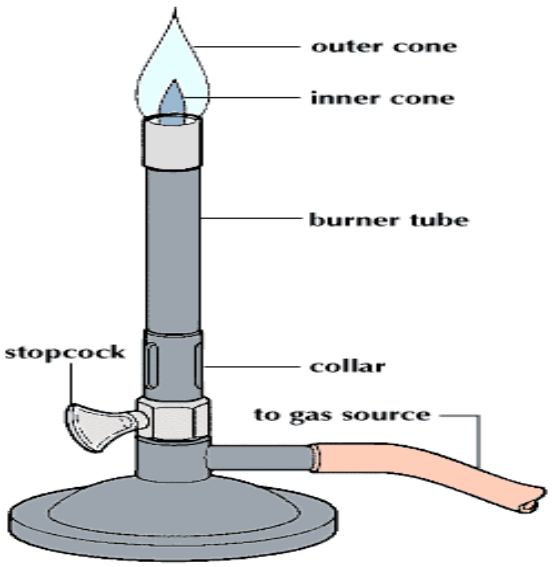
All heating mantels are particularly vulnerable to spillage of liquids, and with constant use this can lay bare the wires within the heating element.

9.11.3. The Bunsen burner

A **Bunsen burner**, named after Robert Bunsen, is a common piece of laboratory equipment that produces a single open gas flame, which is used for heating, sterilization, and combustion.

The device in use today safely burns a continuous stream of a flammable gas such as natural gas (which is principally methane) or a liquefied petroleum gas such as propane, butane, or a mixture of both.

The hose barb is connected to a gas nozzle on the laboratory bench with rubber tubing. Most laboratory benches are equipped with multiple gas nozzles connected to a central gas source, as well as vacuum, nitrogen, and steam nozzles. The gas then flows up through the base through a small hole at the bottom of the barrel and is directed upward. There are open slots in the side of the tube bottom to admit air into the stream via the Venturi effect, and the gas burns at the top of the tube once ignited by a flame or spark. The most common methods of lighting the burner are using a match or a spark lighter.



How to use a Bunsen burner

i. Connect the Bunsen burner to a gas tap using a piece of rubber tubing.
ii. Close the needle valve (counter clockwise with burner in standing position) and the air vent (turn chimney clockwise until closed)
iii. Open the gas valve.
iv. Open the needle valve 1-2 full turns (clockwise in upright position), listening for the sound of escaping gas.
v. Using the striker, light the burner.
vi. The flame of the Bunsen burner should be yellow and irregular in shape.
vii. Open the air-hole slowly. The color of the flame changes to blue.
viii. When not using the Bunsen burner for a while, close the air-hole. This will change the blue flame back to a yellow flame. The yellow flame is the safety flame.
ix. Never leave the flame unattended
x. Turn off the gas valve after use.

CAUTION: It is possible for the burner to strike back and begin burning in the stack or hose. Turn off the gas at the outlet valve, let the burner cool, then close the air vent and repeat the lighting procedure.

TO ADJUST THE FLAME:

1. Use the needle valve to attain a yellow flame approximately 7.5 cm (3 in.) long.
2. Open the air vents by turning the chimney counter-clockwise until the flame loses its yellow color and changes to a pale blue.
3. Reduce the gas flow with the needle valve until the flame is about 2.5cm (1 inch) long high with a distinct inner cone of a deeper blue.

TO TURN OFF THE BURNER:
Turn the handle on the gas outlet valve until it is perpendicular to the outlet nozzle.

9.12. Cooling of the Reaction Mixtures

9.12.1. Methods of cooling

It is often necessary to cool a reaction, particularly when a reaction is rapid and highly exothermic, or when an intermediate formed in a reaction is thermally labile. Temperatures near 0 ^{o}C can be maintained by the use of an ice-water bath. Temperatures down to about -20 ^{o}C can be maintained by the use of ice-salt baths formed by the addition of salt (sodium chloride) to ice. Lower temperatures can be maintained by the addition of dry ice (solid carbon dioxide) to various solvents, Very low temperatures can be maintained by the use of liquid nitrogen (b.p. -196 ^{o}C).

The addition of dry ice or liquid nitrogen to any solvent must be carried out with caution; the addition causes excessive bubbling by the evaporation of gaseous carbon dioxide or nitrogen. The dry ice or liquid nitrogen is slowly added to the solvent contained in a wide-mouth vacuum flask or any other suitably insulated container until a slush is formed. Additional quantities of dry ice or liquid nitrogen are added to maintain the slush. Solvents of low flammability are recommended for use in such cooling baths, although some of the most useful cooling baths employ highly flammable solvents because of the desired accessible temperatures and due precautions must be exercised.

Bath	Temperature (^oC)	Bath	Temperature (^oC)
Liquid nitrogen	-196	Acetone/CO_2	-77
Isopentane/N_2	-160	Ethanol/CO_2	-72
Pentane/N_2	-131	Chloroform/CO_2	-61
Isooctane/N_2	-107	Diethyl carbitol/CO_2	-52
Methanol/N_2	-98	Acetonitrile/CO_2	-41
Toluene/N_2	-95	Carbon tetrachloride/CO_2	-23
Ethyl acetate/N_2	-84	Ethylene glycol/CO_2	-15

Caution must also be used in the used of liquid nitrogen in that if the cooled system is not protected from the air, liquid oxygen (b.p. -176 ^oC) may be condensed in the apparatus which on warming may result in the formation of very high internal pressures. Useful combinations of coolant and solvent are listed in the table above.

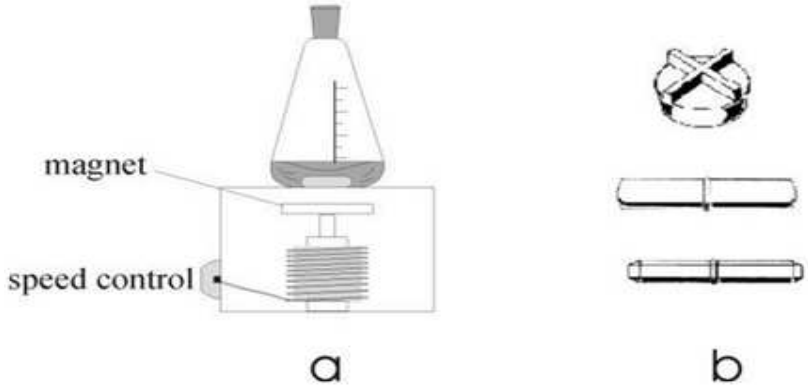
Sometimes a heated circulator is combined with a refrigerator unit. Powerful, quiet- running cooling compressors cool them. Refrigerated circulators and cryostats are mainly used when below-ambient temperatures must be reached or maintained or when it is wished to cycle between two temperatures at a control rate. Refrigerated circulators and cryostats feature a wide temperature range (i.e., –90 °C to 150 °C). They are especially suited for controlling open and closed circuits due to their extremely powerful pressure/suction pumps.
These coolers are suitable for individual cooling applications:

- for cooler smaller volumes down to –90 °C,
- for removing reaction heat, and
- for replacing tap-water cooling.

The lowest reliable temperature depends on the quantity of liquid, the type of liquid and its viscosity, and the bath insulation.

9.13. Stirring

Most of the chemical reactions need stirring to mix the reagents or to aid heat transfer. There are three main ways to agitate a mixture: by hand, with a magnetic stirrer, and with a mechanical stirrer; but when a constant stirring is needed for a sustained period, a stirrer motor should be used. Magnetic stirring has many applications, but the most important is probably stirring in closed systems. Magnetic stirrers are easy to use, and have the advantage that they are often combined with a hotplate. Hot-plate stirrers allow you to keep solutions at a constant temperature while stirring.
A rotating field of magnetic force is employed to induce a variable-speed stirring action. The principle of magnetic stirring is shown in Figure a below. The stirring is accomplished with the aid of a small magnetic bar—coated with Teflon or Pyrex—that is available in various sizes and forms depending on the volume and the viscosity of the liquid. Although bar magnets can be obtained with several sorts of coating, only Teflon-coated stirrers are universally useful (see Figure b).



Schematic (a) magnetic stirrer and (b) magnetic bars

There are two types of magnetic stirrers: mechanical and electronic. Most manufacturers of magnetic stirrers use a mechanical approach. They use steel and aluminum for the structural material, and outdated methods of controlling the speed. Adjusting the stirring speed with a mechanical stirrer is fairly inaccurate.

Electronic controls allow the stirrer to control the speed with greater accuracy, and if the motor is running at the maximum operating speed with a load, and the load is suddenly removed, the circuitry will not allow the motor to increase in speed, which would damage the unit. It is advantageous to stir in two directions to obtain homogeneous results. This cannot be done with a mechanical stirrer.

Liquid behavior is important to consider when you begin a mixing project. If the reaction mixture is very viscous or heterogeneous—with a large amount of suspended solid—the magnetic stirrer motor, with its relative low torque, will not be suitable for the purpose, and in these cases a mechanical stirrer should be used.

The stirrer is simply attached to a motor by a flexible connection made out of a sort of pressure tubing. A typical collection of mechanical stirrers is illustrated in the figure below. The stirrer—either rod or paddle—may be made of glass, metal, or Teflon, depending on the characteristic of the liquid to be stirred.

Several useful types of mechanical stirrer

Proper selection of a suitable motor and type of stirrer requires that you know certain application variables:

- Container volume;
- Liquid viscosity (the viscosity of most liquids varies inversely with temperature);
- torque requirements (the rotation force required of the mixer measured in lbs*ft or N*m);
- Horsepower (hp) requirements (the efficiency required of the mixer motor with regard to torque and to rotation speed); and

- Rotational speed (measured in rpm).

The effect of the liquid's viscosity can present problems when the liquid is subjected to a force. Different types of liquid display characteristics when force is applied. The most common types of viscous liquids are:

- *Newtonian liquids*: viscosity remains constant regardless of changes in shear rate or agitation. Liquids displaying Newtonian behavior include water, hydrocarbons, mineral oil, and syrup.
- *Pseudoplastic liquids*: viscosity decreases as shear rate increases, but initial viscosity may be sufficiently great to prevent mixing. Typical pseudoplastic liquids are gels and latex paints.
- *Dilatant liquids*: viscosity increases as shear rate increases. Mixers can bog down and stall after initially mixing such liquids. Dilatant liquids include slurries, clay, and candy compounds.
- *Thixotropic liquids*: viscosity decreases as shear rate or agitation increases, but when agitation is stopped or reduced, hysteresis occurs and viscosity increases. Often the viscosity will not return to the initial value. Thixotropic liquids include soaps, tars, vegetable oils, inks, glue, and peanut butter.

10.0. SUMMARY

The study of chemistry requires a student to explore many diverse laboratory exercises. Even though the experiments may be diverse, they usually involve similar laboratory techniques. A student completing an introductory chemistry course should have an understanding of basic chemistry laboratory techniques in areas of measuring, mixing transferring, separating, heating, etc.

11.0. TUTOR-MARKED ASSIGNMENTS

1. Why is it necessary to use a wire gauze on an iron ring when using a beaker/flask to heat or boil liquids? what would you use to secure the beaker/flask from falling?
2. What does a yellow flame in the bunsen burner indicate? how can this be corrected?
3. Why use a funnel to transfer large volume of liquid from one container to another?

12.0. REFERENCES AND FURTHER READING

1. Gelosa, D and Sliepcevich, A, *"Chemical laboratory techniques"* in "The World of Chemistry", ed. S. Carrà in Encyclopedia of Life Support Systems (EOLSS) Developed under the auspices of the UNESCO, Eolss Publishers, Oxford,UK, [http://www.eolss.net]

2. Brewster R., Vanderwerf C.A., and McEwen W.E., *Unitized Experiments in Organic Chemistry. Fourth Edition*, 577 pp. New York: D. Van Nostrand Co. 1977. [A versatile and flexible manual of organic chemistry featuring a rich variety of tested experiments.]

UNIT 4 - WEIGHING IN THE LABORATORY

7.0. INTRODUCTION

The world of weighing scale is very fascinating and interesting. These did not come to existence overnight. They have evolved from a barter system where units were absent compared to the modern day standards. When the standard units were accepted by businesses and governments across different regions, an industrial revolution was set to begin and everyone benefited from that. Today the old age classical weighing scales have become obsolete but they served the humanity for a long time. In the modern times, one can see the weighing scales ruling the industrial and domestic space.

8.0. OBJECTIVE

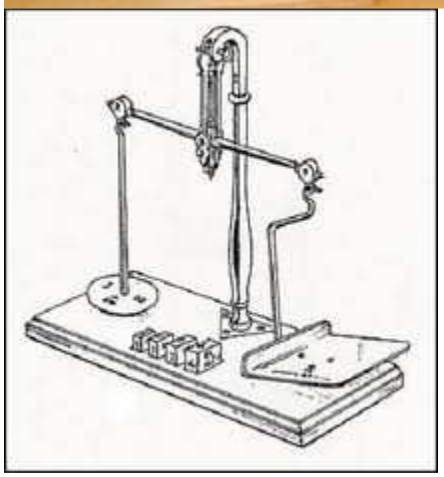
After studying this unit, you should be able to:

- Identify different equipment used in laboratory weighing
- Differentiate between a to top-loading balance and an analytical balance
- Carry out simple weighing experiments in the laboratory

9.0. MAIN CONTENT

9.1. Types of weighing balances

Here is a list of different products that one must know about.



1. The conventional **wooden or iron scale** was the oldest weighing machine invented by man. It consisted of two containers attached to each other through a pulley system. These weighing scales were meant for domestic purposes and they often reported wrong measurements. It was more of a weighing balance than a weighing scale.

2. The next stage was the **spring balance**. A spring connected to a fixed and rigid system that was used to weigh mass of objects by hanging them with the spring. The spring will develop tension in it and it will reflect a standard value on the rigid body attached to it. The value will reflect the weight of the object in standard unit of mass. Precision and accuracy were still missing in this case.

3. The modern **digital weighing scales** are the third kind of weighing machines invented so far. These are the products that are electronically operated and display results very precisely. They have many types, styles and weigh ranges. Broadly, they can be categorized into two categories namely Industrial Scales and Domestic Scales.

4. **Hydraulic Scales** are used mainly in the laboratories and industrial units. It made use of liquid displacement technique to weigh the mass of heavy objects. The object placed on the top of weighing panel displaces the liquid and the volume change indicates the weight of the object.
Today, the weighing scales have changed a lot over a course of time. Most of the modern invented equipments are precise, accurate and minimize the errors while weighing. These weighing machines constitute of electronic circuits, digital chips, electronic sensors and motion sensors. They are portable, lightweight and easy to operate and even an unskilled person can operate these devices.

9.2. Weighing

When mass amounts are specified in chemical procedures the following terms are commonly used:
a. "Weigh out about 2 g of ..... " This statement means that you are required to weigh an amount of approximately two grams. The accuracy to which this mass amount needs to be known is not high and the top-loading balance will suffice.

b. "Accurately weigh out about 0.2 g of ...." This statement means that you should, with the aid of the analytical balance, weigh out an amount that is close to 0.2 g, but you must know the exact amount to an accuracy of ± 0.1 mg. Note that this does not mean that you must weigh out exactly 0.2000 g. An amount between 0.1900 g and 0.2100 g is perfectly acceptable. However, you must know the exact amount to the nearest tenth of a milligram. When weighing out triplicate samples it is not necessary that all three weights be exactly the same, indeed, it is poor procedure to attempt to do so.

In this unit, you will be asked to make a variety of weighings. It is important for you to realize with what sort of accuracy these weighings should be made. Depending on the desired accuracy you should use the proper balance to make your weighings. There are two types of balances available to you in this course:

A. The Top-Loading Balance
The top-loading balance digitally displays a mass reading, in grams, to 2 decimal places. The uncertainty in a single reading on the top -loading balance is 0.05 g.

B. The Analytical Balance
The analytical balance is more accurate than the top-loading balance. Its digital display gives mass, in grams, to 4 places after the decimal. The uncertainty in a single reading on the analytical balance is ~ 0.0002 g. You will use this balance if the experiment calls for accurate measurement of mass. Never use the analytical balance if the top-loading balance will do. The analytical balances are usually located in the “BALANCE room” of a laboratory.

9.3. WEIGHING ON AN ANALYTICAL BALANCE

Weighing is a frequent step in analytical procedures, and the balance is an essential piece of laboratory equipment in most analyses. In spite of this, weighing is a common source of error that can be difficult to detect in the final analytical results. The procedure described here applies directly to electronic balances; therefore, certain portions of the procedure are not applicable to other types of balance. The weighing procedure can be separated into three basic steps: planning, checking the balance, and weighing the material.

9.3.1. Planning

The initial step is to assemble the proper equipment, such as containers for weighing, receiving vessels, forceps, pipettes, spatulas of proper size, and so forth. Use containers of size such that the loading capacity of the

balance is not exceeded. Make sure that the containers selected to receive the weighed material are clean and dry. Assemble the necessary chemicals if solutions or reagents are required. Preparation of the material to be weighed is often necessary. The material may require grinding or drying. Some materials may have been heated or stored in a refrigerator. Materials must be brought to the temperature of the balance before they are weighed. To avoid condensation of moisture, refrigerated materials must be allowed to come to room temperature before the container is opened.

9.3.2. Checking the balance

In the next step it is important to remember that, unless the balance is checked before each weighing operation is performed, errors can easily occur, resulting in faulty analytical data. The balance user should check the Balance Environment, Calibration, and Balance Uncertainties. Do not assume that the balance has been left in the proper operating condition by the previous user.

9.3.3. Balance Environment

The balance is placed in a suitable location with sufficiently low levels of vibration and air current. It must have a constant electrical supply. The balance and the surrounding work area have to be kept neat and tidy. It is good practice to use a camel's hair brush or its equivalent to dust the balance pan before any weighing so as to remove any materials that may have been left by the previous operator. [NOTE—Individuals must clean up debris, dispose of any spilled materials or paper, and remove the vessels and apparatus used in making the measurements.] When a balance is moved, it must be allowed to adjust to the temperature of its new environment and be recalibrated.

9.3.4. Calibration

If necessary, turn on the power, and allow the balance to equilibrate for at least 1 hour before proceeding with the calibration. (Microbalances may require up to 24 hours to reach equilibrium). If the balance power has gone off and then has come back on, as in a power outage, certain types of balance may display a message indicating that the balance must be calibrated before a weighing is made. If the operator touches the balance bar, the message may be cleared and the balance may display zeros; however, the balance will not give the correct weighing until it has been calibrated. Electronic analytical balances have an internal calibration system based on an applied load. The calibration applies for the current ambient temperature.

9.3.5. Balance Uncertainties

Drift Reduction - Drift is one of the most common errors, and it is also one of the easiest to reduce or eliminate. Balance drift can be present without the operators being aware of the problem. Check the sample, the balance, and the laboratory environment for the following causes of errors, and eliminate them:

- A balance door is open.
- Temperatures of the balance and the material to be weighed are not the same.
- The sample is losing or gaining weight.
- The balance has been recently moved but has not been allowed to equilibrate to its surroundings or has not been recalibrated.
- Air currents are present in the laboratory.
- Temperatures in the laboratory vary.
- The balance is not properly leveled.
- Laboratory operations are causing vibration.
- Hysteresis of the mechanical parts occurs during weighing.

9.3.6. Rules for Weighing

- Do not handle objects to be weighed with bare hands. Use tongs or paper towels if no appropriate tongs are available.
- Never weigh chemicals directly on the balance pan; use a glass container or weighing paper or filter paper.
- If you spill a chemical on the top -loading balance, clean it immediately. Never spill chemicals inside the analytical balance enclosure. Keep the weighing chamber and weighing pan clean.
- Before using the balance, be sure that the pan is clean. If it is dirty report it to your instructor, then brush it off with the brush provided.
- Do not overload the balance. The maximum capacity of the top -loading balance is 620 g. The maximum capacity for the analytical balance is 110 g.
- Do not weigh hot or cold objects on the balance. Hot objects will give erroneously low readings due to buoyancy of hot air, while cold objects will give high readings.
- Check to be sure that the balance is level. It is level if the bubble in the Level Indicator is in the centre while the balance is "OFF". Your instructor may need to adjust the leveling Feet.

9.3.7. Weighing by difference to overcome the problem of balance calibration errors

How accurate are your balance readings? There is no way for you to know. In order to overcome the problem of inaccurate readings due to lack of calibration or miscalibration, chemists designed a method called weighing by difference. It does not matter how far off each reading of your balance is if you weigh your sample by difference.

Example: Weighing a solid sample by difference from a beaker: To find the mass of the sample in the beaker, first the empty beaker is placed on the balance and the mass is read. Then the solid is added to the beaker and the mass of beaker with solid is read. The mass of the solid sample is the difference between the two readings.

Notice that the weighing pan of the analytical balance is enclosed in glass. This glass case is designed to protect the balance from temperature fluctuations and air currents that cause the balance to drift - that is, the digital display continues to change in one direction (up or down). To make sure that the temperature of the air in the balance does not change, keep your hands (which are warmer than the air in the balance) out of the enclosure as much as possible, and keep the balance doors closed.

Using Balance 1, a calibrated balance, the following masses are recorded:
Mass of Empty Beaker 24.7423 g
Mass of Beaker + Solid 26.7587 g
Mass of sample: 26.7587g – 24.7423 g = 2.0164 g

Using Balance 2, an uncalibrated balance (all readings are low by 0.5000 g) the following masses are recorded:
Mass of Empty Beaker 24.2423 g
Mass of Beaker + Solid: 26.2587 g
Mass of sample: 26.2587g – 24.2423 g = 2.0164 g
Does it matter which balance, 1 or 2, you choose to use? _____
Explain: _____

Throughout the weighing process you should protect the object you are weighing from coming in contact with your hands by handling it with a paper towel.

9.4. Sample Weighing Experiment

3.4.1. Using the Top-loading balance

1. Get a numbered metal block from the side bench. Record its number on the Report Sheet.
2. Locate the top-loading balances in your lab.
3. Check the balance for dust or chemicals, brushing off any that you see.
4. Check the balance level. Look for the Level Indicator at the back of the balance. The bubble should be in the centre of the circle. If it is not, ask your instructor to level the balance for you. Students should not attempt to adjust the level themselves.
5. Turn the balance ON. Wail until it displays "0.00 g", and then place your metal block on the pan.
Read the balance and record the mass directly onto your Report Sheet. (USE INK!)
Remember, if you record a datum incorrectly on your report sheet, just cross it
out with a single line and write the correct value next to or above it.
6. Turn off the balance. Please always remember to turn off the top -loading balance when finished.
7. Using the same metallic block, repeat steps 2 -6 using two other top-loading balances.

3.4.2. Using the Analytical balance

1. Get a numbered metal block from the side bench. Record its number on the Report Sheet.
2. Locate the analytical balances in your lab.
3. Check the balance for dust or chemicals, brushing off any that you see.
4. Check the balance level. Look down through the glass top to the floor of the balance. The levelling bubble is visible through a hole in the floor. If the bubble is not centred, ask your instructor to level the balance for you. Students should not attempt to adjust the level themselves.
5. Push the ON button. Allow the balance to calibrate. After about 5 seconds, it will display "0.0000g".
The balance is now ready to be used. If the balance does not read zero, push the T (tare) button. The balance should read 0.0000 g.
6 Slide open the door and carefully place the metal block on the centre of the pan to avoid corner load errors.
7. Close the balance door and after about 5 seconds read and record the mass of the metal block on your Report Sheet. (Remember: all data must be recorded in INK.)
8. Carefully remove the object and close the door, then push the "OFF" button.
9. Repeat steps 2 to 8 using two other different analytical balances to find the mass of the same metal block. Record your data on your Report Sheet.

3.4.3. Weighing by difference using the Tare function of the Analytical balance

1. Clean the outsides of three 125 ml Erlenmeyer flasks. Make sure the Erlenmeyer flasks are completely dry on the outside. Label the flasks: 1, 2, and 3 by writing the numbers on the white patches on the flasks.
Reminder: Throughout the weighing process you should protect the Erlenmeyer flasks from your hands with a paper towel.
2. Take the following items with you to the weighing room: Erlenmeyer flask #1, Report Sheet, pen.
3. Turn the analytical balance on and allow it to calibrate.
4. Once it reads 0.0000g, place flask #1 on the balance pan and close the balance door.
5. Push the T (tare) button and the display will read 0.0000g, even though the flask is on the balance pan. Report this reading as the Initial reading on your report sheet.
6. There is some NaCl in a plastic weighing boat next to the analytical balance. Use the long-handled plastic spoon provided to carefully transfer, without spilling, a small amount of NaCl into flask #1. Tap the handle of the spoon with your finger to get it to fall out of the spoon. Continue to add NaCl a little at a time until the display reads approximately 0.3 g. Close the balance door and record the reading on your Report Sheet. Remember that the analytical balance reads and should be reported to 4 decimal places.

7. When you are finished, remove the Erlenmeyer flask, close the balance door, touch the tare bar then turn the balance off.
8. Repeat steps 1 through 7, weighing samples of about 0.3 g into each of the remaining flasks, #2 and #3.
9. Wash your Erlenmeyer flasks (do a final rinse with deionized water) and store them for your next lab period.

3.5. REPORT SHEET

EXPERIMENT: Weighing

DATA:

A. TOP–LOADING BALANCE
Metal block # _____
Readings: 1. _____ 2. _____ 3. _____

Instructor's initials _____

B. ANALYTICAL BALANCE
Readings: 1. _____ 2. _____ 3. _____

Instructor's initials _____

C. WEIGHING BY DIFFERENCE USING THE ANALYTICAL BALANCE.

	Sample #1	Sample #2	Sample #3
Initial reading			
Final reading			
Calculated mass of NaCl sample			

10.0. CONCLUSION

By carefully following the procedures outlined above, laboratory personnel will eliminate many errors that might be introduced into weighing procedures. However, it is important for each balance to be serviced and calibrated regularly by a specially trained internal or external service person. The balance should be tested using appropriate weights traceable to standardization. No repairs should be made to any balance by anyone other than a qualified maintenance person.

11.0. TUTOR-MARKED ASSIGNMENTS

1. If you weigh an object whose mass is about 13 g, how many significant figures will there be in its mass if you weigh it on the top-loading balance?

2. If a procedure tells you weigh the sample accurately, which balance would you use, the analytical or the top-loading balance?

3. _____

4. An analytical reads to 0.1 milligram. How many grams is that?
5. _____

6. If a procedure tells you to "Add about 3 g of ammonium sulfate" which balance would you use, the analytical or the top-loading balance?
7. _____

8. If a procedure tells you to "Weigh to the nearest tenth of a gram about 3 grams," which balance would you use, the analytical or the top -loading balance?

9. If a procedure says "Weigh accurately about 0.8 g", which balance would you use, the analytical or the top-loading?
10. _____

11. The vessel to be weighed should be placed (off to the side, or in the centre) of the balance pan.
12. _____

13. After heating a sample in a dish should you weigh it immediately to save time or should you allow it to cool completely before weighing it?
14. _____

15. During the same experiment, one must be careful to always use (the same, different) balance.
16. _____

17. To avoid the errors in mass due to the use of balances that are not calibrated, one should weigh by a method called…
18. _____

19. If you weigh a hot object on the balance, will the reading be higher, the same, or lower than it will be after it cools to room temperature?
20. _____

21. If you weigh a cold object on the balance, will the reading be higher, the same, or lower than it will be after it warms up to room temperature?
22. _____

12.0. REFERENCES

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- Pasto, D.J. , Johnson, C.R. and Miller, M.J., *Experiments and Techniques in Organic Chemistry*, Prentice Hall, New Jersey, 1992.

MODULE 2

Unit 1
Distillation – Separation of a Mixture

Purpose:

- To purify a compound by separating it from a non-volatile or less-volatile material.
- To separate a mixture of two miscible liquids (liquids that mix in all proportions) with different boiling points.

Equipment / Materials:

large test tubes(3)	test tube rack (1)	10-mL graduated cylinder	50-mL round bottom flask
clamp (1 or 2)	heating mantle	condenser (1 or 2)	thermometer
ringstand	glass adaptor	Grease	rubber tubing (2)
boiling chips	thermometer adaptor	50- mL round bottom flask	unknowns (A and B)

Discussion:

Distillation is one of the oldest and still most common methods for both the purification and the identification of organic liquids. It is a physical process used to separate chemicals from a mixture by the difference in how easily they vaporize. As the mixture is heated, the temperature rises until it reaches the temperature of the lowest boiling substance in the mixture, while the other components of the mixture remain in their original phase in the mixture. The resultant hot vapour passes into a condenser and is converted to the liquid, which is then collected in a receiver flask. The other components of the mixture remain in their original phase until the most volatile substance has all boiled off. Only then does the temperature of the gas phase rises again until it reaches the boiling point of a second component in the mixture, and so on.

The boiling point of a substance—determined by distillation—is a useful physical property for the characterization of pure compounds.

At any given temperature a liquid is in equilibrium with its vapour. This equilibrium is described by the vapour pressure of the liquid. **The vapour pressure** is the pressure that the molecules at the surface of the liquid exert against the external pressure, which is usually the atmospheric pressure. The vapour pressure is a very sensitive function of temperature. It does not increase linearly but in fact increases exponentially with temperature. The vapour pressure of a substance roughly doubles for every increase in 10 °C, Figure 1.
When the vapour pressure of the liquid equals the applied pressure, the liquid boils. Thus, **the boiling point** of a liquid is the temperature at which the vapour pressure equals the applied pressure. The **normal boiling point** of a liquid is the temperature at which the vapour pressure of a liquid equals atmospheric pressure (1 atm).
The boiling point of a liquid is a measure of its volatility.

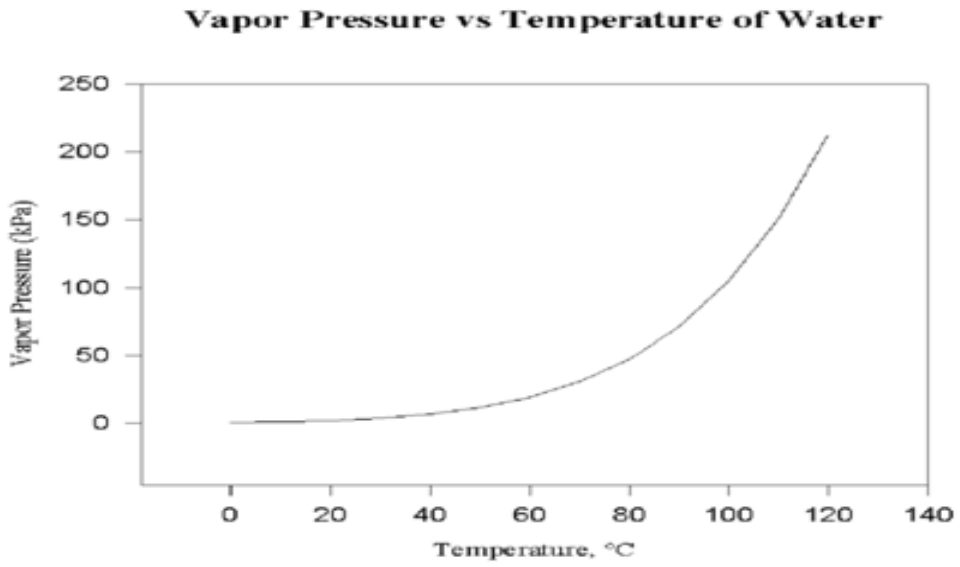


Fig. 1: Vapour pressure dependence on temperature for water

The successful application of distillation techniques depends on several factors. These include the difference in vapour pressure (related to the difference in the boiling points) of the components present, the size of the sample, and the distillation apparatus. Distillation relies on the fact that the vapour above a liquid mixture is richer in the more volatile component in the liquid, the composition being controlled by Raoult's law:

In an ideal solution the partial pressure (P_A) of component *A* at a given temperature is equal to the vapor pressure P_A^o of pure *A* multiplied by the mole fraction of *A* (X_A) in solution.
Consider an ideal solution of *A* and *B*:

$$X_A = \frac{n_A}{n_A + n_B}, \quad X_B = \frac{n_B}{n_A + n_B}, \qquad \text{and } X_A + X_B = 1$$

n_A and n_B represent the number of moles of components A and B.
$P_A = X_A P_A^o$, $P_B = X_B P_B^o$ and P_T (total vapour pressure) = $P_A + P_B$
This relationship, derived from Raoult's law, is capable of describing the boiling point behaviour of compound A in a mixture of compounds under a variety of different circumstances. The boiling point of the solution is reached when P_T is equal to the pressure applied to the surface of the solution.

There are two major types of distillation are to be considered:

Simple Distillation
Used frequently in the organic chemistry teaching labs, **Figure 2**. It is often considered when:

c. the liquid is relatively pure to begin with (e.g., no more than 10% liquid contaminants)
d. essentially a pure material is separated from a non-volatile or from a solid contaminant
e. the liquid is contaminated by a liquid with a boiling point that differs by at least 70°C

Simple distillation involves a single equilibration between the liquid and vapour. This distillation is referred to as involving one theoretical plate.

Fractional Distillation

The principle of fractional distillation is based on the establishment of a large number of theoretical vaporization-condensation cycles (theoretical plates): the apparatus of a simple distillation is modified by inserting a fractionating column between the distillation flask and the distillation head, **Figure 3**. The fractionating column provides a large surface area in which the initial distillate is redistilled and condensed again. This process continues as the vapours rise up the column until the vapours finally make it into the condenser. These vapours and the final distillate will contain a greater percentage of the lower boiling liquid. Continuous repetition of the redistillation process in fractional distillation gives good separation of the volatile liquid components.

A **simple distillation** apparatus (**Fig. 2**) is less efficient than a **fractional distillation** apparatus (**Fig. 3**), but is used to purify materials containing only small amounts of impurities with much higher or lower boiling points.

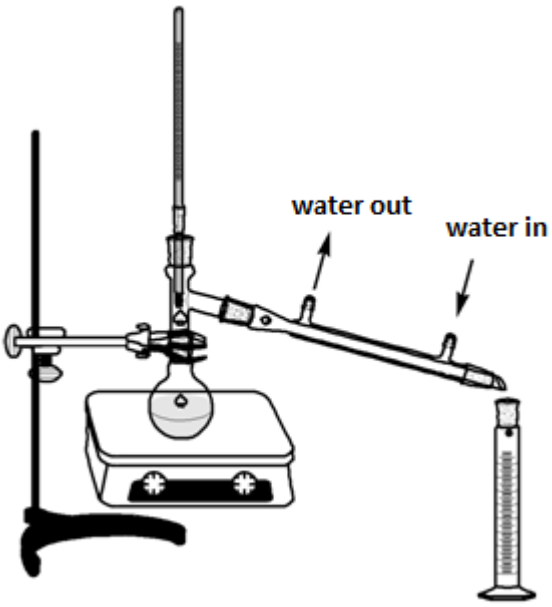


Fig. 2: - The apparatus used in simple distillation

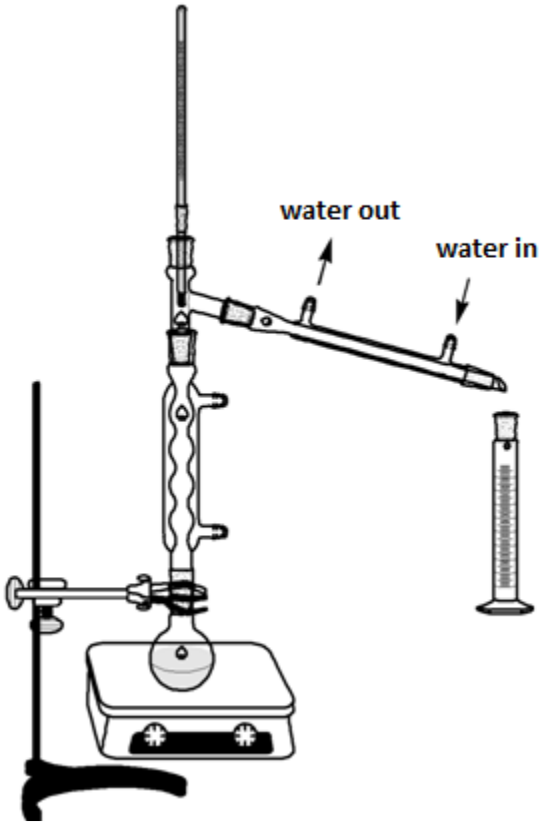
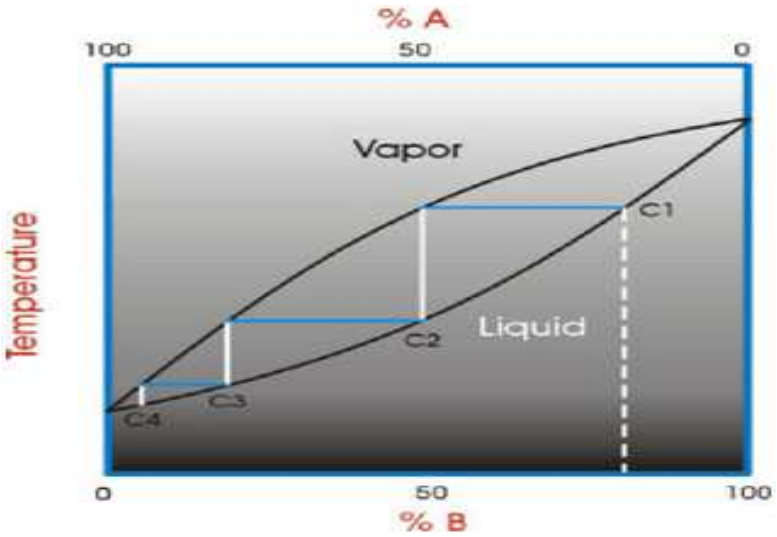


Fig. 3: - The apparatus used in fractional distillation

Not all mixtures of liquids obey Raoult's law, such mixtures; called **azeotropes**, mimic the boiling behaviour of pure liquids. These mixtures, when present at specific concentrations, usually distil at a constant boiling temperature and cannot be separated by distillation. Examples of such mixtures are 95% ethanol-5% water (bp 78.1 °C).

To understand the nature of simple distillation, fractional distillation and azeotropes, we need to look at vapour/liquid diagrams for pairs of solvents. The graph below (Fig. 4) shows such a diagram for two solvents, A and B. A is the lower boiling material. The bottom of the graph shows the liquid state and the top of the

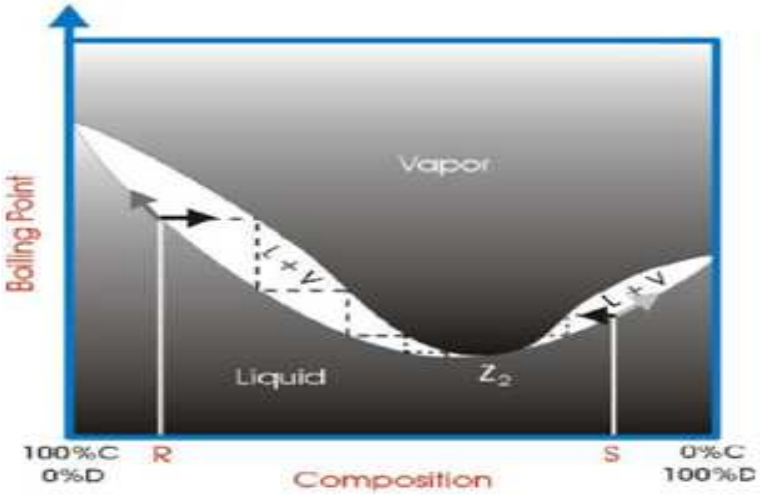
graph shows the vapour state. The area in between the two curves shows what is happening in the distillation column. If we start with a mixture of A and B that corresponds to the dashed white line on the graph and the letter C1 (concentration 1), (The mixture is vaporized (distilled) follow the horizontal blue line until it reaches the vapour curve. This is concentration C2 in the diagram. This process has improved the concentration of A, the lower boiling component. The material is then condensed. Follow the white vertical line down to the liquid curve.



If this was simple distillation, we could stop now. It can be seen that the purification effected by the simple distillation of such a mixture of volatile liquids is very imperfect.

However, in fractional distillation the distillation process continues. The condensed material is vaporized again. Follow the blue horizontal line across from the liquid curve at C2 to the vapour curve. There is another improvement in the concentration of the lower boiling component A. The vapour is condensed again. Follow the white vertical line down to the liquid curve.

This number of times that the process of vaporization and condensation occurs depends on the efficiency of the distillation column. The more efficient the distillation column, the more times this happens and the purer the final product will be.
With azeotropes, the vapour liquid curves are not ideal and have a point where the vapor curve meets the liquid curve. This point is called the azeotrope point.



Example (1)

Calculate the mole fraction of each compound in a mixture 95.0 g ethanol, CH_3CH_2OH and 5.0 g water.

Answer

n(ethanol) = $n_e = \frac{95.0}{46} = 2.07$ mole ethanol n(water) = $n_w = \frac{5.0}{18} = 0.28$ mole water

n(total) = n_t = n(ethanol) + n(water) = 2.07 + 0.28 = 2.35 mole

X (mole fraction of ethanol) = $X_e = \frac{n_e}{n_t} = \frac{2.07}{2.35} = 0.88$

X (mole fraction of water) = $X_w = \frac{n_w}{n_t} = \frac{0.28}{2.35} = 0.12$

Example (2)

Given the following mole fraction and vapour pressures for miscible liquids A and B, calculate the composition (in mole percentage) of the vapour from a distilling an ideal binary solution at 150 ^oC and 760 mm Hg, for the solution.

Answer

$X_A = 0.40$, $P_A^o = 1710$ mmHg

$X_B = 0.60$, $P_A^o = 127$ mmHg

$P_{total} = P_A + P_B = X_A . P_A^o + X_B . P_B^o = (0.40 \times 1710) + (0.60 \times 127) = 760$ mmHg

% A = $\frac{(0.40 \times 1710)}{760} \times 100\% =$ **90%**

% B = $\frac{(0.60 \times 127)}{760} \times 100\% =$ **10%**

In this experiment you will perform several distillations. You will compare distillations of a mixture of methanol and methylene dichloride using the glassware set-up for a **simple distillation** and one for **fractional distillation**. You will also measure the boiling point-range for an unknown compound and use this information as an aid in its identification from Table 1 of possible substances. Pairs of students will perform this experiment. Each pair will conduct either the simple distillation procedure or the fractional distillation procedure.

Results will be shared between two groups, so that everyone has data for both simple and fractional distillation.

Also remember methanol and methylene dichloride are flammable. Never add boiling chips to hot liquid and never distil the flask to dryness. Some organic compounds form peroxide, which explode upon dryness and concentration. All glassware must be clamped in place.

Substance	**Boiling Point (^oC)**	**Substance**	**Boiling Point (^oC)**
Pentane	36.1	Methanol	65
Hexane	69	Ethanol	78-79
Toluene	111	Propanol	97-98
Octane	125.7	2-Propanol (isopropanol)	82-83
Methyl acetate	57	Water	100
Tetrahydrofuran	65	t-Butyl alcohol	83
1-propanol	97	Cyclohexane	80.7
3-Ethylpentane	93.5	Methylene dichloride	40
2,2,4-Trimethylpentane	99.2	Bromoform	146-150
Acetone	56- 57		

EXPERIMENT – Distillation of a Mixture

Experimental Procedures

Place 30 ml of an unknown liquid mixture (15 ml A + 15 ml B) that is to be purified by simple distillation and for which the boiling point range is to be determined.

Assemble the assigned distillation apparatus (simple or fractional). Transfer the unknown liquid to a 50 mL round bottom flask (this will be the distilling pot). Add one boiling stone, and proceed to distil the liquid into a 10 mL graduated cylinder (this will be the receiver). Check the position of the thermometer (the bulb of the thermometer must be below the arm of the distillation head) and make sure that the bottom of the distilling pot touching the heating surface of the heating mantel. Securely attach a piece of condenser tubing to each condenser outlets. Securely connect the other end of the "water in" tubing to the water jet in the sink (or hood). Place the other end of the "water out" tubing in the sink (or back of the hood). Plug in the heating mantle and before heating your distillation apparatus or turning on the water for cooling the condenser, have your laboratory instructor check your distillation apparatus.

After your laboratory instructor has checked your apparatus, slowly turn on the water for condenser, and begin heating. Adjust the heating mantle to maintain a distillation rate of one drop per second. As the lower boiling component is distilled, the boiling point of the mixture in the distillation flask will increase.

Record the temperature after the first drop is collected and again after every 2 ml of distillate is collected. After the 10 ml of distillate has been collected, you will have to empty the graduated cylinder into a test tube as it fills. Cover and label the test tube first fraction (component A). **KEEP IT**.

Collect the next 10 ml of distillate, again recording the temperature after every 1 ml of distillate. After the second 10 ml of distillate has been collected, you will have to empty the graduated cylinder into a test tube as it fills and **DISCARD IT** in organic waste container.

Collection of last portion of distillate should continue until the temperature remains constant. If the distillation flask is approaching dryness, remove the heat source immediately and after cooling, transfer the distillate and any remaining liquid from the flask to the third test tube (component B). **KEEP IT**.

Determine the boiling point range of the first fraction of the collected liquid and the third portion of the collected liquid. Identify the unknowns by their boiling points using the possible boiling points of compounds.

Construct a table like that given below, to record the temperature at the distillation "head" as a function of volume distilled. You will record your data in report form. Plot distillate temperature (^{o}C) vs. volume of distillate (ml) collected for the mixture with and without the fractionating column and use a graph sheet to determine the boiling points of the two compounds in the mixture and identify the compound in the mixture. Both sets of data will be plotted on the same graph, using different symbols (colours). Label the two curves.

Volume distilled (mL)	0	2	4	6	8	10	12	14
Temperature (^{o}C, simple or fractional)								
Volume distilled (mL)	16	18	20	22	24	26	28	30
Temperature (^{o}C, simple or fractional)								

Separating a Mixture by Distillation
REPORT SHEET

Name _____

Instructor _____ **Date** _____

Volume distilled (mL)	0	2	4	6	8	10	12	14
Temperature (^{o}C, simple or fractional)								
Volume distilled (mL)	16	18	20	22	24	26	28	30
Temperature (^{o}C, simple or fractional)								

point of the first compound (A): _____ ^{o}C
range

Literature boiling point of the first compound (A): _____ ^{o}C
range

Observed boiling point of the second compound (B): _____ ^{o}C
range

Literature boiling point of the second compound (B): _____ ^{o}C
range

Identity of Mixture

Compound A is _____ Compound B is _____
(name) (name)

Pre-laboratory questions - Due before lab begins.

1. The normal boiling point of cyclohexane is 81.0 ^{o}C. What is the vapor pressure of cyclohexane at 81.0 ^{o}C?
2. A mixture of two miscible liquids with a widely different boiling point is distilled. The temperature of distilled liquid is observed to plateau and then drop before rising again. Explain the temperature drop.
3. What effect would a decrease or increase in barometric pressure have on the boiling point?
4. If a mixture distilled rapidly, the separation of its compound is poorer than if the mixture is distilled slowly. Explain.
5. A chemist has a small amount of compound (b.p. = 65 ^{o}C) that must be fractionally distilled. Yet, the Chemist does not want to lose any of the compound to hold up on the column. What should the chemist do?

Post-laboratory questions - Due after completing the lab.

1. One mole of compound A, with vapor pressure 400 mmHg at 50 ^{o}C, mixed with 3 moles of compound B, with vapor pressure 480 mmHg at 50 ^{o}C to form a homogeneous solution. What is the vapor pressure of mixture at 50 ^{o}C?
2. Why should a distilling flask be filled not less than 1/3 filled or more than 2/3 full?
3. A 50% aqueous solution of ethanol (50 ml total) is distilled and collected in 10ml fractions. Predict the boiling range of each fraction.

Fractions	Vol. Collected (mL)	Temperature (^{o}C) predicted
1	10	78
2	20	78
3	30	78 – 100
4	40	100
5	50	100

4. What is the mole fraction of each component if 3.9 g of benzene (C_6H_6) is dissolved in 4.6 g of toluene (C_7H_8)?
5. Define the following terms
    a. Reflux
    b. Dalton's law
    c. Raoult's law

Unit 2

Purification – Recrystallization of Benzoic acid

Purpose:

a) To purify samples of organic compounds that are solids at room temperature
b) To dissociate the impure sample in the minimum amount of an appropriate hot solvent

Equipment / Materials:

Hot plate	digital scales	50 mL beaker	impure benzoic acid
Büchner funnel	Mel-temp apparatus	weighing paper	benzoic acid
tubing (hose)	stirring rod	boiling stones (chips)	ice
25 mL graduated cylinder	spatula	filter paper	
125-mL Erlenmeyer flask	rubber		

Discussion:

The products of chemical reactions can be impure. Purification of your products must be performed to remove by-products and impurities. Liquids are customarily purified by distillation, while solids are purified by recrystallization (sometimes called simply "**crystallization**").

Recrystallization is a method of purifying a solid. There are two types of impurities: those more soluble in a given solvent than the main component and those less soluble. (If there are any impurities that have the same solubility as the main component, then a different solvent needs to be chosen).

When organic substances are synthesized in the laboratory or isolated from plants, they will obviously contain impurities. Several techniques for purifying these compounds have been developed. The most basic of these techniques for the purification of organic solids is recrystallization, which relies on the different solubilities of solutes in a solvent. Compounds, which are less soluble, will crystallize first. The crystallization process itself helps in the purification because as the crystals form, they select the correct molecules, which fit into the crystal lattice and ignore the wrong molecules. This is of course not a perfect process, but it does increase the purity of the final product.

The solubility of the compound in the solvent used for recrystallization is important. In the ideal case, the solvent would completely dissolve the compound to be purified at high temperature (usually the boiling point of the solvent), and the compound would be completely insoluble in that solvent at room temperature or at zero ^{o}C. In addition, the impurity either would be completely insoluble in the particular solvent at the high temperature, or would be very soluble in the solvent at low temperature. In the former case, the impurity could be filtered off at high temperature, while in the latter case the impurity would completely stay in solution upon cooling. In the real world, this will never happen and recrystallization is a technique that has to be practiced and perfected.

Regardless of crystallization method, the purity of the solid can be verified by taking the melting point.

A good (suitable) recrystallization solvent will dissolve a large amount of the impure compound at temperatures near the boiling point of the solvent. Small amount of compound being purified should remain in solution at low temperatures, between approximately 25 and –5 ^{o}C. Low solubility at low temperatures minimizes the amount of purified compound that will be lost during recrystallization.

A suitable recrystallization solvent should also be partially volatile in order to be easily removed from the purified crystals. The solvent should not react with the compound being purified and it should have the boiling point below the melting point of the compound being purified, in order to prevent the compound from melting before dissolving (oiling out). In selecting a good recrystallization solvent one should also consider flammability, toxicity, and expense.

In selecting a solvent consider that "like likes like". Polar compounds dissolve polar compounds and non-polar compounds dissolve non-polar compounds. The most commonly used recrystallization solvents are presented in the following table.

solvent	**formula**	**polarity**	**boiling point (^oC)**
water	H_2O	very polar	100
ethanol	CH_3CH_2OH	polar	78
methanol	CH_3OH	polar	65
dichloromethane	CH_2Cl_2	slightly polar	40
diethyl ether	$(CH_3CH_2)_2O$	slightly polar	35

Organic compounds with one polar functional group and a low number of carbon atoms such as methanol, ethanol, and n-propanol are highly soluble (miscible) in water. These alcohols form hydrogen bond with water due to the polar –OH functional group. As the number of carbons per polar functional group increase, solubility decreases. The solubility of alcohols with four to five carbons is given in the following table.

alcohol	**Formula**	**Solubility (g/100 ml H_2O)**
n-butanol	$CH_3CH_2CH_2CH_2OH$	8
n-pentanol	$CH_3CH_2CH_2CH_2CH_2OH$	2
n-hexanol	$CH_3CH_2CH_2CH_2CH_2CH_2OH$	0.5
n-pentanol	$CH_3CH_2CH_2CH_2CH_2CH_2CH_2OH$	0.1

Compounds with six or more carbons for each polar group will not be very soluble in polar solvents but will be soluble in non-polar solvents such as benzene and cyclohexane.

If a single solvent cannot be found that is suitable for recrystallization, a solvent pair often used. The solvents must be miscible in one another. Some commonly used solvent pairs are water-ethanol, acetic acid – water, ether-acetone. Typically, the compound being recrystallized will be more soluble in one solvent than the other. The compound is dissolved in a minimum amount of the hot solvent in which it is more soluble.

The following formulae are used in solubility problems:

$$\%\ \text{loss in cold solvent} = \frac{\text{solubility in cold solvent}}{\text{solubility in hot solvent}} \times 100\ \%$$

$$\%\ \text{recovery of solid} = \frac{\text{mass recovered}}{\text{mass dissolved}} \times 100\ \%$$

Example (1)

The solubility of solid "X" in hot water (5.50 g/100 ml at 100 ^{o}C) is not very great, and its solubility in cold water (0.53 g/100 ml at 0 ^{o}C) is significant. What would be the maximum theoretical percent recovery from crystallization of 5.00 g of solid "X" from 100 ml water? Assuming the solution is chilled at 0 ^{o}C.

Answer

$$\% \text{ loss in cold solvent} = \frac{\text{solubility in cold solvent}}{\text{solubility in hot solvent}} \text{ x } 100\ \%$$

$$\% \text{ loss in cold solvent} = \frac{0.53}{5.50} \text{ x } 100\ \% = 9.64\%$$

$$\% \text{ recovery of solid} = \frac{\text{mass recovered}}{\text{mass dissolved}} \text{ x } 100\ \%$$

$$\% \text{ recovery of solid} = \frac{(5.50 - 0.53)}{5.50} \text{ x } 100\ \% = 90.4\%$$

Example (2)

The solubility of compound "X" in ethanol is 0.80 g per 100 ml at 0 ^{o}C and 5.00 g per 100 ml at 78^{o}C. What is the minimum volume of ethanol needed to recrystallize a 12.00 g sample of compound "X"? How much would be lost in the recrystallization, that is, would remain in the cold solvent?

Answer

$$\text{Volume of ethanol needed at 78 °C} = \frac{12.00\text{ g}}{5.00\text{ g}} \text{ x } 100\text{ ml} = 240\text{ ml}$$

$$\text{Mass of sample remaining in the cold solvent at 0 °C} = \frac{240\text{ ml}}{100\text{ ml}} \text{ x } 0.80\text{ g} = 1.9\text{ g}$$

or

$$\% \text{ loss} = \frac{0.80}{5.00} \text{ x } 100\% = 16\% \Rightarrow 12.00 \text{ x } 16\% = 1.92\text{ g}$$

The actual laboratory we will do is the recrystallization of benzoic acid from water using the temperature gradient method. Benzoic acid is not very soluble in cold water, but it is soluble in hot water. The purpose of this experiment is to learn the technique of recrystallization by purifying benzoic acid.

EXPERIMENT – Recrystallization of Benzoic Acid

Experimental Procedures

Using a weighing paper, weigh out about 1.00 g of "impure Benzoic acid for recrystallization" and transfer it to a 125-ml Erlenmeyer flask. Add about 20 ml distilled water, using a graduated cylinder, to the flask and bring the mixture to the boiling point by heating on a hot plate, while stirring the mixture and boiling gently to dissolve benzoic acid completely. (Fig. 1)

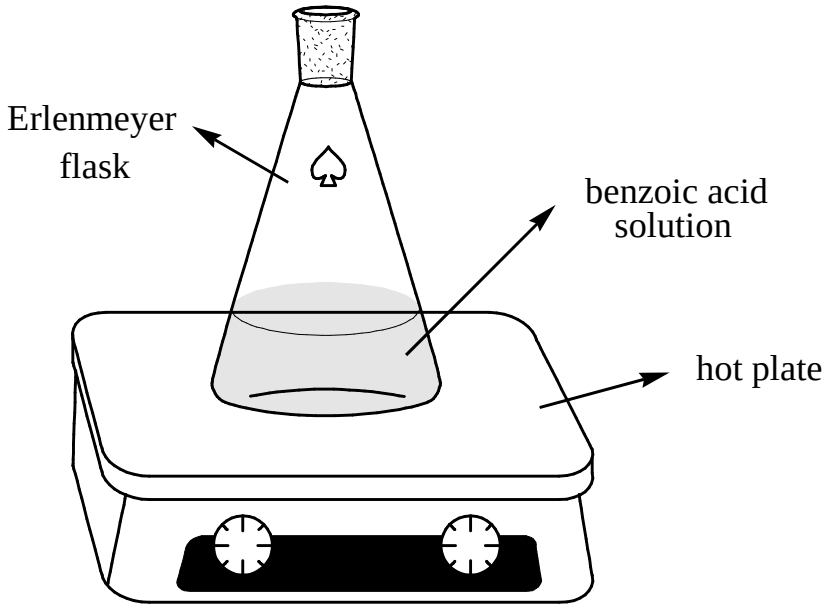


Fig. 1: Dissolving benzoic acid

Remove the flask from the hot plate and examine the solution. If there are particles of benzoic acid still undissolved, then add an additional amount of hot or cold water in small increments and resume heating the solution. The objective is to dissolve the entire solid in only as much as hot or near boiling solvent (water) as is necessary. Do not add too much water or the solution will not be saturated and the yield of purified benzoic acid will be reduced. Keep adding water in small amounts (several drops at a time from a Pasteur pipette) until all of the benzoic acid is dissolved and the solution is boiling.

If the solution is completely clear (though not necessarily colourless) and no solid benzoic acid is visible, then add additional 10-15 ml water to the mixture and place the Erlenmeyer flask on a countertop where it will not be disturbed and cover with an upside-down small beaker (to prevent dust contamination). Allowing the flask to cool slowly will give the best-shaped crystals after about 5-10 minutes. If crystallization does not occur after 10 minutes, scrape the sides of the flask above the level of the solution with the sharp end of a glass rod hard enough to audibly scratch the interior surface of the flask. This may dislodge some undetectable, small crystals that will drop into the solution and "seed" the solution, helping to induce crystallization. A seed crystal can serve as a nucleation point for the crystallization process. Cooling the solution in an ice bath may also help at this point.

When the crystals have formed completely (may required ice bath), collect your solid chemical by setting up a vacuum (suction) filtration on a properly fitted filter paper in a clean Büchner funnel apparatus as described by your instructor. (Fig. 2)

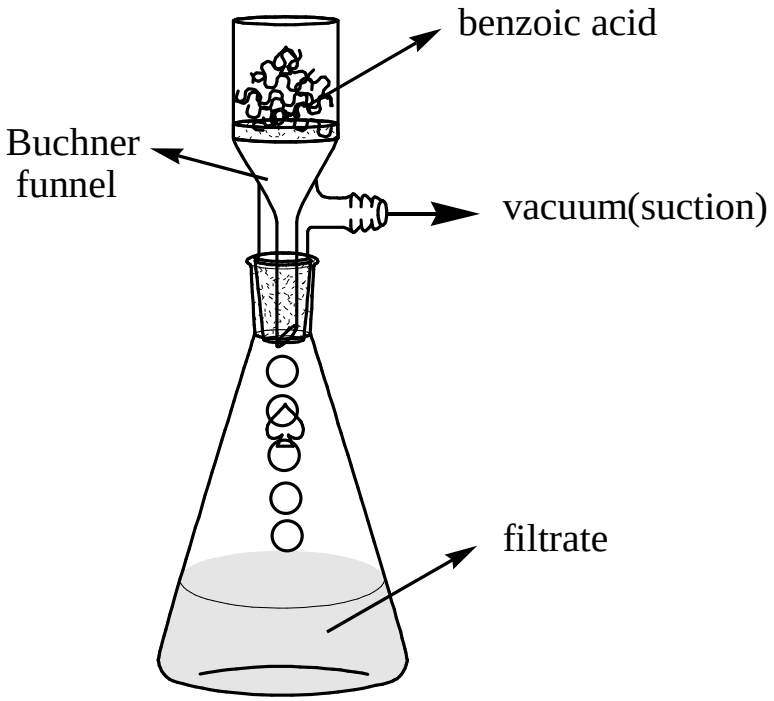


Fig. 2: Büchner funnel and suction flask

Pour the chilled mixture into the Buchner funnel. The water should filter quickly - if not, check for vacuum leaks. Get all the crystals out of the flask using a spatula or stirring rod. Rinsing with 1 or 2 ml of **cold** water helps get the crystals out of the flask, and rinsing helps remove impurities.

Let the aspirator run for a few minutes to start air-drying the crystals. Then use a spatula to lift the filter paper and crystals out of the Buchner funnel, then press them as dry as possible on a large clean paper towel (hand dry), allow them to dry completely, and transfer the dry sample to a pre-weigh weighing paper. Determine the weigh the DRY crystals of recovered benzoic acid.

Calculate the percent recovered using the following *written* formula and determine the melting point of your recrystallized benzoic acid.

$$\% \text{ Recovered} = \frac{\text{Mass of benzoic acid obtained after recrystallization}}{\text{Mass of benzoic acid before recrystallization}} \text{ x } 100$$

Note: Submit product to the instructor in a properly labelled container.

Data and Results (Recrystallization)

REPORT SHEET

Name _____

Instructor _____

Date_____

1. Sample name _____

2. Data on the impure Benzoic acid

a. Mass of the benzoic acid + weighing paper _____ **g**

b. Mass of weighing paper _____ **g**

c. Mass of impure benzoic acid _____ **g**

3. Data for recrystallized benzoic acid

a. Mass of recrystallized benzoic acid + weighing paper _____**g**

b. Mass of weighing paper _____ **g**

c. Mass of recrystallized benzoic acid _____**g**

d. Calculation of percentage recovery

(show calculation)

e. Melting point of recrystallized benzoic acid _____ **^{o}C**

f. Structural formula of the benzoic acid

Pre-laboratory Questions - Due before lab begins.

1. What is the ideal solvent for crystallization of a particular compound? What is the primary consideration in choosing a solvent for crystallizing a compound?
2. Impure benzoic acid was dissolved in hot water. The container of solution was placed in an ice-water bath instead of being allowed cooling slowly. What will be the result of cooling the solution in this manner?
3. Outline the successive steps in the crystallization of an organic solid from a solvent and state the purpose of each operation.
4. Compound X is quite soluble in toluene, but only slightly soluble in petroleum ether. How could these solvents be used in combination in order to recrystallize X?
5. 0.12 g of compound "Y" dissolves in 10 ml of acetone at 25 ^{o}C and 0.85 g of the same compound dissolves in 10 ml of boiling acetone. What volume of acetone would be required to purify a 5.0 g sample of compound?

Post-laboratory Questions - Due after completing the lab.

1. Give some reasons why Suction filtration (vacuum) is to be preferred to gravity filtration.
2. A student recrystallized some impure benzoic acid and isolated it by filtration. He scraped the purified benzoic acid off the filter paper after it had dried and took the melting point as a test for purity. He was surprised that most of the white solid melted sharply between 121 and 122^{o}C but that a small amount remained unmelted even at temperatures above 200^{o}C. Explain this behaviour.
3. What does the term "oiling out" mean? How can one prevent oiling out?
4. What are the purposes of the following in recrystallization of solids?
   I) boiling stones II) activated carbon III) seed crystals –
5. Give one reason why we cannot reuse boiling chips?
6. 0.12 g of compound "Y" dissolves in 10 ml of acetone at 25 ^{o}C and 0.85 g of the same compound dissolves in 10 ml of boiling acetone. If 5.0 g of compound "Y" were to be recrystallized from 75 ml acetone, what will be the next maximum amount of "Y" that will be recrystallized?

Unit 3

Extraction
Determination of Distribution Coefficient

Purpose:

a) To purify samples of organic compounds that are solids at room temperature
b) To dissociate the impure sample in the minimum amount of an appropriate hot solvent

Equipment / Materials:

Separatory funnel	burette	spatula
125 ml Erlemeyer flask	burette clamp	Benzoic acid solution
10 and 50 mL graduated cylinders	ring& ring stand	0.1 M NaOH (or 0.02 M) solution
50 and 100 mL beakers	funnel	methylene dichloride

Discussion: Crystallization, purification, and isolation (may only be restricted to a solid) are insufficient ways to separate mixtures of compounds. Extraction is the recovery of a substance from a mixture by bringing it into contact with a solvent, which dissolves the desired material. Partitioning is the separation between two distinct phases (immiscible liquids) and also called fractional separation.

Like recrystallization and distillation, **extraction** is a separation technique frequently employed in the laboratory to isolate one or more components from a mixture. Unlike recrystallization and distillation, it does not yield a pure product; thus, the former techniques may be required to purify a product isolated by extraction. In the technical sense extraction is based on the principle of the **equilibrium distribution** of a substance (solute) between two immiscible phases, one of which is usually a solvent. The solvent need not be a pure liquid but may be a mixture of several solvents or a solution of some chemical reagent that will react with one or more components of the mixture being extracted to form a new substance soluble in the solution. The material being extracted may be a liquid, a solid, or a mixture of these. Extraction is a very general, highly versatile technique that is of great value not only in the laboratory but also in everyday life.

Extraction is a convenient method for separating an organic substance from a mixture, such as an aqueous reaction mixture or a steam distillate. The extraction solvent is usually a volatile organic liquid that can be removed by evaporation after the desired component has been extracted.

The extraction technique is based on the fact that if a substance is insoluble to some extent in two immiscible liquids, it can be transferred from one liquid to the other by shaking it together with the two liquids. For example, acetanilide is partly soluble in both water and ethyl ether. If a solution of acetanilide in water is shaken with a portion of ethyl ether (which is immiscible with water), some of the acetanilide will be transferred to the ether layer. The ether layer, being less dense than water, separates out above the water layer and can be removed and replaced with another portion of ether. When this in turn is shaken with the aqueous solution, more acetanilide passes into the new ether layer. This new layer can be removed and combined with the first. By repeating this process enough times, virtually all of the acetanilide can be transferred from the water to the ether.
As we stated above, the substance being extracted may be a solid. Extractions of this type will not be conducted here, but they are probably already part of your own experience. The brewing of tea from tea leaves (or the tea bag that combines extraction and filtration) and of coffee from the ground bean are excellent examples of the extraction of a solid mixture with a hot solvent (water).

In the laboratory one of the more important applications of the extraction process has been its use to remove an organic compound from a solution when distillation is not feasible. Extraction is accomplished by shaking the solution in a **separatory funnel** with a second solvent that is immiscible with the one in which the compound is dissolved, but dissolves the compound more readily. Two liquid layers are formed, and the layer that has most of the desired product in it can be separated from the other. Sometimes not the entire product is extracted in a single operation and the process must be repeated once or twice more to assure a clean separation. It has been found that when two immiscible solvents are shaken together, the solute distributes itself between them in a ratio roughly proportional to its solubility in each. The ratio of the concentration of the solute in each solvent at equilibrium is a constant called the **distribution ratio (d) or partition coefficient (K_d).**

The larger the value of K_d, the more solute will be transferred to the ether with each extraction, and the fewer portions of ether will be required for essentially complete removal of the solute.

$$K_d = \frac{[\text{solute}]_o}{[\text{solute}]_{aq}} = \frac{C_o}{C_{aq}} = \frac{m_o/V_o}{m_{aq}/V_{aq}}$$

Where o and aq refer to the organic (ether) and aqueous layers, respectively; and m_o and m_{aq} are the masses in grams of material dissolved in each respective layer.
C_o = concentration of organic solution and C_{aq} = concentration of aqueous solution

Example (1)
At 20 ^oC, only 0.24 g of an organic acid "A" dissolves in 100 ml of water, but 2.70 g of the same acid dissolves in 100 ml of ether.

a) calculate the value of partition coefficient.

Answer

$$K_d = \frac{[\text{solute}]_o}{[\text{solute}]_{aq}} = \frac{C_o}{C_{aq}} = \frac{m_o/V_o}{m_{aq}/V_{aq}}$$

$$K_d = \frac{m_o/V_o}{m_{aq}/V_{aq}} = \frac{2.70\ g/100\ ml}{0.24\ g/100\ ml} = 11.25$$

b) Calculate the percentage of extraction if 0.12 g of acid extracted in 100 ml of aqueous solution.

Answer

$K_d = \frac{X_o/100}{0.12/100}$. i.e., $11.25 = \frac{X_o}{0.12}$

X_o = 1.35 g (acid extracted in organic phase)

$X_{total} = X_o + X_{aq}$ = 1.35 g + 0.12 g = 1.47 g

$$\%\ \text{Extraction} = \frac{X_o}{X_{total}} \times 100\% = \frac{1.35}{1.47} \times 100\% = 92\ \%$$

c) Calculate the volume of ether required to extract 85% of a 3.00 g sample of acid "A" in a 100 ml aqueous solution.

X_o = (85%) x 3.00 g = 2.55 g

X_{aq} = 3.00 g – 2.55g = 0.45 g remained in aqueous solution

Answer

$$11.25 = \frac{2.55/V_o}{0.45/100}$$

V_o = 51 ml

d) Calculate the total amount of acid extracted by a double extraction of 50 ml ether in each extraction in part (c).

Answer

First extraction (first 50 ml ether)

$$11.25 = \frac{X_{o1}/50}{X_{w1}/100}$$

$$11.25 = \frac{X_{o1}/50}{(3.00 - X_{o1})/100}$$

Solving for X_{o1},

X_{o1} **= 2.55 g (**acid extracted in first extraction), i.e., $\frac{2.55}{3.00}$ x 100% = □□.□**%**

X_{w1} = 3.00 g – 2.55 g = **0.45g** (acid remained in aqueous solution after first extraction)

Second extraction (second 50 ml ether)

$$11.25 = \frac{X_{o2}/50}{X_{w2}/100}$$

$$11.25 = \frac{X_{o2}/50}{(0.45 - X_{o2})/100}$$

Solving for X_{o2},

X_{o2} **= 0.38 g (**acid extracted in second extraction)

$X_{o\,(total)} = X_{o1} + X_{o2}$ = 2.57 g + 0.38 g = 2.95 g (acid extracted during the 1st and the 2nd extractions)

i.e., $\frac{2.93}{3.00}$ x 100% = □□.□**%**

X_{w2} = 3.00 g – 2.93 g = **0.07 g** (acid remained in aqueous solution after second extraction)

Or use the following formula:

$$\% \text{ Extraction} = \left[1 - \left(\frac{V_{aq}}{V_{aq} + K_d.V_o}\right)^n\right] \text{x } 100\%.$$ Where **n** is the number of extractions.

Using this equation for the second extraction above, we have:

$$\% \text{ Extraction} = \left[1 - \left(\frac{100}{100 + (11.25 \text{ x } 50)}\right)^2\right] \text{x } 100\% = \textbf{97.7 \%}$$

Example (2)

Isobutyric acid has a solubility in water (at 25 ^oC) about one-third of its solubility in diethyl ether; thus K_d = 3.0. Imagine 4.0 g of isobutyric acid dissolved in a mixture of 35.0 ml of diethyl ether and 100.0 ml of water.

a) What mass of acid is in each layer at equilibrium after first extraction and second extraction?

b) Calculate the percentage extraction after second extraction.

Answer

Solubility of butyric acid in water = X g/ml Solubility of butyric acid in diethyl ether = 3X g/ml

(a) First Extraction

$$K_d = \frac{X_{o1}/35}{X_{w1}/100}. \quad \text{i.e.,} \quad 3 = \frac{X_{o1}/35}{(4 - X_{o1})/100}$$

Solving, X_{o1} = **2.05 g** (extracted by diethyl ether after first extraction)

Therefore, X_{w1} = (4 – 2.05) g = **1.95 g** (remained in water after first extraction).

(b) Second Extraction

$$3 = \frac{X_{o2}/35}{X_{w2}/100}$$

$$3 = \frac{X_{o2}/35}{(1.95 - X_{o2})/100}$$

Solving for X_{o2},

X_{o2} = **0.999 g** (Isobutyric acid extracted in second extraction)

$X_{o\ (total)} = X_{o1} + X_{o2}$ = 2.05 g + 0.999 g = 3.049 g (acid extracted during the 1st and the 2nd extractions)

i.e., $\frac{3.049}{4.00}$ x 100% = □□.□**%**

Extraction solvents

If a solvent is to be used to extract an organic compound from aqueous mixture or solution, it must be virtually insoluble in water, and it should have a low boiling point so that the solvent can be more soluble in the extraction solvent than in water, since otherwise too many extraction steps will be required to remove all of the solute.

Ethyl ether is the most common extraction solvent. It has a very low boiling point (34.5 ^{o}C) and can dissolve a large number of organic compounds, both polar and non-polar. However, ethyl ether must be used with great care, since it is extremely flammable and tends to form explosive peroxides on standing.

Methylene Chloride (dichloromethane) has most of the advantage of ethyl ether; in addition, it is non-flammable and denser than water. However, it has a tendency to form emulsions, which can make it difficult to separate the layers cleanly. Other useful solvents and their properties are listed in the following table. Various grades of petroleum ether (a mixture of low boiling hydrocarbons) can be used in place of pentane.

From the foregoing discussions some of the desirable properties of an organic extraction solvent become apparent:

i. It must readily dissolve the substance being extracted but must not dissolve to any appreciable extent in the solvent from which desired substance is being extracted.
ii. It should extract neither the impurities nor other substances present in the original mixture.
iii. It should not react with the substance being extracted.
iv. It should be readily separated from the desired solute after extraction.

Few solvents will meet all of these criteria, and in some cases a completely satisfactory solvent cannot be found. Therefore, the scientist must select a solvent system that most nearly approaches the ideal.

Some of the solvents commonly used for extracting aqueous solutions or mixtures include diethyl ether, methylene chloride, chloroform, carbon tetrachloride, benzene, n-pentane, n-hexane, and various mixtures of saturated hydrocarbons from petroleum (petroleum ether, ligroin, etc.). Each of these has a relatively low boiling point so that it may be fairly easily separated from the solute by evaporation or distillation. Methanol and

ethanol are not good solvents for extracting aqueous solutions or mixtures because of their solubility in water; however, if an aqueous solution can be saturated with potassium carbonate without affecting the solute, ethanol can be used to extract polar solutes from the solution.

Solvent	**b.p. (^{o}C)**	**D (g/ml)**	**Comments**
Ethyl acetate	77	0.90	Absorbs much of water good general solvent
Ethyl ether	34.5	0.90	absorb some water, easy to remove; very flammable; vapour should not be inhaled
Methylene dichloride	40	1.34	Good general solvent; easy to dry and remove; suspected carcinogen
Chloroform	62	1.48	Can form emulsion easy to dry and remove; health hazard; suspected carcinogen
1,1,2-trichlorotrifluroethane (Freon TF)	48	1.58	Maybe substituted for carbon tetrachloride
Pentane	36	0.63	Easy to dry & remove; very flammable
Hexane	69	0.66	Easily dried

Criteria for selecting an extracting solvent

i. It should be insoluble or slightly soluble with the solvent of the solution being extracted.

ii. It should have a favourable distribution coefficient for the substance being extracted and an unfavourable distribution.

iii. It should be able to be easily removed from the extracted substance after the extraction. Since the removal is often by distillation, the solvent should therefore have a reasonably low boiling point.

iv. It should be chemically inert to the extracted substance, other components in the mixture, and the solvent to the solution being extracted.

v. It should be reasonably safe to work with and relatively inexpensive.

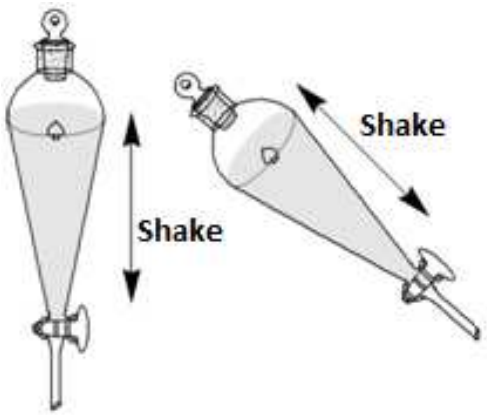
Use of the Separatory Funnel

The procedure in this experiment involves the use of the separatory funnel. It is important that you learn how to use this piece of equipment properly, for an efficient separation and for safety. It is made of thin glass and is easily broken unless handled carefully. Unfortunately, in various student manuals you will find descriptions of ways of holding the separatory funnel. Probably for you there is some best method, depending on the size of your hands, the strength of your fingers, your manual dexterity, and the size and shape of the funnel. The following are important rules to observe.

1. Hold the funnel firmly but gently in both hands so that it can be turned from the vertical to horizontal direction and back again easily and can be shaken vigorously while observing (2) and (3).
2. Keep the stopper tightly seated with one hand at all times, using the forefinger of that hand, the base of the forefinger, or the palm of the hand.
3. Keep the stopcock tightly seated with the fingers of the other hand in such a way that the fingers can open and close the stopcock quickly to release the pressure that may be built up from solvent vapour or evolved gases.

The use of the separatory funnel is a skill and is best learned by practice with an empty funnel while watching your instructor demonstrate the technique. Two slightly different methods of handling the separatory funnel are shown in the figure below. In the first method the stem of the funnel projects between the thumb and first finger of the left hand (for a right-handed person). The stopcock is held in place and operated with the thumb and first finger. The stopper is kept in place by pressure against the base of the first finger of the right hand.

In the second method the stem of the funnel projects between the first and second fingers of the left hand. The stopcock is held in place by the pressure from these fingers and is operated by them in conjunction with the thumb. The stopper is held in place by pressure against the middle of the palm of the right hand. (Fig 1)



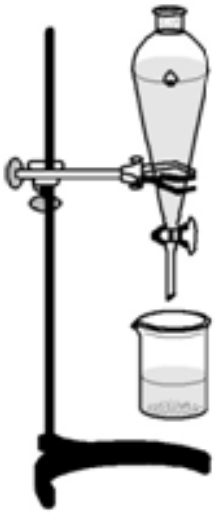


Fig. 1: methods for holding and shaking the separatory funnel. **Fig. 2: support and use of the separatory funnel.**

Support the separatory funnel in a ring on ring stand. Close the stopcock and add the liquids to the funnel to be separated. Insert the stopper, and immediately invert the funnel. **Point the barrel away from your face and that of your neighbours.** ***Open the stopcock to release the pressure***, which may have accumulated inside the funnel (volatile solvents such as ether develop considerable pressure).

Close the stopcock and, holding the funnel horizontally; shake the funnel two or three times. Invert the funnel and release the pressure as before. Repeat this process until opening the stopcock causes no further pressure release. Close the stopcock and shake the funnel 15-20 times. Replace the funnel in the holder (ring on ring stand) and ***remove the stopper*** (Fig. 2). Allow the liquids to stand until the layers have completely separated. Draw the lower layer into a flask or beaker of proper size (Fig. 2).

Do not draw the liquid through the stopcock too rapidly. Slow the flow carefully as the boundary between the two layers approaches the stopcock. Stop the flow of liquid completely just as the upper layer enters the hole in the stopcock. Pour the upper layer *through the neck* of the funnel into a second flask. *Never discard either layer until you are absolutely certain which the proper layer to keep is*. Usually, one layer will be an aqueous layer or solution, and the other will be an organic liquid. The one of greater density will be at the bottom.

To check the identity of a layer, should you be in doubt, withdraw a few millilitres of the lower layer into a test tube containing an equal volume of water. If the lower layer in the separatory funnel is water or an aqueous solution it will be homogeneous (only one layer). If the layer being tested is the organic layer, the sample withdrawn will fall to the bottom of your test tube and also form two liquid layers. In either event, return the test mixture to the separatory funnel.

EXPERIMENT – Extraction, Determination of Distribution Coefficient

Experimental Procedures

Part 1 – Standardization of NaOH solution

Use a 10 mL graduated cylinder measure 10.0 ml of the acid solution and transfer the solution to a 125 mL Erlenmeyer flask. Add 2-3 drops of phenolphthalein and titrate to the end point (light pink) with a standardized (≈ 0.1M or 0.02 M) sodium hydroxide solution. Record in report form the number of millilitres of base required to neutralize this volume of acid solution. Calculate the molarity of the NaOH.

Discard the neutralized acid solution and rinse your flasks. *REPEAT. (TWO TRIALS)*

Part 2 – Single extraction

Use a 50 mL graduated cylinder to measure out a second 50.0 ml volume of acid solution and transfer it to your separatory funnel. Add 10 ml of methylene dichloride, CH_2Cl_2, to the funnel and extract according to the procedure outlined in the part 1 of this experiment. Separate the *bottom* layer (organic phase) in a 100 mL beaker and collect the *top* layer (aqueous) into a 125 mL Erlenmeyer flask and add 2-3 drops of indicator. Record the volume of the sodium hydroxide solution in the burette and titrate to the phenolphthalein end point (light pink). Again record the volume of base required and calculate a – g below. Discard the neutralized acid solution and the methylene dichloride layer into the large bottle marked **"Organic Waste".**

NOTE: This lab has been modified in that methylene dichloride is now used in place of ether as the organic phase. This avoids the problem of ether fumes and explosions. However, the extraction with methylene dichloride is not as clean because methylene dichloride is more miscible in water than ether. As a result, you will find that your aqueous layer is cloudy after extraction. You can still titrate the aqueous layer to a light pink endpoint.

Part 3 - Multiextraction

Repeat the procedure from Step 2, but this time, extract 50 ml of fresh acid solution with two 5 ml portions of methylene dichloride. Separate the aqueous layer into a flask and dispose of the organic layer. Transfer the aqueous layer back into the empty, cleaned, separatory funnel and extract it with a second 5 ml portion of fresh methylene dichloride. Separate the extracted aqueous layer, add indicator as before and titrate to the end point. Record the volume of standard base required and calculate a – f below. Dispose of the organic layer extracts as directed and clean your separatory funnel.

Data and Results (Extraction of Benzoic Acid)

REPORT SHEET

Name _____

Instructor _____

Date_____

Part 1: Standardization of NaOH Solution

a. Volume of base required to neutralize 10.0 ml of the benzoic acid solution:
   Trial 1: _____ml Trial 2: _____ml Average: _____ml
b. Molarity of the NaOH solution:
   (ml of base) (M of base) = (ml of acid) (M of acid) _____ml

Part 2: Distribution Coefficient

a. Volume of base required to neutralize 50 ml of benzoic acid solution after a single extraction with 10.0 ml of CH_2Cl_2 _____ml
b. Moles of benzoic acid neutralized: (2a in 1L) (1b) _____mole
c. Grams of benzoic acid neutralized: (2b) (MW of benzoic acid) _____g
d. Grams of benzoic acid originally present in 50 ml before extracting with $CH_2 Cl_2$: 0.122 g
e. Grams of benzoic acid extracted in CH_2Cl_2 : (2d – 2c) _____g
f. Percent of benzoic acid in extracted in CH_2Cl_2 : (2e/2d) x 100 _____%
g. Distribution coefficient $K_d = C\ C_{H2\ Cl2}/C_{H2O}$: (2e/10 ml)/ (2c/50ml _____

Part 3: Multiple Extraction

a. Volume of base required to neutralize 50 ml of benzoic acid solution after two extraction with 5.0 ml of CH_2Cl_2 each: _____ml
b. Moles of benzoic acid neutralized: (3a in L) (1b) _____mole
c. Grams of benzoic acid neutralized: (3b) (MW of benzoic acid) _____g
d. Grams of benzoic acid originally present in 50 ml before extracting with $CH_2 Cl_2$ 0.122 g
e. Grams of benzoic acid extracted in CH_2Cl_2: (3d – 3c) _____g
f. Percent of benzoic acid in extracted in CH_2Cl_2: (3e/3d) x 100 _____%

Calculate the mass of benzoic acid which would be extracted in two 5.0 ml extractions with CH_2Cl_2 based on your K_d obtained in part 2

Pre-Laboratory Questions - Due before lab begins

1. The distribution coefficient, K, is 10 for compound Y. What mass of compound Y would be removed from a solution of 4.0 g of Y in 100 ml water by a single extraction with 100 ml of ether?
2. Reconsider question 1 above; what mass of compound Y would be removed by two (double) extractions using 50 ml of ether each time?
3. A student during an extraction experiment lost track of which layer is the aqueous layer. How could the student determine which layer is which by a simple test?
4. Why must the stopper be removed from the separatory funnel before the lower layer is removed?
5. An aqueous solution containing 5.0 g of solute in 100 ml is extracted with three 25 ml portion of diethyl ether. What is the total amount of solute that will be extracted by the ether, K = 1.0?

Post-Laboratory Questions - Due after completing the lab

1. What are the advantages and disadvantages of using ether as a solvent for the extraction of organic compounds?
2. What volume of an organic solvent must be used to effect 90% separation in one extraction when only 2.7 g of a certain compound dissolves in 100 ml of water? (K= 15)
3. What percentage of the organic compound could be recovered if two extractions were made, each time using half of the volume calculated 2?
4. An organic compound can be extracted from a water layer by an organic solvent more efficiently if the water layer is saturated with an inorganic salt such as sodium chloride. This effect, called “salting out”, increase the participation coefficient in favour of the organic compound. (explain)
5. The pain reliever phenacetin is soluble in cold water to an extent of 1.0 g/1310 ml and soluble in diethyl ether to an extent of 1.0 g /90ml.
   a) Determine the approximate distribution coefficient for phenacetin in those two solvents.
   b) If 150 g of phenacetin were dissolved in 100 ml of water, how much ether would be required to extract 90% of phenacetin in a single extraction?
   c) What percent of the phenacetin would be extracted the aqueous solution in part (b) by two 25ml portions of ether?

MODULE 3

Unit 1
Measurement and Density

Learning Objectives

After studying this unit, you should

- Become familiar with laboratory equipment and glassware
- Begin to see the link between measurement and chemical knowledge Begin to understand how scientists communicate with significant figures Engage in proper measurement technique and the data collection process
- Begin to make the connection between macroscopic observations and the sub-microscopic level through drawing
- Understand the concept of density and explore methods for measuring density in the lab Begin to see the power of a "class data set".
- Experience the culture of teamwork and individual responsibility to a group.

Purpose

Section 1 — By calculating the density of a known substance (water), determine the relative precision and accuracy of different glassware items.

Section 2 — Determine the density of a salt water solution using the most precise and accurate piece of glassware determined in section 1

Equipment / Materials:

Weighing balance	Water	Unknown metal cube
100 mL beaker	Unknown liquid	
100 mL graduated cylinder	Metal cylinder	

Introduction

The ability to make accurate and detailed observations is crucial in science. This lab will focus on quantitative observations, more specifically, measurements. A measurement is defined by a number and a scale or unit. The scale used is often varied. Due to convenience, the metric system is often used in many countries. The universal scale, however, used by scientists is the SI unit. In this unit, we will focus on making accurate and detailed observations in measurements using the metric system while obeying the laws of significant figures.

In addition to quantities defined by a single unit, quantities can also be defined by a combination of units. One such example is density. Density is merely one way to characterize a substance. Density (d) is defined as mass (m) per unit volume (V).

Thus, units of both mass (i.e. g) and volume (i.e. mL) are necessary in order to determine density.

D = m/V

Examples

1. A cube of copper was found to have a mass of 0.630 kg. What are the dimensions of the cube? (The density of copper is 8.94 g/cm3.)

Solution:

a) Determine the volume of the cube (note that kg have been converted to g):

8.94 g/cm3 = 630 g / volume

volume = 70.47 cm3

b) Each side of a cube is equal in length, so take cube root of the volume for length of cube side:

[cube root of] 70.47 cm3 = 4.13 cm

2. A cube of copper was found to have a mass of 0.630 kg. What are the dimensions of the cube? (The density of copper is 8.94 g/cm3.)

Solution:

a) Determine the volume of the cube (note that kg have been converted to g):

8.94 g/cm3 = 630 g / volume

volume = 70.47 cm3

b) Each side of a cube is equal in length, so take cube root of the volume for length of cube side:

[cube root of] 70.47 cm3 = 4.13 cm.

3. A graduated cylinder is filled to the 40.00 mL mark with mineral oil. The masses of the cylinder before and after the addition of mineral oil are 124.966 g and 159.446 g. In a separate experiment, a metal ball bearing of mass 18.713 g is placed in the cylinder and the cylinder is again filled to the 40.00 mL mark with the mineral oil. The combined mass of the ball bearing and mineral oil is 50.952 g. Calculate the density of the ball bearing.

Solution:

a) Determine the density of the mineral oil:

159.446 g minus 124.966 g = 34.480 g

34.480 g / 40.00 mL = 0.8620 g/mL

b) Determine the volume of the ball bearing:

50.952 g minus 18.713 = 32.239 g (this is the mass of mineral oil)

32.239 g divided by 0.8620 g/mL = 37.40 mL (this is the volume of mineral oil)

40.00 mL - 37.40 mL = 2.60 mL

c) The density of the ball bearing is 7.197 g/mL. This came from 18.713 g divided by 2.60 mL.

EXPERIMENT – Measurement and Density

Experimental Procedures

Part A, Density of liquids:

i) Density of water:

1. Collect about 100 mL of water in a beaker; let it sit until its temperature is stabilized. Record its temperature.
2. Weigh a clean, dry 10 mL graduated cylinder and record its mass.
3. Now add water from the beaker to the cylinder, so that the level is above 5mL, but below 10 mL and record the volume accurately to the correct significant figures.
4. Wipe off any water droplets adhering to the outside as well as above of the water level inside.
5. Record the mass of the cylinder with water.
6. Now repeat step 2-4 two more times, each time with a different volume, but still between 5-10 mL.
7. Now you have 3 sets of data, calculate the average density of water at this temperature.
8. Calculate the % error, once you know the true value of density of water from literature.

$$\% \text{ error} = \frac{\text{experimental value} - \text{true value}}{\text{true value}} \text{ x } 100\%$$

ii) Density of an unknown liquid:

Start with another dry graduated cylinder and repeat the mass and volume measurements (above steps 1-7) with an unknown liquid (A, B or C assigned by the instructor). Calculate the density of the liquid, identify it (after discussing with the instructor), and calculate the % error.

Part B, Density of solids:

i) Density of an unknown metal cylinder:

1. Weigh the metal cylinder and record the mass. Also record the cylinder number.

2. Now measure its height and the diameter of the circular base to the correct significant figures, using a ruler. Calculate the volume of the cylinder.
3. Alternately, you can also determine the volume of the metal cylinder using volume displacement method as described below:
4. Add water from the beaker to a 50 mL graduated cylinder so that the lever is between 25 and 30 mL and record the accurate volume (significant figures please).
5. Hold the graduated cylinder in an angle and slide the metal cylinder gently through the side, without splashing any water or breaking the glass cylinder.

You will see the level of the water inside the graduated cylinder has risen. Record the new volume now (significant figures again). The difference in the volume levels gives you the volume of the metal cylinder

Now calculate the density of the metal from volume from 2 different methods.

ii) Density of an unknown metal cube:

1. Weigh the metal cube and record the mass. Also record the cube number.
2. Now measure its height and the width and depth to the correct significant figures using a ruler. Calculate the volume of the cube and calculate its density.

DATA and CALCULATIONS:

Part A : Density of liquids:

(i) Density of water:

Temperature of water : _____

Mass of empty graduated cylinder: _____

	Trial 1	Trial 2	Trial 3
Volume of water (mL)			
Mass of water + graduated cylinder (g)			
Mass of water alone (g)			
Density of water (g/mL)			
Density from literature			
% error			

(ii) Density of unknown liquid : _____ (A/B/C)

Temperature of liquid : _____

Mass of empty graduated cylinder: _____

	Trial 1	Trial 2	Trial 3
Volume of liquid (mL)			
Mass of liquid + graduated cylinder (g)			
Mass of liquid alone (g)			
Density of liquid (g/mL)			
Density from literature			
% error			

Part B, Density of solids:

Density of an unknown metal cylinder # : ___

Density from diameter and height measurement:

Mass of metal cylinder (g)	Diameter (cm)	Height (cm)	Volume = _____(cm3)	Density of metal (g/cm3)	Density from literature (g/cm3)	% error

Density from volume displacement:

Volume of water before dropping metal cylinder (mL)	Volume of water after immersing the metal cylinder (mL)	Volume of metal cylinder (mL)	Density of metal (g/cm3)	% error

ii) Density of an unknown metal cube # : ___

Mass of metal cube (g)	Depth (cm)	Height (cm)	Width (cm)	Volume = _____(cm3)	Density of metal (g/cm3)	Density from literature (g/cm3)	% error

Pre-Laboratory Questions - Due before lab begins

1. You are given a bottle that contains 4.59 cm3 of a metallic solid. The total mass of the bottle is 35.66 g. The empty bottle weighs 14.23 g. What is the density of the solid?
2. Mercury is traded by the "flask", a unit that has a mass of 34.5 kg. What is the volume of a flask of mercury if the density of mercury is 13.6 g/ml?

Post-Laboratory Questions - Due after completing the lab

1. A metal cylinder with 26.0 mm diameter and 75.0 mm height has a density of 8.60 g/mL . Calculate its mass.
2. A metal sphere weighing 18.48 g is added to 20.00 mL of water in a graduated cylinder. If the density of the metal is 4.50 g/mL, what will be new level of water in the graduated cylinder?
3. In this experiment, could you have used volume displacement method with water for finding the volume of the wood cube, assuming the cube can fit into the cylinder? Explain why or why not.
4. How will you find the volume of an irregularly shaped object that would dissolve in water?

Unit 2

Micro Method Determination of Boiling Point of Hydrocarbons

Purpose:

a) To become acquainted with procedure in evaluating physical properties such as boiling point and the use of boiling point in identifying liquid.
b) To determine the boiling points of various organic compounds and to use these to identify unknowns.

Equipment / Materials:

Hot plate	Closed end capillary tube	Small test tube	Liquid organic compounds
Thermometer	400 mL beaker	Beaker tongs	

Discussion

The boiling point of a compound is the temperature at which it changes from a liquid to a gas. This is a physical property often used to identify substances or to check the purity of the compound.

It is difficult, though, to determine **boiling *point***. Usually, chemists can only obtain a boiling range of a 2-3 ^{o}C accuracy. This is usually sufficient for most uses of the boiling point.

The boiling point of a liquid is an important physical property. A liquid's boiling point is the temperature at which its vapour pressure is equal to the atmospheric pressure. Normally, the boiling point is measured at one atmosphere (101 kPa or 760 mmHg or 760 torr). Like melting points, boiling points are characteristic properties of pure materials. Boiling points are approximately related to molecular weights; the higher the molecular weight, the higher the boiling point.

Boiling point is the temperature at which the vapour pressure of the liquid exactly equals the pressure exerted on it, causing the liquid to "boil" or change to the gas phase. For purposes of this laboratory experiment, the boiling point of an organic liquid is the ***temperature range*** over which the state of the organic compound changes from the liquid phase to the gas phase at 760 mm of pressure. While the boiling point is a characteristic physical property of a compound, many compounds may have the same boiling point.

The molecules of compounds that exist in the liquid state are relatively close together, compared to those of gaseous compounds. The close proximity of molecules in the liquid state allows these molecules to interact via ***non-covalent interactions*** (dipole-dipole, H-bonding, van der Waals forces). In general, these interactions are favourable and help to hold the molecules together in a defined volume, but still allow free motion or "flow". Conversely, molecules of a gaseous compound are much farther away from each other and are not confined to a specific volume by non-covalent interactions (Fig. 1). If enough energy (often in the form of heat) is provided to the liquid, the molecules begin to move away from each other by "breaking" the non-covalent forces that hold the compound in the liquid state.

Thus, the boiling point is the temperature range over which enough energy is provided to a liquid compound so that its molecules can separate sufficiently to transform to a gaseous state by breaking *non-covalent interactions*. ***No covalent bonds are broken during a change from the liquid phase to the gas phase.***

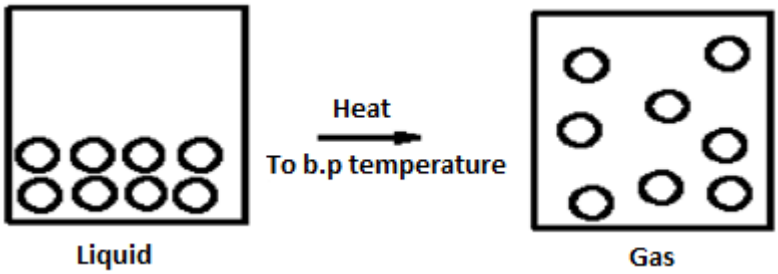


Fig.1: Phase Change from Liquid to Gas at Boiling Point Temperature Range

The vapour pressure, P_{vapor} , exerted by a liquid is directly proportional to the temperature. Thus if the atmospheric pressure, P_{atm} , is lowered, the temperature to which the liquid must be heated in order for P_{vapor} to P_{atm} is lowered. The boiling point will change by approximately 0.5 ^{o}C for each 10 mm (10 mmHg) change in pressure.

Thus the change in b.p. = $\Delta T_b = \frac{(P_{vapour} - P_{atm})\ mm}{10\ mm}$ x 0.5 □

The corrected b.p. = $\mathbf{T_b}$ = **normal b.p.** + $\mathbf{\Delta T_b}$

Example

What will be the boiling point of ethanol at 700 mmHg when its normal point at 760 mmHg is known to be 78.3 ^{o}C?

Answer

$\Delta T_b = \frac{(700 - 760)}{10}$ x 0.5 □ = -3 ^{o}C.

T_b = normal b.p. + ΔT_b = 78.3 ^{o}C + (-3 ^{o}C) = 75.3 ^{o}C.

In theory when a liquid is at its boiling point, one should observe bubbles of vapour forming as the liquid changes to the vapour phase. However in practice, this is usually not the case. Typically the liquid becomes superheated as its temperature climbs above the true boiling point commences. Then the solution suddenly "bumps" or boils with tremendous vigour, bumping the hot liquid out of the container. Steps must be taken to guard against this process.

In order to promote smooth boiling, the solution can be stirred, or boiling stones (boiling chips) can be added to the liquid. These glassy type devices work by providing a sharp surface upon which bubbles naturally form, which promotes smooth generation of bubbles (prevent bumping and formation of large bubbles).
If the volume of the liquid is small, it is more advantageous to use the micro method, as in this experiment. However, if the volume of the liquid is large, its boiling point can be determined by distillation.

Factors Influencing Boiling Point

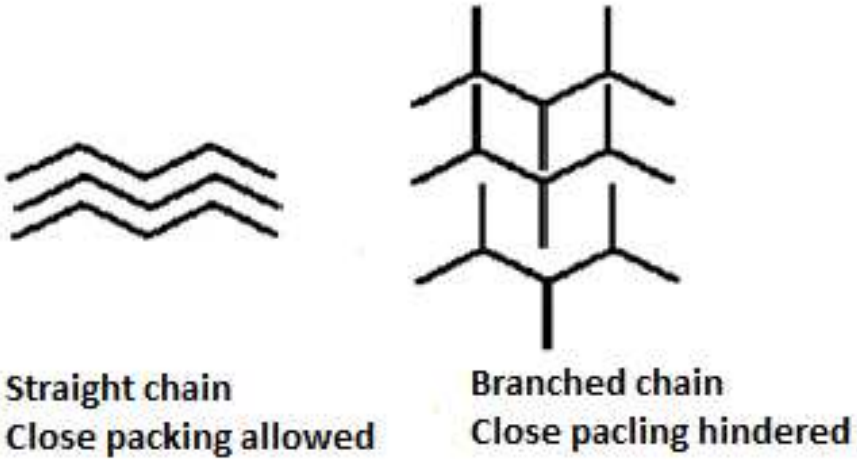
Structural features of a compound influence the boiling point by increasing or decreasing the molecules' ability to establish and maintain non-covalent interactions that hold the molecules close together in the liquid state. The structural features of a compound that influence boiling point are:

a) ***Polarity*** - Increased H-bonds, polar covalent bonds or formal charges in a molecule tend to increase the boiling point. More polar elements in a molecule increase the total number of dipole-dipole, ion-dipole

and/or H-bonding interactions. More energy (higher boiling point temperature) is necessary to break these interactions and allow the molecules to move away from each other into a gaseous state.

b) ***Molecular Weight:*** Increased molecular weight increases boiling point. A higher molecular weight compound has more atoms that can be involved in non-covalent interactions. The greater the number of non-covalent interactions, the more energy (higher boiling point temperature) that is necessary to break the non-covalent interactions to transform the compound from the liquid phase to the gas phase.

f. ***Branching:*** Branching decreases boiling point. Branching blocks molecules from packing together too closely. The closer the molecules are, the stronger the non-covalent interactions. Thus, molecules that are forced to be farther away from each other due to branching have weaker non-covalent interactions. Less energy (lower temperatures) is needed to induce a phase change from the liquid phase to the gas for branched compounds relative to straight chain compounds. (Fig. below)



Experiment - Micro Method Determination of Boiling Point of Hydrocarbons

Experimental Procedure

Place about 5 ml of the liquid is placed in a small test tube. A capillary tube, sealed at one end, is placed open-end down into the liquid (Fig. 1). The test tube is firmly attached to a thermometer by means of a rubber band, and this entire assembly is immersed in a water bath (oil bath for samples with boiling point higher than 100 ^{o}C) (Fig. 3). As the temperature is slowly increased, a rapid evolution of bubbles from the end of the tube begins. Continue heating for about 5-10 seconds to be sure that all of the air has been expelled from the capillary, and the vapours of the liquid remains in the capillary. Remove the heat, but do not take the assembly out of water bath (or oil bath), and carefully watch the capillary. Bubbles continue to be seen until the pressure exerted by the vapour of the liquid becomes equal to the atmospheric pressure. As the temperature decreases, the bubbles will slow down and at some point, the liquid will rise into the capillary. The boiling point of the sample is reached when the bubbles stops. Read the thermometer and record the temperature. The temperature observed when this happens should be the observed boiling point of the liquid. Compare your experimental result to the literature value (Table below) of the boiling point for the liquid used. If your technique is good, your experimental value should not differ from the known value (literature value) by more than 2-3 ^{o}C. Repeat the procedure with the known liquids. Each time you perform the procedure, you must use a new capillary. It will also be necessary to

allow the hot bath to cool at least 15 - 20 ^{o}C below the suspected boiling point before repeating your experiment.

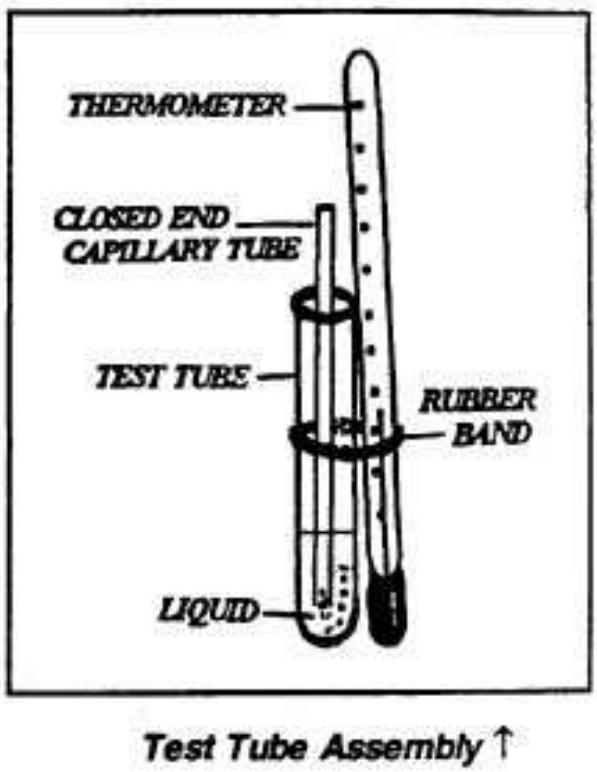


Fig. 2: Small test tube and capillary, sealed at one end

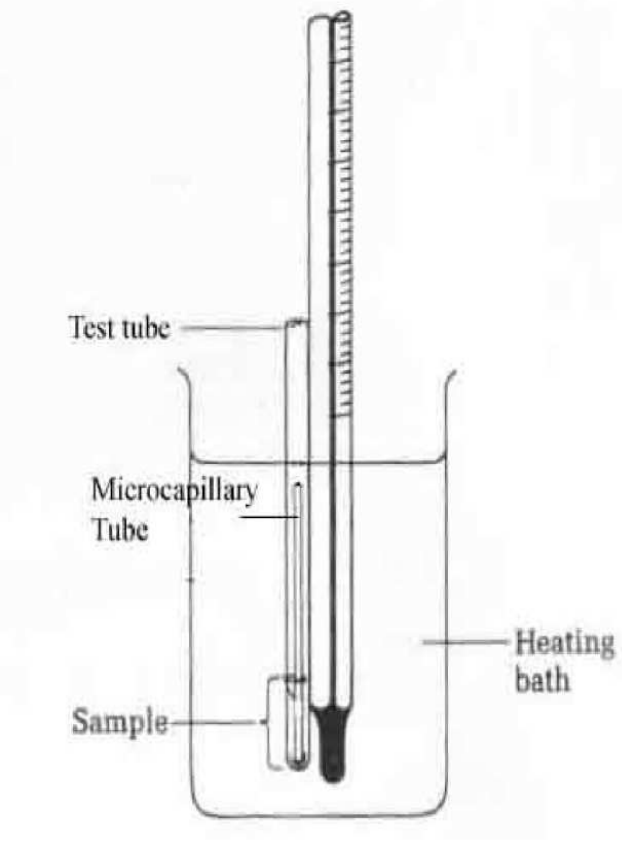


Fig. 3: Small Scale Boiling Point Apparatus

Table 1

Substance	**Boiling Point (^{o}C)**	**Substance**	**Boiling Point (^{o}C)**
Pentane	36.1	Methanol	65
Hexane	69	Ethanol	78-79
Heptane	98.4	Propanol	97-98
Octane	125.7	2-Propanol (isopropanol)	82-83
2-Methylheptane	117.7	Water	100
3-Methylheptane	119	t-Butyl alcohol	83
2,2-Dimethylhexane	106.8	Cyclohexane	80.7
3-Ethylpentane	93.5	Methylene chloride	39.8
Acetone	56- 57	Bromoform	146-150

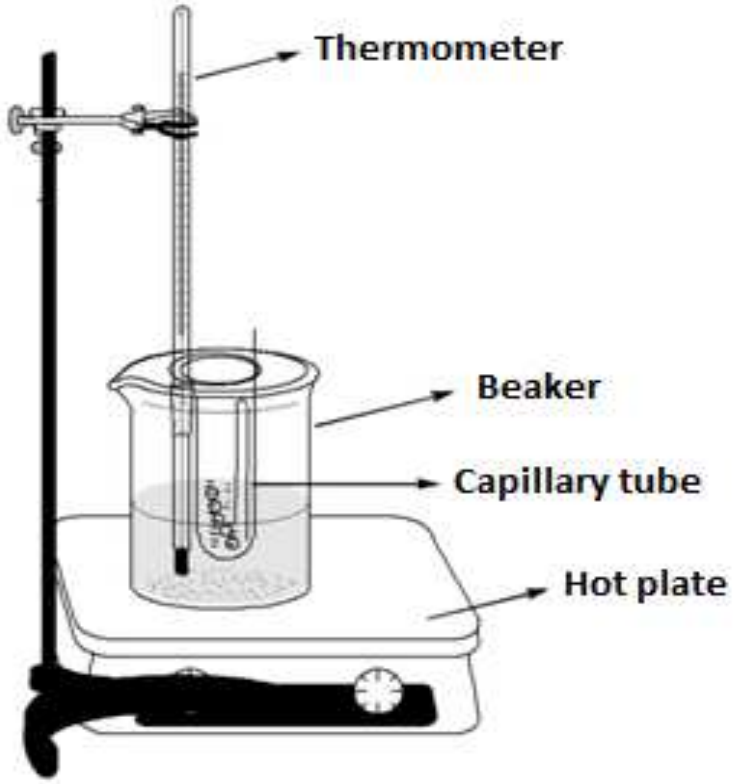


Fig. 4 - Boiling Point Apparatus Set Up

EXPERIMENT - Boiling Point Determination
RESULT SHEET
Data and Results (Boiling Point)

Name _____

Instructor _____

Date _____

Part I. Boiling Points of known compounds

(Practice compounds)

	Name of compound	B.P., ^{o}C (range) (Experimental)	B.P., ^{o}C (Literature)
e.	_____	(____-____)	_____
f.	_____	(____-____)	_____
g.	_____	(____-____)	_____

Part II. Boiling Point of an unknown Sample

(Unknown)

Unknown Sample Number = _____

Boiling Point of unknown sample

Trial 1: _____-_____ ^{o}C (range)

Trial 2: _____-_____ ^{o}C (range)

Trial 3: _____-_____ ^{o}C (range)

My unknown compound is _____
(name of compound)

Pre-laboratory Questions - Due before lab begins

1. Which of the compounds, ethanol, CH_3CH_2OH, or methanol, CH_3OH, should have the higher boiling point? Why?
2. How would the boiling point change if the atmospheric pressure increased or decreased?
3. What is the effect of a small amount of impurity on the boiling point of an organic compound?
4. List five physical properties of organic compounds that are often measured by organic chemists.
5. The boiling point of acetic acid is usually stated to be 118^{o}C. Is it possible to have acetic acid boil at 90 ^{o}C? Explain

Post-laboratory Questions - Due after completing the lab

c) What is the difference between evaporation and condensation? Define each.

d) Boiling point determination can be used for several purposes. What are those purposes?

e) What effect on the boiling point is produced by:
   a soluble non-volatile impurity, such as table salt
   an insoluble foreign substance, such as sand

f) A liquid "X" has a normal boiling point of 75 ^{o}C. What will be the approximate boiling point of the liquid at 800 mmHg pressure?

g) The density of a liquid whose boiling point is 63-65 ^{o}C was determined to be 0.74 ± 0.05 g/ml. What is the liquid?

Unit 3
Melting Point Determination

Purpose

- Determine the purity of a substance using melting point as physical property
- Identify an unknown compound using its melting point
- Identify an unknown compound using mixture melting point
- Learn how to obtain an accurate melting point using a Mel-Temp apparatus

Discussion

Identifying an unknown compound can be a tedious and exacting task. In identifying a compound, a chemist often measures several physical properties (melting point, boiling point, density, etc.) and observes a few chemical properties (reactivity, acidity, basicity, etc.) of the compound. The reason for determining several chemical and physical properties of the compound is that it is quite possible for two different compounds to have a few physical and or chemical properties in common; but it is highly unlikely for the two compounds to have very many identical physical and chemical properties.

Useful physical properties that are often utilized by chemists in identifying an organic compound include colour, odour, physical state, melting point (M.P.), boiling point (B.P.), density (d), infrared (IR) spectrum, nuclear magnetic (NMR) spectrum and ultraviolet (UV) spectrum.

Physical constants are numerical values measured at the time certain physical properties are observed. As long as the physical constants are determined under standard conditions (temperature, pressure, etc.), they are invariant and, therefore, useful in helping to determine the identity of unknown substances.

Chemists regard a table of physical properties and physical constants to be extremely helpful in identifying unknown compounds. There are a number of reference books that contain tables of physical properties and physical constants of compounds. If the physical properties of an unknown compound are identical to the physical properties of a compound listed in the tables, the two compounds are probably the same. Thus, a colourless, liquid compound with a melting point of 5.5 ^{o}C, a boiling point (at 760 mm) of 80.1 ^{o}C, is likely to be benzene, although we might want to make a few more observations to be sure.

It should be pointed out, however, that it is not possible to accurately predict the physical properties of newly synthesized or isolated compounds. Therefore, tables of physical properties are only useful in identifying previously known compounds. However, useful information as to the compound's identity and its purity can often be obtained from its melting point.

The **melting point of a solid** is defined as the **temperature** at which the liquid and solid phases are in equilibrium.

The **freezing point of a liquid** is the same **temperature** as the melting point of its solid. However, freezing points are rarely measured in practice because they are more difficult to determine. One reason for this is that solidification may not occur at the correct temperature due to the phenomenon of **supercooling.** Supercooling occurs when a liquid is cooled below its freezing point and does not solidify.

Determination of the temperature at which the solid and liquid phases of a substance are in equilibrium is tedious and time consuming; it is also quite difficult with a small amount of sample. Thus, in practice, most

melting points are determined as capillary melting points, which can be done quickly with a small amount of sample in a capillary tube. A **capillary melting point** is defined as the temperature range over which a small amount of solid in a thin walled capillary tube first visibly softens (first drop of liquid) and then completely liquefies.

A solid is said to melt **sharply** if the melting point range is 0.5 - 1.0 ^{o}C (narrow melting point range). A pure solid will generally melt sharply because the forces of attraction between its particles are the same. However, the presence of a foreign particle in a crystal lattice interrupts its uniform structure and the forces of attraction are weakened.

An impure solid melts at a lower temperature and over a wider range. Thus, a solid's melting point is useful not only as an aid in identification but also as an indication of purity.

Suppose two compounds X and Y have identical melting points of 131- 132 ^{o}C and appear to be identical. We can easily determine whether or not X and Y really are the same compound by mixing a small amount of Y with X (or vice versa) and taking the melting point of the mixture. (The melting point of a mixture is called the **mixture melting point**). **If X and Y are the same compound,** the mixture melting point will be the same as the melting point of pure X or pure Y. **If X and Y are not the same compound**, one will act as an impurity in the other and the mixture melting point will be lower and more spread out (wide range 120-125 ^{o}C in this case) than the individual melting points of pure X or pure Y.

It should be noted, however, that there is one unique mixture of two compounds, X and Y, which has a lower melting point than any other mixture of the two compounds. This particular mixture is called the **eutectic mixture**. The melting point of the eutectic mixture is called the **eutectic point.** A mixture whose composition corresponds exactly to its eutectic mixture will have a relatively sharp melting point. Thus, there is a possibility that a eutectic mixture could be mistaken for a pure compound. However, if a small amount of either X or Y (assuming they are both known) is added to the mixture, the melting point of the resulting mixture will be **higher** and more spread out than the melting point of the eutectic mixture.

Some solids pass directly from the solid state to the gaseous state without first liquefying; this phenomenon is called **sublimation.** The temperature at which sublimation occurs is called the **sublimation point**. Other solids decompose rather than melt. The temperature at which a solid decomposes is the **decomposition point**. While both sublimation points and decomposition points are useful helping to identify compounds, neither is very helpful in establishing the purity of a compound.

Some solids begin to "sweat" a few degrees below their true melting points. Other solids suddenly shrink just before melting. Such shrinkage of a solid being heated is called **sintering**.

Soluble impurities (that is, impurities that are included within the crystal matrix) tend to lower the observed melting point and broaden the melting point range. Insoluble impurities have no effect on the melting point.

MEL-TEMP OPERATING INSTRUCTIONS

CAUTION: Never assume the unit is cold! Wait for the heating block to cool if the temperature is not <20^{o} C below the melting point (m.p) of your compound. Allow the block to cool to room temperature if you have no idea what the approximate melting point of your compound is.

g. Set voltage to obtain the desired heating rate at the anticipated melting point range. ***The voltage control controls the rate of heating, not the temperature!*** The higher the setting, the faster the temperature rise. Use the heating rate charts below to select the correct voltage and estimate the amount of time to obtain a temperature within 20 ^{o}C of the melting point.
NOTE: It is always a good idea to determine the approximate value for the m.p in a literature source prior to lab. If the approximate value is impossible to obtain, save time by first obtaining a quick melting range, then repeat slowly with another sample.

h. Obtain a sample or prepare a sample by packing capillary tube 3-4 mm high with thoroughly dried, finely powdered, densely packed sample. Larger, loose samples will heat unevenly! Insert the loaded m.p. tube in one of the (three) channels in the opening at the top of the unit.

i. Insert thermometer into thermometer well of instrument (bulb first).

j. Turn on power switch, making sure that apparatus is plugged in. Set the power according to the heating rate chart (below).

k. Observe samples with the eye about 6" from lens. Turn down the voltage control to get a 2 ^{o}C per minute rise when you are within 20 ^{o}C of the melting range (see example below right). Be patient!

l. Record the temperatures of the melting *range*.
T_1: Temperature at which 1st drop of liquid appears.
T_2: Temperature at which the last crystal *just* disappears.

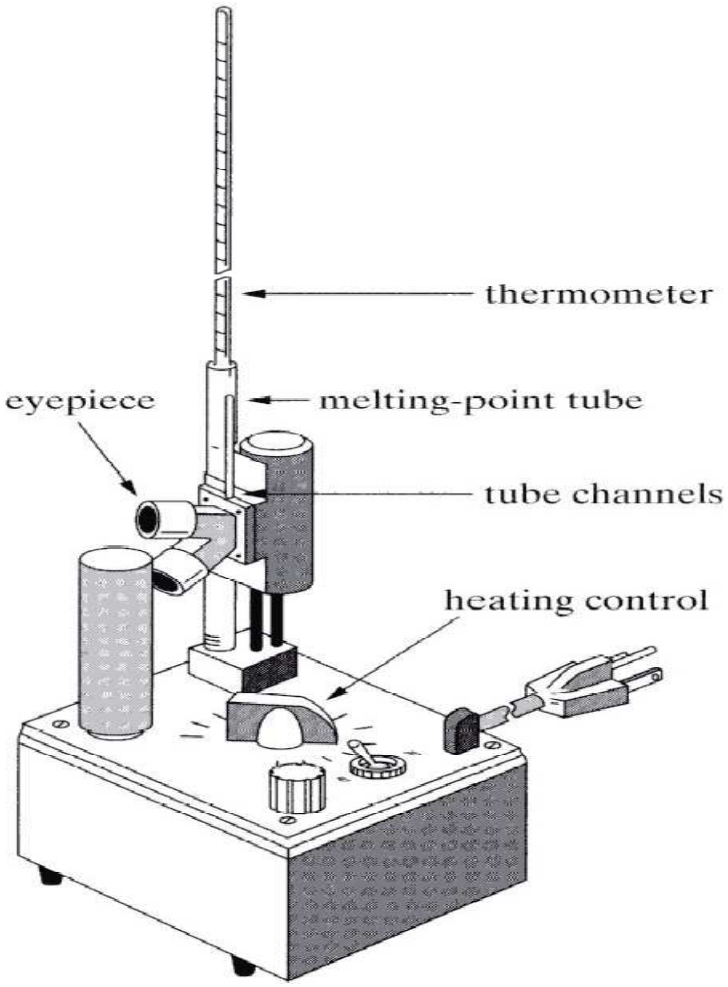
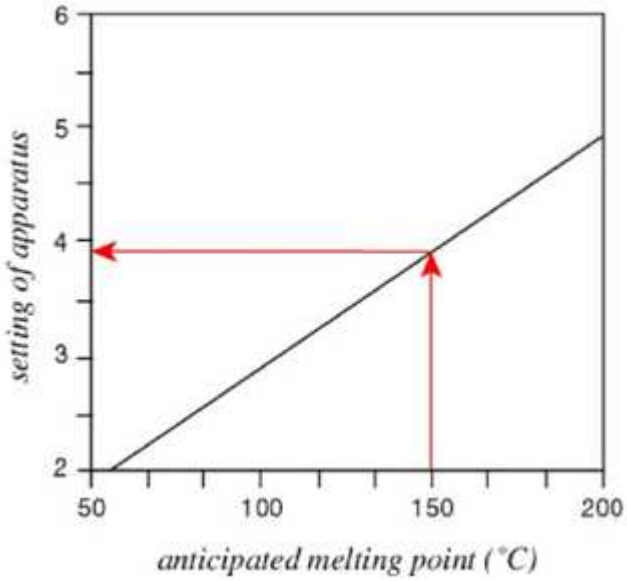


Fig. 1: Mel-Temp apparatus

Set the voltage control to zero and turn off the power switch when finished. Leave apparatus on lab bench until cool. Discard the sample in the glass disposal box.

Chart for setting the Mel-Temp melting point apparatus



Experimental Procedures

A. Preparing the Sample

Place a pea-size mound of one of the listed compounds on a piece of paper and grind it to a fine powder using a spatula. Use the spatula to push a small amount of the solid into the open end of a capillary tube. Then drop the capillary down several times to fill and pack the sample well to cause the solid to fall to the bottom. Repeat this step until you have accumulated a sample 2-4 mm (≈ 0.5 cm) high in the bottom of the tube (see Fig. 2).

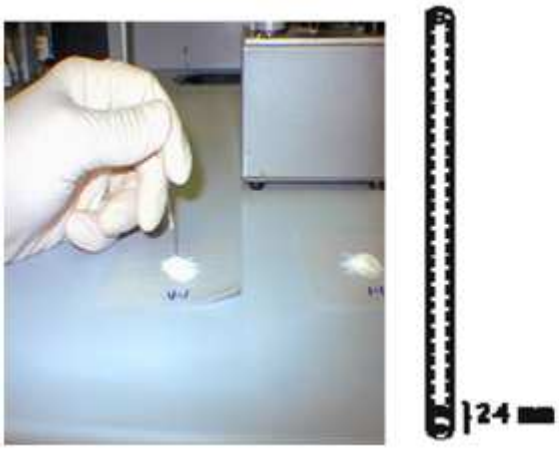


Fig. 2 – Preparing the sample

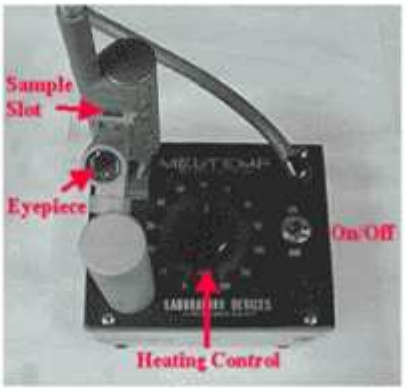


Fig. 3 - Taking a Melting Point

B. Taking a Melting Point

To record the melting point range of a compound fill two capillary tubes each to a depth of 2 -4 millimeters with the compound of interest. First, get a rough idea of the approximate melting point range (see Fig. 3). Place one of the tubes in the Mel-Tem apparatus and raise the temperature a relatively fast rate, about 10 °C/minute. Record the range of temperature from the first visible evidence of liquid (the sample appears moist, or a tiny drop of liquid is observed) to the complete liquefaction of the sample.

Note the temperature at which the compound first begins to melt. Allow the melting point apparatus to cool to about 20 °C below that temperature, and then insert the second tube. Raise the temperature more slowly this time, at the rate of about 2 °C/min.

Note the temperatures at which: I. the first crystals melt, and; II. at which the sample has completely melted. This is the melting point range. For example, a mp range of 164-168 °C average of 166 °C indicates the sample softened or began to melt at 164 °C and that transition to a liquid was complete at 168 °C.

C. Mixture Melting Point

Take approximately equal amounts of the two compounds for which you have already determined melting points and grind them together very thoroughly. Prepare a capillary containing a sample of this mixture as described in Part B and determine its melting point.

List of Melting Points for Standard Compounds

Compound	**M.P. degrees C**
o-toluic acid	103-105
Acetanilide	113-114
Fluorene	114 -115
dl-Mandelic Acid	117-118
Benzoic Acid	121-122
2-Naphthol	121-122
Urea	132-133
trans-Cinnamic Acid	132-133
Benzoin	136-137
Maleic Acid	136-137
Vanilin	81-82
Cholesterol	148-150
Biphenyl	70-71
Phenylbenzoate	69-70
Benzhydrol	68 –69
Benzophenone	48- 49

D. Determination of the Identity of an Unknown

Obtain an unknown compound from your instructor. Prepare two capillaries containing the unknown. Determine an approximate melting point for it using the first tube and a heating rate of 15-20 degrees per minute. Then let the thermometer and Mel-Temp apparatus cool to at least 20 degrees below this approximate melting point and use the second tube to obtain an accurate melting point with a heating rate of no more than 3 degrees per minute.

From the found melting point of your unknown, decide which of the listed compounds it might possibly be. There may well be more than one reasonable possibility. Prepare a mixture of your compound with the compound that is the most likely of the possible choices and take a melting point of t his mixture. If its melting point is the same as that of the unknown, it is likely that your unknown and the compound with which you mixed it are the same. If the melting point of the mixture is lower than that of the unknown, you should prepare a mixture of your unknown with the next most likely choice. You must continue to experiment until you have found a substance that does not lower the melting point of your unknown. When you have confirmed the identity of your unknown, record your findings in the report sheet.

EXPERIMENT – Melting Point Determination

Name _____

REPORT SHEET

Instructor _____

Date _____

I. Melting Points of Pure compounds

Name of compound	M.P., ^{o}C (Experimental)	M.P., ^{o}C (Literature)
h. _____		
i. _____		

II. Melting Point of an Impure Substance

Melting Point of a Mixture of (a) and (b) _____ ^{o}C

III. Identification of an Unknown Solid from its Melting Point

Unknown Number = _____

M.P. of PURE unknown = _____ ^{o}C

Trial 1: M.P.of unknown when mixed with _____ (name of compound) = _____ ^{o}C

Trial 2: M.P. of unknown when mixed with _____ (name of compound) = _____ ^{o}C

Trial 3: M.P. of unknown when mixed with _____ (name of compound) = _____ ^{o}C

My unknown compound is _____ (name of compound)

Pre-Laboratory Questions - Due before lab begins

2. List six physical properties of organic compounds that are often measured by organic chemists in attempting to identify a compound.
3. Melting point determination can be used for several purposes. What are those purposes?
4. Define the following terms:
   melting point –
   sublimation –
   sintering –
   eutectic mixture –
5. What is the effect of a small amount of impurity on the melting point of an organic compound?
6. What is the difference between the capillary melting point and true melting point?

Post-Laboratory Questions - Due after completing the lab

1. What would be the effect on the observed melting point if sample were:
   a) too small –
   b) too large –
   c) poorly packed –
   d) heated too rapidly –
2. Some compounds sublime in the capillary and some decompose before melting. How do you determine melting point of these compounds?
3. A student was given a white solid for an unknown. Its melting point range was 119 – 121 ^{o}C. The student has previously worked with benzoic acid, and had observed that it was a white crystalline solid with a melting point of 122 ^{o}C.
   (a) Can the student conclude that the unknown is benzoic acid on the basis of her work to this? Why or why not?
   (b) What additional experimental work should be done to verify this compound?
4. You and your lab partner take melting points of the same sample. You observe a melting point of 101-107°C, while your partner observes a value of 110-112°C. Explain how you can get two different values with exactly the same sample.
5. An unidentified compound is observed to melt sharply at 111 °C with the vigorous evolution of a gas. The sample then solidifies and does not melt until the temperature reaches 155 °C, at which time it again melts sharply. Briefly explain these observations.

Module 4

Unit 1

Structures of Hydrocarbons - Experiment with Models

Purpose

- Draw formulas for alkanes from their three dimensional models.
- Write the names of alkanes from their structural formulas.
- Construct models of isomers of alkanes.
- Write structural formulas for cycloalkanes and haloalkanes.

Discussion

The saturated hydrocarbons represent a group of organic compounds composed of carbon and hydrogen. Alkanes and cycloalkanes are called saturated hydrocarbons because their carbon atoms are connected by only single bonds. In each type of alkane, each carbon atom has four valence electrons and must always have four single bonds.

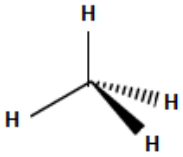
To learn more about the three-dimensional structure compounds, it is helpful to build models using a ball-and-stick model kit. In the kit are plastic (or wooden) balls and sticks, which represent typical elements and chemical bonds respectively in organic compounds.

Elements and bonds represented in the Organic Model Kit

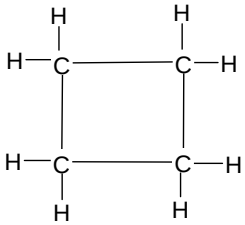
Color	Element	Number of bonds
Black	carbon	4
White	hydrogen	1
Red	oxygen	2
Yellow	nitrogen	3
Green	chlorine	1
Blue	bromine	1

The first model to build is methane, CH_4, a hydrocarbon consisting of one carbon and four hydrogen atoms. The model of methane shows the three-dimensional shape, a tetrahedron, around a carbon atom.

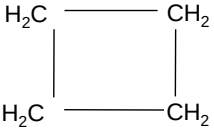
CH_4 (methane)



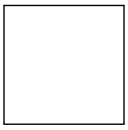
C_4H_8 (cyclobutane) Molecular formula



Structural formula



Condensed formula

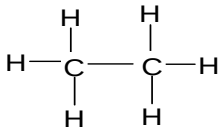


Skeletal formula

CH_2

Empirical formula

To represent this model on paper, its shape is flattened, and the carbon atom is shown attached to four hydrogen. This type of formula is called a *complete structural formula.* However, it is more convenient to use a shortened version called a *condensed structural formula*. To write a condensed formula, the hydrogen atoms are grouped with their carbon atom. The number of hydrogen atoms is written as a subscript. The complete structural formula and the condensed structural formula for C_2H_6 are shown below:



Complete Structural formula

CH_3——CH_3 or CH_3CH_3

Condensed Structural formula

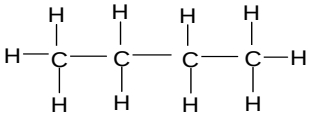
Naming Alkanes

The names of alkanes all end with ***–ane***. The names of organic compounds are based on the names of the alkane family.

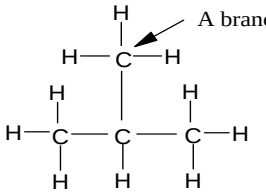
Name	**Formula**	**Name**	**Formula**
Methane	CH_4	Hexane	$CH_3CH_2CH_2CH_2CH_2CH_3$
Ethane	CH_3CH_3	Heptane	$CH_3CH_2CH_2CH_2CH_2CH_2CH_3$
Propane	$CH_3CH_2CH_3$	Octane	$CH_3CH_2CH_2CH_2CH_2CH_2CH_2CH_3$
Butane	$CH_3CH_2CH_2CH_3$	Nonane	$CH_3CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_3$
Pentane	$CH_3CH_2CH_2CH_2CH_3$	Decane	$CH_3CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_3$

Constitutional Isomers

Constitutional isomers are present when a molecular formula can represent two or more different structural (or condensed) formulas. One structure cannot be converted to the other without breaking and forming bonds. The isomers have different physical and chemical properties. One of the reasons for the vast array of organic compounds is the phenomenon of *isomerism*.



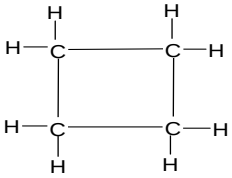
n-Butane



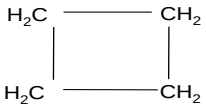
2-Methylpropane

Cycloalkanes

In a cycloalkane, and alkane has a cyclic or ring sructure. There are no end carbon atoms. The structural formula of a cycloalkane indicates all of the carbon and hydrogen atoms. The condensed formula groups the hydrogen atoms with each of the carbon atoms. Another type of notation called the ***geometric structure*** is often used to depict a cycloalkane by showing only the bonds that outline the geometric shape of the compound. For example, the geometric shape of cyclopropane is triangle, and the geometric shape of cyclobutane is square. Examples of the various structural formulas for cyclobutane are shown below.



Structural formula

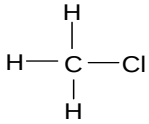
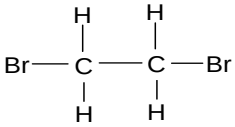


Condensed formula

Skeletal formula

Haloalkanes

In haloalkanes, a halogen atom such as chlorine (Cl) or bromine (Br) replaces a hydrogen atom of an alkane or a cycloalkane.

Complete structural formula	Condensed formula	Name
	CH_3Cl	Chloromethane (methyl chloride)
	$BrCH_2CH_2Br$	1,2-Dibromoethane

Experimental Procedures

Using an organic model kit, construct a ball-and-stick model of the following molecules. Draw the three-dimensional shape of the molecules and write the complete structural formulae and condensed formulae for all the isomers if any. Be sure to name all compounds.

(I) Alkanes

A) Methane, CH_4 B) Ethane, C_2H_6 C) Propane, C_3H_8

D) Butane, C_4H_{10} E) Pentane, C_5H_{12} F) Hexane, C_6H_{14}

(II) Cycloalkanes

G) Cyclopropane, C_3H_6 H) Cyclobutane, C_4H_8 I) Cyclopentane, C_5H_{10}

J) Cyclohexane, C_6H_{12}

(III) Haloalkanes

K) 1,2-dichloropropane L) Bromoethane M) Dibromopropane

N) 1,2-dichlorocyclopentane O) 2- methylpropane

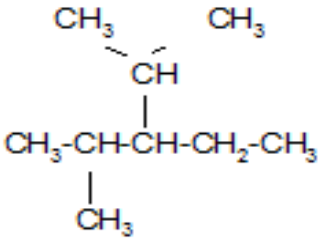
Pre-Laboratory Questions - Due before lab begins

m. How do you distinguish between molecular formula, empirical formula (simplest), and structural formula of benzene? (Draw structures)

n. How do you distinguish between geometrical and structural isomers? Give examples.

o. Why should the properties of structural isomers differ?

p. Draw skeletal formula for the following compound.



5. Draw all possible cyclic isomers for C_4H_8 and name all isomers.

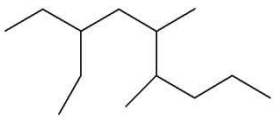
Post-Laboratory Questions - Due after completing the lab

j. Write the condensed structural formulas and names for all the constitutional isomers with the formula C_4H_9Br.

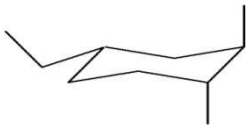
k. Write the correct names of the alkanes and cycloalkane

a) CH_3- C $(CH_3)_2$ – $CH(CH_3)$ CH_3

b)



c)



Br

7. Write condensed formulas for the followings:

2,3,3,4 –tetramethylnonane

1-butyl-4-methylcyclodecane

neopentane

8. n-butane and isobutane are constitutional isomers. What is the boiling point of each compound?

9. Define geometrical isomers and give examples. (structural formulas and names)

Unit 2
Reactions of Hydrocarbons

Properties and Identification of Hydrocarbons

Purpose:

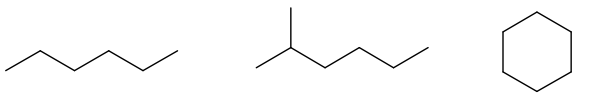
- To identify saturated and unsaturated hydrocarbons using properties and reactions.
- Study substitution and addition reactions.

Equipments	**Materials**	
Test tube rack (1)	Cyclohexane	n-Hexane
Test tube holder (2)	0.50 % $KMnO_4$	Conc. H_2SO_4
100 mL beaker	Toluene	Br_2/CCl_4 or Br_2/CH_2Cl_2
Medium test tube (6)	10 % Na_2CO_3	Unknown hydrocarbons

Discussion

The number of known organic compounds totals into the millions. Of these compounds, the simplest types are those that contain only hydrogen and carbon atoms. These are known as *hydrocarbons*. Because of the number and variety of hydrocarbons that can exist, some means of classification is necessary.

One means of classification depends on the way in which carbon atoms are connected. *Chain* aliphatic hydrocarbons are compounds consisting of carbons linked either in a single chain or in a branched chain. *Cyclic* hydrocarbons are aliphatic compounds that have carbon atoms linked in a closed polygon (also referred to as a *ring*). For example, hexane (single) and 2-methylpentane (branched) are chain aliphatic molecules, while cyclohexane is a cyclic aliphatic compound.



Hexane 2-Methylpentane Cyclohexane

Another means of classification depends on the type of bonding that exists between carbons. Hydrocarbons that contain only carbon-to-carbon single bonds are called *alkanes*. These are also referred to as *saturated* molecules. Hydrocarbons containing at least one carbon-to-carbon double bond are called *alkenes*, and compounds with at least one carbon-to-carbon triple bond are called *alkynes*. Alkenes and alkynes are referred to as *unsaturated* molecules. Finally, a class of cyclic hydrocarbons that contain a closed loop (sextet) of electrons is called *aromatic*. With so many compounds possible, identification of the bond type is an important step in establishing the molecular structure. Quick, simple tests on small samples can establish the physical and chemical properties of the compounds by class.

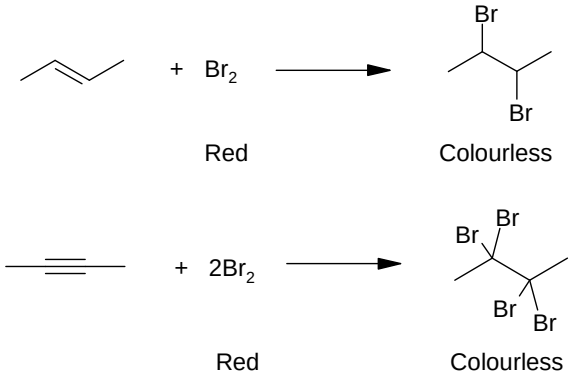
Some of the observed physical properties of hydrocarbons result from the nonpolar character of the compounds. In general, hydrocarbons do not mix with polar solvents such as water or ethanol (ethyl

alcohol). On the other hand, hydrocarbons mix with relatively nonpolar solvents such as ligroin (a mixture of alkanes), carbon tetrachloride (CCl_4), or dichloromethane (CH_2Cl_2). Because the density of most hydrocarbons is less than that of water, they will float. Crude oil and crude oil products (home heating oil and gasoline) are mixtures of hydrocarbons; when spilled on water, these substances spread quickly along the surface because they are insoluble in water.

The chemical reactivity of hydrocarbons is determined by the type of bond in the compound. Unsaturated hydrocarbons (i.e., alkenes and alkynes) react by *addition* of reagents to the double or triple bonds. The addition products become saturated, with fragments of the reagent becoming attached the carbons of the multiple bond. Aromatic compounds, with a higher carbon-to-hydrogen ratio than nonaromatic compounds, undergo *substitution* in the presence of catalysts rather than an addition reaction.

1. *Reaction with bromine.*

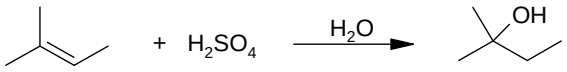
Unsaturated hydrocarbons react rapidly with bromine in a solution of carbon tetrachloride or cyclohexane. The reaction is the addition of the elements of bromine to the carbons of the multiple bonds.



The bromine solution is red; the product that has the bromine atoms attached to carbon is colorless. Thus, a reaction has taken place when there is a loss of color from the bromine solution and a colorless solution remains. Because alkanes have only single C- C bonds present, no reaction with bromine is observed; the red color of the reagent would persist when added. Aromatic compounds resist addition reactions because of their "aromaticity": *the possession of a closed loop (sextet) of electrons which imparts extreme stability.* These compounds can react with bromine but require the presence of a catalyst such as iron fillings or aluminum chloride.

2. *Reaction with concentrated sulfuric acid.*

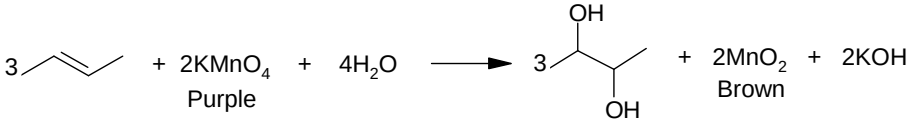
Alkenes react with cold concentrated sulfuric acid by addition. Alkyl sulfonic acids form as products and are soluble in H_2SO_4; subsequent water work-up results in an "-OH" on the more substituted carbon (as demonstrated in lecture).



Saturated hydrocarbons are unreactive (additions are not possible); alkynes react slowly and require a catalyst (H_2SO_4); due to their inherent stability, aromatic compounds are also unreactive.

3. *Reaction with potassium permanganate*.

Dilute or alkaline solutions of $KMnO_4$ oxidize unsaturated compounds. Alkanes and aromatic compounds are generally unreactive. Evidence that a reaction has occurred is observed by the loss of the purple color of $KMnO_4$ and the formation of the brown precipitate manganese dioxide, MnO_2.



Note that the product formed (which contains two "-OH" groups) is called a glycol.

Experimental Procedure

Assume the organic compounds are highly flammable. Use only small quantities. Keep away from open flames. Assume the organic compounds are toxic and can be absorbed through the skin. Avoid contact; wash if any chemical spills on your person. Handle concentrated sulfuric carefully. Flush with water if any spills on your person. Potassium permanganate and bromine are toxic; bromine solutions are also corrosive. Although the solutions are diluted, they may cause burns to the skin. Wear gloves when working with these chemicals. Also consider the following:

1. The hydrocarbons hexane, cyclohexene, and toluene (alkane, alkene and aromatic, respectively) are available in dropper bottles.
2. The reagents 1% Br_2 in cyclohexane, 1% aqueous $KMnO_4$, and concentrated H_2SO_4 are available in dropper bottles.
3. Unknowns are in dropper bottles labeled A, B, and C. They may include an alkane, an alkene, and/or an aromatic compound.
4. Test tubes will be suitable for all the tests; mix thoroughly.
5. Dispose of all organic wastes as directed by the instructor. *Do not pour them into the sink!*

Physical Properties of Hydrocarbons

1. *Water solubility of hydrocarbons*. Label six test tubes with the name of the substance to be tested. Place into each test tube 5 drops of the appropriate hydrocarbon: hexane, cyclohexene, toluene, unknown A, unknown B, and unknown C. Add about 5 drops of water dropwise into each test tube. Water is a polar solvent. Is there any separation of components? Which component is on the bottom; which component is on the top? Mix the contents. What happens when the contents are allowed to settle? What do you conclude about the density of the hydrocarbon? Is the hydrocarbon *more* dense than water or *less* dense than water? Record your observations. Save these solutions for comparison with the next part.

2. *Solubility of hydrocarbons in ligroin*. Label six test tubes with the name of the substance to be tested.

Place into each test tube 5 drops of the appropriate hydrocarbons: hexane, cyclohexene, toluene, unknown A, unknown B, and unknown C. Add about 5 drops of ligroin dropwise into each test tube. Ligroin is a nonpolar solvent. Is there a separation of components? Is there a bottom layer and a top layer? Mix the contents. Is there any change in the appearance of the contents before and after mixing? Compare these test tubes with those from the previous part. Record your observations. Can you make any conclusion about the density of the hydrocarbons from what you actually see?

Chemical Properties of Hydrocarbons

i. *Reaction with bromine*. Results provided on data sheet.

ii. *Reaction with $KMnO_4$* (Baeyer's test). Label six clean, dry test tubes with the name of the substance to be tested. Place into each test tube 5 drops of the appropriate hydrocarbon: hexane, cyclohexene, toluene, unknown A, unknown B, and unknown C. Carefully add (dropwise) 1% aqueous $KMnO_4$ solution; after each drop, shake to mix the solutions. Keep count of the number of the drops needed to have the colour of the permanganate solution persist; do not add more than 10 drops. Record your observations.

iii. *Reaction with concentrated H_2SO_4*. Label six clean, dry test tubes with the name of the substance to be tested. Place into each test tube 5 drops of the appropriate hydrocarbon: hexane, cyclohexene, toluene, unknown A, unknown B, and unknown C. Place all of the test tubes in an ice bath. Wear gloves and carefully add (with shaking) 3 drops of cold, concentrated sulfuric acid to each test tube. Note whether the solution has become homogeneous or whether a color is produced. (The evolution of heat, the formation of a homogeneous solution, or the appearance of a color is evidence that a reaction has occurred.) Record your observations.

iv. *Unknowns*: By comparing the observations you made for your unknowns with that of the known hydrocarbons, you can identify unknowns A, B, and C. Record their identities on your data sheet.

Data and Results (Properties of Hydrocarbons)

REPORT SHEET

Physical properties of hydrocarbons

Solubility: Does the hydrocarbon mix with the solvent, *soluble*, or not mix with solvent, *insoluble*? Use the observations you make for the solubility tests and determine whether the hydrocarbons are polar or nonpolar substances.

Density: For water, is the density *greater* than water (sinks) or *less* than water (floats)? For ligroin, can you tell anything about the relative densities?

	H_2O		**Ligroin**	
Hydrocarbon	**Solubility**	**Density**	**Solubility**	**Density**
Hexane				
Cyclohexene				
Toluene 				
Unknown A				
Unknown B				
Unknown C				

Chemical properties of hydrocarbons (note: results for bromine test are provided)

Hydrocarbon	**Bromine Test**	**$KMnO_4$ Test**	**H_2SO_4 Test**
Hexane			
Cyclohexene			
Toluene 			
Unknown A			
Unknown B			
Unknown C			

Unknown A is _____.

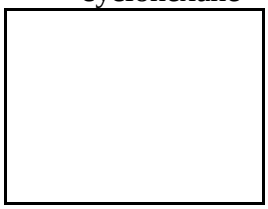
Unknown B is _____.

Unknown C is _____.

Pre-Laboratory Questions - Due before lab begins

1. Draw skeletal (line –bond) structures for the following compounds.

cyclohexane



cyclohexene

toluene

2. What are the general formulas for alkanes and cycloalkanes?

Alkanes

cycloalkanes

3. Write equations for the reaction of 1-butene with the following reagents.

a) Br_2/CH_2Cl_2

c) $KMnO_4$/ hot

4. How could you distinguish octane from 1-octene by a simple chemical test?

5. What would you expect the difference between reactivity of the following pairs? Please explain your answers

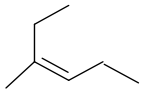
a) hexane and Cyclohexane b) hexane and cyclohexene

Post-Laboratory Questions - Due after completing the lab

Below are four organic compounds. The reagent shown is added to the compound. Based on your studies in this lab, determine the products (if any) that you should observe when the reactants below are mixed together:

A.  + Br_2 ⟶

B.  + $KMnO_4$ ⟶

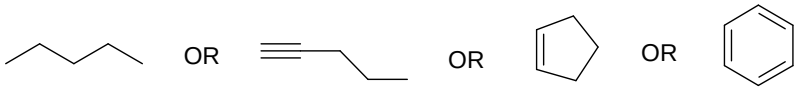
C.  + H_2SO_4 $\xrightarrow{H_2O}$

D.  + $KMnO_4$ ⟶

2. A student has two compounds in two separate bottles but with no labels on either one. One is an unbranched alkane, octane (C_8H_{18}); the other is 1-hexene (C_6H_{12}), an unbranched alkene. Based on your observations in this experiment, tell what you should observe via the following tests:

		Octane	**1-Hexene**
A.	Water solubility		
B.	Ligroin solubility		
C.	Density versus water		
D.	Bromine test		
E.	Permanganate test		

3. An unknown compound, believed to be a hydrocarbon, showed the following behavior: no heat or color appeared when sulfuric acid was added; permanganate solution remained purple; and the red color of bromine solution was lost only after a catalyst was added. From the compounds below, circle the ONE that fits the observations.



4. Draw condensed structure formulas for the following compounds.

cyclohexane

cyclohexene

toluene

5. Write equationrs for the reaction of cyclohexene with the following reagents:

a. Br_2/CH_2Cl_2

b. $KMnO_4$/cold

h) How could you distinguish 1-octanol from 1-octene by a simple chemical test?

Unit 3

Alkene Synthesis from Alcohol

Preparation of Cyclohexene From Cyclohexanol

Purpose:

4. Preparation of an alkene by dehydration (elimination of water) of an alcohol in the presence of an acid catalyst.
5. Calculation of percentage recovery of product.
6. Test for purity and identification of alkenes

Equipment:

10-mL graduated cylinder	Condenser	Glass adaptor (2)
Round bottom flask (25 mL, 50 mL)	Thermometer	Rubber tubing (2)
Heating mantle	Thermometer adaptor	Grease

Chemicals:

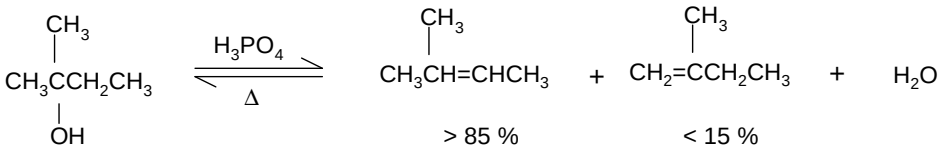
Cyclohexanol	Phosphoric acid (85 %)	10 % Na_2CO_3
Br_2/CCl_4	0.5 % $KMnO_4$	Drying agent ($CaCl_2$)

Discussion:

Dehydration is an elimination reaction of an alcohol. The elimination reaction involves the loss of an OH from one carbon and an H from an adjacent carbon. Overall, this amounts to the elimination of a molecule of water, resulting in a pi-bond formation of an alkene or alkyne. The loss of water from a molecule is called dehydration. In many cases alcohol dehydration requires an acid catalyst and heat. Phosphoric acid (H_3PO_4) and sulfuric acid (H_2SO_4) are the most commonly used acid catalysts.

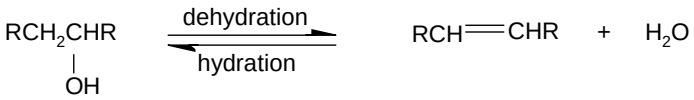
When more than one elimination product can be formed, the major product is the more substituted alkene - the one obtained by removing a proton from the adjacent carbon that has fewer hydrogens (Recall Zaitsev's rule). The more substituted alkene is the major product because it is the more stable alkene, so it has the more stable transition state leading to its formation.

Dehydration of 2-methyl-2-butanol produces primarily 2-methyl-2-butene, a tri-substituted alkene, rather than 2-methyl-1-butene, a di-substituted alkene:



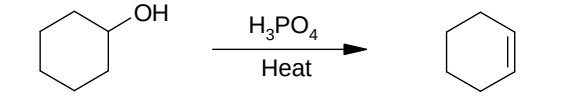
Alkenes can be hydrated (water molecules added) in the presence of an acid catalyst:

The hydration of an alkene is the reverse of the acid-catalyzed dehydration of an alcohol:



To prevent the alkene formed in the dehydration reaction from reforming back the alcohol, the alkene can be removed by distillation as it is formed, because it has a much lower boiling point than the alcohol. Removing a product displaces the reaction to the right. (Recall Le-Chatelier's principle).

In this experiment, Cyclohexanol is dehydrated to cyclohexene according to the following reaction:



b.p. = 160 - 161 °C b.p. = 83 °C

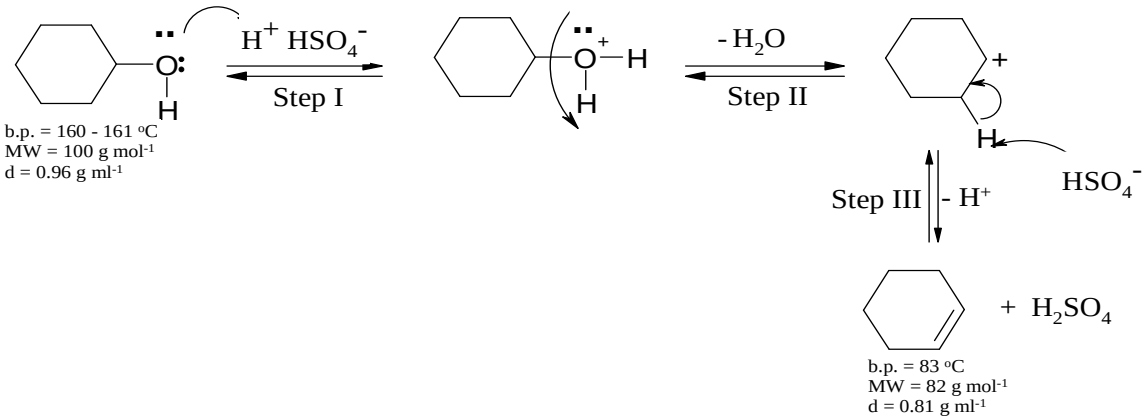
Because the OH group is a very poor leaving group, an alcohol is able to undergo dehydration only if its OH group is converted into a better leaving group.

One way to convert an OH group into a good group is to protonate it.

- In the first step of dehydration reaction, protonation changes the very poor leaving group –OH into a good leaving group $–OH_2^+$.
- In the second step, water departs, leaving behind a carbocation.
- In the third step, the base HSO_4^- removes a proton from the carbon adjacent to the positively charged carbon, forming an alkene and regenerating the acid catalyst H_2SO_4.

Because the cyclohexene has lower boiling point than the cyclohexanol, the cyclohexene can be distilled as it forms.

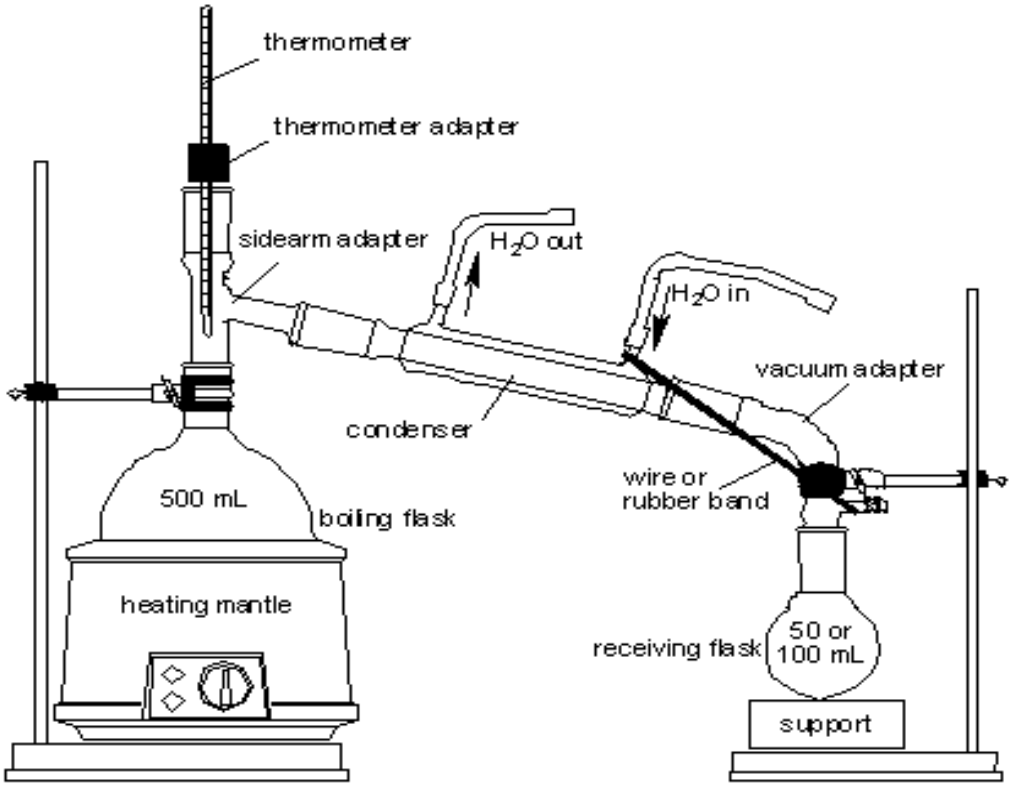
<u>Mechanism:</u>



The reaction is conducted in a distillation apparatus. As the reaction mixture is heated, the lower boiling products (cyclohexene, b.p. 83^{o}C, and water, b.p. = 100 ^{o}C) distill out and are collected in the receiving flask. Any unreacted cyclohexanol (the starting material) and Phosphoric acid (the catalyst) are left in the distilling flask because of their high boiling points (161^{o}C and 213^{o}C, respectively). However, since a small amount of phosphoric acid still appears in the receiving flask, the product is washed with aqueous sodium carbonate to neutralize the acid. Cyclohexene is insoluble in water and thus is not lost during the washing with aqueous sodium carbonate solution. The last traces of water are removed from the crude cyclohexene using anhydrous sodium sulphate (a salt which forms a hydrate).

Experimental Procedure

Step I - Measure 10 mL of cyclohexanol into 50.0 mL round -bottom flask. Carefully add 3 mL of 85% phosphoric acid; H_3PO_4, the boiling chips, and mount the flask for simple distillation (see Fig. Below: either use column packing or low hold-up packing). Slowly heat the mixture until it comes to a gentle boil. After 10 minutes of gentle boiling, increase the heat sufficiently to cause distillation (the temperature of the distilling vapor should not exceed 100 ^{o}C) and collect the distillate in a cooled 25 mL round bottom flask as a receiver.



Laboratory display of distillation

To the distillate, add 1 mL of 10% sodium carbonate solution, Na_2CO_3, to neutralize any traces of acid, which have being carried over. Transfer the liquid to a separatory funnel, add 5 ml of cold water, swirl the mixture gently, and drain off the lower aqueous layer.
Pour the upper organic layer into a small, dry 50 mL Erlenmeyer flask, and dry it over anhydrous calcium chloride (add about on tea spoon) for 5 to 10 minutes with a cover.

Step II (ask instructor if necessary) - Decant the dried cyclohexene into a small distilling flask, add a boiling stone, attach the flask to a simple distillation assembly, and distill carefully. Collect the material distilling at 80 to 85 ^{o}C. Determine the weight of the product and calculate the percentage yield.

Step III – Use 5-10 drops of product in two small test tubes and test with drop-wise bromine (decoloration) and drop-wise potassium permanganate (dark brown precipitate) for purity and identification of alkene.

Alkene Synthesis from Alcohol
Data and Results

REPORT SHEET

Name _____

Instructor _____

Date _____

Reaction Equation

Amount of reactant (cyclohexanol) used _____ g _____ ml

Theoretical number of moles cyclohexanol (calculation) _____ mole

Theoretical number of moles cyclohexene (calculation) _____ mole

Amount of product theoretically obtainable (calculation) _____ g

Actual yield _____ g

Percentage yield (calculation) _____ %

Pre-Laboratory Questions - Due before lab begins

1. Define the following terms Dehydration –
   Catalysis –
   Chaser solvent -

2. What is the function of each of the following reagents in this experiment: phosphoric acid; anhydrous sodium sulphate; sodium carbonate solution, and saturated sodium chloride solution.

3. Outline the mechanism for the dehydration of 2-methylcychohexanol
   Name or draw the structure, whichever is appropriate, of each of the following compounds.
   a. cyclohexene b. CH_3-CH_2-CH=CH_2 c. 2-methyl-1-pentane
   d. CH_3-CH_2-OH e. cyclopentanol

Post-Laboratory Questions - Due after completing the lab

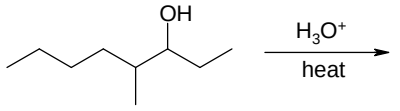
1. Outline mechanism for the dehydration of 1-methyl-1-cyclohexanol.

2. What is the major disadvantage of using concentrated sulphuric acid (or hydrochloric acid) rather than 85% phosphoric acid for the dehydration of alcohols?

3. Why is the receiving flask supposed to be kept on ice during the preparation of cyclohexene?

4. If 0.138g of cyclohexene (C_6H_{10}) was obtained from 0.240g of cyclohexanol ($C_6H_{12}0$), what is the percentage yield of cyclohexene?

5. Complete each of the following reactions by drawing the structures of the organic products.

a. 

b. 

c.