

# ***Chemistry 1009 Lab Manual***

*University of Louisiana  
at Monroe  
Department of Chemistry*

*2010*



Version 2.0

## Contents

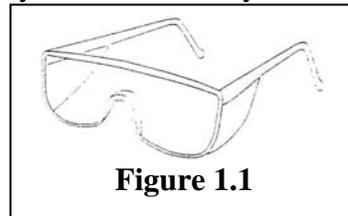
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### To the Students:

Each experiment write-up has blanks within the procedures in which you should enter requested information (such as calculated results or other responses). Also provided at the end of each experiment is a Report Form, on which the same entry blanks are duplicated. At the end of a laboratory period, your instructor may simply inspect the completed blanks in the body of the experiment to verify that you have completed the work. Alternatively, s/he may have you copy the results neatly onto the Report Form to be handed in at the end of the period.

## Lab Session 1: Laboratory Safety Rules and Check In

These rules are designed to ensure that all work done in the laboratory will be safe for you and your fellow students:



- Wear safety goggles at all times when you are in the laboratory! Your instructor will specify what type of goggles/glasses is required.
- Bare skin must be minimized while in the laboratory by wearing clothing that covers one's feet, legs and body completely. Hence, closed shoes and full-length pants are required, since broken glass and spilled chemicals are all too common on the floors of chemistry laboratories. Sandals, flip-flops, skirts, shorts, three-quarter length pants, bare midribs, bare backs and bare shoulders are not allowed. Long-sleeve shirts are recommended but not required. Long hair should be tied back. Hats are not allowed.
- No horseplay, joking or playing is permitted in the lab. Failure to obey this rule is cause for immediate expulsion from the lab.
- No eating or drinking is permitted in the lab or in the prelab classroom.
- No visitors are allowed in the lab. Your friends should visit with you before or after, but not during the lab session.
- Read all labels carefully. Never use materials from unmarked bottles. Most bottles will have short paragraphs on the hazards of a chemical. Most will have a safety sticker showing whether or not the chemical is poisonous or corrosive, and indicating hazards and precautions. See Figure 1.2 for a sample.
- Report all accidents to your laboratory instructor.
- Report all spills or breakage to your laboratory instructor. Clean spills promptly with materials provided by your instructor.
- Inquire about the location of eye-wash and shower stations in your lab.
- Locate spill control materials in your lab.
- Locate all fire extinguishers in your lab.



- Do not throw chemicals down the drain without first consulting your lab instructor. Never put solid material in the sink or down the drain. Solids are to be disposed of in the trash containers.
- Read the safety chart in the lab. This chart contains useful information and recommends safe laboratory procedures.
- Do not pipet liquids by mouth. Use a bulb to siphon liquids into a pipet.
- Use the fume hoods to pour noxious or irritating chemicals, and to run chemical reactions that generate noxious or irritating products.
- Never work in the laboratory unsupervised.
- Read the corresponding chapters of this manual *before* coming to class. This will familiarize you with any potential hazards that may exist or evolve during the exercise. Pay particular attention to any information concerning handling or safety of particular chemicals or solutions.

## **Fire**

Your first responsibility is to get out of harm's way and inform those around you and your lab instructor of the situation. If possible, turn off all gas cocks and remove flammable materials from the area. Usually a wet towel thrown over the fire will extinguish the blaze. Do not throw water on the fire; use the fire extinguisher if possible. In case of fire on your clothing, WALK, DO NOT RUN, to the nearest fire blanket and wrap it around you. Inform the instructor as soon as possible about the incident.

## **Injury**

Report all accidents to the laboratory instructor immediately. In all but the most trivial cases, a visit to the infirmary is required. Chemical splashes may require the use of the shower located in each room. Chemical injury requires immediate attention. Wash the area thoroughly with soap and water and report to the lab instructor. If chemicals get in your eye, flush the eye with water for several minutes and do not touch the eye. When there is a question of physical injury, through chemical or mechanical means, modesty should not be a factor. Showers and eyewash stations are there to be used.

## **Spills**

All spills must be reported to the stockroom. All chemical spills are a potential hazard. For acid spills, use sodium bicarbonate or spill material provided in a hood in each lab. For base spills, use a weak acid and water to neutralize the base and clean up the spill. When cleaning a spill, wear gloves, apron, and goggles. Always ventilate the area when cleaning a spill. Mercury spills, including broken thermometers, are to be treated by stockroom personnel. Do not attempt to clean mercury spills or handle mercury!

## ***Cleanliness***

It is important to keep the laboratory as clean as possible, for safety reasons as well as aesthetic reasons. Each pair of students is responsible for their immediate desk area. Before leaving the laboratory, students should make sure that the area of the laboratory bench near their assigned drawer is clean and dry, that Bunsen burners and other shared equipment are put away in the appropriate space, and that the trough to the sink is free of any solid material. Laboratory instructors will check work areas before approving completion of the experiment.

Pairs of students will be assigned dates for which they are responsible for cleaning the reagent shelves, balances and balance tables in the weighing room, and surfaces under the hoods. This duty will be rotated so that each pair of students will be responsible for general laboratory cleanliness at least once during the semester.

# Desk Assignment Sheet (Chemistry 1009)

Please

Print

Name \_\_\_\_\_ ULM ID \_\_\_\_\_

Name \_\_\_\_\_ ULM ID \_\_\_\_\_

Section # \_\_\_\_\_ Room # \_\_\_\_\_ Desk # \_\_\_\_\_

- |   |  |
|---|--|
| <input type="checkbox"/> 3 Beaker, 100 or 150 ml                    | <input type="checkbox"/> 2 Flask, Erlenmeyer, 125 ml |
| <input type="checkbox"/> 2 Beaker, 250 ml                           | <input type="checkbox"/> 1 Flask, Erlenmeyer, 250 ml |
| <input type="checkbox"/> 1 Beaker, 400 ml                           | <input type="checkbox"/> 1 Flask, Florence, 500 ml   |
| <input type="checkbox"/> 2 Bottles, one 4 oz<br>and one 8 oz, round | <input type="checkbox"/> 1 Funnel, Narrow Stem       |
| <input type="checkbox"/> 1 Wash Bottle                              | <input type="checkbox"/> 2 Watch Glass               |
| <input type="checkbox"/> 1 Clamp, Pinchcock                         | <input type="checkbox"/> 1 Spatula                   |
| <input type="checkbox"/> 1 Clamp, Test-Tube                         | <input type="checkbox"/> 1 Combustion Spoon          |
| <input type="checkbox"/> 1 Crucible and Cover                       | <input type="checkbox"/> 1 Test Tube, 25 × 150       |
| <input type="checkbox"/> 1 Cylinder, Graduated 10 ml                | <input type="checkbox"/> 10 Test Tube, 15 × 125      |
| <input type="checkbox"/> 1 Cylinder, Graduated, 100 ml              | <input type="checkbox"/> 1 Thermometer               |
| <input type="checkbox"/> 1 Evaporating Dish                         | <input type="checkbox"/> 1 Tongs                     |
|   | <input type="checkbox"/> 1 Triangle, Wire            |

*See pictures of items on the following pages*

Check In:

Signature \_\_\_\_\_

Signature \_\_\_\_\_

Checked in by \_\_\_\_\_ Date \_\_\_\_\_

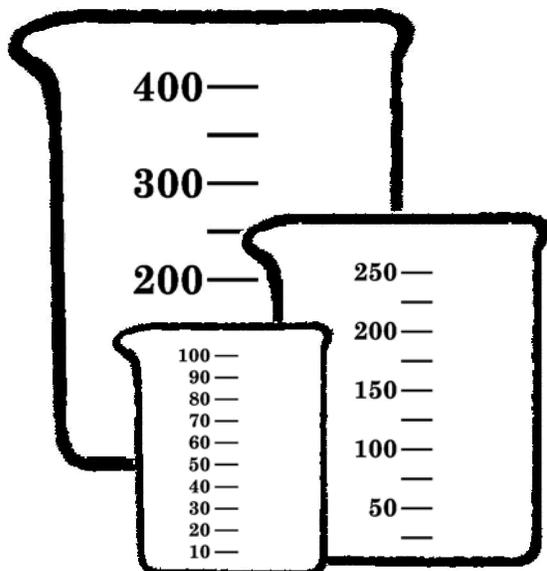
**I MUST WEAR SAFETY GOGGLES DURING EACH LAB SESSION.**

Initials \_\_\_\_\_ Date \_\_\_\_\_

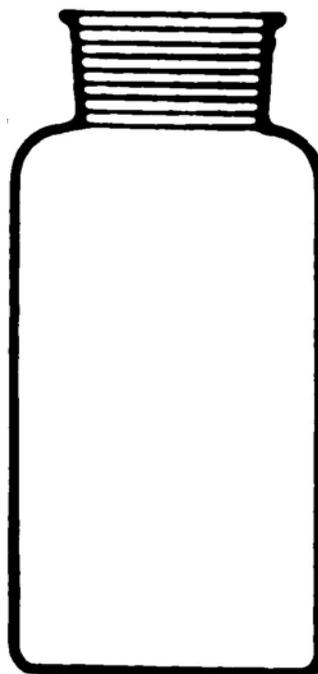
Initials \_\_\_\_\_ Date \_\_\_\_\_

Check Out:

Checked out by \_\_\_\_\_ Date \_\_\_\_\_



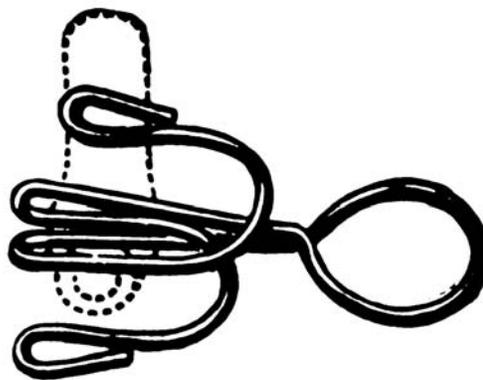
Beakers



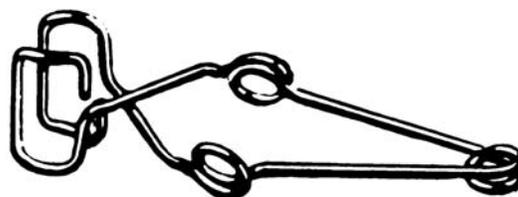
Bottle



Wash Bottle



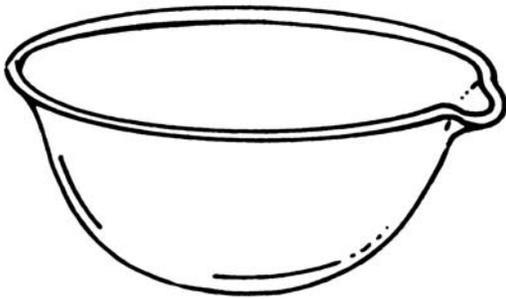
Clamp, Pinchcock



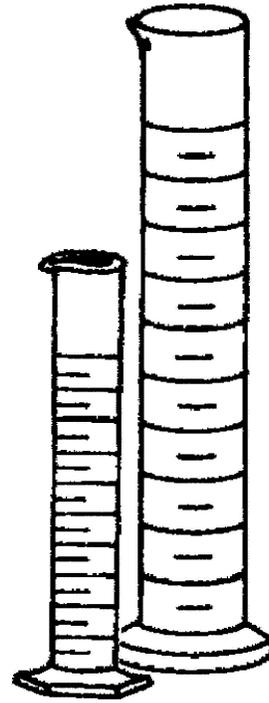
Clamp, Test tube



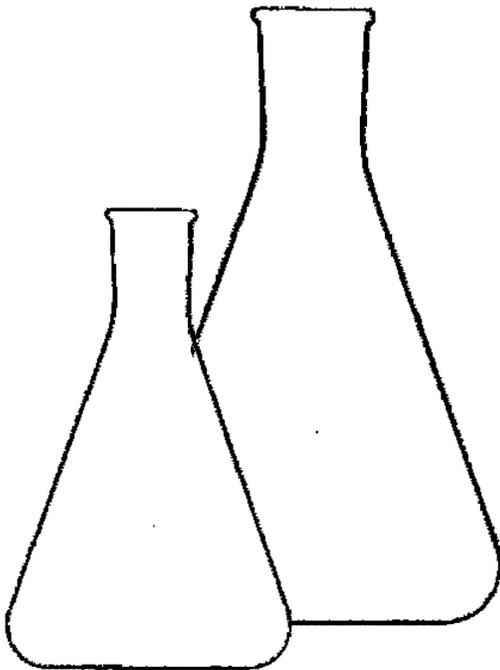
Crucible and Cover



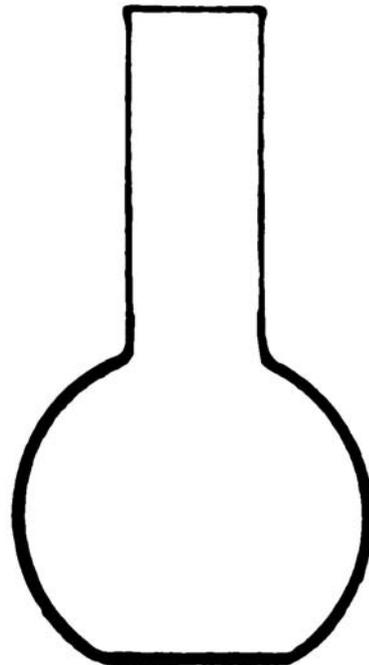
Evaporating Dish



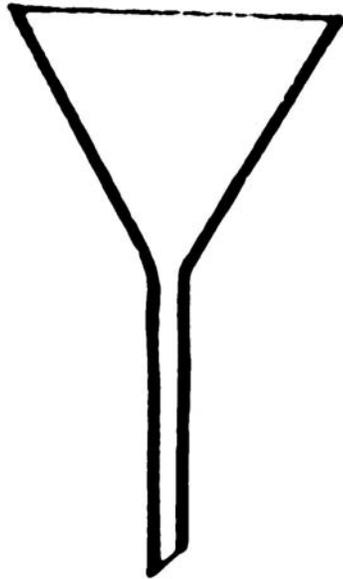
Cylinder, Graduated



Flask, Erlenmeyer



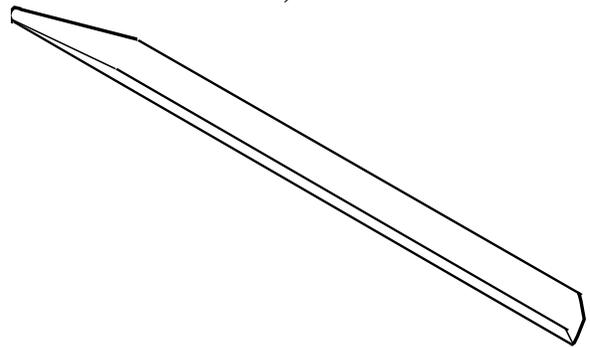
Flask, Florence



Funnel, Narrow Stem



Glass, Watch



Spatula



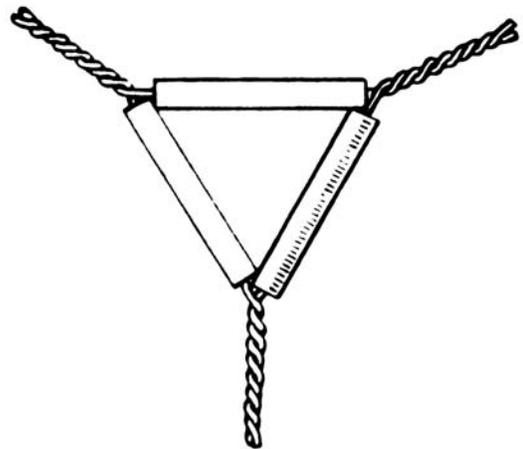
Combustion  
Spoon



Test Tubes

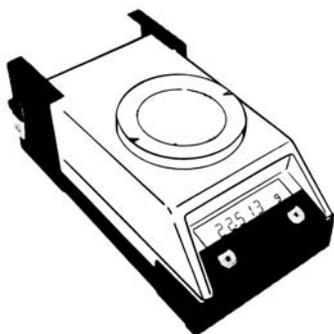


Tongs



Wire Triangle

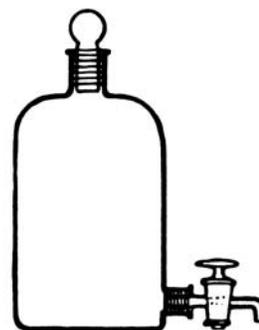
**Commonly Used Equipment (not in the desk drawer)**



**Top Loading Balance**



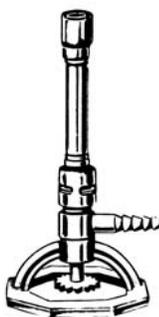
**Test Tube Brush**



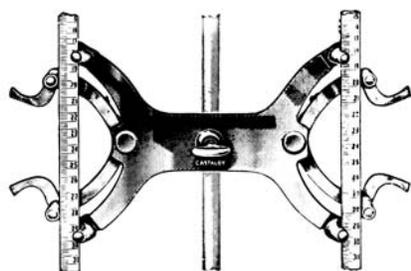
**Dispensing Bottle**



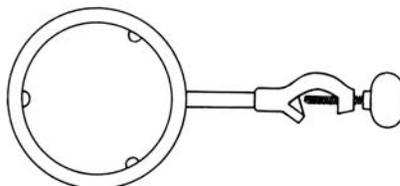
**Squeeze Bulb**



**Bunsen Burner**



**Burette Clamp**



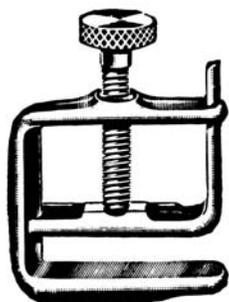
**Ring Clamp**



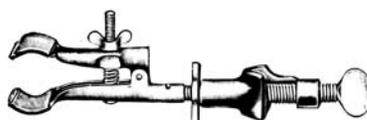
**Ring Stand Clamp**



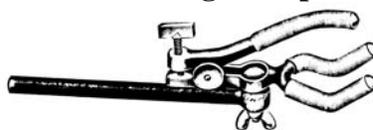
**Burette**



**Screw Clamp**



**Two-Prong Clamp**



**Three Prong Clamp**



**Eye Dropper  
(Suction Pipette)**



**Fire Blanket**



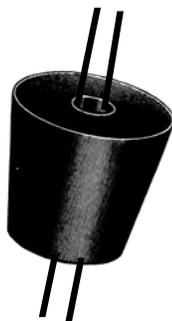
**Fire Extinguisher**



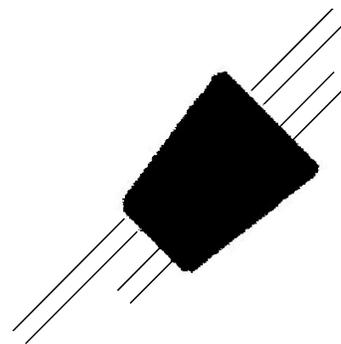
**Plastic Ruler**



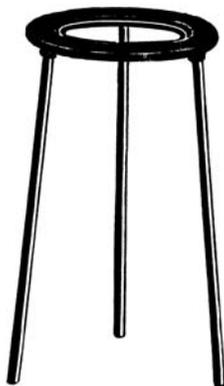
**Striker**



**One-hole Stopper**



**Two-hole Stopper**



**Tripod**



**Ring Stand**



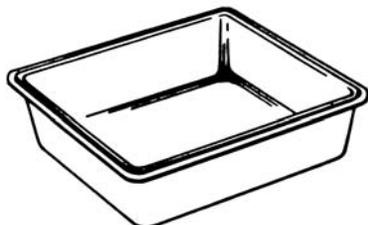
**Glass  
Pipette**



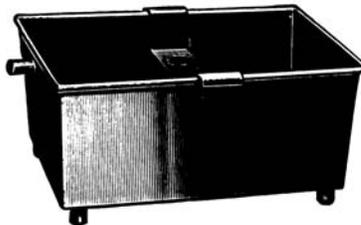
**Thermometer**



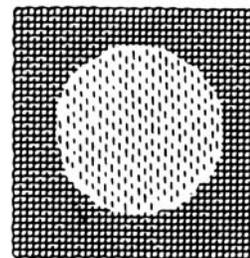
**Thistle  
Tube**



**Metal or Plastic Trough**



**Pneumatic Trough**



**Wire Gauze**

## Lab Session 2, Experiment 1: Introductory Exercises

These experiments are meant to provide your first experience in the laboratory. If you have prior laboratory experience, these exercises constitute good review. Conduct the experiments in whatever order seems convenient. Keep all results to show to your instructor before you leave, or if requested, to turn in on the separate Report Form.

### Weighing

You must learn how to use laboratory balances. As always, the *limit* of readable precision of the scale should be recorded.

When approaching the balance you will need the following:

1. The substance to be weighed;
2. Any container or holder for the sample while on the balance;
3. Any sample handling device such as a spatula; and
4. Your notebook and a pencil or pen to record your measurement.

Balances in this laboratory have a semi-automatic tare (an allowance for mass of the container or holder). On an electronic balance, "tare equals zero" is set by depressing the bar, which is also the on/off switch: up for off, down for on, down again to tare. These balances have an automatic range selector that will change the readout precision automatically to  $\pm 0.01$  g when the gross mass on the pan is over 35 g. The measurement precision for small masses is best if very light containers are used, such as the glassine weighing paper for dry solid samples. The precision for samples less than 30 g gross mass is  $\pm 0.001$  g.

## 1A Experiment: Mass

1. Select five pennies from your resources. Include a new shiny one if possible. Dates are useful for keeping them in order.
2. Without taring, place a sheet of paper on the pan. Read and record its mass in the table on this page. When possible, use the cover to protect the pan from air drafts to obtain higher precision.
3. Add the pennies to the pan one at a time, reading the mass after each addition. Enter each in the table below in the column entitled "Cumulative Mass." Keep the pennies in order. Calculate the mass of each penny by subtracting; record the differences in the column entitled "Mass by Difference."
4. Remove all the coins and weigh each individually, taring to zero. Record the mass of each coin in the table in the column entitled "Direct Weighing."
5. Calculate the mean or average mass ( $\bar{m}$ ) of the pennies:  $\bar{m} = (\sum m)/n$ .
6. Calculate the absolute deviation ( $d = |m - \bar{m}|$ ) from the mean of each mass (direct weighing) and then determine the Average Deviation:  $\bar{d} = (\sum d)/n$ .

Source	Cumulative Mass	Mass by Difference	Direct Weighing	Deviation from Mean
Weighing Paper	g			
1 <sup>st</sup> penny	g	g	g	g
2 <sup>nd</sup> penny	g	g	g	g
3 <sup>rd</sup> penny	g	g	g	g
4 <sup>th</sup> penny	g	g	g	g
5 <sup>th</sup> penny	g	g	g	g
<b>Average:</b>			g	g

Consider two hypotheses:

1. All Lincoln-head pennies are manufactured with equal mass (within  $\pm 0.001$  g), but their various histories result in different masses when measured.
2. New Lincoln-head pennies are lighter than older ones.

Observation is complicated by the various histories of the pennies. Some typical problems in chemistry are illustrated here. Most obviously, the state of corrosion of the pennies represents an uncontrolled experimental variable which can be important. Had we used non-circulated coins, we would have expected better precision. On the other hand, pennies may not be very uniform even when new. Another question arises: How much experimental difference is sufficient and how consistently must it be observed for us to consider two data sets, or groups of data sets as distinctly different? This is an important question for which statistical methods provide answers. Which hypothesis do you choose, and why?

## Density

Density is defined as the ratio of the mass of a sample to its volume. Mass and volume are extensive properties of matter -- properties that depend on the quantities of substances. Such properties are not of themselves useful in characterizing substances.

Intensive properties, on the other hand, are useful in characterizing substances. Intensive properties are often determined by ratioing two extensive properties measured at constant temperature (T) and pressure (P). Density is an example of this kind of intensive property. When measured under known conditions of T and P, density can be used to characterize substances. Of course, two or more substances may have the same density, but for a given substance there is only one density (at constant T and P). If you determine that a colorless liquid has a density of 1.00 g/mL at 4° C and 1 atm, this does not prove the liquid is water. This fact is simply one piece of evidence that the substance may be water.

### 1B Experiment: The Density of Water at Room Temperature

1. Collect a small beaker of deionized water and measure its temperature.  
T = \_\_\_\_\_ °C

2. Place a clean dry 10 mL graduated cylinder on the balance and tare it to zero. Carefully transfer nearly 10 mL (but less) of water into the cylinder, taking care not to splash water up on the sides (read the mass to  $\pm 0.001$  g).

$$\text{"Weight"} = \text{Mass of H}_2\text{O} = \text{_____ g}$$

3. Carefully read the volume occupied by the water at the bottom of the meniscus holding the cylinder at eye level. The volume should be read to  $\pm 0.1$  mL.

$$\text{Volume of H}_2\text{O} = \text{_____ mL}$$

4. Calculate the density of water at the current temperature, noting the number of significant figures.

$$D = \text{Mass of H}_2\text{O} / \text{volume of H}_2\text{O} = \text{_____ g/mL}$$

5. Consult the Table of Water Densities (below) and calculate the percent error in your determination according to the following formula.

$$\% \text{Error} = [ |D_{\text{TAB}} - D_{\text{EXP}}| \div D_{\text{TAB}} ] \times 100 = \text{_____ \%Error}$$

**Table of Water Density at Various Temperatures**

T, °C	D, g/mL	T, °C	D, g/mL	T, °C	D, g/mL
15	0.9991	20	0.9982	25	0.9970
16	0.9989	21	0.9980	26	0.9967
17	0.9987	22	0.9978	27	0.9965
18	0.9986	23	0.9975	28	0.9962
19	0.9984	24	0.9973	29	0.9960

## 1C Experiment: The Density of a Metal

Different metals are furnished for this determination, either as cylindrical rods or as pellets or shot. In this experiment, you will determine the mass of a metal sample and its volume, then calculate its density.

1. Start with a 10 mL graduated cylinder about half filled with H<sub>2</sub>O. Read the volume of water in the cylinder. Volume of H<sub>2</sub>O = \_\_\_\_\_ mL

2. Put a cylindrical metal rod or a sample of metal pellets or shot into the water so that the entire sample is submerged. Read the volume occupied by the water and the metal. Obtain the greatest change in the water level consistent with having all the metal sample submerged with no bubbles adhered to the surface of the sample. You may have to thump or jostle the cylinder to get rid of bubbles.

$$\text{Volume of H}_2\text{O} + \text{Volume of metal} = \text{_____ mL}$$

3. The difference in the two volumes is the volume of the metal sample.

$$\begin{aligned} \text{Volume of H}_2\text{O} + \text{Volume of metal} &= \text{_____ mL} \\ - \text{Volume of H}_2\text{O} &= \text{_____ mL} \\ = \text{Volume of metal} &= \text{_____ mL} \end{aligned}$$

4. Thoroughly dry the metal pieces and weigh them. Use brown paper towels to absorb most of the water, then a hair dryer if available.

$$\text{"Weight"} = \text{Mass of metal} = \text{_____ g}$$

5. Calculate the density of the metal and pick the metal from the Table of Metal Densities (below).

$$D = \text{Mass of metal} \div \text{Volume of metal} = \text{_____ g/mL}$$

Metal name \_\_\_\_\_ Metal symbol \_\_\_\_\_

**Table of Metal Densities (g/mL)**

Metal	D, g/mL	Metal	D, g/mL	Metal	D, g/mL
Aluminum	2.70	Iron	7.87	Tin	7.29
Antimony	6.62	Lead	11.34	Titanium	4.51
Bismuth	9.80	Magnesium	1.74	Tungsten	19.30
Copper	8.94	Molybdenum	10.22	Zinc	7.13

## 1D Experiment: Length

1. While you are thinking about the precision of reading a balance, select a wooden splint and measure its length on the inch scale and the centimeter scale.

$$\text{Length} = \text{_____ inches.} \qquad \text{Length} = \text{_____ cm}$$

2. Convert the length in inches to length in centimeters using the factor 2.54 cm/in. Pay attention to the significant figures in your results.

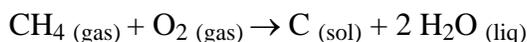
$$\text{Length in inches converted to length in centimeters} = \text{_____ cm}$$

## Adjusting a Bunsen Burner

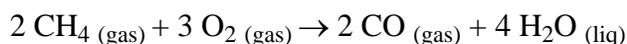
When selecting a burner, check to see that the gas needle valve on the bottom will close completely. Also check to see that the barrel of the burner will screw in and out so that the air supply to the flame can be controlled.

With the burner gas valve off and the hose connected to the burner and the bench gas cock (see Figure 2.1), turn the bench gas cock fully on and check for leaks around the burner with a match. With the air vents closed, open the burner gas valve and light the flame. The flame should be yellow and luminous.

Stick a test tube into the flame briefly. You should observe a deposit of carbon black on the tube. When hydrocarbons such as methane [CH<sub>4</sub> (natural gas)] burns in too little air (oxygen), the reaction is:



With a bit more air, the flame becomes hotter and blue, but carbon monoxide is formed:



Now adjust the air supply -- you may also have to adjust the gas with the burner valve -- until the flame resembles Figure 2.2. This is the hottest flame and is characterized by a blue inverted cone shape within the flame that is the so-called reducing flame. A little above the apex of the cone is the hottest area in the flame, reaching temperatures around 1500°C.

Towards the top of the flame, conditions are oxidizing (high temperatures, excess O<sub>2</sub>). The well-adjusted flame completely converts methane and oxygen to carbon dioxide and water:

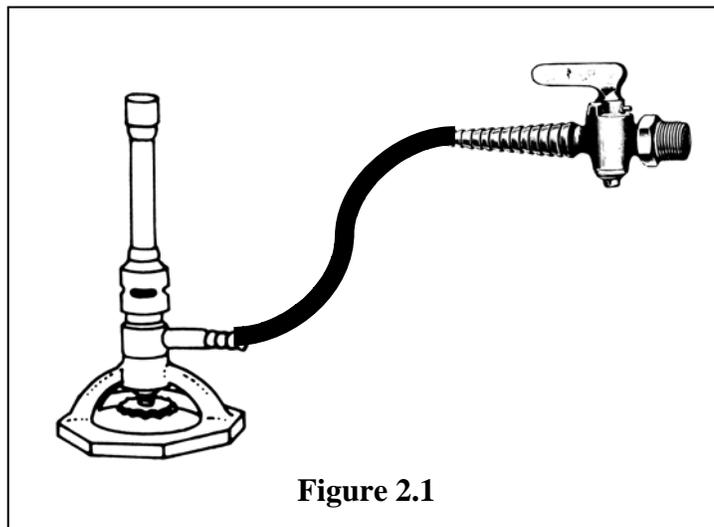
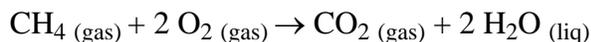


Figure 2.1

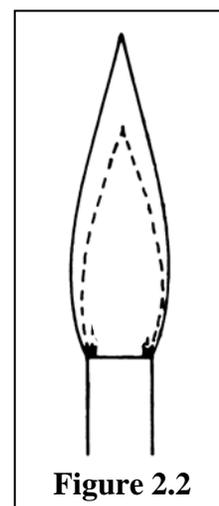


Figure 2.2

## 1E Exercise: Understanding Flames

Take a wooden splint and hold it with its edge resting on the top of the burner. Notice how the splint is burned only on the edges of the flame. The flame under the cone is relatively cool (about 350 °C). Higher in the flame, the splint ignites uniformly. Show the splint to your instructor along with the rest of today's results.

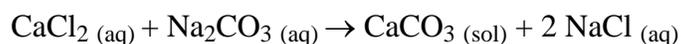
To turn off the Bunsen burner, execute the lighting procedure in reverse. Shut the gas valve at the base of the burner, then close the bench gas cock.

## Reactions and Separations

A very common and useful type of reaction is the double displacement reaction (also called a metathesis or exchange reaction). This occurs readily among ionic compounds in solution when two ions that form an "insoluble" salt are mixed. When the clear (not necessarily colorless) solutions are mixed, a solid forms that "falls out" of solution or precipitates. The solid that is formed, called the precipitate, may be separated from the remaining solution, which still contains the counter-ions of the two salts that formed the precipitate. Upon evaporation, the other more soluble product is recovered.

## 1F Experiment: Reactions and Separations I

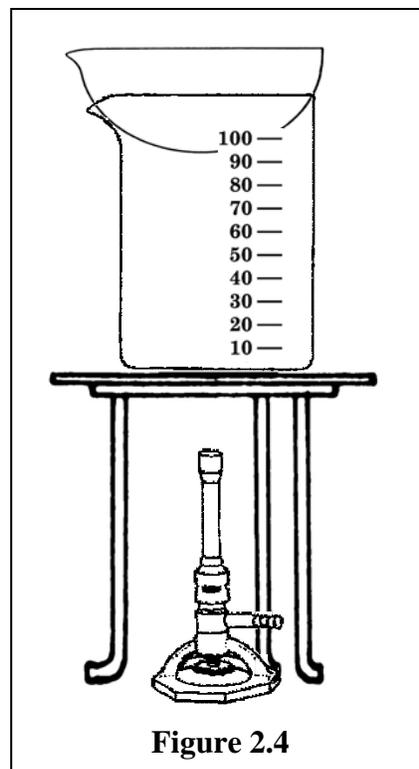
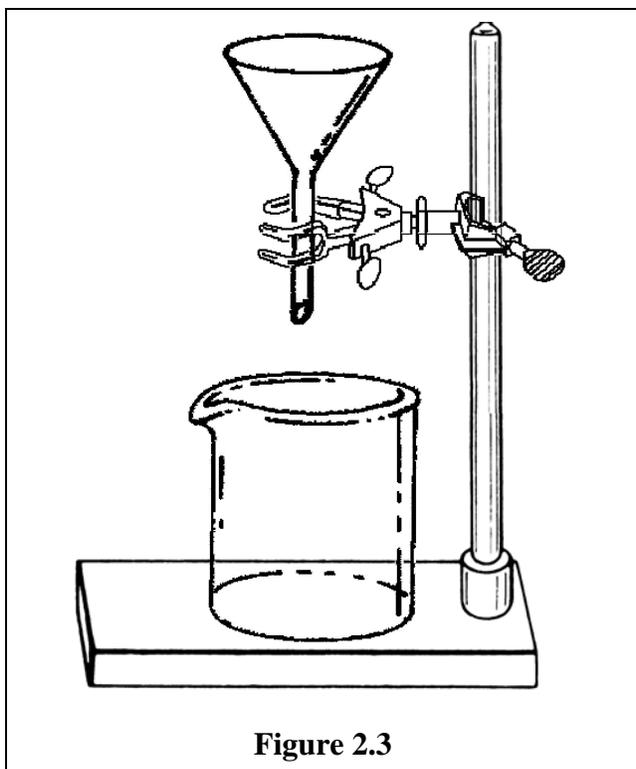
Equal volumes of Na<sub>2</sub>CO<sub>3</sub> and CaCl<sub>2</sub> solutions will be mixed. The concentrations of these solutions are such that equal volumes contain exact reacting quantities. The reaction is:



After mixing 5mL of each solution, note the formation of a white solid. Set up a filter as shown in Figure 2.3. Catch the filtrate (the solution which passes through the filter paper), which should be clear. Keep the precipitate that has been caught on the filter paper to show to your instructor. What is the formula of the precipitate caught by the filter paper?

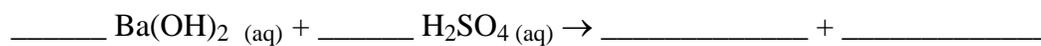
---

Set up an evaporating dish on a steam bath as shown in Figure 2.4 and evaporate a portion of the filtrate. Keep the residue in the evaporating dish to show to your instructor. Be sure you know what these compounds are.



### 1G Exercise: Reactions and Separations II

Barium sulfate,  $\text{BaSO}_4$  is very insoluble. Suppose solutions of  $\text{Ba}(\text{OH})_2$  and  $\text{H}_2\text{SO}_4$  (aq) are mixed. Complete the equation below:



**Report Form 1: Introductory Exercises** Name \_\_\_\_\_

Partner \_\_\_\_\_ Section # \_\_\_\_\_

Source	Cumulative Mass	Mass By Difference	Direct Weighing	Deviation From Mean
Weighing Paper	g			
1 <sup>st</sup> penny	g	g	g	g
2 <sup>nd</sup> penny	g	g	g	g
3 <sup>rd</sup> penny	g	g	g	g
4 <sup>th</sup> penny	g	g	g	g
5 <sup>th</sup> penny	g	g	g	g
<b>Average:</b>			g	g

**1B Experiment: The Density of Water at Room Temperature**

1. T = \_\_\_\_\_ °C
2. "Weight" = Mass of H<sub>2</sub>O = \_\_\_\_\_ g
3. Volume of H<sub>2</sub>O = \_\_\_\_\_ mL
4. D = Mass of H<sub>2</sub>O / volume of H<sub>2</sub>O = \_\_\_\_\_ g/mL
5. %Error =  $[(D_{\text{TAB}} - D_{\text{EXP}}) \div D_{\text{TAB}}] \times 100 =$  \_\_\_\_\_ %Error

**1C Experiment: The Density of a Metal**

6. Volume of H<sub>2</sub>O = \_\_\_\_\_ mL
  7. Volume of H<sub>2</sub>O + Volume of metal = \_\_\_\_\_ mL
  8. Volume of metal = \_\_\_\_\_ mL
  9. "Weight" = Mass of metal = \_\_\_\_\_ g
  10. D = Mass of metal ÷ Volume of metal = \_\_\_\_\_ g/mL
- Metal name \_\_\_\_\_  
Metal symbol \_\_\_\_\_

**1D Experiment: Length**

11. Length = \_\_\_\_\_ inches                      Length = \_\_\_\_\_ cm
12. Length in inches converted to length in centimeters = \_\_\_\_\_ cm

**1E Exercise: Understanding Flames**

Show the splint to your instructor along with the rest of today's results.

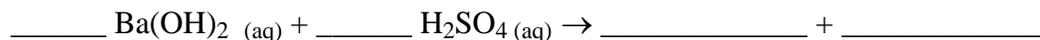
**1F Experiment: Reactions and Separations I**

Keep the precipitate that has been caught on the filter paper to show to your instructor.

What is the formula of the precipitate caught by the filter paper? \_\_\_\_\_

Keep the residue in the evaporating dish to show to your instructor.

**1G Exercise: Reactions and Separations II**



## Lab Session 3, Experiment 2: Oxygen

Oxygen is the most abundant element on or near the surface of the earth. Oxygen is present in large quantities in the air, water, rocks, and minerals. Oxygen combines with almost all other elements to form compounds that are called oxides of these elements.

Oxygen is not highly active (or reactive) at room temperature, but at higher temperatures its activity (or reactivity) increases markedly.

Oxygen may be prepared in a number of ways. Liquefied air is the source of commercial quantities of oxygen. Electrolysis of water is sometimes used to obtain small quantities of oxygen (and hydrogen). The most common laboratory method is the thermal decomposition of oxygen-containing compounds. *This does not imply that any compound that contains oxygen is a ready source of oxygen.*

Metal oxides that are soluble in or react with water give basic solutions. These oxides are called "basic oxides" or "basic anhydrides". Oxides of nonmetals that are soluble in or react with water give acidic solutions, and these oxides are called "acidic oxides" or "acid anhydrides".

In this lab session, you will explore several factors that influence the speed of a chemical reaction. These are temperature, concentration, and use of a catalyst. A catalyst is a substance that changes the speed of a reaction, but is not consumed and may be recovered at the end of the reaction. Ideally then, a catalyst can be used over and over again.

***Procedural Note:*** This is the first experiment in which you must acquire a sample of a solid crystalline powder from a reagent bottle. Certain rules should be followed when securing reagents from a reagent bottle to prevent contamination of the reagent. It is *bad* procedure (thus it is *forbidden*) to insert a spatula into a reagent bottle. It is equally *improper* to return unused portions of a chemical to a reagent bottle. Thus, when securing a solid reagent (such as  $\text{BaO}_2$ ,  $\text{PbO}_2$ ,  $\text{KClO}_3$  or  $\text{MnO}_2$  in this experiment), you should take a beaker or watch glass with you and pour the needed amount of solid reagent into the beaker or watch glass. Alternatively, you may take a piece of filter paper or weighing paper to the reagent shelf and pour the solid on the paper. Excess solid reagent should be disposed of rather than returned to the reagent bottle. If the solid in the reagent bottle cannot easily be poured because it has acquired moisture and lumped or hardened, ask your lab instructor for help.

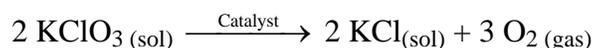
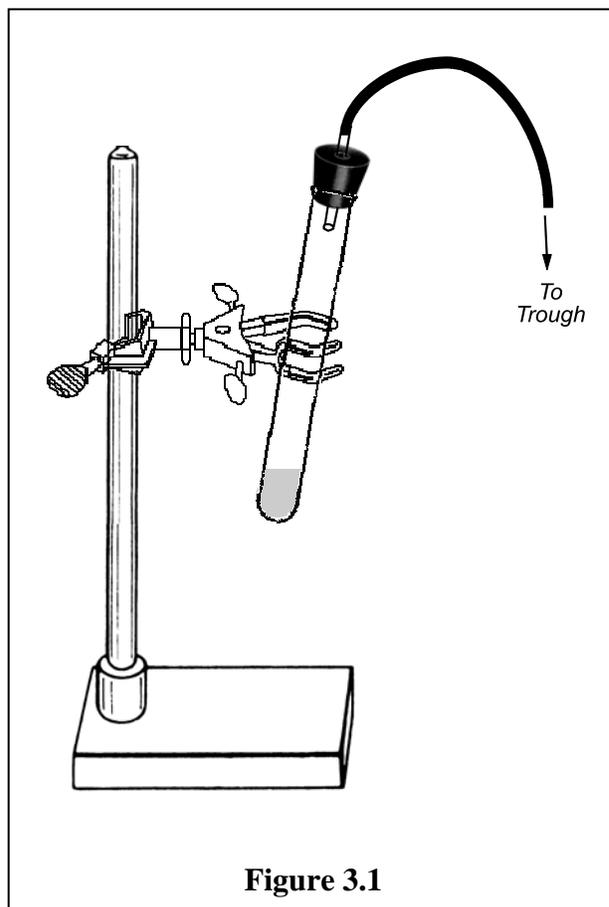
The same rules apply to liquid reagents. When obtaining a liquid reagent (either a solution or a pure liquid) from a reagent bottle, no objects (such as eyedroppers or pipettes) should be inserted into the reagent bottle. Liquid reagents should be poured from the reagent bottle into an appropriate container, or secured through the delivery/dispensing system provided. Excess liquid should not be returned to the reagent bottle.

## 2A Experiment

1. Place BaO<sub>2</sub> in a small test tube to a height of about 1 cm.
2. Using another small test tube, do the same with PbO<sub>2</sub>.
3. Heat each of the test tubes in the flame of your burner. Use the "glowing splint" test to show that oxygen is produced.



4. *Clean and dry* your large test tube. Add KClO<sub>3</sub> to the test tube to a height of 2 - 3 cm; add, using a spatula, a pinch of catalyst, MnO<sub>2</sub>. Mix well. Mount the test tube to a ring stand as shown in Figure 3.1. Check to make sure that the glass tube through the rubber stopper fits tightly, that the rubber stopper fits tightly in the test tube, and that the rubber tube for delivery to the pneumatic trough fits tightly on the glass tube through the rubber stopper. The rubber tubes may need to be secured by a hose clamp or by wire windings. Tight fits prevent oxygen loss to the atmosphere.
5. Prepare to collect oxygen by displacement of water. You will need two 8 oz bottles of oxygen, so before initiating the decomposition of KClO<sub>3</sub>, fill, invert, and position in the pneumatic trough both bottles. Heat the test tube, gently at first, to start the decomposition. After each bottle is filled, cover the mouth of the bottle with a watch glass, remove it from the trough, and set it right side up on the desk.



- (a) What does the collection of oxygen by displacement of water indicate about the solubility of O<sub>2</sub> in water?
-

(b) List four physical properties of oxygen.

---

---

(c) What is the purpose of  $\text{MnO}_2$ ?

---

6. Add about 10 mL of water to each of the oxygen-filled bottles. Move the watch glass aside slightly and lower a glowing splint into the bottle. Allow the combustion to continue for several seconds. Remove the splint, cover the bottle, and swirl to mix the water and gas.

(d) What gas was produced by the combustion?

---

(e) Write the equation for the reaction between the gas produced and the water.

---

(f) Test the solution formed with litmus paper. What do you observe?

---

(g) Classify the oxide formed by the combustion as acidic or basic:

---

7. *Under the hood*, place a *small amount* of sulfur in a combustion spoon. Ignite the sulfur with the burner flame. Lower the burning sulfur into the second oxygen bottle.

(h) Record your observations.

---

---

(i) What gas is produced by the combustion?

---

(j) Write the equation for the reaction between the gas and water.

---

(k) Test the solution in the bottle with litmus paper. What do you observe?

---

(l) Classify the oxide formed by the combustion of sulfur as acidic or basic:

---

**Report Form 2: Oxygen**

Name \_\_\_\_\_

Partner \_\_\_\_\_ Section # \_\_\_\_\_

(a) Is O<sub>2</sub> soluble or insoluble in water? \_\_\_\_\_

(b) List four physical properties of oxygen. \_\_\_\_\_  
\_\_\_\_\_

(c) What is the purpose of MnO<sub>2</sub>? \_\_\_\_\_  
\_\_\_\_\_

(d) What gas was produced by the combustion? \_\_\_\_\_  
\_\_\_\_\_

(e) Write the equation for the reaction between the gas produced and the water.  
\_\_\_\_\_

(f) Test the solution formed with litmus paper. What do you observe?  
\_\_\_\_\_

(g) Classify the oxide formed by the combustion as acidic or basic:  
\_\_\_\_\_

(h) Record your observations.  
\_\_\_\_\_  
\_\_\_\_\_

(i) What gas is produced by the combustion?  
\_\_\_\_\_

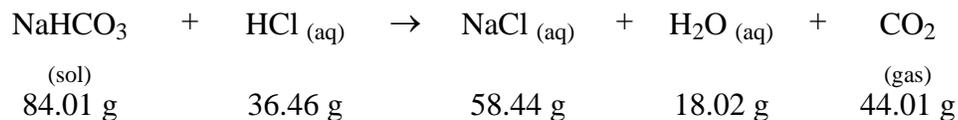
(j) Write the equation for the reaction between the gas and water.  
\_\_\_\_\_

(k) Test the solution in the bottle with litmus paper. What do you observe?  
\_\_\_\_\_

(l) Classify the oxide formed by the combustion of sulfur as acidic or basic:  
\_\_\_\_\_

## Lab Session 4, Experiment 3: Preparation of Sodium Chloride

Sodium chloride will be synthesized by reacting sodium bicarbonate with hydrochloric acid. The reaction equation is shown below:



The quantitative interpretation of the reaction is as follows: 84.01 g (1 mol) of sodium bicarbonate reacts with 36.46 g (1 mol) of hydrochloric acid to generate 58.44 g (1 mol) of salt, 18.02 g (1 mol) of water, and 44.01 g (or 1 mol) of carbon dioxide. Of course the starting quantity of  $\text{NaHCO}_3$  may be more or less than 84.01 g, but a proportionate quantity of the hydrochloric acid will be consumed, and proportionate quantities of the products will be formed. For example, should 100.00 g of  $\text{NaHCO}_3$  react with excess acid, the theoretical mass of salt produced would be calculated as follows:

$$\frac{58.44 \text{ g sodium chloride}}{84.01 \text{ g sodium bicarbonate}} \left| \frac{100.00 \text{ g sodium bicarbonate}}{\phantom{84.01 \text{ g sodium bicarbonate}}} \right. = 69.55 \text{ g sodium chloride}$$

Note that when a bicarbonate is reacted with excess acid, the salt produced is the only substance not readily volatile. That is, the unreacted acid, the water, and the carbon dioxide are easily removed by heating.

### 3A Experiment

Chemicals needed: sodium bicarbonate, concentrated hydrochloric acid.

1. Accurately weigh an empty, dry evaporating dish, and record its mass in blanks (b) and (e) in the table below.
2. Add to the dish about 5 g of  $\text{NaHCO}_3$  and weigh again. Record the mass in blank (a) in the table below.
3. Add 5 to 6 mL of distilled water to the dish to wet the bicarbonate. Cover the dish with a watch glass.
4. Move the watch glass aside slightly and add, in small portions, about 6 mL of concentrated hydrochloric acid from a 10 mL graduated cylinder. These small portions of acid should be added so that the acid runs down the inside wall of the evaporating dish. After the addition of 6 mL of acid, continue adding acid only as long as  $\text{CO}_2$  (gas) continues to be evolved.
5. Remove the watch glass and evaporate to dryness over a water bath (the evaporating dish is placed on top of a beaker containing boiling water).
6. Next, heat the dish on wire gauze with the burner for about 3 minutes. Allow the dish to cool and weigh accurately. Again, heat the dish, cool, and weigh. Continue heating and weighing until the dish reaches constant mass. Record this constant mass in blank (d) in the table below.

(a) Mass of dish and $\text{NaHCO}_3$	g
(b) Mass of empty dish	g
(c) Mass of $\text{NaHCO}_3$ [(a)–(b)]	g
(d) Mass of dish and residue	g
(e) Mass of empty dish (b)	g
(f) Mass of NaCl residue [(d)–(e)]	g

7. In the reaction studied, what reactant was present in limiting quantity?
- 

### 3B Calculations

1. Calculate the theoretical yield of NaCl.

$$\frac{58.44 \text{ g NaCl}}{84.01 \text{ g NaHCO}_3} \left| \frac{\text{_____ g NaHCO}_3}{\text{_____ g NaCl}} \right. = \text{_____ g NaCl}$$

2. Calculate the percentage yield of NaCl.

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

### 3C Exercises

In the following problems, show calculations:

3. What theoretical mass of NaCl would result from reacting 60.00 g of NaHCO<sub>3</sub> with excess HCl (aqueous)?

$$\frac{58.44 \text{ g NaCl}}{84.01 \text{ g NaHCO}_3} \left| \frac{60.00 \text{ g NaHCO}_3}{1} \right. = \text{_____ g NaCl}$$

4. What theoretical mass of NaCl would result from reacting 3.00 moles of NaHCO<sub>3</sub> with excess HCl (aqueous)?

$$\frac{3.00 \text{ mol NaHCO}_3}{1} \left| \frac{1 \text{ mol NaCl}}{1 \text{ mol NaHCO}_3} \right| \frac{58.44 \text{ g NaCl}}{1 \text{ mol NaCl}} = \text{_____ g NaCl}$$

5. What mass of CO<sub>2</sub> would be generated along with 35.00 g of NaCl?

$$\frac{35.00 \text{ g NaCl}}{58.44 \text{ g NaCl}} \left| \frac{44.01 \text{ g CO}_2}{1} \right. = \text{_____ g CO}_2$$

**Report Form 3: Preparation of Sodium Chloride**

Name \_\_\_\_\_

Partner \_\_\_\_\_ Section # \_\_\_\_\_

**3A Experiment**

(a) Mass of dish and NaHCO <sub>3</sub>	_____
(b) Mass of empty dish	_____
(c) Mass of NaHCO <sub>3</sub> [(a)-(b)]	_____
(d) Mass of dish and residue	_____
(e) Mass of empty dish (b)	_____
(f) Mass of NaCl residue [(d)-(e)]	_____

In the reaction studied, what reactant was present in limiting quantity?

\_\_\_\_\_

**3B Calculations**

1. Theoretical yield of NaCl. \_\_\_\_\_

2. Percentage yield of NaCl. \_\_\_\_\_

**3C Exercises**

3. What theoretical mass of NaCl would result from reacting 60.00 g of NaHCO<sub>3</sub> with excess HCl (aqueous)? \_\_\_\_\_

4. What theoretical mass of NaCl would result from reacting 3.00 mol of NaHCO<sub>3</sub> with excess HCl (aqueous)? \_\_\_\_\_

5. What mass of CO<sub>2</sub> would be generated along with 35.00 g of NaCl? \_\_\_\_\_

## Lab Session 5, Experiment 4: Law of Definite Proportions

The law of definite proportions states that when two or more elements combine to form a given compound, they do so in fixed proportions by mass. This is the same as saying the composition of a compound is fixed. For example, sodium chloride contains 39.3% by mass sodium and 60.7% by mass chlorine.

In this experiment a sample of  $\text{KClO}_3$  will be decomposed thermally, and the oxygen produced will be expressed as a percentage of the original mass of  $\text{KClO}_3$ . Experimental results will then be compared to the theoretical percentage of oxygen in  $\text{KClO}_3$ .

### 4A Experiment: Decomposition of a Salt

1. Add approximately 0.1 g of  $\text{MnO}_2$  to a clean, dry crucible and weigh the crucible including the catalyst. Enter the mass on line (b) below.
2. Add  $\text{KClO}_3$  to the crucible until it is about one-third full. Mix the  $\text{KClO}_3$  and  $\text{MnO}_2$  thoroughly. Weigh again and enter the mass on line (a) below.
3. Place the crucible on a wire triangle supported on a ring clamp. Adjust the ring clamp to a height that allows the crucible to be heated with the flame of your burner.
4. Heat the crucible slowly at first. Then regulate heating to prevent boil-over from the crucible. The objective is to completely decompose the chlorate to the chloride and oxygen; therefore, heat for about ten minutes after apparent total decomposition. Heating should be so vigorous as to cause the bottom of the crucible to glow red. Heat until all purple color is gone and the contents are a uniform gray color.
5. Allow the crucible to cool. Weigh the crucible and enter the mass on line (d) below.
6. Compute the following: mass of oxygen, mass of  $\text{KCl}$ , experimental percentage of oxygen, theoretical percentage of oxygen, and percent error in the experimental percentage of oxygen, calculated as  $[(\text{actual yield} - \text{theoretical yield}) / \text{theoretical yield}] \times 100$ .

(a) Mass of crucible with $\text{MnO}_2$ and $\text{KClO}_3$	g
(b) Mass of crucible with $\text{MnO}_2$	g
(c) Mass of $\text{KClO}_3$ [(a)-(b)]	g
(d) Mass of crucible and contents after heating	g
(e) Mass of oxygen [(a)-(d)]	g
(f) Mass of $\text{KCl}$ [(c)-(e)]	g
(g) Experimental percentage of oxygen	%
(h) Theoretical percentage of oxygen	%
(i) Percent error in experimental percentage of oxygen	%

7. Now assume that the compound you decomposed has the formula  $\text{KClO}_x$ . Note that "x" is the ratio of moles of O to moles of  $\text{KCl}$ . Complete the following table.

(j) Experimental moles of O	mol
(k) Experimental moles of $\text{KCl}$	mol
(l) Calculated value of above ratio	
(m) Formula of compound decomposed	

## Hydrates

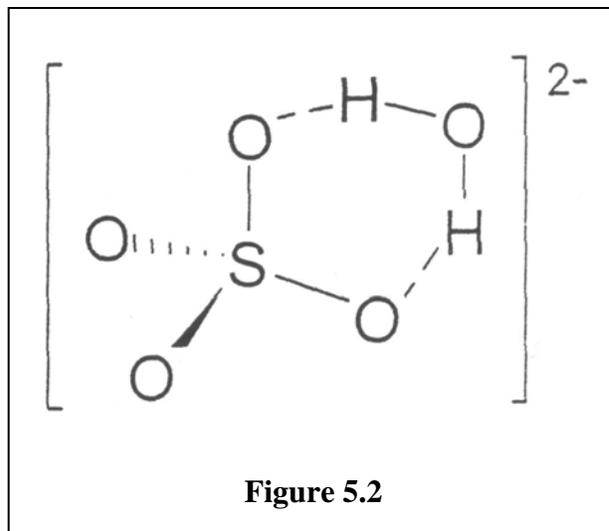
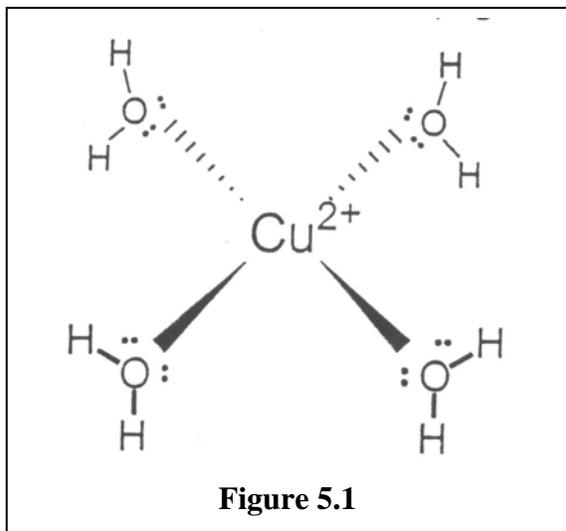
Hydrates are substances formed when water combines chemically in definite proportions with a salt. The ratio of water molecules to the ions of the salt is a constant. Hydrates are not mixtures. The anhydrous (without water) form of the salt is produced when all the water of hydration is lost. Some examples of hydrates are listed below:

<u>Formula</u>	<u>Names</u>
$(\text{CaSO}_4)_2 \cdot \text{H}_2\text{O}$	calcium sulfate hemihydrate "plaster of paris"
$\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$	calcium sulfate dihydrate "gypsum"
$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$	copper (II) sulfate pentahydrate "blue vitriol"
$\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$	magnesium sulfate heptahydrate "epsom salt"
$\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$	sodium carbonate decahydrate "washing soda"

The • in the formula indicates a kind of chemical bond that usually can be easily broken. For example, copper (II) sulfate pentahydrate can be converted to anhydrous copper (II) sulfate by heating:



In  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  the bonding involves four water molecules coordinatively bound to the  $\text{Cu}^{2+}$  ion in a square planar structure (Figure 5.1) and one molecule of water bound to the sulfate ion by hydrogen bonds (Figure 5.2).



Loss of hydration water to the atmosphere is called **efflorescence**. The property of some salts to collect moisture from the air and dissolve in it is called **deliquescence**. A compound is **hygroscopic** if absorption of water from the atmosphere occurs without dissolution of the compound.

## 4B Experiment: Composition of a Hydrate

In the following experiment, record all masses in the data table.

1. Weigh your evaporating dish.
2. Introduce about 5 g of pulverized hydrated  $\text{CuSO}_4$ . Note the appearance and color of the solid; weigh the dish and contents.
3. Place the evaporating dish on the wire gauze and heat slowly until the color disappears. Prolonged heating may result in the decomposition of the anhydride. What color change occurred? \_\_\_\_\_
4. When cool, weigh the dish and anhydride.
5. From the data, calculate the following:  
 the percentage of water in the hydrate  $[(\text{mass of H}_2\text{O}) \div (\text{mass of hydrate})] \times 100$ ,  
 the number of moles of water  $[(\text{mass of H}_2\text{O}) \div \text{molar mass of H}_2\text{O}]$ ,  
 the number of moles of anhydride  $[\text{mass of anhydride} \div \text{molar mass of anhydride}]$ , and  
 the formula of the hydrate  $\text{CuSO}_4 \cdot x \text{H}_2\text{O}$  where  $x = [(\text{moles of water}) \div (\text{moles of anhydride})]$ .
6. Add water a few drops at a time to convert the anhydride back to the hydrate. Do you notice a change in temperature? (Check with a thermometer.) \_\_\_\_\_
7. What evidence of a chemical change did you observe? \_\_\_\_\_

	Formula of Copper Sulfate Hydrate	
	Experiment	Duplicate
(a) Mass of crucible or dish and hydrate	g	g
(b) Mass of empty crucible or dish	g	g
(c) Mass of hydrate [(a) – (b)]	g	g
(d) Mass of crucible or dish and anhydride	g	g
(e) Mass of anhydride [(d) – (b)]	g	g
(f) Mass of water in hydrate [(c) – (e)]	g	g
(g) Percentage of water in hydrate [(f) ÷ (c)] × 100	%	%
(h) Moles of water [(f) ÷ molar mass of H <sub>2</sub> O]	mol	mol
(i) Moles of anhydride [(e) ÷ molar mass of anhydride]	mol	mol
(j) Moles of water per mole of anhydride [(h) ÷ (i)]		
(k) Theoretical moles of water per mole of anhydride		
(l) % error $[ (j) - (k)  \div (k)] \times 100$		
(m) Formula of hydrate		
(n) Equation for decomposition of your hydrate		

#### 4C Experiment: Properties of hydrates

1. Place crystals of calcium chloride, washing soda, and lithium chloride on a watch glass and expose them to the air of the laboratory.
  2. Note the appearance of each sample towards the end of the lab. Which crystals deliquesce?
- 

Which crystals effloresce?

---

Explain why one sample gained water and why one lost water.

---

#### 4D Experiment

1. Introduce one spatula of each of the following into separate test tubes: sodium chloride, potassium dichromate, aluminum sulfate, cobalt chloride, and nickel sulfate. Record color before heating in the table below.
  2. Heat each sample in turn, holding the tube at an angle of about 30° so that any water will condense in the cool part of the tube. Record color after heating in the table below.
  3. When you heated salts in this experiment, which ones lost water?
- 
4. Can the original color be restored by addition of a few drops of water to the cooled tube?
- 

List any hydrates that changed color upon heating, but for which the color came back when water was added.

---

	<b>Color Before Heating</b>	<b>Color After Heating</b>
(a) Sodium Chloride		
(b) Potassium Dichromate		
(c) Aluminum Sulfate		
(d) Cobalt Chloride		
(e) Nickel Sulfate		

**Report Form 4: Law of Definite Proportions**

Name \_\_\_\_\_

Partner \_\_\_\_\_ Section # \_\_\_\_\_

**4A Experiment: Decomposition of a Salt**

(a) Mass of crucible with MnO <sub>2</sub> and KClO <sub>3</sub>	g
(b) Mass of crucible with MnO <sub>2</sub>	g
(c) Mass of KClO <sub>3</sub> [(a)–(b)]	g
(d) Mass crucible and contents after heating	g
(e) Mass of oxygen [(a)–(d)]	g
(f) Mass of KCl [(c)–(e)]	g
(g) Experimental percentage of oxygen	%
(h) Theoretical percentage of oxygen	%
(i) Percent error in experimental percentage of oxygen	%
(j) Experimental moles of O	mol
(k) Experimental moles of KCl	mol
(l) Calculated value of above ratio	
(m) Formula of compound decomposed	

**4B Experiment: Composition of a Hydrate**

What color change occurred? \_\_\_\_\_

Do you notice a change in temperature? (Check with a thermometer.) \_\_\_\_\_

What evidence of a chemical change did you observe? \_\_\_\_\_

	Experiment	Duplicate
(a) Mass of crucible or dish and hydrate	g	g
(b) Mass of empty crucible or dish	g	g
(c) Mass of hydrate [(a) – (b)]	g	g
(d) Mass of crucible or dish and anhydride	g	g
(e) Mass of anhydride [(d) – (b)]	g	g
(f) Mass of water in hydrate [(c) – (e)]	g	g
(g) Percentage of water in hydrate [(f) ÷ (c)] × 100	%	%
(h) Moles of water [(f) ÷ molar mass of water]	mol	mol
(i) Moles of anhydride [(e) ÷ molar mass of anhydride]	mol	mol
(j) Moles of water per mole of anhydride [(h) ÷ (i)]		
(k) Theoretical moles of water per mole of anhydride		
(l) % error [( (j) – (k)  ÷ (k)) × 100]		
(m) Formula of hydrate		
(n) Equation for decomposition of your hydrate		

### 4C Experiment: Properties of hydrates

Which crystals deliquesce? \_\_\_\_\_

Which crystals effloresce? \_\_\_\_\_

Explain why one sample gained water and why one lost water. \_\_\_\_\_

---

### 4D Experiment

When you heated salts in this experiment, which ones lost water?

---

Can the original color be restored by addition of a few drops of water to the cooled tube?

---

List any hydrates that changed color upon heating, but for which the color came back when water was added. \_\_\_\_\_

	Color Before Heating	Color After Heating
(a) Sodium Chloride		
(b) Potassium Dichromate		
(c) Aluminum Sulfate		
(d) Cobalt Chloride		
(e) Nickel Sulfate		

## Lab Session 6, Experiment 5: Hydrogen and the Activity Series of Metals

An activity series is a listing of chemical species in order of increasing (or decreasing) reactivity.

Metals (be sure you can locate them on the periodic table) tend to react by losing one or more electrons (per atom). Non-metals tend to gain electrons when they react. When a metal, or any chemical species, loses electrons, it is said to be *oxidized*. The word "oxidized" is used by chemists to refer to the process that usually happens when atoms react with oxygen. Metals are considered more active since they are more easily oxidized. One way an activity series may be determined is to observe the spontaneity of reaction of a set of metals with a single oxidizing agent.

The oxidizing agent used in most of the experiments in this lab session will be hydrochloric acid (aqueous hydrogen chloride). In fact, any strong acid would do because the chemical entity actually taking the electrons from the metals is the hydronium ion,  $\text{H}_3\text{O}^+_{(\text{aq})}$ , which is present in all strong aqueous acids. Frequently, this species is simply called the hydrogen ion, or even the hydrated proton,  $\text{H}^+_{(\text{aq})}$ , since in aqueous solution it is hydrated.

The product formed when  $\text{H}^+_{(\text{aq})}$  is the oxidizing agent is  $\text{H}_{2(\text{gas})}$ , neutral molecular hydrogen, which can only be formed if  $\text{H}^+_{(\text{aq})}$  can take an electron from the metal.

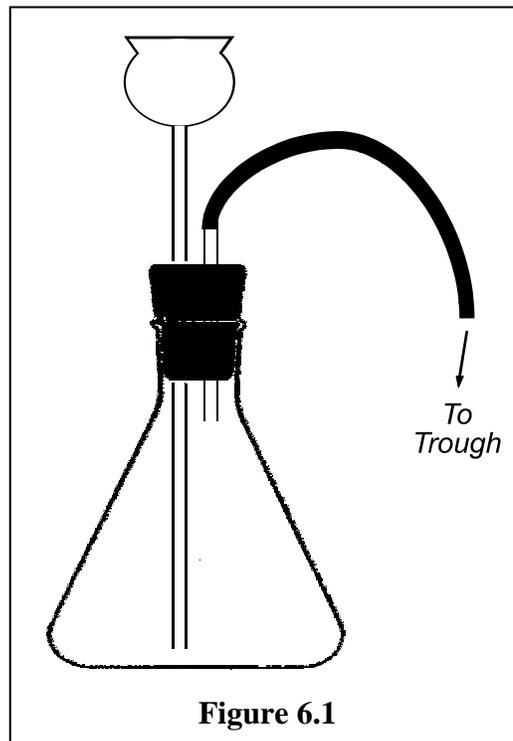
The ion formed from the metal in the reaction depends on the chemical nature of the metal. Periodic Groups IA, IIA, and IIIA are very consistent in this matter. Group IA metals form +1 ions, etc. Transition metals are not as predictable, but the charges on their cations are known from experiment. This knowledge is imparted to you by the charge shown in parentheses after these metals on the activity series furnished below.

# Activity Series for Metals

Oxides reduced by H <sub>2</sub> (gas)	K (K <sup>1+</sup> )	Metal + cold H <sub>2</sub> O → H <sub>2</sub> (gas) + hydroxide	
	Ba (Ba <sup>2+</sup> )		
	Sr (Sr <sup>2+</sup> )		
	Ca (Ca <sup>2+</sup> )		
	Na (Na <sup>1+</sup> )		
	Mg (Mg <sup>2+</sup> )		
	Al (Al <sup>3+</sup> )		
	Mn (Mn <sup>2+</sup> )		
	Zn (Zn <sup>2+</sup> )		
	Cr (Cr <sup>3+</sup> )		
Oxides <u>not</u> reduced by H <sub>2</sub> (gas)	Fe (Fe <sup>2+,3+</sup> )	Metal + hot water → H <sub>2</sub> (gas) + oxide or hydroxide	
	Cd (Cd <sup>2+</sup> )		
	Co (Co <sup>2+</sup> )		
	Ni (Ni <sup>2+</sup> )		
	Sn (Sn <sup>2+</sup> )		
	Pb (Pb <sup>2+</sup> )		
	H <sub>2</sub> (H <sup>1+</sup> )		
	Sb (Sb <sup>3+</sup> )		
	As (As <sup>3+</sup> )		
	Bi (Bi <sup>3+</sup> )		
Oxides reduced by heat alone	Cu (Cu <sup>2+</sup> )	Metal + acid → H <sub>2</sub> (gas) + salt	
	Ag (Ag <sup>1+</sup> )		
	Pd (Pd <sup>2+</sup> )		
	Hg (Hg <sup>2+</sup> )		
	Pt (Pt <sup>2+</sup> )		
	Au (Au <sup>3+</sup> )		
	Forms oxides only indirectly		
	Metal + O <sub>2</sub> (1 atm) → oxide		

## 5A Experiment

1. A single example of an acid reacting with a metal will be performed and the products noted.
2. Add 50 mL of concentrated HCl (12 M) to 50 mL of water to make 100 mL of 6 M HCl (save excess for later).
3. Using a 125 mL Erlenmeyer flask and a two-hole stopper fitted with a glass tube, assemble the hydrogen generator as shown in Figure 6.1. Make sure all fittings (thistle tube through the stopper, glass tube through the stopper, and rubber tubing to the glass tube) are tight. A hose clamp or wire winding may be needed.



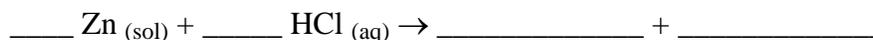
4. Place 5 g of granulated zinc in the flask, close it and add 25 mL 6 M HCl through the thistle tube. Be sure your thistle tube's lower end is submerged. Copious bubbles should form on the metal surface with release of heat.
5. Begin catching the effluent of this apparatus with a small test tube by letting it bubble into the water-filled, inverted tube under water in a pneumatic trough.
6. Being very careful to take the tube away from the generator in an *inverted* position, light the gas inside with a small flame. A small explosion will occur, making a barking sound. Continue this procedure periodically. Eventually, as the oxygen of the air is swept out of the generator, the product gas, hydrogen, will burn evenly with a nearly invisible flame. Make observations on the properties of hydrogen.

Density as compared to air \_\_\_\_\_

Solubility in water \_\_\_\_\_

Reactivity with oxygen (write an equation) \_\_\_\_\_

7. Now we turn our attention to the other product of the reaction. When you have finished testing the hydrogen, set your generator in the hood, open it and allow it to run down. Allow all the hydrogen to escape as it forms and to not build up. Later, each group of two students should decant a few mL of the solution off the metal and evaporate it over a steam bath. The solid residue is the salt zinc chloride. Complete the following reaction:



Be sure you can write a similar equation for each metal in our activity series that reacts with acid.

## 5B Experiment

1. The relative activity of some metals with acid will be observed.
2. While your hydrogen generator from Experiment 6A is exhausting itself, pick up a small sample of each of the following metals: granulated zinc, tin, lead, copper, iron, aluminum, and magnesium. If granules are not available, magnesium ribbon and aluminum turnings or wire will do. Place each metal in its own test tube. **The smallest amount you can see is the right amount.** Using too much of an active metal may cause the reaction contents to bubble out of the test tube.
3. To each in succession add 2 mL of 6 M HCl and observe the reaction.
4. When you have tested all the metals, list them in order of decreasing reactivity and compare your results with the order given in the activity series furnished. Judge the activity by speed of gas evolution and heat given off.

Most Least  
Active \_\_\_\_\_ Active

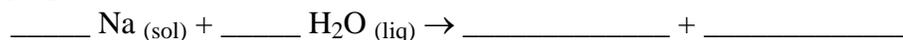
## 5C Experiment

A very active metal such as sodium reacts with cold water. Water is a considerably weaker oxidizing agent than 6 M HCl. Your instructor may choose to demonstrate this experiment.

1. The sodium is placed in a beaker of water placed behind a safety shield. Notice that the reaction is so exothermic that it will ignite the hydrogen released. If no ignition occurs, place the sodium on a small piece of filter paper floated on the water. The residual molten sodium oxide sphere has a tendency to burst into pieces some time after the reaction seems to be over.

**BE CAREFUL -- Be sure you have your eye protection on while watching this!**

2. Test the solution produced with red litmus paper. In addition to  $H_2$  (gas), a strongly basic metal hydroxide is produced.
3. Complete the following equation:



Be sure you can write a similar equation for each of the very active metals of the activity series that will react with cold water. This experiment may be repeated with calcium metal, if available.

## 5D Experiment

An active metal reacts with oxygen and also reacts with hot water. As shown on the activity series, most metals will react with oxygen at one atmosphere (in air, the partial pressure of O<sub>2</sub> is only 0.2 atm) to produce an oxide. Less active metals, which do not react with cold water, will react with hot water to release H<sub>2</sub>.

1. Hold a 3 inch piece of magnesium ribbon by one end with your tongs. Light the other end in the burner flame. (Careful, do not look directly at the flame; it is very bright.) Now quickly plunge the burning ribbon into a 400 mL beaker of boiling water. How can it continue to burn without the oxygen of the air? \_\_\_\_\_

2. Complete the two following equations:



At an elevated temperature, Mg(OH)<sub>2</sub> will dehydrate to MgO as shown in the activity series, but here the sparingly soluble base, Mg(OH)<sub>2</sub>, may be detected in water by turning litmus blue. Since either a metal hydroxide or oxide will react with an acid to form a salt (and water, in the case of hydroxide), the salt will always be a product when metal oxidation is done by acid, even if the most active metals are involved.

## 5E Experiment

Apparently, activity is affected by conditions of concentration and temperature.

*Part 1:* More concentrated acid is a stronger oxidizing agent than dilute acid.

1. Put 1 mL of 6 M HCl in a 10 mL graduated cylinder and add 9 mL of water, then mix.
2. Place two similarly sized pinches of granulated zinc in two test tubes. To one add 6 M HCl and to the other the dilute HCl.
3. What do you observe?  
\_\_\_\_\_

*Part 2:* If the metal is more finely subdivided, more contact between metal and acid occurs.

1. Place a pinch of zinc *dust* in a test tube and add 6 M HCl.
2. Compare this reaction to that of granulated zinc treated with 6 M HCl.  
\_\_\_\_\_

*Part 3:* The reaction rate activity should be enhanced at increased temperature.

1. Take a pinch of granulated tin and add 2 mL of 6 M HCl.
2. After noting the activity, heat carefully in a hot water bath.
3. What do you observe?  
\_\_\_\_\_

**Report Form 5: Hydrogen and the Activity Series of Metals**

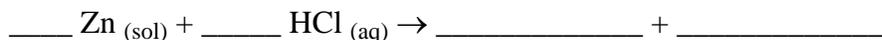
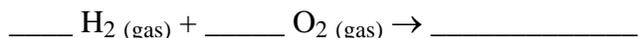
Name \_\_\_\_\_

Partner \_\_\_\_\_ Section # \_\_\_\_\_

**5A Experiment**

Density of H<sub>2</sub> gas as compared to air \_\_\_\_\_

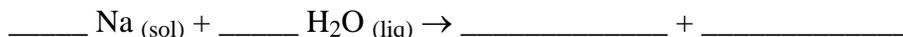
Solubility of H<sub>2</sub> gas in water \_\_\_\_\_



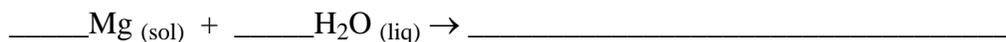
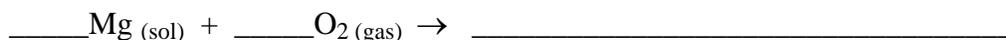
**5B Experiment**

Most Active \_\_\_\_\_ Least Active \_\_\_\_\_

**5C Experiment**



**5D Experiment**



**5E Experiment**

Part 1: Observation \_\_\_\_\_

Part 2: Observation \_\_\_\_\_

Part 3: Observation \_\_\_\_\_

## Lab Session 7, Experiment 6: Acid-Base Titration

Molarity (M) and normality (N) are two means of expressing solute concentrations. Molarity is defined as moles of solute per liter of solution, and in a similar way, normality is defined as equivalents (eq) of solute per liter of solution. From these definitions we may write the following:

$$M = \frac{\text{moles}}{L}, \text{ or by rearrangement, } M \times L = \text{moles}$$

$$N = \frac{\text{eq}}{L}, \text{ or by rearrangement, } N \times L = \text{eq}$$

$$N = \frac{\text{millequivalents (or meq)}}{\text{mL}}, \text{ or by rearrangement, } N \times \text{mL} = \text{meq}$$

In this experiment, we will consider only acid-base reactions. Your instructor will define the quantity of an acid that is an equivalent and the quantity of a base that is an equivalent. Let us stress here that one eq (or meq) of an acid reacts exactly with one eq (or meq) of a base. Whatever the number of eq (or meq) of acid, the same number of eq (or meq) of base will be consumed in the reaction. Thus,

$$\text{meq}_{\text{acid}} = \text{meq}_{\text{base}}$$

$$\text{mL}_{\text{acid}} \times N_{\text{acid}} = \text{mL}_{\text{base}} \times N_{\text{base}}$$

Assume that 20.00 mL of 0.3000 N acid requires 30.00 mL of base. Then,

$$N_{\text{base}} = \frac{\text{mL}_{\text{acid}} \times N_{\text{acid}}}{\text{mL}_{\text{base}}} = \frac{20.00 \times 0.3000}{30.00} = 0.2000 \text{ N}$$

Our assumed case may be stated as follows: The titration of 20.00 mL of 0.3000 N acid requires 30.00 mL of 0.2000 N base.

Titration is the precise measurement of the volume of one reagent required to react with a mass or volume of another reagent. As in the titration described above, the solution of base would be added from a burette to the acid until the acid is just neutralized. You will use the indicator phenolphthalein, which is colorless in acid and pink in base, to indicate when the acid is just neutralized. Two or three drops of phenolphthalein are added to the acid before the titration is begun. This colorless solution turns pink when, as a result of base addition from the burette, the solution changes from acidic to barely basic. When the color change appears, the burette is read to obtain the volume of base added.

## 6A Experiment

In this experiment, you will use a standardized NaOH solution to titrate against an aqueous acetic acid unknown in order to determine the normality of the acetic acid solution.

- You will be provided with three chemicals:
  - An NaOH standard solution of known normality (approximately 0.21 N). The exact normality should be recorded in the data table.
  - Acetic acid,  $\text{CH}_3\text{CO}_2\text{H}$ , of unknown normality. This is to be prepared by the stockroom personnel by diluting 12 mL of concentrated acetic acid to one liter.
  - Phenolphthalein in a dropper bottle.
- Clean and empty the burette, rinse with and then fill with the acetic acid.
- Draw off three 15.00 mL samples of acetic acid into three Erlenmeyer flasks. You may use either size flask, 125 mL and/or 250 mL.
- Add 2-3 drops of indicator to each sample.
- Clean and empty the burette, rinse with and then fill with the NaOH solution.
- Titrate each acid sample to the phenolphthalein end-point. Complete the data table below for each of the three titrations.

		Titrations			
		1	2	3	
$N_{\text{base}}$					
$\text{mL}_{\text{acid}}$					
$\text{mL}_{\text{base}}$					
$N_{\text{acid}}$					
% deviation					
				<b>Averages</b>	
				$N_{\text{acid}}$	
				% deviation	

## 6B Exercise

- Calculate the grams of acetic acid in one liter of solution.
- The density of the acetic acid solution is 1.00 g/mL. Calculate the percentage of acetic acid in the solution.
- Give the equations for the neutralization of  $\text{HCl}_{(\text{aq})}$  and  $\text{CH}_3\text{CO}_2\text{H}_{(\text{aq})}$  with  $\text{NaOH}_{(\text{aq})}$ .

**Be sure to clean your burette and rinse it with deionized water before you return it to the hood.**

**Report Form 6: Acid-Base Titration** Name \_\_\_\_\_

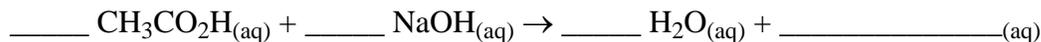
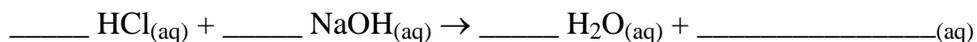
Partner \_\_\_\_\_ Section # \_\_\_\_\_

### 6A Experiment

Titrations						
		1	2	3		
N <sub>base</sub>						
mL <sub>acid</sub>						
mL <sub>base</sub>					Averages	
N <sub>acid</sub>					N <sub>acid</sub>	
% deviation					% deviation	

### 6B Exercise

1. Grams of acetic acid in one liter of solution \_\_\_\_\_ g
2. Percentage of acetic acid in the solution \_\_\_\_\_ %
3. Balanced Neutralization equations



**Note:** In the above table, if N<sub>1</sub>, N<sub>2</sub> and N<sub>3</sub> are the values of N<sub>acid</sub> for the individual titrations,

$$\text{Average } N_{\text{acid}} = \frac{N_1 + N_2 + N_3}{3}$$

Defining N<sub>avg</sub> = average N<sub>acid</sub>, the % deviations for the individual titrations are

$$\% D1 = \frac{|N_1 - N_{\text{avg}}|}{N_{\text{avg}}} \times 100, \quad \% D2 = \frac{|N_2 - N_{\text{avg}}|}{N_{\text{avg}}} \times 100, \quad \% D3 = \frac{|N_3 - N_{\text{avg}}|}{N_{\text{avg}}} \times 100$$

The average % deviation, therefore, is

$$\text{Average \% deviation} = \frac{\%D1 + \%D2 + \%D3}{3}$$

## Lab Session 8, Experiment 7: Antacids

The stomach secretes hydrochloric acid, which is necessary for digestion of food. However, for a number of reasons the stomach may secrete excess hydrochloric acid that causes discomfort. A number of commercial antacids are available to neutralize the excess acid. It should be remembered that antacids should be consumed in limited amounts in order that the stomach not be made alkaline. In this experiment, several brands of antacid tablets will be compared for their capacity to neutralize hydrochloric acid.

### 7A Experiment

In this experiment, you will add an antacid tablet to a known quantity of 0.50 M HCl that exceeds the amount required for reaction with the tablet. Standardized NaOH solution will be added to neutralize the excess acid. The millimoles of acid neutralized by the antacid will be the initial number of millimoles of acid minus the number of millimoles of excess acid, which is the number of millimoles of NaOH required.

*Chemicals Required:* Antacid tablets  
0.50 M HCl  
NaOH solution  
Phenolphthalein indicator

**CAUTION:**  
**DO NOT TASTE THE ANTACID**  
**TABLETS USED IN THIS LAB!**

1. Dissolve the antacid tablet in 50.00 mL of the 0.50 M HCl solution. Heat very gently and swirl the container if necessary to dissolve the tablet. A small part of the tablet may not dissolve.
2. After dissolving as much of the tablet as possible, add 3 drops of phenolphthalein solution. Since an excess of aqueous HCl solution has been added, the indicator should be colorless. If it is not colorless but rather turns red, add 10.0 mL more of the HCl solution, and note that the total volume of acid is 50.00 mL + 10.00 mL = 60.00 mL.
3. Titrate to the end point with the standardized NaOH solution. The molarity of the NaOH solution will be given on the label of the reagent bottle.

(a) Mass of antacid tested	g
(b) Milliliters of HCl used	mL
(c) Molarity of HCl	mol/L
(d) Millimoles of HCl used = $(\text{mL}_{\text{HCl}} \times M_{\text{HCl}})$	mmol
(e) Milliliters of NaOH used to neutralize excess acid	mL
(f) Molarity of NaOH	mol/L
(g) Millimoles of NaOH used = $(\text{mL}_{\text{NaOH}} \times M_{\text{NaOH}})$	mmol
(h) Millimoles of HCl (total used)	mmol
(i) Millimoles of excess HCl*	mmol
(j) Millimoles of HCl neutralized by antacid [(h)–(i)]	mmol
(k) Mass of CaCO <sub>3</sub>	g
(l) % CaCO <sub>3</sub> in tablet	%

\*Millimoles of excess acid = millimoles of base required to neutralize excess acid.

*Different types of antacid tables may be studied in your class. If so, you are encouraged to compare the results.*

### **7B Exercise**

1. The active ingredient in one brand of antacid is  $\text{CaCO}_3$ .

(a) Write the equation for the reaction between  $\text{HCl}_{(aq)}$  and  $\text{CaCO}_{3(s)}$ .

---

(b) How many millimoles of  $\text{HCl}_{(aq)}$  are neutralized by 0.35 g of  $\text{CaCO}_{3(s)}$ ? \_\_\_\_\_ mmol

2. Which household substance is frequently used as an antacid? \_\_\_\_\_

(a) Write the equation for the reaction between this substance and  $\text{HCl}_{(aq)}$ .

---

(b) How many millimoles of  $\text{HCl}_{(aq)}$  are neutralized by 5.0 g of this substance? \_\_\_\_\_ mmol

3. Suggest an explanation for why an antacid tablet may not be completely soluble in acid solution:

---

---

## Report Form 7: Antacids

Name \_\_\_\_\_

Partner \_\_\_\_\_ Section # \_\_\_\_\_

### 7A Experiment

(a) Mass of antacid tested	g
(b) Milliliters of HCl used	mL
(c) Molarity of HCl	mol/L
(d) Millimoles of HCl used = $(\text{mL}_{\text{HCl}} \times M_{\text{HCl}})$	mmol
(e) Milliliters of NaOH used to neutralize excess acid	mL
(f) Molarity of NaOH	mol/L
(g) Millimoles of NaOH used = $(\text{mL}_{\text{NaOH}} \times M_{\text{NaOH}})$	mmol
(h) Millimoles of HCl (total used)	mmol
(i) Millimoles of excess HCl*	mmol
(j) Millimoles of HCl neutralized by antacid [(h)–(i)]	mmol
(k) Mass of $\text{CaCO}_3$	g
(l) % $\text{CaCO}_3$ in tablet	%

\*Millimoles of excess acid = millimoles of base required to neutralize excess acid

### 7B Exercise

1. The active ingredient in one brand of antacid is  $\text{CaCO}_3$ .

(a) Write the equation for the reaction between  $\text{HCl}_{(\text{aq})}$  and  $\text{CaCO}_{3(\text{sol})}$ .

\_\_\_\_\_

(b) How many millimoles of  $\text{HCl}_{(\text{aq})}$  are neutralized by 0.35 g of  $\text{CaCO}_{3(\text{sol})}$ ? \_\_\_\_\_ mmol

2. Which household substance is frequently used as an antacid? \_\_\_\_\_

(a) Write the equation for the reaction between this substance and  $\text{HCl}_{(\text{aq})}$ .

\_\_\_\_\_

(b) How many millimoles of  $\text{HCl}_{(\text{aq})}$  are neutralized by 5.0 g of this substance? \_\_\_\_\_ mmol

3. Suggest an explanation for why an antacid tablet may not be completely soluble in acid solution:

\_\_\_\_\_

\_\_\_\_\_

## Lab Session 9, Experiment 8: Calorimetry, Heat of Reaction

**Specific heat** is an intensive property of a single phase (solid, liquid or gas) sample that describes how the temperature of the sample changes as it either absorbs or loses heat energy. Specific heat is generally a function of temperature, but, to a good approximation, it can be treated as being constant for a single phase over a moderate temperature range. The table below lists the specific heats at 25°C of liquid water and selected metal solids.

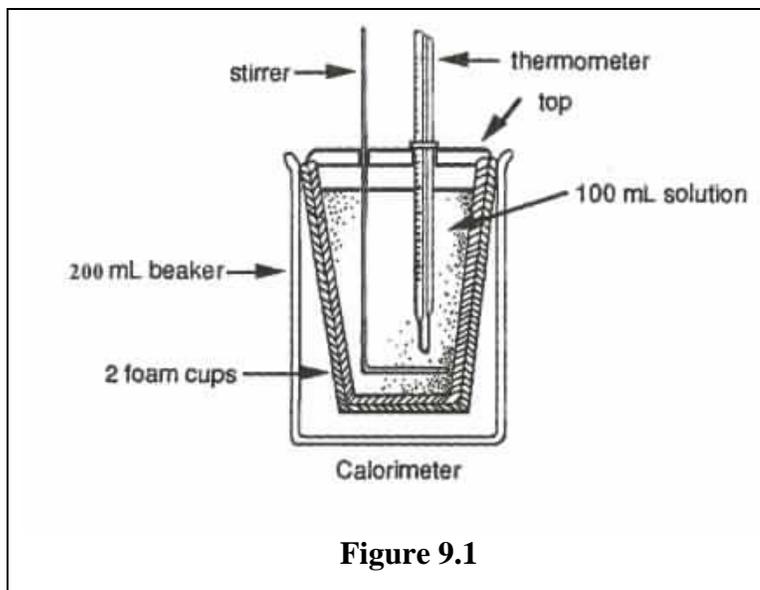
The Zeroth Law of Thermodynamics states: “If two samples of matter, initially at different temperatures ( $T_H$  and  $T_C$ , respectively), are placed in thermal contact, heat will be lost by the hotter sample ( $T_H$ ) and gained by the cooler one ( $T_C$ ). This exchange of heat will take place until both samples achieve the same final temperature,  $T_F$ , so that  $T_H > T_F > T_C$ .” The First Law of Thermodynamics states: “During heat exchange (or, as we shall see, during a chemical reaction), heat is neither created nor destroyed.” Thus, heat lost equals heat gained, if the heat exchange can be sufficiently insulated from the surroundings so that not very much heat escapes to the surroundings. A vessel that provides adequate heat insulation from its surroundings, and in which temperature changes are measured in order to determine specific heats (or heats of chemical reactions) is called a **calorimeter**. A perfect calorimeter absorbs no heat from the solution that it contains, nor loses any heat to the surroundings. No calorimeter is perfect, however. A typical calorimeter used in freshman chemistry labs is made of two nested Styrofoam cups, and looks like the one shown in Figure 9.1.

If we place  $m_{\text{metal}}$  grams of a hot metal, at a Celsius temperature  $T_H$ , into a nearly perfect calorimeter, along with  $m_{\text{water}}$  grams of water, at a lower Celsius temperature  $T_C$ , then the metal and the water will thermally equilibrate at a final intermediate Celsius temperature,  $T_F$ .

Since the heat lost by the metal is equal to the heat gained by the water within the calorimeter, the following equation holds ( $m$  is in the units g,  $T$  is in the units °C and sp ht is in the units J/g °C):

$$[(m_{\text{metal}}) \times (T_H - T_F) \times (\text{sp ht}_{\text{metal}})] = [(m_{\text{water}}) \times (T_F - T_C) \times (\text{sp ht}_{\text{water}})] .$$

Substance	Specific Heat, J/(g°C)
water (liquid)	4.184
aluminum (solid)	0.901
chromium (solid)	0.448
copper (solid)	0.386
iron (solid)	0.450
lead (solid)	0.129
nickel (solid)	0.443
tin (solid)	0.217
zinc (solid)	0.386



**Figure 9.1**

We can possibly identify an unknown metal by determining its specific heat using the preceding heat exchange formula with the specific heat of water taken to be 4.184 J/g °C. From the table above, it is obvious that we cannot distinguish between copper and zinc, or between chromium and nickel, on the basis of specific heat alone. Additional information is needed, such as color. (Copper has the characteristic "copper" color, whereas zinc is gray.) Rearranging the heat exchange formula and inserting the specific heat of water gives

$$\text{sp ht}_{\text{metal}} = \frac{(m_{\text{water}})(T_{\text{F}} - T_{\text{C}})(4.184 \text{ J/g}^\circ\text{C})}{(m_{\text{metal}})(T_{\text{H}} - T_{\text{F}})}$$

A major source of error in the preceding example is the assumption that no heat went into warming the calorimeter vessel and that no heat was lost to the surroundings. We can take this into account by defining the **heat capacity** of the calorimeter  $C$ , which has the units  $\text{J}/^\circ\text{C}$ . By making a similar heat exchange measurement in which we mix  $m_{\text{H}}$  grams of hot water, initially at temperature  $T_{\text{H}}$ , with  $m_{\text{C}}$  grams of water initially at a lower temperature  $T_{\text{C}}$  (perhaps at or near room temperature). The value of the calorimeter's heat capacity reflects the calorimeter's efficiency. (A more efficient calorimeter has a smaller value of  $C$ .) With this correction for the calorimeter and surroundings, the heat exchange relationship becomes:

heat lost by the hot water = heat gained by the cold water

+ heat gained by the calorimeter and surroundings,

or

$$[(m_{\text{H}})(T_{\text{H}} - T_{\text{F}})(4.184 \text{ J/g}^\circ\text{C})] = [(m_{\text{C}})(T_{\text{F}} - T_{\text{C}})(4.184 \text{ J/g}^\circ\text{C}) + (C)(T_{\text{F}} - T_{\text{C}})]$$

Rearranging, we have

$$\begin{aligned} C &= \frac{(m_{\text{H}})(T_{\text{H}} - T_{\text{F}})(4.184 \text{ J/g}^\circ\text{C}) - (m_{\text{C}})(T_{\text{F}} - T_{\text{C}})(4.184 \text{ J/g}^\circ\text{C})}{(T_{\text{F}} - T_{\text{C}})} \\ &= \left[ \frac{(m_{\text{H}})(T_{\text{H}} - T_{\text{F}}) - (m_{\text{C}})(T_{\text{F}} - T_{\text{C}})}{(T_{\text{F}} - T_{\text{C}})} \right] (4.184 \text{ J/g}^\circ\text{C}). \end{aligned}$$

This heat capacity is most reliable for the temperature range in which it was determined (i.e., for temperature changes that occur just above room temperature in this case). The heat which is absorbed and thereby increases the temperature of the water and the calorimeter can be generated by an exothermic chemical reaction occurring within the calorimeter. For example, if we mix equimolar amounts of an aqueous strong acid (e.g.,  $\text{HCl}_{(\text{aq})}$ ) with an aqueous strong base (e.g.,  $\text{NaOH}_{(\text{aq})}$ ), the neutralization reaction produces heat, which causes the temperature in the calorimeter to increase.

If we mix 50.0 mL of a 2.00 M aqueous HCl solution with 50.0 mL of a 2.00 M aqueous NaOH solution, the neutralization reaction  $\text{HCl}_{(\text{aq})} + \text{NaOH}_{(\text{aq})} \rightarrow \text{H}_2\text{O}_{(\text{aq})} + \text{NaCl}_{(\text{aq})}$  will produce a measurable change in temperature. After complete reaction, the calorimeter will contain a dilute solution of sodium chloride. We can assume that the specific heat is still that of water, namely

4.184 J/g °C, without introducing much error. We can also assume that the density of the dilute salt solution is that of water, 1.00 g/mL. The heat exchange equation in this case is:

$$\begin{aligned} \text{heat given off by the reaction} &= \text{heat gained by the water} \\ &+ \text{heat gained by the calorimeter and surroundings.} \end{aligned}$$

If the calorimeter, the HCl solution, and the NaOH solution all start at the same initial temperature  $T_I$  and warm to a final temperature  $T_F$ , then

$$\begin{aligned} \text{heat given off by the reaction} &= (m_{\text{water}})(T_F - T_I)(4.184 \text{ J/g } ^\circ\text{C}) + (C)(T_F - T_I) \\ &= [(V_{\text{water}} \text{ in mL})(1.00 \text{ g/mL})(4.184 \text{ J/g } ^\circ\text{C}) + (C)](T_F - T_I). \end{aligned}$$

(Note:  $V_{\text{water}} = 100 \text{ mL}$ )

At constant pressure, the heat given off by the reaction is equal to the change in enthalpy ( $\Delta H$ ) for the reaction. Since the reaction involves  $(2.00 \frac{\text{mol}}{\text{L}}) \left( \frac{10^{-3} \text{ L}}{1 \text{ mL}} \right) (50.0 \text{ mL}) = 0.100 \text{ mol}$  of HCl = 0.100 mol of NaOH, we can calculate the change in enthalpy for the acid/base reaction as follows (the minus sign is inserted to indicate that the reaction is exothermic):

$$\Delta H = \left( \frac{-(\text{heat}) \text{ J}}{0.100 \text{ mol}} \right) \left( \frac{1 \text{ kJ}}{10^3 \text{ J}} \right) = \left( \frac{-(\text{heat})}{100} \right) \text{ in kJ/mol.}$$

## 8A Experiment: Determination of Calorimeter Constant

1. Obtain or assemble a calorimeter as shown in Figure 9. The experiment will require two thermometers, one for the calorimeter and one for the heated water.
2. Using a graduated cylinder, measure 50.0 mL of water and pour it into the calorimeter. Measure an additional 50.0 mL of water and pour it into a clean, previously dried beaker.
3. One lab partner should stir the calorimeter contents for at least 5 minutes and then record the temperature inside the calorimeter as  $T_C$ .
4. Meanwhile, the other lab partner should heat (bunsen burner) and stir the water in the beaker until it reaches a temperature of 55-60 °C. After removing the burner, stir and record the exact temperature of the water in the beaker as  $T_H$ .
5. At that point, pour the hot water into the calorimeter, replace the top and stir the contents well while recording the temperature at 15 second intervals. The highest temperature should occur within 2 minutes. Record this maximum temperature as  $T_F$ .
6. Calculate the heat capacity  $C$  of the calorimeter in J/°C using the formula given in the preceding section with  $m_H = m_C = (50.0 \text{ mL})(1.00 \text{ g/mL}) = 50.0 \text{ g}$ .
7. Repeat steps 1 through 6 in order to make a second determination of  $C$ . Reverse the thermometers for the second determination of  $C$ .

First Determination of $C$		Second Determination of $C$	
$T_C =$	°C	$T_C =$	°C
$T_H =$	°C	$T_H =$	°C
Temperature inside calorimeter at 15 s intervals:		Temperature inside calorimeter at 15 s intervals:	
°C	°C	°C	°C
°C	°C	°C	°C
°C	°C	°C	°C
°C	°C	°C	°C
°C	°C	°C	°C
°C	°C	°C	°C
$T_F =$	°C	$T_F =$	°C
$C =$	J/°C	$C =$	J/°C
		Average $C =$	J/°C

## 8B Experiment: Determination of Reaction Enthalpy

1. Dry the calorimeter and reassemble it.
2. Using a clean, dry graduated cylinder, measure 50.0 mL of a 2.0 M aqueous NaOH solution and pour it into the calorimeter. (*Caution:* NaOH is corrosive and, if spilled on the skin, should be washed away immediately with copious amounts of water.)
3. Using a clean, dry graduated cylinder, measure 50.0 mL of a 2.0 M aqueous HCl solution and pour it into a clean, dry beaker. (*Caution:* HCl is corrosive and, if spilled on the skin, should be washed away immediately with copious amounts of water.)
4. Stir the two solutions (the NaOH (aq) in the calorimeter, and the HCl(aq) in the beaker) until they exhibit the same temperature. Record this temperature as  $T_I$ .
5. Pour the HCl solution into the calorimeter, replace the top, and stir while recording the temperature at 15 second intervals.
6. Record the highest temperature reached as  $T_F$ .
7. Use these temperatures and the value of  $C$  determined for the calorimeter in the equations presented in the previous section to calculate the heat evolved and  $\Delta H$  of the reaction. (# of moles = (0.0500 L)(2.0 mol/L) = 0.10 mol.  $\Delta H$  = heat/moles.)
8. Repeat steps 1 through 7 with the thermometers reversed in order to make a second determination of the heat evolved and of  $\Delta H$ . ( $\Delta H$  should equal  $-55.8$  kJ/mol.)

First Determination of $\Delta H$		Second Determination of $\Delta H$	
$T_I =$ °C		$T_I =$ °C	
Temperature inside calorimeter at 15 s intervals:		Temperature inside calorimeter at 15 s intervals:	
°C	°C	°C	°C
°C	°C	°C	°C
°C	°C	°C	°C
°C	°C	°C	°C
°C	°C	°C	°C
°C	°C	°C	°C
$T_F =$ °C		$T_F =$ °C	
heat = J		heat = J	
$\Delta H =$ kJ/mol		$\Delta H =$ kJ/mol	
		Average $\Delta H =$ kJ/mol	
$\% \text{ error} = [( \Delta H  - 55.8)/55.8] \times 100 =$		%	

## 8C Exercises

1. Suppose that one were to mix 30.0 g of aluminum pellets, originally at 97.0 °C, with 100 grams of water, originally at 23.0 °C, in a perfect calorimeter. What will be the equilibrium temperature  $T_F$  in the calorimeter?  $T_F =$  \_\_\_\_\_ °C.
2. Repeat the calculation of Exercise 1, but in the calorimeter you used in lab rather than in a perfect calorimeter.  $T_F =$  \_\_\_\_\_ °C.
3. When 100 g of an unknown metal at 98.0 °C were mixed in a perfect calorimeter with 50.0 g of water at 22.0 °C, the final temperature  $T_F$  was observed to be 26.4 °C. Presuming that the metal is one of those listed in the table of specific heats given above, which one is it?  
Unknown metal = \_\_\_\_\_.

**Report Form 8: Calorimetry,  
Heat of Reaction**

Name \_\_\_\_\_  
Partner \_\_\_\_\_ Section # \_\_\_\_\_

**8A Experiment: Determination of Calorimeter Constant**

First Determination of $C$		Second Determination of $C$	
$T_C =$	°C	$T_C =$	°C
$T_H =$	°C	$T_H =$	°C
Temperature inside calorimeter at 15 s intervals:		Temperature inside calorimeter at 15 s intervals:	
°C	°C	°C	°C
°C	°C	°C	°C
°C	°C	°C	°C
°C	°C	°C	°C
°C	°C	°C	°C
°C	°C	°C	°C
$T_F =$	°C	$T_F =$	°C
$C =$	J/°C	$C =$	J/°C
		Average $C =$	J/°C

**8B Experiment: Determination of Reaction Enthalpy**

First Determination of $\Delta H$		Second Determination of $\Delta H$	
$T_I =$	°C	$T_I =$	°C
Temperature inside calorimeter at 15 s intervals:		Temperature inside calorimeter at 15 s intervals:	
°C	°C	°C	°C
°C	°C	°C	°C
°C	°C	°C	°C
°C	°C	°C	°C
°C	°C	°C	°C
°C	°C	°C	°C
$T_F =$	°C	$T_F =$	°C
heat =	J	heat =	J
$\Delta H =$	kJ/mol	$\Delta H =$	kJ/mol
		Average $\Delta H =$	kJ/mol
		% error = $[( \Delta H  -  55.8 )/55.8] \times 100 =$	%

**8C Exercises**

- $T_F =$  \_\_\_\_\_ °C.
- $T_F =$  \_\_\_\_\_ °C.
- Unknown metal = \_\_\_\_\_ .

## Lab Session 10, Experiment 9: Charles' Law

The purpose of this experiment is to study the changes in the volume of a gas with changes in temperature at constant pressure.

### 9A Experiment

1. Use a thoroughly dried 125 mL Erlenmeyer flask for this experiment. If it is not dry, rinse the flask with a small amount of acetone or ethanol and place it upside-down on a paper towel to dry.
2. Fit the flask with a one-hole rubber stopper inserted with a short piece of dry glass tubing. Add rubber tubing to the end of the glass tube and assemble the apparatus as shown in Figure 10.1, using the 125 mL flask and a 400 mL beaker. Be sure that the stopper fits tightly in the flask, the glass tubing fits tightly in the rubber stopper, and the rubber tubing fits tightly on the glass tubing. (The latter may require a hose clamp or wire winding.) Leave a 1 cm gap between the bottom of the flask and the beaker.

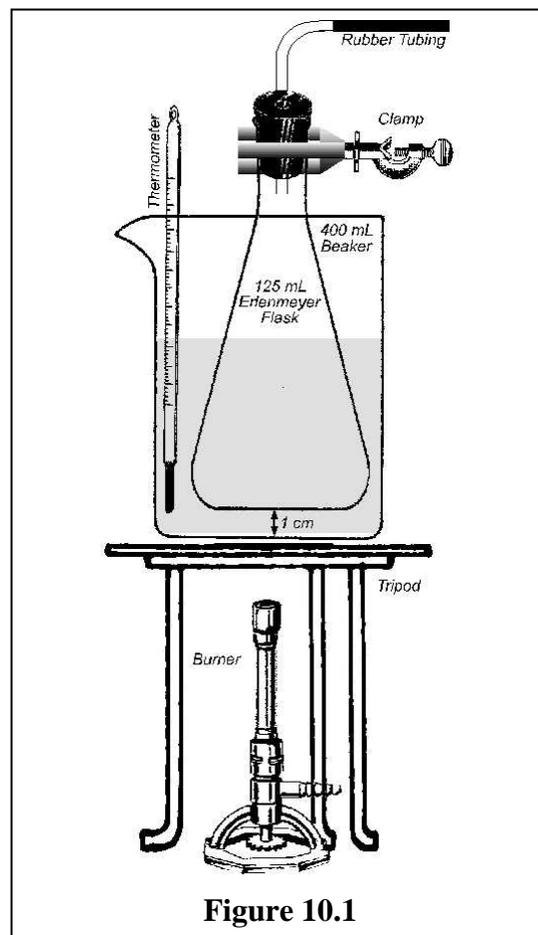


Figure 10.1

3. Pour water into the beaker until as much of the flask is covered as possible.
4. Using a Bunsen burner, heat the water in the beaker until it boils and then continue to heat for 5 minutes. At this point determine the temperature of the water by means of a 110 °C thermometer. Read the thermometer while its bulb is immersed in the water and record the reading as entry (c) in the data table below. Make sure the thermometer does not touch the beaker.
5. After the water in the beaker has been boiling for about 5 minutes, place a screw clamp (or a pinch clamp) on the rubber tubing. While the water is still boiling, close the clamp in order to make the flask airtight. Turn off the Bunsen burner by closing the gas valve at its base, then turn off the gas at the bench gas cock.
6. Loosen the ring stand clamp, remove the flask from the beaker, and immerse it in the water in the stoppered end sink nearest your work area. It should be cooled upside-down in the sink for about 5 minutes, with the stopper and tubing completely immersed in water throughout this period. At the end of this time, loosen the screw clamp while the end of the tube is completely submerged and let water be forced into the flask.
7. Holding the rubber tubing closed with your fingers, remove the flask from the sink, and determine the volume of the water in the flask by pouring it into a 100 mL graduated cylinder. Record this volume as entry (a) in the data table.

8. Determine the temperature of the water in the sink and record it as entry (d) in the data table. Your instructor may opt to prepare ice/water baths in the end sinks, in which case the temperature should be close to 0°C. You should measure it with your thermometer and record it anyway.
9. To determine the volume of gas (air) used in the beginning, it is necessary to accurately determine the volume of the Erlenmeyer flask. *Completely* fill the flask with water and place the rubber stopper (and tubing) in its previous position. Remove the stopper and measure the volume of the water in the flask with a 100 mL graduated cylinder. Record this volume as entry (b) in the data table.

(a) Volume of water forced into the flask	mL	
(b) Initial volume of the air (measured volume of the flask/stopper)	mL	
(c) Initial temperature of the air (temperature of the hot water)	°C	K
(d) Final temperature of the air (temperature of the water in the sink)	°C	K
(e) Final volume of the air [(b)–(a)] (measured volume of the flask minus the volume of water forced into the flask)	mL	

To summarize, a given volume of air was taken at the temperature of boiling water. The air was cooled, causing it to contract. The new volume was determined experimentally.

### 9B Exercise

The accuracy of the experimental determination of the final volume (entry (e) in the data table) can be checked by calculating this volume with Charles' Law. Change the temperature reading into the absolute (Kelvin) scale and enter your data into the following formula:

$$\frac{\text{Initial volume (mL)} \times \text{Final Temperature (K)}}{\text{Initial Temperature (K)}} = \text{Final Volume (mL)}$$

Your calculations:

$$\underline{\hspace{10em}} \times \underline{\hspace{10em}} = \underline{\hspace{10em}}$$

Complete the following table.

(a) What was the experimental final volume?	mL
(b) According to Charles' Law, what should have been the final volume?	mL
(c) What is the difference between the experimental and calculated volumes?	mL
(d) Assuming the calculated volume to be correct, determine the percent error of the experiment.	%

**Report Form 9: Charles' Law**

Name \_\_\_\_\_

Partner \_\_\_\_\_ Section # \_\_\_\_\_

**9A Experiment**

(a) Volume of water forced into the flask	mL	
(b) Initial volume of the air (measured volume of the flask/stopper)	mL	
(c) Initial temperature of the air (temperature of the hot water)	°C	K
(d) Final temperature of the air (temperature of the water in the sink)	°C	K
(e) Final volume of the air [(b)-(a)] (measured volume of the flask minus the volume of water forced into the flask)	mL	

**9B Exercise**

(a) What was the experimental final volume?	mL
(b) According to Charles' Law, what should have been the final volume?	mL
(c) What is the difference between the experimental and calculated volumes?	mL
(d) Assuming the calculated volume to be correct, determine the percent error of the experiment.	%

## Lab Session 11, Experiment 10: Determination of the Molar Mass of Oxygen

The molar mass (sometimes called the gram molar mass, or the molecular weight) of a gaseous compound can be calculated if the following are known: the mass, volume, temperature, and pressure of the gas. In this experiment, the molar mass of oxygen will be determined. Potassium chlorate will be decomposed to produce oxygen. The mass of oxygen generated will equal the difference between the mass of  $\text{KClO}_3$  before decomposition and the mass of the residue after decomposition. The volume of the oxygen will be the volume of water displaced from a bottle initially filled with water. The total gaseous pressure ( $P_{\text{oxygen}} + P_{\text{water}}$ ) will be assumed to be barometric pressure. The temperature of the oxygen will be assumed to be the same as that of the water displaced.

### 10A Experiment

Record all measurements in the data table provided.

1. Fill  $\text{KClO}_3$  into your large test tube to a depth of 2-3 cm.  
Add a small quantity of  $\text{MnO}_2$  and weigh the test tube with contents.

2. Mix well. Mount the test tube as shown in Figure 11.1.

*Note:* All connections must be tight. This includes glass tubes through stoppers, stoppers in vessels, and rubber tubing connections to glass tubes. The latter may require a hose clamp or wire winding.

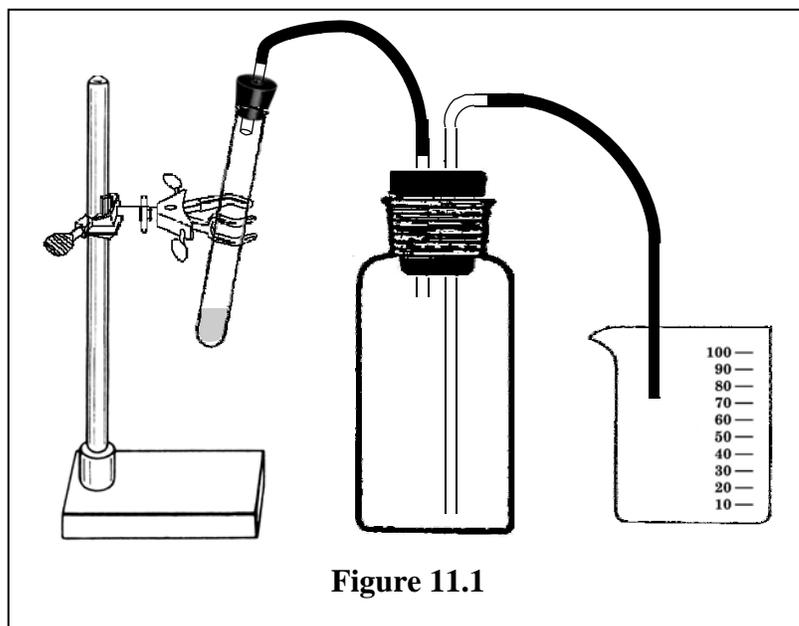


Figure 11.1

3. Before heat is applied to the test tube, the assembly must be checked to make sure that water will not be transferred from the bottle to the beaker by siphoning. Proceed with this check as follows. Remove the test tube from the assembly by removing the rubber stopper from it. Insert the glass tube extending from the stopper into the opening of a rubber bulb that you get from the stockroom. Squeeze the rubber bulb gently to fill the delivery tube between the bottle and the beaker with water. While the delivery tube is filled, close a pinch-clamp over the connecting rubber tubing between the test tube and the bottle. Empty the beaker; a small quantity of water will be transferred to the beaker when filling the delivery tube. Put the large test tube back in place. Remove the pinch-clamp and observe for siphoning. If water flows into the beaker, there is a leak around the stopper that must be corrected before proceeding. If there is no evidence of siphoning, proceed to the next step.

- Apply heat from your burner, slowly at first, to begin the decomposition of the chlorate. After a little more than half the water in the bottle has been transferred to the beaker, discontinue the heating.
- Allow the test tube to cool to room temperature, then place a pinch-clamp over the rubber tube. Remove and weigh the test tube. Measure the volume and temperature of the water in the beaker, which is equivalent to the volume and temperature of oxygen generated.
- Obtain the vapor pressure of water from the table below and calculate the pressure of dry oxygen. You will have to convert the vapor pressure of water from Torr to atm. [1 atm = 760 Torr]

### Vapor Pressure of Water

°C	Torr	°C	Torr	°C	Torr	°C	Torr	°C	Torr	°C	Torr
18.0	15.477	20.0	17.535	22.0	19.827	24.0	22.377	26.0	25.209	28.0	28.349
18.2	15.673	20.2	17.753	22.2	20.070	24.2	22.648	26.2	25.509	28.2	28.680
18.4	15.871	20.4	17.974	22.4	20.316	24.4	22.922	26.4	25.812	28.4	29.015
18.6	16.071	20.6	18.197	22.6	20.565	24.6	23.198	26.6	26.117	28.6	29.354
18.8	16.272	20.8	18.422	22.8	20.815	24.8	23.476	26.8	26.426	28.8	29.697
19.0	16.477	21.0	18.650	23.0	21.068	25.0	23.756	27.0	26.739	29.0	30.043
19.2	16.685	21.2	18.880	23.2	21.324	25.2	24.039	27.2	27.055	29.2	30.392
19.4	16.894	21.4	19.113	23.4	21.583	25.4	24.326	27.4	27.374	29.4	30.745
19.6	17.105	21.6	19.349	23.6	21.845	25.6	24.617	27.6	27.696	29.6	31.102
19.8	17.319	21.8	19.587	23.8	22.110	25.8	24.912	27.8	28.021	29.8	31.461

- Use the ideal gas law to calculate the gram-molar mass of oxygen. See the review of the ideal gas law in the table below.

Review of the Ideal Gas Law	
$PV = nRT$	$M = (\text{mass} \times RT) \div PV$
$PV = (\text{mass} \div M) \times RT$	where $M$ is g/mol of gas
$PVM = \text{mass} \times RT$	$R = 0.08206 \text{ L atm mol}^{-1} \text{ K}^{-1}$

(a) Mass of test tube and contents before heating	g	
(b) Mass of test tube and contents after heating	g	
(c) Mass of oxygen in flask [(a)–(b)]	g	
(d) Volume of water transferred = Volume of O <sub>2</sub>	mL	L
(e) Temperature of water = Temperature of O <sub>2</sub>	°C	K
(f) Barometric pressure = P <sub>atm</sub>	Torr	atm
(g) Vapor pressure of water	Torr	atm
(h) Pressure of dry oxygen [(f)–(g)]	Torr	atm
(i) Molar mass of oxygen	g/mol	
(j) % error = $[(i) - 32.00] / 32.00 \times 100$	%	

## 10B Exercise

1. Why is it necessary to allow the test tube to cool before it is removed for weighing?

---

2. Why must the water delivery tube extend nearly to the bottom of the flask?

---

3. When water flow ceases, what can be assumed as the relationship between barometric pressure ( $P_{\text{atm}}$ ) and the pressure inside the flask ( $P_{\text{oxygen}} + P_{\text{water}}$ )?

---

4. Is it necessary to decompose all the chlorate?

---

**Report Form 10: Determination  
of the Molar Mass of Oxygen**

Name \_\_\_\_\_  
Partner \_\_\_\_\_ Section # \_\_\_\_\_

**10A Experiment**

(a) Mass of test tube and contents before heating	g	
(b) Mass of test tube and contents after heating	g	
(c) Mass of oxygen in flask [(a)-(b)]	g	
(d) Volume of water transferred = Volume of O <sub>2</sub>	mL	L
(e) Temperature of water = Temperature of O <sub>2</sub>	°C	K
(f) Barometric pressure = P <sub>atm</sub>	Torr	atm
(g) Vapor pressure of water	Torr	atm
(h) Pressure of dry oxygen [(f)-(g)]	Torr	atm
(i) Molar mass of oxygen	g/mol	
(j) % error = $[(i) - 32.00] / 32.00 \times 100$	%	

**10B Exercise**

1. Why is it necessary to allow the test tube to cool before it is removed for weighing?

\_\_\_\_\_

2. Why must the glass water delivery tube extend nearly to the bottom of the bottle?

\_\_\_\_\_

3. When water flow ceases, what can be assumed as the relationship between barometric pressure (P<sub>atm</sub>) and the pressure inside the flask (P<sub>oxygen</sub> + P<sub>water</sub>)?

\_\_\_\_\_

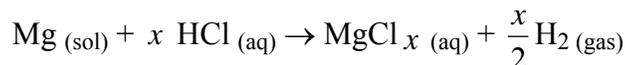
4. Is it necessary to decompose all the chlorate?

\_\_\_\_\_

## Lab Session 12, Experiment 11: Valence, the Combining Capacity of Elements and Molecules

One molar mass of an element or compound that produces or reacts with 1.008 grams of hydrogen has a valence of I. It follows that the same quantity producing or reacting with 2.016 grams of hydrogen is valence II, and producing or reacting with 3.024 grams of hydrogen is valence III, etc.

In this experiment, the magnesium metal will be reacted with concentrated hydrochloric acid to produce hydrogen gas according to the following chemical equation:



The hydrogen gas produced will be collected, its volume measured, and the mass in grams calculated using the ideal gas law:

$$PV = nRT$$

When using this equation, the units of the gas constant (R) must be consistent with the units used for the other quantities in the equation. The Handbook of Chemistry and Physics lists the value of R as

$$R = 0.08206 \text{ L} \times \text{atm} \times \text{mol}^{-1} \times \text{K}^{-1}$$

Thus, pressure (P) must be expressed in atmospheres, volume (V) in liters, and temperature (T) in Kelvin.

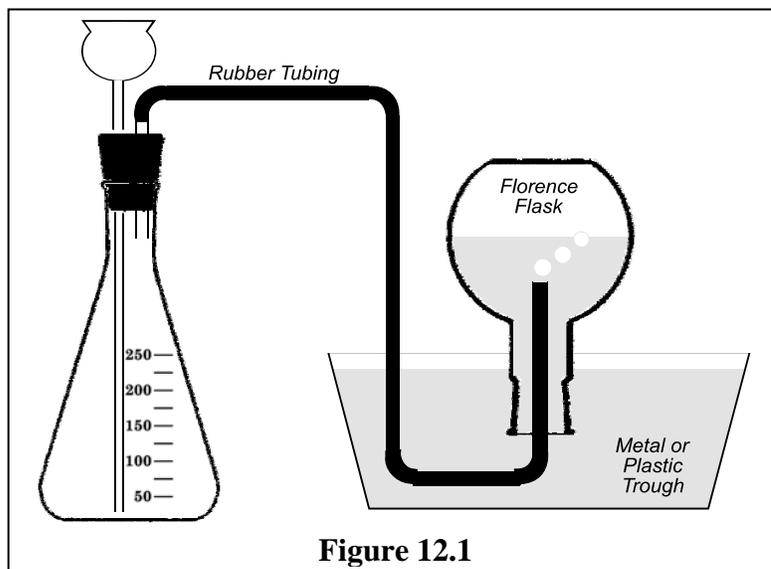
### 11A Experiment

A sample of magnesium metal with an approximate mass of 0.24 grams will be reacted with excess hydrochloric acid. The hydrogen gas produced will be collected, its volume measured, and finally its mass in grams will be calculated.

*Chemicals Required:* 6M hydrochloric acid                      Magnesium metal,  
*Note: 12M HCl<sub>(aq)</sub> must be diluted.*                      ribbon or granules

*Apparatus Required:* 500 mL Florence flask                      125 mL Erlenmeyer flask  
Glass tubing                      Metal or plastic trough  
Watch glass                      100 mL graduated cylinder  
Rubber tubing                      Thermometer

The apparatus should be set up as shown in Figure 12.1. Make sure the bottom of the thistle tube almost touches the bottom of the Erlenmeyer flask. You may be instructed to use the end sink rather than a metal or plastic trough. Be sure that all fittings are tight. This includes the fittings of the rubber stopper in the Erlenmeyer flask, the glass tube through the stopper, and the rubber tubing to the glass tube. (The latter may require a hose clamp or wire winding.)



**Figure 12.1**

1. Record the exact mass of the magnesium as entry (a) in the data table.
2. Remove the two-hole stopper from the Erlenmeyer flask and add enough water to cover the bottom of the thistle tube. If the magnesium is ribbon, form it into a ball or tight coil, and place it in the flask. Reinsert the rubber stopper tightly.
3. Add 10 mL of 6M hydrochloric acid solution by pouring it into the thistle tube. Put a rubber stopper in the top of the thistle tube to avoid back-splashing acid or escape of hydrogen gas.
4. After the reaction is complete, allow a few minutes for the flask to cool. Be sure to keep the end of the rubber tubing above the water level in the Florence flask. The gas collected ( $H_2$  and water vapor) must be at the same pressure as the barometric pressure. To equalize pressures, remove the gas delivery tube and raise or lower the Florence flask so that the water levels in the trough (or end sink) and in the Florence flask are equal. (You may have to tilt the flask to equalize levels. Be careful that the mouth of the Florence flask stays below the water level in the trough.) Cover the Florence flask opening with a watch glass, invert the Florence flask and place it on the desktop.
5. Using a graduated cylinder, measure the volume of water that must be added to the Florence flask to fill it. It will be assumed that this is the volume of the gas collected. It should be recorded as item (e) in the data table.
6. Measure the temperature of the water in the trough with your thermometer and record it as item (b) below. It will be assumed that this is the temperature of the collected gas.
7. Record the barometric pressure as item (c) below. This is the total pressure of the hydrogen and water vapor.

(a) Mass of magnesium	g
(b) Temperature of water in trough	$^{\circ}C$
(c) Barometric pressure	Torr
(d) Vapor pressure of water at the temperature recorded in (b)	Torr
(e) Volume of gas collected = Volume of $H_2O$ added	mL

## 11B Exercise: Converting Data to the Correct Units

1. Calculate the Pressure P = \_\_\_\_\_ atm

$$\text{Pressure [atm]} = \frac{\text{Barometric Pressure} - \text{Vapor Pressure of H}_2\text{O}}{760 \text{ Torr/atm}}$$

2. Calculate the Volume V = \_\_\_\_\_ L

$$\text{Volume in L} = \text{Volume in mL} \div 1000$$

3. Calculate the Temperature T = \_\_\_\_\_ K

$$\text{Absolute temperature in Kelvins} = \text{ }^\circ\text{C} + 273.15$$

4. Calculate the mass of hydrogen Mass = \_\_\_\_\_ g

Calculating the grams of hydrogen produced using the ideal gas law:

$$PV = \text{Mass of hydrogen produced} \div \text{molar mass of hydrogen} \times 0.08206 \text{ (R)} \times T$$

Rearranged:

$$\text{Mass in grams of hydrogen produced} = \frac{P \times V \times \text{Molar Mass of H}_2}{0.08206 \text{ (R)} \times T}$$

5. From the generalized balanced equation:  $\text{Mg} + x \text{HCl} \rightarrow \text{MgCl}_x + \frac{1}{2} x \text{H}_2$ , we can calculate how many grams of hydrogen should be produced from the number of grams of magnesium reacted, as follows:

$$\text{g}_{\text{hydrogen}} = (\text{g}_{\text{Mg}}) \left( \frac{1 \text{ mole Mg}}{24.305 \text{ g Mg}} \right) \left( \frac{(x/2) \text{ moles H}_2}{1 \text{ mole Mg}} \right) \left( \frac{2.016 \text{ g H}_2}{1 \text{ mole H}_2} \right) = \frac{x}{24.11}$$

Rearrangement yields the following ratio.

$$\frac{\text{g}_{\text{hydrogen}}}{\text{g}_{\text{Mg}}} = \frac{x}{24.11}. \text{ Thus, } x = \left( \frac{\text{g}_{\text{hydrogen}}}{\text{g}_{\text{Mg}}} \right) (24.11). \quad x = \left( \frac{\text{_____ g}_{\text{H}_2}}{\text{_____ g}_{\text{Mg}}} \right) (24.11) = \text{_____}.$$

$$\% \text{ error} = \frac{|x - 2|}{2} \times 100 = \text{_____} \%$$

Give a balanced equation for the reaction of magnesium with hydrochloric acid:

---

6. Verification that HCl is the excess reagent by completing the following calculations:

$$\frac{\text{g Mg}}{\text{24.3 g Mg}} \left| \frac{1 \text{ mole Mg}}{\text{24.3 g Mg}} \right| \frac{2 \text{ mole HCl}}{1 \text{ mole Mg}} = \text{_____ moles HCL needed}$$

$$\frac{10 \text{ mL}}{1 \text{ mL}} \left| \frac{10^{-3} \text{ L}}{1 \text{ mL}} \right| \frac{6.0 \text{ moles HCl}}{\text{L}} = \text{_____ moles HCL added}$$

$$\text{_____ moles HCl added} - \text{_____ moles HCl needed} = \text{_____ moles of HCl unreacted}$$

### 11C Alternate Calculations

$$1. \text{ Moles of H}_2 = \frac{PV}{RT} = \frac{(\text{_____})(\text{_____})}{(\text{_____})(\text{_____})} = \text{_____ mol.}$$

$$2. \text{ Moles of Mg} = \frac{g_{\text{Mg}}}{M_{\text{Mg}}} = \frac{(\text{_____})}{(\text{_____})} = \text{_____ mol.}$$

$$3. \text{ Mole ratio of H}_2 \text{ to Mg} = \frac{\text{Moles of H}_2}{\text{Moles of Mg}} = \frac{(\text{_____})}{(\text{_____})} = \text{_____}.$$

$$4. x_{\text{exp}} = 2 \times \text{ratio} = 2 \times (\text{_____}) = \text{_____}.$$

$$5. \% \text{ error} = \frac{x_{\text{exp}} - 2.00}{2.00} = \frac{(\text{_____}) - 2.00}{2.00} = \text{_____} \%$$

### 11D Exercise: Nomenclature

Give the formula of each of the following.

calcium sulfide	Iron (II) chloride
sodium sulfate	nickel (II) nitrate
calcium phosphate	magnesium hydroxide
lithium oxide	Tin (IV) bromide

**Report Form 11: The Combining Capacity of Elements and Molecules**

Name \_\_\_\_\_  
 Partner \_\_\_\_\_ Section # \_\_\_\_\_

**11A Experiment**

(a) Mass of magnesium	g
(b) Temperature of water in trough	° C
(c) Barometric pressure	Torr
(d) Vapor pressure of water at the temperature recorded in (b)	Torr
(e) Volume of gas collected = Volume of H <sub>2</sub> O added	mL

**11B Exercise: Converting Data to the Correct Units**

- Calculate the Pressure P = \_\_\_\_\_ atm
- Calculate the Volume V = \_\_\_\_\_ L
- Calculate the Temperature T = \_\_\_\_\_ K
- Calculate the Mass of hydrogen Mass = \_\_\_\_\_ g
- $x = \left( \frac{\text{_____ g}_{\text{H}_2}}{\text{_____ g}_{\text{Mg}}} \right) (24.11) = \text{_____} . \quad \% \text{ error} = \text{_____} .$

Give a balanced equation for the reaction of magnesium with hydrochloric acid:

\_\_\_\_\_

- Verification that HCl is the excess reagent by completing the following calculations:

\_\_\_\_\_ moles HCl added – \_\_\_\_\_ moles HCl needed = \_\_\_\_\_ moles of HCl unreacted

**11C Alternate Calculations**

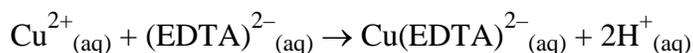
- Moles of H<sub>2</sub> = \_\_\_\_\_ mol.
- Moles of Mg = \_\_\_\_\_ mol.
- Mole ratio of H<sub>2</sub> to Mg = \_\_\_\_\_.
- $x_{\text{exp}} = 2 \times \text{ratio} = \text{_____} .$
- % error = \_\_\_\_\_ %.

**11D Exercise: Nomenclature**

calcium sulfide	Iron (II) chloride
sodium sulfate	nickel (II) nitrate
calcium phosphate	magnesium hydroxide
lithium oxide	Tin (IV) bromide

## Lab Session 13, Experiment 12: Copper Analysis by Complexometric Titration

A quantitative analysis of copper in a soluble copper salt will be performed by complexometric titration. The complexing agent will be ethylenediaminetetraacetic acid (EDTA) in the form of its disodium dihydrate salt ( $\text{Na}_2\text{C}_{10}\text{H}_{18}\text{N}_2\text{O}_{10}$ ), with a molar mass of  $372.24 \text{ g mol}^{-1}$ . Since EDTA forms complexes with many metal ions, this particular method can only be used in the absence of such ions as  $\text{Ca}^{2+}$ ,  $\text{Ni}^{2+}$ , etc. The reaction of complexation is:



The stoichiometry is one metal cation to one EDTA anion. However, for  $\text{Cu}^{2+}$  (since it has lost 2 electrons), the equivalent mass is  $63.546/2 = 31.773 \text{ g eq}^{-1}$ , and since  $(\text{EDTA})^{2-}$  is a dianion its equivalent mass is  $372.24/2 = 186.12 \text{ g eq}^{-1}$  (for the disodium dihydrate salt). The equation above represents two equivalents reacting with two equivalents. The complex dianion is formed with the release of two moles of  $\text{H}^+$  from EDTA, with the indicator being released from the copper ion.

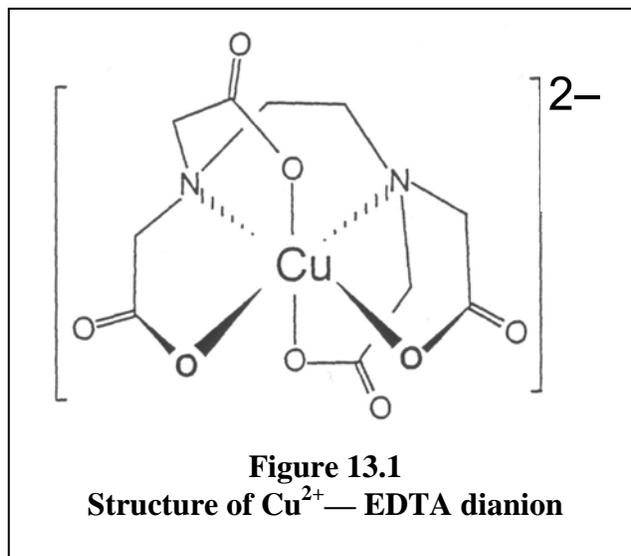
The complex dianion has the structure shown in Figure 13.1. Note that the anion completely surrounds the cation, forming six coordinate covalent bonds to copper and a very stable complex. The bonding to the copper ion is nearly octahedral.

The indicator used for the titration is called murexide. This indicator is highly colored and will complex with the copper ion to give a different colored species. During the titration, the  $\text{EDTA}^{2-}$  forms a more stable complex and frees the indicator, which then displays its original color. The appearance of the free indicator means that all metal ions have been complexed by  $\text{EDTA}^{2-}$ , which signals the end point. At the end point, the following equation applies:

$$\begin{aligned} N_{\text{EDTA}}V_{\text{EDTA}} &= N_{\text{Cu(II)}}V_{\text{Cu(II)}} = \# \text{eq Cu(II)}, \text{ if } V \text{ is given in L} \\ &= \# \text{meq Cu(II)}, \text{ if } V \text{ is given in mL} \end{aligned}$$

The mass of Cu equals  $(\# \text{eq Cu(II)}) \times (\text{equivalent mass of Cu(II)})$ , and

$$\frac{\text{mass Cu(II)}}{\text{mass Cu(II) salt}} \times 100 = \% \text{ Cu}$$



## 12A Experiment

1. Rinse your burette and fill it with standardized  $\text{Na}_2\text{EDTA}\cdot 2\text{H}_2\text{O}$  solution (7.445 g  $\text{Na}_2\text{EDTA}\cdot 2\text{H}_2\text{O}$  per liter of water).
2. Weigh accurately three approximately 0.1 g samples of the copper salt.
3. Dissolve each sample in 50 mL of de-ionized water.
4. Add *exactly* the same amount of indicator to each sample, three drops to start off with. If the indicator solution is not strong enough, add more but always the same for all samples. (The indicator's concentration should be 100 mg/100 mL  $\text{H}_2\text{O}$ )
5. Titrate each sample with the standardized EDTA. The light yellow solution turns green near the end point, then suddenly purplish blue at the end point. This end point is fairly hard to see, so put a white sheet of paper under your beaker and watch carefully. The distinctly purplish hue, due to free murexide, is the key to observing the end point.
6. For each titration, calculate the number of equivalents or (milliequivalents) of Cu(II) found.
7. For each titrated sample, calculate the mass of copper in that sample.
8. For each titration, calculate the % copper content in the sample, then average them.

	Sample 1	Sample 2	Sample 3	
(a) Normality of EDTA				
(b) grams of Cu(II) sample				
(c) mL of EDTA solution				
(d) eq (or meq) of Cu(II)				<b>Average % copper content</b>
(e) mass of copper				
(f) % copper content				

## 12B Exercise

In the experiment on hydrates, we found that copper sulfate was a hydrate which contained 36.1% by mass water. Since the only other component is the sulfate ion,  $\text{SO}_4^{2-}$ , we can now determine the complete formula of copper sulfate.

### **NOTE Solutions preparation:**

Either weigh the EDTA analytically or standardize the solution. Label the bottles with the normality of EDTA. Use deionized water. About 1 liter will be used by 20 students, 10 groups. Make these solutions up fresh, including the murexide solution.

**Report Form 12: Copper Analysis  
by Complexometric Titration**

Name \_\_\_\_\_

Partner \_\_\_\_\_ Section # \_\_\_\_\_

**12A Experiment**

	<b>Sample 1</b>	<b>Sample 2</b>	<b>Sample 3</b>	
(g) Normality of EDTA				<b>Average % copper content</b>
(h) grams of Cu(II) sample				
(i) mL of EDTA solution				
(j) eq (or meq) of Cu(II)				
(k) mass of copper				
(l) % copper content				

**12B Exercise**

Complete formula of copper sulfate. \_\_\_\_\_