

A LABORATORY MANUAL  
FOR  
HARTNELL CHEMISTRY 22:  
THE SCIENCE OF CHEMISTRY

Fourth Revision



Experiments Adapted for Hartnell College with Permission from  
Victor Krimley's "Introductory Chemistry Laboratory Manual"

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August 2011

For permission to use copyrighted material, grateful acknowledgment is made to  
Dr. Victor Krimsley.

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## ACKNOWLEDGMENTS

I would like to thank the present and past faculty, staff and administration of Hartnell College for their support of this project. Without their interest, this project would not have been possible. I hope that the students taking Chemistry 22 will benefit from our efforts.

I offer special thanks to:

Chanté Davis for writing the original edition of this lab manual with me.

Victor Krimsley, who developed the original experiments and materials that Chanté and I adapted for this laboratory manual. He has generously given his full permission for use of copyrighted materials taken from his text.

Lawrence Yee and Susan Hovde, who developed the safety information included herein.

Crystal Gonzalez for use of the report sheets she created for this lab manual.

College Cost Reduction and Access Act (CCRAA) grant for funding this project.

Amy thanks her parents, who gave her that first Gilbert Chemistry Set when she was in elementary school, and the rest of her family for their understanding of the time required for this project. She would especially like to thank her husband, Albert, for cheerfully submitting to reading the entire manuscript many times over.



# LABORATORY SAFETY

Each laboratory experiment gives you the opportunity to confront the unknown and to gain a better understanding of how the “real” world works. Every experiment holds many secrets waiting for you to discover. These lab exercises were designed to help you grasp a better understanding of the principles discussed in lecture.

The experiments presented in this manual have been designed with your safety in mind. Nevertheless, whenever you work in a chemistry laboratory, potential hazards exist. Knowledge of the most common sources of hazards, as well as the safety precautions routinely observed in the laboratory, will help to avoid any serious accidents. One of the most important things to remember about working in any laboratory is to use common sense and exercise good judgment. The sheer number of students working, and the number of lab set-ups necessitate vigilance and care at all times. Use logical precautions, and read and understand the instructions for each lab exercise before attempting the experiment. If at any time you have a question as to what is to be done, or how a piece of equipment is used or the proper way to use and dispose of chemicals ASK YOUR INSTRUCTOR. Most procedures, instructions, and precautions are just common sense practices.

## SAFETY EQUIPMENT

Several pieces of safety equipment are found in most laboratories. You should be aware of the safety equipment that is available in your laboratory, where is located, and how to use it. Many instructors like to quiz beginning students about the location and use of such equipment. Look in **Appendix B** for locations.

**EYE PROTECTION AND SAFETY GOGGLES:** Your eyes are one of your most precious sensory organs, and you should protect them at all times when you are in the laboratory, even if you are not doing any experimental work yourself. Safety goggles **MUST** be worn for each lab session when **ANYONE** in the lab is working with chemicals or equipment. Chemicals that are handled improperly can splash up, and if such a mishap occurs, these chemicals can get in your eyes. Similarly, glassware can splinter upon heating, flying into your eye, and test tubes can fly out of a centrifuge. However, the harmful effects of such accidents can be virtually eliminated by wearing safety goggles whenever you are in the laboratory. Safety goggles must be worn over regular prescription glasses, even if they have plastic or shatter proof lenses, because of their small size. Sunglasses are not allowed because dark lenses limit vision in the lab. Always wash and dry your hands first before you touch or rub your face, eyes, nose, etc.

**EYEWASH FOUNTAIN:** It is unlikely that chemicals will get in your eyes while you are wearing safety glasses. However, if some chemicals should get in your eyes, most laboratories are equipped with an eyewash fountain. The eyewash fountain in your laboratory is located next to the entrance door to the lab. It resembles a drinking fountain, somewhat, but with two faucets directed towards one another. These faucets flush water over both eyes (with the eyes held open) when the head is held between them. If chemicals splash your eyes, use the fountain to flush them for 15 minutes, thereby washing the chemicals out of the eyes. Always report such an accident to your instructor, who may wish to have you see a doctor.

**SAFETY SHOWER:** In your laboratories, the safety shower is found next to the eyewash fountain. It showers large quantities of water on an individual using it. It may be used to put out fires or to douse a person who has suffered a large chemical splash over their body. Never activate a safety shower except in an emergency.

**FIRE EXTINGUISHER:** Look around the room to locate the fire extinguisher nearest you in your laboratory. In M23 and M24 there is one located next to the balance room door. In M26 there are three fire extinguishers. One is on the wall between the windows and the hoods, the second is on the wall next to the bulletin board,

and the third is on the side of the counter next to the eyewash fountain. Most fire extinguishers contain carbon dioxide under pressure and can be used to smother most fires. To use most fire extinguishers, open the door of the cabinet, lift the extinguisher off the hook, pull out the safety pin from the handle, point the nozzle at the fire, and squeeze the handle. Be sure you know how to use the particular type of fire extinguishers found in your laboratory.

**FIRE BLANKET:** The fire blanket is in a long, metal box secured to the wall. A fire blanket may be used to wrap around a victim, who has caught fire, thereby smothering the fire. In M24 the fire blanket is on the wall next to the balance room door. In M26 the fire blanket is on the window wall between the windows and the bulletin board.

**FIRST-AID KIT:** For M23 and M24 there is a first-aid kit in the preparatory room between the two rooms on top of the file cabinets. For M26 there is a first-aid kit secured to the wall in the preparatory room containing the ice machine. *Always report any injury requiring use of the first-aid kit to your instructor* because follow-up treatment may be necessary.

**FUME HOODS:** Six fume hoods are located along one wall in each of the labs. Each hood consists of a partially enclosed laboratory bench equipped with exhaust fans that draw fumes out of the area and expel them into the open air above the building. Any experiments you do that produce toxic fumes should be carried out in a fume hood. Your instructor or the experiment directions usually tell you which experiments require this, but any time an obnoxious or choking odor is produced, you need not wait to move over to the hood to do your work. If your nose does not like the smell of your lab work, it is likely that your body will not like it either. Always use the fume hood in such situations.

**GLOVES:** When working with caustic chemicals, it is a good idea to wear protective gloves. Gloves are provided in all the labs. If you are in doubt, ask your instructor.

## **MISCELLANEOUS HAZARDS**

There are several types of hazards that are commonly encountered in the laboratory. If you and your fellow workers are careful and follow the directions given in this manual or by your instructor, you will find the laboratory a very safe place. However, the remarks accompanying each hazard will prepare you in the event that any of the following accidents should occur.

**FIRES:** Fire rarely occurs in a freshman chemistry lab, but you need to be prepared in case a fire does occur. Fires are more apt to occur in organic chemistry labs, because of the flammable solvents with which one is working. Minor fires can be extinguished by simple means, such as simply allowing it to burn out, if not much solvent is involved, or immediately dousing with water in your lab sink. However, fire extinguishers should be used without hesitation for more serious fires. If your clothing catches fire, the safety shower or the fire blanket should be used. If you see another person's clothing catch fire, immediately push him/her under the safety shower and douse him/her until the fires is completely extinguished. Alternatively, the fire blanket can be wrapped around the victim to smother the fire.

**CHEMICAL SPILLS: ACIDS, BASES, AND OTHER CAUSTIC CHEMICALS:** Occasionally, you may spill a caustic chemical on yourself. If the chemical is spilled in a relatively small amount, simply flush the exposed area for several minutes with tap water from a sink. If a burning sensation accompanies the spill, report it immediately to your instructor, who may recommend further action or send you to a doctor if the burn seems serious. Whenever a burning sensation accompanies a chemical spill, be sure to inform your instructor. Some chemical burns begin with only a minor burning sensation, but develop into a more serious injury if not treated promptly. Your instructor will be able to identify whether you have come into contact with such chemicals.

Small amounts of chemicals spilled on lab benches and other surfaces can be wiped up with a paper towel. Dispose of the contaminated paper towel properly – not in the trash. Larger amounts may need to be neutralized before being removed. See your instructor for proper removal of large amounts of chemicals. Any spill should be cleaned up immediately. Even a small amount may harm another student, who inadvertently touches it and then does not know how to treat the resulting chemical burn, because he/she does not know what it is.

**DILUTING CONCENTRATED ACIDS:** Whenever preparing solutions of dilute acids from a more concentrated solution, always add the concentrated acid to water; never the reverse. When water is added to concentrated acid, the solution will become extremely hot and may spatter acid on the worker. Spattering is much less likely to occur if the acid is added slowly to the water. Recall the mnemonic expression "when you're doing what you oughter, add the acid to the water."

**INSERTING GLASS TUBING IN STOPPERS:** Whenever inserting glass tubing into a hole in a rubber stopper or cork, be sure that the hole is the proper size. In addition, the use of either glycerol (glycerin) or soap to lubricate the end being inserted will help the glass to slide freely. Never force a piece of glass tubing into a hole. The glass may snap, and the jagged edges on the broken glass can cause a serious cut, requiring stitches. Instead, hold the glass near the end being inserted, and twist the glass into the hole.

**SPATTERING FROM TEST TUBES:** When heating solutions in a test tube, spattering may occur. Therefore, never point a test tube being heated toward another person. To minimize the danger of splattering, heat the test tube near the liquid surface, and agitate the contents back and forth.

**HOT EQUIPMENT:** Use a wire gauze or heat resistant pad under the object. Do not place directly on the bench. The object may stick, become contaminated with the bench surface, or cause damage to the bench surface.

#### GLASSWARE:

1. Broken glassware should be immediately cleaned up using the dustpan and brush in the tall cabinet near the entrance door.
2. To help prevent breaking glassware, never try to dry graduated cylinders, burets, or volumetric flasks with your Bunsen burner. They are not made of resistant glass, as are beakers, test tubes, and Erlenmeyer flasks.
3. To help prevent burns, remember hot glass looks the same as cold glass.

**FOOD AND DRINK:** Any and all foods and drinks are prohibited in a chemistry lab. Never taste any chemical and consider all chemicals hazardous unless instructed otherwise. Always wash your hands with soap and water before eating after working in the chemistry lab. There is a table located outside the labs for you to leave your food and drink.

**DETECTING ODORS:** When detecting odors in the laboratory, never inhale the odors directly, as they may be quite strong. Instead, using your hand, waft the odors gently toward your nose in a controlled fashion. To avoid over-inhalation of the fumes, partially fill your lungs with air before inhaling the odors.

**TASTING:** Never taste anything prepared in a chemistry laboratory. Newly prepared chemicals all have the potential for toxicity. Unless we know that the equipment being used and the substances being prepared are absolutely free of toxic chemicals, we never risk the danger of tasting substances prepared in the laboratory.

**ORGANIC CHEMICALS:** Many commonly used organic compounds turn to vapor easily and also tend to burn easily or explode, if used around flames. When organic solvents are used, we normally do not use open flames (Bunsen burners). We use hot plates or sand baths. Be sure you know where the fire extinguishers are located in the lab, just in case.

**HORSEPLAY:** The laboratory is no place for horseplay, because there is always a danger of breaking or spilling something. Fooling around in the laboratory is an invitation for serious accident.

## **ACCIDENTS**

1. Clean up all spills and broken glass immediately.
2. If a mercury thermometer breaks, do not touch the mercury and notify lab staff immediately. Broken alcohol thermometers may be treated like normal broken glass.
3. In case of contact with a chemical, wash the affected area immediately and thoroughly with water. Notify lab staff.
4. OOPS: Always report ANY injury, accident, or spill, no matter how minor, to your instructor or laboratory staff immediately!

## **GENERAL LABORATORY PROCEDURES AND CONDUCT**

Anyone working in the chemistry laboratory must follow these procedures and practices for laboratory safety. This includes students, instructors and laboratory staff. Disregard of these procedures will result in disciplinary action.

1. An INSTRUCTOR must be present, whenever you work in the lab. If it is not a lab in which you are scheduled, you must get the instructor's permission.
2. Protective goggles or safety glasses with side shields must be worn at all times in the laboratory when performing or observing an experiment or when near others performing experiments.
3. Learn the primary, secondary and handicapped escape routes from the laboratory.
4. Learn the locations of the fire extinguishers, safety shower, eyewash stations, fire blankets, and hoods.
5. Never perform unauthorized experiments.
6. Eating, drinking, and smoking in the laboratory are forbidden. Do not bring food or drink into the laboratory.
7. Become familiar with the use and operation of laboratory equipment and instruments. Drawings of common laboratory equipment may be found in Appendix I.
8. Never taste a chemical.
9. If instructed to smell a chemical, do so by gently fanning the vapors toward your nose.
10. Never point a test tube that is being heated toward yourself or others.
11. Never pipette by mouth. Pipette filler bulbs are available and their use will be demonstrated when appropriate.
12. Never carry a reagent bottle to your desk because other students will not be able to find it. Carry liquids in clean glass containers and solids in weighing boats or on weighing paper.
13. Read chemical labels carefully, TWICE. Be sure that you are using the chemical required.

14. Take only the amount of chemical from the reagent bottles needed for an experiment. Do not return unused chemicals to reagent bottles. This will prevent contamination.
15. Dispose of excess hazardous chemicals in designated containers. Dispose of nonhazardous solids in wastepaper baskets, not in the sink. Nonhazardous liquids can go in the sink. If in doubt, ask your instructor or stockroom personnel.
16. When pouring liquids out of a bottle hold the palm of your hand over the label to prevent possible drops from running down the bottle and defacing the label. Always clean any drips on the bottle when you have finished pouring. This also helps to protect your hand from chemicals spilled on the sides of the bottle.
17. When obtaining chemicals, use the spatula or dropper provided and taped in a test tube to the side of the bottle. Be sure to replace the spatula or dropper in the holder on the bottle from which it came, so you do not accidentally contaminate the contents by using the wrong spatula or dropper.
18. Do not lay stoppers down on the counter. This allows them to become contaminated. Always replace caps and stoppers to the original bottle. Always recap tightly immediately after use, since moisture in the air can affect many of the chemicals.
19. Footwear should cover the feet completely. No open-toed shoes are allowed. Clothing should cover the body to the knees. Long pants are preferred. Long hair and loose clothing should be secured. Wear appropriate clothing.
20. When diluting, ALWAYS add acid to the water.
21. READ the lab experiment procedures before coming to lab.
22. WORK independently, unless instructed to do otherwise.
23. RECORD DATA directly into your report tables, report sheet, or notebook in ink. Do not recopy from another piece of paper. If you make an error, neatly cross the data out with a single line, so it may still be legible, and write in the corrected data nearby. Do not use “white out” to obscure mistakes.
24. BALANCE: A balance is a fragile, expensive, and accurate piece of equipment. Never place chemicals directly on the balance pan. Use a weighing boat, piece of paper, paper cup, or piece of binder paper between the chemical and the balance pan. Weigh objects at room temperature. Do not weigh hot or warm objects.
25. EQUIPMENT AND DRAWER: You are responsible for keeping your equipment and drawer clean during the semester. It must also be clean when your turn in everything at the end of the semester or if you drop the class. A \$25 fine will be assessed if you drop the class and do not return your locker.
26. Be economical in the use of reagents, deionized water, and detergent.
27. When the experiment is completed, wipe the laboratory table; clean and dry equipment; compare the equipment and chemicals in the tray with the checklist; when complete, return materials you have checked out to the stockroom. Return all ring stands, hot plates, Bunsen burners, etc. to their proper places. Keep balances clean. If you spill chemicals on a balance, immediately clean them off to prevent damage.

## **EMERGENCY EVACUATION**

You need to know the possible exits from your laboratory, in case of an emergency evacuation. Follow your instructor's directions to safely evacuate the building. Remember to use the stairwells and not the elevator.



## KEEPING A LABORATORY NOTEBOOK

Notebooks, or other formally kept records, are essential tools in many professions, e.g., crime scene investigators, field biologists, medical professionals, researchers, or safety inspectors. Whatever field you decide upon for your career, keeping accurate and objective records is often critical. Learning the good habits required to keep a laboratory notebook takes sustained effort, but remember that what you learn about keeping records will be useful in the future, even if you do not become a scientist. Your laboratory instructor will have specific requirements and formats for keeping your laboratory notebook. Following are some general guidelines to help you develop good record-keeping skills.

1. Your laboratory notebook is meant to be a permanent document. It is intended that you write all your observations and records directly in the notebook. Do not write notes on pieces of paper and transfer them later into your notebook.
2. As a permanent record, do not tear pages out of your notebook. If you make errors - just cross out errors neatly. Do not obscure errors with “white-out” or markers. Your instructor should be able to see the original information that was written.
3. All records must be written in blue or black permanent ink. Your records should be neatly and legibly written or printed.
4. Note the date on each page. Data and measured quantities should be clearly labeled and written with units. Calculations used to generate results should always be written out.
5. If your instructor makes changes to the laboratory manual procedures, these should be noted in your notebook.
6. Your laboratory notebook should contain enough details of the experimental procedures and explanatory details that someone else could duplicate your results. Ask yourself - “If I only had my laboratory notebook and not my lab manual, could I carry out this experiment?”



# EXPERIMENT 1: BASIC MEASUREMENT TECHNIQUES

## Objectives

1. Use common laboratory measurement devices for determining length, mass and volumes of various materials. These skills are important to learn for this and other experiments.
2. Find the density of unknown liquids and solids using these basic measurement techniques.



## Materials and Equipment



Ruler, meter stick, 10 mL graduated cylinder, 25 and 50 mL graduated cylinders, small and large test tube, 50 mL and 150 mL beakers, 50 mL Erlenmeyer flask, digital top-loading balance, unknown liquids for density determination, irregular solids for density determination, safety glasses.

## Introduction

The International System of Measurement (SI) is used worldwide and has been adopted as the official system of measurement by most countries. It is commonly called the *metric system*. Our traditional American/English system of measurement (e.g., miles, quarts, pounds) requires many conversion factors. Take length, for example – there are inches, feet, yards, rods, chains, and miles! The metric system is much different. It is based on standard units that can be easily converted by multiplying or dividing by factors of ten. Engineers and scientists most often use these standard metric units: the meter, for length; the gram, for mass (or weight); the liter, for volume; and the degree Celsius (or less often Kelvin) for temperature.

## Estimating and Uncertainty

Whenever you take a scientific measurement, you are making a *quantitative* observation. When you report your data, you usually are estimating the last *significant figure*. You can look at Appendix E if you need help with significant figures. In other words, you will report the digits in the measurement that you are certain about plus one additional digit that are you allowed to estimate. Here are typical uncertainties of common measuring devices:

Measuring Device	Uncertainty
12 cm ruler	$\pm 0.05$ cm
triple-beam balance	$\pm 0.05$ g
analytical balance	$\pm 0.0001$ g
10 ml graduated cylinder	$\pm 0.05$ ml
100 ml graduated cylinder	$\pm 0.5$ ml
50 ml buret	$\pm 0.02$ ml
25 ml volumetric flask	$\pm 0.02$ ml
25 ml transfer pipet	$\pm 0.02$ ml

The last digit in your measurement is the digit that is considered *uncertain* because it is estimated. For example, if you are measuring the length of a piece of metal with a ruler that has

marks down to units of 0.1 cm, you could estimate the length down to the 0.05 cm, in other words, between tenths of a cm. The number of significant figures gives us an idea of the accuracy of a measuring device. In this and future labs in this class, you will be expected to keep track of significant figures for performing calculations and reporting results. Appendix F provides rules for rounding off the results of calculations with significant figures.

### Accuracy and Precision

In common English, we often use the terms “accuracy” and “precision” interchangeably, to indicate how “correct” an answer is. However, in science the two terms have different meanings. *Accuracy* is a measure of how closely an observation is to the “true” or “accepted” value.

*Precision* is a measure of how closely a group of observations are to one another. If you think about a dartboard, “accurate” would be hitting the bulls-eye or center of the target; “precise” would mean that all of your darts hit the target close to one another, without reference to whether or not you hit the bulls-eye. So, it is possible to be precise (all the darts close together) but not accurate (missing the bulls-eye). Of course, we would like to be both accurate and precise in our laboratory measurements. Appendix D (Useful Math Relationships) gives you some mathematical ways of reporting accuracy and precision.



### Random and Systematic Errors

As we can see from the information above, each measurement has a certain amount of uncertainty associated with it, which means that each measurement has a certain amount of error. Errors refer to the calculated difference between a measured value and the “true” value. There are actually two kinds of error: *random* error and *systematic* error. *Random errors* result from the uncertainty of your measurement device. They are not caused by a mistake in your technique, but are caused by unpredictable or imperceptible factors that are beyond your control as an experimenter. An example would be two different experimenters determining the mass of an object using two kinds of balances that have different sensitivities for mass.

Errors that have definite causes are called *systematic errors*. In general, systematic errors are generally reproducible and will result in values that are always higher than the true value or lower than the true value. An example would be a thermometer that is not calibrated correctly and always gives a reading that is lower than the actual or true value.

Random errors are always present – but you want to eliminate or minimize the systematic errors in carrying out your experiments.

## Procedures

### Part 1. Length Measurements

Measure the length and width of your laboratory notebook in centimeters. Record your observations. Convert these measurements into millimeters and into inches. Conversion factors are included in Appendix A.

### Part 2. Temperature Measurements

1. Determine the Celsius temperature in the laboratory by reading your thermometer. Record your observation. Convert this temperature to Kelvin and its Fahrenheit equivalent. Note: it is not necessary to “shake down” a laboratory thermometer to read the temperature it is measuring.
2. Prepare an ice water bath by filling a 150 mL beaker with a 50:50 mixture of ice and deionized water. Carefully immerse your thermometer in the ice bath and stir gently without hitting the sides of the beaker. Record the lowest temperature. Note: if your temperature reading differs from 0°C by more than two degrees, let your lab instructor know.

### Part 3. Volume Measurements



1. Look at this diagram of a 50 mL graduated cylinder. You will see that the surface of a liquid in a cylinder forms a curved surface. This surface is known as the meniscus. The arrow is pointing to the meniscus in the graduated cylinder. The very bottom of the meniscus is where you will take your readings. Make sure that the meniscus is at your eye level to avoid systematic errors.
2. Fill a 50 mL graduated cylinder about half full with deionized water and make a sketch of the graduated cylinder and meniscus you observe. Label your sketch with the level at which you would take your reading. Empty the graduated cylinder.
3. Next, take a small test tube and fill it to the very top with deionized water. Transfer (pour) the water into a 10 mL graduated cylinder. Record the volume of water that the small test tube contained. Empty the graduated cylinder.
4. Now fill a large test tube to the very top with deionized water. Transfer the water into a 50 mL graduated cylinder and record the volume. Empty the graduated cylinder.
5. Fill your 50 mL beaker to the 40 mL mark with deionized water. Pour the water into a 50 mL graduated cylinder and record the volume. Empty the graduated cylinder.
6. Repeat step 5 with a 50 mL Erlenmeyer (conical) flask. Remember to record the volume.

## Part 4. Mass Measurements



1. You will be using digital top-loading balances in this laboratory. Your instructor will provide you with detailed instructions for the particular balances you have in your laboratory. Here are some general guidelines for using any scientific balance.
  - a. Never move the balance from where it has been placed. It has been calibrated for a particular location and has been leveled to read accurately.
  - b. Never place anything wet or hot on the balance. Everything to be weighed must be at room temperature. Protect the top pan of the balance with a sheet of weighing paper or a plastic “weigh boat”.
  - c. Be gentle when adding or removing an object from the pan of the balance. It is a delicate piece of equipment.
  - d. Keep the balance and area around it clean. There are small brushes and wipes provided for this purpose.
  - e. Always use the same balance throughout an experiment for better results.
2. Weigh a large test tube, a 50 mL beaker, and a 150 mL beaker on a digital top-loading balance. Record your data.

## Part 5. Density Determinations

**Proper Waste Disposal:** recycle the unknown liquid as directed by your laboratory instructor. There sh

1. Density is calculated using the following formula

$$d = \frac{m}{V} \quad \text{where } d \text{ is density in g/mL, } m \text{ is mass in g, } V \text{ is volume in mL.}$$

2. Weigh a dry 10 mL graduated cylinder. Record the weight. Measure about 2 mL of the unknown liquid provided by your instructor into the pre-weighed graduated cylinder. Now reweigh the graduated cylinder. Record both the **actual** volume of liquid contained in the graduated cylinder and the weight of the graduated cylinder with the liquid. Calculate the density of the unknown liquid using the formula above. Empty the unknown liquid into the recycling container and dry the graduated cylinder.
3. Repeat step 2, adding about 10 mL of the unknown liquid this time. Record your data as in step 1, calculate the density of the unknown liquid, empty the unknown liquid into the recycling container and dry the graduated cylinder.
4. Report the density reported to your instructor and obtain the accepted density of the unknown liquid. Calculate the percent error for each of the densities you calculated in steps 2 and 3. Use the equation in Appendix D.
5. You will next use the following steps to determine the density of an irregularly shaped solid. The method uses a technique known as *volume by displacement*.
  - a. Obtain an unknown solid from your instructor. Record the unknown number. Remove the solid from its container and measure its mass on a balance. Record this mass.

- b. Next fill a 25 mL graduated cylinder about halfway with water and record the volume. You want the level of the water in the graduated cylinder to be higher than the height of your solid object.
- c. Carefully tilt the graduated cylinder so you can gently slide your object into the cylinder. Don't let it drop, as you can break out the bottom of the cylinder. Record the new volume. The difference between the volume in step 5b and in step 5c represents the volume of the irregular solid.
- d. Calculate the density of the irregular solid using the density equation given above.

**Clean Up:** Dry the solid unknown with a paper towel and place it back into its container. Return this to your instructor.

### Observations and Data

Make sure to record the following information in your lab notebook or on your report sheet.

#### Part 1. Length Measurements

1.  Length of notebook in centimeters
2.  Width of notebook in centimeters

#### Part 2. Temperature Measurements

1.  Room temperature in °C
2.  Ice bath temperature in °C

#### Part 3. Volume Measurements

1.  Sketch of the graduated cylinder, meniscus and indication where the volume is to be read
2.  Volume of water from small test tube
3.  Volume of water from large test tube
4.  Volume of water from 50 mL beaker
5.  Volume of water from 50 mL Erlenmeyer flask

#### Part 4. Mass Measurements

1.  Type of balance used
2.  Mass of large test tube
3.  Mass of 50 mL beaker

- Mass of 150 mL beaker

#### Part 5. Density Determinations

Data for 2 mL of the unknown liquid:

- Mass of empty 10 mL graduated cylinder
- Approximate volume added
- Actual volume added
- Mass of cylinder plus unknown liquid
- Accepted value for the unknown liquid

Data for 10 mL of the unknown liquid:

- Mass of empty 10 mL graduated cylinder
- Approximate volume added
- Actual volume added
- Mass of cylinder plus unknown liquid
- Accepted value for the unknown liquid

Data for the unknown solid:

- Unknown number
- Mass of irregular solid
- Volume of water in graduated cylinder
- Volume of water plus object

#### **Calculations**

You will need to carry out the following calculations in your lab notebook or on your report sheet. Show your work.

#### Part 1. Length Measurements

- Length of notebook in inches
- Width of notebook in inches

3. Length in millimeters

4. Width in millimeters

#### Part 2. Temperature Measurements

1. Room temperature in K

2. Room temperature in °F

3. Ice bath temperature in K

4. Ice bath temperature in °F

#### Part 5. Density Determination

1. Density of the unknown liquid based on a 2 mL sample and percent error

2. Density of the unknown liquid based on a 10 mL sample and percent error

3. Density of the irregular solid

#### Questions

1. What is the mathematical relationship between mL and  $\text{cm}^3$ ?

2. Which of the following measurement devices do you think would be accurate enough to use for precise measurement of volumes: 50 mL beaker; 50 mL Erlenmeyer flask; 50 mL graduated cylinder?

3. Suppose a student makes an error of 0.1 mL in measuring 1.0 mL of liquid, i.e., he records the value as 1.1 mL when it is actually 1.0 mL. What is the percent error? Show your work.

4. Suppose another student makes an error of 0.1 mL in measuring 10.0 mL of liquid, i.e., she records 9.9 mL when it is actually 10.0 mL. What is the percent error? Show your work.

5. Based on your answers to questions 3 and 4 above, which of the determinations of density for your unknown liquid would you expect to be more accurate – the 2 mL sample or the 10 mL sample?

6. Which of your two values actually was more accurate?

#### Additional Resources

Check Appendix M for additional resources for this experiment.

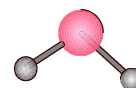


## EXPERIMENT 2: MOLECULAR MODELS

### Objectives

1. To acquire skills in understanding molecular bonding, structure, and isomerism.
2. To use Lewis structure and molecular models to predict polarity, molecular geometry, relative water solubility and hydrogen bonding capability.
3. To use molecular models to study classes of organic compounds.

### Materials and Equipment



Molecular model sets

COLOR CODE FOR MODELS	
COLOR	ATOM REPRESENTED
Black	Carbon (or any atom where four bonds are needed)
Yellow	Hydrogen (1 bond)
Red	Oxygen (2 bonds)
Blue	Nitrogen or phosphorous (3 bonds)
Green	Chlorine (1 bond)
Purple	Iodine (1 bond)
Orange	Bromine (1 bond)

### Introduction

Elements that are classified as non-metals tend to form **covalent bonds** through sharing electrons between other non-polar elements. Covalent bonds are strong and very stable. A particularly stable structure is one in which all of the atoms have a share in eight **valence** electrons (with the exception of hydrogen, which can share a maximum of two electrons). It is important for you to understand and appreciate that atoms and molecules have three-dimensional shapes, since most drawings seen in texts and lab manuals try to show three-dimensional forms, which still look flat. A study of the subtle differences in the three dimensional nature of molecules will be useful to help you understand why some chemicals are “food”, others pass through the body untouched as “non-food”, and others may be acutely poisonous. The geometry of a molecule influences whether it is polar or non-polar. The geometry of a molecule is influenced by the number of valence electrons which an atom has, and whether those electrons tend to be involved in bonding, or whether they tend to remain unshared, and non-bonding. If the electrons are non-bonding, then they tend to try to be as far apart as possible, and so satisfy the valence shell electron pair repulsion rule (VSEPR).

An important characteristic of molecular compounds is that of polarity. A bond between two unlike atoms is always polar, however the molecule as a whole may be **nonpolar** if the complete molecule is **symmetrical**. A molecule is **polar** if it is structurally **asymmetrical**, i.e., the molecule is composed of two different elements, or the atoms are unevenly arranged around the central atom. HF would be polar since two elements are joined by a covalent bond, but the electrons are not shared equally. H<sub>2</sub> would be nonpolar and symmetrical, since both atoms of the molecule are of the same element, so there is equal sharing of the electron cloud. Polar molecules can also be made of more than two elements and more than three atoms. In all cases,

the degree (or amount) of polarity depends on the position of the atoms which are unevenly arranged around the central atom.

Molecules are definitely three-dimensional. They have a characteristic shape, form, bulk, and many of their properties result from their bulky shapes. Unsaturated lipids are usually liquid at room temperature, because the molecules do not fit closely next to each other, hence have less molecular attraction. The shape and polarity of molecules, as well as its total molecular mass, determines the melting point and boiling point of a molecule, as well as solubility properties.

Water is a very polar molecule because it has two unshared pairs of electrons, and two hydrogen atoms covalently bonded to an oxygen atom. The oxygen atom is strongly electronegative so it strongly attracts the electron clouds on the hydrogen atoms. In doing so, the hydrogen atoms have less of the electron cloud and develop a partial positive charge, noted by the symbol  $\delta^+$ . The oxygen, having taken most of the electron cloud from the hydrogen atoms, now is indicated by  $\delta^-$ . Molecules that are polar tend to dissolve in water, whereas those that are nonpolar tend to be repelled by water. If one was to compare molecular compounds of a similar molecular mass, their properties would be very different depending on whether they are nonpolar, polar, or capable of dissociating, as organic acids are capable of doing.

You will be using atomic models for this exercise. Use the same size sticks between atoms of the same color in order to preserve relative shapes of the molecules. If you predict a double bond between atoms, use two springs to demonstrate the double bond (sticks do not bend very well). A triple bond would require three springs.

In constructing models of molecules, draw the Lewis dot structure first. Then put the molecule together. As an example, in constructing a molecule of water, connect an oxygen atom to two hydrogen atoms. Note the angle between the hydrogen atoms, and examine the molecule from the front angle and from the side. Many texts draw the molecule from the “front” so that the hydrogen atoms appear to be symmetrical in relation to the oxygen. If you examine the molecule from the side, you will see that the molecule is distinctly asymmetrical due to the unseen unshared pairs of electrons. You might wish to remake the molecule using a black “carbon” atom that has four evenly spaced holes. Place two hydrogen atoms onto the central “oxygen” as before, and then place two short sticks into the unused two holes to represent the unshared pairs of electrons. In this way you can fully appreciate the importance of unshared pairs of electrons in the geometry of a molecule.

A study of molecular geometry and polarity helps one understand how polyatomic ions “happen”. Polyatomic ions are difficult to remember and use in reactions (at least on paper), and yet are constantly being used in chemical experiments. This exercise will help to show that they are logically constructed, and make sense on a chemical level.

The following table may help you decipher some of the material explained above.

### Five Types of Molecular Geometry

Molecular Geometry	Lewis Structure	Number of Atoms Bound to Central Atom	Number of Lone Pairs on Central Atom	3-D shape
<b>Linear</b> 180°		1		H—Cl
<b>Linear</b> 180°		2	0	O—C—O
<b>Bent</b> (Angular or V-shaped) 120°		2	1	
<b>Bent</b> (Angular or V-shaped) 109° (105°)		2	2	
<b>Trigonal Planar</b> 120°		3	0	
<b>Trigonal Pyramidal</b> 109° (107°)		3	1	
<b>Tetrahedral</b> 109°		4	0	

### Procedure

1. The formula of the molecule to be constructed is listed in the first column.
2. Using the formula, draw its Lewis structure (include pairs of dots to indicate any unshared pairs of electrons).
3. Assemble each model using the colored balls and draw the shape in the space for three-dimensional (3-D) shape and geometry.

4. Write the name of the geometrical shape which you predict from building the model, and tell whether the molecule has polarity or not (nonpolar or polar). Polarity is based on molecular asymmetry. If the molecule looks the same from all angles, it is symmetrical and therefore nonpolar. If one side is different from the rest, it is asymmetrical, and therefore is polar.

Using a similar set-up and spacing as in the example on the report form below. In each row, next to the formula for each molecule, draw its Lewis structure and 3-D shape as seen from your model, then determine the geometry, and decide whether it is polar or not. Ask for help if you are having difficulty.

### Observations and Data

FORMULA OF MOLECULE	LEWIS STRUCTURE	3-D SHAPE (show lone pairs)	MOLECULAR GEOMETRY	MOLECULAR POLARITY
example: CH <sub>4</sub>	<pre>       H             H-C-H               H           </pre>		tetrahedral	nonpolar
H <sub>2</sub> O				
H <sub>3</sub> O <sup>+</sup>				
C <sub>2</sub> H <sub>6</sub>				
C <sub>2</sub> H <sub>4</sub>				
C <sub>2</sub> H <sub>2</sub>				
CO <sub>2</sub>				
H <sub>2</sub> O <sub>2</sub>				
CH <sub>3</sub> Cl				

FORMULA OF MOLECULE	LEWIS STRUCTURE	3-D SHAPE (show lone pairs)	MOLECULAR GEOMETRY	MOLECULAR POLARITY
CH <sub>2</sub> Cl <sub>2</sub>				
CHCl <sub>3</sub>				
CCl <sub>4</sub>				
H <sub>2</sub> S				
NH <sub>3</sub>				
NH <sub>4</sub> <sup>+</sup>				
Br <sub>2</sub>				
N <sub>2</sub>				
CH <sub>3</sub> OH				
CH <sub>2</sub> O				
HCl				
H <sub>2</sub> CO <sub>3</sub>				

FORMULA OF MOLECULE	LEWIS STRUCTURE	3-D SHAPE (show lone pairs)	MOLECULAR GEOMETRY	MOLECULAR POLARITY
$\text{HCO}_3^-$				
$\text{CO}_3^{2-}$				
$\text{SO}_2$				
$\text{SO}_3$				
$\text{H}_2\text{SO}_4$				
$\text{SO}_4^{2-}$				

### Questions

- Explain why the model for each of the following atoms has a specific number of bonds:
  - Hydrogen: 1 bond
  - Oxygen: 2 bonds
  - Nitrogen: 3 bonds
  - Carbon: 4 bonds
- Discuss the shapes of  $\text{SO}_2$  and  $\text{SO}_3$ . Why is one polar and the other is not.
- Explain why an ion such as  $\text{NH}_4^+$ ,  $\text{CO}_3^{2-}$ , and  $\text{SO}_4^{2-}$  can be nonpolar, but charged?
- Explain how molecules of  $\text{H}_3\text{O}^+$  and  $\text{NH}_4^+$  form from uncharged molecules of  $\text{H}_2\text{O}$  and  $\text{NH}_3$ .

### Additional Resources

Check Appendix M for additional resources for this exercise.

## EXPERIMENT 3: THE GAS BURNER



### Objectives

1. Learn proper procedures for the operation of Tirrill-type burners. You will be using such burners in this and other laboratories.
2. Discover factors influencing combustion.
3. Detect products of complete and incomplete combustion.

### Materials and Equipment

Tirrill type burner, rubber tubing, matches or “striker”, iron ring stand, iron ring, wire gauze, beaker, water, porcelain crucible, crucible tongs, safety goggles.



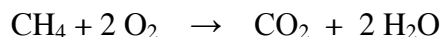
### Introduction

The most commonly used gas burners are the Bunsen or Tirrill types. They are similar in that they both have an air adjustment at the base of the barrel; they differ in that the Tirrill type also has a gas adjustment screw at the bottom of the burner. Any of the Hartnell laboratory burners uses natural gas, which is primarily methane (CH<sub>4</sub>), for fuel. Most people refer to both types of gas burners as Bunsen burners.

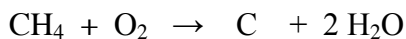
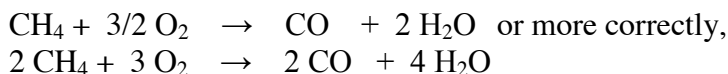
In the burner, methane mixes with air entering through the holes, called “air ports”, in the collar at the base of the barrel. When ignited, the methane then combines with the oxygen in the air during the process of burning, which is called combustion, to form water vapor and carbon dioxide.

If the air ports are partially or completely closed, there may be insufficient air for complete combustion of the methane. Instead of carbon dioxide forming, carbon monoxide or just carbon will form. When carbon forms, as happens when the air ports are completely closed, the burner burns with a yellow sooty flame, called a “luminous flame” because it is so easily seen. When the air ports are open to allow the proper amount of air to enter, the burner burns with a blue, almost invisible, flame, called a “non-luminous flame”. If there is too much air coming in from the air ports, the flame may “roar” or lift up off the barrel or even “strike back”, that is, the flame will travel down the tube and burn at the small opening in the base of the burner. The collar and tube of the burner may get quite hot and you may smell melting or burning rubber from where the rubber tubing attaches to the burner. Turn off the gas supply and partially close the air ports by turning the collar of the burner after it has cooled, then relight the burner.

The balanced chemical equation when the correct amount of oxygen is available for complete combustion of methane to form carbon dioxide and water follows:



The balanced chemical equations for incomplete combustion of methane when there is not enough oxygen for complete combustion, leading to the formation of carbon monoxide or carbon, follow:



## Procedure

1. Observe the gas outlet on the laboratory bench. It is off when it is parallel to the pipe (turned either to the right or to the left.) It is on when it is turned 90 degrees toward you in the perpendicular position.
2. Observe the movable collar at the base of the barrel and the gas adjustment screw at the very base of the burner. Disassemble your burner by unscrewing the barrel and the gas adjustment screw. Draw and label each part of your burner in your lab notebook.
3. Reassemble your burner, attach one end of the rubber tubing to the burner and the other end to the gas outlet. Light the burner as follows:
  - a. Light your match and hold it just to the side of the top of the barrel and open the gas outlet to the perpendicular position.
  - b. Bring the lighted match to the top of the burner from the side and adjust the air supply by turning the collar until a non-luminous flame appears.
  - c. Adjust the screw at the base of the burner until you have a flame with a double bluish cone. The inner cone is a bright light blue, and the outer one a more diffuse and darker blue. You may need to adjust the air again to get the best results.
4. Observe the differences in temperature within the flame by inserting the corner of your wire gauze into various parts of the flame and comparing the amount of time it takes for the wire to glow in the different parts. Test (a) the very tip of the flame, (b) the tip of the inner blue cone and (c) the flame next to the top of the barrel. Record.
5. Place a wire gauze on a ring stand and place a beaker of cold water on the wire gauze. "Dash" the flame of your lighted burner across the side of the beaker once or twice. You should be able to see evidence of one of the products of combustion of methane. Record your observations.
6. Turn the collar of your burner until the air ports are closed all the way. This should produce a yellow (luminous) flame. Hold a cold porcelain crucible in the luminous flame with crucible tongs for about a minute and observe what happens. Record.

Cool and clean the crucible, adjust the collar to open the air ports to produce the blue (nonluminous) flame and repeat the experiment. Observe and record what happens this time.

## Observations and Data

Record the following information in your lab notebook or on your report sheet.

- 1-2.  Sketch your burner. Label the base, the barrel, the collar, the air ports, and the gas adjustment screw.

- 3-4.  Sketch the double non-luminous flame, draw lines to and label the top of the flame, the tip of the inner blue cone, and the base of the flame. Also note the comparative temperatures of these locations according to your observations.
5.  What appears on the glass when the flame is dashed across the beaker of cold water?
6.  a. What occurs when the crucible is held in the luminous (yellow) flame?  
 b. What occurs when the crucible is held in the non-luminous (blue) flame?

### Questions

Refer to the Introduction, the Procedures, your instructor's directions and your observations to answer these questions.

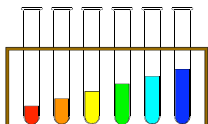
1. Why should you not hold your lighted match over the top of the burner while you turn on the gas?
2.
  - a. Suppose when you lighted your burner, it had a luminous flame. What should you do to get a properly adjusted flame?
  - b. Suppose when you lighted your burner, it had a loud "roaring" sound. What should you do to correct it?
  - c. Suppose when you lighted your burner, the flame disappeared down into the barrel. How do you obtain a properly adjusted flame?
3.
  - a. What product of combustion is shown to be present by the results of dashing the flame across the cold beaker surface?
  - b. What product of combustion is shown to be present by the results of holding the crucible in the luminous flame?
  - c. Why did the deposit in b not appear when you held the crucible in the non-luminous (blue) flame?

### Additional Resources

Check Appendix M for additional resources for this experiment.



## EXPERIMENT 4: DETERMINING CHEMICAL PROPERTIES TO SEPARATE A MIXTURE



### Objectives

1. Investigate chemical and physical properties of pure chemical substances.
2. Look up information about the physical properties of pure substances in the “Handbook of Chemistry and Physics”.
3. Use information gained through observation and resources to separate the components of a mixture.

### Materials and Equipment

Dilute sodium hydroxide (6M NaOH)

Dilute hydrochloric acid (6M HCl)

Magnesium metal (Mg)

Zinc, granular (Zn)

Copper (II) carbonate ( $\text{CuCO}_3$ )

Sodium chloride (NaCl)

150 mL beaker, 250 mL beaker, test tubes, vacuum filtration apparatus (filter funnel and filter flask assembly), filter paper, wash bottle, glass stirring rod, safety goggles.



Copper metal (Cu)

Magnesium oxide (MgO)

Copper (II) nitrate ( $\text{Cu}(\text{NO}_3)_2$ )

unknown mixture of  $\text{CuCO}_3$  and NaCl

### Introduction

A property of a substance is a distinguishing characteristic that can be used to describe that material. Common physical properties of pure substances include boiling point, color, density, hardness, melting point, odor, solubility in different solvents, and taste. Measuring a physical property does not involve any chemical changes in the substance. Chemical properties describe the substance's ability to undergo chemical change. In other words, a chemical reaction has to occur. A common chemical property of a pure substance is flammability or its ability to combine with oxygen. These physical and chemical properties can be used to help identify a compound or determine its purity.

In this experiment you will examine several physical and chemical properties for certain elements and compounds. You will then be asked to separate two compounds mixed together, using the information you have collected.

### Procedure

Part 1. Observation of Chemical and Physical Properties

**Proper Waste Disposal:** Dispose all waste solids and solutions as directed by your instructor.

1. Obtain 2 mL of dilute hydrochloric acid in a clean test tube and 2 mL of dilute sodium hydroxide in a second clean test tube. You will use these test tubes as a source of the two

chemicals used in testing the following elements and compounds: magnesium, copper, zinc, magnesium oxide, copper (II) carbonate, copper (II) nitrate, and sodium chloride.

2. Carry out each of the following steps for each of the materials to be tested. Complete all the testing steps for each material before proceeding to test the next material. Record your data in a table for ease of reading.
  - a. Place several crystals (no more than a grain of rice) of the substance to be tested in each of four clean small test tubes. Examine and record the appearance.
  - b. For test tube 1:  
Heat the first test tube and observe the effect of heat, if any, on the substance. Notice if you see any gas being evolved. Remember to record your observations.
  - c. For test tube 2:  
Add about 2 mL of deionized water from your wash bottle to the sample in the second test tube. Stir with a clean glass rod. Observe the solubility of the material in cold water. Record your observations.  
Gently heat the water just to boiling in the test tube to observe the solubility of the material in hot water. Record your observations.  
Allow the solution to cool and test it with litmus paper – note if the paper turns red for acid or blue for base.
  - d. For test tube 3:  
Add a few drops of dilute hydrochloric acid to the sample in the third test tube. Stir with a clean glass rod. Record any reaction (change of appearance) or solubility.
  - e. For test tube 4:  
Add a few drops of dilute sodium hydroxide to the sample in the fourth test tube. Stir with a clean glass rod. Record any reaction (change of appearance) or solubility.
3. Check your observations against the data in the “Handbook of Chemistry and Physics” and note any discrepancies. You will find information listed in the section titled “Physical Constants of Inorganic Compounds”.

## Part 2. Separation of a Mixture

Note: You will be provided with a vacuum filtration apparatus. A diagram of this apparatus is provided in Appendix J Figure 1. Your instructor will provide you with more detailed instructions on its use.

1. Obtain a sample of a mixture containing sodium chloride and finely ground copper (II) carbonate. Make a note of the identification number or letter of your mixture.
2. Accurately weigh about 2 g of the mixture as follows:
  - a. Weigh an empty 250 mL beaker and record the weight.
  - b. Add about 2 g of the mixture to the beaker.
  - c. Reweigh the beaker and record the weight of the beaker plus mixture.
3. Use the solubility data you collected in Part 1 to see which one of the substances will dissolve in water and which one will not. Make a note of this information.
4. Separate the mixture as follows:
  - a. Add about 50 mL of deionized water to the mixture in the beaker and stir for about 5 minutes with a clean glass rod.

- b. Weigh a 150 mL beaker. Weigh a piece of dry filter paper. Record these data.
- c. Put together the filtration apparatus as shown in Appendix J and place the piece of filter paper in the filter funnel. Wet the paper with a small amount of deionized water from your water bottle.
- d. Turn on the aspirator and pour the mixture through the filter paper, collecting the filtrate in filter flask. Wash the insoluble solid with a small amount of deionized water.
- e. After all the liquid has come through the filter, shut off the aspirator and pour the filtrate (liquid) from the filter flask into your pre-weighed 150 mL beaker.
- f. Evaporate the water from the filtrate by heating gently over a hot plate or Bunsen burner. Let the beaker cool and record its weight. Note the appearance of any solid remaining.
- g. Allow the solid on the filter paper to dry overnight at room temperature or for 1 hour at 110°C in a drying oven. Let the filter paper cool and record its weight. Note the appearance of any solid remaining.

5. Which solid was sodium chloride? Which solid was copper (II) carbonate? Make a note.
6. Calculate the mass of each substance and the percentage of each substance in the original mixture.

**Proper Waste Disposal:** The dried solid that remains in the beaker in step 4e can be dissolved in tap water and disposed of by washing it down the drain with tap water. Dispose of the solid that remains in the filter paper and the filter paper in the waste container designated by your instructor.

### Observations and Data

Part 1. Organize your observations in a table in your lab notebook.

Part 2.

1.  Mass of 250 mL beaker
2.  Mass of 250 mL beaker plus mixture
3.  Mass of filter paper
4.  Mass of 150 mL beaker
5.  Mass of 150 mL beaker plus soluble solid
6.  Identity of soluble solid
7.  Mass of filter paper plus insoluble solid
8.  Identity of insoluble solid

### Calculations

1. Mass of mixture

2. Mass of soluble component
3. Percentage of soluble component in mixture
4. Mass of insoluble component
5. Percentage of insoluble component in mixture

### **Questions**

1. Extrapolating from what you have learned in this experiment, how would you separate a mixture of magnesium oxide from copper metal?
2. Suppose you could not tell whether a material was insoluble or slightly soluble in water, just from the appearance of the mixture. Explain what you would do to determine whether this substance is slightly soluble.

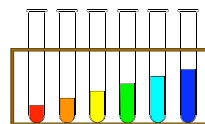
### **Additional Resources**

Check Appendix M for additional resources for this experiment.

## EXPERIMENT 5: USING QUALITATIVE ANALYSIS TO IDENTIFY IONS

### Objectives

1. Identify some commonly occurring ions using qualitative tests.
2. Write equations used for their identification.



### Materials and Equipment

#### CAUTION

Do not get **silver nitrate** ( $\text{AgNO}_3$ ) on your skin. It will leave a dark brown-black stain that may take a week to fade. It will permanently stain clothing and paper.

Small test tubes, disposable pipettes, marking crayons and safety glasses plus the following reagents –



#### Part 1. Anions

Solutions containing the following anions to be tested:

- Bromide,  $\text{Br}^-$  (from 0.1 M  $\text{NaBr}$ )
- Carbonate,  $\text{CO}_3^{2-}$  (from 0.1 M  $\text{Na}_2\text{CO}_3$ )
- Chloride,  $\text{Cl}^-$  (from 0.1 M  $\text{NaCl}$ )
- Iodide,  $\text{I}^-$  (from 0.1 M  $\text{NaI}$ )
- Nitrate,  $\text{NO}_3^-$  (from 0.1 M  $\text{NaNO}_3$ )
- Phosphate,  $\text{PO}_4^{3-}$  (from 0.1 M  $\text{Na}_3\text{PO}_4$ )
- Sulfate,  $\text{SO}_4^{2-}$  (from 0.1 M  $\text{Na}_2\text{SO}_4$ )
- Sulfide,  $\text{S}^{2-}$  (from 0.1 M  $\text{Na}_2\text{S}$ )

Reagents to be used for testing the preceding anions:

- Dilute hydrochloric acid, 6 M  $\text{HCl}$
- Dilute nitric acid, 6 M  $\text{HNO}_3$
- Silver nitrate solution, 0.1 M  $\text{AgNO}_3$
- Dilute nitric acid, 6 M  $\text{HNO}_3$
- Freshly prepared chlorine water
- Hexane
- Saturated calcium hydroxide,  $\text{Ca}(\text{OH})_2$
- Iron (II) sulfate crystals,  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$
- Concentrated sulfuric acid, 18 M  $\text{H}_2\text{SO}_4$
- Ammonium molybdate solution, 0.2 M  $(\text{NH}_4)_2\text{MoO}_4$
- Barium chloride solution, 0.5 M  $\text{BaCl}_2$
- Lead acetate paper

#### Part 2. Cations

Solutions containing the following cations to be tested:

- Aluminum,  $\text{Al}^{3+}$  (from 0.1M  $\text{Al}(\text{NO}_3)_3$ )
- Ammonium,  $\text{NH}_4^+$  (from 0.1 M  $\text{NH}_4\text{NO}_3$ )
- Calcium,  $\text{Ca}^{2+}$  (from 0.1 M  $\text{Ca}(\text{NO}_3)_2$ )

Copper (II),  $\text{Cu}^{2+}$  (from 0.1 M  $\text{Cu}(\text{NO}_3)_2$ )  
Iron (III),  $\text{Fe}^{3+}$  (from 0.1 M  $\text{Fe}(\text{NO}_3)_3$ )  
Lead (II),  $\text{Pb}^{2+}$  (from 0.1 M  $\text{Pb}(\text{NO}_3)_2$ )  
Silver,  $\text{Ag}^+$  (from 0.1 M  $\text{AgNO}_3$ )  
Zinc,  $\text{Zn}^{2+}$  (from 0.1 M  $\text{Zn}(\text{NO}_3)_2$ )

Reagents to be used for testing the preceding cations:

Dilute hydrochloric acid, 6 M  $\text{HCl}$

Dilute nitric acid, 6 M  $\text{HNO}_3$

Aqueous ammonia, 6 M  $\text{NH}_3$  (aq)

Sodium hydroxide, 2 M  $\text{NaOH}$

Ammonium acetate, 3 M  $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$

Aluminon reagent (aurin tricarboxylic acid)

Sodium sulfide, 0.2 M  $\text{Na}_2\text{S}$

Ammonium oxalate, 1 M  $(\text{NH}_4)_2\text{C}_2\text{O}_4$

Potassium hexacyanoferrate (II), 0.2 M  $\text{K}_4\text{Fe}(\text{CN})_6$  (potassium ferrocyanide)

Potassium chromate, 1 M  $\text{K}_2\text{CrO}_4$

**Proper Waste Disposal:** Dispose all waste solids and solutions as directed by your instructor. Elements you may encounter in your labs that are often regulated as hazardous metals are: Ag, As, Cd, Co, Cr, Cu, Hg, Mn, Ni, Pb, Sn, and Zn – there may be special containers provided for such waste. Solutions containing the organic solvent hexane must also be disposed in special containers.

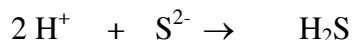
## Introduction

A branch of chemistry that is concerned with the identification of chemical substances is *qualitative analysis*. Through qualitative analysis you can determine whether or not a material is present or absent, but you won't necessarily be able to tell how much of that material there is in a sample.

In Part 1 of this experiment, you will learn to identify some of the common *anions* (negative ions) frequently encountered in the laboratory. You will be looking at reactions with three groups of anions:

1. Anions that will not precipitate (form a solid) in the presence of silver nitrate;
2. Anions that react with silver nitrate to form precipitates that dissolve in nitric acid;
3. Anions that react with silver nitrate to form precipitates that do not dissolve in nitric acid.

In addition, you will also use gas evolution as a clue to identifying certain anions. Net ionic equations for two of these anions are:



After studying the reactions of preliminary tests and then carrying out specific confirmatory tests for the known anion solutions, you will be asked to deduce the identity of an unknown anion in solution. A confirmatory test is carried out only after you have narrowed down the

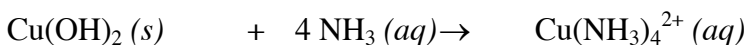
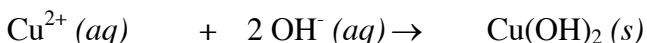
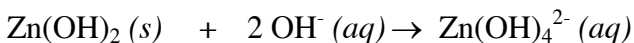
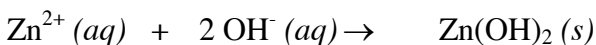
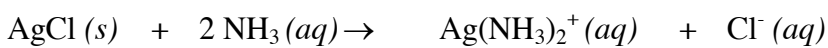
possible list of anions by doing the preliminary tests. Each confirmatory test is specific and gives characteristic results for only one anion.

In Part 2 of this experiment, you will learn to identify some of the more common *cations* (positive ions) frequently encountered in the laboratory. You will be looking at five groups of cations:

1. Cations that precipitate with hydrochloric acid.
2. Cations that precipitate with small amounts of sodium hydroxide but redissolve with larger amounts of sodium hydroxide.
3. Cations that react with small amounts of sodium hydroxide to form precipitates that do not dissolve with larger amounts of sodium hydroxide.
4. Cations that react with small amounts of sodium hydroxide to form precipitates that dissolve in ammonium hydroxide.
5. Cations that react with small amounts of aqueous ammonia to form precipitates that do not dissolve in larger amounts of ammonium hydroxide.

After studying the reactions of preliminary tests and then carrying out specific confirmatory tests for the known cation solutions, you will be asked to deduce the identity of an unknown cation in solution. A confirmatory test is carried out only after you have narrowed down the possible list of cations by doing the preliminary tests. Each confirmatory test is specific and gives characteristic results for only one cation.

The following equations may be helpful for you to understand some of the chemical reactions you will encounter:



## Procedure

*Reminder:* Be careful in selecting the reagent solutions – make certain you are using the right bottle. Some of the solutions have very similar names, so read the label twice! To avoid cross-contamination, do not touch the pipette tips to any surface. Use the crayons for marking your test tubes.

### Part 1. Anions

You will need three clean small test tubes for the testing for each anion. It is OK if the test tubes are wet with deionized water. Carry out the following tests for each anion.

1. Test tube 1: Place about 1 mL of the anion solution to be tested in a clean small test tube. Add about 1 mL of dilute hydrochloric acid (6 M HCl) to the anion solution and mix well.

Make a note of any reaction that occurs, e.g., bubbling, odor of any gas evolved, appearance of the solution. Write the net ionic equation for any reaction that produces a gas.

2. Test tube 2: Take a clean tube and add about 1 mL of the anion solution to be tested.
  - a. Add a few drops of silver nitrate, 0.1 M  $\text{AgNO}_3$  solution and mix well. Note the appearance of any precipitate that forms. Write the net ionic equation for any reaction that produces a precipitate.
  - b. Acidify each test tube with dilute nitric acid (6 M  $\text{HNO}_3$ ) and mix well. Note which precipitates dissolve and which remain.
3. Test tube 3: In a clean test tube, carry out the specific confirmatory test for just the anion you are testing. For example, if you are studying  $\text{Cl}^-$ , only perform the confirmatory test for  $\text{Cl}^-$ .

### **$\text{Br}^-$ or $\text{I}^-$**

To 2 mL of the anion solution add 0.5 mL of hexane and several drops of chlorine water. Stopper and shake well, then allow the upper hexane layer to separate. Note the color in the hexane layer. A brown or gold color indicates the presence of bromine; a reddish-violet or pink color indicates iodine.

**Proper Waste Disposal:** Dispose of the wastes from this test into the Halogenated Solvents container.

### **$\text{CO}_3^{2-}$**

Add dilute hydrochloric acid (6 M  $\text{HCl}$ ) to 5 mL of the anion solution until it tests acid with litmus paper. Carbonates produce an odorless gas. If you see bubbles and there is no odor, this indicates the presence of carbonate. Record your observations.

### **$\text{Cl}^-$**

To 2 mL of the anion solution add 0.5 mL of silver nitrate, 0.1 M  $\text{AgNO}_3$  solution. A white precipitate that easily dissolves when the solution is made basic (test with litmus paper) with aqueous ammonia, 6 M  $\text{NH}_3$  (aq) solution indicates the presence of the chloride ion. Record your observations.

### **$\text{NO}_3^-$**

To 2 mL of the anion solution add two or three crystals of iron (II) sulfate. Stopper and shake until the crystals are dissolved. Hold the test tube at a slant with a test tube holder and cautiously pour 0.5 mL of concentrated sulfuric acid, 18 M  $\text{H}_2\text{SO}_4$  into the tube. DO NOT SHAKE the mixture. A brown ring of  $\text{FeNO}^{2+}$  will form at the interface of the two layers if nitrate ions are present. Make note your observations.

### **$\text{PO}_4^{3-}$**

Add dilute nitric acid, 6 M  $\text{HNO}_3$  to 2 mL of the anion solution until it tests acid with litmus paper. Add 1 mL of ammonium molybdate, 0.2 M  $(\text{NH}_4)_2\text{MoO}_4$  solution. Gentle warming over a Bunsen burner may be necessary to produce a yellow precipitate. Record your results.

## $\text{SO}_4^{2-}$

Add 1 mL of 1 M  $\text{BaCl}_2$  solution to 2 mL of the anion solution. A white precipitate that does not dissolve when HCl is added indicates the presence of sulfate. Record your observations.

## $\text{S}^{2-}$

Add dilute hydrochloric acid (6 M HCl) to 2 mL of the anion solution until it tests acid with litmus paper. The rotten egg odor of hydrogen sulfide ( $\text{H}_2\text{S}$ ) should be apparent. Warm the solution slightly and hold a piece of moist lead acetate paper at the mouth of the test tube. If the paper turns black from the formation of  $\text{PbS}$ , this indicates the presence of sulfide. Note the appearance of the lead acetate paper.

4. Group the anions into the four groups listed in the Introduction.
5. Obtain an unknown anion solution and identify the unknown, using information you have collected on the known anion solutions.

## Part 2. Cations

You will need four clean small test tubes for testing for each cation. It is OK if the test tubes are wet with deionized water. Carry out the following steps for each cation.

1. Test tube 1: Add 2 mL of the cation solution to be tested to a clean small test tube. Add about 1 mL of dilute hydrochloric acid, 6 M HCl. Record your results. If a precipitate forms, go directly to step 4. If a precipitate forms, write the net ionic equation.
2. Test tube 2: If no precipitate formed in step 1, add 2 mL of the cation solution to a second test tube.
  - a. Add two to three drops of sodium hydroxide, 2 M NaOH solution. Stir. Record your results. If a precipitate forms, write the net ionic equation and carry out part b of this step. If no precipitate forms, go directly to step 3.
  - b. Add two or three mL of sodium hydroxide, 2 M NaOH solution. Stir. Observe whether the precipitate formed in part a now dissolves.
3. Test tube 3: If no precipitate was formed with HCl in step 1, add 2 mL of the cation solution to a third test tube. Do not carry out this step if a precipitate forms in step 1.
  - a. Add two to three drops of aqueous ammonia, 6 M  $\text{NH}_3$  (aq) and stir. Record your results. If a precipitate forms, carry out part b of this step. If no precipitate forms, go directly to step 4. If a precipitate is formed, write the net ionic equation.
  - b. Add two or three mL of aqueous ammonia, 6 M  $\text{NH}_3$  (aq) and stir. Observe whether the precipitate formed in part a now dissolves.
4. Test tube 4: Carry out the specific confirmatory test for just the cation you are testing.

**Al<sup>3+</sup>**

Add about 0.5 mL of 3 M ammonium acetate solution and 0.5 mL of Aluminon reagent (aurin tricarboxylic acid) solution to 2 mL of the cation solution to be tested in a small clean test tube. Stir the mixture. Add just enough drops of aqueous ammonia, 6 M NH<sub>3</sub> (*aq*) to turn the solution basic – test the solution with litmus paper. Warm the resulting mixture very gently – do not boil. Record your observations.

**NH<sub>4</sub><sup>+</sup>**

Put about 2 mL of the cation solution to be tested into a small test tube and make the solution basic with sodium hydroxide (2 M NaOH) solution. Use litmus paper to test if you have added enough sodium hydroxide. Warm the mixture gently – do not boil. Test for ammonia fumes by holding a piece of moist red litmus paper just above the mouth of the test tube while you are warming the solution. Be careful not to touch the litmus paper on the test tube or you may get erroneous results. Record your observations.

**Ca<sup>2+</sup>**

Add a few drops of ammonium oxalate, 1 M (NH<sub>4</sub>)<sub>2</sub>C<sub>2</sub>O<sub>4</sub> solution to 2 mL of the cation solution to be tested in a small clean test tube. Record your results.

**Cu<sup>2+</sup>**

In a small clean test tube, acidify 2 mL of the cation solution to be tested with dilute hydrochloric acid, 6 M HCl, and add a few drops of potassium hexacyanoferrate (II), 0.2 M K<sub>4</sub>Fe(CN)<sub>6</sub> solution. Make note of your observations.

**Fe<sup>3+</sup>**

Add a few drops of potassium hexacyanoferrate (II), 0.2 M K<sub>4</sub>Fe(CN)<sub>6</sub> solution to 2 mL of the cation solution to be tested in a small clean test tube. Record your results.

**Pb<sup>2+</sup>**

Add a few drops of potassium chromate, 1 M K<sub>2</sub>CrO<sub>4</sub> solution to 2 mL of the cation solution to be tested in a small clean test tube and record your observations.

**Ag<sup>+</sup>**

Place 2 mL of the cation to be tested into a small clean test tube and add enough dilute hydrochloric acid, 6 M HCl to form a precipitate. Dissolve the precipitate by making the solution basic with aqueous ammonia, 6 M NH<sub>3</sub> (*aq*). Test the solution for basicity with litmus paper. Then add dilute nitric acid, 6 M HNO<sub>3</sub> until the precipitate reappears. Make note of your observations.

**Zn<sup>2+</sup>**

Add excess sodium hydroxide (2 M NaOH) solution to 2 mL of the cation solution to be tested in a small clean test tube. Add a few drops of sodium sulfide, 0.2 M Na<sub>2</sub>S solution and record your observations.

## Observations and Data

### Part 1. Anions

Here is an example table format you can follow to collect your observations.

Ion being tested		Observations			
Name	Symbol	Test tube 1	Test tube 2		Test tube 3
		With HCl	With AgNO <sub>3</sub>	After addition of HNO <sub>3</sub>	Confirmatory test

Unknown number

Specific confirmatory tests performed

Identification of unknown anion

### Part 2. Cations

Here is an example table format you can follow to collect your observations.

Cation being tested		Observations					
Name	Symbol	Test tube 1	Test tube 2		Test tube 3		Test tube 4
		With HCl	2 – 3 drops NaOH	Excess NaOH	2 – 3 drops aqueous NH <sub>3</sub>	Excess aqueous NH <sub>3</sub>	Confirmatory test

Unknown number

Specific confirmatory tests performed and identification of unknown cation

## Questions

1. For Part 1, write the net ionic equations for any precipitates that formed or gases evolved. For Part 2, write the net ionic equations for any precipitations that occurred with chloride ion or hydroxide ion.
2. Group the anions tested according to the three groups listed in the Introduction, plus those anions that produce gases upon addition of HCl.
3. Other than a specific confirmatory test, give a simple test that would allow you to distinguish between the following pairs of anions. Tell what you would do and what you should observe for each anion.
  - a.  $\text{CO}_3^{2-}$  and  $\text{NO}_3^-$
  - b.  $\text{Br}^-$  and  $\text{NO}_3^-$
  - c.  $\text{Cl}^-$  and  $\text{SO}_4^{2-}$
  - d.  $\text{S}^{2-}$  and  $\text{I}^-$
4. Group the cations tested according to the five groups listed in the Introduction. A cation may belong to more than one group.
5. Other than using a specific confirmatory test, give a simple test that would allow you to distinguish between the following pairs of cations. Tell what you would do and what you should observe for each cation.
  - a.  $\text{Ag}^+$  and  $\text{Cu}^{2+}$
  - b.  $\text{Ca}^{2+}$  and  $\text{Zn}^{2+}$
  - c.  $\text{Fe}^{3+}$  and  $\text{Al}^{3+}$
  - d.  $\text{Al}^{3+}$  and  $\text{Zn}^{2+}$

## Additional Resources

Check Appendix M for additional resources for this experiment.

## EXPERIMENT 6: COMPARING CHEMICAL AND PHYSICAL CHANGES USING A TEMPERATURE PROBE

### Objectives

1. Compare the relative amount of energy that is involved in a physical change versus a chemical change for the same substance.
2. Use a computer-interfaced temperature probe to collect data.



### Materials and Equipment

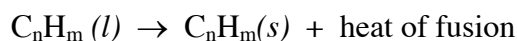


Stainless steel temperature probe, LabPro Data Logger, computer workstation, plain wax candle in a 400 mL beaker, a large test tube filled with about 10 g of candle wax (paraffin), hot plate, iron ring stand, iron ring, clay triangle, beakers, tin can, water, safety goggles.

### Introduction

Candle wax (paraffin) consists of long chains of hydrocarbons. Hydrocarbons are chemical compounds that contain only the elements hydrogen and carbon. One way of representing candle wax is to use the general formula  $C_nH_m$ , where the subscripts  $n$  and  $m$  can have a variety of values.

When you melt wax by heating it, which is a physical change, the heat required for melting to occur is called the *heat of fusion*. No new chemical compounds are created – there is only a change of phase, from solid to liquid, or from liquid to solid. We can write a chemical equation to describe the solidification of melted wax as follows:



In this equation, the heat of fusion is the amount of heat that is released to the environment by the molten wax as it cools and solidifies.

When you combust or burn wax, which is a chemical change, the heat given off during combustion is referred to as the *heat of combustion*. In this case, new chemical compounds are created – bonds are being broken and formed. The following general equation describes the combustion process:



In this case, the heat of combustion is the amount of heat that is released to the environment as the candle burns.

In order to compare the different amounts of heat given off by combustion and fusion, you have to measure the heat in some way. In this experiment, you will use water to capture the heat evolved by the two different processes. The following information will be useful to you in doing your calculations.

There are two units of heat that are commonly used in the chemical laboratory: the calorie (cal) and the joule (J). *One calorie* is defined as the amount of heat required to raise the temperature of 1.00 g of water by 1.00 °C. From this definition, for 1.00 g of water to warm up by 10.0 °C, the water must absorb 10.0 cal of heat from the environment. Note that this is different than the food Calorie – which is 1000 calories (or 1 kcal). The joule is the SI unit of heat and is related to the calorie by the equivalency: 1 cal = 4.184 J.

The general formula for heat is given by:

$$q = m \cdot c \cdot \Delta T$$

where

q = heat

m = mass of the substance involved

c = specific heat of the substance involved

$\Delta T$  = the change in temperature of the substance.

The *specific heat* of a substance is the amount of heat required to raise the temperature of one gram of that substance by one degree Celsius. The water, which is the substance you will be using to capture heat in the experiment, the specific heat is defined as 1.000 cal. In SI units, this would be equivalent to 4.184 J.

g · °C

g · °C

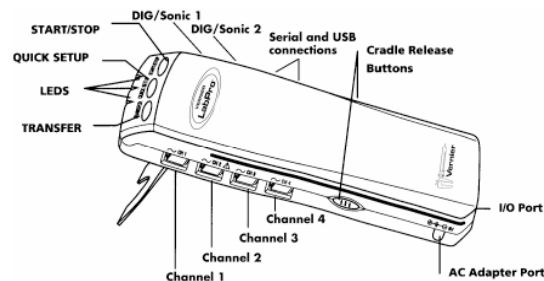
In order to determine how much heat is given off by the wax during each process (combustion or fusion), you will need to know the mass of water heated, the specific heat (which you have been given above), and the change in water temperature that you observe.

Finally, if you know how much wax is involved for each process (combustion or fusion), you will be able to calculate the heat given off for each gram of wax. In this way, you will be able to directly compare the heat involved in combustion and fusion.

## Procedure

### Part 1. Heat of Combustion

1. Weigh to the nearest 0.01 g a 400 mL beaker containing a candle that is standing upright. You need the total weight, not the individual weights. Record this value.
2. Weigh the tin can provided to the nearest 0.1 g. Add about 150 mL of tap water to the tin can and reweigh. You will need both these weights.
3. Set up your equipment according to the diagram provided in Appendix J Figure 2. Make sure that the temperature probe is centered in the tin can and is not touching the sides.
4. Follow the instructions below for use of the LabPro Data Logger:



- a. The Vernier Data Logger (LabPro) will already be connected to a computer workstation.
  - b. Make sure power source is connected to the side of the LabPro.
  - c. Connect the Temperature Probe plug into the Ch 1 port on the same side as the power plug on the LabPro (see diagram above).
  - d. Check that you have set up the heat measuring equipment per the illustrations in Appendix J “Sample Experimental Set-ups”.
  - e. Open LoggerPro software (icon should be on the computer Desktop).
  - f. Click on “Experiment” on top menu bar.
  - g. Select “Data Collection”.
  - h. Under “Collection” tab, select “Time based” mode; set length for 20 minutes; Sampling Rate at 1 sample per minute; 1 minute per sample.
  - i. Close Data Collection box.
  - j. Start data collection by going the Experiment menu and selecting “Start Data Collection”.
  - k. You should start to see data being collected on the Left side of your screen.
  - l. Light the candle and stir the water occasionally with a glass stirring rod, being careful not to strike the temperature probe.
  - m. You can stop collecting data after the temperature has risen 20 degrees.
  - n. Extinguish the candle.
  - o. To stop data collection, return to the Experiment menu and select “Stop Data Collection”.
  - m. Print your data to one of the network printers for inclusion in your lab report.
5. Reweigh the 400 mL beaker and candle.
  6. Calculate the heat absorbed by the water, the mass of candle wax that you burned, and the heat of combustion of the candle wax.

## Part 2. Heat of Fusion

1. You will be given a corked large test tube filled with about 10 g of wax. The exact weight of the empty test tube is marked on the side of the test tube. Removing the cork, weigh the test tube with wax to the nearest 0.01 g. You will need both weights to determine the mass of wax inside.
2. Weigh a small styrofoam cup to the nearest 0.1 g. Add about 150 mL of tap water at room temperature and reweigh the cup. You will need both weights to determine the mass of water in the cup. This water will be used to cool the test tube after you have melted the wax inside the test tube in step 3 below.
3. Heat a 250 mL beaker of water to boiling and place the wax-filled test tube in the hot water to melt the wax.
4. Follow the instructions below for use of the LabPro Data Logger.
  - a. The Vernier Data Logger (LabPro) will already be connected to a computer workstation.
  - b. Make sure power source is connected to the side of the LabPro.
  - c. Connect the Temperature Probe plug into the Ch 1 port on the same side as the power plug on the LabPro (see diagram above).
  - d. Check that you have set up the heat measuring equipment per the illustration for heat of fusion in Appendix J figure 2.
  - e. Open LoggerPro software (icon should be on the computer Desktop).

- f. Click on “Experiment” on top menu bar.
  - g. Select “Data Collection”.
  - h. Under “Collection” tab, select “Time based” mode; set length for 20 minutes; Sampling Rate at 1 sample per minute; 1 minute per sample.
  - i. Close Data Collection box.
  - j. Start data collection by going the Experiment menu and selecting “Start Data Collection”.
  - k. You should start to see data being collected on the Left side of your screen.
  - l. Using a test tube holder, remove the test tube containing the melted wax from the hot water and allow the wax to cool until you see the first solid wax being formed.
  - m. Quickly place the test tube into the styrofoam cup and stir the water gently with the test tube. Continue stirring until the wax has completely solidified.
  - n. You can stop collecting data after the wax has completely solidified. To stop data collection, return to the Experiment menu and select “Stop Data Collection”.
  - o. Print your data to one of the network printers for inclusion in your lab report.
5. Note the highest temperature you observed.
  6. Calculate the heat that was absorbed by the water, the mass of candle wax solidified and the heat of fusion of the candle wax.

### **Observations and Data**

#### Part 1. Heat of Combustion

1.  Initial mass of 400 mL beaker with wax candle
2.  Mass of the tin can empty
3.  Mass of the tin can plus water
4.  Initial temperature of water
5.  Final temperature of water
6.  Final mass of 400 mL beaker with wax candle

#### Part 2. Heat of Fusion

1.  Mass of empty test tube
2.  Mass of test tube plus candle wax
3.  Mass of styrofoam cup
4.  Mass of styrofoam cup plus water
5.  Initial temperature of water in styrofoam cup
6.  Highest temperature of water in styrofoam cup

## Calculations

### Part 1. Heat of Combustion

1. Mass of water heated
2. Temperature change of water
3. Heat absorbed by water in J
4. Mass of candle burned
5. Heat of combustion of the candle

### Part 2. Heat of Fusion

1. Mass of water heated in styrofoam cup
2. Temperature change of water
3. Heat absorbed by water in J
4. Mass of candle solidified
5. Heat of fusion of candle wax

## Questions

1. For Part 1, list ways where heat could be lost and consequently not raise the temperature of the water.
2. Calculate the ratio of the heat of combustion to the heat of fusion.
3. Based on your answer to question 2, what conclusion can you make about the amount of energy it takes for a chemical change compared to a physical change?

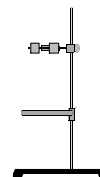
## Additional Resources

Check Appendix M for additional resources for this experiment.


## EXPERIMENT 7: CALCULATING AN EMPIRICAL FORMULA

### Objective

Determine the empirical formula of the product formed by reacting copper metal with elemental sulfur in the absence of oxygen.



### Materials and Equipment

 Porcelain crucible with cover, clay triangle, crucible tongs, ring stand, Bunsen burner, digital top-loading balance, copper beads (approximately 1 g), sulfur powder (approximately 1 g), small beaker, matches or striker, safety goggles.

### CAUTION

Remember that hot objects look the same as cool objects. Always be cautious in handling heated equipment – you can get severe burns.

**Proper Waste Disposal:** Your instructor will provide you with instructions for disposing of wastes generated during this experiment. The solid product you create will be disposed into a marked container in the waste disposal area.

### Introduction

The *empirical formula* of any compound gives the simplest whole number ratio of the atoms of each element that is present in that compound. For example, if we were to look at the molecular formula for sugar, it would be  $C_6H_{12}O_6$ . Thus, if we were to write the empirical formula for sugar, it would be  $CH_2O$ . Notice that there could be many compounds that have the same empirical formula, e.g., formaldehyde has the same empirical formula,  $CH_2O$ , as sugar. Experimentally, it is often easier to determine the ratios of the moles of the elements – which essentially allow you to determine the empirical formula.

In this experiment you will react elemental copper, which is a metal, with sulfur, which is a non-metal. By making accurate measurements of the mass of sulfur incorporated into the final product, you will be able to determine the empirical formula of the compound. The basic reaction is:

Copper + Sulfur  $\rightarrow$  a sulfide of copper.

This reaction will be carried out by adding an excess of sulfur to a known quantity of copper. All of the copper should be incorporated into the final product (the sulfide of copper), while only a portion of the sulfur will be incorporated. The excess sulfur will be burned off at the end of the reaction. The mass of sulfur can then be calculated, based on the change of mass of the solid left behind as product. If you know the mass of each of the elements used, you can convert the

masses into moles. The mole ratio of the elements will provide you with the information you need to determine the empirical formula.

## Procedure

1. Place a crucible with its cover on a clay triangle, supported by an iron ring stand. A diagram of this apparatus is provided in Appendix J figure 3. The cover must be ajar, so that you can drive off any impurities and moisture. Before heating the crucible, practice removing and replacing the cover with the crucible tongs. The crucible must be heated for several minutes over a non-luminous (hot) flame to cause the porcelain to glow.
2. Allow the crucible to cool – remember not to place the hot crucible directly on the lab bench, but set it on wire gauze, otherwise you may break the crucible. Weigh the crucible *without* its cover on a digital top-loading balance. Record the weight. Use the same balance throughout the experiment.
3. Weigh out approximately 1 g of copper beads and place them in the crucible. Weigh the crucible without its cover but with the copper beads and record its weight accurately. Note and record the color of the copper.
4. Weigh about 1.0 g of sulfur powder into a small beaker and add this into the crucible. There is no need to reweigh the crucible at this point. (Can you think of the reason why this is the case?) Make a note of the appearance of the sulfur.
5. **IMPORTANT:** Now reassemble your ring stand, Bunsen burner, clay triangle and crucible under the hood.

### **CAUTION**

Since the decomposition of excess sulfur creates sulfur dioxide, which is a toxic, choking gas, this part of the experiment **must** be carried out in the fume hood.

6. Cover the crucible with the crucible cover and heat it with a non-luminous (hot) flame for 10 minutes. When heating the copper-sulfur mixture, the crucible cover must completely cover the crucible or you will not create the desired compound.
7. At the end of 10 minutes, remove the crucible cover with the crucible tongs and continue to heat the crucible until the combustion of excess sulfur is no longer visible. You will see a blue flame at the top of the crucible as long as there is sulfur being combusted. It is this reaction that creates the toxic gas sulfur dioxide (SO<sub>2</sub>) and the reason for carrying out this part of the experiment in the hood:



- Heat the crucible for an additional two minutes after the blue flame disappears, and then allow the crucible to cool.
- Reweigh the crucible – it will contain the copper sulfide produced. Record the weight. Note and record the appearance of the copper sulfide.
- Use the mass data you recorded to calculate the moles of copper and the moles of sulfur in the final product. From the ratio of moles, write the empirical formula of the sulfide created.

### **Observations and Data**

You will need to record the following information in your lab notebook.

- Mass of crucible without cover
- Mass of crucible plus copper beads
- Approximate mass of sulfur used
- Appearance of copper beads
- Appearance of sulfur powder
- Mass of crucible without cover plus copper sulfide after heating
- Appearance of copper sulfide formed

### **Calculations**

- Mass of copper used
- Mass of sulfur incorporated
- Moles of copper
- Moles of sulfur
- Simplest ratio of moles of copper to sulfur in the copper sulfide (divide the smaller number of moles into the larger)
- Empirical formula of the copper sulfide

### **Questions**

1. Why was the crucible heated before you took its initial weight?
2. Before heating, you weighed the crucible empty, and then weighed it again after you added the copper beads. Why was it not necessary to reweigh the crucible immediately after you added the sulfur powder?
3. How would the copper to sulfur ratio be affected if you had added twice as much sulfur powder to the crucible?
4. What would happen to the copper to sulfur ratio if you only used  $\frac{1}{4}$  as much sulfur?
5. Color is a physical property. But a color change can be the result of either a chemical change or a physical change. Was the color change in this experiment the result of a chemical change or a physical change? Explain your reasoning.

### **Additional Resources**

Check Appendix M for additional resources for this experiment.

## EXPERIMENT 8: PRODUCTION AND INVESTIGATION OF FOUR GASES

### Objectives

1. Investigate some physical and chemical properties of a group of gases.
2. Determine the identification of these gases based upon their properties.

### Materials and Equipment

Dilute hydrochloric acid (6 M HCl)  
Concentrated hydrochloric acid (12 M HCl)  
Concentrated nitric acid (15 M HNO<sub>3</sub>)  
Calcium carbonate  
Saturated calcium hydroxide (limewater) solution  
Mossy zinc  
Manganese dioxide  
10% hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>)  
Granulated copper metal



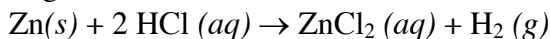
Gas-generating apparatus (see Appendix J Figure 4), glass bottles, glass plates, large test tubes, test tube clamp, wooden splints, matches, plastic straws.



### Introduction

There are several gases that are common in laboratories. You are going to use the physical and chemical properties of four gases to identify them. The four gases you will investigate and the reactions to form them in this experiment are:

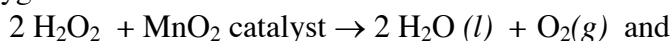
Hydrogen:



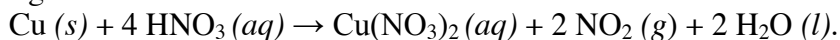
Carbon dioxide:



Oxygen:



Nitrogen dioxide:



Note: a *catalyst* is a material that accelerates a chemical reaction but is not used up or incorporated into a product. The same amount of a catalyst will be present at the beginning and end of a reaction.

You will generate small amounts of the first three gases in test tubes and collect the gases using the gas collection apparatus. Gas bubbles are less dense than water, so they will bubble to the surface under water. Think about the bubbles that are exhaled by divers under water. The gases you generate can be collected into a bottle or test tube for further investigation. Your instructor will produce nitrogen dioxide as a demonstration.

## Procedure

### Part 1. Hydrogen

1. Assemble the gas-generating apparatus as shown in Appendix J “Example Experimental Set-ups”. Remove the stopper with the gas delivery tube for later use. Fill the pneumatic trough with tap water.
2. Fill a second large test tube to the very top with water. Hold your thumb over the open end of the test tube and lower the open end of the test tube into the pneumatic trough as shown in the picture in Appendix J “Example Experimental Set-ups”. Invert the test tube completely and set the mouth of the test tube over the delivery tube.
3. Add about 5 mL of deionized water and a small piece of mossy zinc to the large test tube in the gas-generating apparatus.
4. Add 5 mL of dilute hydrochloric acid (6 M HCl) to the test tube containing the water and mossy zinc. If the reaction does not produce a constant stream of hydrogen bubbles, add more acid.
5. Insert the stopper with the gas delivery tube (see step 1) into the gas-generating test tube. Run the delivery tube into the inverted test tube in the pneumatic trough (see step 2). Collect bubbles until hydrogen gas has replaced the water in the test tube – the test tube will look “empty”.
6. Light a wooden splint with a match, remove the inverted test tube containing hydrogen from the pneumatic tube, and quickly push the splint into the mouth of the test tube. You should hear and observe something happen.
7. Record your observations.

**Proper Waste Disposal:** Dispose all waste solids and solutions as directed by your instructor. Dispose of your waste hydrochloric acid in Acid/Base Waste. Dispose of the solid zinc into the specially marked container.

### Part 2. Carbon Dioxide

1. Prepare your gas-generating apparatus for reuse.
2. Fill three wide-mouth glass bottles to the very top with tap water. Place a glass plate over the top of each bottle and invert each bottle into the pneumatic trough. Remove the glass plates.
3. Place a few pieces of calcium carbonate into the gas-generating test tube. Add dilute hydrochloric acid until you see a steady stream of carbon dioxide bubbles. If the evolution of gas slows down, you can add small amounts of concentrated hydrochloric acid to speed up the reaction.
4. Connect the stopper with the gas delivery tube as you did in the previous part.
5. When carbon dioxide bubbles appear in the pneumatic trough, place an inverted bottle containing water over the stream of bubbles. Fill each of the water-filled bubbles with the

carbon dioxide from the gas generator. As each bottle is filled with carbon dioxide, cover the open end of the bottle with a glass plate and place it on the lab bench until you are ready for testing the carbon dioxide.

6. Note the appearance and odor, if any, of the gas in the first bottle of carbon dioxide. Record your observations.
7. Light a wooden splint and push it into the open mouth of the first bottle of carbon dioxide. What happens to the splint? Record your observations.
8. Limewater is a solution that is saturated with calcium hydroxide. It can be used as a test for the presence carbon dioxide. Pour about 10 mL of clear limewater into the second bottle of carbon dioxide and place the glass plate back on the bottle. Shake the bottle several times. What happens to the solution? Record your observations.
9. Pour about 10 mL of limewater into an empty bottle (one containing only air). Take the third bottle containing carbon dioxide, remove the glass plate and invert it over the bottle containing the limewater, as if you were pouring water out of the bottle. Cover the bottle containing the limewater and shake it several times. What happens to the solution? Does it look like the bottle in step 8? Record your observations.
10. Pour about 5 mL of limewater into a large test tube. Insert a clean plastic straw into the test tube so that the end is under the surface of the limewater. Blow into the limewater for several (at least five) minutes. Do you observe any change? Record your observations.

**Proper Waste Disposal:** Dispose your waste hydrochloric acid and calcium carbonate chunks as directed by your instructor. There will be a specially marked container for the calcium carbonate chunks – do not throw them in the trash or into the sink. The used limewater can be diluted and rinsed down the drain.

### Part 3. Oxygen

1. Prepare your gas-generating apparatus for reuse.
2. Place about 0.1 g of manganese dioxide (enough to fill the tip of a micro-spatula) into the gas generation tube.
3. Add enough 10% hydrogen peroxide to fill the test tube about half full.
4. Connect the stopper containing the gas delivery tube as before.
5. Do not collect the bubbles generated for the first minute, then collect the oxygen gas into a water-filled bottle.
6. Invert the bottle containing the oxygen. Light a wooden splint with a match and blow out the flame so the splint only glows. Immediately push the glowing splint into the bottle containing the oxygen. What happens? Record your observations.

**Proper Waste Disposal:** Dispose of your waste from this part of the laboratory into the bottle marked “MnO<sub>2</sub>/H<sub>2</sub>O<sub>2</sub> Waste”.

#### Part 4. Nitrogen Dioxide (instructor demonstration)

### CAUTION

1. Concentrated nitric acid is highly corrosive. If spilled, clean it up immediately and tell your instructor. If you get any on your skin, wash it off thoroughly and tell your instructor.
2. Nitrogen dioxide ( $\text{NO}_2$ ) is a highly toxic, irritating gas. Do not inhale fumes!

Your instructor will carry out this demonstration in the fume hood, because a toxic, irritating gas (nitrogen dioxide) is generated. The gas will not be collected. You will observe what happens and record your observations.

1. About 3 mL of concentrated nitric acid is placed into a large dry test tube.
2. Eight to 10 granules of copper metal are added.
3. Observe the gas ( $\text{NO}_2$ ) and solution that results. Record your observations.

**Proper Waste Disposal:** Your instructor will dispose of the waste generated by this demonstration.

### Observations and Data

#### Part 1. Hydrogen

1. Reagents used for preparation
2. Color of gas
3. Effect upon lighted splint

#### Part 2. Carbon Dioxide

1. Reagents used for preparation
2. Color and odor of gas
3. Effect upon lighted splint
4. Addition of limewater to carbon dioxide
5. Result of “pouring” carbon dioxide into limewater
6. Result of blowing into limewater

### Part 3. Oxygen

1. Reagents used for preparation
2. Color of gas
3. Effect upon glowing splint

### Part 4. Nitrogen Dioxide

1. Reagents used for preparation
2. Color of gas

### Questions

1. What observation did you make that makes you believe that gases are less dense than water?
2. Is carbon dioxide less dense or denser than air? What evidence do you have for your choice?
3. If you fill a balloon with helium, it rises, because the helium is lighter or less dense than air. What do you think would happen to a balloon filled with carbon dioxide? Hint: consider your answer to question 2.
4. Which of the gases you observed today would be the most helpful in putting out a fire? What evidence do you have to support this choice?
5. Based on what you learned today, tell how you could distinguish between the following pairs of gases:
  - a. carbon dioxide and nitrogen dioxide
  - b. hydrogen and carbon dioxide
  - c. oxygen and hydrogen?
6. Now suppose you were given a sample of one of the four gases you observed today.
  - a. If the sample gas is colorless, which gas does this eliminate?
  - b. If a portion of the same sample gas is bubbled through limewater and nothing happens, what gas is eliminated?
  - c. If a glowing splint reignites when it is placed in another portion of the same sample gas, what is the identity of the gas?

### Additional Resources

Check Appendix M for additional resources for this experiment.



## EXPERIMENT 9: GAS LAWS – DETERMINING THE MOLAR VOLUME OF HYDROGEN

### Objective

Determine the volume of 1 mole of a gas at standard temperature and pressure (STP).

### Materials and Equipment

Magnesium ribbon  
Dilute hydrochloric acid, 6 M HCl  
Gas-measuring tube, with two-holed stopper  
Piece of copper wire  
250 mL or 400 mL beaker, large graduated cylinder, scissors, meter stick, thermometer, barometer, latex gloves, safety goggles.



### CAUTION

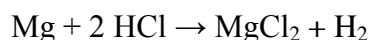
Remember that that hydrochloric acid is extremely corrosive – you can get burns. Wear protective gloves.

### Introduction

According to Avogadro's ideal gas law, equal volumes of gases under the same conditions of temperature and pressure will contain the same number of moles. The *molar volume* of a gas is the volume occupied by 1 mole of gas. It is typically reported at *standard temperature and pressure (STP)*, which are:

$$0\text{ }^{\circ}\text{C} = 273\text{ K and }1.000\text{ atm} = 760.0\text{ torr.}$$

In this experiment you will generate a known number of moles of hydrogen gas from the reaction of magnesium with hydrochloric acid.



If you know the number of moles of magnesium consumed during the reaction, you know how many moles of hydrogen gas are evolved. Since the hydrogen gas will be collected over water, the pressure of “wet” hydrogen gas produced must be corrected by subtracting out the pressure due to water vapor that is collected at the same time.

$$P_{\text{barometric}} = P_{\text{Hydrogen}} + P_{\text{Water}}$$

Then the volume of the “dry” hydrogen gas is corrected to STP conditions using the *combined gas law*. If you need help with the combined gas law, refer to your lecture textbook.

$$P_1V_1/T_1 = P_2V_2/T_2$$

By replacing the values for condition 2 with the values for STP, we can obtain the following equation:

$$V_{\text{STP}} = (P_1 * V_1 * 273 \text{ K}) / (T_1 * 760 \text{ torr}).$$

Remember that for an ideal gas at STP, the molar volume is 22.4 L/mole

The molar volume at STP is simply the ratio of the volume of dry gas collected corrected to STP to the moles of hydrogen gas produced (which is calculated from the moles of magnesium consumed). The table below provides the vapor pressure of water that must be subtracted from the barometric pressure to do the correction at several temperatures.

Temperature (°C)	Pressure (torr)	Temperature (°C)	Pressure (torr)
15	12.8	23	21.0
16	13.6	24	22.4
17	14.5	25	23.8
18	15.5	26	25.2
19	16.5	27	26.7
20	17.5	28	28.3
21	18.6	29	30.0
22	19.8	30	31.8

### Procedure

1. Measure out and cut a piece of magnesium ribbon between 4.0 cm and 4.5 cm. Do not use more than 4.5 cm or you will not be able to determine the volume of hydrogen gas produced! Record the length of the magnesium ribbon to the nearest 0.1 cm. Obtain and record the mass of the magnesium ribbon per centimeter – it should be marked on the container holding the ribbon. From these data you will be able to calculate the total mass of magnesium you use.
2. Coil the magnesium ribbon into a small ball. Use a piece of copper wire to create a cage around the magnesium and secure it to the stopper as shown in Appendix J figure 5. Set this aside until it is needed.
3. Pour 10 mL of 6 M hydrochloric acid into the gas tube. Tilt the tube and slowly fill the gas tube with water. The object is to not disturb the layer of hydrochloric acid at the bottom of the gas tube. Fill the gas tube to the very top with water.
4. Using gloves, stopper the tube with the rubber stopper you prepared in step 2. There should be no air bubbles anywhere in the stoppered gas tube.
5. Still using gloves, hold a finger over the holes in the stopper, invert the gas tube and place it in a 250 mL or 400 mL beaker filled about half full of water. You can use an iron ring stand to help hold the gas tube vertical.
6. As the hydrochloric acid drifts down to the magnesium, you will see bubbles of hydrogen gas form and float to the top of the gas tube. As more hydrogen gas is evolved, the gas pressure at the top of the tube will push the water down and out through the holes in the rubber stopper. Eventually, the bubbling will cease when all the magnesium is consumed.

7. Wait about 5 minutes after the reaction is over to let the gas come to room temperature. In the meantime, obtain the barometric pressure of the laboratory.
8. Transfer the gas tube to a large graduated cylinder filled with room temperature water to aid in equalizing the pressure inside the gas tube to barometric pressure as shown in Appendix J figure 5. Remember to use gloves and hold a finger over the holes in the rubber stopper when you do this. Let the temperature in the gas tube equilibrate to the temperature of the water in the graduated cylinder. Record this information.
9. Equalize the pressure inside the gas tube with the external barometric pressure. To do this, raise or lower your gas collection tube so the liquid level inside the tube is the same as the surrounding liquid. Read the volume of the gas inside the tube to the nearest 0.1 mL.

**Proper Waste Disposal:** Dispose of the magnesium-containing hydrochloric acid solution left in your beaker and gas tube as directed by your instructor.

### Observations and Data

1.  Mass of magnesium ribbon per cm
2.  Length of magnesium strip
3.  Water temperature
4.  Barometric pressure of the laboratory (torr)
5.  Volume of “wet” hydrogen gas collected

### Calculations

1. Mass of magnesium used
2. Moles of magnesium used
3. Moles of hydrogen evolved
4. Pressure of “dry” hydrogen
5. Volume of “dry” hydrogen corrected to STP
6. Molar volume of hydrogen at STP
7. Determine the percentage error between your experimental value and the accepted value of 22.4 L/mol at STP

### Questions

1. What effect would using twice as much hydrochloric acid have on the volume of hydrogen gas produced?

2. What effect would using half as much magnesium have on the volume of hydrogen produced?
3. What effect would the use of half as much magnesium have on the molar volume determined?
4. The accepted molar volume at STP is 22.4 L/mole. What is the molar volume at 25°C and 1 atm?

### **Additional Resources**

Check Appendix M for additional resources for this experiment.



## EXPERIMENT 10: STOICHIOMETRY – PRODUCING AN INSOLUBLE IONIC COMPOUND



### Objectives

1. Write a balanced molecular equation to produce a desired compound.
2. Use stoichiometry to prepare a given amount of an insoluble ionic compound.

### Materials and Equipment

Barium chloride dihydrate,  $\text{BaCl}_2 \cdot 2 \text{H}_2\text{O}$

Calcium chloride dihydrate,  $\text{CaCl}_2 \cdot 2 \text{H}_2\text{O}$

Strontium nitrate,  $\text{Sr}(\text{NO}_3)_2$

Potassium carbonate 1.5 hydrate,  $\text{K}_2\text{CO}_3 \cdot 1.5 \text{H}_2\text{O}$

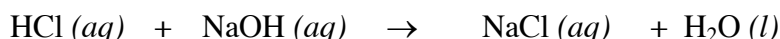
Two 100 mL or 150 mL beakers, one 250 mL beaker, vacuum filtration apparatus (filter funnel and filter flask assembly), filter paper, wash bottle, glass stirring rod, safety goggles.



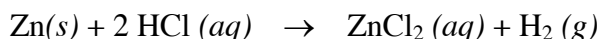
### Introduction

There are several methods that can be used to form ionic compounds, sometimes referred to as *salts*. Sometimes the salts are soluble, but sometimes the product formed is insoluble. Insoluble ionic compounds are referred to as *precipitates*. Four of the more common types of reactions to produce salts are provided below, with examples. You can find more examples in your lecture textbook.

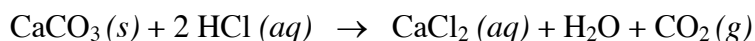
An acid with a base:



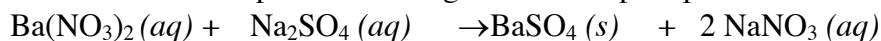
A metal with an acid:



A carbonate with an acid:



Two soluble ionic compounds forming at least one precipitate:



If you know the correct balanced molecular equation, you can use the ratios of moles to determine how much starting material you will need to form a particular amount of product. Suppose you wanted to use the last example reaction to make 3.00 g of insoluble  $\text{BaSO}_4$ . Here is one way to proceed.

- a. Write a balanced equation for this reaction.
- b. Decide how many moles of  $\text{BaSO}_4$  is equal to 3.00 g. Hint: you will need to know the molar mass of  $\text{BaSO}_4$ .
- c. Calculate the mass of solid  $\text{Ba}(\text{NO}_3)_2$  you will need. Hint: you will need to know the molar mass of  $\text{Ba}(\text{NO}_3)_2$ .

d. Calculate the mass of solid  $\text{Na}_2\text{SO}_4$  you will need. As before, you will need to know the molar mass of this reactant.

Realize that if one of your starting materials contains waters of hydration in the molecular formula, such as potassium carbonate 1.5 hydrate,  $\text{K}_2\text{CO}_3 \cdot 1.5 \text{H}_2\text{O}$ , you need to add the mass of the molecules of water to the mass of the compound. In this way you are correcting for the fact that not all of the material that you weigh out is, for this example,  $\text{K}_2\text{CO}_3$  – some of the material is water that is part of the crystal matrix. Therefore the molar mass in this example would include 1.5 times the molar mass of water.

Note that just because the experimental goal in this example was 3.00 g of product, this is just the *theoretical yield*, the maximum amount that would be created if everything worked perfectly. However, realistically, this is not what you would expect to produce. We use the *percentage yield* as a measure of how well the reaction worked:

$$\% \text{ yield} = \frac{\text{Actual yield}}{\text{Theoretical yield}} \times 100\%$$

### Procedure

1. You will be assigned an insoluble compound to prepare. Determine which of the chemicals provided could be used to produce that compound.
2. Write a balanced molecular equation. You can use the solubility table in Appendix H to help you with the solubility of any of the reactants or products.
3. Your assignment is to prepare 2.00 g of product. Determine how many moles of product this is. Using the balanced molecular equation, calculate the mass of each reactant required to prepare the desired quantity of product.
4. Weigh each reactant into a separate 100 mL or 150 mL beaker. Record the masses that you used.
5. Add 35 mL of deionized water to each beaker. Use a glass stirring rod to stir each solution until the solute (solid) is totally dissolved. If a solute does not dissolve within five minutes, add an additional 10 mL of deionized water, then continue to stir until the solute is totally dissolved. The resulting solutions should be clear and colorless.
6. Pour the two solutions from the smaller beakers into the 250 mL beaker. Rinse each of the smaller beakers with no more than 10 mL of deionized water and add this rinse water to the 250 mL beaker. What is the appearance of the solution? Record your observations.
7. Allow the precipitate (product) to form for at least 15 minutes, stirring the mixture frequently with a glass stirring rod.
8. While the precipitation reaction is proceeding, weigh a piece of filter paper to use for the vacuum filtration. Record the weight. Prepare your vacuum filtration apparatus, if you have not already done so, as shown in Appendix J Figure 1 and place the piece of pre-weighed filter paper in the filter funnel.

9. When you are ready to filter your reaction mixture, wet the paper with a small amount of deionized water from your water bottle.
10. Turn on the aspirator and pour the mixture through the filter paper, collecting the filtrate in the filter flask and the precipitate on the filter paper. Wash the insoluble precipitate with a small amount of deionized water. What is the appearance of the filtrate? What is the appearance of the precipitate? Record your observations.
11. After all the liquid has come through the filter, shut off the aspirator and carefully remove the filter paper with precipitate and place it on a paper towel.
12. Allow the solid on the filter paper to dry overnight at room temperature or for 30 minutes at 100°C in a drying oven. Let the filter paper cool and record its weight. Note the appearance of any solid remaining.
13. Calculate the percentage yield of your product.
14. Submit your weighed product to your instructor with your report.

***Proper Waste Disposal:*** Dispose all waste solids and solutions as directed by your instructor.

### **Pre-experiment Calculations**

1. Compound to be prepared
2. Desired mass of salt to be prepared
3. Reaction to be performed (balanced molecular equation)
4. Moles of product desired
5. Mass of each reactant to be used

### **Observations and Data**

1.  Mass of each empty beaker
2.  Mass of first beaker plus reactant 1
3.  Mass of second beaker plus reactant 2
4.  Mass of filter paper
5.  Mass of filter paper plus product

### **Calculations**

1. Actual mass of each reactant used

2. Mass of product isolated
3. Percentage yield

### Questions

Write a balanced molecular equation that would produce each of the following salts by using the type of reaction indicated. Use your lecture textbook for help.

1.  $K_2SO_4$  from an acid and a base:
2.  $AlBr_3$  from a metal and an acid:
3.  $Pb(NO_3)_2$  from a carbonate and an acid:
4.  $MgCl_2$  from a metal and an acid:
5.  $MgCl_2$  from a carbonate and an acid:
6.  $MgCl_2$  from a metal hydroxide (base) and an acid:
7.  $NH_4I$  from a carbonate and an acid:

### Additional Resources

Check Appendix M for additional resources for this experiment.

## EXPERIMENT 11: STUDYING IONIC REACTIONS

### Objectives

1. Study the nature of ionic reactions.
2. Write net ionic equations for precipitation reactions you observe.

### Materials and Equipment

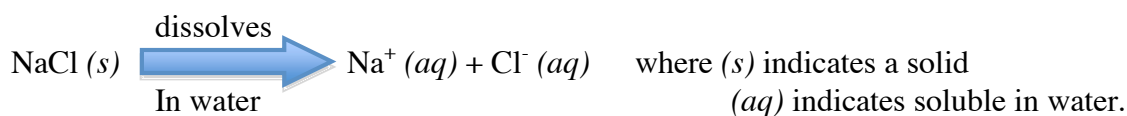
Acetate sheets, 250 mL beaker for waste, wash bottle, safety goggles.  
The following five sets of aqueous ionic solutions:



	Set I	Set II	Set III	Set IV	Set V
Solution 1	Ba(NO <sub>3</sub> ) <sub>2</sub>	Na <sub>2</sub> SO <sub>4</sub>	FeCl <sub>3</sub>	NiCl <sub>2</sub>	BaCl <sub>2</sub>
Solution 2	BaCl <sub>2</sub>	Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub>	Co(NO <sub>3</sub> ) <sub>2</sub>	MgCl <sub>2</sub>	Sr(NO <sub>3</sub> ) <sub>2</sub>
Solution 3	Na <sub>2</sub> CrO <sub>4</sub>	Sr(NO <sub>3</sub> ) <sub>2</sub>	CoCl <sub>2</sub>	K <sub>2</sub> SO <sub>4</sub>	Na <sub>2</sub> CrO <sub>4</sub>
Solution 4	K <sub>2</sub> CrO <sub>4</sub>	BaCl <sub>2</sub>	NaOH	NaOH	Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub>
Solution 5	NaNO <sub>3</sub>	Ba(NO <sub>3</sub> ) <sub>2</sub>	KOH	Ba(OH) <sub>2</sub>	K <sub>2</sub> CrO <sub>4</sub>
Solution 6	KCl	AlCl <sub>3</sub>	NaNO <sub>3</sub>	MgSO <sub>4</sub>	AgNO <sub>3</sub>

### Introduction

*Aqueous solutions* are those in which water is the solvent. So, aqueous ionic solutions are solutions of ionic compounds in water. The solutions you will be provided will be of ionic compounds that are soluble in water. Soluble ionic compounds separate into *cations* (positively charged ions) and *anions* (negatively charged ions) – this process is known as *dissociation*. So, if we dissolved table salt, NaCl, in water, the process can be represented as follows:

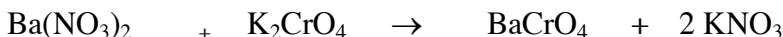


NaCl (aq) could also be used to indicate an aqueous sodium chloride solution, instead of writing out the individual ions as above.

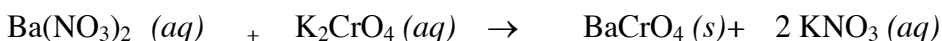
In this experiment, you will take a series of two different clear aqueous solutions of ions and mix them together. When the two solutions are mixed, the ions of both solutions are all available to react with each other. Sometimes this type of reaction is categorized as a *double replacement reaction*. If you see a solid or cloudiness form, the solid product that is formed is referred to as a *precipitate*. A precipitate indicates that at least one of the products formed is insoluble in water. The symbol (s) can also be used to designate a precipitate. A precipitate can also be represented by the down arrow ↓ or by the abbreviation ppt. You can use the solubility table in Appendix H to decide what product has come out of solution as a solid. Some general principles apply to double replacement reactions:

1. Any soluble ionic substance will never be observed as a precipitate.
2. If a given pair of positive and negative ions are observed together in any solution without forming a precipitate, they will never precipitate together.
3. Some kinds of ions do not react and will remain in solution – these are called *spectator ions*.
4. If no precipitate forms, no reaction has occurred.

Let us look at a simple example from Set I to help understand precipitation reactions. If we take an aqueous solution of  $\text{Ba}(\text{NO}_3)_2$  and mix it with an aqueous solution of  $\text{K}_2\text{CrO}_4$  and a solid forms, we can represent the overall reaction as:

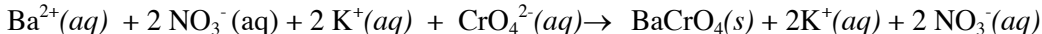


We know that the symbols for the reactants on the left-hand side of the equation will both be (*aq*), since the ionic compounds were both soluble, i.e., they were in solution. But which one or ones of the products is the solid precipitate that formed? If we look at the table of solubilities in Appendix H, we see that  $\text{KNO}_3$  is marked as *aq* or soluble.  $\text{BaCrO}_4$  is marked as *I* or insoluble. So we can modify the equation to become:

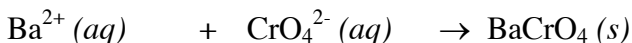


This representation would be the balanced *molecular equation*, showing the molecular formulas of the reactants and products.

But we know that the ionic compounds that are in solution have dissociated and are in the form of ions. So the *total ionic equation* could be written like this:



If we wanted to just represent the ions that are actually coming together to create a new compound – the precipitate – we could rewrite the equation as follows:



This form of the reaction would be referred to as the *net ionic equation* and the spectator ions, those ions that do not form a new product, are not written out.

## Procedure

1. You will be given five sets of ionic solutions – each set will contain six solutions as listed in the Materials and Equipment section. Draw data tables such as the examples given under Observations and Data to organize your work.
2. For each set of solutions, mix all of the possible combinations two at a time. You will use acetate sheets to mix two drops of each of the solutions together. Do not touch the dropper to the sheet or to any liquid on the sheet. Observe if any precipitates form.
3. After you have completed one set of solutions, rinse your sheet into a 250 mL beaker. You will later transfer your waste into the designated waste container. Wash off your acetate sheet with deionized water and dry with a paper towel between sets.

4. After all possible combinations have been mixed, determine the formula of each unique precipitate formed and write the net ionic equation for the reaction.

*Important note: NaOH solution easily absorbs carbon dioxide from air. In Set V, if you see a precipitate with barium hydroxide, indicating the production of barium carbonate, ignore the result.*

**Proper Waste Disposal:** Dispose all waste solids and solutions as directed by your instructor.

### Observations and Data

Set I

You can set up a table similar to this example to record your observations:

	K <sup>+</sup> /Cl <sup>-</sup>	Na <sup>+</sup> /NO <sub>3</sub> <sup>-</sup>	K <sup>+</sup> /CrO <sub>4</sub> <sup>2-</sup>	Na <sup>+</sup> /CrO <sub>4</sub> <sup>2-</sup>	Ba <sup>2+</sup> /Cl <sup>-</sup>
Ba <sup>2+</sup> .NO <sub>3</sub> <sup>-</sup>					
Ba <sup>2+</sup> /Cl <sup>-</sup>					
Na <sup>+</sup> /CrO <sub>4</sub> <sup>2-</sup>					
K <sup>+</sup> /CrO <sub>4</sub> <sup>2-</sup>					
Na <sup>+</sup> /NO <sub>3</sub> <sup>-</sup>					

Write the net ionic equation for each unique precipitate that forms.

Make similar tables for Sets II through V, and write net ionic equations for those sets.

### Questions

- Make up your own solubility chart by using your observations to fill out the table below. Use the same symbols that are used in Appendix H. Check your results against the solubility table in Appendix H.

	Cl <sup>-</sup>	CrO <sub>4</sub> <sup>2-</sup>	NO <sub>3</sub> <sup>-</sup>	OH <sup>-</sup>	SO <sub>4</sub> <sup>2-</sup>
Ag <sup>+</sup>					
Al <sup>3+</sup>					
K <sup>+</sup>					
Mg <sup>2+</sup>					
Na <sup>+</sup>					
Sr <sup>2+</sup>					

- Referring to the table you have prepared, make generalizations if you can about the solubility of compounds containing:
  - The nitrate anion.
  - The sodium cation.
  - The potassium cation.

**Additional Resources**

Check Appendix M for additional resources for this experiment.

## EXPERIMENT 12: TITRATIONS OF ACIDS AND BASES

### Objectives

1. Develop proficiency in the use of a buret and the techniques of titration.
2. Prepare a standard solution with a volumetric flask for use in titration.
3. Standardize a sodium hydroxide solution by titration.
4. Determine the concentration of an unknown acid.

### Materials and Equipment

Oxalic acid dihydrate,  $\text{H}_2\text{C}_2\text{O}_4 \cdot 2 \text{H}_2\text{O}$  (126.0 g/mol)

6 M NaOH

Phenolphthalein indicator solution

Unknown oxalic acid solutions

Two burets and a buret clamp, iron ring stand, 100 mL volumetric flask, two 250 mL Erlenmeyer flasks with stoppers, one 50 mL beaker or weigh boat, one 125 mL Erlenmeyer flask, small plastic funnels, top-loading balance, wash bottle, glass stirring rod, disposable droppers, safety glasses.



### Introduction

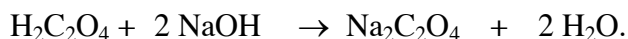
Chemists refer to the branch of chemistry that deals with the composition of matter as *analytical chemistry*. There are two sub-branches of analytical chemistry: *qualitative analysis* and *quantitative analysis*. *Qualitative analysis* allows you to determine whether or not a material is present or absent, but doesn't give you information on how much is present. If you have completed Experiment 5 in this lab manual, you have already performed qualitative analysis.

*Quantitative analysis* gives you information about the amounts of compounds or elements in a sample. Quantitative analysis can be performed on gaseous, liquid or solid samples. For example, if you ever have received a report from your doctor on medical laboratory work done to test your blood for levels of physiological compounds, your samples were analyzed by quantitative analysis. If you have read a food label and it tells you how much sodium is contained in a serving, that information also was the result of quantitative analysis. If you have received a report from your water company about the levels of different metals in your water, this information was generated using quantitative analysis.

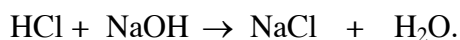
In this two-week laboratory, you will be using a basic form of quantitative analysis to determine how much acid or base is present in aqueous samples. Determining the concentration of a solution, e.g., how many moles of solute there are per Liter of solution, is called *standardization*. One of the common laboratory techniques used for standardization is *titration*. In *titration*, you compare the concentrations of two solutions by measuring the ratio of volumes required in a known reaction. For this experiment, you will be using the neutralization reaction of an acid with a base. This experiment will be carried out over two weeks and is broken down into four parts:

1. Prepare an oxalic acid solution of known concentration, to be used as a primary standard, using the top-loading balance and volumetric flask.

- Dilute a concentrated solution of sodium hydroxide to make a solution of approximate concentration.
- Determine the precise concentration of the sodium hydroxide solution you made in Part 2, by neutralizing it with the oxalic acid solution from Part 1. The technique used to carry out the neutralization in this experiment is *titration*. You will be using burets to make precise measurements of volume to carry out the titration. More detail about titration is given later in this Introduction. The neutralization reaction you will be using in Part 3 is:



- Use the sodium hydroxide solution with the now known concentration from Part 3 to titrate a hydrochloric acid solution of unknown concentration. Part 4 will also require the use of the burets. The neutralization reaction you will be using in Part 4 is:

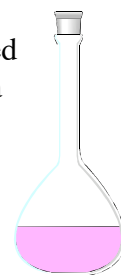


Parts 1 through 3 can be carried out in one lab period. The sodium hydroxide solution from Part 3 will be used to carry out the titrations in Part 4 during a second lab period.

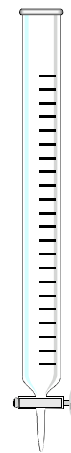
### Volumetric Equipment and Standard Solutions

*Volumetric glassware* is equipment that has been calibrated allow you to make very accurate and precise volume measurements. In other words, volumetric glassware has a smaller amount of uncertainty than regular glassware, like beakers and graduated cylinders. The two kinds of volumetric equipment you will use in this experiment are the volumetric flask and burets.

The volumetric flask you will use in this experiment has been individually calibrated to hold the stated volume (100 mL) to the closest milliliter. There is an illustration of a volumetric flask next to this paragraph and also in Appendix J (Example Experimental Set-ups). Notice that it has a very narrow neck and a calibration mark on the neck. To properly use a volumetric flask, the bottom of the meniscus of the liquid you are using must be level with the calibration mark. Remember, as always, that your eye must be level with the meniscus to get the most accurate reading.



You use the volumetric flask to make *standard solutions*. A *standard solution* is one that you know the concentration accurately. One of the most common ways of preparing a standard solution is to use a solid as the standard, as it is easier to accurately weigh out a solid than to pour out a given amount of a liquid. In this experiment you will use crystals of oxalic acid dihydrate,  $\text{H}_2\text{C}_2\text{O}_4 \cdot 2 \text{H}_2\text{O}$ , to make up a standard oxalic acid solution. As in Experiment 10, the waters of hydration or crystallization are important in calculating the molar mass (molecular weight) of the compound when it is in the solid form. Why? Because only a portion of the mass that you weigh out actually contains the oxalic acid – the rest is the mass of water. Once the solid is dissolved in solution, you no longer care about the waters of hydration – they will have no effect in the experiment. So in calculating how much oxalic acid is actually in solution, this is an important point to remember.



A buret is a cylindrical graduated tube with a stopcock (valve) that you turn to control the flow of solution into another container. Note that if the handle of the stopcock is parallel to the lab counter, it will be closed; if it is vertical, in line with the buret itself, it will be totally open. You can look at the buret as a variation of the graduated cylinder, as it has markings for volume similar to a graduated cylinder. But in a buret, the numbers are largest at the bottom, so the volume dispensed by a buret is determined by the difference in the volume readings obtained before and after the solution is dispensed into another container. Just like a graduated cylinder or the volumetric flask, it is important to read the volume of the liquid at the bottom of the meniscus. Your 50 mL buret is calibrated in tenths of milliliters, so you can estimate the volume to the nearest hundredth of a milliliter (0.01 mL) when reading the buret.

Your laboratory instructor will give you more information about using the volumetric flask and burets. There are also details included in the Procedures section. Just be aware that these are delicate pieces of glassware and quite easy to break, so please be careful with them.

### Dilution of a Concentrated Solution

Sometimes it is not easy or possible to make a standard solution of a compound, as you will with oxalic acid. In the case of sodium hydroxide, there are several factors that make it difficult to weigh out an accurate amount of sodium hydroxide and make a standard solution: Sodium hydroxide pellets absorb water very quickly. You can actually see the water from the air being absorbed onto the surface of the pellets while you are trying to weigh them; Sodium hydroxide will react with carbon dioxide from the air to create sodium carbonate. This will result in a solution that has at least a small amount of carbonate and less hydroxide than you would expect from the mass of sodium hydroxide pellets you added.

Thus, in the case of sodium hydroxide solutions, you only obtain an approximate concentration by weighing out the pellets and dissolving them in water. So the concentrated NaOH solution you will be using to make a dilute solution of NaOH only has an approximate concentration of 6 M NaOH. The concentration of the dilute solution has to be determined by titration, as in Part 3 of the experiment.

However, you can determine the approximate concentration of your diluted solution by using the following concentration relationship:

$$V_{\text{dil}} M_{\text{dil}} = V_{\text{con}} M_{\text{con}}$$

where  $V_{\text{dil}}$  is the volume of dilute solution  
 $M_{\text{dil}}$  is the molarity of dilute solution  
 $V_{\text{con}}$  is the volume of concentrated solution  
 $M_{\text{con}}$  is the molarity of concentrated solution.

You will be making 250 mL of a dilute sodium hydroxide solution by diluting 9 mL of 6M NaOH with deionized water. You will use the technique of titration in Part 3 to accurately determine the concentration of your dilute solution.

### Standardizing a Solution by Titration

We use the term *standardization* in this experiment to indicate the process of determining the concentration of a solution. One of the most common techniques of standardization is called *titration*. In this method, you compare the ratios of volumes of two solutions that react together

to form a known product. As was mentioned earlier in this Introduction, you will be using two different acid/base reactions in Parts 3 and 4 to make these comparisons. In each case, you will know the concentration of one of the solutions and will determine the concentration of the other. In Part 3, you will know the concentration of the oxalic acid solution that you made in Part 1 and will determine the concentration of the sodium hydroxide solution you made in Part 2. In Part 4, you will know the concentration of the sodium hydroxide solution from the titration you carried out in Part 3. You will use that sodium hydroxide solution to carry out the titration in Part 4 to determine the concentration of a hydrochloric acid solution.

In Parts 3 and 4, we are relying on the neutralization reaction to let us know when we have added an equal amount of  $\text{H}^+$  ions from the acid and  $\text{OH}^-$  ions from the base. Each oxalic acid molecule can produce two  $\text{H}^+$  ions. In other words, two molecules of sodium hydroxide are required to react with each molecule of oxalic acid

How will you know when you have added an equal amount of base to acid? Since you will be working with colorless solutions, you need to use an *indicator solution* that tells you when the reaction is complete. In both Parts 3 and 4 you will use phenolphthalein, a compound that turns from colorless to pink when exposed to a basic solution.

If you have not already covered neutralization reactions and stoichiometry during lecture, your instructor will provide you with additional information to help you with the titration calculations.

## Procedures

**Proper Waste Disposal:** Your instructor will provide you with detailed instructions for disposing the wastes generated in this laboratory. The neutral waste solutions generated in Parts 3 and 4 may be discarded down the drain.

### Part 1. Preparing a Standardized Solution

**Safety Note:** Use gloves when handling oxalic acid dihydrate – it can produce an allergic rash if you are sensitive.

1. Use a plastic weigh boat or 50 mL beaker. Tare the container on a top-loading balance. Add between 1.50 and 1.75 g of oxalic acid dihydrate,  $\text{H}_2\text{C}_2\text{O}_4 \cdot 2 \text{H}_2\text{O}$  to your tared container and record the precise mass.
2. Carefully transfer the oxalic crystals to a clean 100 mL volumetric flask. Use a funnel to help you transfer the solid without loss.
3. Use a small amount of deionized water to rinse any crystals from your tared container and the funnel into the volumetric flask.
4. Add enough deionized water to fill the volumetric flask about one-half to two-thirds full. You want to leave plenty of room to allow you to swirl the volumetric flask to help the oxalic acid crystals dissolve.

5. Swirl the volumetric flask until all the crystals are completely dissolved. You should not see any solid residue.
6. Carefully add more deionized water until the bottom of the meniscus comes nearly to the etched calibration mark on the narrow neck of the volumetric flask. When you get close, add the water drop-wise until the bottom of the meniscus is right at the calibration mark.
7. Place the stopper firmly in the volumetric flask. Hold the stopper in and invert the volumetric flask. Shake the flask several times. Turn the flask back upright. Repeat this process about ten times to make sure the solution is completely mixed and homogeneous.
8. Put the solution just prepared into a clean, dry 250 mL Erlenmeyer flask and stopper it tightly. Label the flask with the name of the substance, concentration, date and your drawer number. Keep this solution in your lab drawer until you are ready to use it in Part 3.

#### Part 2. Preparing a Solution by Dilution

1. Pour 9 mL of 6 M sodium hydroxide solution into another clean 250 mL Erlenmeyer flask.
2. Add enough deionized water to bring the level of solution to the 250 mL mark on the flask. Use a clean glass stirring rod to mix the solution thoroughly. Stopper the flask tightly. Label the flask with the name of the substance, approximate concentration, date and your drawer number. Keep this solution in your lab drawer until you are ready to use it in Part 3. (Can you think of the reason why the amount of water added is not critical in this part?)

### Part 3. Standardizing a Sodium Hydroxide Solution by Titration

1. Clean two burets thoroughly. First wash them with tap water, and then rinse them several times with small amounts of deionized water. Make certain to run some of the water through the tip of the buret so that it is also cleaned. A diagram of this apparatus is provided in Appendix J figure 6.
2. Next rinse one buret with several 5 mL portions of your standard oxalic acid solution you made in Part 1. Again, rinse some of the solution through the tip of the buret. You want to make certain that no deionized water has been left in the buret. Fill this buret with more of the standard oxalic acid solution. Drain a little of the solution out of the tip of the buret, making sure that there are no air bubbles in the buret.
3. Rinse the other buret with several 5 mL portions of dilute sodium hydroxide solution you made in Part 2. In the same manner as in step 2 above, rinse some of the solution through the tip of the buret. Fill this buret with more of the dilute sodium hydroxide solution. Drain a little of the solution out of the tip of the buret, again making sure that there are not air bubbles left in the buret.
4. Read each buret and record these as your initial readings.
5. Run about 20 mL of the oxalic acid solution from the buret into a clean 125 mL Erlenmeyer flask. It does not have to be dry. Then read the buret and record this as your final acid reading for this titration.
6. Put two to three drops of phenolphthalein indicator into the solution in your Erlenmeyer flask. There should be no color change at this point.
7. Add sodium hydroxide solution from the second buret at a moderate rate. Swirl the Erlenmeyer flask to make certain that the sodium hydroxide is evenly distributed. You will start to see a flash of pink color where the sodium hydroxide solution is in higher concentration – this color should rapidly disappear at first. The more sodium hydroxide solution is added, the more time it will take for the pink color to disappear. As the disappearance of the pink color takes longer, slow down the rate you add the sodium hydroxide solution. When the pale pink color persists, add the sodium hydroxide one drop at a time. The point at which there is an equal amount of acid and base (the *end point*), the pale pink color should persist for at least 15 seconds.
8. Read and record the reading from the second buret. This will be your final base reading for this titration.
9. Calculate the volume of each solution used and the concentration of the sodium hydroxide solution.
10. Rinse out the Erlenmeyer flask, and carry out two more titrations. Refill the burets if necessary.
11. Calculate the average molarity of the sodium hydroxide solution. The percentage error of your results should be no more than 5%. If it is higher, consult your instructor.

- Label the 250 mL Erlenmeyer flask containing your **dilute sodium hydroxide solution** with the calculated concentration. **Stopper tightly and place it in your lab drawer until it is needed for Part 4.** You may dispose of the standard oxalic acid solution, only after you are certain that you have completed all titrations necessary for this section of the experiment. If in doubt, ask your lab instructor.

#### Part 4. Standardizing an Oxalic Acid Solution by Titration

- Obtain an unknown oxalic acid solution from your instructor.
- Set up the two burets as in Part 3 and as shown in Appendix J figure 6. This time fill one buret with the unknown oxalic acid and the other with the dilute sodium hydroxide solution you standardized in Part 3. Remember to rinse each buret with small amounts of the solution that will be dispensed.
- Carry out three titrations between the unknown acid and the standardized sodium hydroxide solution. Add the 20 mL of acid solution into your 250 mL or 125 mL Erlenmeyer flask first, add the phenolphthalein indicator, then begin adding the sodium hydroxide solution. Remember to take both the initial and final readings of each buret for every titration.
- Calculate the molarity of the unknown oxalic acid solution from each titration and the average for the three titrations. The percentage error of your results should be no more than 5%. If it is higher, consult your instructor.

**Proper Waste Disposal:** Your instructor will provide you with detailed instructions for disposing the wastes generated in this laboratory. The neutral waste solutions generated in Parts 3 and 4 may be discarded down the drain.

#### Observations and Data

##### Part 1 Preparing a Standardized Solution

- Volume of volumetric flask
- Mass of oxalic acid dihydrate

##### Part 2. Preparing a Solution by Dilution

- Volume of 6 M NaOH used
- Final volume of diluted solution

Part 3. Standardizing a Sodium Hydroxide Solution by Titration

Oxalic Acid	Trial 1 (mL)	Trial 2 (mL)	Trial 3 (mL)
Initial Volume			
Final Volume			
Volume Used			

Sodium Hydroxide	Trial 1 (mL)	Trial 2 (mL)	Trial 3 (mL)
Initial Volume			
Final Volume			
Volume Used			

Part 4. Standardizing an Oxalic Acid Solution by Titration

Oxalic Acid	Trial 1 (mL)	Trial 2 (mL)	Trial 3 (mL)
Initial Volume			
Final Volume			
Volume Used			

Sodium Hydroxide	Trial 1 (mL)	Trial 2 (mL)	Trial 3 (mL)
Initial Volume			
Final Volume			
Volume Used			

**Calculations**

Part 1 Preparing a Standardized Solution

1. Moles of oxalic acid dihydrate
2. Molarity of oxalic acid solution

Part 2. Preparing a Solution by Dilution

1. Approximate concentration of diluted sodium hydroxide solution

Part 3. Standardizing a Sodium Hydroxide Solution by Titration

	Trial 1	Trial 2	Trial 3
Moles oxalic acid			
Moles NaOH			
Volume NaOH			
Molarity NaOH			

Average molarity of NaOH solution

Percentage difference between high and low results

#### Part 4. Standardizing an Oxalic Acid Solution by Titration

	Trial 1	Trial 2	Trial 3
Moles NaOH			
Moles Oxalic Acid			
Volume Oxalic Acid			
Molarity HCl			

Average molarity of oxalic acid solution

Percentage difference between high and low results

#### Questions

1. Why is the exact amount of water used to prepare the dilute sodium hydroxide solution in Part 2 not critical to know?
2. Suppose some deionized water was left in the buret before it was filled with the oxalic acid solution you were using as a primary standard. When you perform the standardization of sodium hydroxide in Part 3, how would the excess water affect the concentration of sodium hydroxide you calculate?
3. In Parts 3 and 4, why is it not necessary to dry the Erlenmeyer flask you use for titrations?

#### Additional Resources

Check Appendix M for additional resources for this experiment.



## EXPERIMENT 13: OBSERVING PHYSICAL PROPERTIES – BOILING POINT, SOLUBILITY AND CONDUCTIVITY

### Objectives

1. To determine the boiling point of a liquid.
2. To use different resources to obtain the boiling points of other substances.
3. To investigate the solvent properties of polar and nonpolar liquids.
4. To investigate the conductivity of aqueous solutions of ionic and covalent substances.

### Materials and Equipment



Stainless steel temperature probe, Vernier Data Logger (LabPro), computer workstation, large test tube, iron ring stand, clamp, hot plate, beakers, deionized water, safety goggles.

### Introduction

A property of a substance is a distinguishing characteristic that can be used to describe that material. Common physical properties of pure substances include boiling point, color, density, hardness, melting point, odor, solubility in different solvents, and taste. Measuring a physical property does not involve any chemical changes in the substance. Chemical properties describe the substance's ability to undergo chemical change. In other words, a chemical reaction has to occur. A common chemical property of a pure substance is flammability or its ability to combine with oxygen. These physical and chemical properties can be used to help identify a compound or determine its purity.

The *boiling point* of a compound is the temperature at which a liquid is converted to the vapor phase. Therefore that point, there are both liquid and vapor phases are present. The boiling point of a substance can be used to help identify the compound and determine its purity. The narrower the boiling point range and the closer the observations are to the established values, the more pure the substance is. In this experiment, you will determine the boiling point of a pure liquid with a temperature probe apparatus.

Similarly, the *melting point* of a compound is the temperature at which the solid is converted to the liquid phase. At that point, there are both solid and liquid phases present. As with boiling point, the melting point can be used to help identify the compound and determine its purity. The narrower the melting point range and the closer to the observations are to established values, the more pure the substance is.

A *solution* is a homogenous mixture of two substances. The two substances can have the same or different physical states. An example of a solution that you are probably very familiar with is sugar dissolved in tea or coffee. The substance being dissolved, or which is smaller in quantity is called the *solute*. The substance which is greater in quantity is called the *solvent*. What is the solvent in one situation may be the solute in another.

Substances can also be classified by the physical property of *polarity*. Polarity is a measure of how evenly electrons are distributed in the molecules that make up the substance. A *polar* substance is one where there is a non-uniform distribution of electrical charge within the

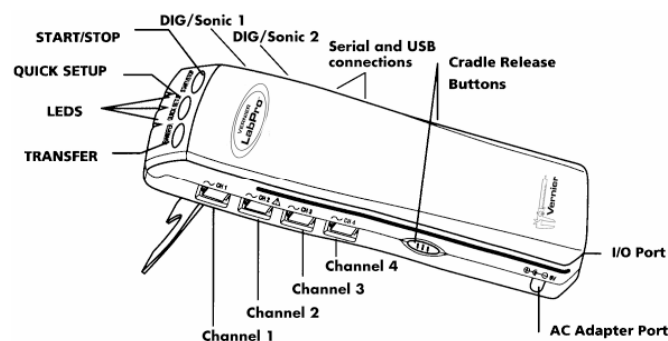
molecule. A *non-polar* substance is one where the molecules exhibit a uniform or symmetrical distribution of electrical charge. Polarity is related to the physical property of solubility. A general rule of thumb is that “like dissolves like”. In other words, a polar solute more readily dissolves in a polar solvent; a non-polar solute dissolves better in a non-polar solvent. *Miscible* is special term that is used when two liquids are mutually soluble in any proportion. Solutions prepared when solutes are dissolved with water as the solvent are referred to as *aqueous* solutions. This experiment will require you to determine the solubility or miscibility of a series of solutes and solvents.

When you dissolve an ionic compound such as table salt (NaCl) in water, the positive and negative ions separate (*dissociate*) and become surrounded by water molecules. As a result, these kinds of aqueous solutions can conduct electricity. The compounds that have this ability are referred to as *electrolytes*. Substances that do not conduct electricity when dissolved in water are called *non-electrolytes*. You will use a simple conductivity meter in this experiment to detect the presence of ions.

## Procedure

### Part 1. Boiling Point Determination

1. Pour about 5 mL of 2-propanol into a clean, dry large test tube. Add two or three small glass beads as boiling chips. This will ensure even boiling. Set this aside.
2. Use a 600 mL beaker for a water bath and fill it about half full with water – tap water is OK to use. Place the 600 mL beaker on the hot plate and assemble the rest of boiling point measuring equipment per the figures in Appendix J Figure 7.
3. The Vernier Data Logger (LabPro) will already be connected to a computer workstation.
4. Make sure power source is connected to the side of the LabPro.



5. Connect the Temperature Probe plug into the Ch 1 port on the same side as the power plug on the LabPro (see diagram above).
6. Make certain the tip of the temperature probe is about 1” from the surface of the 2-propanol.
7. Open LoggerPro software (icon should be on the computer Desktop).
8. Click on “Experiment” on top menu bar.
9. Select “Data Collection”.
10. Under “Collection” tab, select “Time based” mode; set length for 20 minutes; Sampling Rate at 1 sample per minute; 1 minute per sample.
11. Close Data Collection box.

12. Start data collection by going the Experiment menu and selecting “Start Data Collection”.  
Turn the hot plate on to a setting of about 3.5.
13. You should start to see data being collected on the Left side of your screen.
14. You can stop collecting data after the temperature has stopped rising. To stop data collection, return to the Experiment menu and select “Stop Data Collection”.
13. Print your data to one of the network printers for inclusion in your lab report.
14. Let your apparatus cool down, disassemble and clean.
15. Look up the accepted value for the boiling point of 2-propanol in the organic section of the “Handbook of Chemistry and Physics” or on one of the on-line resources at the end of this experiment.
16. Look up the accepted values for boiling point and molecular masses of the following hydrocarbons: methane, ethane, propane, butane, pentane, hexane, heptane, octane, nonane, and decane.

## Part 2. Miscibility

[Note: These may be provided to you.]

1. Label six clean, dry large test tubes 1 – 6.
2. Place the following substances in the different test tubes:  
Test Tube 1: 3 mL deionized water and 3 mL ethyl acetate;  
Test Tube 2: 3 mL deionized water and 3 mL methanol;  
Test Tube 3: 3 mL deionized water and 3 mL hexane;  
Test Tube 4: 3 mL methanol and 3 mL ethyl acetate;  
Test Tube 5: 3 mL methanol and 3 mL hexane;  
Test Tube 6: 3 mL ethyl acetate and 3 mL hexane.
3. Stopper and shake the contents of each test tube. Which of these combinations are miscible and which are not miscible. Record your observations in a table.

## Part 3. Solubility of Solids in Liquids

[Note: These may be provided to you.]

1. Label four clean, dry large test tubes 1 – 4.
2. Put about 3 mL of deionized water in test tube 1; 3 mL methanol in test tube 2; 3 mL of hexane in test tube 3; 3 mL of ethyl acetate in test tube 4.
3. Add one crystal of iodine to each of the test tubes, stopper, shake and observe. Record your observations in a table.
4. Repeat steps 1 – 3 with the following solids instead of iodine: sodium chloride, sucrose, potassium nitrate and paraffin.

## Part 4. Electrolytes

[Note: These may be provided to you.]

1. Fill the small plastic beakers provided with the materials in each Nalgene bottle.

2. Test the conductivity of each material by immersing the electrodes of an unplugged conductivity apparatus in one of the beakers. Connect the plug to a power strip. Record the appearance of the light bulb. Does it glow brightly, glow dimly, or not glow at all?
3. Unplug the conductivity meter and remove the electrodes from the beaker.
4. Record your observations in a table.

### **Observations and Data**

#### Part 1. Boiling Points

1.  Observed boiling point of 2-propanol.
2.  Accepted value from handbook.
3.  Boiling points and molecular masses of hydrocarbons from the handbook or other resource: methane, ethane, propane, butane, pentane, hexane, heptane, octane, nonane, and decane

#### Part 2. Miscibility

- Organize your data in tabular form.

#### Part 3. Solubility of Solids in Liquids

- Organize your data in tabular form.

#### Part 4. Electrolytes

- Organize your data in tabular form.

### **Questions**

1. What trends do you observe in boiling point and molecular mass for the hydrocarbons that you checked in the “Handbook of Chemistry and Physics”?
2. What generalizations can you make about the nature of solvents that dissolved ionic solids the best?
3. What generalizations can you make about solids that are electrolytes?
4. The four liquids that you tested may be ranked in order of decreasing polarity as follows: (most polar) water > methanol > ethyl acetate > hexane (least polar). Based on the data you gathered, what generalizations can you make about the relationship of polarity and miscibility?

5. Based on your answer to question 4, which of the four liquids would probably best dissolve a polar solid such as NaCl?
6. Which would best dissolve a nonpolar solid such as paraffin?

### **Additional Resources**

Check Appendix M for additional resources for this experiment.



## APPENDIX A: CONVERSION FACTORS

### USING CONVERSION FACTORS DIMENSIONAL ANALYSIS (UNIT FACTOR ANALYSIS)

*For example: How many feet are in 60 inches?*

1. Identify the unknown.  
(What are you looking for?)

$$ft? =$$

2. Identify the given quantity.  
(What kind of number are you given to start with?)

$$ft? = 60 in$$

3. Find the appropriate equality or equalities to solve the problem.  
(What equation relates the two kinds of units? In some cases, there may not be just one equation that relates the two kinds of units and you may have to repeat the process.)

$$1 ft = 12 in$$

4. Make a fraction (conversion factor) that will cancel the first kind of unit and bring you closer to the desired kind of unit when you multiply.

$$\frac{1 ft}{12 in} \quad \text{or} \quad \frac{12 in}{1 ft} \quad ???$$

Remember that each equality can generate two conversion factors or fractions.
---

$$ft? = 60 in \times \frac{1 ft}{12 in}$$

5. Multiply to solve the problem.

$$ft? = 60 in \times \frac{1 ft}{12 in} = 5 ft$$

Note: You may have to repeat these steps if you did not find one equality that contained both kinds of units.

## SELECTED EQUALITIES TO USE IN CONVERSION FACTORS

### UNITS OF LENGTH: meter (m)

1 kilometer	=	1000 meters
1 meter	=	10 decimeters
1 meter	=	100 centimeters
1 meter	=	1000 millimeters
1 centimeter	=	10 millimeters
2.54 centimeter	=	1 inch (exact)

### UNITS OF MASS: gram (g)

1 kilogram	=	1000 grams
1 gram	=	1000 milligrams
1 kilogram	=	2.20 pounds (not exact)
1 pound	=	454 grams (not exact)

### UNITS OF TEMPERATURE: degrees Celsius ( $^{\circ}\text{C}$ )

$T_{\text{F}}$	=	$(1.8) T_{\text{C}} + 32$	$(T_{\text{F}}$ in $^{\circ}\text{F}$ , $T_{\text{C}}$ in $^{\circ}\text{C}$ )
$T_{\text{K}}$	=	$T_{\text{C}} + 273$	$(T_{\text{K}}$ in Kelvin, $T_{\text{C}}$ in $^{\circ}\text{C}$ )

### UNITS OF VOLUME: liter (l)

1 kiloliter	=	1000 liters
1 liter	=	1000 milliliters
1 liter	=	1.06 quarts (not exact)
1 quart	=	946 milliliters (not exact)

### PREFIXES

Prefix	Symbol	Number	Power of 10
mega-	M	1,000,000	$1 \times 10^6$
kilo-	k	1,000	$1 \times 10^3$
hecto-	h	100	$1 \times 10^2$
deca-	da	10	$1 \times 10^1$
deci-	d	0.1	$1 \times 10^{-1}$
centi-	c	0.01	$1 \times 10^{-2}$
milli-	m	0.001	$1 \times 10^{-3}$
micro-	$\mu$	0.000001	$1 \times 10^{-6}$

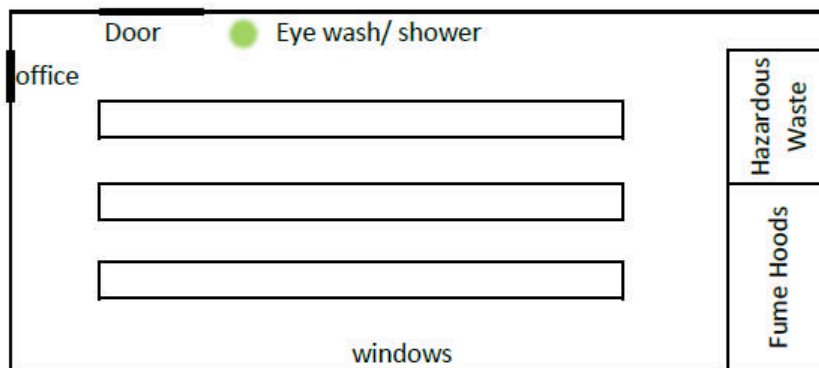
### OTHER RELATIONSHIPS:

length and volume  
 $1 \text{ cm}^3 = 1 \text{ mL}$  for any substance

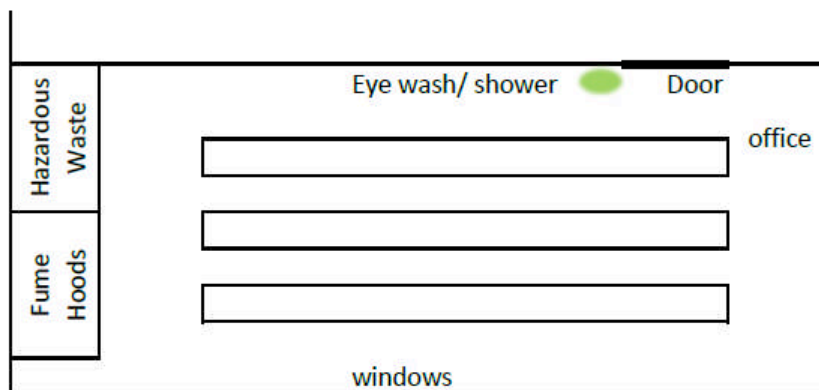
volume and mass  
 $1 \text{ mL} = 1 \text{ g}$  for  $\text{H}_2\text{O}$  at  $3.9^{\circ}\text{C}$

## APPENDIX B: LAB LAYOUTS WITH LOCATIONS OF SAFETY EQUIPMENT

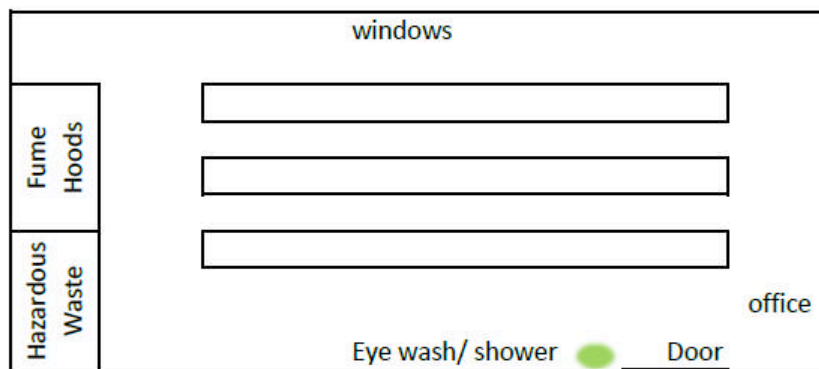
M 23



M 24



M 26



## APPENDIX C: PROPER WASTE DISPOSAL

Several types of waste will be generated throughout the course of chemistry 22 and are separated in clearly labeled containers. When the contents of any waste container reaches the full mark (located on the outside of each container) notify your instructor or lab technician who will immediately prepare another container for your use. Do not fill any waste container past the full mark. Overfilling containers will create a hazard for yourself, your classmates, your instructor and the lab technician. For any broken thermometers please notify your instructor and the lab technician so that your thermometer can be replaced.

### Liquid Waste:

Acid Base waste: Please put all acidic and basic waste in this container

Heavy metals waste: Please put all waste that contains a heavy metal in this container. (examples: Iron, Copper, Silver, Nickel, Chromium, Cobalt, Manganese)

Halogenated Organic Solvent waste: Put all waste that contains halogen (examples: Chlorine, Bromine, and Iodine)

Non-halogenated Organic Solvent waste: Put all waste that contains hexane without a halogen into this container

MnO<sub>2</sub> and H<sub>2</sub>O<sub>2</sub> waste: Put all waste that contains Manganese Oxide and Hydrogen Peroxide in this container

KI and I<sub>2</sub>: Put all waste that contains Potassium Iodide, Iodine and \_\_\_\_\_ in this container

### Solids:

Zinc chips, Marble chips: Put all zinc and Marble chips in this container. Drain liquid into \_\_\_\_\_ waste container before adding the chips to their appropriate containers.

Magnesium/sulfur/copper: All magnesium and copper in various states (shots, powder, ribbon) are to be disposed of in this container including copper with sulfur residue.

Inorganic waste:

Broken Thermometers: All broken thermometers are to be disposed of in this container

Capillary tubes: All capillary tubes are to be disposed of in this container

### Glass Waste:

Notify your instructor if glass is broken in the classroom. There are dust pans and brooms in the laboratory coat closet and in each scale room. Use caution when sweeping and disposing of glass so that others are not at risk of injury. The glass disposal box is located in a corner of each classroom and can be located in Appendix B.

## Chemical Labels

Each container that contains a hazardous chemical must be labeled by the manufacturer or distributor before it is sent to downstream users. Each label will contain the identity of the hazardous chemical(s) by common or chemical name. Appropriate hazard warnings. The name and address of the manufacturer, distributor, or the responsible party. You will see three types of labels in the classroom, 1) The right to know label, 2) Manufacture labels and 3) Chemistry stockroom labels

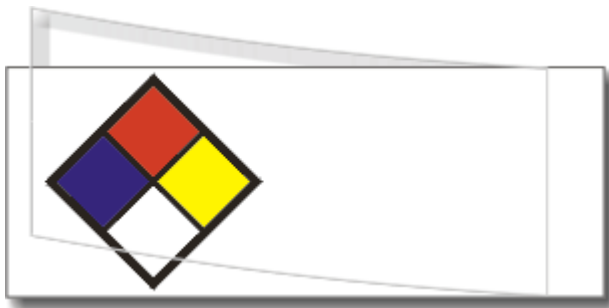
- 1) The right to know label: A standardized color coded label that will identify specific hazard, the strength of the hazard, Chemical name, molecular formula and CAS. These labels can be printed and hand written.

Blue: Health

Red: Flammability

Yellow:

White: reactivity



- 2) Manufacturers labels:  
These labels are not standardized and vary a great deal. The important information printed on these containers is the chemical name/common name/molecular formula, concentration (liquids/aq), a few potential hazards, and the manufacturers name. Often these labels will also have a chemistry stockroom label or right to know label added.
- 3) Chemistry stockroom label:  
These labels are generated by Laboratory technicians and are hand written. These labels will contain chemical name or molecular formula, concentration (liquids/aq),

## APPENDIX D: USEFUL MATH RELATIONSHIPS

### Absolute Error and Percent Error

Accuracy refers to the closeness of an experimental value to its “true” value. Quantitatively, accuracy is represented by absolute error and percent error.

Absolute error is the difference between the experimental value and the true value:

$$\text{Absolute error} = \text{experimental value} - \text{true value} \quad (1).$$

The percent error is the absolute error relative to the true value:

$$\% \text{ error} = \frac{\text{absolute error}}{\text{"true" value}} \times 100 \quad (2).$$

### Deviation and Average Deviation

Precision refers to the closeness of a set of data to each other. Precision can be quantified by calculating the individual deviation of a result and the average deviation of a set of results:

$$\text{Deviation} = \text{individual result} - \text{average value} \quad (3).$$

$$\text{Average deviation} = \frac{\sum |\text{individual result} - \text{average value}|}{\text{Number of measurements}} \quad (4)$$

### Percentage Error

If you have to compare two or more values to one another without knowing what is the “true” value, you can use the percentage error. A percentage difference between two (or more) numbers may be obtained by taking the difference between the high and low values, dividing that number by the average value, and then multiplying by 100%:

$$\% \text{ difference} = \frac{\text{high value} - \text{low value}}{\text{average value}} \times 100\% \quad (5).$$

## APPENDIX E: RULES FOR COUNTING SIGNIFICANT FIGURES

**1. Nonzero integers:** any digit from 1 through 9 always counts as a significant figure.

Example: 1234.56789 has 9 significant figures.

**2. Zeros:** there are three kinds of zeros:

a. Leading zeros (zeros to the left of nonzero digits) never count as significant figures.

Example: 0.0025 has 2 significant figures.

b. Captive zeros (zeros between nonzero digits) always count as significant figures

Example : 1.008 has 4 significant figures.

c. Trailing zeros – zeros to the right of nonzero digits only count if the number contains a decimal point.

Example: 100 has 1 significant figure.

Example:  $1.00 \times 10^2$  has 3 significant figures.

Example : 100. has 3 significant figures.

**3. Exact numbers** have an infinite number of significant figures. There are two kinds of exact numbers:

a. Counted numbers

Example: 10 experiments, 3 apples or 8 molecules have an infinite number of significant figures.

b. Defined numbers

Example: 1 kilogram = 1,000 grams have infinite numbers of significant figures.

Hint : Sometimes writing numbers in proper scientific notation will make counting significant figures less ambiguous.

### Additional Resources

Bishop “An Introduction to Chemistry, Atoms First”, Chapter 2, Unit Conversions  
[http://en.wikipedia.org/wiki/Significant\\_figures](http://en.wikipedia.org/wiki/Significant_figures)

## APPENDIX F: RULES FOR ROUNDING OFF

**1. After calculating a number using multiplication or division,** you must round off the answer to the correct number of significant figures.

a. Determine whether each value is *exact*, and ignore exact values in counting significant figures.

**Remember:** *exact* numbers are of two types:

counted numbers (e.g., 12 objects = 1 dozen exactly) or  
defined numbers (e.g., 1000 mL = 1 L exactly). Look in Appendix E for help.

b. Determine the number of significant figures for each value that is measured.

c. Round off the answer (considering rule 3 below) to the same number of *significant figures* as the measured value with the fewest number of significant figures.

Example:  $1.83 \text{ cm} \times \frac{1 \text{ in.}}{2.54 \text{ cm}} = 0.72047244 \text{ in.} = 0.720 \text{ in.}$

**2. After calculating a number using addition or subtraction,** you must round off the answer to the correct number of decimal places.

a. Determine whether each value is exact, and ignore exact values.

b. Determine the number of decimal places for each value that is not exact.

c. Round off the answer (considering rule 3 below) to the same number of *decimal places* as the measured value with the fewest decimal places. Remember that this rule is different than the rule for multiplication and division.

Example:  $43.6 \text{ g} + 132.31 \text{ g} = 175.9 \text{ g}$

**3. The following two rules always apply when you are rounding off:**

a. When the first digit of those to be dropped is less than 5, leave the preceding digit unchanged.

Example: the number 56.748 rounded off to the nearest 0.1 becomes 56.7.

b. If the first digit of those to be dropped is 5 or greater than 5, raise the preceding digit by 1.

Example: the number 2.146 rounded off to the nearest 0.01 becomes 2.15.

## **Additional Resources**

Bishop “An Introduction to Chemistry, Atoms First”, Chapter 2, Unit Conversions

<http://chemed.chem.purdue.edu/genchem/topicreview/bp/ch1/sigfigs.html>

[http://en.wikipedia.org/wiki/Significant\\_figures](http://en.wikipedia.org/wiki/Significant_figures)

## APPENDIX G: FORMULAS AND CHARGES FOR SELECTED IONS

	POSITIVE IONS (CATIONS)			NEGATIVE IONS (ANIONS)	
1+	Ammonium Copper (I) Cuprous Hydrogen Lithium Potassium Silver Sodium	NH <sub>4</sub> <sup>+</sup> Cu <sup>+</sup>  H <sup>+</sup> Li <sup>+</sup> K <sup>+</sup> Ag <sup>+</sup> Na <sup>+</sup>		Acetate Bromate Bromide Chlorate Chloride Chlorite Cyanide Fluoride Dihydrogen phosphate Hydride Hydrogen carbonate (Bicarbonate) Hydrogen sulfate (Bisulfate) Hydrogen sulfite (Bisulfite) Hydroxide Hypochlorite Iodate Iodide Nitrate Nitrite Perchlorate Permanganate Thiocyanate	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> <sup>-</sup> BrO <sub>3</sub> <sup>-</sup> Br <sup>-</sup> ClO <sub>3</sub> <sup>-</sup> Cl <sup>-</sup> ClO <sub>2</sub> <sup>-</sup> CN <sup>-</sup> F <sup>-</sup> H <sub>2</sub> PO <sub>4</sub> <sup>-</sup> H <sup>-</sup> HCO <sub>3</sub> <sup>-</sup> HSO <sub>4</sub> <sup>-</sup> HSO <sub>3</sub> <sup>-</sup> OH <sup>-</sup> ClO <sup>-</sup> IO <sub>3</sub> <sup>-</sup> I <sup>-</sup> NO <sub>3</sub> <sup>-</sup> NO <sub>2</sub> <sup>-</sup> ClO <sub>4</sub> <sup>-</sup> MnO <sub>4</sub> <sup>-</sup> SCN <sup>-</sup>
2+	Barium Cadmium Calcium Chromium(II) (Chromous) Cobalt (II) (Cobaltous) Copper (II) (Cupric) Iron (II) (Ferrous) Lead (II) (Plumbous) Magnesium Manganese (II) Mercury(II) (Mercuric) Nickel (II) Strontium Tin (II) (Stannous) Zinc	Ba <sup>2+</sup> Cd <sup>2+</sup> Ca <sup>2+</sup> Cr <sup>2+</sup>  Co <sup>2+</sup>  Cu <sup>2+</sup>  Fe <sup>2+</sup>  Pb <sup>2+</sup>  Mg <sup>2+</sup> Mn <sup>2+</sup> Hg <sup>2+</sup>  Ni <sup>2+</sup> Sr <sup>2+</sup> Sn <sup>2+</sup>  Zn <sup>2+</sup>	1-		
3+	Aluminum Antimony (III) Arsenic (III) Bismuth (III) Chromium (III) (Chromic) Cobalt (III) (Cobaltic) Iron (III) (Ferric) Titanium (III)	Al <sup>3+</sup> Sb <sup>3+</sup> As <sup>3+</sup> Bi <sup>3+</sup> Cr <sup>3+</sup>  Co <sup>3+</sup>  Fe <sup>3+</sup>  Ti <sup>3+</sup>	2-	Carbonate Chromate Dichromate Monohydrogen phosphate Oxalate Oxide Peroxide Silicate Sulfate Sulfide Sulfite	CO <sub>3</sub> <sup>2-</sup> CrO <sub>4</sub> <sup>2-</sup> Cr <sub>2</sub> O <sub>7</sub> <sup>2-</sup> HPO <sub>4</sub> <sup>2-</sup>  C <sub>2</sub> O <sub>4</sub> <sup>2-</sup> O <sup>2-</sup> O <sub>2</sub> <sup>2-</sup> SiO <sub>3</sub> <sup>2-</sup> SO <sub>4</sub> <sup>2-</sup> S <sup>2-</sup> SO <sub>3</sub> <sup>2-</sup>
4+	Manganese (IV) Tin (IV) (Stannic) Titanium (IV) (Titanic)	Mn <sup>4+</sup> Sn <sup>4+</sup>  Ti <sup>4+</sup>	3-	Arsenate Borate Nitride Phosphate Phosphide Phosphite	AsO <sub>4</sub> <sup>3-</sup> BO <sub>3</sub> <sup>3-</sup> N <sup>3-</sup> PO <sub>4</sub> <sup>3-</sup> P <sup>3-</sup> PO <sub>3</sub> <sup>3-</sup>
5+	Antimony (V) Arsenic (V)	Sb <sup>5+</sup> As <sup>5+</sup>			

## APPENDIX H: SOLUBILITIES OF SELECTED SALTS

Cations	Anions														
	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> <sup>-</sup>	AsO <sub>4</sub> <sup>-</sup>	Br <sup>-</sup>	CO <sub>3</sub> <sup>2-</sup>	Cl <sup>-</sup>	CrO <sub>4</sub> <sup>2-</sup>	OH <sup>-</sup>	I <sup>-</sup>	NO <sub>3</sub> <sup>-</sup>	C <sub>2</sub> O <sub>4</sub> <sup>2-</sup>	O <sup>2-</sup>	PO <sub>4</sub> <sup>3-</sup>	SO <sub>4</sub> <sup>2-</sup>	S <sup>2-</sup>	SO <sub>3</sub> <sup>2-</sup>
Al <sup>3+</sup>	<i>aq</i>	I	<i>aq</i>	-	<i>aq</i>	-	I	<i>aq</i>	<i>aq</i>	-	I	I	<i>aq</i>	d	-
NH <sub>4</sub> <sup>+</sup>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	-	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>
Ba <sup>2+</sup>	<i>aq</i>	I	<i>aq</i>	I	<i>aq</i>	I	<i>sl. aq</i>	<i>aq</i>	<i>aq</i>	I	<i>sl. aq</i>	I	I	d	I
Bi <sup>3+</sup>	-	<i>sl. aq</i>	d	I	d	-	I	I	<i>aq</i>	I	I	<i>sl. aq</i>	d	I	-
Ca <sup>2+</sup>	<i>aq</i>	I	<i>aq</i>	I	<i>aq</i>	<i>aq</i>	I	<i>aq</i>	<i>aq</i>	I	I	I	I	d	I
Co <sup>2+</sup>	<i>aq</i>	I	<i>aq</i>	I	<i>aq</i>	I	I	<i>aq</i>	<i>aq</i>	I	I	I	<i>aq</i>	I	I
Cu <sup>2+</sup>	<i>aq</i>	I	<i>aq</i>	I	<i>aq</i>	I	I	-	<i>aq</i>	I	I	I	<i>aq</i>	I	-
Fe <sup>2+</sup>	<i>aq</i>	I	<i>aq</i>	<i>sl. aq</i>	<i>aq</i>	-	I	<i>aq</i>	<i>aq</i>	I	I	I	<i>aq</i>	I	<i>sl. aq</i>
Fe <sup>3+</sup>	I	I	<i>aq</i>	I	<i>aq</i>	I	I	-	<i>aq</i>	<i>aq</i>	I	I	<i>aq</i>	I	-
Pb <sup>2+</sup>	<i>aq</i>	I	I	I	I	I	I	I	<i>aq</i>	I	I	I	I	I	I
Mg <sup>2+</sup>	<i>aq</i>	d	<i>aq</i>	I	<i>aq</i>	<i>aq</i>	I	<i>aq</i>	<i>aq</i>	I	I	I	<i>aq</i>	d	<i>sl. aq</i>
Hg <sub>2</sub> <sup>2+</sup>	<i>sl. aq</i>	I	I	I	I	<i>sl. aq</i>	-	I	<i>aq</i>	I	I	I	I	I	-
Hg <sup>2+</sup>	<i>aq</i>	I	I	I	<i>aq</i>	<i>sl. aq</i>	I	I	<i>aq</i>	I	I	I	d	I	-
Ni <sup>2+</sup>	<i>aq</i>	I	<i>aq</i>	I	<i>aq</i>	I	I	<i>aq</i>	<i>aq</i>	I	I	I	<i>aq</i>	I	I
K <sup>+</sup>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>
Ag <sup>+</sup>	<i>sl. aq</i>	I	I	I	I	I	-	I	<i>aq</i>	I	I	I	I	I	I
Na <sup>+</sup>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>	<i>aq</i>
Sr <sup>2+</sup>	<i>aq</i>	-	<i>aq</i>	I	<i>aq</i>	I	<i>aq</i>	<i>aq</i>	<i>aq</i>	-	-	I	I	I	-
Zn <sup>2+</sup>	<i>aq</i>	I	<i>aq</i>	I	<i>aq</i>	I	I	<i>aq</i>	<i>aq</i>	I	I	I	<i>aq</i>	I	I

Key: *aq* = Soluble in water  
*sl. aq* = Slightly soluble in water

I = Insoluble in water (less than 1 g/100 g H<sub>2</sub>O)  
d = Decomposes in water

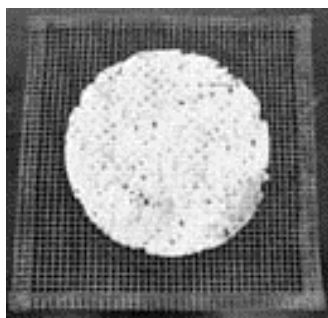
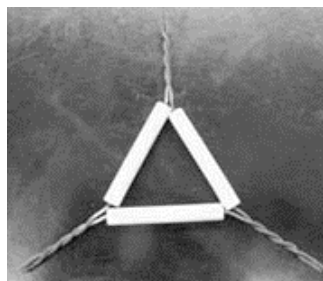
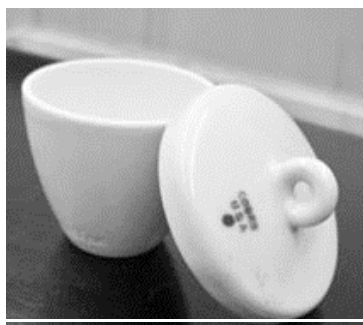
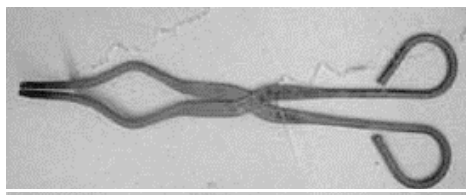
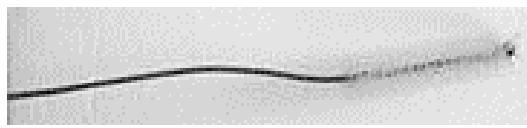
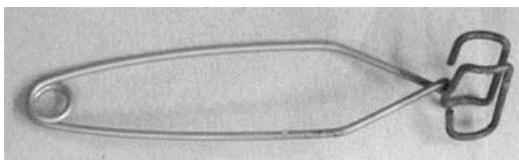
It is useful to remember:

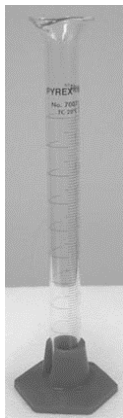
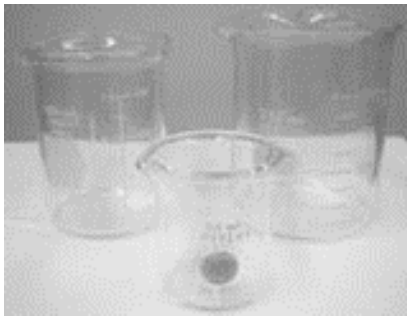
- salts with Group I or ammonium cations are always soluble no matter what the anions are
- salts with nitrate anions are always soluble no matter what the cations are (except for Bi<sup>3+</sup>)

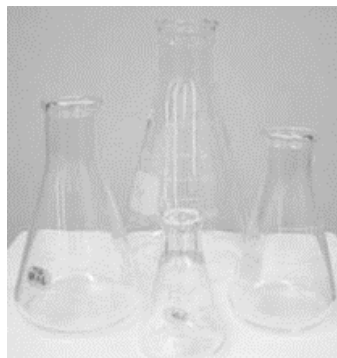
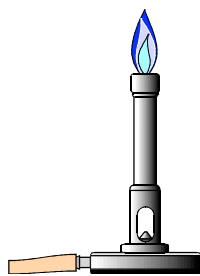
### Additional Resources

[http://en.wikipedia.org/wiki/Solubility\\_chart](http://en.wikipedia.org/wiki/Solubility_chart)  
<http://www.csudh.edu/oliver/chemdata/solrules.htm>

# APPENDIX I: COMMON PIECES OF EQUIPMENT USED IN STUDENT CHEMISTRY LABORATORIES







## APPENDIX J: EXAMPLE EXPERIMENTAL SET-UPS

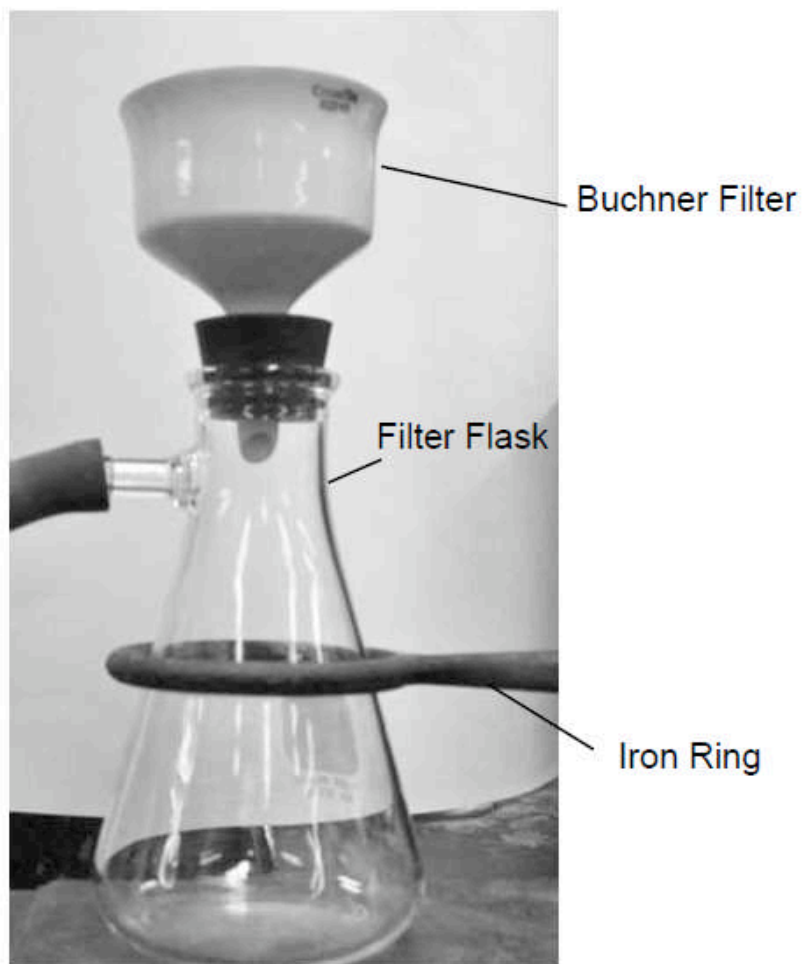
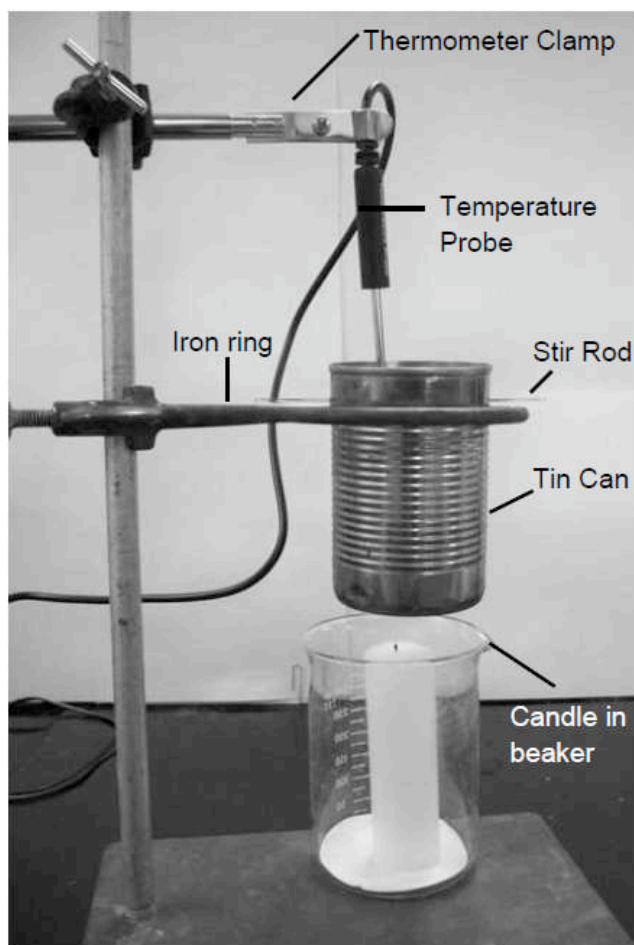


Figure 1: Experimental set up for experiment 4: determining chemical properties to separate a mixture and experiment 10: stoichiometry – producing an insoluble ionic compound

### Part 1: Heat of Combustion



### Part 2: Heat of Fusion

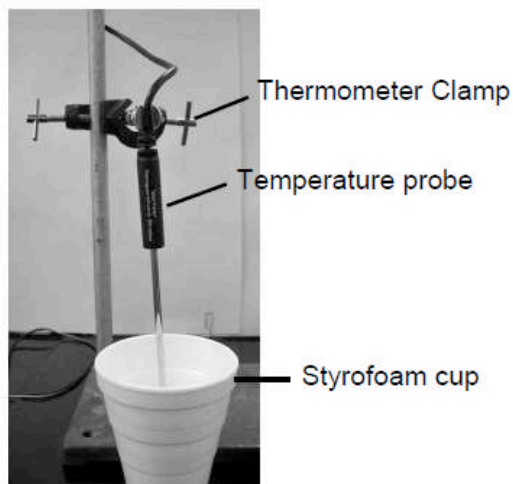


Figure 2: Experimental set up for experiment 6: comparing chemical and physical changes using a temperature probe.

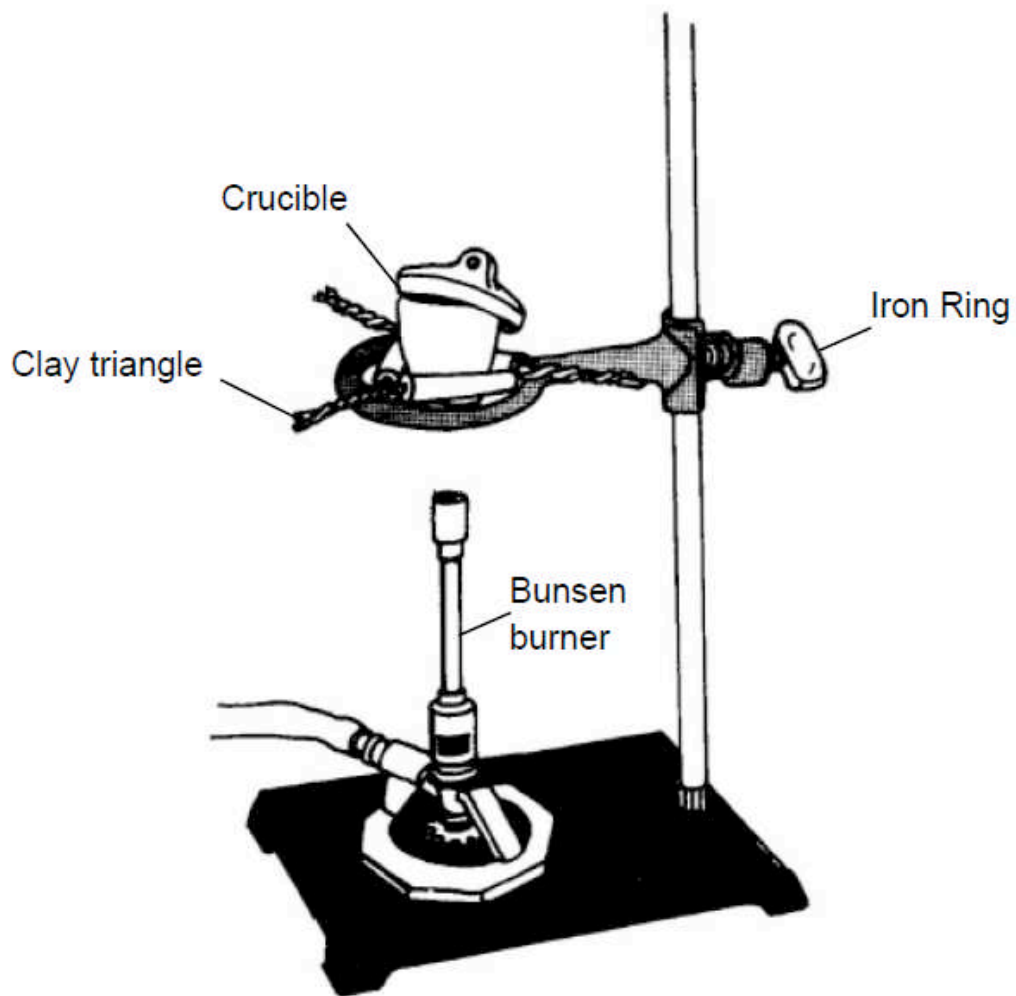


Figure 3: Experimental set up for experiment 7: calculating an empirical formula.

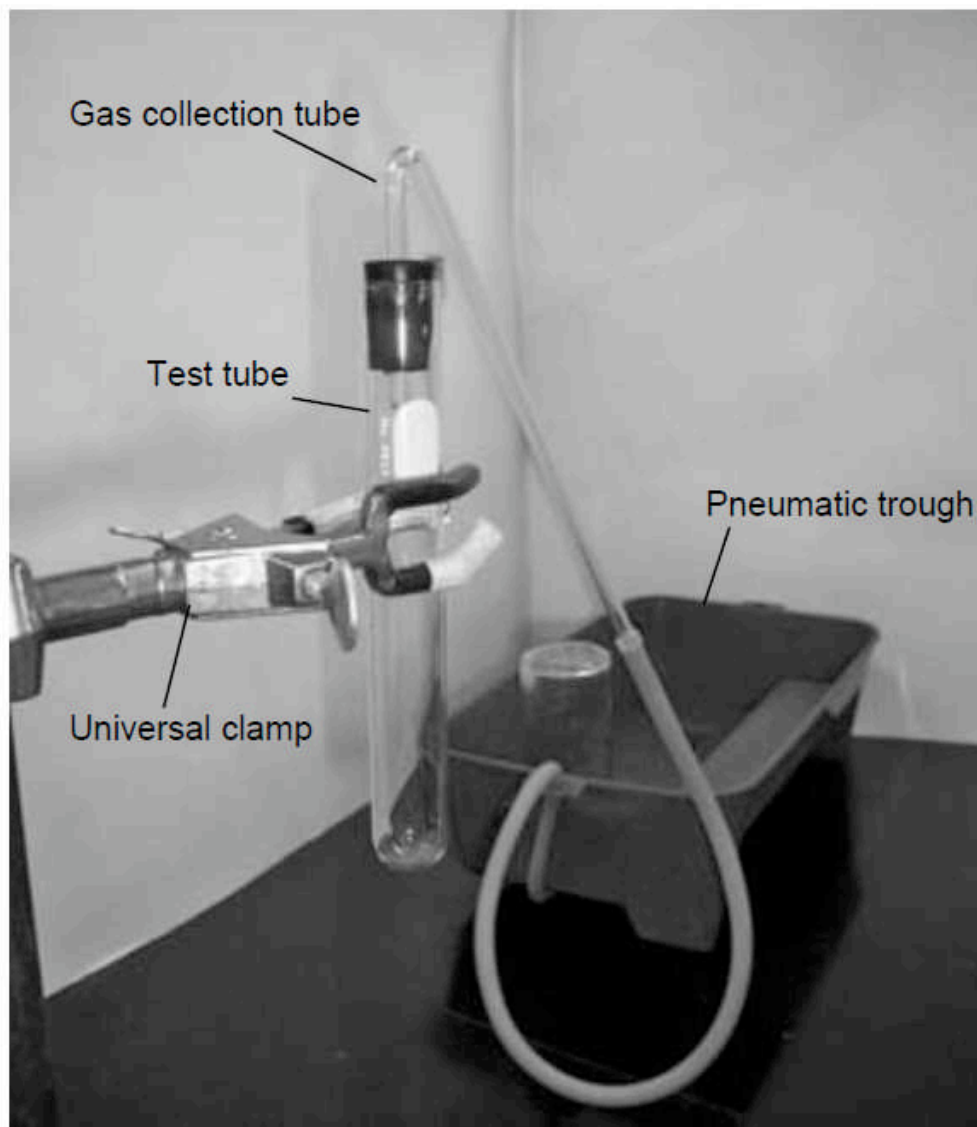
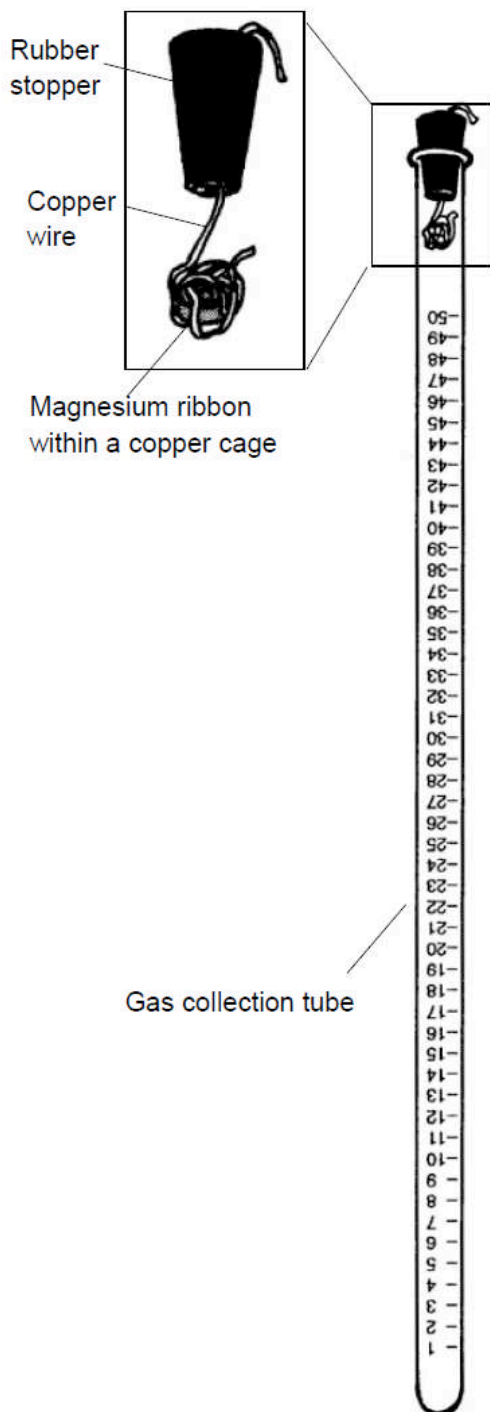


Figure 4: Experimental set up for experiment 8: production and investigation of four gases

Part 1:



Part 2:

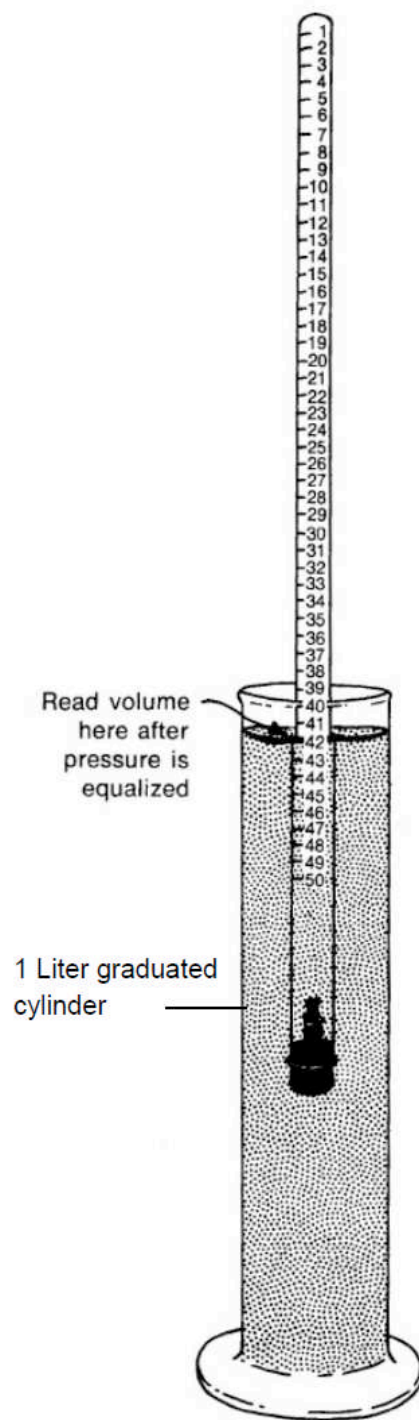


Figure 5: Experimental set up for experiment 9: gas laws—determining the molar volume of Hydrogen.

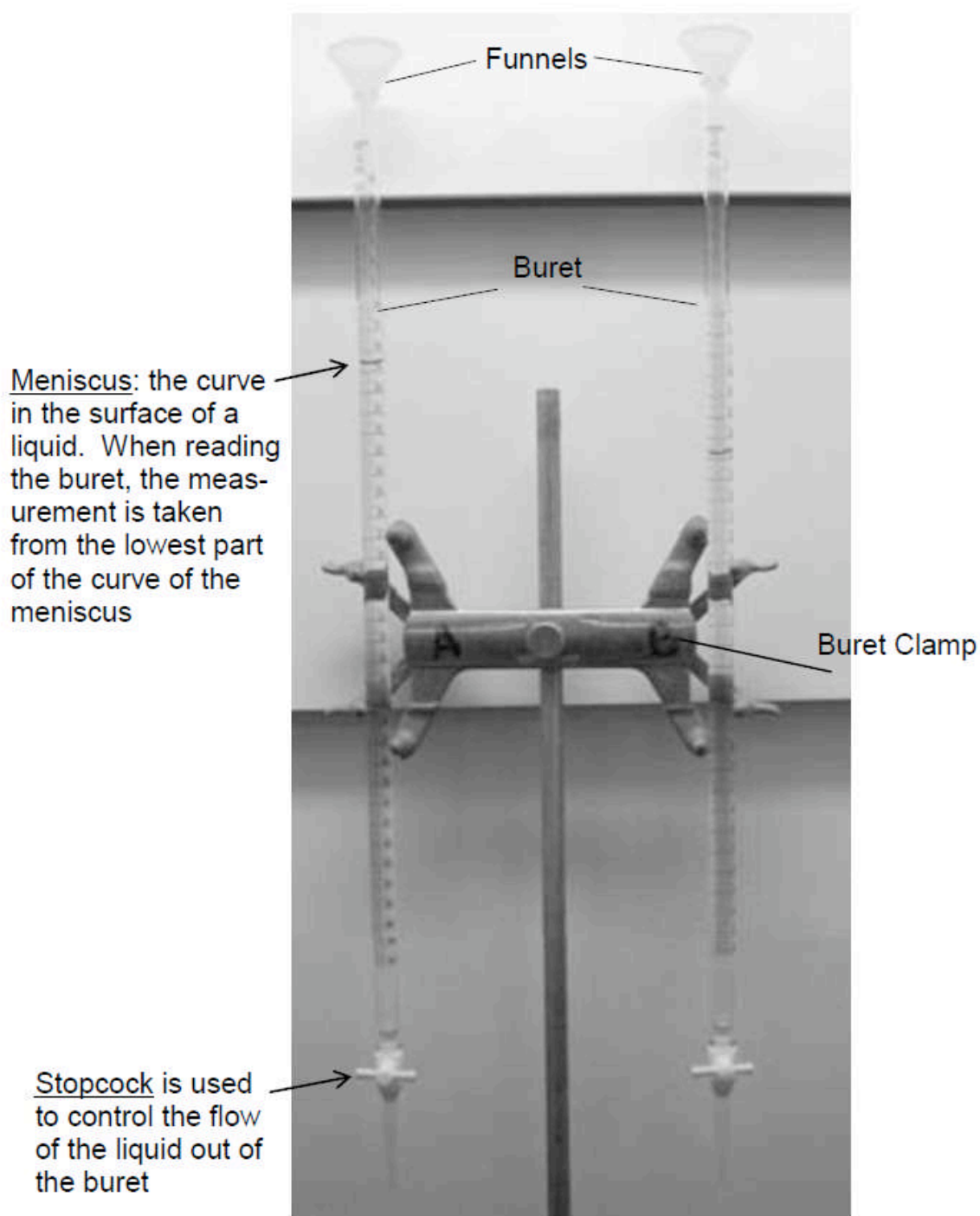


Figure 6: Experimental set up for experiment 12: titrations of Acids and Bases. **Note:** for instructions on how to read the meniscus refer to ch 3 part 3 or your laboratory notebook.

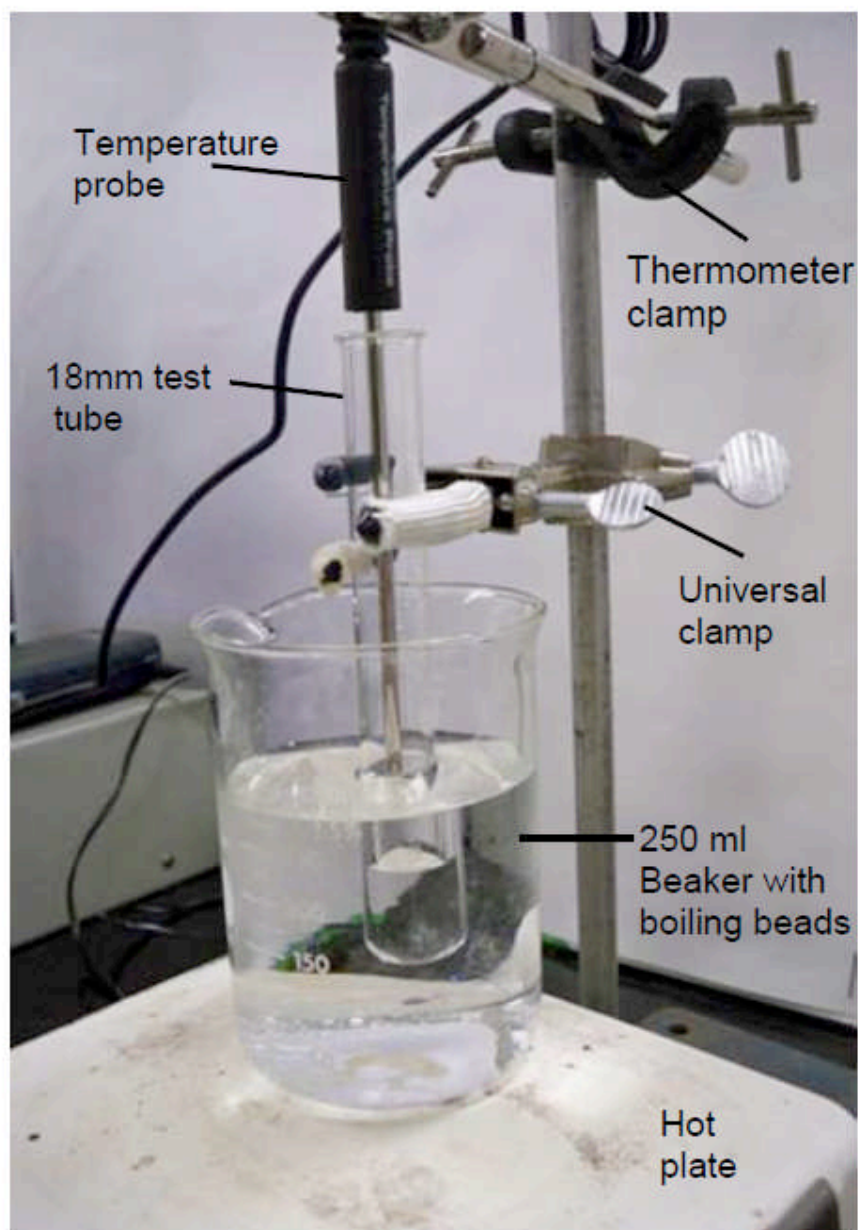


Figure 7: Experimental set up for experiment 13: observing physical properties—boiling point, solubility and conductivity. **Note:** the temperature probe does not touch the sides of the test tube while the experiment is in progress.

## APPENDIX K: LOCKER CHECK-OUT AND CHECK-IN PROCEDURES

### Locker Check OUT: beginning of the semester

- 1) Laboratory Technicians are:
  - a. \_\_\_\_\_
  - b. \_\_\_\_\_
- 2) Go through all materials that are in the locker assigned to you, following along with the Lab technician
  - a. Check for cracks and nicks in all glassware and have any unusable equipment replaced if necessary
  - b. Hold glassware up to the light to see if there are smudges or chemicals on/in glassware
- 3) Chemistry locker rules
  - a. \$25 fee will be charged for failure to clean locker at the end of the semester or once you have dropped the class. This fee is sent to the cashier who will place a hold on your record preventing you from ordering transcripts and registering for classes until the fee is paid.
  - b. Do not put shared class materials in any student locker because other classes use these materials
  - c. Fill soap bottles by the sinks using the soap dispenser at the back of the classroom under the window.
- 4) Waste
  - a. All waste has to be placed in the appropriate container as outlined in Appendix C

### Locker Check In: End of semester

- 1) Remove all materials from the locker assigned to you as the beginning of the semester
- 2) Replace paper towels on bottom of the drawer and wipe the drawer out
- 3) Using the protocol for washing glassware outlined in Appendix L, wash all glassware and dirty equipment
- 4) To check if glassware is clean, hold each piece up to the light – it should sparkle
- 5) A \$25.00 fee is charged to students who do not check out their lockers

## APPENDIX L: LABORATORY EQUIPMENT LIST

Equipment	Qt	Unit Price	Equipment	Qt	Unit Price
Beaker, griffin, low form			Flask, Erlenmeyer		
CAP: 50ml	1	\$3.59	CAP: 50ml	1	\$5.44
CAP: 150ml	1	\$3.94	CAP: 125ml	1	\$4.75
CAP: 250 ml	1	\$4.19	CAP: 250 ml	1	\$4.40
			CAP: 500ml	1	\$5.90
Bottle, polyethylene	1	\$3.72	Litmus Paper, blue & red	2	\$0.00
Brush, test tube	1	\$2.49	Padlock, combination type	1	\$14.35
Burner, macro, tirrill type	1	\$67.75	Rule, plastic	1	\$0.45
Clamp, test tube holder	1	\$22.26	Spatula, micro, stainless steel	1	\$8.51
Iron wire triangle	1	\$9.16	Stopper – #7	1	0.00
Crucible cover, “F”	1	\$8.11	Test tube, with lip		
Crucible, porcelain, “o”	1	\$3.97	SIZE: 13 x 100 mm	8	\$0.06
Crucible tong	1	\$9.39	SIZE: 18 x 150 mm	2	\$0.14
Cylinder, graduated, 10ml	1	\$12.56	Test tube rack	1	\$27.00
Dropper bulbs, disposable	2	\$0.00	Thermometer, -20° + 150°	1	\$19.17
			Wire gauze, ceramic center	1	\$1.07

## APPENDIX M: ADDITIONAL RESOURCES

### Experiment 1

Bishop “An Introduction to Chemistry, Atoms First”, Chapter 2, Unit Conversions

[http://preparatorychemistry.com/Bishop\\_Tutorials.htm](http://preparatorychemistry.com/Bishop_Tutorials.htm)

[http://en.wikipedia.org/wiki/Conversion\\_of\\_units](http://en.wikipedia.org/wiki/Conversion_of_units)

<http://physics.nist.gov/cuu/Units/units.html>

[http://www.dartmouth.edu/~chemlab/techniques/top\\_balance.html](http://www.dartmouth.edu/~chemlab/techniques/top_balance.html)

<http://www.uwplatt.edu/chemep/chem/chemscape/labdocs/labdrwr/labequip.htm>

<http://www.uwplatt.edu/chemep/chem/chemscape/labdocs/catofp/measurea/volume/gradcyl/gradcyl.htm>

### Experiment 2

Bishop “An Introduction to Chemistry, Atoms First”, Chapter 5, Chemical Compounds

[http://preparatorychemistry.com/Bishop\\_Tutorials.htm](http://preparatorychemistry.com/Bishop_Tutorials.htm)

[http://en.wikipedia.org/wiki/Molecular\\_geome](http://en.wikipedia.org/wiki/Molecular_geome)

### Experiment 3

[http://en.wikipedia.org/wiki/Bunsen\\_burner](http://en.wikipedia.org/wiki/Bunsen_burner)

<http://www.uwplatt.edu/chemep/chem/chemscape/labdocs/catofp/bunsbur/bunsbur2.htm>

### Experiment 4

CRC “Handbook of Chemistry and Physics”

<http://www.dartmouth.edu/~chemlab/techniques/vfiltration.html>

<http://www.uwplatt.edu/chemep/chem/chemscape/labdocs/catofp/mixpour/filter/filter.htm>

<http://www.uwplatt.edu/chemep/chem/chemscape/labdocs/catofp/mixpour/mixing/mix/mixb.htm>

### Experiment 5

<http://chemistry.about.com/library/weekly/aa091001a.htm>

[http://en.wikipedia.org/wiki/Qualitative\\_inorganic\\_analysis](http://en.wikipedia.org/wiki/Qualitative_inorganic_analysis)

<http://www.uwplatt.edu/chemep/chem/chemscape/labdocs/catofp/mixpour/mixing/mix/mixb.htm>

### Experiment 6

<http://www.uwplatt.edu/chemep/chem/chemscape/labdocs/catofp/measurea/calorimt/calorimt.htm>

<http://www.vernier.com/>

### Experiment 7

Bishop “An Introduction to Chemistry, Atoms First”, Chapter 5, Chemical Compounds

<http://legacyweb.chemistry.ohio-state.edu/betha/nealChemBal/>

### Experiment 8

<http://legacyweb.chemistry.ohio-state.edu/betha/nealChemBal/>

#### Experiment 9

<http://chemed.chem.purdue.edu/genchem/topicreview/bp/ch4/gaslaws3.html>

#### Experiment 10

[http://preparatorychemistry.com/Bishop\\_Tutorials.htm](http://preparatorychemistry.com/Bishop_Tutorials.htm)

<http://legacyweb.chemistry.ohio-state.edu/betha/nealChemBal/>

<http://www.dartmouth.edu/~chemlab/techniques/vfiltration.html>

<http://www.uwplatt.edu/chemep/chem/chemscape/labdocs/catofp/mixpour/filter/filter.htm>

<http://www.uwplatt.edu/chemep/chem/chemscape/labdocs/catofp/mixpour/mixing/mix/mixb.htm>

#### Experiment 11

<http://dl.clackamas.edu/ch105-04/equation.htm>

#### Experiment 12

<http://www.dartmouth.edu/~chemlab/techniques/titration.html>

<http://www.uwplatt.edu/chemep/chem/chemscape/labdocs/catofp/measurea/concentr/titrate/titration.htm>

<http://www.uwplatt.edu/chemep/chem/chemscape/labdocs/catofp/mixpour/volflask/volflask.htm>

#### Experiment 13

CRC “Handbook of Chemistry and Physics”

<http://www.vernier.com/>

## **APPENDIX N: SAMPLE FORMS**



Name: \_\_\_\_\_ Date: \_\_\_\_\_ Lab Section \_\_\_\_\_

## Experiment 1: Basic Measurement Techniques

### REPORT SHEET

Objectives of Experiment:

Data Part 1: Length Measurements (all values must include units)

A. Length of notebook in cm: \_\_\_\_\_

B. Width of notebook in cm: \_\_\_\_\_

Calculations Part 1 (show your work for full credit):

1. Length of notebook in in.:

2. Width of notebook in in.:

3. Length in mm:

4. Width in mm:

Data Part 2: Temperature Measurements (all values must include units)

A. Room temperature in °C: \_\_\_\_\_

B. Ice Bath temperature in °C: \_\_\_\_\_

Calculations Part 2 (show your work for full credit):

1. Room temperature in K:

2. Room temperature in °F:

3. Ice bath temperature in K:

4. Ice bath temperature in °F:

**Data Part 3: Volume Measurements (all values must include units)**

A. Sketch of graduated cylinder with meniscus in box ->:

(indicate with an arrow where the volume should be read)

B. Volume of H<sub>2</sub>O from small test tube: \_\_\_\_\_

C. Volume of H<sub>2</sub>O from large test tube: \_\_\_\_\_

D. Volume of water from 50-mL beaker: \_\_\_\_\_

E. Volume of water from 50-mL Erlenmeyer Flask: \_\_\_\_\_

**Data Part 4: Mass Measurements (all values must include units)**

A. Type of balance used: \_\_\_\_\_

B. Mass of large test tube: \_\_\_\_\_

C. Mass of 50-mL beaker: \_\_\_\_\_

D. Mass of 150-mL beaker: \_\_\_\_\_

**Data Part 5: Density determinations (all values must include units)**

**Data for 2-mL sample of unknown liquid:**

A. Mass of empty 10-mL graduated cylinder: \_\_\_\_\_

B. Approximate volume of unknown liquid added: \_\_\_\_\_

C. Actual volume of unknown liquid added (remember sig figs): \_\_\_\_\_

D. Mass of cylinder plus unknown liquid: \_\_\_\_\_

E. Mass of liquid alone: \_\_\_\_\_

- Calculate density of 2-mL unknown liquid sample using data from C and E above: (include units)

Accepted (textbook) density value for unknown liquid (obtain from instructor): \_\_\_\_\_

- Calculate % error between accepted density and experimental (your) value:

$$\% \text{ error} = \frac{|\text{Absolute error}|}{\text{Accepted value}} \times 100$$

% error for 2-mL sample: \_\_\_\_\_

Data for 10-mL sample of unknown liquid:

- A. Mass of empty 10-mL graduated cylinder: \_\_\_\_\_
- B. Approximate volume of unknown liquid added: \_\_\_\_\_
- C. Actual volume of unknown liquid added (remember sig figs): \_\_\_\_\_
- D. Mass of cylinder plus unknown liquid: \_\_\_\_\_
- E. Mass of liquid alone: \_\_\_\_\_

- Calculate density of 10-mL unknown liquid sample using data from C and E above: (include units)

Accepted (textbook) density value for unknown liquid (obtain from instructor): \_\_\_\_\_

- Calculate % error between accepted density and experimental (your) value:

$$\% \text{ error} = \frac{|\text{Absolute error}|}{\text{Accepted value}} \times 100$$

% error for 10-mL sample: \_\_\_\_\_

Data for the unknown solid

- A. Unknown number: \_\_\_\_\_
- B. Mass of irregular solid: \_\_\_\_\_
- C. Volume of water in graduated cylinder: \_\_\_\_\_
- D. Volume of water plus object: \_\_\_\_\_
- E. Volume of object alone: \_\_\_\_\_

- Calculate the density of the irregular solid using data from B and E above:



**6. Which of your two values actually was more accurate?**



## Report Sheet Experiment 2

FORMULA OF MOLECULE	LEWIS STRUCTURE	3-D SHAPE	GEOMETRY	MOLECULAR POLARITY
example: CH <sub>4</sub>	$  \begin{array}{c}  \text{H} \\    \\  \text{H}-\text{C}-\text{H} \\    \\  \text{H}  \end{array}  $		tetrahedral	nonpolar
H <sub>2</sub> O				
H <sub>3</sub> O <sup>+</sup>				
C <sub>2</sub> H <sub>6</sub>				
C <sub>2</sub> H <sub>4</sub>				
C <sub>2</sub> H <sub>2</sub>				
CO <sub>2</sub>				
H <sub>2</sub> O <sub>2</sub>				
CH <sub>3</sub> Cl				
CH <sub>2</sub> Cl <sub>2</sub>				
CHCl <sub>3</sub>				
CCl <sub>4</sub>				

FORMULA OF MOLECULE	LEWIS STRUCTURE	3-D SHAPE	GEOMETRY	MOLECULAR POLARITY
H <sub>2</sub> S				
NH <sub>3</sub>				
NH <sub>4</sub> <sup>+</sup>				
Br <sub>2</sub>				
N <sub>2</sub>				
CH <sub>3</sub> OH				
CH <sub>2</sub> O				
HCl				
H <sub>2</sub> CO <sub>3</sub>				
HCO <sub>3</sub> <sup>-</sup>				
CO <sub>3</sub> <sup>2-</sup>				
SO <sub>2</sub>				

FORMULA OF MOLECULE	LEWIS STRUCTURE	3-D SHAPE	GEOMETRY	MOLECULAR POLARITY
SO <sub>3</sub>				
H <sub>2</sub> SO <sub>4</sub>				
SO <sub>4</sub> <sup>2-</sup>				

### Questions

- Explain why the model for each of the following atoms has a specific number of bonds:
  - Hydrogen: 1 bond
  - Oxygen: 2 bonds
  - Nitrogen: 3 bonds
  - Carbon: 4 bonds
- Discuss the shapes of SO<sub>2</sub> and SO<sub>3</sub>. Why is one polar and the other is not.
- Explain why an ion such as NH<sub>4</sub><sup>+</sup>, CO<sub>3</sub><sup>2-</sup>, and SO<sub>4</sub><sup>2-</sup> can be nonpolar, but charged?
- Explain how molecules of H<sub>3</sub>O<sup>+</sup> and NH<sub>4</sub><sup>+</sup> form from uncharged molecules of H<sub>2</sub>O and NH<sub>3</sub>.



Name: \_\_\_\_\_ Date: \_\_\_\_\_ Lab Section \_\_\_\_\_

## Experiment 3: The Gas Burner

### REPORT SHEET

Objectives of Experiment:

Observations and Data 1 & 2: Sketch your burner in Box 1 below. Label the base, the barrel, the collar, the air ports and the gas adjustment screw.

Box 1	Box 2

Observations and Data 3 & 4: Sketch the non-luminous flame in Box 2 above. Indicate (by drawing a line to) the location of the top of the flame, the tip of the inner blue cone, and the base of the flame. Also, note the comparative temperatures of these locations according to your observations.

Time required to turn wire gauze red (include units):

_____	_____	_____
Tip of flame	tip of inner blue cone	flame next to top of barrel

5. What appears on the glass when the flame is dashed across the beaker of cold water? (Hint: check products of combustion in introduction)

6a. What occurs when the crucible is held in the luminous (yellow) flame?

6b. What occurs when the crucible is held in the non-luminous (blue) flame?

Questions: Refer to the Introduction of the experiment, the Procedures, your instructor's directions, and your observations to answer these questions.

1. Why should you not hold your lighted match over the top of the burner while you turn on the gas?
  
2. A) Suppose when you lighted your burner, it had a luminous flame. What should you do to get a properly adjusted flame?  
  
B) Suppose when you lighted your burner, it had a loud "roaring" sound. What should you do to correct it?  
  
C) Suppose when you lighted your burner, the flame disappeared down into the barrel. How do you obtain a properly adjusted flame?
  
3. A) What product of combustion is shown to be present by the results of dashing the flame across the cold beaker surface?  
  
B) What product of combustion is shown to be present by the results of holding the crucible in the luminous flame?  
  
C) Why did the deposit in (B) not appear when you held the crucible in the non-luminous (blue) flame?

Name: \_\_\_\_\_ Date: \_\_\_\_\_ Lab Section \_\_\_\_\_

**Experiment 4: Determining Chemical Properties to Separate a Mixture**

**Objectives of Experiment:**

**Part 1: Physical and Chemical Properties**

Part 1 Data:	Test Tube #1		Test Tube #2			Test Tube #3	Test Tube #4
Substance Name, Formula	Color	Effect of Heat	Cold H <sub>2</sub> O	Hot H <sub>2</sub> O	Litmus Test	dilute HCl	dilute NaOH
Magnesium, Mg							
Copper, Cu							
Zinc, Zn							
Magnesium Oxide, MgO							
Copper(II)Carbonate, CuCO <sub>3</sub>							
Copper(II)Nitrate, Cu(NO <sub>3</sub> ) <sub>2</sub>							
Sodium Chloride, NaCl							

**Part 2: Separation of a Mixture of Copper(II)Carbonate and Sodium Chloride**

**Data**

- A. ID # of Sample: \_\_\_\_\_
- B. Mass of empty 250-mL beaker: \_\_\_\_\_
- C. Mass of 250-mL beaker plus mixture sample: \_\_\_\_\_
- D. Exact mass of mixture sample (C minus B): \_\_\_\_\_
- E. Identity of soluble mixture component: \_\_\_\_\_
- F. Identity of Insoluble mixture component: \_\_\_\_\_
- G. Mass of dry filter paper: \_\_\_\_\_
- H. Mass of empty 150-mL beaker: \_\_\_\_\_
- I. Mass of filter paper plus insoluble mixture component: \_\_\_\_\_
- J. Mass of 150-mL beaker plus soluble mixture component: \_\_\_\_\_

**Part 2: Calculations** (must show your work for full credit)

1. Mass of soluble component of mixture (J minus H):

2. **Percentage of soluble component in mixture** (answer from 1 divided by D x 100):
  
3. **Mass of insoluble component of mixture** (I minus G):
  
4. **Percentage of insoluble component in mixture** (answer from 3 divided by D x 100):
  
5. **Sum of percentages** (add percentages from 2 and 4):

**\*\*If your answer to # 5 is more than 100%, you need to explain why and how this happens.**

**Answer Questions 1 and 2 on a separate sheet of paper.**





Name: \_\_\_\_\_ Date: \_\_\_\_\_ Lab Section \_\_\_\_\_

**Experiment 6: Comparing chemical and physical changes  
Using a temperature probe  
REPORT SHEET**

**Objectives of Experiment:**

**Part 1: Heat of Combustion**

**Data (all values must include units)**

- A. Initial Mass of 400 mL beaker plus candle: \_\_\_\_\_
- B. Mass of empty tin can: \_\_\_\_\_
- C. Mass of tin can plus water: \_\_\_\_\_
- D. Initial temperature of water: \_\_\_\_\_
- E. Final temperature of water: \_\_\_\_\_
- F. Final mass of 400 mL beaker plus candle: \_\_\_\_\_

**Calculations (show your work for full credit)**

- 1. Mass of water heated (C minus B):
- 2. Temperature change of water (E minus D):
- 3. Heat absorbed by water (remember the specific heat of water is  $4184 \text{ J/g}\cdot^{\circ}\text{C}$ )
- 4. Mass of candle burned (A minus F):
- 5. Heat of combustion of the candle in J/g (divide answer to 3 by answer to 4):

**Part 2: Heat of Fusion**

**Data (all values must include units)**

- A. Mass of empty test tube: \_\_\_\_\_
- B. Mass of test tube plus candle wax: \_\_\_\_\_
- C. Mass of empty Styrofoam cup: \_\_\_\_\_
- D. Mass of Styrofoam cup plus water: \_\_\_\_\_

E. Initial temperature of water: \_\_\_\_\_

F. Final temperature of water: \_\_\_\_\_

**Calculations (show your work for full credit)**

1. Mass of water heated (D minus C):
2. Temperature change of water (F minus E):
3. Heat absorbed by water (remember the specific heat of water is  $4.184 \text{ J/g}\cdot^{\circ}\text{C}$ ):
4. Mass of candle solidified (B minus A):
5. Heat of fusion of candle wax in  $\text{J/g}$  (divide answer to 3 by answer to 4):

**Questions** (answer on a separate sheet of paper, attach, and turn in with this report sheet)

1. For Part 1, list ways where heat could be lost and consequently not raise the temperature of the water.
2. Calculate the ratio of the heat of combustion to the heat of fusion. (Hint: divide the heat of combustion by the heat of fusion. Your answer should not have units.)
3. Based on your answer to question 2, what conclusion can you make about the amount of energy it takes for a chemical change compared to a physical change?

Experiment 7 Report Sheet  
Empirical Formula: A sulfide of Copper

## Objectives:

## Observations and Data

- A. Mass of Crucible (without cover): \_\_\_\_\_
- B. Mass of Crucible plus copper: \_\_\_\_\_
- C. Approximate mass of sulfur used: \_\_\_\_\_
- D. Appearance of Copper beads: \_\_\_\_\_
- E. Appearance of sulfur powder: \_\_\_\_\_
- F. Mass of Crucible (without cover) plus mass of copper sulfide after heating: \_\_\_\_\_
- G. Appearance of Copper Sulfide formed: \_\_\_\_\_

## Calculations (show work for full credit)

1. Mass of copper used (B minus A):
  
  
  
  
  
  
  
  
  
  
2. Mass of sulfur incorporated (F minus B):
  
  
  
  
  
  
  
  
  
  
3. Moles of copper (answer from 1 / MW of copper):
  
  
  
  
  
  
  
  
  
  
4. Moles of sulfur (answer from 2 / MW of sulfur):
  
  
  
  
  
  
  
  
  
  
5. Simplest ratio of moles of copper to sulfur in the copper sulfide (divide the smaller number of moles into the larger):
  
  
  
  
  
  
  
  
  
  
6. Empirical formula of the copper sulfide:

**Questions 1 - 5: answer on a separate sheet of paper, attach and turn in.**

Experiment 8 Report Sheet  
Properties of Gases

## Objectives:

## Observations and Data:

Part 1 – Hydrogen, H<sub>2</sub>

- A. Reagents used for preparation: \_\_\_\_\_
- B. Color of gas: \_\_\_\_\_
- C. Effect upon lighted splint: \_\_\_\_\_

Part 2 – Carbon Dioxide, CO<sub>2</sub>

- A. Reagents used for preparation: \_\_\_\_\_
- B. Color of gas: \_\_\_\_\_
- C. Effect upon lighted splint: \_\_\_\_\_
- D. Addition of limewater to carbon dioxide: \_\_\_\_\_  
\_\_\_\_\_
- E. Result of “pouring” carbon dioxide into limewater: \_\_\_\_\_  
\_\_\_\_\_
- F. Result of blowing into limewater: \_\_\_\_\_

Part 3 – Oxygen, O<sub>2</sub>

- A. Reagents used for preparation: \_\_\_\_\_
- B. Color of gas: \_\_\_\_\_
- C. Effect upon glowing splint: \_\_\_\_\_

Part 4 – Nitrogen Dioxide, NO<sub>2</sub>

- A. Reagents used for preparation: \_\_\_\_\_
- B. Color of gas: \_\_\_\_\_

## Questions

1. What observation did you make that makes you believe that gases are less dense than water?
2. Is carbon dioxide less dense than air? What evidence do you have for your choice?
3. If you fill a balloon with Helium, He, it rises because He is lighter or less dense than air. What do you think would happen to a balloon filled with CO<sub>2</sub>? Hint: consider your answer to question 2.
4. Which of the gases you observed today would be the most helpful in putting out a fire? What evidence do you have to support this choice?
5. Based on what you learned today, tell how you could distinguish between the following pairs of gases:
  - a. CO<sub>2</sub> and NO<sub>2</sub>
  - b. H<sub>2</sub> and CO<sub>2</sub>
  - c. O<sub>2</sub> and H<sub>2</sub>
6. Now suppose you were given a sample of one of the four gases you observed today.
  - a. If the sample gas is colorless, which gas does this eliminate?
  - b. If a portion of the same sample gas is bubbled through limewater and nothing happens, what gas is eliminated?
  - c. If a glowing splint reignites when it is placed in another portion of the same sample gas, what is the identity of the gas?

## Experiment 9: Determining the molar volume of Hydrogen

## Objectives:

## Data:

- A. Mass of Mg per centimeter of ribbon: \_\_\_\_\_ g/cm
- B. Length of Mg strip: \_\_\_\_\_
- C. Water temperature: \_\_\_\_\_
- D. Temperature of Laboratory: \_\_\_\_\_
- E. Barometric pressure: \_\_\_\_\_
- F. Volume of "wet" H<sub>2</sub> gas collected: \_\_\_\_\_

## Calculations (show your work for full credit):

1. Mass of magnesium, Mg (hint: use B and A above):
  
  
  
  
  
  
  
  
  
  
2. Moles of magnesium, Mg (use answer from 1 and MW of Mg to solve):
  
  
  
  
  
  
  
  
  
  
3. Moles of hydrogen gas, H<sub>2</sub> (use the mole ratio from the balance eqn for the reaction to solve):
  
  
  
  
  
  
  
  
  
  
4. Pressure of "dry" H<sub>2</sub> (use the table from your lab manual to find the partial pressure of the gas due to water, H<sub>2</sub>O, and subtract it from the barometric pressure to find your answer):
  
  
  
  
  
  
  
  
  
  
5. Volume of "dry" H<sub>2</sub> corrected to STP (use the combined gas law as shown in your manual. P<sub>1</sub>, V<sub>1</sub> and T<sub>1</sub> are all data you have collected above).

6. Determine the molar volume at STP (divide your answer from 5 by the # of moles of H<sub>2</sub>):

7. Calculate the % error between your experimental value from 6 above and the accepted value of 22.4 L/mol at STP:

$$\% \text{ error} = \frac{|\text{accepted value} - \text{experimental value}|}{\text{Accepted value}} \times 100$$

**\*\*\* Answer questions 1 – 4 on a separate sheet and turn in with your report.\*\*\***

CHEM22 Section \_\_\_\_\_

Name \_\_\_\_\_

Experiment 10 Report Sheet

Date \_\_\_\_\_

Stoichiometry: Producing an insoluble compound

Objectives:

Pre-experiment Calculations:

- A. Compound to be prepared: \_\_\_\_\_
- B. Desired mass of salt to be prepared: \_\_\_\_\_
- C. Reaction to be performed (balanced equation): \_\_\_\_\_
- D. Moles of product desired (calculate MW of product and calculate how many moles in 3.0g):
  
- E. Mass of each reactant to be used (calculate MW of each reactant and calculate how many grams of each is needed from the molar ratio of product):

**Data and Calculations:**

- | <b>F. Mass of reactants:</b>   | <b>Reactant 1</b> | <b>Reactant 2</b> |
|--------------------------------|-------------------|-------------------|
| a. Mass of beaker:             | _____             | _____             |
| b. Mass beaker plus reactants: | _____             | _____             |
| c. Mass reactant used:         | _____             | _____             |
- G. Mass of filter paper (dry): \_\_\_\_\_
- H. Mass of filter paper plus product: \_\_\_\_\_
- I. Mass of Product isolated(H minus G): \_\_\_\_\_
- J. Percentage yield ( I divided by B X 100):

**\*\*\*Warning: if percent yield is greater than 100%, you must explain factors that could have affected your experiment.**

**Answer Questions 1 – 7 on a separate sheet of paper and turn in with your report.**

## Studying Ionic Reactions

Objectives:

Observations and Data: Provide both possible combinations, check appendix H for solubility.

Set I	K <sup>+</sup> / Cl <sup>-</sup>	Na <sup>+</sup> / NO <sub>3</sub> <sup>-</sup>	K <sup>+</sup> /CrO <sub>4</sub> <sup>2-</sup>	Na <sup>+</sup> /CrO <sub>4</sub> <sup>2-</sup>	Ba <sup>2+</sup> / Cl <sup>-</sup>
Ba <sup>2+</sup> / NO <sub>3</sub> <sup>-</sup>					
Ba <sup>2+</sup> / Cl <sup>-</sup>					
Na <sup>+</sup> / CrO <sub>4</sub> <sup>2-</sup>					
K <sup>+</sup> / CrO <sub>4</sub> <sup>2-</sup>					
Na <sup>+</sup> / NO <sub>3</sub> <sup>-</sup>					

Net ionic equations for set I:

Set II	Al <sup>3+</sup> /Cl <sup>-</sup>	Ba <sup>2+</sup> / NO <sub>3</sub> <sup>-</sup>	Ba <sup>2+</sup> / Cl <sup>-</sup>	Sr <sup>2+</sup> / NO <sub>3</sub> <sup>-</sup>	Al <sup>3+</sup> /SO <sub>4</sub> <sup>2-</sup>
Na <sup>+</sup> / SO <sub>4</sub> <sup>2-</sup>					
Al <sup>3+</sup> / SO <sub>4</sub> <sup>2-</sup>					
Sr <sup>2+</sup> / NO <sub>3</sub> <sup>-</sup>					
Ba <sup>2+</sup> / Cl <sup>-</sup>					
Ba <sup>2+</sup> / NO <sub>3</sub> <sup>-</sup>					

Net ionic equations for set II:

Set III	Na <sup>+</sup> /NO <sub>3</sub> <sup>-</sup>	K <sup>+</sup> / OH <sup>-</sup>	Na <sup>+</sup> / OH <sup>-</sup>	Co <sup>2+</sup> / Cl <sup>-</sup>	Co <sup>2+</sup> / NO <sub>3</sub> <sup>-</sup>
Fe <sup>3+</sup> /Cl <sup>-</sup>					
Co <sup>2+</sup> / NO <sub>3</sub> <sup>-</sup>					
Co <sup>2+</sup> / Cl <sup>-</sup>					
Na <sup>+</sup> / OH <sup>-</sup>					
K <sup>+</sup> / OH <sup>-</sup>					

Net ionic equations set III:

Set IV	Mg <sup>2+</sup> /SO <sub>4</sub> <sup>2-</sup>	Ba <sup>2+</sup> / OH-	Na+ / OH-	K+ / SO <sub>4</sub> <sup>2-</sup>	Mg <sup>2+</sup> / Cl-
Na+ / Cl-					
Mg <sup>2+</sup> / Cl-					
K+ / SO <sub>4</sub> <sup>2-</sup>					
Na+ / OH-					
Ba <sup>2+</sup> / OH-					

Net ionic equations set IV:

Set V	Ag+ / NO <sub>3</sub> <sup>-</sup>	K+ / CrO <sub>4</sub> <sup>2-</sup>	Al <sup>3+</sup> / SO <sub>4</sub> <sup>2-</sup>	Na+ / CrO <sub>4</sub> <sup>2-</sup>	Sr <sup>2+</sup> / NO <sub>3</sub> <sup>-</sup>
Ba <sup>2+</sup> / Cl-					
Sr <sup>2+</sup> / NO <sub>3</sub> <sup>-</sup>					
Na+ / CrO <sub>4</sub> <sup>2-</sup>					
Al <sup>3+</sup> / SO <sub>4</sub> <sup>2-</sup>					
K+ / CrO <sub>4</sub> <sup>2-</sup>					

Net ionic equations set V:

\*\*\*\*Answer questions 1 and 2 on a separate sheet of paper and please attach, along with your scratch work for the ion crosses. Full credit will not be given without showing your work.\*\*\*\*

Experiment 12 Report Sheet  
Titrations of Acids and Bases

## Objectives:

## Part 1 Data, Preparing a Standardized solution:

A. Volume of volumetric flask: \_\_\_\_\_

B. Mass of Oxalic acid dihydrate: \_\_\_\_\_

## Part 1 Calculations (Show your work for full credit):

1. Moles of oxalic acid dihydrate (use mass from above, and MW of  $\text{H}_2\text{C}_2\text{O}_4 \cdot 2 \text{H}_2\text{O}$ ):

2. Molarity of oxalic acid solution (use moles you found in 1, and the volume from A):

## Part 2 Data, Preparing a solution by Dilution:

A. Volume of 6 M NaOH used: \_\_\_\_\_

B. Final volume of diluted solution: \_\_\_\_\_

Part 2 Calculation: Using the information from A and B above, calculate the approximate concentration of your diluted NaOH solution (Hint:  $M_1V_1 = M_2V_2$ , show your work for full credit):

## Part 3 Data, Standardizing a Sodium Hydroxide solution by Titration:

Oxalic Acid	Trial 1 (mL)	Trial 2 (mL)	Trial 3 (mL)
Initial Volume:			
Final Volume:			
Volume Used:			

Sodium Hydroxide	Trial 1 (mL)	Trial 2 (mL)	Trial 3 (mL)
Initial Volume:			
Final Volume:			
Volume Used:			

Summary of Calculations for Part 3: (Show your work and attach to this sheet):

	Trial 1	Trial 2	Trial 3
Mol of oxalic acid:			
Moles of NaOH:			
Volume NaOH (L):			
Molarity of NaOH:			

1. Average Molarity of NaOH solution (add all 3 trials, divide by 3): \_\_\_\_\_

2. Percentage difference between high and low results: \_\_\_\_\_

$$\% \text{ Diff} = \{(\text{High value} - \text{Low value}) / \text{Average}\} \times 100$$

Part 4 Data, Standardizing an Oxalic Acid Solution by Titration:

Oxalic Acid	Trial 1 (mL)	Trial 2 (mL)	Trial 3 (mL)
Initial Volume:			
Final Volume:			
Volume Used:			

Sodium Hydroxide	Trial 1 (mL)	Trial 2 (mL)	Trial 3 (mL)
Initial Volume:			
Final Volume:			
Volume Used:			

Summary of Calculations for Part 4: (Show all your work and attach to this sheet):

	Trial 1	Trial 2	Trial 3
Moles NaOH			
Moles Oxalic Acid:			
Vol Oxalic Acid (L):			
Molarity $\text{H}_2\text{C}_2\text{O}_4$ :			

1. Average Molarity of Oxalic Acid solution (add all 3 trials, divide by 3): \_\_\_\_\_

2. Percentage difference between high and low results: \_\_\_\_\_

$$\% \text{ Diff} = \{(\text{High value} - \text{Low value}) / \text{Average}\} \times 100$$

\*\*\*\* Answer Questions 1-3 on a separate sheet of paper and attach to this sheet.\*\*\*\*

Name: \_\_\_\_\_ Date: \_\_\_\_\_ Lab Section \_\_\_\_\_

## Experiment 13: Observing Physical Properties – Boiling Point, Solubility and Conductivity

### REPORT SHEET

**Objectives of Experiment:**

**Observations and Data Part 1: Boiling Point Determination (all values must contain units)**

A. Observed boiling point of 2-propanol: \_\_\_\_\_

B. Accepted value of 2-propanol: \_\_\_\_\_

C. Boiling points, molecular masses and chemical formula of hydrocarbons: (fill in table below)

Hydrocarbon	Chemical formula	molecular mass (g/mol)	Boiling Point (°C)
Methane			
Ethane			
Propane			
Butane			
Pentane			
Hexane			
Heptane			
Octane			
Nonane			
Decane			

**Question 1:** What trends do you observe in boiling point and molecular mass for the hydrocarbons you checked in the “Handbook of Chemistry and Physics”? (If you didn’t use CRC, please list website(s).)

**Observations and Data Part 2: Miscibility (all values must contain units).**

Test Tube #	Liquid 1 (name &vol)	Liquid 2 (name &vol)	Miscible or Immiscible?

**Observations and Data Part 3: Solubility of Solids in Liquids.** Label each solid as soluble or insoluble in the appropriate solvent. Describe what you see.

Liquid sample (3-mL)	Iodine I <sub>2</sub>	sodium chloride NaCl	Sucrose C <sub>12</sub> H <sub>22</sub> O <sub>11</sub>	Potassium Nitrate KNO <sub>3</sub>	Paraffin wax
1. DI water					
2. methanol					
3. hexane					
4. ethyl acetate					

**Observations and Data Part 4: Electrolytes.** For each electrolyte you test, list the name of the material, and tell if the light bulb glows brightly, glow dimly, or not at all.

Electrolyte Name	Light Bulb?	Electrolyte Name	Light Bulb?
1.		6.	
2.		7.	
3.		8.	
4.		9.	
5.		10.	

**Questions: Answer these questions on a separate sheet, attach and turn in with your report.**

- What generalizations can you make about the nature of solvents that dissolved ionic solids the best?
- What generalizations can you make about the solids that are electrolytes?
- The four liquids that you tested may be ranked in order of decreasing polarity as follows:  
(most polar) water > methanol > ethyl acetate > hexane (least polar)  
Based on the data you gathered, what generalizations can you make about the relationship between polarity and miscibility?
- Based on your answer to question 4, which of the four liquids would probably best dissolve a polar solid such as NaCl?
- Which liquid would best dissolve a nonpolar solid such as paraffin?



## *Commitment to Laboratory Safety*

Name \_\_\_\_\_ Student ID \_\_\_\_\_

I have carefully read the laboratory preparation and safety instructions. I understand the importance of good safety practices for my own welfare and of all people in the laboratory and I will comply with the safety rules by carrying out the following:

- \_\_\_\_\_ Read laboratory instructions ahead of lab time.
- \_\_\_\_\_ Know the locations of eyewash fountains, fire extinguishers, safety showers, and exits.
- \_\_\_\_\_ Wear safety goggles or safety glasses in the laboratory at all times.
- \_\_\_\_\_ Use the proper equipment for laboratory procedures.
- \_\_\_\_\_ Never perform any unauthorized experiments or work alone in the laboratory.
- \_\_\_\_\_ Remember what is hot if I have used the Bunsen burner or hot plate.
- \_\_\_\_\_ Clean up chemical spills and broken glass immediately.
- \_\_\_\_\_ Immediately inform the instructor of a chemical spill or accident in the laboratory.
- \_\_\_\_\_ Never eat or drink in the laboratory.
- \_\_\_\_\_ Wear sensible clothing and closed shoes, and tie back long hair in the laboratory.
- \_\_\_\_\_ Read the labels on reagent bottles carefully, remove only small amounts of reagent with the proper tools, and never return unused chemicals to the bottle.
- \_\_\_\_\_ Dispose of broken glass and waste chemicals in the appropriate waste container.
- \_\_\_\_\_ Wash my hands and leave a clean work area when I leave the laboratory.
- \_\_\_\_\_ Never leave an experiment when substances are heating or reacting.

Date: \_\_\_\_\_

Signature: \_\_\_\_\_

Laboratory Class and Section \_\_\_\_\_

