King Khalid University

College of Science
Department of Chemistry

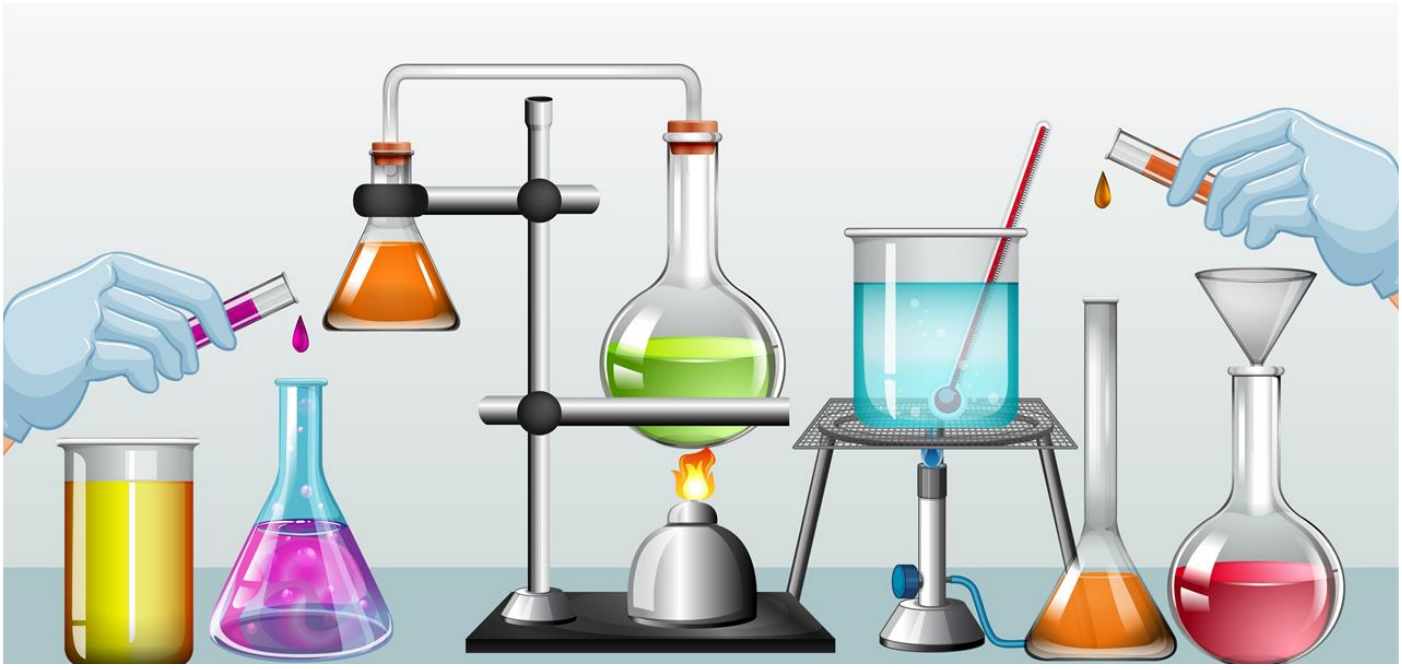
جامعة الملك خالد

كلية العلوم
قسم الكيمياء

GENERAL CHEMISTRY 1

Laboratory Manual

1101Chem



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LABORATORY SAFETY RULES

The following safety rules must be followed at all times in the laboratory. The chemical laboratory is not necessarily a dangerous place. Intelligent precautions and a proper understanding of techniques to be followed make the chemistry laboratory no more dangerous than any other classroom.

1. Safety goggles (department approved) must be worn in the lab at all times. Glasses and contact lenses are not acceptable eye protection. Students who do not follow this rule will be asked to leave the lab immediately.

2. **Never eat or drink in the lab**. Food may pick up toxic chemicals.

3. **Never inhale fumes or vapors**. Use fume hoods for dangerous or irritating chemicals. Always waft odors toward your nose with your hand.

4. **Never taste any chemical.** Some chemicals are very corrosive and poisonous in very small quantities.

5. **Never perform an unauthorized experiment and never work in the lab without an instructor in charge.** An accident may happen when mixing simple chemicals. 6. Never remove anything (chemicals, glassware, etc.) from the lab. It is illegal!

7. **Label all containers to identify their contents**.

8. **Never put anything back into a reagent bottle**. Once a reagent has passed the mouth of its container, it has passed the point of no return. Always take as little of a chemical as possible. Use only clean, dry spatulas for removing chemicals from bottles. Properly dispose of excess chemicals.

9. **Leave chemicals in their proper place**. Do not carry original containers of chemicals to your benchtop.

10. **Avoid touching hot objects**. Burns are a common accident in the chemistry lab. Be careful when using hot plates and objects which have been heated on them. Use beaker tongs to remove hot containers from the hot plate.

11**. Rinse spills off skin immediately**. Rinse off any chemicals spilled on the skin immediately with large amounts of water.

12. **Clean up broken glassware immediately**. Place it in the labeled crock at the front of the lab. Obtain replacement glassware from the instructor.

13. **Properly dispose of waste chemicals**. Certain liquids can be poured into the sink and flushed with water while others are poured into designated waste containers. Most solid wastes are placed in designated crocks. Your instructor will provide disposal instructions each lab.

14. **Notify your instructor immediately of all accidents**.

15. **Learn to locate and operate** (if applicable), the safety shower, fire extinguisher, eye-wash fountain, fire blanket, and fire exit.

LAB PROCEDURES

The following are procedures that must be followed for all lab sessions. Many are safety issues as well.

1. **Dress properly for lab.** Wear clothing that covers as much skin as possible. Sandals are not permitted. If possible, wear older clothes on lab day. All loose clothing and long hair must be confined.

2. **Read the entire experiment before coming to lab**. The instructor will briefly discuss the experiment at the beginning of each lab.

3. **Arrive on time for lab.** If a student arrives late for lab and misses a substantial portion of the introductory discussion and safety precautions, the student may be a threat to themselves and others in the lab. The instructor may deny the student the privilege of completing the assigned experiment.

4. **Work independently** unless otherwise instructed

5. **Keep the benchtop uncluttered**. Only those personal items pertinent to the lab work (lab manual, etc.) are to be on the benchtop at a student's work station. Book bags, coats, etc. are not to be placed on the benchtop or on the floor close to the lab benches. Place all such items in the designated areas near the entrance to the lab.

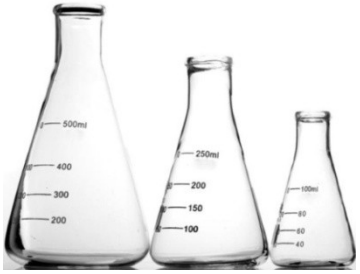
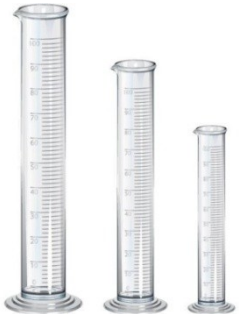
6. **Keep drawers closed**. Drawers and cabinets are to be kept closed except when items are being taken from or returned to these drawers.

7. **Take only planned breaks**. If the need arises to take a short break, you may do so at any time during the experiment with these points in mind; try to plan the break during a less critical time in the experiment (e.g., while something is cooling); make sure that your hot plate is turned off; inform a neighbor and the instructor.

8. **Do not come to lab under the influence of drugs.** If, in the judgement of the instructor, a student presents a safety hazard to himself or his fellow students because the student is affected by medication, alcohol or other factors, the instructor may refuse to allow the student to continue working in the lab that day. If the situation is noted more than once, the student may be permanently removed from the course.

9. **Clean up at the end of lab**. At the end of all lab sessions return clean glassware to your drawer, clean your benchtop and finally wash your hands thoroughly. Be sure all electrical devices and water are turned off.

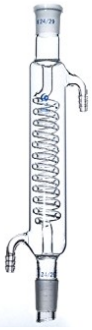
KNOW YOUR LAB EQUIPMENTS

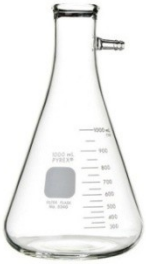
Test tube A test tube, also known as a sample tube, is a common piece of laboratory glassware consisting of a finger-like length of glass or clear plastic tubing, open at the top and closed at the bottom.	
Beakers Beakers are used as containers. They are available in a variety of sizes. Although they often possess volume markings, these are only rough estimates of the liquid volume. The markings are not necessarily accurate.	
Erlenmeyer flask Erlenmeyer flasks are often used as reaction vessels, particularly in titrations. As with beakers, the volume markings should not be considered accurate.	
Volumetric flask Volumetric flasks are used to measure and store solutions with a high degree of accuracy. These flasks generally possess a marking near the top that indicates the level at which the volume of the liquid is equal to the volume written on the outside of the flask. These devices are often used when solutions containing dissolved solids of known concentration are needed.	
Graduated cylinder Graduated cylinders are used to transfer liquids with a moderate degree of accuracy.	

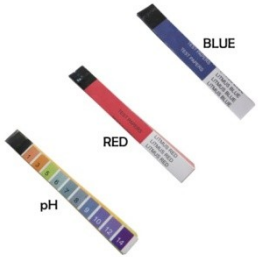
Pipette Pipettes are used for transferring liquids with a fixed volume and quantity of liquid must be known to a high degree of accuracy.	
Graduated pipette These Pipettes are calibrated in the factory to release the desired quantity of liquid.	
Disposable pipette Disposable transfer. These Pipettes are made of plastic and are useful for transferring liquids dropwise.	
Burette Burettes are devices used typically in analytical, quantitative chemistry applications for measuring liquid solution. Differing from a pipette since the sample quantity delivered is changeable, graduated Burettes are used heavily in titration experiments. In Burette and Pipettes it is common to measure the volume delivered. This means that two volume measurements will be made. The volume delivered is the difference in these two volumes, V_{final} - $V_{initial}$.	
Burette clamp Burette clamp is scientific equipment which used specifically to hold and secure a burette on a stand, so that a burette is fixed and more convenient for the experiment. Burette clamp can be made by many materials such as plastic and cast iron. However, iron clamp with rubber knob to hold burette are likely to be more durable. Usually Burette clamp comes in double, which means it can hold two burettes.	
Funnel A tube or pipe that is wide at the top and narrow at the bottom, used for guiding liquid or powder into a small opening.	

Buchner Funnel A Büchner funnel is a piece of laboratory equipment used in filtration. It is traditionally made of porcelain, but glass and plastic funnels are also available. On top of the funnel-shaped part there is a cylinder with a fritted glass disc/perforated plate separating it from the funnel.	
Buchner funnel vacuum filtration setup Suction filtration (vacuum filtration) is the standard technique used for separating a solid-liquid mixture when the goal is to retain the solid (for example in crystallization). Similar to gravity filtration, a solid-liquid mixture is poured onto a filter paper, with the main difference being that the process is aided by suction beneath the funnel.	
Clamp It is used to hold round laboratory glassware, such as a beaker, and flasks, etc. This type of clamp is made from stainless steel.	
Test tube brush Test tube brush or spout brush is a brush used for cleaning test tubes and narrow mouth laboratory glassware, such as graduated cylinders, burettes, and Erlenmeyer flasks.	
Test tube holder A test tube holder is used to hold test tubes. It is used for holding a test tube in place when the tube is hot or should not be touched.	
Test tube stand Test tube racks are laboratory equipment used to hold upright multiple test tubes at the same time.	
Bunsen burner A small adjustable gas burner used in laboratories as a source of heat.	

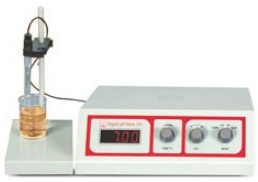
Petri dish A petri dish is a small dish shaped like a cylinder.	
Glass rod A glass stirring rod, glass rod, stirring rod or stir rod is a piece of laboratory equipment used to mix chemicals and liquids for laboratory purposes. They are usually made of solid glass, about the thickness and slightly longer than a drinking straw, with rounded ends.	
Graduated Dropper A dropper or Pasteur pipette is an instrument used to transfer small quantities of liquid.	
Tongs Tongs are a type of tool used to grip and lift objects instead of holding them directly with hands.	
Utility clamp A utility clamp is a laboratory apparatus resembling a pair of scissors. The screw in the middle works as the wide adjustment of 2-prong. It is composed of 3 parts: 2-prong adjust, metal rod, and clamp down (attach the clamp to the ring stand for adjust the height).	
Spot test plate A spot plate, also called a reaction plate is a laboratory tool made either from ceramics.	

Tripod for Bunsen burner A laboratory tripod is a three-legged platform used to support flasks and beakers.	
Wash Bottle A wash bottle is a squeeze bottle with a nozzle, used to rinse various pieces of laboratory glassware, such as test tubes and round bottom flasks. Wash bottles are sealed with a screw-top lid.	
Wire Mesh Gauze (Ceramic Centre) Wire mesh (wire gauze) is used for uniform distribution of flame heat in base of a heated body. It is made of iron wire strands with or without ceramic interior cores.	
Spatula In laboratories, spatulas and microspatulas are small stainless steel utensils, used for scraping, transferring, or applying powders and paste like chemicals or treatments. Many spatula brands are also resistant to acids, bases, heat, and solvents, which make them ideal for use with a wide range of compound.	
Round-bottom flasks Round-bottom flasks (also called round-bottomed flasks or RB flasks) are types of flasks having spherical bottoms used as laboratory glassware, mostly for chemical or biochemical work.	
Glass Condenser A condenser is an apparatus or item of equipment used to condense (change the physical state of a substance from its gaseous to its liquid state). In the laboratory, condensers are generally used in procedures involving organic liquids brought into the gaseous state through heating, with or without lowering the pressure (applying vacuum)—though applications in inorganic and other chemistry areas exist.	

Filter paper Filter paper is a semi-permeable paper barrier placed perpendicular to a liquid or air flow. It is used to separate fine substances from liquids or air. It is used in science labs to remove solids from liquids.	
Separatory funnel A separatory funnel, also known as a separation funnel, separating funnel, or colloquially sep funnel, is a piece of laboratory glassware used in liquid-liquid extractions to separate (partition) the components of a mixture into two immiscible solvent phases of different densities.	
Filtering flask A filtering flask is a piece of labware used to separate solids from fluids in filtration operations. To filter substances, the flask is used with a filtering funnel, such as a Buchner funnel, which incorporates a fritted glass disc or perforated plate fitted with a piece of filter paper to filter particles.	
Watch glass A watch glass is a circular concave piece of glass used in chemistry as a surface to evaporate a liquid, to hold solids while being weighed, for heating a small amount of substance and as a cover for a beaker. The latter use is generally applied to prevent dust or other particles entering the beaker; the watch glass does not completely seal the beaker, so gas exchanges still occur.	
Fusion tube Fusion tube is laboratory tube used much in the same way as boiling tubes expect not being as large and thick walled. A fusion tube made of thinner glass because it is intended to be broken into a container of water at the end of the fusion tube.	
Mortar and pestle Mortar and pestle are implements used since ancient times to prepare ingredients or substances by crushing and grinding them into a fine paste or powder in the laboratory.	

Reagent bottle Reagent bottles, also known as media bottles or graduated bottles, are containers made of glass, plastic, borosilicate or related substances, and topped by special caps or stoppers and are intended to contain chemicals in liquid or powder form for laboratories and stored in cabinets or on shelves.	
Litmus and pH paper Litmus or pH paper contains a chemical that changes color as it makes contact with an acid or base. The paper will turn red in acids and blue in bases.	
Sample containers Sample containers are very useful for storing chemicals solid samples. Very useful for transport purposes as it allows leak proof and airtight closures.	
Desiccator Desiccators are sealable enclosures containing desiccants used for preserving moisture-sensitive chemicals. A common use for desiccators is to protect chemicals which are hygroscopic or which react with water from humidity.	
Crucible A crucible is a ceramic or metal container in which metals or other substances may be melted or subjected to very high temperatures.	
Kipp's apparatus A laboratory apparatus for producing a gas, usually hydrogen sulphide, by the action of a liquid on a solid without heating.	

Mechanical Shaker It is a piece of laboratory equipment used to mix, blend, or agitate substances in a bottle or flask by shaking them.	
Digital Balance The digital mass balances in the General Chemistry labs are very sensitive instruments used for weighing substances to the milligram (0.001 g) level. Please treat them with care. Use containers when weighing chemicals and always weigh objects at room temperature. Keep the draft shields closed. Do not jar the instruments or change the levels. Always clean the area around the pan with a sable brush after use and inform the lab staff if any liquids or solids spill onto the balance.	
Magnetic Stirrer A magnetic stirrer is a device widely used in laboratories and consists of a rotating magnet or a stationary electromagnet that creates a rotating magnetic field. This device is used to make a stir bar, immerse in a liquid, quickly spin, or stirring or mixing a solution.	
Water bath A water bath is laboratory equipment made from a container filled with heated water. It is used to incubate samples in water at a constant temperature over a long period of time.	
Vacuum Pump It can evacuate chemically aggressive gases and vapors.	
Digital Colorimeter A colorimeter is a light-sensitive device used for measuring the transmittance and absorbance of light passing through a liquid sample. The device measures the intensity or concentration of the color that develops upon introducing a specific reagent into a solution.	
Digital Conductometer A conductivity meter measures the electrical conductivity in a solution. It has multiple applications in research and engineering.	

Digital pH meter A pH meter is a scientific instrument that measures the hydrogen-ion activity in water-based solutions, indicating its acidity or alkalinity expressed as pH.	
Digital Potentiometer A potentiometer is an instrument for measuring voltage by comparison of an unknown voltage with a known reference voltage. If a sensitive indicating instrument is used, very little current is drawn from the source of the unknown voltage. Since the reference voltage can be produced from an accurately calibrated voltage divider, a potentiometer can provide high precision in measurement.	

Experiment 1: – Measurements

INTRODUCTION

In all sciences, *measurements* are essential. The most fundamental properties that can be measured are length, mass, and time. In chemistry, temperature is also treated as a fundamental property. Other properties of matter such as volume, area, and density are ratios or products of the fundamental properties. For example, (a) units of area are length x width, or $(\text{distance})^2$ (b) units of volume are length x width x height, or $(\text{distance})^3$ (c) units of density are mass / volume, or mass / $(\text{distance})^3$ The metric system is used almost exclusively in all sciences. The meter, kilogram, and the second are the basic units in the International System of Units (le Système International d'Unités, SI), but the meter and the kilogram are generally too large for convenient use in the chemistry laboratory. Units in metric system are related to each other as multiples of ten and associated with prefixes such as, **pico** (10^{-12}), **nano** (10^{-9}), **micro** (10^{-6}), **milli** (10^{-3}), **centi** (10^{-2}), **deci** (10^{-1}), **kilo** (10^{3}), and **mega** (10^{6}). This experiment has been designed to acquaint you with several types of measurements and measuring devices. One of the fundamental properties of any sample of matter is its **density**. This property is dependent on the **mass** and the **volume** of the sample. The relationship between density, mass, and volume is,

Density = mass / volume or D = M / V

The density of a liquid or of a solution is usually reported in units of grams per milliliter, or g/mL. The density of a solid is reported in units of grams per cubic centimeter (g/cm^3). Because 1 mL is equivalent to 1 cm^3, these units are interchangeable. The density of water is 1.00000 g/cm^3 at 4°C and is slightly less at room temperature (0.9970 g/cm^3 at 25°C).

For any density determination, two quantities must be determined: the mass and the volume of a given quantity of matter. The mass can easily be determined by weighing a sample of the substance on a balance.

The mass of a sample of liquid in a container can be found by taking the difference between the mass of the container plus the liquid and the mass of the empty container.

The volume of a liquid can easily be determined by means of "graduated" containers such as graduated cylinders, pipettes, or others for routine measurements. The volume of a solid can be determined by direct measurement if the solid has a regular geometric shape such as a cube, rectangle, or cylinder.

A convenient way to determine the volume of a solid (usually irregular-shaped) is to measure accurately the volume of liquid displaced (raised) when an amount of the solid is dropped (immersed) in the liquid. The volume of the solid will equal the volume of liquid which it displaces.

Experimental Procedure

A. Mass Measurements

After cleaning the pan and zeroing the balance, take a coin (penny) and measure its mass to +/– 0.01 g. Record the mass on the report sheet in grams and milligrams. Repeat the measurement for two other coins.

B. Volume Measurements

I. Read the volume of liquid in the graduated cylinder designated by instructor to +/– 0.1 mL.
II. Using a dropper, measure the volume of 20 drops of tap water in a 10-mL graduated cylinder.

III. Using a 10-mL pipette, transfer 10 mL of tap water to a 25-mL graduated cylinder and read the volume of tap water in the graduated cylinder.

IV. Using a ruler or meter stick, measure the length, width, and height (thickness) of a given wooden block and calculate the volume of the block. You will use this same block for Part D. V. Measure the volume of a given irregular shaped solid by the water displacement method (refer to the illustration in Part D, below). Record the type of solid used on the report sheet.

C. Temperature Measurements

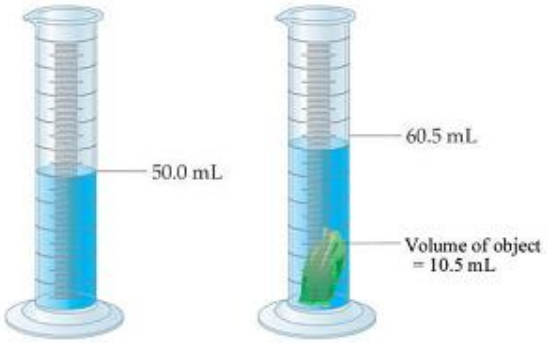
Using a thermometer, determine the temperature of the following:

I. 50 mL of tap water in a 150-mL beaker.
II. About 100 mL of ice in a 250-mL beaker, with a small amount of water added.
III. 100 mL of boiling deionized water (from a deionizing filter) in a 250-mL beaker.

D. Density Measurements

Method I for Solids

Weigh a given solid on a balance to the nearest 0.01g. Make sure you zero the balance before proceeding with each measurement. Record the type of solid used on the report sheet. Fill a 50 or 100-mL graduated cylinder about half full with tap water and record the volume as accurately as possible. Next, place the solid object in the graduated cylinder and record the water level after the solid is immersed. When making volume measurements, you should carefully read the lowest point of the water meniscus (the concave liquid surface exhibited by water and aqueous solutions when measured in glass containers). Calculate the density of the solid.



Method II for Solids

Weigh the wooden block you used in Part A on a balance to the nearest 0.01g. Make sure the balance is properly zeroed before weighing. From the mass and previously determined volume, calculate the density of the solid.

Method for Liquids Weigh a 50-mL Erlenmeyer flask. Measure 10 mL of rubbing alcohol (isopropyl alcohol) in a 10-mL graduated cylinder, add the alcohol to the flask, and reweigh.

Repeat a second time. From your data, calculate the average density of the alcohol.

Experiment 1 – Measurements

REPORT FORM

Name _____

Instructor _____

Date _____

A. Mass Measurement

Mass of a penny _____ g _____ mg

Mass of a nickel _____ g _____ mg

Mass of a quarter _____ g _____ mg

B. Volume Measurements

I. Volume of water in graduated cylinder (on the teacher's desk) _____ mL

II. Volume of 20 drops water _____ mL

III. Volume of water delivered by pipet in graduated cylinder _____ mL

IV. Dimensions: L = _____ cm W = _____ cm H = _____ cm Volume = _____ cm^3 (show your calculation)

V. Volume of solid by water displacement method: Type of solid: _____, _____ mL

C. Temperature Measurements

I. Temperature of tap water _____ °C _____ °F (show calculation for conversion of °C to °F) II. Temperature of tap water + ice _____ °C _____ °F

III. Temperature of boiling water _____ °C _____ °F

D. Density Measurements

Solid, Method I

Type of Solid: _____

Mass of solid _____ g

Initial volume of water (V_1) _____ mL

Final volume of water (V_2) _____ mL

Volume of solid (V_2 - V_1) _____ mL

Density of solid (show your calculation) _____ g / mL

Solid, Method II Type of Solid: Wood Block

Mass of wooden block used in Part A: _____ g

Dimensions of solid: L = _____ cm W = _____ cm H = _____ cm

Volume of solid (show your calculation) V = _____ cm^3

Density of solid (show your calculation) D = _____ g / cm^3

Liquid

Mass of empty 50-mL flask _____ g

Mass of flask + 10.0 mL alcohol _____ g

Mass of alcohol _____ g

Density of alcohol _____ g / mL

(show your calculations)

Average density of alcohol (show your calculation) _____ g / mL

Experiment 2: Identification of a Substance by Physical Properties

Objective: To learn the use of physical properties such as solubility, density, boiling point, and melting point in identifying liquids and solids substances.

INTRODUCTION

Every substance has a unique set of properties that allow us to differentiate one from another. These properties can be classified as either **physical properties** or **chemical properties**. Physical properties are those that can be determined or measured without changing the composition or identity of the substance. These properties include color, odor, taste, density, melting point, boiling point, conductivity, and hardness. Chemical properties tell us how a substance interacts with other substances. Such properties include reaction with oxygen (oxidation), chlorine, metals, etc. Determination of chemical properties results in the change of the identity of the substance. Some properties, such as solubility, melting point, boiling point, and density are independent of the amount of substance being examined. These properties are known as **intensive properties** and are used to identify a substance. **Extensive properties** such as mass and volume depend on the amount of substance present and are not useful in the identification of a substance. In this experiment, we will use three properties to identify a liquid substance - solubility, density and boiling point.

Solubility

The **solubility** of a solute (a dissolved substance) in a solvent (the dissolving medium) is the most important chemical principle underlying three major techniques you will study in organic chemistry laboratory crystallization, extraction, and chromatography

The **solubility** of a substance is usually defined as the mass (g) of that substance which will dissolve in a fixed amount of solvent (usually 100g of a liquid) at a given temperature. Depending on its molecular structure, a solute will have different solubilities in different solvents.

For example, sodium chloride (table salt) is an ionic compound. It is soluble in polar solvents such as water, but insoluble in non-polar solvents such as cyclohexane or toluene. Sodium chloride may only be slightly soluble in a solvent that has both polar and non-polar properties. Such a solvent is a weakly polar solvent.

In this experiment, we will use three solvents to compare solubilities: water (polar), cyclohexane (non-polar), and ethanol (weakly polar). The solubilities will be recorded as soluble (completely dissolved), slightly soluble (partially dissolved), or insoluble (not dissolved at all). We will use the following terms: “S” for soluble, “I” for insoluble, and “SLS” or “SS” for slightly soluble.

Part of this experiment will involve determining whether an organic solid compound is **soluble** or **insoluble** in a given solvent. You should keep in mind that this is actually an over simplification since some solids may be **partially soluble** in a given solvent.

If an organic liquid compound dissolves in a solvent, it is sometimes more appropriate to say that the compound and the solvent are **miscible** (mix homogeneously in all proportions). Likewise, if an organic liquid compound is insoluble in the solvent, then they are **immiscible** (do not mix and forms two liquid phases).

The concentration of a solution can be derived by expressing solubility in quantitative terms. An example is the solubility of table salt in water which is 35.9 g/100 cm^3 of water.

Density

Density = mass / volume or D = m / v

Density is defined as mass per unit volume. Mass is usually measured in g and volume in mL or cm^3. Since two substances rarely have the same density, it is a useful physical property in order to identify unknown substances. "Heavy" elements such as lead and gold have high densities while elements that are "light in weight" typically have low densities. For most substances, the variation in density with temperature is negligible. It is usually expressed at 20°C, which is considered to be room temperature. The density of water from 0° to 30°C, is tabulated in Table 1.

The density of a liquid is usually reported in units of grams per milliliter (g/mL). The density of a solid is reported in units of grams per cubic centimeter (g/cm^3). Because 1 mL is equivalent to 1 cm^3, these units are interchangeable. The density of water is 1.00000 g/cm^3 at 4°C and is slightly less at room temperature (0.9970 g/cm^3 at 25°C).

Boiling point

Boiling point is defined as the temperature at which the vapor pressure of a liquid becomes equal to the pressure at the surface of the liquid. The boiling point of a liquid can change if the pressure at the surface of the changes. Since pure substances have a distinct boiling point, boiling points are sometimes used to determine the purity of substances.

A liquid gets converted in to its gaseous state when the temperature of the liquid reaches its boiling point. This is indicated by bubbles of its vapor rising in all parts of the liquid. This is the temperature at which the pressure of the saturated vapor of the liquid is equal to the pressure of the atmosphere under which the liquid boils. Normally, boiling points are determined at standard pressure: 760 mm Hg (torr) or 1 atm.

The boiling point of a liquid is sensitive to changes in atmospheric pressure and varies directly with it. At approximately normal pressure, the boiling point of a liquid will drop about 0.5 C° for every 10-mm Hg drop in pressure. At much lower pressures, close to 10 mmHg, the temperature will drop about 10 C° when the pressure is halved.

Melting point

The **melting point** of a solid is defined as the temperature at which the liquid and solid phases are in equilibrium at a given pressure. At this temperature, a solid is converted to liquid. This is an important property of solids. The melting point of solids, like the boiling point of liquids, is often used for the identification of substances. The **normal melting point** of a solid is defined as the temperature at which the solid and liquid phases of a solid are in equilibrium at a total pressure of 1atm.

Pure crystalline substances have clear, sharply defined melting points. During the melting process, all of the energy added to a substance is consumed as heat of fusion, and the temperature remains constant. A pure substance melts at a precisely defined temperature, characteristic of every crystalline substance and dependent only on pressure.

In contrast to the volume change that accompanies the vaporization of a liquid, the change in volume that takes place upon the melting of a solid is very small. This makes the melting point of a solid, unlike the boiling point of a liquid, practically independent of any ordinary pressure change. The melting point of a solid can be easily and accurately determined with small amounts of material, making this physical property the most used for the identification and characterization of solids.

SAFETY PRECAUTIONS

- The unknown liquids used in this experiment are flammable.
- Although the danger of fire is greatly reduced by the use of small samples, it is not eliminated.
- Keep all liquid samples away from open flames.
- Avoid inhaling vapors from volatile liquids.
- The vapors from the liquids used in this experiment may be irritating and can be toxic if you are exposed to them for long periods of time.
- These problems are minimized by using small amounts of the liquids.
- If you are bothered by the odors of any liquids, work in the fume hood.
- Avoid skin contact with the volatile liquids.

EXPERIMENTAL PROCEDURE

Materials:

Hot plate	Digital balance	Capillary tubes (3)
Thermometer	Beakers (250 mL)	
Watch glass	Graduated Cylinder (10 mL)	
Mel-Temp apparatus	Test tubes (2 small, and 1 large)	
Test tube rack (1)		

I. LIQUID - Identification of a liquid substance utilizing the physical properties of solubility, (miscibility), density and boiling point.

PART A. *Solubility*

1. Take two test tubes and set them in a rack. Add 2 ml of water to the first test tube and 2 ml of ethanol to the second test tube. Add 2 ml of your unknown to each test tube, shake well, and observe whether the two liquids mix completely, partially or not at all.

2. If the two liquids mix completely, it will appear as if only one liquid is present. The two liquids are soluble (miscible) in each other.

3. If the two liquids do not mix at all, indicated by a visible line of separation after shaking and allowing to stand for about a minute. The liquids are insoluble (immiscible).

4. The unknown liquid may be „partially soluble", as judged by the amount remaining separate after shaking as compared to the amount initially added. Record the solubility of your unknown as "s" for soluble, "i" for insoluble, or "sls" for slightly soluble.

PART B. *Density*

1. Place a 10 ml graduated cylinder on the balance and tare.

2. Transfer about 5 ml of your unknown liquid to the 10 ml graduated cylinder.

3. Record the mass and record the exact volume.

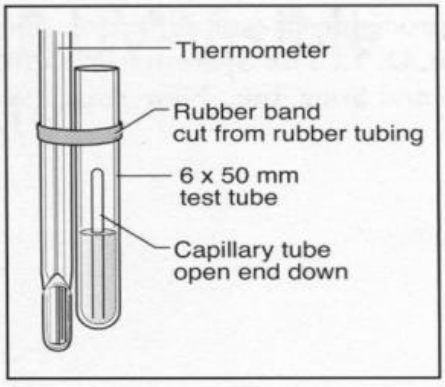
4. Calculate the density of the liquid and record. 5. Save the liquid.

PART C. *Boiling point*

1. To determine the boiling point of your unknown liquid, pour about 5 ml (retained from your density determination above) into a large test tube. (See Fig. below).

2. Add a boiling chip to the liquid.

3. Start a water bath by filling a 250 ml beaker with tap water and place on a hot plate. 4. Using a ring stand and clamp to support the test tube containing the unknown liquid, place a thermometer into the liquid. Make sure that the bulb of the thermometer is submerged into the liquid. Submerge the test tube in the boiling water bath.



5. Heat the unknown liquid until it begins to boil, monitoring the thermometer readings. Record the temperature at which the liquid boils. This will be the point where the liquid is boiling but the temperature reading is stable. Do not allow the liquid to boil to dryness.

II. SOLID - Identification of a solid utilizing the physical properties of solubility in water, solubility in ethanol, density, and melting point.

PART A. Solubility

1. Take two test tubes and set them in a rack. Add 2 ml of water to the first test tube and 2 ml of ethanol to the second test tube.

2. Add a FEW crystals of your unknown solid to each of the test tubes. Shake briefly.

3. Observe whether the crystals dissolve completely, dissolve to some extent, or do not dissolve.

4. Record on your data sheet "s" for soluble, "sls" for slightly soluble, or "i" for insoluble.

PART B. Density

1. Fill your 10 ml graduated cylinder to the 5 ml mark with the liquid in which your unknown was not soluble in Part A.

2. Read the volume to the nearest 0.05 ml. Record this initial volume on your data sheet. 3. Place the 10 ml graduated cylinder + liquid on the balance and tare the balance (re-set to zero).

4. While the graduated cylinder is on the balance, add a few crystals of your unknown to the liquid in the graduated cylinder until the balance reads about 0.5g. Record the mass of your crystals on the data sheet.

5. Making sure that the crystals are completely submerged, read the final volume of the liquid in the cylinder. Record this final volume on your data sheet.

6. To find the volume of your solid, subtract the initial volume of the liquid from the final volume of the liquid.

7. Calculate the density of your solid and record. D = m/v

8. Pour most of the liquid into the sink and dispose of the solid part of the sample in the solid waste container provided by your instructor.

PART C. Melting point

1. Obtain 3 capillary tubes.

2. Place some of your unknown on a watch glass and push some unknown into the open end of the capillary tube until you have about ¼ inch of unknown in the tube. Tap the crystals down to the closed end until they are well packed.

3. Using the Mel-Temp apparatus, set the plateau temperature to 100^0C. Place 2 capillary tubes into the apparatus and press start. (See Fig 2 or Fig. 3 using hot water bath). View your samples through the viewfinder. Record on the data sheet the temperature at the instant the first crystal begins to melt. This is your melting point. To verify the melting point temperature, you may wish to repeat the procedure with your last test tube,



Fig. 2

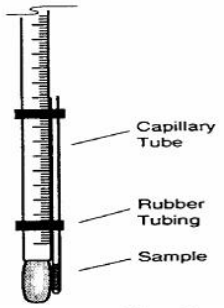


Fig. 3

4. Discard the capillary tubes in the "Glass Disposal" containers. Fig. 2 Fig. 3

Reference Table

Substance	Density (g/ml)	Melting Point °C	Boiling Point °C	Solubility	
				Water	Ethanol
Acetone	0.79	-95	56	s	s
Heptane	0.58	-91	98	i	sls
Isopropyl Alcohol	0.79	-86	83	s	s
Methanol	0.79	-98	65	s	s
Naphthalene	1.15	80	218	i	sls
Potassium Alum	1.76	92	n/a	s	i
Benzil	1.5	95	347	i	s
Benzophenone	1.09	49	306	i	s
Acetamide	1.16	81	222	s	s
1-Propanol	0.8	-127	97	s	s
Cyclohexane	0.78	6.5	81.4	i	s
Benzoic Acid	1.07	1.22	249	i	s

Experiment 2: Identification of a Substance by Physical Properties

REPORT SHEET

Name: _____ Instructor: _____

Date: _____

LIQUID

PART A. Solubility

Liquid: Unknown Number _____

Solubility: Water _____ Ethanol _____

PART B. Density

Weight of the Liquid _____ g Volume of the Liquid _____ml

Density of the Liquid _____ g/ml

Calculation:

PART C. Boiling Point

Boiling Point _____ ^{o}C (range) **Unknown Liquid Identity** _____

SOLID

PART A. Solubility

Solid: Unknown Number _____

Solubility: Water _____ Ethanol _____

PART B. Density

Mass of the solid _____ g

Initial Volume of the Liquid _____ ml Final Volume of the Liquid _____ml
Volume of the Solid Unknown _____ ml Density of the Solid Unknown _____ g/ml
Calculation:

PART C. Melting Point

Melting Point : _____ ^{o}C (range) **Unknown Solid Identity** _____

Experiment 3: Paper Chromatography of Gel Ink Pens

Objective:

- To use paper chromatography to identify whether certain colored inks are pure substances or mixtures.
- To obtain a paper chromatogram of various gel inks
- To identify components of inks by calculating R values

Introduction:

Chromatography is a method of physically separating *mixtures* into its individual components. It is a common laboratory technique used to identify unknown components in mixtures.

There are several types of chromatography; all types employ a *mobile phase* or *eluent* (it can be liquid or gas), which is forced through a *stationary phase* (a solid or semi-solid). Mixtures are separated because some components will be more attracted to the stationary phase (and stick to it) while some components will be more attracted to the mobile phase (and travel with it).

By eye, we cannot know if each ink color is a mixture or pure substance. Using chromatography, the components in a sample will migrate along the filter paper at different rates such that they become spread out and separated from each other. The mobile phase takes advantage of differing solubility or polarity of the components in order to separate them. This component traveling process is called *elution*. Chromatography can be used to separate inks, dyes, pigments in plants, or used to determine the chemical composition of many substances.

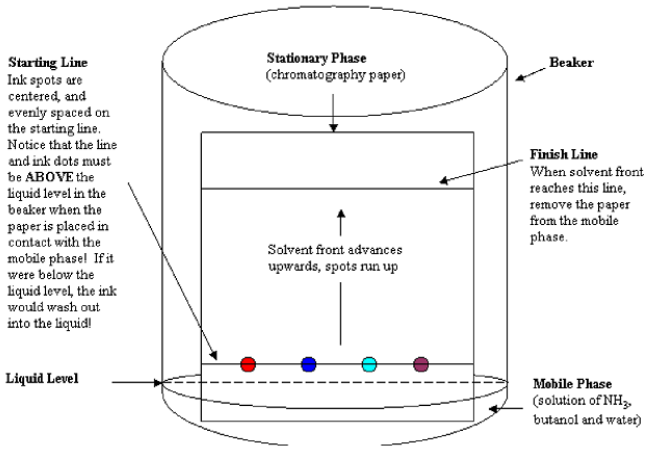
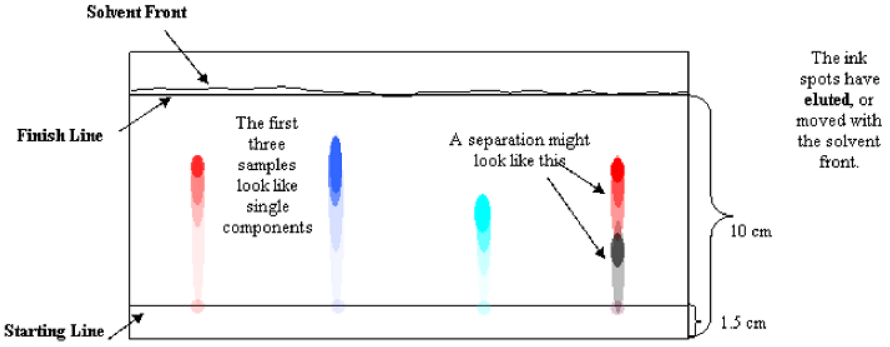


Figure 1 shows a beaker containing mobile phase and a prepared paper stationary phase. A line drawn at the bottom edge of the paper is the *starting line*. The starting line and ink dots *must be above* the level of the mobile phase when the paper is placed inside the beaker. If the starting line is below the liquid level, the inks will wash out into the mobile phase rather than elute up the stationary phase. Another line is drawn about 10 cm above the bottom edge of the paper. This is the *finish line*. Its location was chosen for this experiment because when the eluting solution reaches that line, any inks that are mixtures should be clearly separated.

Note: When the solvent front reaches the finish line, the paper should be removed immediately from contact with the mobile phase.

Figure 2 shows a typical paper chromatogram. There are a few difficulties commonly encountered in the elution process. One problem is that spots tend the spread out as they elute, and can bleed into each other as they proceed up the paper. This can be confusing when interpreting the chromatogram. To avoid this problem, space the spots of sample far apart and make repeated, tiny applications of sample to prevent spreading. Another problem is an uneven solvent front. This can happen if the beaker is nudged – if the mobile phase sloshes inside, the elution trails may travel diagonally, which makes interpretation very difficult. This can also happen if the two edges of the chromatogram are allowed to touch when they are stapled or taped together to form a cylinder.



A component with a given solubility travels along with the mobile phase at one rate, regardless of what other components are present in the sample. If the red part of purple ink travels at the same rate as pure red ink, and both stop in the same place, the two should be the same red ink. The two red spots should have the same *Retention*

Factor, R . The R is the distance, , traveled by the spot divided by the distance traveled by the eluting solution, or *Solvent Front, F* .

Rf = D/F

Comparing the Rf values allows the confirmation of a component in multiple samples because unique components have unique Rf values.

EXPERIMENTAL PROCEDURE

Materials and Equipment

Materials: chromatography paper, gel pens, and eluting solution.

Equipment: 600-mL beaker, pencil, ruler, plastic wrap, tape and paper towels.

Safety: Wear safety goggles at all times. Use eluting solution only in the hood. Do not breathe fumes from the eluting solution. Be sure to handle only the dry part or the chromatogram when removing it from the beaker. Wash hands thoroughly if the eluting solution touches your skin. Personal protective equipment (PPE) required: lab coat, safety goggles, closed-toe shoes, gloves

Part A: Preparation of Chromatography Paper

1. Wash your hands thoroughly to remove excess oils from your skin. Obtain a ruler and a piece of chromatography paper from your instructor. Handle the paper only on the edges to avoid leaving fingerprints, as these may hinder the elution process.
2. Place the chromatography paper on a sheet of clean notebook paper or paper towel to avoid picking up dirt or contaminants from the bench top. Orient the paper into a "landscape" position and write your name on the top edge of the paper in one corner. Using a pencil and ruler to measure accurately, draw a straight line across the paper, about 1.5 cm above the bottom edge. This is the **starting line**. Draw another line about 10 cm above the bottom edge. This is the **finish line**.
3. On the starting line, measure in from one side about 2.5 cm and lightly draw a small "X" centered on the starting line. Draw seven more, 1.5 cm apart.
4. In the center of each X, make a small spot of ink color in this order: **black, burgundy, red, pink, violet, turquoise, green, blue** When you have finished, you should have something that looks like Figure 3.

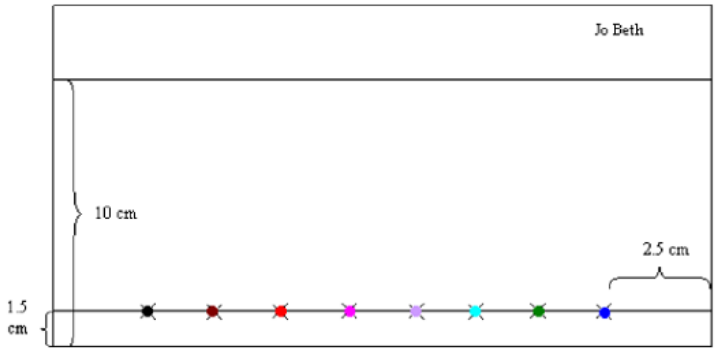


Figure 3: Prepared Chromatography Paper

5. Go back over each ink spot a second time to ensure there is enough ink in the spot.
6. Obtain a small piece of tape and gently curl the paper into a cylinder, with the spots on the outside. Tape the ends together near the top and bottom, taking care that the two edges of the paper do not touch. If they do touch, the eluent will creep on a diagonal, and the spots will run together, or not in straight lines.

Part B: Acquisition of Chromatogram

1. Take a 500-mL beaker and pour about 25-mL of eluting solution into the beaker. Obtain a piece of plastic wrap to cover the top.
2. Gently place the paper cylinder into the beaker and cover the top with the plastic wrap. Remember that the spots must be above the liquid level for the experiment to work. Watch the eluent creep up the paper until it begins to move some of the ink. It will take about 45-90 minutes for the solvent front to reach the finish line.
3. When the solvent front reaches the finish line, remove the paper from the beaker, being careful to touch only the top. Let excess eluent drip into the beaker. Gently remove the tape and lay the chromatogram on a piece of paper towel in the hood. Leave the paper in the fume hood, where it will dry completely. If needed, use a heat lamp (in the fume hood) to dry the chromatogram; if using the heat lamp, allow 5-10 minutes to dry.

Part C: Interpretation of Chromatogram

1. Write the names of the original ink colors beneath each "X" mark (from the order you added the colors). Each ink sample should no longer be on the "X" mark, and have travelled

up the paper, becoming one or more separate color spots between the starting and finish lines. Circle around each color spot.

2. Use a ruler and draw a plus sign in the center of each spot. Measure the distance from the starting line to each plus sign. Record this distance for each spot on your lab report. These are the values, in cm.

3. Measure the distance between the starting line and the finish line or, the farthest up that the solvent front reached. Record this distance. This is the value, in cm.

4. Calculate the retention factor (R) for each spot and record the values in your lab report.
5. You and your lab partner will hand in your lab reports at the same time, with the paper chromatogram stapled to the lab reports.

Experiment 3: Paper Chromatography of Gel Ink Pens

REPORT SHEET

Name: _____ Instructor: _____

Date: _____

Experimental Data

F value (the distance traveled by the eluting solution):

Record D values for each eluted spot to the nearest 0.1 cm. Draw an X through any unused boxes.

Ink color	Distance(s) traveled by Eluted Spot(s), D		

Calculate and record the R_f value for each eluted spot, using Equation given previously:

Ink color	R_f value for Eluted Spot		

Show calculations for R_f below for the purple marker:

Data Analysis

1. This lab employs chromatography to separate the components in ink. What other applications can we use chromatography for?

2. Are any of the R values in the table above the same (or similar, within 0.1)?

What would the same or similar R_f values indicate?

3. What are the units for R_f values?

4. Record ink colors in the appropriate column; for mixtures, list the colors of the components.

Mixtures/Component Colors	**Single-Component Colors**

5. Which color travelled farthest with the mobile phase?

What does this indicate about that component's attraction:

- to the mobile phase?

- To the stationary phase?

6. Which of the materials tested (gel ink pen and colored markers) had an R_f value of zero?

What does this indicate about that component's attraction

- to the mobile phase?

- To the stationary phase?

7. Why is it necessary to use a pencil to mark the lines and X's on the paper?

Experiment 4 – Separation of a Mixture

I. Introduction:

The physical separation of the components of a mixture is the goal of this experiment. Each component in a mixture retains its individual physical properties. That is, they do not react with each other (a chemical change) in the mixture or during the separation process. Product mixtures are a common result in chemical reactions. Indeed, it is rare that a reaction results in a single, pure product directly. The real challenge often is the separation of the desired product from the mixture and its subsequent purification. You will rely on differences in properties to separate silicon dioxide (SiO_2, sand), sodium chloride (NaCl, table salt), and ammonium chloride (NH_4Cl), from a mixture of these solid compounds. Three commonly used methods of separation are given below:

(a) SUBLIMATION

Sublimation is the process by which a solid change from the solid to the gaseous state directly without forming a liquid. *Melting* is a process by which a solid change to a liquid by heating. In the mixture used in this experiment, one compound, ammonium chloride sublimes easily, while the other two components do not.

(b) SOLUBILITY

The extent to which a substance is soluble in a solvent depends upon the chemical structure of both the substance and the solvent. In general, polar compounds, such as sugar and alcohol, and ionic compounds, such as KCl, NaCl, NH_4Cl, and NH_4NO_3, are soluble in polar solvents such as water. Nonpolar substances such as grease, wax, and oil, are soluble in nonpolar solvents such as toluene or kerosene. Extracting (dissolving) a soluble substance out of a mixture with a appropriate solvent is a common separation technique. In this experiment you will use solubility to extract a solid, NaCl, which is soluble in water, from another solid, SiO_2, which is insoluble in water.

(c) DISTILLATION

If two components have very different boiling points, the substance with the lower boiling point will evaporate more rapidly at a given temperature than the substance with a higher boiling point, and so they can be separated on this basis. Solid NaCl with a very high boiling point can be separated from a solution ($NaCl–H_2O$ solution) by simply evaporating the water, which has a much lower boiling point. The solid NaCl will remain in the dish as a dry residue. If the water vapor is condensed to a liquid and is collected, the process is called *distillation*.

The separation techniques of sublimation, extraction, and evaporation usually do not change the chemical composition of a substance, and as such, are standard separation techniques used by chemists.

II. Experimental Procedure

Part I

Obtain a sample of "unknown mixture" from your instructor and record its identity number or letter on the report form. Weigh a clean, dry evaporating dish to the nearest 0.01 g. Record the

mass on the report form. Measure approximately 2 g of your mixture by carefully adding it directly to the evaporating dish while it is on the balance. The mass of sample does not have to be exactly two grams, but you should measure the mass to within +/– 0.01 g.

Part II

Heat the evaporating dish and its contents on an electric hot plate in the fume hood. Ammonium chloride, NH_4Cl, will sublime from the mixture and appear as a white smoke. Continue heating until the white smoke is no longer produced. At this point, you can stir the hot mixture with a glass stirring rod to ensure complete sublimation of the ammonium chloride. Allow the evaporating dish and its contents to cool to room temperature and weigh them together to the nearest 0.01 g. Record this mass on the report form. From the differences in masses before and after heating, determine the mass of NH_4Cl and calculate it's percent by mass.

Percent by mass of "Z" = [mass of Z/ total mass of the sample] X 100

Part III

Add about 20 mL of deionized water to the residue in the evaporating dish to dissolve the NaCl. Stir the mixture well for a few minutes with a glass stirring rod to dissolve the NaCl completely. Next, *decant* the water (now a solution of dissolved NaCl) into a second, pre-weighed evaporating dish, leaving the undissolved solid (sand, SiO_2) behind in the original evaporating dish. Add about 10 mL of deionized water to the wet sand, stir well with the stirring rod as before, and decant the liquid into the second evaporating dish, which will now contain about 30 mL of water with dissolved NaCl. Set this dish and solution aside for Part IV.

Decantation:

Heat the evaporating dish containing the wet sand on the hot plate to dry the sand. Use *crucible* tongs (not *beaker* tongs!) to quickly lift the dish from the plate when needed, as the last remainder of water can evaporate suddenly and cause the sand to "popcorn" out of the dish like popping popcorn! Alternately, you can cover the dish with a watch glass (concave side downward) to prevent any sand from spattering out, but some sand may stick to the watch glass. Allow the evaporating dish and dry sand to cool to room temperature, and if necessary, carefully scrape any sand sticking to the watch glass back into the dish. Weigh the dish and sand to the nearest 0.01 g and record this mass on the report form. Determine the mass of SiO_2 and calculate its percent by mass in the sample.

Part IV

Heat the second evaporating dish containing the salt water solution on the hot plate. You should not heat so strongly that the solution splashes out of the container when it is boiling, but it is not necessary to cover the dish with a watch glass, as this will inhibit the evaporation of the water. The goal is to evaporate the water completely, leaving a residue of dry NaCl in the dish. However, when the salt is nearly dry, it has a strong tendency to "popcorn" (see note above), causing loss of NaCl to occur. There are two ways to prevent this. You can use your crucible tongs to lift the dish off and on the hot plate to control the rate of evaporation of the last of the water, or, you can place a watch glass on the evaporating dish and let any spattering salt stick to the underside of the watch glass. Either way, after the salt appears dry and no more spattering is occurring, continue heating the open dish, and the watch glass also if it has any salt adhering to it, for about 10 minutes. This extra heating is done to ensure that the salt is completely dry. Allow the evaporating dish and dry NaCl, and the watch glass if it was used, to

cool to room temperature. Carefully scrape any salt adhering to the dry watch glass back into the dish. Weigh the dish and salt to the nearest 0.01 g and record this mass on the report form. Calculate the mass of NaCl and its percent by mass in the sample.

Part V Add up the masses of NH_4Cl, SiO_2, and NaCl from Parts II, III, and IV, and record this total mass on the report form. From this, calculate the total percentage of mixture recovered in this experiment.

Percent Recovery = [(mass of NH_4Cl + mass of SiO_2 + mass of NaCl)/ original mass of sample] X 100

Ideally, your percent recovery should be in the neighborhood of 100 percent. If your percentage recovery is less than 100 percent, *or more* than 100 percent, give an explanation for the error on the report sheet.

Experiment 4 – Separation of a Mixture

REPORT FORM

Name _____

Instructor _____

Date _____

Part I

1) Sample Number _____

2) Mass of empty evaporating dish _____ g

3) Mass of dish + mixture _____ g

4) Mass of mixture _____ g

Part II

5) Mass of dish + sand + salt (after subliming the NH_4Cl) _____ g

6) Mass of ammonium chloride [(3) – (5)] _____ g

7) Percentage of NH_4Cl _____ %

(show your calculation)

Part III

8) Mass of dish + dry SiO_2 (after heating) _____ g

9) Mass of dry SiO_2 [(8) – (2)] _____ g

10) Percentage of SiO_2 _____ %

(show your calculation)

Part IV

11) Mass of empty second evaporating dish _____ g

12) Mass of evaporation dish + dry NaCl (after heating) _____ g

13) Mass of dry NaCl [(12) – (11)] _____ g

14) Percentage of NaCl _____ %

(show your calculation)

Part V

15) Total mass of NH_4Cl, SiO_2, and NaCl _____ g

16) Percent recovery _____ %

Explanation:

Experiment 5: Determination of chemical formula

Object: Object of the experiment is to determine the formula of the compound with the general formula CuxCly.zH2O, where x, y and z are integers which, when known, establish the formula of the compound

I. Introduction

In order to determine the formula of a compound we should find the mass of each element in weighed sample. When the mass of each element is known, we can determine the formula of the compound as the atom ratio in a compound is equal to the mole ratio. In this experiment the formula of compound with the general formula CuxCly.zH2O will be determined.

Importance:

By performing this experiment, it is straightforward to understand how the formula of an unknown compound can be determined experimentally. This method can be useful in determining the formulae of compounds of transition metals, which can show various Oxidation states.

II. Procedure:

Determining the mass of the water: A clean and dry crucible is weighed on an analytical balance. The small amount of the unknown hydrated copper is placed in the crucible and accurately weighed. The measurements are written on the Data page.

The sample is heated on the hot plate until the blue-green crystals tun to brown. When all the Crystals became brown, the sample heated for additional two minutes. Then the sample is set aside to let it cool with a cover to prevent it from rehydration.

Determining the mass of the copper:

After 15 minutes the brown crystals are transferred to an empty 50-mL beaker by rinsing it out the crucible with two 5- to 7-mL portions of distilled water. The beaker is swirled gently until the brown solid dissolved completely. The measured 20 cm of 20-gauge aluminum wire is formed into a loose spiral coil and placed into the solution (completely immersed), The evolution of hydrogen gas took place. The copper metal formed on the Al wire. After the completion of the reaction (after about 30 minutes), the solution became colorless. The 5 drops of 6 M HCI is added to dissolve any insoluble aluminum salts. The copper is removed from wire by using glass stirring. The formed copper metal is separated from solution by using weighed filter paper. The filter paper is placed on the funnel and solution is decanted into the funnel. All the copper is transferred to the funnel. While the suction is turned off, 10 mL of 95% ethanol is added. The suction was turned on for additional 5 minutes. The paper is lifted with a spatula and weighed accurately.

III. Results, Discussions and conclusions

Results:

	Copper	Chlorine	Water
Mass(g)	0.3854	0.4485	0.2133
Moles, [mol]	0.06	0.013	0.012

Mole ratio (x: copper)	-	2:1	2:1

Calculations:

Mole (copper) =0.3854 g / 63.546 g mol= 0.06 mol

Mole (chlorine) =0.4485 g/35.453 g mol'=0.013 mol

Mole (water) = 0.2133 g/ 18.01528 g mol'= 0.012 mol

Formula of dehydrated sample (round to nearest integer): CuCl2

Formula of hydrated sample: CuCl2. 2H20

Discussions:

The compound studied in the experiment was an ideal option for learning the way of Discussions:

The compound studied in the experiment was an ideal option for learning the way of determining the formula of a substance. The change in color (from blue-green to brown) indicated that the sample became dehydrated, which is convenient for determining the mass of the water by difference.

The masses determined during the experiment could be inaccurate as some copper metal could stay on the walls of the beaker. In order to prevent these inaccuracies in mass values the beaker should be drained thoroughly with water.

Conclusions:

The experiment has revealed that the unknown substance's formula is CuCl2. 2H20. From the experiment I have learnt that by determining the mass of each element the mole ratio can be calculated and the formula of a compound can be determined. The aim of the experiment is met as the formula of a sample of hydrated copper chloride is determined.

Experiment 5 – Determination of Chemical formula

REPORT FORM

Name _____

Instructor _____

Date _____

Experiment

Data and Calculations: Determination of a Chemical Formula

Atomic masses: Copper_____Cl: _____, H: _____O: _____

Mass of crucible _____g

Mass of crucible and hydrated sample _____g

Mass of hydrated sample _____g

Mass of crucible and dehydrated sample: _____g

Mass of dehydrated sample _____g

Mass of filter paper _____g

Mass of filter paper and copper: _____g

Mass of copper: _____g

No. moles of copper _____moles

Mass of water evolved: _____g

No. moles of water: _____moles

Mass of chlorine in sample (by difference): _____ g

No, moles of chlorine: _____moles

Mole ratio, chlorine: copper in sample: _____:1

Mole ratio, water: copper in hydrated sample: _____:1

Formula of dehydrated sample (round to nearest integer): _____

Formula of hydrated sample: _____

Experiment 6: Gravimetric Analysis

Objectives:

- To experimentally analyze an unknown sulfate salt via a precipitation reaction, using the techniques associated with Gravimetric Analysis to collect and weigh the precipitate, and
- To calculate the percentage by mass of in the unknown sulfate salt via a stoichiometric analysis of the collected precipitate, and then use this percentage to identify the metal "M" present in the sulfate salt.

I. Introduction:

Gravimetric analysis is a quantitative method for accurately determining the amount of a substance by selective precipitation of the substance from an aqueous solution. The precipitate is separated from the remaining aqueous solution by filtration and is then weighed. Assuming that the chemical formula for the precipitate is known and that the precipitation reaction goes all the way to completion, then the mass of the substance in the original sample can be determined.

In this experiment, the percentage by mass of sulfate in an unknown sulfate salt will be determined by gravimetric analysis. First, a pre-weighed sample of the unknown sulfate salt will be dissolved in water. Next, an excess of aqueous barium chloride is added to the aqueous solution of the unknown salt. This will result in the precipitation of all the sulfate ions as barium sulfate:

Metal sulfate (aq) + Barium chloride (aq)⟶ Barium sulfate (s) + Metal chloride (aq)

The barium sulfate precipitate is collected by filtration, dried and weighed. Since barium chloride is added in excess, and since the precipitation reaction goes to completion, we can assume that *all of the sulfate* is transferred from the original unknown sample to the precipitate. The mass of sulfate in the collected precipitate can be calculated via its percent composition. This also yields the mass of sulfate in the original unknown since:

Finally, using the mass of sulfate along with the initial mass of unknown used, the percentage by mass of sulfate in the original sample may now be calculated.
In order to obtain the best results, the collected crystals should be as large as possible. This considerably aids the filtration process (larger crystals are less likely to be pass through the filter paper), and it also minimizes the amount of impurities adsorbed onto the crystals (smaller surface area). In general, larger crystals are obtained when the rate of precipitation is as low as possible. The rate of precipitation is minimized by slowly adding the solution to the aqueous mixture containing the unknown salt while continuously stirring the mixture. The rate of precipitation can be decreased even further by slightly increasing the solubility of the. This may be achieved by lowering the pH with 6 M and by increasing the temperature. The resulting decrease in the yield of the is insignificant.

II. Procedure

Chemicals
Unknown solid sulfate salt, 6 M HCl solution, and 0.1 M $BaCl_2$ solution

Equipment
250-mL beaker, analytical balance, stirring rod, 100-mL graduated cylinder, stand with ring clamp, wire screen, Bunsen burner, wash bottle with distilled water, crucible and lid, crucible tongs, ash-less filter paper, large funnel, 500-mL Erlenmeyer flask, clay triangle

Safety: Be very careful when handling 6 M (*aq*). If this acid comes in contact with your skin or eyes you should immediately rinse the affected area with water for several minutes. Also remember that items heated in the Bunsen burner are very hot (especially the crucible), and to allow ample time for them to cool before touching.

Instructions
1. Weigh a clean, dry 250-mL beaker to the nearest 0.001 g using the analytical balance, and record this mass on your lab report. Next, add 0.30 – 0.35 grams of your unknown sample to the beaker. Record the combined mass of the beaker plus sample on your lab report.
2. Add 50 mL of distilled water, followed by 20 drops of 6 M HCl(*aq*), to the sample in the beaker. Stir the contents of the beaker until the sample has entirely dissolved. **Leave the stirring rod in the beaker.**

3. Obtain a stand with a ring clamp from the back of the lab. Place your wire screen on the ring and the beaker containing your dissolved sample on the wire screen. Use the Bunsen burner to heat the solution until it is nearly (but not quite) boiling. Turn the Bunsen burner off before the solution boils.

4. While heating the solution, measure out 25 mL of 0.1 M $BaCl_2$(*aq*) using a 100-mL graduated cylinder. The graduated cylinder used should be clean (rinse with distilled water) but does not need to be dry.

5. Slowly add small portions of $BaCl_2$(*aq*) (*aq*) to the beaker containing the hot solution. You should observe the formation of a white precipitate of $BaSO_4$(*s*). Stir the contents of the beaker as you add the solution. **The addition of the** $BaCl_2$ **must be performed very slowly – this step should take you at least 3 minutes to complete!** When finished, rinse any precipitate that remains on the stirring rod into the solution with a small amount of distilled water, and then allow the precipitate to settle in the beaker for about 20 minutes.

6. While the precipitate settles, prepare your crucible by heating it in the hottest part of the Bunsen burner flame for about 2 minutes (use the crucible tongs, cradling the crucible as demonstrated by the instructor – *never pick up a crucible by pinching the walls*). Repeat with the lid. Place the hot crucible and lid on the metal base of the stand to cool. Once they have cooled to room temperature weigh the crucible **without the lid** using the analytical balance, and record this mass on your lab report. You do not need to weigh the lid.

7. Obtain a piece of ash-less filter paper from your instructor and fold it into quarters. Open the folded paper into a cone and place it into your large funnel. Wet the filter paper with a small amount of distilled water so that it adheres to the funnel. Sit the funnel in the mouth of a 500-mL Erlenmeyer flask, which will be used to collect the filtrate.

8. After 20 minutes has passed, slowly pour the mixture containing the precipitate down your stirring rod into the funnel. Be careful that the level of liquid in the funnel is never more than three-fourths of the way to the top of the filter paper. When the transfer is complete use your wash bottle (filled with distilled water) to rinse the residual precipitate from the beaker and the stirring rod into the funnel.

9. After all the liquid has drained from the funnel, wearing gloves **very carefully** press the top edges of the filter paper together, and gently fold the filter paper into a compact package that will fit into the crucible. It is important that you do not use too much force in order to avoid tearing the filter paper. Place the folded filter paper into the crucible.
10. Take your stand, ring clamp and Bunsen burner over to a **fume hood.** Place your clay triangle on the ring and the crucible in the clay triangle for support. *Gently heat the crucible without the lid to remove the water*. Once the paper appears to be dry (after several minutes), *heat the crucible more vigorously so that the filter paper begins to char* (turning from white, to brown, to black) – but not so vigorously that the filter paper bursts into flame. If the filter paper bursts into flame, you should cover it with the crucible lid to put out the flame, then reduce the amount of heat and remove the lid. Continue to heat moderately with the lid off until all of the filter paper has turned black.

11. Once all the filter paper has turned black, *vigorously heat the crucible without the lid* in the hottest part of the Bunsen burner flame so that the bottom of the crucible is **red hot**. The center of the flame should now be directly on the crucible. The charred filter paper (carbon) will gradually combust and be converted into gas. When the filter paper is entirely combusted only the white should remain in the crucible. Continue to heat the crucible vigorously until no charred filter paper remains. This should take 5 - 10 minutes.

12. Allow the crucible to cool to room temperature (this takes at least 5 minutes). Weigh the crucible **without the lid** and its contents on the analytical balance. Record this mass.
13. Place the crucible and its contents back in the clay triangle and heat vigorously with the lid off for an additional 5 minutes. Then allow it to cool again and reweigh the crucible and its contents **without the lid**. If the mass is within 0.005 grams of that in step

12, then record this mass on your lab report. If the mass has decreased by more that 0.005 grams, then either the is still wet or not all of the filter paper has combusted and you should repeat this step until you achieve a consistent mass, making sure the crucible is in the hottest part of the flame.

14. Discard the in the proper waste container, then clean up as directed by your instructor.

Experiment 6 – Gravimetric Analysis

REPORT FORM

Name _____

Instructor _____

Date _____

Experimental Data

Unknown Sulfate ID Code:

Mass of empty 250-mL beaker	
Mass of empty 250-mL beaker + unknown sulfate	
Mass of Unknown sulfate	
Mass of empty crucible(without lid)	
Mass of empty crucible(without lid) and barium sulfate	
Mass of Barium sulfate	

Calculations and Conclusions

1. Calculate the mass of sulfate in the unknown sample. Clearly show each step of your calculation.

2. Calculate the percentage by mass of sulfate in the unknown sample. Show your work.

3. Calculate the percentage by mass of the metal in the unknown sample. Show your work.

4. The cation in your unknown metal sulfate is one of the following:
   Al^{3+} ; Na^{+} ; Ni^{2+} ; K^{+} ; NH^{4+} ; Cd^{2+}

Use this information along with your experimental results to determine which cation it is. Unknown Sulfate ID Code:
Identity of Cation:
Show all of your work with clear, logical steps below. Clearly explain how your calculations here along with your experimental results for #2 and/or #3 allowed you to identify the cation in the metal sulfate.

Experiment 7: Heat of Neutralization

Objectives: To calculate enthalpy change of a reaction by using calorimeter and understand the difference between endothermic and exothermic reactions.

I. Introduction

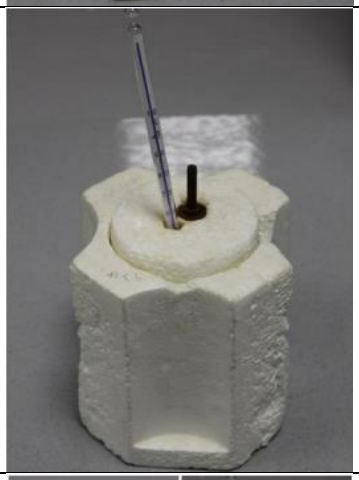
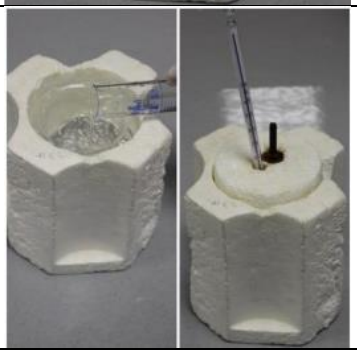
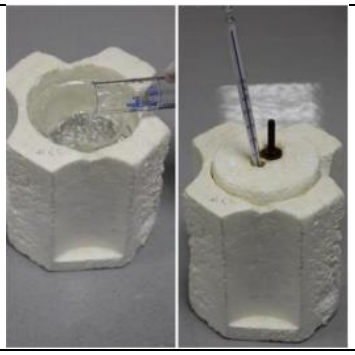
In chemical reactions, energy change is observed. This energy change is usually in the form of heat and at constant pressure it is defined as **heat of reaction** or **enthalpy change (ΔH).** To form 1 mole of compound from its constituent elements, necessary amount of enthalpy change occurs and this change is defined as **enthalpy of formation**. If heat is released during the reaction, ΔH is shown with negative sign and the reaction is called **exothermic reaction.** If heat is absorbed during the reaction, ΔH is shown with positive sign and the reaction is called **endothermic reaction.** Direct measurement of enthalpies of formation is difficult experimentally, so indirect methods involving enthalpies of reaction are used. Hess's Law states that the change in a thermodynamic property such as enthalpy depends on the initial and final states and is independent of path followed. An example for Hess's Law is given below. N_2 (g) + H_2 → NH_3 (g) $\Delta H_{formation}$ (NH_3(g)) = ΔH_1 NH_3 (g) → NH_3 (aq) $\Delta H_{dissolving}$ = ΔH_2 Assume that ΔH_1 and ΔH_2 are known. If first and second reactions are added, net reaction becomes; N_2 (g) + H_2 → NH_3 (g) $\Delta H_{formation}$ (NH_3(aq)) = $\Delta H_1 + \Delta H_2$ which is also formation reaction of NH_3(aq). Given that ΔH_1 = - 45.8 kJ/mol and ΔH_2 = - 35.4 kJ/mol, we can calculate the ΔH formation of NH_3(aq) as -81.2 kJ/mol. The heat is measured experimentally by allowing the reaction to take place in a thermally insulated vessel called as **calorimeter**. If the calorimeter is perfectly insulated, no heat change occurs between system and surrounding and the system is defined as adiabatic (Q=0). Consequently, at constant pressure, ΔH_{system} is also equal to zero. The formulation of enthalpy changes of the system, ΔH_{system}, is shown as in Equation (1). ΔH_{system} = ΔT (heat capacity of calorimeter + heat capacity of contents) (1) For endothermic reaction in adiabatic system, Equation (1) can be written as below. $\Delta H_{system} = n\Delta H_{reaction} + C_p\Delta T$

$0 = n\Delta H_{reaction} + C_p\Delta T$; $n\Delta H_{reaction} = - C_p\Delta T$

In a similar manner, for exothermic reaction in an adiabatic system, Equation (1) can be simplified as: $\Delta H_{system} = -n\Delta H_{reaction} + C_p\Delta T$ $0 = -n\Delta H_{reaction} + C_p\Delta T$ $n\Delta H_{reaction} = C_p\Delta T$ In this experiment, you will determine the heat of formation of various ammonium salts NH_4X(s) where X is Cl, NO_3 or SO_4 by combining measurements of the heat for the neutralization reaction; NH_3(aq) + HX(s) → NH_4X(aq) ΔH_{neut} And the heat of the dissolution reaction; NH_4X(s) + H_2O → NH_4X(aq) ΔH_{diss} with known heats of formation of NH_3(aq) and HX(aq).

II.Materials Nitric acid (HNO_3); 250 mL beaker; Graduated cylinder; Ammonia (NH_3); Thermometers; Ammonium nitrate (NH_4NO_3); Styrofoam cups

III.Procedure

PART A: Heat of Neutralization	
1. Obtain a sytrofoam cup. In the first cup, place 50 mL of 1.5 M NH_3	
2. Place a thermometer in the cup containing the NH_3 and record temperature at 30 seconds intervals	
4. Add the acid solution to the NH_3 and swirl to mix. Continue taking temperature data at 30 seconds intervals while swirling the solution occasionally	
PART B: Dissolving	
1. Place a volume of distilled water equal to the final volume of solution from part (A) in a Styrofoam cup and record temperature data at 30 seconds intervals	

2. Weigh out that mass of NH_4NO_3 salt into a clean, dry beaker	
3. Immediately, add the weighed amount of salt, swirl to dissolve (use stirring rod if necessary), and continue taking temperature data at 30 seconds intervals	

Calculations

1. Plot temperature versus time graph using your data and determine ΔH_{neut} for (a) and ΔH_{diss} for (b).

2. Take ΔH_f of 1.5 M NH_3 as -81.2 kJ/mol and ΔH_f of 1.5 M HCl as -165.1 kJ/mol, calculate the ΔH_f of $NH_4Cl(s)$.

3. Do the same calculations for $NH_4NO_3(s)$ using -206.0 kJ/mol for the ΔH_f of 1.5 M HNO_3.

4. Calculate ΔH_f of $(NH_4)_2SO_4$ using -884.2 kJ/mol for the ΔH_f of 1.5 M H_2SO_4. [Note that all ΔH_f are per mol (not per 1.5 mol)]

Experiment 7: Heat of Neutralization

REPORT FORM

Name _____

Instructor _____

Date _____

Prelaboratory Work

1. Volume of 1.5 M HNO_3 solution:

2. Mass of NH_4NO_3:

Time (s)	Temperature of NH_3 solution in ◦C before adding 1.5 M HNO_3	Temperature of distilled water in ◦C before NH_4NO_3
0		
30		
60		
90		
120		
150		
180		
Time (s)	Temperature of NH_3 + 1.5 M HNO_3 solution in ◦C	Temperature of NH_4NO_3 solution in ◦C
0		
30		
60		
90		
120		
150		
180		

Results

1. Calculate $\Delta H_{neutralization}$:

2. Calculate $\Delta H_{dissociation}$:

3. Calculate $\Delta H_{formation}$ of NH_4NO_3:

Experiment 8: Reactions in Aqueous Solutions: Metathesis Reactions and Net Ionic Equations

Objectives

- Careful observation and detailed description of chemical reactions in solution
- Inferring from observation and from other information whether or not a reaction actually takes place
- Writing the reactions that take place in the shorthand language of chemistry: chemical equations (molecular, complete ionic, and net ionic)

Predicting the Products of Metathesis Reactions

Products of metathesis ("double replacement") reactions are obtained by interchanging the ions produced by dissociation of the reactants.

$AB + CD \longrightarrow AD + CB$

$AgNO_3 + NaI \longrightarrow AgI + NaNO_3$

Example: Predict reaction products given reactants Na_2SO_4 and $AlCl_3$:

1. Identify reactant ions

   $2\ Na^+ + SO_4^{2-}$ and $Al^{3+} + 3\ Cl^-$

2. Interchange the ions: $Na^+ + Cl^-$ and $Al^{3+} + SO_4^{2-}$
3. Write correct formulas for the products (i.e., form the ions into compounds): $NaCl$ from $Na^+ + Cl^-$

   $Al_2(SO_4)_3$ from $Al^{3+} + SO_4^{2-}$

4. Write the complete **molecular equation** for the reaction

   $Na_2SO_4 + AlCl_3 \longrightarrow NaCl + Al_2(SO_4)_3$

5. In order to write a **complete ionic equation** for the reaction, write all soluble reactants and products as separated ions, for that is how they exist in solution: $Na^+ + SO_4^{2-} + Al^{3+} + Cl^- \longrightarrow Na^+ + Cl^- + Al_2(SO_4)_3$
6. Note: in general, all of the reactants are soluble but not all of the products if an overall reaction occurred
   Note: Don't worry about balancing the equation, but get the formulas of the species involved right.
7. In order to write a **net ionic equation**, remove the spectator ions from the complete ionic equation. The spectator ions are the free ions that are unchanged in the reaction, so they appear on both sides of the complete ionic equation. $SO_4^{2-} +$ Al^{3+} --> $Al_2(SO_4)_3$
   Note: the net ionic equation involves only the product of the reaction that is the driving force of the reaction (see below), and the reactant ions that lead directly to that product.

The Net Chemical Reaction

Observation of one of the following is evidence for a net chemical reaction:

- Heat is evolved
- A precipitate forms (i.e., one product is insoluble in water)
- A weak electrolyte is produced (most important example, H_2O)
- A gas is evolved

These may be understood in terms of the "driving force" of a chemical reaction. That is, the rearrangement of ions in a double replacement reaction is likely to occur if the rearrangement leads to products that are energetically favored over the reactants; otherwise, no rearrangement is likely, and no reaction occurs. Formation of precipitates or gases is a sign that new bonds formed from the free ions in solution, resulting in a more stable product that comes out of solution. Evolution of heat is also a sign that the product (whatever it is) is lower in energy (more stable) than the reactants. Thus, we can infer that a reaction occurs **if rearrangement of the ions produces one of the above observable results**.

List of Chemicals

- *0.1 M ammonium chloride
- *0.2 M ammonium oxalate
- *0.1 M barium chloride
- *0.1 M calcium chloride
- solid copper
- *0.1 M copper (II) nitrate
- deionized (DI) water
- *1.0M hydrochloric acid
- **6.0 M hydrochloric acid
- *0.5 M silver nitrate
- *1.0 M sodium hydroxide
- **6 M sodium hydroxide
- *0.1 M sodium sulfate
- solid zinc

List of Equipment and Glassware

- one glass stirring rod
- one large beaker labeled as waste
- one test tube rack (if plastic, do not place a very hot test tube in it)
- test tubes
- one Bunsen burner
- one splint (a thin piece of wood)

• one test tube holder

• one medium size beaker (100-mL or 250-mL)

Experimental Procedure

1. Be sure to carefully read the label on each chemical bottle.

2. Use 13 mm x 100 mm test tubes unless stated otherwise. Wash and rinse each test tube with deionized water. Dry the outside. These can be used wet, just shake to remove excess water.
3. When approximate volumes are needed, use the dropper on the bottles: 20 drops are approximately 1 mL.

4. Pour one reactant into the other - the order does not matter unless stated otherwise.
5. Mix all reactions thoroughly with a glass rod. After using the glass rod, rinse it with deionized water. Collect rinses in your waste beaker. No need to dry it before reusing.
6. Record all observations. Some observations to include (if applicable) are:

• physical description: color, phase, hardness, crystallinity, shininess

• temperature change (touch the test tubes before mixing and again after mixing;

DO NOT TOUCH A TEST TUBE AFTER HEATING IT IN THE BUNSEN BURNER

• gas (bubbling or effervescence is evidence of gas formation)

• odor

• solid (observed as a precipitate – bottom of test tube, or cloudiness –suspended in the solution)

• any other observations you notice

7. Did a reaction occur? If so, write yes in the table, and classify the reaction based on the Reaction Types listed earlier in this experiment. If there is no reaction, then simply write no, and write a dash in the reaction type. Then move on.

8. If a chemical reaction occurs, write the molecular, ionic, and net ionic equations. Write these before moving on to the next reaction, unless stated otherwise. There is no need to try to write an equation if a reaction did not occur.

Experiment 8: Reactions in Aqueous Solutions: Metathesis Reactions and Net Ionic Equations (REPORT FORM)

Name _____ **Section** _____

Instructor _____

Date _____

1. 6 M hydrochloric acid + zinc granules. Perform splint test.

Observations	
Reactants before combining	After combining and mixing
Reacted?	Reaction type:

ME:_____
IE:_____
NIE: _____

2. 0.2 M ammonium oxalate (the oxalate ion is $C_2O_4^{2-}$) + 0.1 M calcium chloride.

Observations	
Reactants before combining	After combining and mixing
Reacted?	Reaction type:

ME: _____

IE: _____

NIE: _____

3. Copper metal + 6 M hydrochloric acid. Observe reaction several times for 10 - 15 minutes.

Observations	
Reactants before combining	After combining and mixing
Reacted?	Reaction type:

ME: _____

IE: _____

NIE: _____

4. Copper metal + 0.5 M silver nitrate. Observe reaction several times for 10 - 15 minutes.

Observations	
Reactants before combining	After combining and mixing
Reacted?	Reaction type:

ME: _____

IE: _____

NIE: _____

5. 0.1 M barium chloride + 0.1 M sodium sulfate.

Observations	
Reactants before combining	After combining and mixing
Reacted?	Reaction type:

ME: _____

IE: _____

NIE: _____

6. 1.0 M sodium hydroxide + hydrochloric acid. Measure temperature and pH before and after.

Observations	
Reactants before combining	After combining and mixing
Reacted?	Reaction type:

ME: _____

IE: _____

NIE: _____

7. 0.1 M ammonium chloride + 0.1 M copper (II) nitrate.

Observations	
Reactants before combining	After combining and mixing
Reacted?	Reaction type:

ME: _____

IE: _____

NIE: _____

9. 6.0 M sodium hydroxide dropwise + 0.1 M copper (II) nitrate.

Observations	
Reactants before combining	After combining and mixing
Reacted?	Reaction type:

ME: _____

IE: _____

NIE: _____

Experiment 9 – Reaction Stoichiometry and Percent Yield

I. Introduction

Stoichiometry calculations are about calculating the amounts of substances that react and form in a chemical reaction. The word "stoichiometry" comes from the Greek *stoikheion* "element" and *metriā* "measure." Based on the balanced chemical equation, we can calculate the amount of a product substance that will form if we begin with a specific amount of one or more reactants. Or, you may have a target amount of product to prepare. How much starting compounds are needed to prepare this amount? These are practical calculations that are done frequently by chemists. In this experiment, you will prepare copper metal from the reaction of aluminum metal with a solution of copper (II) sulfate (cupric sulfate). From the amounts of the reactants, you will determine which reactant is the **limiting reactant**, and from this amount, calculate the **theoretical yield** of copper metal. From the actual amount of copper obtained, you can then calculate your **percent yield** of copper.

The calculation method

For practically all stoichiometry calculations, we want to use the F**abulous Four Steps**

Step 1: Write the balanced chemical equation for the reaction.

Step 2: Calculate the *moles* of "given" substance. If more than one reactant amount is given, calculate the moles of each to determine which is the *limiting reactant*.

Step 3: Calculate the moles of "desired" substance from your answer in Step 2 using the coefficients from the balanced chemical equation. If more than one reactant was given originally, you can calculate the moles of product *twice*, based on the moles of each reactant. The reactant that gives the **smaller** moles of product is the **limiting reactant**. Keep this answer for Step 4.

Step 4: Convert your answer in Step 3 to the units the problem asks for. Usually this is grams, but it could be volume (for gases or liquid solutions) or concentration (such as molarity, for solutions).

Again, in brief:

1. Balanced reaction.

2. Moles of "given" substance(s).

3. Moles of "desired" substance such as a product.

4. Convert Step 3 answer to the units asked for.

Sample calculations

Eample 1. How many grams of iron (III) oxide (ferric oxide), Fe_2O_3, are formed from the reaction of 5.00 g of iron metal with excess oxygen gas?

Step 1: Balanced reaction.

4 Fe (s) + **3** O2 (g) ⟶ **2** Fe_2O_3 (s)

Step 2: Moles of "given" substance. The given substance is the iron metal, since its amount is given. moles of Fe = grams of Fe / molar mass of Fe = 5.00 g / 55.845 g/mol = 0.08953353 mol (not rounding yet). Note that gram units cancel, leaving the unit of moles for the answer $moles\ of\ Fe = \frac{500\ g\ Fe}{1} \times \frac{1\ mol\ Fe}{55.847\ g\ Fe} = 0.08953353$

Step 3: Moles of "desired" substance. This is the product, ferric oxide. Here is where the balanced reaction comes in. The coefficients in the balanced reaction represent the moles of the substances that react and form. As such, balanced reactions are "in" moles by default. This is why we always have to convert amounts to moles when working stoichiometry problems.

$$moles\ of\ Fe_2O_3 = \frac{5.00\ g\ mole\ Fe}{1} \times \frac{2mol\ Fe_2O_3}{4mol\ Fe} = 0.044766765\ mol\ Fe_2O_3$$

Step 4: Grams of Fe_2O_3.

Grams of $Fe_2O_3 = moles\ of\ Fe_2O_3 \times molar\ mass\ of\ Fe_2O_3 = 0.044766765\ moles \times 159.6882\ g/mol = 7.15g$ rounded to 3 significant figures.

And that's it! We can also do steps 2 through 4 like a conversion problem. Once you are familiar with the steps, you can work these problems more quickly this way:

$$\frac{5.00\ g\ Fe}{1} \times \frac{1\ mol\ Fe}{55.847\ g\ Fe} \times \frac{2mol\ Fe_2O_3}{4mol\ Fe} \times \frac{159.6882\ g\ Fe_2O_3}{mol\ Fe_2O_3} = 7.15g\ of\ Fe_2O_3$$

When working stoichiometry problems, always do the following:

a) Write the units on all numbers.

b) Check that the units cancel properly.

c) Give the correct unit for the answer.

d) Avoid rounding numbers too much during the calculation, or you will have roundoff error in your answer.

e) Round your final answer to the correct number of significant figures.

Practice makes perfect! Working stoichiometry problems properly will strengthen your skills in working many other types of chemistry problems as well.

PERCENT YIELD

The percent yield of a reaction tells us how well the reaction worked in terms of forming a desired product.

$$\textbf{\textit{Percent Yield}} = \frac{\textbf{\textit{Actual (experimental) Yield of product}}}{\textbf{\textit{Theoritical (calculated) Yield of product}}} \times \mathbf{100}$$

The unit of the amounts may be in grams or moles. Usually we use grams, since we will have weighed the product on a balance in gram units.

Example. In the above example, let's say that the student obtained 6.75 g of ferric oxide product. The theoretical yield of ferric oxide calculated with the Fabulous Four Steps was 7.15 g. The percent yield of ferric oxide from the reaction is therefore,

$$Percent\ Yield\ of\ Fe_2O_3 = \frac{6.75\ g}{7.15} \times 100 = 94.4\%$$

Generally, less than 100% yields are obtained. The reaction may not have sufficient time to go to completion, and if the product is transferred from on container to another, or is filtered, there is always some small loss. Therefore, the less "handling," the better the yield will be. If undesired "side reactions" occur in addition to the desired reaction, the yield will be affected. The use of impure reactants can adversely affect the percent yield also. On the other hand, it is impossible to obtain more than a 100% yield! If your calculated percent yield is more than 100%, then, aside from weighing errors, there is some additional substance present in your product making it weigh more than it should. If a solid product is prepared in aqueous solution, incomplete drying is the usual culprit, since the wet solid, which may appear dry, will weigh more than the dry solid product. Chemists use the method of heating to constant weight to avoid this error. If other impurities are present in the product, other methods must be used to remove them, such as extraction, recrystallization, distillation, and chromatography. These techniques are often used by chemists to isolate and purify the desired product from the reaction mixture as effectively and efficiently as possible.

II. Experimental Procedure

★ **Safety Note:** *Hydrochloric acid is a strong acid that is harmful to the skin and especially to your eyes. Wear your safety glasses or goggles during the entire procedure. The reaction also produces flammable hydrogen gas (H2), so Bunsen burners should not be used while the reaction is in progress. Also see the procedural notes on the next page.*

1) Weigh a clean, dry 150 mL beaker and record its weight on the report form.
2) Carefully add copper (II) sulfate pentahydrate, $CuSO_4 \cdot 5H_2O$, until 2.00 g have been added.
3) Measure 10 mL of deionized water in a small graduated cylinder and add the water to the beaker to dissolve the copper (II) sulfate pentahydrate with the aid of a glass stirring rod.

Record the color of the solution on the report sheet.

4) Measure 2.0 mL of 6 M(HCl) in your graduated cylinder, add it to the solution, and mix well.
5) Weigh 0.25 g of dry aluminum foil in small pieces and record the weight on the report sheet.
6) Add the pieces of Al foil a little at a time. Use the stirring rod to mix the solution during the reaction. (CAUTION: Exothermic reaction!) Note the color of the solution after the added piece of aluminum no longer darkens on its surface. Add the remaining few pieces of aluminum foil, and add an additional 5 mL of 6 M(HCl) to facilitate the reaction of any excess aluminum with the hydrochloric acid.

7) After all of the aluminum foil has reacted, allow the solid particles of copper product to settle, and carefully decant the solution from the solid (leaving the copper behind in the beaker). Add 20 mL of deionized water to the solid, stir well with the stirring rod, and decant again. Repeat this washing with 20 mL of water once more. Finally, add 10 mL of methanol to the solid, stir, and decant.

8) Heat the beaker on an electric hot plate at medium heat (a setting of about 4 out of 10) until the solid and beaker are thoroughly dry. Allow the beaker and its contents to cool, and then weigh and record the weight on the report form. If time allows, you may re-heat the beaker for an additional 10 minutes and reweigh the beaker and contents to ensure that drying is complete (heating to constant weight).

9) On the report sheet, record the moles of Al and $CuSO_4 \cdot 5H_2O$ used, and determine which is the limiting reactant. Based on the limiting reactant, calculate the theoretical yield of copper metal product. From your actual yield of copper, calculate the percent yield of copper product obtained from the reaction.

10) Place your copper metal in the collection container on the front desk (do not wash it down the sink!), rinse your glassware well, and return it and your other equipment to their proper storage locations.

EXPERIMENT 9 – Reaction Stoichiometry and Percent Yield

REPORT FORM

Name _____ Section _____

Instructor _____

Date _____

1. Mass of empty 100 mL beaker _____ g

2. Mass of beaker plus CuSO4 · 5 H2O _____ g

3. Color of solution _____

4. Mass of CuSO4 · 5 H2O [2] – [1] _____ g

5. Mass of Al foil used (small pieces) _____ g

6. Color of solution after reaction is complete _____

7. Mass of beaker and copper product First weighing _____ g (after heating and cooling) Second weighing _____ g

8. Mass of copper metal product [7] – [1] _____ g

9. Moles of Al used _____ mol (show your calculation)

10. Moles of CuSO4 · 5 H2O used _____ mol (show your calculation)

11. Moles of copper product based on moles of Al _____ mol (show your calculation)

12. Moles of copper product based on moles of $CuSO_4 . 5H_2O$ _____ mol (show your calculation)

13. Limiting reactant _____

14. Grams of Cu product based on the limiting reactant (theoretical yield) _____ g (show your calculation)

15. Percent yield of Cu (2 to 3 significant figures) _____ % (show your calculation)

EXPERIMENT 10: Chemicals in Everyday Life

Purpose

The purpose of this experiment is to observe chemical reactions of common household substances.

Learning Objectives

Make and record observations of chemical reactions of known substances.

Identify common chemicals by comparing observations of chemical reactions with those of known substances.

Equipment

150 mL beaker	droppers
Bunsen burner and hose	Test tube rack
Six 3 in. test tubes	Red and blue litmus paper
Spatula	
Three medicines	Test tube clamp

Chemicals

Household ammonia	18.0 M H_2SO_4
Household chlorine bleach	1.0 M NH_4Cl
8.0 M NaOH	
Baking soda	3.0 M HNO_3
Vinegar	$Ba(OH)_2$ (sat. soln.)
Table salt	0.1 M $AgNO_3$
Chalk	0.2 M $BaCl_2$
Epsom salts	2.0 M HCl
Mineral oil	Solid unknown
Chemical fertilizer containing: CO_3^{2-}, NaI	Cl^-, SO_4^{-}, or I^-
ion $(NH_4)_2CO_3$	

Introduction

One important aspect of chemistry is the identification of substances. The identification of minerals for example, fool's gold as opposed to genuine gold, was and still is of great importance to prospectors. The rapid identification of a toxic substance ingested by an infant may be the determining factor in saving the child's life. Substances are identified by the use of instruments, by reactions characteristic of the substance, or both.

A reaction that is characteristic of a substance is frequently referred to as a ***test***. For example, you may test for oxygen with a glowing splint; if the splint bursts into flame, oxygen is probably present. You test for chloride ions by adding silver nitrate to an acidified solution. The formation of a white precipitate suggests the presence of chloride ions. Because other substances may yield a white precipitate under these conditions, one can "confirm" the presence of chloride ions by observing that this precipitate dissolves in ammonium hydroxide while the other white precipitates do not. This last step is known as a **confirmatory test**. Commonly, in order to test for an ion present in an unknown solution, two experiments need to be performed. The first test is a test to give a clue of the possible ion present in the unknown solution, and the second is a confirmatory test. Determining the identity of a substance is referred to as **qualitative analysis**. Determining the amount of a substance is **quantitative analysis**.

In this experiment, students will perform tests on or with substances they are likely to encounter in everyday life, such as table salt, bleach, smelling salts, and baking soda. While you may not think of these as chemicals, they most certainly are. Their chemical names are sodium chloride, chlorine, ammonium carbonate, and sodium bicarbonate, respectively.

After observing some reactions of these household chemicals, students will partially identify an unknown. The task will be to determine whether the substance contains the carbonate (CO_3^{2-}), chloride (Cl^-), sulfate (SO_4^{2-}), or iodide (I^-) ion.

Ammonia, NH_3

Ammonium salts are converted to ammonia, $\mathbf{NH_3}$, by the action of strong bases. Thus, it is possible to test for the ammonium ion, $\mathbf{NH_4^+}$, by adding the strong base, sodium hydroxide,

NaOH. A reaction can be detected by the familiar odor of ammonia, NH_3, or by using red litmus paper.

The net ionic reaction is given in Reaction CL.1:

$$NH_4^+(aq) + OH^-(aq) \rightleftharpoons NH_3(aq) + H_2O(l) \qquad \text{(Reaction CL.1)}$$

Most ammonium salts are stable, which is why it is necessary to add sodium hydroxide to the ammonium chloride solution to detect the ammonia using litmus. However, $(NH_4)_2CO_3$ is quite unstable and decomposes to ammonia and carbon dioxide as seen in reaction CL.2:

$$(NH_4)_2CO_3(s) \xrightarrow{\Delta} 2\,NH_3(g) + CO_2(g) + H_2O(g) \qquad \text{(Reaction CL.2)}$$

Household sources of ammonia include smelling salts, which contain ammonium carbonate and many different types of household cleaning solutions, especially those used to clean glass and kitchen counters.

Carbonate Ion, CO_3^{2-}

Substances that contain the carbonate ion, CO_3^{2-}, react with acids to liberate carbon dioxide, CO_2, which is a colorless and odorless gas. Carbon dioxide is released from baking soda by acids, such as those found in lemon juice and buttermilk, and forms bubbles in the cake batter that expand in the hot over, causing a cake to rise. Acids release H^+ in aqueous solution, so the reaction of carbon dioxide with acids is written as shown in Reaction CL.3:

$$NaHCO_3(s) + H^+ \longrightarrow CO_2(g) + H_2O(l) + Na^+(aq) \qquad \text{(Reaction CL.3)}$$

A **confirmatory test** for CO_2 is to allow it to react with $Ba(OH)_2$, barium hydroxide, solution. A white precipitate of $BaCO_3$, barium carbonate, is produced (reaction CL.4):

$$CO_2(g) + Ba(OH)_2(aq) \longrightarrow BaCO_3(s) + H_2O(l) \qquad \text{(Reaction CL.4)}$$

Many substances, such as eggshells, oyster shells, limestone, and washing soda all contain the carbonate ion. A closely related ion, bicarbonate, HCO_3^-, that also reacts with acids to produce carbon dioxide, is found in baking soda and baking powder.

Chloride Ion, Cl^-

Chloride salts react with concentrated sulfuric acid to liberate hydrogen chloride, which is a pungent and colorless gas that turns moist blue litmus red (Reaction CL.5:

$$2\,Cl^-(aq) + H_2SO_4(aq) \longrightarrow 2\,HCl(g) + SO_4^{2-}(aq) \qquad \text{(Reaction CL.5)}$$

This reaction will occur whether the substance is $BaCl_2$, KCl, or $ZnCl_2$; the only requirement is that the salt be a chloride. For KCl, the complete equation is shown in reaction CL.6:

$$KCl(s) + H_2SO_4(aq) \longrightarrow 2\,HCl(g) + K_2SO_4(aq) \qquad \text{(Reaction CL.6)}$$

The **confirmatory test** for chloride ion is a well-known characteristic reaction of the chloride ion with silver nitrate to form silver chloride, AgCl, which is a white, *insoluble* substance (reaction CL.7):

$$Cl^-(aq) + AgNO_3(aq) \longrightarrow AgCl(s) + NO_3^-(aq) \qquad \text{(Reaction CL.7)}$$

Chloride ions can be found in table salt and road salt.

Sulfate Ion, SO_4^{2-}

The following tests are characteristic of the sulfate ion, SO_4^{2-}.

Barium sulfate is a white insoluble substance that forms when barium chloride is added to a solution of any soluble sulfate salt, such as Epsom salts, as seen in Reaction CL.8:

$$SO_4^{2-}(aq) + BaCl_2(aq) \longrightarrow BaSO_4(s) + 2\,Cl^-(aq) \qquad \text{(Reaction CL.8)}$$

Barium chloride solution is then used as a confirmatory test for sulfate ion.

Sulfate ions can be found in Epsom salts, $MgSO_4 \cdot 7\,H_2O$, which are used as a laxative and also in a soaking solution for tired, aching feet.

Iodide Ion, I^-

Iodide ion can be converted to elemental iodine by reacting with chlorine gas (Reaction CL.9).

$$Cl_2(aq) + 2\,I^-(aq) \longrightarrow I_2(aq) + 2\,Cl^-(aq) \qquad \text{(Reaction CL.9)}$$

Iodine gives a reddish-brown color to water; it is more soluble in mineral oil than in water, and it imparts a violetcolor to mineral oil. Thus, chlorine can be used to identify iodide salts.

Elemental chlorine behaves very differently than chloride ions. Elemental chlorine is a pale yellow-green, highlytoxic gas and, thus, is inappropriate for use in an introductory chemistry experiment. Fortunately, commercial bleach, which is a 5% solution of sodium hypochlorite, NaOCl in water, can be used in place of elemental chlorine.

Another way to identify iodide ions uses concentrated sulfuric acid. Solid iodide salts react with concentrated sulfuric acid by turning dark brown and emitting a gas that, when exposed to moist air, appears violet. Pure solidiodine is known to form a violet vapor.

An additional characteristic reaction of iodides is that they form pale-yellow precipitates when treated with silvernitrate solution, shown in reaction CL.10:

$$I^-(aq) + AgNO_3(aq) \longrightarrow AgI(aq) + NO_3^-(aq) \qquad \text{(Reaction CL.10)}$$

Silver nitrate solution is then used as a confirmatory test for iodide ion.

Iodine, an important nutrient, can be found in seafood and dairy products, as well as iodized salt.

Sodium Ion, Na^+

Sodium ions are ubiquitous (found everywhere). The presence of sodium ions can be easily detected because sodium ions emit a characteristic yellow color in a flame. When salted water boils over on a gas stove, the temporary yellow flame color is due to sodium ions. The yellow flames of a campfire indicate the presence of sodiumions in the wood.

Unknown Ion or Ions

Your solid unknown contains **one** of the following ions: carbonate, chloride, sulfate, or iodide. Table CL.1 sum-marizes the behavior of these ions toward H_2SO_4.

Table CL.1: Reaction of Solid Salts with H_2SO_4

Ion	Reaction	Confirmatory Test
CO_3^{2-}	Colorless, odorless gas, CO_2, evolved	Barium hydroxide solution
Cl^-	Colorless, pungent gas, HCl, evolved,which turns blue litmus red	Silver nitrate solution
SO_4^{2-}	No observable reaction	Barium chloride solution
I^-	Violet or red-brown vapors of I_2 formed	silver nitrate solution

Procedure

Safety Precautions

Even though household chemicals may appear innocuous, you should **never** mix them. Innocuous chemicals when combined may produce noxious gases, explosions or other hazardous reactions.

CAUTION: Concentrated sulfuric acid, H_2SO_4, causes severe burns. Do not get it on your skin. If you comein contact with it, immediately wash the area

A. Household Ammonia, NH_4OH

1. Obtain ~1 mL of household ammonia in a 150 mL beaker.
   - Hold a dry piece of red litmus paper over the beaker, being careful not to touch the paper with the sides of the beaker or the solution. Record your observations on the Report Sheet.
   - Repeat the operation using a piece of red litmus paper that has been moistened with tap water. Record your observations about household ammonia and litmus paper on the Report Sheet.

2. Place ~1 mL of 1.0 M NH_4Cl, ammonium chloride, in a test tube.
   - Hold a moist piece of red litmus in the mouth of the tube. Record your observations about NH_4Cl and litmus.
   - Now add about 1 mL of 8.0 M NaOH and repeat the test. If the litmus paper does not change color, gently warm the test tube, but do not boil the solution. Record your observations about NH_4Cl, NaOH and litmus paper.

     > Do not allow the litmus paper to touch the sides of the tube because it may come in contact with NaOH, which will turn the litmus paper blue.

3. Test fertilizer for the presence of ammonia by placing an amount about the size of a pea, in a test tube.
   - add 1 mL of 8.0 M NaOH and test as above using moist litmus paper. Record your observations.
   - Does the fertilizer contain ammonium salts? Record your answer.
4. Hold a moist piece of red litmus over the mouth of an open jar of ammonium carbonate ($(NH_4)_2CO_3$).
   - Record your observations about ammonium carbonate and litmus paper on the Report Sheet.
   - Carefully fan your hand over the jar and see if you can detect a familiar odor. Record your observations.

CAUTION:

Do not place any sample being tested for odor directly under your nose. Instead, waft the air above the sample toward you.

5. Place the chemicals used in the designated waste receptacles.

B. Baking Soda, $NaHCO_3$

1. Place in a small, dry test tube an amount of solid baking soda about the size of a small pea.
   - Add 1 or 2 drops of concentrated H_2SO_4.
   - Record your observations about baking soda and H_2SO_4.

2. Repeat the above procedure using vinegar instead of the sulfuric acid. Record your observations about vine-gar and baking soda.

3. Place a small piece of chalk in a dry test tube.
   - Add a few drops of 2.0 M HCl. Record your observation.
   - Test the escaping gas for CO_2 by carefully holding a drop of $Ba(OH)_2$, suspended from the tip of amedicine dropper or a wire loop, a short distance into the mouth of the test tube.
   - Record your observation.
   - Clouding of the drop is due to the formation of $BaCO_3$ and indicates the presence of carbonate ion.Does chalk contain carbonate ion? Record your answer on the Report Sheet.

     Breathing on the drop will cause it to cloud, because your breath contains CO_2 .

4. Place the chemicals used in the designated waste receptacles.

C. Table Salt, NaCl

1. Place in a small, dry test tube an amount of sodium chloride about the size of a small pea.

- Add 1 or 2 drops of concentrated H_2SO_4.
- Hold a piece of moistened blue litmus paper over the test tube, being careful not to touch the paper withthe sides of the test tube or the solution. Record your observations about NaCl andH_2SO_4 on the ReportSheet.
- On the Report Sheet, complete and balance the equation $NaCl(s) + H_2SO_4(aq) \longrightarrow ?$

2. Place a small amount (about the size of a pea) of NaCl in a small test tube.

   - add 15 drops of DI water and 1 drop of 3.0 M HNO_3.
     - Then add 3 or 4 drops of 0.1 M $AgNO_3$ and mix the contents. Record your observations about NaCl and 0.1 M $AgNO_3$ on the Report Sheet.

3. Could you have replaced the DI water with tap water for this test? Record your answer on the Report Sheet.

4. Confirm your answer by testing tap water for chloride ions:

   - Add 1 drop of 3.0 M HNO_3 to about 2 mL of tap water
   - Add 3 drops of 0.1 M $AgNO_3$. Record your observations?
   - Does this test indicate the presence of chloride ions in tap water? Record your answer on the Report Sheet.

5. Your lab instructor will demonstrate the presence of sodium ions by placing drops of a solution of NaCl orholding NaCl crystals in a flame. Record your observations on the Report Sheet.

6. Place the chemicals used in the designated waste receptacles.

D. Epsom Salts, $MgSO_4 \cdot 7\ H_2O$

1. Place a small quantity of Epsom salts in a small, dry test tube.

   - Add 1 or 2 drops of 18.0 M H_2SO_4 and record your observations.
   - Note (and record!) the difference in the behavior of this substance toward sulfuric acid compared withthe behavior of baking soda toward sulfuric acid.

2. Place some Epsom salts (an amount the size of a small pea) in a small test tube and dissolve it in 1 mL of DIwater.
   - Add 1 drop of 3.0 M HNO_3, then 1 or 2 drops of 0.2 M $BaCl_2$.
   - Record your observations about Epsom salts and $BaCl_2$ on the Report Sheet.

3. Place the chemicals used in the designated waste receptacles.

E. Bleach, NaOCl

1. In a small test tube, dissolve a small amount (about the size of a pea) of sodium iodide, NaI, in 1 mL of DIwater.
   - Add 5 drops of bleach and note the color.
   - Then add several drops of mineral oil, shake, and allow the layers to separate, which takes about 20 sec.
   - Record your observations about bleach and NaI on the Report Sheet.

   > Mineral oil is less dense than water. Thus, the mineral oil layer is the top layer.

2. Dissolve a small amount of sodium iodide in 1 mL of DI water.
   - First add a drop of 3.0 M HNO_3 and then add 3 or 4 drops of 0.1 M $AgNO_3$ solution.
   - Record your observations about NaI and $AgNO_3$ on the Report Sheet.
   - Balance and complete the equation.

3. Place a small amount (about the size of a pea) of sodium iodide in a small, dry test tube and carry it to thehood.
   - *Carefully, while under the hood,* add 1 or 2 drops of 18.0 M H_2SO_4.
   - Record your observations about NaI and H_2SO_4 on the Report Sheet.
4. Place the chemicals used in the designated waste receptacles.

F. Unknown

Your solid unknown will contain only one of the following ions: carbonate, chloride, sulfate, or

iodide. Table CL.1summarizes the behavior of these ions toward H_2SO_4

1. Place a small amount of your unknown (save some for further tests) in a small, dry test tube.
    - Carefully add a drop of 18.0 M (concentrated) H_2SO_4.
    - Record your observations and the identity of the unknown ion on the Report Sheet.

2. Confirm the identity of the unknown. Carry out the appropriate confirmatory test and record your observa-tions on the Report Sheet.

3. Place the chemicals used in the designated waste receptacles.

Name: _____

Section:_____Date: _____

A. Household Ammonia, NH_4OH

Effect of household ammonia on dry litmus paper _____

Effect of household ammonia on moist litmus paper _____

Effect of NH_4Cl on litmus paper _____

Effect of NH_4Cl + NaOH on litmus paper _____

Effect of fertilizer + NaOH on litmus paper _____

Do fertilizers contain ammonium salts? Yes_____No _____

Effect of smelling salts on litmus paper _____

Odor of smelling _____

What do smelling-salts contain? _____

B. Baking Soda, $NaHCO_3$

Effect of sulfuric acid on baking soda _____

Effect of vinegar on baking soda _____

chalk + HCl _____

Appearance of $Ba(OH)_2$ drop _____

Does chalk carbonate ion? Yes_____No _____

C. Table Salt, NaCl

Effect of H_2SO_4 on table salt _____

$H_2SO_4(aq) + 2\ NaCl(s) \longrightarrow\ ?$ _____

Effect of $AgNO_3(aq)$ on table salt solution _____

Could tap water replace DI water? _____

_____

Effect of $AgNO_3(aq)$ on tap water _____

Chloride ions in tap water: Yes_____No _____

Salt in flame _____

. D. Epsom Salts, $MgSO_4 \cdot 7\ H_2O$

Effect of H_2SO_4 on Epsom salts _____

Compare effects of H_2SO_4 on Epsom salts versus baking soda. _____

_____

$BaCl_2$ + Epsom salts _____

E. Bleach

Effect of bleach on NaI(aq) _____

Effect of $AgNO_3(aq)$ on NaI(*aq*) _____

$AgNO_3(aq) + NaI(aq) \longrightarrow$ _____

Effect of H_2SO_4 on NaI(*aq*) _____

F. Unknown

*Unknown #*_____

Effect of H_2SO_4 on unknown(*aq*) _____

Unknown ion_____

Confirmatory test_____

Results of confirmatory test) _____

Experiment 11: The Thermodynamics of Solubility of Potassium Chlorate

Objectives:

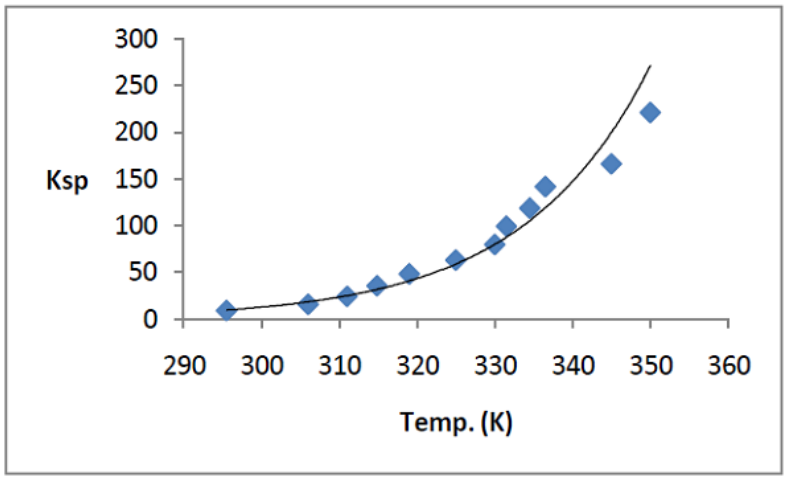
- To observe the effect of temperature on solubility product constant (K_{sp})
- to draw the graph of lnK_{sp} vs 1/T in order to calculate the standard enthalpy change (ΔH°) and standard entropy change (ΔS°) via this graph.

Introduction: The solubility of $KClO_3$ will be measured at various temperatures. From the molar solubility, s: moles of solute per liter of solvent, a value of the solubility product constant, K_{sp}, can be calculated at various temperatures for the following reaction:

$KClO_{3(s)} \rightleftarrows K_{+(aq)} + ClO_{3\,-(aq)}$

$K_{sp} = [K_+][ClO_{3-}] = s^2$

The solubility of a solid is exponential with respect with temperature. A plot of the solubility product constant, Ksp, vs temperature (K) will give an exponential curve.



The relationship between ΔH^0 and the K_{sp} comes from the free energy equations

(1) ΔG^0 = - RT ln K, where R = the gas constant, 8.3143 Joule/mol K and T = absolute temperature in degrees Kelvin.

(2) $\Delta G^0 = \Delta H^0 - T\Delta S^0$, where ΔH^0 is the standard enthalpy change and $\Delta S^0$is the standard entropy change. From (1) and (2) comes the equation

(3) - RT ln K = ΔH^0 - TΔS^0,

Both the molal heat of solution ΔH^0 and the entropy ΔS^0 values can be obtained for the reaction by solving the equation relating the slope of the graph with ΔH^0 and the y- intercept with ΔS^0. The plot of ln K vs 1/T in K^{-1} will give the following equation of a straight line: Each group will

be assigned a different composition of solution to measure. The data from each group will be combined and plotted.

Safety precautions

• $KClO_3$ is flammable and strong oxidizing agent. Therefore, dispose of all wastes by flushing them down the sink with plenty of running water.

• Avoid any contact between the $KClO_3$ and paper.

Review Questions:

Before beginning this experiment in the laboratory, you should be able to answer the following questions:

1. Suppose a reaction has negative ΔH^0 and negative ΔS. Will more or less product be present at equilibrium as the temperature is raised?

2. Fisherman know that on hot summer days, the largest fish will be found in deep sinks in lake bottoms, where the water is coolest. What is the reason of it, think about the temperature dependence of oxygen solubility in water?

3. Nitrogen dioxide reacts with water to produce nitric acid according to following reaction: Predict the sign of ΔS° for this reaction

4. Suggest a reason why the value of ΔH_{soln} for a gas such as CO_2, dissolving in water, is negative.

Procedure:

1.In a test tube, weigh a sample of $KClO_3$ to the nearest 0.01g. The amount of sample will range from 1 to 5 grams in 1-gram increments.

2. Pipet 10 mL of distilled water into the test tubes. Stir the mixture with a glass rod.
3. Gently warm the mixture until the crystals dissolve. For high sample amounts, an evaporating dish can be used as a water bath to transfer heat more evenly. Insert thermometer into test tube and try to note the temperature at which the last crystals go into solution. Then allow the solution to cool until crystals reappear and again note the temperature.
4. Record the saturation temperature you have just determined and enter your values in your data sheet.

Calculations:

A. Ksp values.

1. Write the equation for the equilibrium between solid $KClO_3$ and its ions in solution and the expression for the K_{sp} of $KClO_3$.

2. From the weight-volume data, calculate the molar concentration of $KClO_3$ for each composition. This is the molar solubility at the saturation temperature measured.
3. Calculate the solubility product of $KClO_3$ for each trial. Use the concentrations found in step 1.

4. Tabulate the resulting values of solubility, K_{sp}, and temperature in your lab datasheet.
5. Using Excel, graph temperature (Kelvin) on the horizontal axis (x-axis) versus Ksp on the vertical axis (y-axis).

B. Molar heat of solution of $KClO_3$.

1. Using excel calculate values of 1/T, the reciprocal of the absolute temperature, and ln K_{sp} for each of the points graphed in part A.

2. Make a new graph with ln K_{sp} on the vertical axis and 1/T (K^{-1}) on the horizontal axis. Use the linear curve fit option in excel to obtain the best fit linear fit.

3. From the slope of your graph calculate ΔH^{o}, the heat of solution for $KClO_3$ Also determine $\Delta S°$ from the y-intercept. Tabulate data for the written report and include the curve fit equation with the graph of lnKsp vs 1/T.

QUESTIONS:

1. When a certain solid dissolves in water, the solution becomes cold. Is ΔH_{soln} for this solute positive or negative?

Is the solubility of this substance likely to increase or decrease with increasing temperature?

2. What is the value of equilibrium constant for a reaction for which $\Delta G°= 0$?

What will happen to the composition of the system if we begin the reaction with the products?

3. The solubility of ZnS at 25 °C is 3.5×10^{-12} M. What is the K_{sp} for ZnS? What is $\Delta G°$ at 25°C?

Experiment 11: The Thermodynamics of Solubility of Potassium Chlorate

REPORT FORM

Name _____ **Section**_____

Instructor _____

Date _____

A. K_{sp} Values

1. Write the reaction equation and the solubility product expression for $KClO_3$.

2.

Amount of $KClO_3$(g)	**Molar Concentration of $KClO_3$ (mol/L)**	**Ksp**	**T (°C)**	**T (K)**

3. Draw K_{sp} vs Temperature (K) graph using Excel.

B. Molal Heat of Solution of $KClO_3$

1.

1/T (K^{-1})	**ln Ksp**

2. Draw ln Ksp vs 1/T graph using Excel and calculate ΔH° from the slope and ΔS° from y intercept.

ΔH°=

ΔS° =

Experiment 12: Atomic Spectra and Atomic Structure

Purpose

The purpose of this experiment is to examine and compare the emission spectra of various light sources.

Learning Objectives

Describe and compare emission spectra of incandescent, fluorescent, and LED sources to atomic spectra.

Relate the lines observed in atomic spectra to the internal structure of an atom.

Calculate the energy of observed spectral lines from their approximate

Equipment

- Handheld spectroscope
- Incandescent light
- Fluorescent light
- Three discharge lamps (any element)
- Spot plates or well plates
- Assorted coloredpencils
- Bunsen burner andstriker
- Nichrome wire loop
- Wash bottle of DI water

Chemicals

- LiCl
- NaCl
- KCl
- $CaCl_2$
- $CuCl_2$
- $SrCl_2$

Introduction

During the twentieth century, scientists developed several models of the atom in an attempt to describe its internal structure. Proposed by Niels Bohr in 1913, the Bohr model provides a simplified picture of the energy levels in an atom. While later found to be somewhat incorrect, this model is sufficient for studying some of the most important properties of an atom.

According to the Bohr model (shown in Figure SP.1), protons and neutrons occupy the central core of the atom known as the nucleus, while electrons occupy different energy levels around the nucleus. An atom that has its electrons distributed into the lowest possible energy levels, its lowest-energy electron configuration, is in the **ground** state. If any electrons occupy a higher energy level, the atom has an **excited** state configuration. Electrons may only move from one energy level to another through the processes of excitation (absorbing energy) or emission (giving off energy).

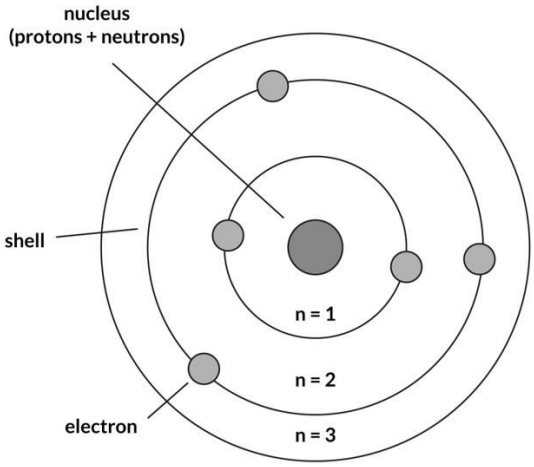


Figure SP.1: Bohr model for a Boron atom.

Excitation occurs when an electron jumps from a lower energy level to a higher energy level by *absorbing* energy from light or electricity. **Emission** occurs when an electron relaxes from a higher energy level to a lower energy level, and it results in the *release* of energy as a photon (quantity of electromagnetic radiation, or light).

Figure SP.2 depicts the helium atom going through each phase of an electron transition. Helium begins in its ground state in Diagram A. In Diagram B, the atom absorbs enough energy to cause an electron to jump from the 1*s* orbital to the 2*s* orbital. Eventually all excited electrons lose the extra energy and relax back to ground state, as shown in Figure SP.2, whereupon the atom emits a photon with a wavelength unique to the precise change in energy levels. An

excited electron emits more energy if it falls from a higher level; for example, a Helium 1s electron could jump to n = 2, 3, 4, 5 or 6. In each case the electron emits a different wavelength of light as it fallsback to the 1s energy level (ground state).

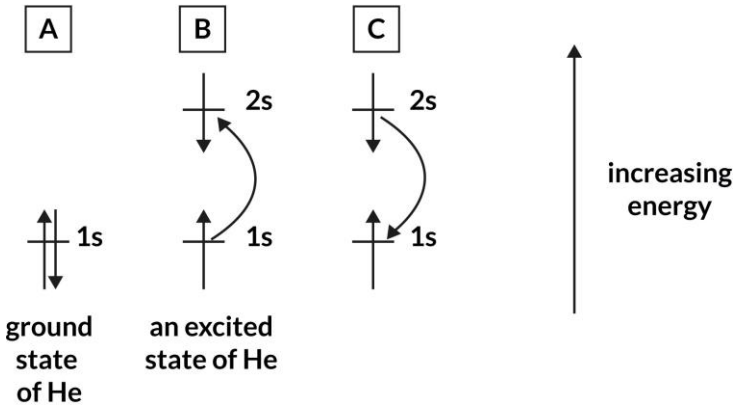


Figure SP.2: Electron excitation and emission in He.

The energy of absorbed or emitted photons exactly matches the difference in energy between the two levels of the electron transition. For example, the difference in energy between orbitals 1*s* and 2*s* is a specific quantity. Any electron jumping from 1*s* to 2*s* must absorb that exact quantity of energy and any electron relaxing from 2*s*to 1*s* emits that exact quantity of energy. The photon absorbed or emitted by the electron is at a wavelength thathas this energy. Thus, the larger the *gap* between orbitals, the greater the amount of energy that is absorbed or emitted by the electron.

In this experiment, you will use the method of spectroscopy to examine the emission spectra of different elements from the periodic table. An emission spectrum is the collection of all emitted wavelengths for an atom ormolecule. Parts A and B utilize a spectroscope, which is an instrument that separates light into its component wavelengths, as does a prism. With each spectrum, you will determine the individual wavelengths of light beingemitted by the atom, seen as distinct colored bands.

Every atom has different electron transitions that are possible, given the number of electrons and orbitals aroundit. The set of transitions an atom has is unique to that particular atom and, therefore, acts as a "fingerprint" for theatom. This is a very important characteristic that allows scientists to identify the elements present in an unknownsubstance, such as determining the

atomic composition of stars and planets in galaxies too far away to analyze by any other method. Because the data in this experiment is limited to the visible region of light, students may observe very similar spectra coming from different elements. If you were able to detect energy in the ultravioletregions, you would would observe a unique spectrum for each element.

The energy (*E*) of each observed wavelength (λ) is calculated using Equation SP.1:

$$E = \frac{hc}{\lambda} \qquad \text{(Equation SP.1)}$$

where *h* is Planck's constant, 6.626 x 10^{-34} J·s, and *c* is the speed of light, 2.998 x 10^{8} m/s.

Because every wavelength (or color of light) has a specific energy associated with it (as discussed above), it alsorefers to a specific electron transition occurring in the atom.

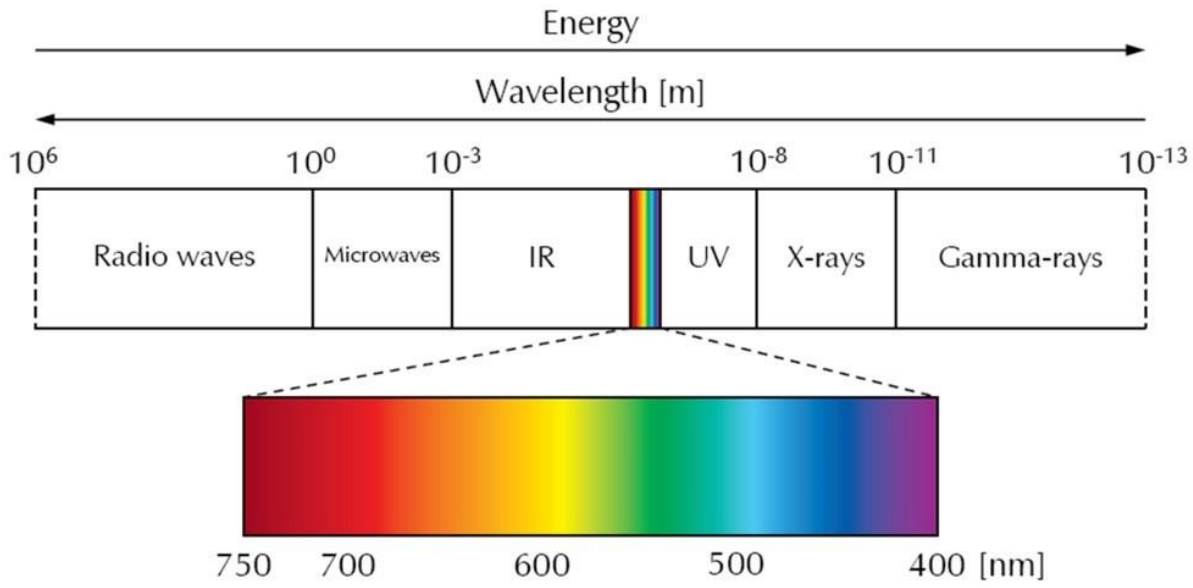


Figure SP.3: The relationship between wavelength and energy.

The electromagnetic spectrum of energy includes many kinds of energy, of which visible light is only a small portion. Figure SP.3 shows the relation between wavelengths and energy in this spectrum, as well as the other kinds of energy waves.

Safety Precautions

Prior to using Bunsen burners, examine gas tubing to ensure that it is free of large cracks and holes, ensuregas line is clear of any objects, pull hair away from face, and place hot items on a safe surface. *Never leave alit Bunsen burner unattended.*

Procedure

Safety Precautions

When working with concentrated acid solutions: WEAR GLOVES, spills must be neutralized, splashes onskin or in eyes must be flushed immediately with cold tap water for a minimum of 15 minutes.

Using a Spectroscope

1. Looking at your handheld spectroscope, locate each of the labeled parts shown in Figure SP.4. Notice the grating has a small window through which you will look to see the spectrum created by each light source.

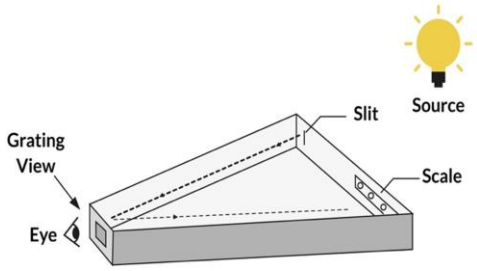


Figure SP.4: Diagram of a handheld spectroscope.

2. On the other end of the scope is a narrow slit. Position the slit so it is lined up with the light source. It is important that you center the slit squarely with the source so no stray radiation enters the scope, since stray radiation will confuse your data.

3. Beginning with the overhead FLUORESCENT light, look through the window of the spectroscope. Youshould be able to see the slit at the other end and a scale to the right of the slit that looks like a ruler.

The scale does not start at 0.0 and it does not give a unit of measure.

4. Along the scale you should also be able to see a series of separate-colored bands. Draw a sketch of the emission spectrum on your report sheet that clearly shows the relative positions of each band on the scale, and label the color of each band. Using the scale, estimate the wavelength of each color band.

If working in pairs, one student should use the spectroscope to collect data on fluorescent light; the other should collect data on incandescent light so that each gains skill in BOTH reading the instrument and in recording the results.

5. Now use the spectroscope to examine the INCANDESCENT light in the desk lamp provided. You should notice a very different looking spectrum from that of the fluorescent lamp; this is because you are looking at"white" light which contains every possible wavelength of the visible range. Using colored pencils provided, draw a sketch of the emission spectrum on your report sheet that clearly describes what you see.

Spectroscope Analysis of Atomic Gases

(performed individually)

Use the spectroscope to view the emission spectrum for each of the gas discharge tubes. Each one contains adifferent atomic gas that is being excited by electricity. Lamps may be viewed in any order.

You will be given three elements of the many that could be tested in the lab. On your report sheet, clearly recordthe identity of your three gasses and the scale position (± 0.1) and color of ALL bands observed for each lamp.

Describe the appearance of each lamp as you view it naturally without the scope. In other words, what color(s)does each one give off as you look at it? Be *descriptive*.

Flame Tests of Metal Ions (performed individually)

Thoroughly clean and rinse a well plate. Do not spend time drying it. Place it on a paper towel and label the towel for each of these solutions: LiCl; NaCl; KCl; $CaCl_2$; $CuCl_2$; and $SrCl_2$. Place ~20 drops of the corresponding 5% solutions into each labeled well. Label an additional well for HCl and add ~2 mL of 1 M HCl to it. This solution will be used for cleaning the nichrome wire between flame tests.

Obtain a Bunsen burner and a striker. *Follow all safety protocols prior to igniting the burner.*

Adjust the burner appropriately until you obtain a sharp blue flame as shown in Figure SP.5.



Figure SP.5: Proper adjustment of burner.

It may be helpful to reduce the lighting in the lab while students are doing the flame tests. This will make colored emissions more visible.

**Dip the looped end of a nichrome wire into your HCl solution; then pull it out of solution and heat in the flame to clean it as shown in Figure SP.5. Repeat this process until the flame burns faintly yellow when the wire is introduced; *other colors indicate the presence of impurities*.

Allow the wire to cool briefly after it is cleaned; dip the loop into your solution labeled LiCl.

Bring the loop into the tip of the inner cone (from the side) and carefully observe what happens. On your ReportSheet, record everything you see, especially noting any color changes. Be as descriptive as possible.

Repeat from the ** above for each of the remaining solutions and the activated charcoal, being careful to com- pletely clean the nichrome loop with HCl between test solutions. When testing the charcoal, wet the loop first with DI water then dip/swirl the wet loop in the charcoal powder.

When you are finished with the last solution, clean the nichrome wire once more with HCl and return it to its storage location. Dispose of all solutions properly.

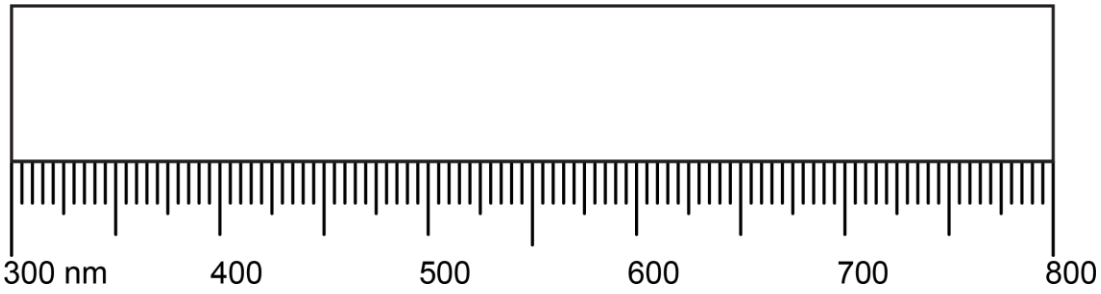
Waste Disposal

1. To dispose of the test solutions, use a DI water squirt bottle to rinse the well plate into a large beaker. Rinsethe well plate several times. Then pour the contents of the beaker into the proper waste container in the hood. Wash the well plate and return it to the storage bin.

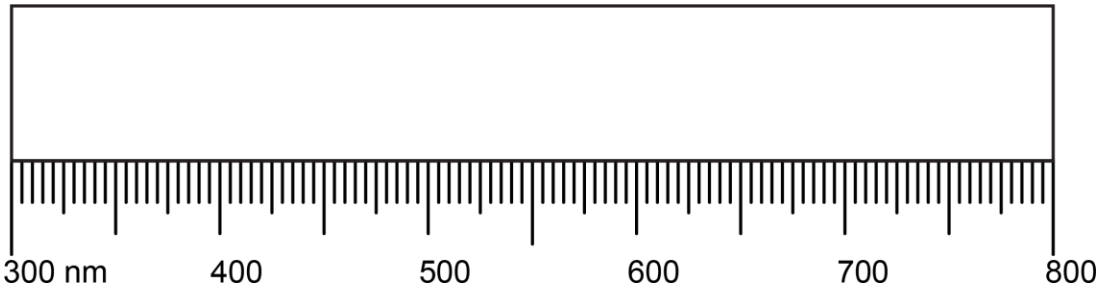
2. Waste containers are in the hood; pay attention to the contents listed on each label!

Using a Spectroscope Data Analysis

Draw the observed spectra for fluorescent and incandescent light in the spaces provided. Use colored pencils toestimate the wavelengths of light that you see.



Report Figure SP.1: Observed spectrum of fluorescent Light

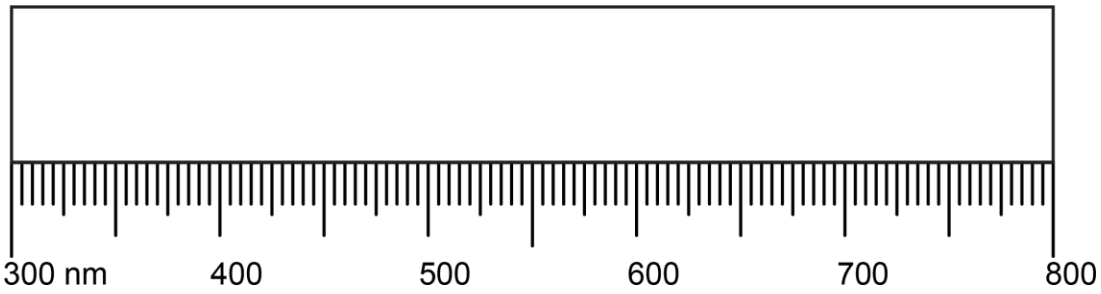


Report Figure SP.2: Observed spectrum of incandescent Light

Atomic Gases Data Analysis

Discharge Tube #1

Element in the first gas discharge tube: _____

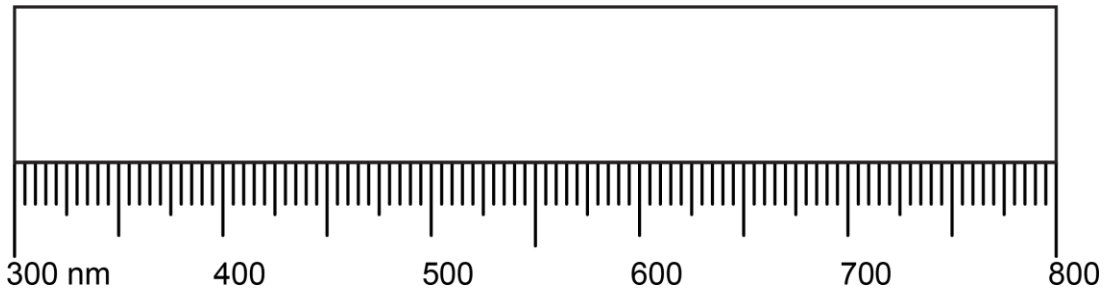


Report Figure SP.3: Observed spectrum of first gas discharge tube

Description:

Discharge Tube #2

Element in second gas discharge tube: _____

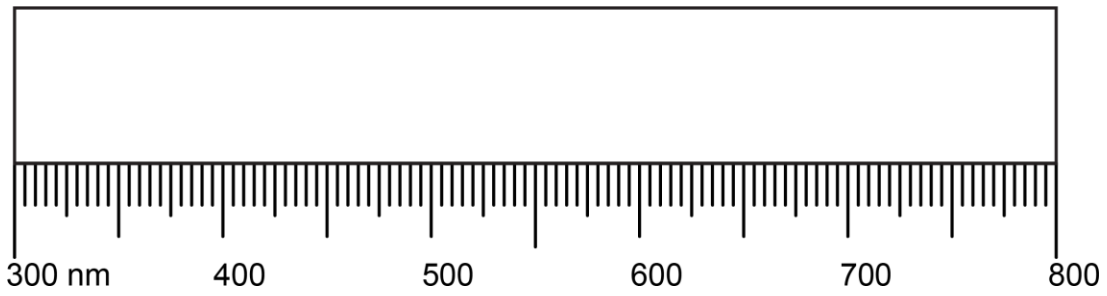


Report Figure SP.4: Observed spectrum of second gas discharge tube

Description:

Discharge Tube #3

Element in the third gas discharge tube: _____



Report Figure SP.5: Observed spectrum of third gas discharge tube

Description:

The known values for Helium electron transitions are given in the table below. Calculate the theoretical energiesof each wavelength determined for the helium lamp using Equation SP.1. Pay attention to the units! *Show yourcalculations here for the VIOLET line and record the results in the table below!*

Report Table SP.1: Energies associated with various colors of light

	Wavelength (nm)	Theoretical Energy (J)
Violet	447	
Blue	471	
Green	501	
Yellow	587	
Red	668	

Flame Tests Data Analysis

Report Table SP.2: Flame Test Observations

	Observations
LiCl:	
Na	

Cl:

KC

l:

Ca

Cl_2:

SrC

l_2:

$CuCl_2$: