School of Science & Engineering

LABORATORY MANUAL FOR GENERAL CHEMISTRY I

CHE1401

Name: _____

Section: _____

LABORATORY MANUAL FOR
GENERAL CHEMISTRY I

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TABLE OF CONTENTS

PREFACE

Most students who take freshman chemistry are not planning for a career in this discipline. As a result, the introductory chemistry course usually serves several functions at various levels. It begins the training process for those who seek to become chemists. It introduces non science students to chemistry as an important, useful, and, we hope, interesting and rewarding part of their general education. This manual has been prepared with these objectives in mind.

To assist the students, we have included review questions to be answered before the experiments are begun. These are designed both to help the student understand the experiment and as an incentive to read the experiment in advance. An effort has been made to minimize the cost of the experiments. We have at the same time striven for a broad representation of the essential principles while keeping in mind that many students gain no other exposure to analytical techniques. Consequently, balances, pH meters, and spectrophotometers are used in some of the experiments. A list of necessary materials is given at the beginning of each experiment.

Each of the experiments contains a detachable report sheet at the end of each experiment.

To the Student

You are about to engage in what for most of you will be a unique experience. You are going to collect experimental data on your own and use your reasoning powers to draw logical conclusions about the meaning of these data. Your laboratory periods are short, and in most instances, there will not be enough time to come to the laboratory unaware of what you are to do, collect your experimental data, make conclusions and/or calculations regarding them, clean up, and hand in your results. Thus, you should read the experimental procedure in advance so that you can work in the lab most efficiently.

After you've read through the experiment, try to answer the review questions we've included at the end of each experiment. These questions will help you to understand the experiment in advance.

Some of your experiments will also contain an element of danger. For this and other reasons, your lab instructor is present to assist you. He is your friend. Treat him well and above all don't be afraid to ask him questions. Within reason, he will be glad to help you.

Chemistry is an experimental science. The knowledge that has been accumulated through previous experiments provides the basis for today's chemistry courses. The information now being gathered will form the basis of future courses. There are basically two types of experiments that chemists conduct:

1   Qualitative – to determine the nature of processes, which are often unanticipated and sometimes unpredictable.
2   Quantitative- to determine the amount of a measurable change in mass, volume, or temperature, for example, including the time rate of change on processes for which the qualitative data are already known.

It is much easier to appreciate and comprehend the science of Chemistry, if you actually participate in experimentation. Although there are many descriptions of the scientific method, the reasoning process involved is difficult to appreciate without performing experiments. Invariably there are experimental difficulties encountered in the laboratory that require care and patience to overcome. There are four objectives for you, the student, in the laboratory:

1   To develop the skills necessary to obtain and evaluate a reliable original result.
2   To record your results for future use.
3   To be able to draw conclusions regarding your results (with the aid of some coaching and reading in the beginning).
4   To learn to communicate your results critically and knowledgeably.

By attentively reading over the experiments in advance, and by carefully following directions and working safely in the laboratory, you will be able to accomplish all these objectives.

Best wishes for an error-free and accident-free term!

SAFETY IN THE LABORATORY

Safety in the laboratory must be emphasized. The compounds you will work with do have some hazards associated with them. Therefore, it is important to follow the safety rules outlined in this lab manual. You should assume that all compounds encountered in the laboratory are toxic and handle them accordingly. Safety goggles for eye protection are recommended and lab coats are to be worn by all students at all times when entering the laboratory. Many chemicals, common in chemical laboratories, will make holes in clothing. Always wash your hands thoroughly when leaving the laboratory. The location and use of the safety equipment in laboratory were already discussed in CHE1401 and will be reminded by your instructor the first day the laboratory class meets. You should become familiar with the proper use of the safety shower, eye-wash fountain, fire blanket and fire extinguisher.

Report any accidents which occur immediately to the laboratory supervisor.

Safety rules to be strictly followed by all students:
1. Wear goggles when required.
2. Do not touch chemicals with your hands. Spatulas will be provided for handling solid materials.
3. Do not eat or drink in the laboratory.
4. Do not taste any chemical.
5. Do not smell any chemicals directly. Use your fingers to waft the odor to your nose.
6. Do not pipet solutions by mouth. Rubber pipet bulbs are provided at each lab station.
7. Do not put flammable liquids near an open flame.
8. When heating a test tube, make certain that the open end of the tube is directed away from the students.
9. When finished with your Bunsen Burner for a given portion of an experiment, turn it off.
10. Do not sit on the lab benches.
11. Do not engage in games in the laboratory. Failure to follow this rule will result in immediate dismissal from the lab and subsequent conduct action.
12. Do not pour any chemicals into a sink without authorization from the instructor.
13. Notify your instructor if a mercury spill should occur.
14. All broken glassware should be cleaned up immediately. The instructor should be notified of all breakage, especially if a thermometer is involved.
15. Do all reactions involving malodorous, noxious or dangerous chemicals in a fume hood.
16. If a chemical gets on your skin, immediately wash the affected area with large quantities of water. The instructor should be notified; no matter how insignificant the incident might seem.
17. When pouring one liquid into another, do so slowly and cautiously. To dilute an acid, pour the acid into the water; never pour water into an acid.
18. No student shall be permitted to work alone in the lab, you should be supervised by a laboratory instructor (or the lab technician during make up sessions).
19. Exercise good housekeeping practices in the laboratory. Be sure that the lab benches remain free of disorder during the experiment. In the event of a spill, clean the area immediately and be sure to use a wet sponge to wipe off the work station at the end of the lab session.
20. Know what you have to do before entering the lab. Read the experiment carefully before coming to the laboratory.

For more information, a booklet titled "Student's Chemistry Laboratory Safety Manual" will be provided to you in your first lab session. Please get acquainted with it.

Be cautious and think about what you are doing!

Safety rules

The laboratory can be but is not necessarily a dangerous place. When intelligent precautions and a proper understanding of techniques are employed, the laboratory is no more dangerous than any other classroom. Most of the precautions are just common-sense practices. These include the following:

1. Wear approved eye protection when required while in the laboratory. Your safety eye protection may be slightly different from that shown, but it must include shatterproof lenses and side shields to provide protection from splashes.

**Typical eyewash**   **Approved eye protections**

The laboratory has an eyewash fountain available for your use. In the event that a chemical splashes near your eyes, you should use the fountain BEFORE THE MATERIAL RUNS BEHIND YOUR EYEGLASSES AND INTO YOUR EYES. The eyewash has a "panic bar," which enables its easy activation in an emergency.

2. Eating, drinking, and smoking are strictly prohibited in the laboratory at all times

3. Know where to find and how to use safety and first-aid equipment.

4. Consider all chemicals to be hazardous unless you are instructed otherwise. **Dispose of chemicals as instructed by your instructor.** Follow the explicit instructions given in the experiments.

5 If chemicals come into contact with your skin or eyes, wash immediately with copious amounts of water and then consult your laboratory instructor.

6. Wear shoes at all times. "Baboosh" shoes are not allowed in the laboratory.

6 Never taste anything. Never directly smell the source of any vapor or gas; instead, by means of your cupped hand, bring a small sample to your nose (see figure below). Chemicals are not to be used to obtain a "high" or clear your sinuses.
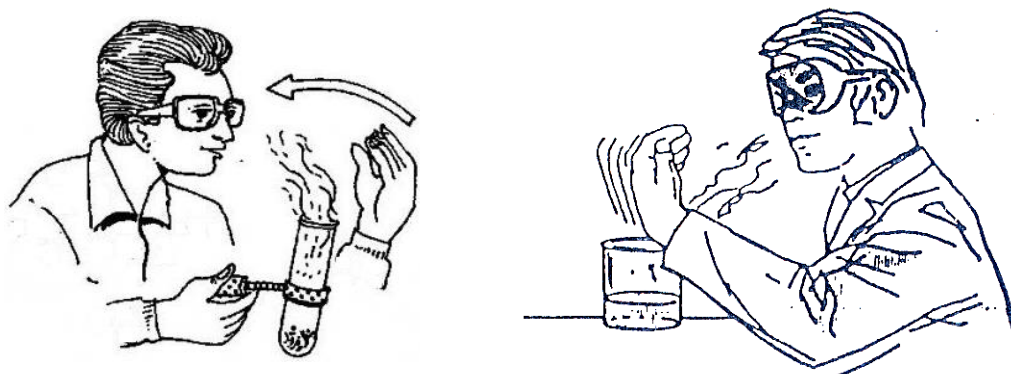
Wafting vapors towards one's nose

8. Perform in the hood any reactions involving skin-irritating or dangerous chemicals and/or ill-smelling chemicals. A typical fume exhaust hood is shown below.
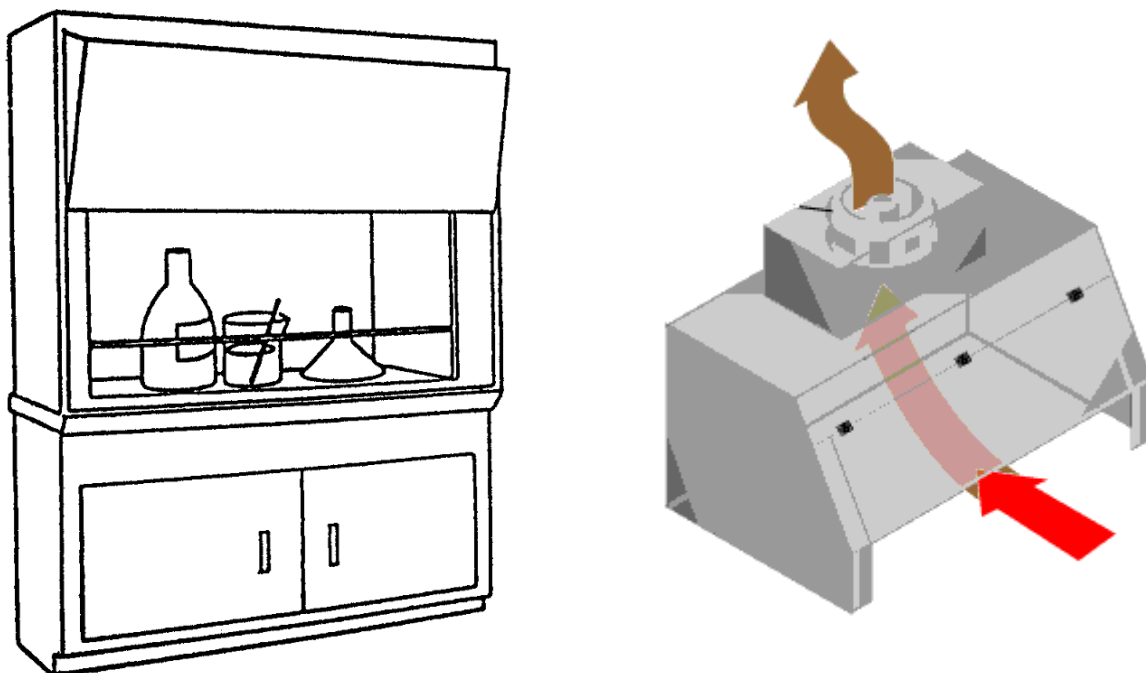
Fume hood found in the laboratory

Exhaust hoods have fans to exhaust fumes out of the hood and away from the user. The hood should be used when noxious, hazardous, and flammable materials are being studied. It also has a shatterproof glass window, which may be used as a shield to protect you from minor explosions. Reagents that evolve toxic fumes are stored in the hood. Return these reagents to the hood after their use.

9. Never point a test tube that you are heating at yourself or your neighbour. It may erupt like a geyser.
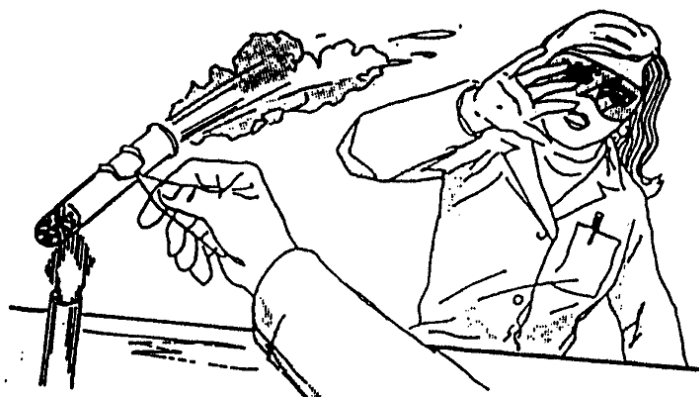
Beware of spattering

10. Do not perform any unauthorised experiments.
11. Clean up all broken glassware immediately.
12. Always pour acids into water, not water into acid, because the heat of solution will cause the water to boil and the acid to spatter.
13. Avoid rubbing your eyes unless you know that your hands are clean.
14. NOTIFY THE INSTRUCTOR IMMEDIATELY IN CASE OF AN ACCIDENT
15. Many common reagents, for example, alcohols, acetone, and especially ether, are highly flammable. Do not use them anywhere near open flames.
16. Observe all special precautions mentioned in experiments.
17. Learn the location of fire protection devices. In the unlikely event that a large chemical fire occurs, a powder extinguisher and a CO_2 extinguisher are available in the lab.

   In order to activate the extinguisher, you must pull the metal safety ring from the handle and then depress the handle. Direct the output of the extinguisher at the base of the flames. The carbon dioxide smothers the flames and cools the flammable material

Powder and CO_2 extinguishers

quickly. If you use the fire extinguisher, be sure to return the extinguisher in at the stockroom so that it can be refilled immediately. If the carbon dioxide extinguisher does not extinguish the fire, evacuate the laboratory immediately and call the security. One of the most frightening and potentially most serious accidents is the ignition of one's clothing. Therefore, certain types of clothing are hazardous in the laboratory and must not be worn. Since sleeves are most likely to come closest to flames, ANY CLOTHING THAT HAS BULKY OR LOOSE SLEEVES SHOULD NOT BE WORN IN THE LABORATORY. Ideally, students should wear laboratory coats with tightly fitting sleeves. Long hair also presents a hazard and must be tied back. If a student's clothing or hair catches fire

his or her neighbours should take prompt action to prevent severe burns. Most laboratories have a water shower for such emergencies. A typical laboratory emergency water shower has the following appearance.

In case someone's clothing or hair is on fire, immediately lead the person to the shower and pull the metal ring. Safety showers generally dump 151 to 190 litres of water, which should extinguish the flames. These showers cannot be shut off once the metal ring has been pulled. Therefore, the shower cannot be demonstrated. (Showers are checked for proper operation on a regular basis, however.)

18. Whenever possible use hot plates instead of Bunsen burners.

A safety shower

COMMON LABORATORY EQUIPMENT

Bunsen burner

Beaker

Erlenmeyer flask

Liquid funnel

Burette

Graduated pipet

Volumetric pipet

Pipet pump

Graduated cylinder

Medicine dropper

Test tube rack

Test tube

Test tube holder

Crucible and cover

Evaporating dish

Watch glass

Crucible tongs

Stirring rods

Spatula

Weighing paper

Wash bottle

Utility clamp

Thermometer clamp

Ring stand

Double burette clamp

Thermometrer

Safety goggles

Safety glasses

Centrifuge

Stirring hot plate

Balance

Vortex mixer

EXPERIMENT 1

Basic laboratory techniques

OBJECTIVE
To learn the use of common, simple laboratory equipment.

Relates to chapter 1 of "Chemistry the Central Science, 12th Ed.".

APPARATUS AND CHEMICALS

Balance	Iron ring and ring stand
150-mL beaker	Bunsen burner and hose
50- or 100-mL graduated cylinder	Clamp
10-mL pipet	Rubber bulb
25-mL Erlenmeyer flask	Thermometer
125-mL Erlenmeyer flask	

INTRODUCTION
Chemistry is an experimental science. It depends upon careful observation and the use of good laboratory techniques. In this experiment you will become familiar with some basic operations that will help you throughout this course. Your success as well as your safety in future experiments will depend upon your mastering these fundamental operations.

Because every measurement made in the laboratory is really an approximation, it is important that the numbers you record reflect the accuracy of the device you use to make the measurement. Our system of weights and measures, the metric system, was originally based mainly upon fundamental properties of one of the world's most abundant substances, water. The system is summarized in **Table 1.1**. Conversions within the metric system are quite simple once you have committed to memory the meaning of the pre-fixes given in **Table 1.2**. Recently, scientists have started to use a briefer version of the metric system of units in which the basic units for length, mass, and time are the meter, the kilogram, and the second. This system of units, known as the International System of Units, is commonly referred to as the SI system and is preferred in scientific work. A comparison of some common SI, metric, and English units is presented in **Table 1.3**. Conversions within the metric system are quite easy if you remember the definitions for the prefixes and use dimensional analysis in problem solving.

Table 1.1 Units of Measurement in the Metric System

Measurement	Unit and definition
Mass or weight	Gram (g) = weight of 1 cubic centimeter (cm^3) of water at 4°C and 760 mm Hg Mass = quantity of material Weight = mass x gravitational force
Length	Meter (m) =100 cm =1000 millimeters (mm) =39.37 in.
Volume	Liter (L) =volume of 1 kilogram (kg) of H_2O at 4° C
Temperature	°C, measures heat intensity: $$^oC = \frac{5}{9}\left(^oF - 32\right) \text{ or } ^oF = \left(\frac{9}{5} \times ^oC\right) + 32$$
Heat	1 calorie (cal), amount of heat required to raise 1 g of water 1°C 1 cal = 4.184 joules (J)
Density	d, usually g/ml, for liquids and g/L for gases: d = mass/unit volume
Specific gravity	Sp gr, dimensionless Sp gr = density of a substance/density of a reference substance

The quantities presented in **Table1.1** are measured with the aid of various pieces of apparatus. A brief description of some measuring devices follows.

Table 1.2 The Meaning of prefixes in the metric system

Prefix	Meaning (power of 10)	Abbreviation
femto-	10^{-15}	f
pico-	10^{-12}	p
nano-	10^{-9}	n
micro-	10^{-6}	μ
milli-	10^{-3}	m
centi-	10^{-2}	c
deci-	10^{-1}	d
kilo-	10^{3}	k
mega-	10^{6}	M
giga-	10^{9}	G

Table 1.3 Comparison of SI, Metric, and English Units

Physical quantity	SI unit	Some common Metric units	Conversion factors
Length	Meter (m)	Meter (m) Centimeter (cm)	$1 m = 10^2$ cm 1 m = 39,37 in. 1 in. = 2.54 cm
Volume	Cubic Meter (m^3)	Liter (L) Milliliter (mL) *	$1 L = 10^3 cm^3$ $1L = 10^{-3} m^3$ 1L = 1.06 qt
Mass	Kilogram (Kg)	Gram (g) Milligram (mg)	$1 kg = 10^3$ g 1 kg = 2.205 lb 1 lb = 453.6 g
Energy	Joule (J)	Calorie (cal)	1 cal = 4.184 J
Temperature	Kelvin (k)	Degree Celsius (°C)	0 K =- 273.15 °C $$^oC = \frac{5}{9}\left(^oF - 32\right)$$

* A mL is the same volume as a cubic centimeter: $1 mL = 1 cm^3$

Laboratory Balance

A laboratory balance is used to obtain the mass of various objects. There are several different varieties of balances, with various limits on their accuracy. Two of these balances are pictured in **Figure 1.1**. Most modern laboratories possess single-pan balances. These are the most accurate balances; generally, they are also the simplest to use and are the most delicate and expensive. The amount of material to be weighed and the accuracy required determine which balance you should use.

Figure 1.1 Digital electronic balances. The balance gives the mass directly when an object to be weighed is placed on the pan.

Graduated Cylinders

Graduated cylinders are tall, cylindrical vessels with graduations scribed along the side of the cylinder. Since volumes are measured in these cylinders by measuring the height of a column of liquid, it is critical that the cylinder has a uniform diameter along its entire height. Obviously, a tall cylinder with a small diameter will be more accurate than a short one with a large diameter. A liter (L) is divided into milliliters (mL) such that 1 mL = 0.001 L and 1 L = 1000 mL.
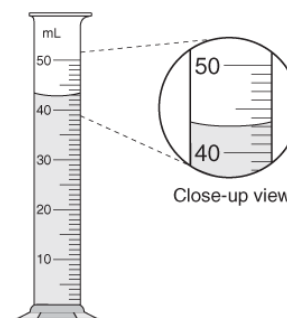
Graduated cylinder

Thermometers

Most thermometers are based upon the principle that liquids expand when heated. Most common thermometers use mercury as the liquid. These thermometers are constructed so that a uniform-diameter capillary tube surmounts a mercury reservoir. To calibrate a thermometer, one defines two reference points, normally the freezing point of water (0°C, 32°F) and the boiling point of water (100°C, 212°F) at 1 atm of pressure (1 atm = 760 mm Hg). Once these points are marked on the capillary, its length is then sub-divided into uniform divisions called *degrees*. There are 100° between these two points on the Celsius, (°C, or centigrade) scale and 180° between those two points on the Fahrenheit (°F) scale.

Pipets

Pipets are glass vessels that are constructed and calibrated so as to deliver a precisely known volume of liquid at a given temperature. The markings on the pipet illustrated in **Figure 1.2** signify that this pipet was calibrated To Deliver (TD) 10.00 mL of liquid at 25°C. Always use a rubber bulb to fill a pipet. NEVER USE YOUR MOUTH! A TD pipet should not be blown empty. It is important that you be aware that every measuring device, regardless of what it may be, has limitations in its accuracy. Moreover, to take full advantage of a given measuring instrument you should be familiar with or evaluate its accuracy. Careful examination of the subdivisions on the device will indicate the maximum accuracy you can expect of that particular tool.
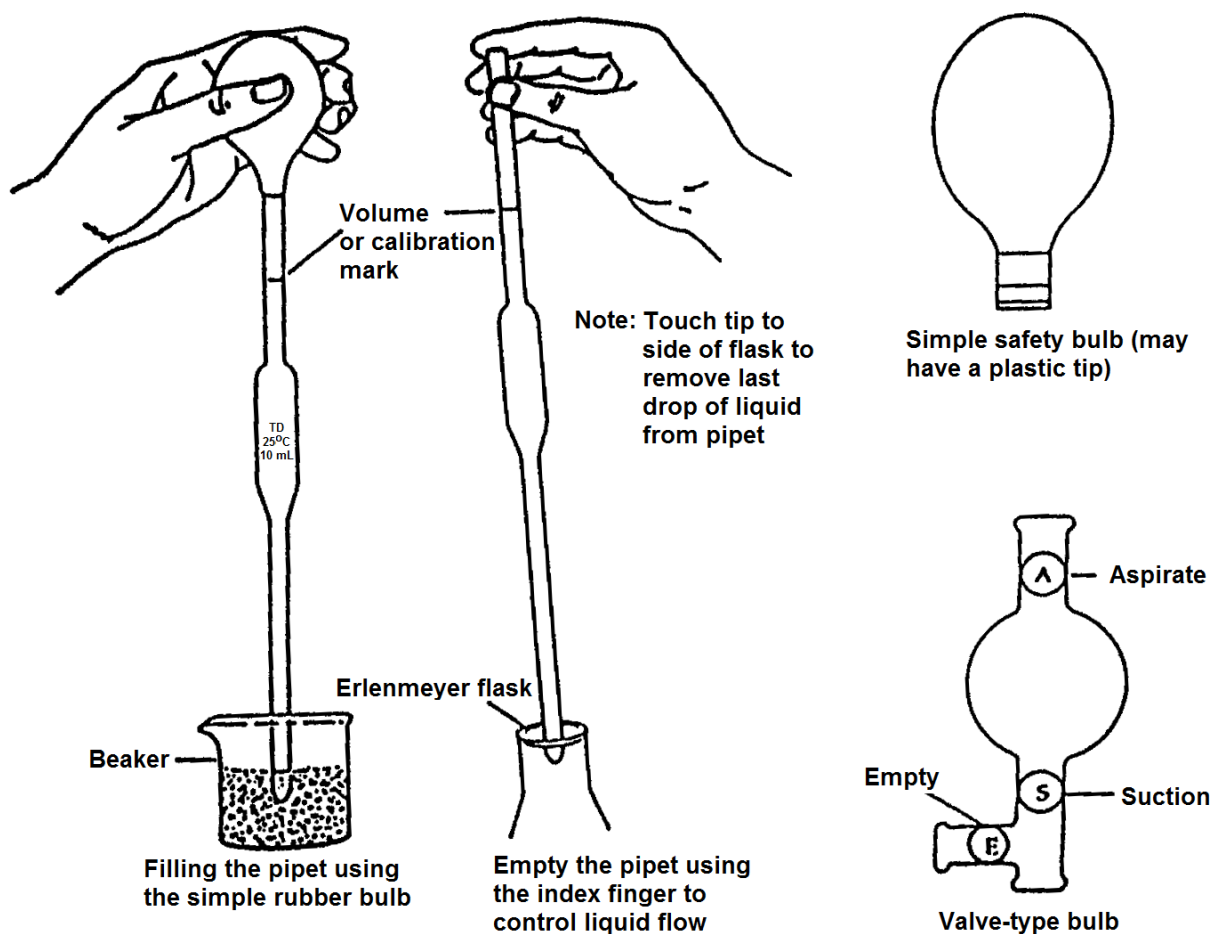
Figure1.2 A typical volumetric pipet, rubber bulbs, and the pipet filling technique.

In this experiment you will determine the accuracy of your 10-mL pipet. The approximate accuracy of some of the equipment you will use in this course is given in **Table 1.4**.

Not only should you obtain a measurement to the highest degree of accuracy that the device or instrument permits, but you should also record the reading or measurement in a manner that reflects the accuracy of the instrument. For example, a mass obtained from an analytical balance should be observed and recorded to the nearest 0.01 g. This is illustrated in **Table 1.5**.

Table1.4 Equipment Accuracy

Equipment	Accuracy
Analytical balance	±0.0001 g (±0.1 mg)
Top-loading balance	±0.001 g (1 mg)
Graduated cylinder	±0.1 mL
Pipet	±0.02 mL
Buret	±0.02 mL
Thermometer	±0.2°C

Table 1.5 Obtaining Significant Figures

Analytical balance	Top loader
85.9 g (incorrect)	85.9 g (incorrect)
85.93 g (incorrect)	85.93 g (incorrect)
85.932 g (incorrect)	85.932 g (correct)
85.9322 g (correct)	

PROCEDURE

A. The Bunsen Burner

Melting points of metals

The Bunsen burner is a convenient source of heat in the laboratory. Although there are several varieties, their principle of operation is the same and is similar to that of the common gas stove. The Bunsen burner requires gas and air, which it mixes in various proportions. The amount of air and gas mixed in the chamber is varied by use of the collar illustrated in **Figure 1.3**. The relative proportions of gas and air determine the temperature of the flame.

Figure1.3 Typical Busen burner.

Examine your burner and locate the gas and airflow adjustments (valves) (see **Figure 1.3**). Determine how each valve operates before connecting the burner to the gas outlet. Close both valves; connect a rubber hose to the gas outlet on the burner and the desk; then open the desk valve about two-thirds of the way. Strike a match or use a gas lighter. Hold the lighted match to the side and just below the top of the barrel of the burner while gradually opening the gas valve on the burner to obtain a flame about 7 or 10 cm high. Gradually open and adjust the air valve until you obtain a pale blue flame with an inner cone as shown in **Figure 1.3**.

Flame temperatures can be observed using the melting points of metals.
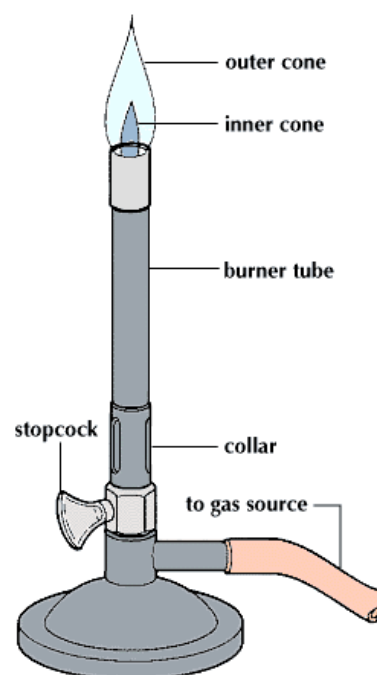
Adjust the burner to a non-luminous flame to measure the temperatures in the various regions of the flame. Use crucible tongs to hold 2-cm strips of iron wire, copper wire, and aluminum wire in the various regions of the flame. The melting point of iron is 1535 °C, that of copper is 1083 °C, and that of aluminum is 660 °C. On the Report Sheet, record the estimated temperature of the flame in the regions designated in **Figure 1.4**.

① Top of the outer cone

② Center of the outer cone

③ Top of the inner cone

④ Center of the inner cone
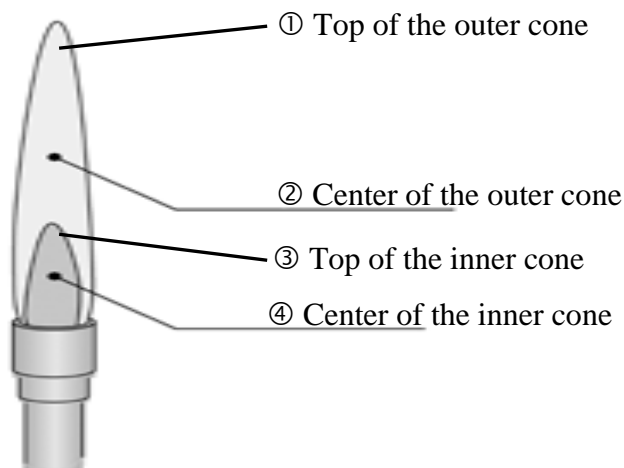
Metal	Melting point (^oC)
Iron (Fe)	1535
Copper (Cu)	1083
Aluminium (Al)	660

Figure 1.4 Regions of the flame for temperature measurement.

B. The Graduated Cylinder

Examine the 100-mL graduated cylinder and notice that it is scribed in milliliters. Fill the cylinder approximately half full with water. Notice that the water meniscus (curved surface of the water) is concave (see **Figure 1.5**). When water is the liquid, the lowest point on the curve is always read as the volume, never the upper level. Avoid errors due to parallax; different and erroneous readings are obtained if the eye is not perpendicular to the scale. Read the volume of water to the nearest 0.1 mL. Record this volume. Measure the maximum amount of water that your 125-mL Erlenmeyer flask will hold. Record this volume.
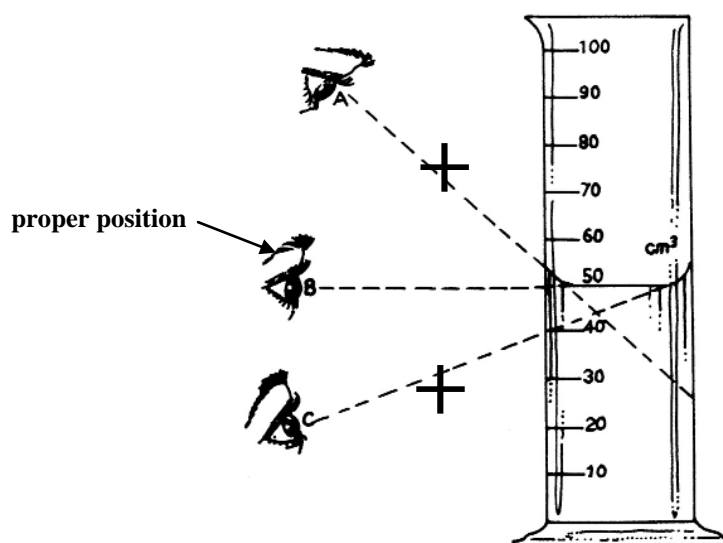
proper position

Figure 1.5   Proper eye position for taking volume readings.
The meniscus reading here is 50.0 mL.

C. Using the balance to calibrate your 10-mL pipet

Weighing an object on a single-pan balance is a simple matter. Because of the sensitivity and the expense of the balance (some cost more than $2500) you must be careful in its use. Directions for operation of single-pan balance vary with make and model. Your laboratory instructor will explain how to use the balance. Regardless of the balance you use, proper care of the balance requires that you observe the following:

1. Do not drop an object on the pan.
2. Center the object on the pan.
3. Do not place chemicals directly on the pan; use a beaker, watch glass, weighing bottle, or weighing paper.
4. Do not weigh hot or warm objects; objects must be at room temperature.
5. Return all weights to the zero position after weighing.
6. Clean up any chemical spills in the balance area.
7. Inform your instructor if the balance is not operating correctly; do not attempt to repair it yourself.

The following method is used to calibrate a pipet or other volumetric glassware. Obtain about 40 mL of distilled water in a 150-mL beaker. Allow the water to sit on the desk while you weigh and record the weight of an empty, dry 25-mL Erlenmeyer flask (tare) to the nearest 0.1 mg. Measure and record the temperature of the water. Using your pipet, pipet exactly 10.00 mL of water into this flask and weigh the flask with the water in it (gross) to the nearest 0.1 mg. Obtain the weight of the water by subtraction (gross − tare= net). Using the equation below and the data given in **Table 1.6**, obtain the volume of water delivered and therefore the volume of your pipet.

$$density = \frac{mass}{volume} = \frac{m}{V}$$

Normally, density is given in units of grams per milliliter (g/mL) for liquids, grams per cubic centimeter (g/cm^3) for solids, and grams per liter (g/L) for gases. Repeat this procedure in triplicate-that is, deliver and weigh exactly 10.00 mL of water three separate times.

**Table 1.6 Density of pure water (g/cm^3) at temperatures ranging from 10.0°C
to 30.9°C by 0.1°C increments (*example*: density at 20.6ºC = 0.998078 g/cm^3)**

		Increment									
		0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
	10	0.999700	0.999691	0.999682	0.999673	0.999664	0.999654	0.999645	0.999635	0.999625	0.999615
	11	0.999605	0.999595	0.999585	0.999574	0.999564	0.999553	0.999542	0.999531	0.999520	0.999509
	12	0.999498	0.999486	0.999475	0.999463	0.999451	0.999439	0.999427	0.999415	0.999402	0.999390
	13	0.999377	0.999364	0.999352	0.999339	0.999326	0.999312	0.999299	0.999285	0.999272	0.999258
	14	0.999244	0.999230	0.999216	0.999202	0.999188	0.999173	0.999159	0.999144	0.999129	0.999114
	15	0.999099	0.999084	0.999069	0.999054	0.999038	0.999023	0.999007	0.998991	0.998975	0.998959
	16	0.998943	0.998926	0.998910	0.998893	0.998877	0.998860	0.998843	0.998826	0.998809	0.998792
	17	0.998774	0.998757	0.998739	0.998722	0.998704	0.998686	0.998668	0.998650	0.998632	0.998613
Temperature (ºC)	18	0.998595	0.998576	0.998558	0.998539	0.998520	0.998501	0.998482	0.998463	0.998444	0.998424
	19	0.998405	0.998385	0.998365	0.998345	0.998325	0.998305	0.998285	0.998265	0.998244	0.998224
	20	0.998203	0.998183	0.998162	0.998141	0.998120	0.998099	0.998078	0.998056	0.998035	0.998013
	21	0.997992	0.997970	0.997948	0.997926	0.997904	0.997882	0.997860	0.997837	0.997815	0.997792
	22	0.997770	0.997747	0.997724	0.997701	0.997678	0.997655	0.997632	0.997608	0.997585	0.997561
	23	0.997538	0.997514	0.997490	0.997466	0.997442	0.997418	0.997394	0.997369	0.997345	0.997320
	24	0.997296	0.997271	0.997246	0.997221	0.997196	0.997171	0.997146	0.997120	0.997095	0.997069
	25	0.997044	0.997018	0.996992	0.996967	0.996941	0.996914	0.996888	0.996862	0.996836	0.996809
	26	0.996783	0.996756	0.996729	0.996703	0.996676	0.996649	0.996621	0.996594	0.996567	0.996540
	27	0.996512	0.996485	0.996457	0.996429	0.996401	0.996373	0.996345	0.996317	0.996289	0.996261
	28	0.996232	0.996204	0.996175	0.996147	0.996118	0.996089	0.996060	0.996031	0.996002	0.995973
	29	0.995944	0.995914	0.995885	0.995855	0.995826	0.995796	0.995766	0.995736	0.995706	0.995676
	30	0.995646	0.995616	0.995586	0.995555	0.995525	0.995494	0.995464	0.995433	0.995402	0.995371

EXAMPLE 1.1

Using the procedure given above, a weight of 10.0025 g was obtained as the weight of
the water delivered by one 10-mL pipet at 22.0°C. What is the volume delivered by
the pipet?

SOLUTION: From the density equation given above, we know that $V = \dfrac{m}{d}$

For mass we substitute our value of 10.0025 g. For the density, consult **Table 1.6**. At
22.0°C, the density is 0.997770 g/mL.

The calculation is:

$$V = \frac{10.0025\,g}{0.997770\,g\,/\,mL} = 10.0249\,mL \approx 10.02\,mL$$

The volume must be rounded off to 10.02, because the pipet's precision can be determined only to within ±0.02 mL.

The precision of a measurement is a statement about the internal agreement among repeated results; it is a measure of the reproducibility of a given set of results. The arithmetic mean (average) of the results is usually taken as the "best" value. The simplest measure of precision is the average deviation from the mean. The average deviation is calculated by first determining the mean of the measurements, then calculating the deviation of each individual measurement from the mean and, finally, averaging the deviations (treating each as a positive quantity). Study **Example 1.2** and then, using your own experimental results, calculate the mean volume delivered by your 10-mL pipet. Also calculate for your three trials the individual deviations from the mean and then state your pipet's volume with its average deviation.

EXAMPLE 1.2
The following values were obtained for the calibration of a 10-mL pipet: 10.10, 9.98, and 10.00 mL. Calculate the mean value and the average deviation from the mean.

SOLUTION:
$$mean = \frac{10.10 + 9.98 + 10.00}{3} = 10.03$$

Deviations from the mean: |value − mean|
|10.10 -10.03| = 0.07
|9.98 - 10.03| = 005
|10.00 - 10.03| = 0.03

Average deviation from the mean $= \dfrac{0.07 + 0.05 + 0.03}{3} = 0.05$

The reported value is therefore $10.03 \pm 0.05\,mL$.

REVIEW QUESTIONS

You should be able to answer the following questions before beginning this experiment:

1. What are the basic units of length, mass, volume, and temperature in the SI system?

2. A liquid has a volume of 1.35 liters. What is its volume in mL? in cm^3?

3. If an object weighs 1.47 g, what is its weight in mg?

4. Why should you never weigh a hot object?

5. What is precision?

6. Define density? Can it be determined from a single measurement?

7. What is the density of an object with a mass of 9.03 g and a volume of 0.1987 mL?

8. Weighing an object three times gave the following results: 10.2 g, 10.1 g, and 10.3g. Find the mean weight and the average deviation from the mean.

9. Normal body temperature is 98.6°F. What is the corresponding Celsius temperature?

11. What is the weight in kilograms of 950 mL of a substance that has a density of 1.274 g/mL?

12. An object weighs exactly five grams on an analytical balance that has an accuracy of 0.1 mg. To how many significant figures should this weight be recorded?

13. What is the dominant color of a properly adjusted flame from a Bunsen burner?

14. How many distinct cones does a properly adjusted non-luminous flame have on a Bunsen burner (one, two or three)?

Experiment 1

Basic laboratory techniques

Name(s) _____

Date _____ Laboratory Instructor _____

REPORT SHEET

A. Bunsen Burner

Indicate the approximate temperature of the following regions of the flame (see **Figure 1.4)**

a**.** region ①: Top of the outer cone _____ °C

b**.** region ②: Center of the outer cone _____ °C

c**.** region ③: Top of the inner cone _____ °C

d**.** region ④: Center of the inner cone _____ °C

B. The graduated cylinder

Volume of water in graduated cylinder _____ mL

Volume of water contained in 125-mL Erlenmeyer flask _____ mL

C. Using the balance to calibrate your 10-mL pipet

Temperature of water _____ °C   Density of water _____ g/cm^3

	Trial 1	Trial 2	Trial 3	
Weight of Erlenmeyer (tare wt)	_____	_____	_____	g
Weight of Erlenmeyer plus ~ 10 mL H$_2$0 (gross wt)	_____	_____	_____	g
Weight of ~ 10 mL of H$_2$O (net wt)	_____	_____	_____	g
Volume delivered by 10-mL pipet	_____	_____	_____	mL

♨ *show calculations overleaf*

Mean volume delivered by 10-mL pipet _____ mL
♨ *show calculations overleaf*

	Trial 1	Trial 2	Trial 3	
Individual deviations from the mean	_____	_____	_____	
Average deviation from the mean		_____		mL

♨ *show calculations overleaf*

Volume delivered by your 10-mL pipet _____ mL ± _____ mL

EXPERIMENT 2

Identification of substances: Physical properties

OBJECTIVE

To become acquainted with procedures used in evaluating physical properties and the use of these properties in identifying substances.

Relates to chapter 1 of "Chemistry the Central Science, 12th Ed.".

APPARATUS AND CHEMICALS

Balance	Capillary tubes (5)
250 mL beaker	Spatula
25 mL Erlenmeyer flask	Ring stand and ring
10 mL graduated cylinder	Utility clamp
10 mL pipet	Thermometer clamp
5 mL pipet	Thermometer
50 mL beakers (2)	Stirring rod
Burner and hose	Apparatus for boiling point determination
Wire gauze	Naphthalene (1 g)
Small rubber bands	Ethyl alcohol (15 mL)
Boiling chips	Cyclohexane (20 mL)
Large test tubes (2)	2 unknowns (liquid and solid)
Small test tubes (6)	Soap solution
Test-tube rack	

DISCUSSION

PROPERTIES are those characteristics of a substance that enable us to identify it and to distinguish it from other substances. Direct identification of some substances can readily be made by simply examining them. For example, we see color, size, shape, and texture, and we can smell odors and discern a variety of tastes. Thus, copper can be distinguished from other metals on the basis of its color.

PHYSICAL PROPERTIES are those properties that can be observed without altering the composition of the substance. Whereas it is difficult to assign definitive values to such properties as taste, color, and odor, other physical properties, such as melting point, boiling point, solubility, density, viscosity, and refractive index, can be expressed quantitatively. For example, the melting point of copper is 1087 °C, and its density is 8.96 g/cm^3. As you probably realize, a specific combination of properties is unique to a given substance, thus making it possible to identify most substances just by careful determination of several properties. This is so important that large books have been compiled listing characteristic properties of many known substances. Two of the most complete references of this type that are readily available today are The *CRC Handbook of Chemistry and Physics* and *Lange's Handbook of Chemistry*.

In this experiment you will use the following properties to identify a substance whose identity is unknown to you: solubility, density, and boiling point.

The solubility of a substance in a solvent at a specified temperature is the maximum weight of that substance that dissolves in a given volume (usually 100 or 1000 mL) of a solvent. It is tabulated in hand-books in terms of grams per 100 mL of solvent; the solvent is usually water.

In the preceding experiment you learned that the density of a substance is defined as

the mass per unit volume: $\qquad d = \dfrac{m}{V}$

A liquid is said to boil when bubbles of vapor from within it, rise rapidly to the surface, and burst. Any liquid in contact with the atmosphere will boil when its vapor pressure is equal to atmospheric pressure-that is, the liquid and gaseous states of a substance are in equilibrium. Boiling points of liquids depend upon atmospheric pressure. A liquid will boil at a higher temperature at a higher pressure or at lower temperature at a lower pressure. The temperature at which a liquid boils at 760 mmHg is called the normal boiling point. To account for these pressure effects on boiling points, people have studied and tabulated data for boiling point versus pressure for a large number of compounds. From these data, nomographs have been constructed.
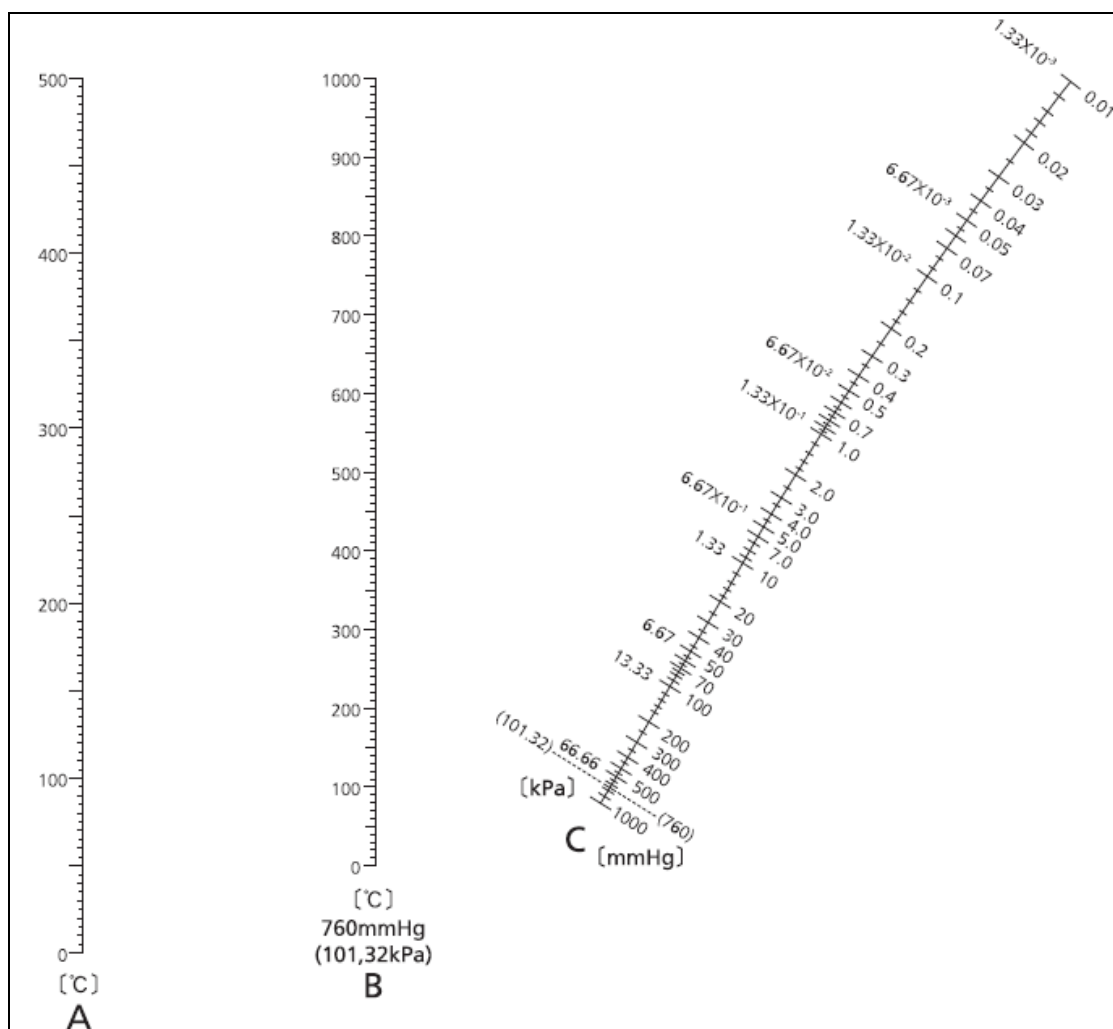

FIGURE 2.1 Nomograph for boiling point correction to 760 mmHg
1) Connect the actual (reduced) pressure on line **C** to its corresponding bp on line **A** using a straight line
2) An intersection found by step 1on line **B** serves as an approximate bp at atmospheric pressure

A nomograph is a set of scales for connected variables (see **Figure 2.1** for an example); these scales are so placed that a straight line connecting the known values on some scales will provide the unknown value at the straight line's intersection with other scales. A nomograph allows you to find the correction necessary to convert the normal boiling point of a substance to its boiling point at any pressure of interest.

Note that the atmospheric pressure in Ifrane (~626 mm Hg) is less than the atmospheric pressure at sea level (760 mm Hg).

PROCEDURE

A. Solubility

Caution: cyclohexane is highly flammable and must be kept away from open flames.

Qualitatively determine the relative solubility of three solutes: water, cyclohexane, and ethyl alcohol. Determine the solubility by adding 5 drops of solute to 2 mL of each of the three solvents in separate, clean, dry test tubes (it is not necessary to measure these volumes accurately). Place some *parafilm* in each test tube and shake briefly. Record your conclusions on the report sheet using the abbreviations *s* (soluble), *sp* (sparingly soluble), and *i* (insoluble).

The formation of two layers indicates immiscibility (lack of solubility) - *i*.

Cloudiness indicates partial solubility - *sp*.

Now repeat these experiments using your liquid unknown as a solute and record your observations.

B. The density of a liquid

Weigh a clean, dry 25-mL Erlenmeyer flask to the nearest 0.01g. Obtain at least 15 mL of the unknown liquid in a clean and dry test tube. Using a 10-mL pipet, pipet exactly 10.0 mL of the unknown liquid into the 25-mL Erlenmeyer flask and quickly weigh the flask containing the 10.0 mL of the unknown to the nearest 0.01 g. Using the weight of this volume of unknown, calculate its density. Record your results and show how (with units) you performed your calculations.

SAVE THE LIQUID FOR YOUR BOILING-POINT DETERMINATION

C. Boiling Point of Liquid Unknown

*Obtain a 260 °C thermometer. Assemble the boiling point apparatus in **Figure 2.3**. Complete two measurements for the boiling point of your unknown.*

a. Assemble the Boiling Point Apparatus.

Place enough of the liquid unknown (use some of the same material you used to determine the density) into a 75-mm test tube, so that the thermometer bulb gets completely immersed.

Caution: The liquid may be flammable - keep it away from an open Bunsen flame.

Position it beside the thermometer bulb using a rubber band. Invert (open end down) a 10-cm capillary tube in the liquid. Place the apparatus into the water bath.

b. **Measure the Boiling Point**.
Place 2-3 boiling chips in the water bath. Slowly heat the water while stirring with the stirring rod. When a rapid and continuous stream of bubbles escapes the capillary tube, discontinue heating the water bath, and gently take the test tube out of the bath. The stream of bubbles slows as the tube cools. When the bubbles cease to escape and before the liquid re-enters the capillary tube, record the observed boiling point (the temperature will become constant at the boiling point of the liquid). Estimate the true (normal b.p. at 1 atm = 760 mmHg) using the pressure correction.

c. Repeat the Measurement.
Add some cold water to the bath and determine the boiling point a second time. The same liquid may be used; however, the capillary tube must be removed, emptied, and inserted before heating resumes.
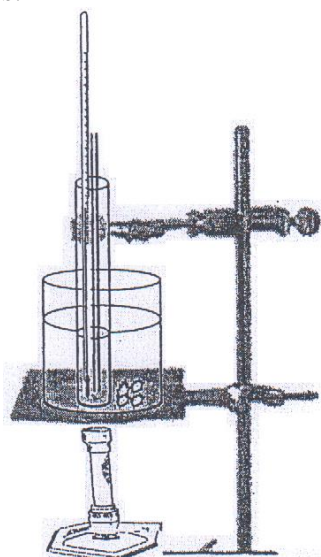

Figure 2.3 Apparatus for determining the boiling point of a liquid.

D. Unknown Identification

Your unknown is one of the substances contained in the table of physical properties enclosed. Compare the properties that you have determined for your unknown with those in the **Appendix I**. Identify your unknown and record your results.

REVIEW QUESTIONS

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

1. List five physical properties?

2. A 8.692 mL sample of an unknown weighed 10.02 g. What is the density of the unknown?

3. Could you determine the density of cadmium nitrate using water? Why or why not?

4. Why do we calibrate thermometers and pipets?

5. Is bromoform miscible with water? with cyclohexane?

6. When water and toluene are mixed, two layers form. Is the bottom layer water or toluene? (see table enclosed)

7. What solvent would you use to determine the density of cadmium nitrate?

8. The density of a solid with melting point of 52 to 54°C was determined to be 1.45 ± 0.02 g/ml. What is the solid?

Experiment 2

Identification of Substances:
Physical Properties

Name(s) _____

Date _____ Laboratory Instructor _____

Unknown n°_____

REPORT SHEET

A.   Solubility

s = soluble *sp* = sparingly soluble *i* = insoluble		Solvent (~2mL)		
		Water	Ethanol	Cyclohexane
Solute (~5 drops)	Water	▨		
	Ethanol	▨	▨	
	Cyclohexane	▨	▨	▨
	Liquid unknown			

B.   Density of Liquid Unknown

Volume of liquid   _____10.0_____ mL

Weight of 25-mL Erlenmeyer   _____ g

Weight of 25-mL Erlenmeyer

   plus 10.0 mL of unknown   _____ g

Weight of liquid   _____ g

Density   _____ g/mL
♨ *show calculations overleaf*

C.   Boiling point of liquid unknown

Observed b.p. 1: _____ °C   Observed b.p. 2: _____ °C   Average b.p. : _____ °C

<u>Note:</u> You should expect a slightly lower boiling point than that given in Appendix I
      (Ifrane is located at 1600 meter above the sea level)

D.   Unknown Identification

          Liquid unknown   _____

EXPERIMENT 3

Separation of the components of a mixture

OBJECTIVE

To become familiar with the methods of separating substances from one another using decantation, extraction, and sublimation techniques.

Relates to chapter 1 of "Chemistry the Central Science, 12th Ed.".

APPARATUS AND CHEMICALS

Balance	Glass stirring rod
Ring stands (2)	50- or 100-mL graduated cylinder
Tongs	Unknown mixture of sodium
Watch glass	chloride, ammonium chloride and
Evaporating dishes (2)	silicon dioxide (2-3g)

DISCUSSION

Materials that are not uniform in composition are said to be impure or heterogeneous and are called mixtures. Most of the materials we encounter in everyday life, such as cement, wood, and soil, are mixtures. When two or more substances that do not react chemically are combined, a mixture results. Mixtures are characterized by two fundamental properties: First, each of the substances in the mixture retains its chemical integrity; second, mixtures are separable into these components by physical means. If one of the substances in a mixture is preponderant-that is, if its amount far exceeds the amounts of the other substances in the mixture-then we usually call this mixture an *impure substance* and speak of the other substances in the mixture as *impurities*.

The preparation of compounds usually involves their separation or isolation from reactants or other impurities. Thus the separation of mixtures into their components and the purification of impure substances are frequently encountered problems. You are probably aware of everyday problems of this sort. For example, our drinking water usually begins as a mixture of silt, sand, dissolved salts, and water. Since water is by far the largest component in this mixture, we usually call this impure water. How do we purify it? The separation of the components of mixtures is based upon the fact that each component has different physical properties. The components of mixtures are always pure substances, either compounds or elements, and each pure substance possesses a unique set of properties. The properties of every sample of a pure substance are identical under the same conditions of temperature and pressure. This means that once we have determined that a sample of sodium chloride, NaCl, is water-soluble and a sample of silicon dioxide (sand), SiO_2, is not, we realize that all samples of sodium chloride are water-soluble and all samples of silicon dioxide are not. Likewise, every crystal of a pure substance melts at a specific temperature, and at a given pressure, every pure substance boils at a specific temperature.

Although there are numerous physical properties that can be used to identify a particular substance (as you learned in the previous experiment), we will be concerned in this experiment merely with the separation of the components and not with their identification.

The methods we will use for the separation depend upon differences in physical properties, and they include the following:

1. *Decantation*: This is the process of separation of a liquid from a solid (sediment) by gently pouring the liquid from the solid so as not to disturb the solid (see **Figure 3.1**).

2. *Filtration:* This is the process of separating a solid from a liquid by means of a porous substance, a filter, which allows the liquid to pass through but not the solid (see **Figure 3.1**). Common filter materials are papers, layers of charcoal, and sand. Silt and sand can be removed from our drinking water by this process.

3. *Extraction*: This is the separation of a substance from a mixture by preferentially dissolving that substance in a suitable solvent. This process is used to separate a soluble compound from an insoluble compound.

4. *Sublimation*: This is the process in which a solid passes directly to the gaseous state without the appearance of the liquid state. Not all substances possess the ability to be sublimed. Iodine, naphthalene, and ammonium chloride (NH_4Cl) are common substances that easily sublime.
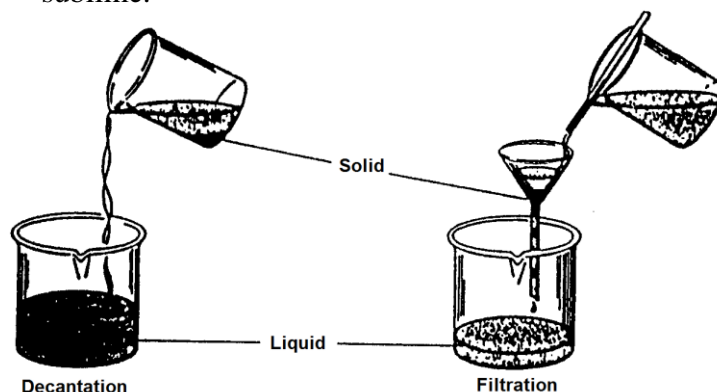

Figure 3.1 Description of the processes of decantation and filtration.

The mixture that you will separate contains three components: NaCl, NH_4Cl, and SiO_2. Their separation will be accomplished by heating the mixture to sublime the NH_4Cl, extracting the NaCl with water, and finally drying the remaining SiO_2, as illustrated in the scheme shown in **Figure 3.2**.
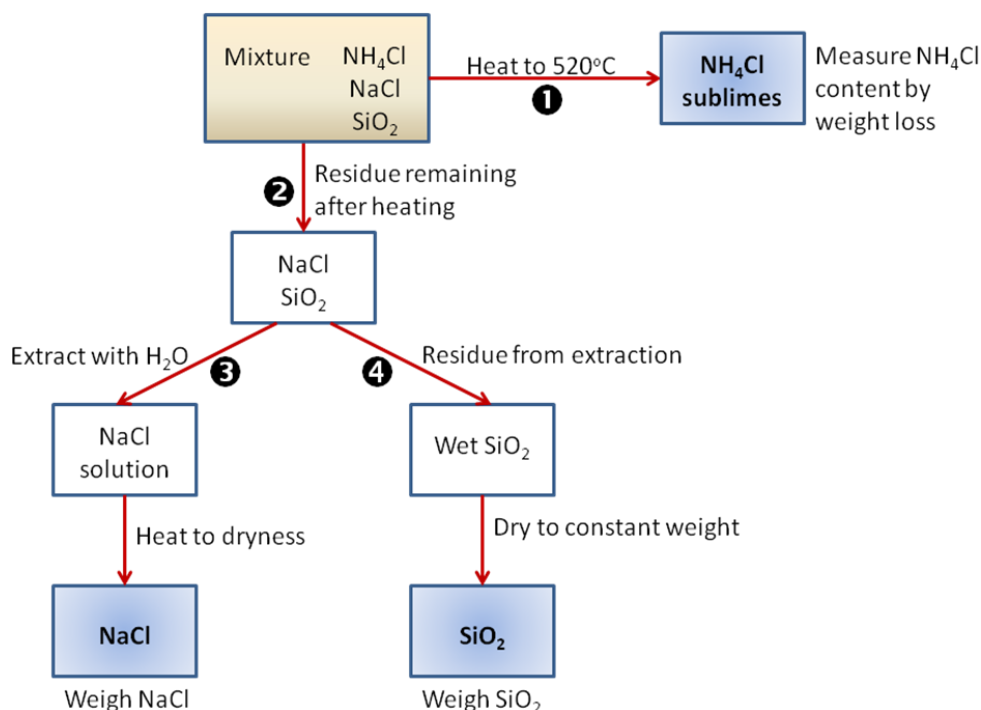
Figure 3.2 Flow diagram for the separation of the components of the mixture

PROCEDURE

Carefully weigh a clean, dry evaporating dish to the nearest 0.01 g. Then obtain from your instructor a 2- to 3-g sample of the unknown mixture in the evaporating dish. Weigh the evaporating dish containing the sample and calculate the sample weight.

Place the evaporating dish containing the mixture on a hot plate under THE FUMEHOOD. Heat the evaporating dish until white fumes are no longer formed (a total of about 15 min). Heat carefully to avoid spattering, especially when liquid is present. After the first 10 min gently stir the mixture with your glass stirring rod; then apply the heat again.

Allow the evaporating dish to cool until it reaches room temperature and then weigh the evaporating dish with the contained solid.

> NEVER WEIGH HOT OR WARM OBJECTS!

The loss in weight represents the amount of NH_4Cl in your mixture. Calculate this weight.

Add 25 mL of water to the solid in this evaporating dish and stir gently for 5 min. Next, weigh another clean, dry evaporating dish and watch glass. Decant the liquid carefully into the second evaporating dish, which you have weighed, being careful not to transfer any of the solids into the second evaporating dish. Add 10 mL more of water to the solid in the first evaporating dish, stir, and decant this liquid into the second evaporating dish as before. Repeat with still another 10 mL of water. This process extracts the soluble NaCl from the sand. You now have two evaporating dishes; one containing wet sand and the second, a solution of sodium chloride.

Place the evaporating dish containing the sodium chloride solution carefully on the hotplate. Begin gently heating the solution to evaporate the water. Take care to avoid boiling or spattering, especially when liquid is present. Near the end, cover the

evaporating dish with the watch glass that was weighed with this evaporating dish, and reduce the heat to prevent spattering. While the water is evaporating you may proceed to dry the SiO_2 in the other evaporating dish as explained in the next paragraph, if you have another hotplate available. When you have dried the sodium chloride completely, no more water will condense on the watch glass, and it, too, will be dry. Let the evaporating dish and watch glass cool to room temperature and weigh them.

The difference between this weight and the weight of the empty evaporating dish and watch glass is the weight of the NaCl. Calculate this weight.

Place the evaporating dish containing the wet sand on the hotplate and cover the evaporating dish with a clean, dry watch glass. Heat slowly at first until the lumps break up and the sand appears dry. Then heat the evaporating dish to dull redness and maintain this heat for 10 min. Take care not to overheat, or the evaporating dish will crack. When the sand is dry, remove the heat and let the dish cool to room temperature. Weigh the dish after it has cooled to room temperature. The difference between this weight and the weight of the empty dish is the weight of the sand. Calculate this weight.

Calculate the percentage of each substance in the mixture using an approach similar to that shown in **Example 3.1**. The accuracy of this experiment is such that the combined total of your three components should be in the neighborhood of 99%. If it is less than this, you have been careless. If it is more than 100%, you have not sufficiently dried the sand and salt.

EXAMPLE 3.1
What is the percentage of SiO_2 in a 7.69 g sample mixture if 3.76 g of SiO_2 has been recovered?

SOLUTION: The percentage of each substance in such a mixture can be calculated as follows:

$$\% \ component = \frac{wt \ component \ in \ grams \times 100}{wt \ sample \ in \ grams}$$

Therefore, the percentage of SiO_2 in this particular sample mixture is

$$\% \ SiO_2 = \frac{3.76 \ g \times 100}{7.69 \ g} = 48.9 \ \%$$

REVIEW QUESTIONS

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

1. What distinguishes a mixture from an impure substance?

2. Define the process of sublimation.

3. How do decantation and filtration differ? Which should be faster?

4. Why does one never weigh a hot object?

5. How does this experiment illustrate the principle of conservation of matter?

6. A mixture was found to contain 3.10 g of SiO_2, 0.38 g of cellulose, and 6.72 g of calcium carbonate. What is the percentage of $CaCO_3$ in this mixture?

7. How could you separate a mixture of zinc chloride and cyclohexane? Consult the table for physical properties, Appendix I.

8. How could you separate zinc chloride from SiO_2?

9. Why is SiO_2 washed with water three times as opposed to only once?

10 A student found that her mixture was 15 percent NH_4C1, 20 percent NaCl, and 75 percent SiO_2. Assuming her calculations are correct, what did she most likely do incorrectly in her experiment?

Experiment 3

Separation of the Components of a Mixture

Name(s) _____

Date _____   Laboratory Instructor _____

Unknown n° _____

REPORT SHEET

A. Weight of evaporating dish   _____ **g**

Weight of evaporating dish and original sample   _____ **g**

Weight of original sample m_{SAMPLE} = _____ **g**

Weight of evaporating dish after subliming NH_4Cl _____ **g**

Weight of NH_4Cl _____ **g**

Percent of NH_4Cl (show calculations)   _____ **%**

B. Weight of evaporating dish and watch glass   _____ **g**

Weight of evaporating dish, watch glass, and NaCl   _____ **g**

Weight NaCl   _____ **g**

Percent of NaCl (show calculations)   _____ **%**

C. Weight of evaporating dish and SiO_2 _____ **g**

Weight of evaporating dish   _____ **g**

Weight of SiO_2 _____ **g**

Percent of SiO_2 (show calculations)   _____ **%**

D. Weight of original sample m_{SAMPLE} = _____ **g**

Weight of determined [NH_4Cl + NaCl + SiO_2], m_{TOTAL}

$m_{TOTAL} = m_{NH4Cl} + m_{NaCl} + m_{SiO2}$ =   _____ **g**

Differences in these weights $|m_{SAMPLE} - m_{TOTAL}|$ =   _____ **g**

Percent recovery of matter

$$= \frac{g \ matter \ recovered}{g \ original \ sample} \times 100 = \frac{m_{TOTAL}}{m_{SAMPLE}} \times 100 = \underline{\hspace{1cm}} \ \%$$

If the percent recovery of matter is not exactly 100%, account for your errors below:

EXPERIMENT 4

Chemical formulas

OBJECTIVE

To become familiar with chemical formulas and how they are determined.

Relates to chapter 3 of "Chemistry the Central Science, 12th Ed.".

APPARATUS AND CHEMICALS

Balance	Copper wire (~ 2 g)
250-mL beaker	Crucible and cover
Hotplate	Evaporating dish
25-mL graduated cylinder	Carborundum boiling chips
Ring stand and ring stirring rod	6 *M* HCl
Granular zinc (~ 1 g)	Powdered sulfur (~3 g)

Derivation of Formulas

Suppose you were working in a hospital laboratory today, and suppose further that this morning the emergency ward admitted a patient complaining of severe stomach cramps and laboured respiration and that this patient died within minutes of being admitted. Relatives of the patient later told you that he may have ingested some rat poison. You therefore had his stomach pumped to verify this or simply to determine the cause of death. One of the more logical things to do would be to attempt to isolate the agent that caused death and perform chemical analyses on it. Let's suppose that this was done, and the analyses showed that the isolated chemical compound contained, by weight, 60.0 percent potassium, 18.5 percent carbon, and 21.5 percent nitrogen. What is the chemical formula for this compound?

One simple and direct way of making the necessary calculations is as follows.

Assume you had 100 g of the compound. This 100 g therefore would contain

$$(100 \ g) \times \left(\frac{60.0}{100} \right) = 60.0 \ g \ \text{potassium}$$

$$(100 \ g) \times \left(\frac{18.5}{100} \right) = 18.5 \ g \ \text{carbon}$$

$$(100 \ g) \times \left(\frac{21.5}{100} \right) = 21.5 \ g \ \text{nitrogen}$$

Chemical formulas tell what elements are present and the ratio of the number of atoms of the constituent elements. Hence, the next step is to determine the number of moles of each element present:

$$\text{Moles potassium} \ = \ n_K = \frac{60.0 \ g}{39.0 \ g / mol} = 1.54 \ mol$$

$$\text{Moles carbon} \ = \ n_C = \frac{18.5 \ g}{12.0 \ g / mol} = 1.54 \ mol$$

$$\text{Moles nitrogen} \quad = \quad n_N = \frac{21.5\ g}{14.0\ g/mol} = 1.54\ mol$$

Hence the chemical formula is $K_{1.54}C_{1.54}N_{1.54}$ but molecules are not formed from partial atoms; therefore, the above numbers must be changed to whole numbers. This is accomplished by division of all subscripts by the smallest subscript. In this case, they are all the same.

$$K_{\frac{1.54}{1.54}}C_{\frac{1.54}{1.54}}N_{\frac{1.54}{1.54}} = KCN$$

The smallest whole-number mole ratio is 1:1:1. Since KCN is a common rat poison, we may justifiably conclude that the relatives' suggestion of rat poison ingestion as the probable cause of death is correct.

The above calculation has given us what is known as the *empirical* formula. There is another type of chemical formula, the *molecular* formula. The distinction between these two is simply that the empirical formula represents the *smallest* whole-number ratio of the combining atoms in a chemical compound, whereas the molecular formula gives the *actual* number of atoms in a molecule. Recall; however, as we stated earlier, that not all compounds exist as discrete molecules. This is true for most ionic compounds, whereas most covalent compounds do exist as discrete molecules. The distinction between empirical and molecular formulas may be clarified by the following example.

Example:

A chemical compound was found by elemental analyses to contain 92.3 percent carbon and 7.7 percent hydrogen by weight and to have a molecular weight of 78. The empirical formula may be obtained just as in the previous example-that is, in 100 g of the compound there are 92.3 g C and 7.7 g H. Hence,

$$\text{Moles C} \quad = \quad n_C = \frac{92.3\ g}{12.0\ g/mol} = 7.7\ mol$$

$$\text{Moles H} \ = \ n_H = \frac{7.7\ g}{1.0\ g/mol} = 7.7\ mol$$

The empirical formula is then $C_{7.7}H_{7.7}$, or CH, whose formula weight is $12 + 1 = 13$. But the molecular weight of the compound is 78. Therefore, there are $\frac{78}{13} = 6$ empirical formula weights in the molecular weight. The molecular formula is then C_6H_6. In this experiment you will determine the empirical formulas of two chemical compounds. One is copper sulfide, which you will prepare according to the following chemical reaction:

$$\textbf{x}\ Cu\ (s) + \frac{\textbf{y}}{8}\ S_8\ (s) \longrightarrow Cu_{\textbf{x}}S_{\textbf{y}}\ (s)$$
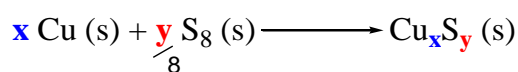
The other is zinc chloride, which you will prepare according to the chemical reaction

$$x \text{ Zn (s)} + y \text{ HCl (aq)} \longrightarrow \text{Zn}_x\text{Cl}_y \text{ (s)} + \frac{y}{2}\text{H}_2 \text{ (g)}$$
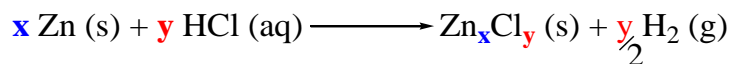
The objective is to determine the combining ratios of the elements (that is, to determine **x** and **y**) and to balance the chemical equations given above.

PROCEDURE

A. Zinc Chloride

> **CAUTION**: *Zinc chloride is caustic and must be handled carefully in order to avoid any contact with your skin. Should you come in contact with it immediately wash the area with copious amounts of water.*

Record the weight of an empty evaporating dish. Obtain about 0.5 g of granular zinc and add it to the weighed evaporating dish. Weigh the evaporating dish containing the zinc and record the total weight. Calculate the weight of the zinc.

Under the fumehood, slowly and with constant stirring, add 15 mL of 6 *M* HCl to the evaporating dish containing the zinc. A vigorous reaction will ensue, and hydrogen gas will be produced.

> NO FLAMES ARE PERMITTED IN THE LABORATORY WHILE THIS REACTION IS TAKING PLACE, SINCE WET HYDROGEN GAS IS VERY EXPLOSIVE.

Stir the mixture gently at room temperature for 5 min. Next, heat the evaporating dish very carefully on a hotplate until all the zinc has dissolved. Finally, keep heating until of the liquid has evaporated. During this last stage, heating should be monitored, otherwise there will be spattering and some loss of product will occur.

> DO NOT HEAT TO THE POINT THAT THE COMPOUND MELTS, OR SOME WILL BE LOST DUE TO SUBLIMATION.

Leave the compound looking somewhat pasty while hot.

Allow the dish to cool to room temperature and weigh it. Record the weigh. After this first weighing, heat the dish again very gently for 5 min; cool it and reweigh it if these weightings do not agree within 0.02g, repeat the heating and weighing until two successive weightings agree. This is known as *drying to constant weight* and is the only way to be certain that all the moisture is driven off. Zinc chloride is very deliquescent and so should be weighed quickly.

Calculate the weight of zinc chloride. The difference in weight between the zinc and zinc chloride is the weight of chlorine. Calculate the weight of chlorine in zinc chloride. From this information you can readily calculate the empirical formula for zinc chloride and balance the chemical equation for its formation. Perform these operations on the report sheet.

B. Copper Sulfide

Record the weight of a clean and dry porcelain crucible and its cover.

Place 1.5-2.0 g of tightly wound copper wire or copper turnings in the crucible and weigh the copper, crucible, and cover. Calculate the weight of copper. Record your results.
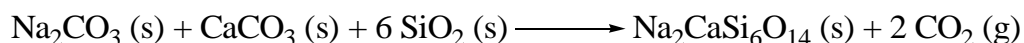
In the fumehood, add sufficient sulfur to cover the copper, place the crucible with cover and heat the crucible. Keep the cover on for the first 5 min to raise the temperature of the crucible quickly, then take it out. Keep heating until sulfur ceases to burn (observation of a blue flame indicates burning). Finally, heat the crucible to dull redness for about 5 min. Allow the crucible to cool to room temperature. This will take about 10 min. Then weigh with the cover in place. Record the weight. Again cover the contents of the crucible with sulfur and repeat the heating procedure. Allow the crucible to cool and reweigh it. Record the weight. If the last two weighings do not agree to within 0.02 g, the chemical reaction between the copper and sulfur is incomplete. If this is found to be the case, add more sulfur and repeat the heating and weighing until a constant weight is obtained.

Calculate the weight of copper sulfide obtained. The difference in weight between the copper sulfide and copper is the weight of sulfur in copper sulfide. Calculate this weight. From this information the empirical formula for copper sulfide can be obtained, and the chemical equation for its production can be balanced. Perform these operations on your report sheet.

REVIEW QUESTIONS

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

1. How do formula weights and molecular weights differ?

2. What is the percentage composition of $CaCO_3$?

3. A substance was found by analysis to contain 65.95 percent barium and 34.05 percent chlorine. What is the empirical formula for the substance?

4. What is the law of definite proportions?

5. How do empirical and molecular formulas differ?

6. What is the weight in grams of one copper atom?

7. Soda-lime glass is prepared by fusing sodium carbonate, Na_2CO_3; limestone. $CaCO_3$; and sand, SiO_2. The composition of the glass varies, but the commonly accepted reaction for its formation is

$$Na_2CO_3 \text{ (s)} + CaCO_3 \text{ (s)} + 6 \text{ SiO}_2 \text{ (s)} \longrightarrow Na_2CaSi_6O_{14} \text{ (s)} + 2 \text{ CO}_2 \text{ (g)}$$

Using this equation, how many kilograms of sand would be required to produce enough glass to make five thousand 400-g wine bottles?

8. Caffeine, a stimulant found in coffee and tea, contains 49.5 percent C, 5.15 percent H, 28.9 percent N, and 16.5 percent O by mass. What is the empirical formula of caffeine? If its molar mass is about 195 g, what is its molecular formula?

9. How many lithium atoms are present in 0.01456 g of lithium?

Experiment 4

Chemical Formulas

Name(s) _____

Date _____ Laboratory Instructor _____

REPORT SHEET

A. Zinc Chloride

1. Weight of evaporating dish   _____ g

2. Weight of evaporating dish and zinc   _____ g

3. Weight of zinc   _____ g

4. Weight of evaporating dish and zinc chloride:
Evaporation to dryness   First weighing   _____ g
                               Second weighing   _____ g

5. Weight of zinc chloride   _____ g

6. Weight of chlorine in zinc chloride   _____ g

7. Empirical formula for zinc chloride   _____
    ⚲ *show calculations overleaf*

8. Balanced chemical equation for the formation of zinc chloride from zinc and HCl

B. Copper Sulfide

1. Weight of crucible and cover   _____ g

2. Weight of crucible, cover, and copper   _____ g

3. Weight of copper   _____ g

4. Weight of crucible, cover, and copper sulfide:
Evaporation to dryness   First weighing   _____ g
                               Second weighing   _____ g

5. Weight of copper sulfide   _____ g

6. Weight of sulfur in copper sulfide   _____ g

7. Empirical formula for copper sulfide   _____
    ⚲ *show calculations overleaf*

8. Balanced chemical equation for the formation of copper sulfide from copper and sulfur

EXPERIMENT 5

Chemical Reactions

OBJECTIVE

To observe some typical chemical reactions, identify some of the products, and summarize the chemical changes in terms of balanced chemical equations.

Relates to chapter 3 of "Chemistry the Central Science, 12th Ed.".

APPARATUS AND CHEMICALS

Crucible and cover
Mossy zinc
Copper wire (2-in. length)
Copper sulfide
0.1 M sodium oxalate
$Na_2C_2O_4$
0.1 M KMnO4
10 M NaOH
0.1 M $Pb(NO_3)_2$
0.1 M $BaCl_2$
1 M K_2CrO_4
0.1 M $NaHSO_3$ (freshly prepared)

6 M HCl
6 M NH_3
0.01 M $CuSO_4$
6 M H_2SO_4
Conc. HNO_3
3 M $(NH_4)_2CO_3$
$KMnO_4$ (solid)
Na_2CO_3 (solid)
Na_2SO_3 (solid)
6-in. test tube

DISCUSSION

Chemical equations represent what transpires in a chemical reaction. For example, the equation below means that potassium chlorate, $KClO_3$, decomposes on heating (Δ is the symbol used for heat) to yield potassium chloride, KCl, and oxygen, O_2.

$$2\ KClO_3\ (s) \xrightarrow{\Delta} 2\ KCl\ (s) + 3\ O_2\ (g)$$
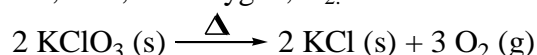
Before an equation can be written for a reaction, someone must establish what the products are. How does one decide what these products are? Products are identified by their chemical and physical properties as well as by analyses. That oxygen and not chlorine gas is produced in the above reaction can be established by the fact that oxygen is a colorless, odorless gas. Chlorine, on the other hand, is a pale, yellow-green gas with an irritating odor.

In this experiment you will observe that in some cases gases are produced, precipitates are formed, or color changes occur during the reactions. These are all indications that a chemical reaction has occurred. To identify some of the products of the reactions, you can consult **Table 5.1**, which lists some of the properties of the substances that could be formed in these reactions.

TABLE 5.1 Properties of reaction products

Water soluble solids		Manganese oxyanions	
KCl	white (colorless solution)	**MnO_4^-**	purple
NH_4Cl	white(colorless solution)	**MnO_4^{2-}**	dark green
$KMnO_4$	purple	**MnO_4^{3-}**	dark blue
$MnCl_2$	pink, (very pale)	**Gases**	
$Cu(NO_3)_2$	blue	**H_2**	colorless, odorless
Water-insoluble solids		**NO_2**	brown, pungent odor (TOXIC)
CuS	very dark blue or black	**NO**	colorless, slight pleasant odor
Cu_2S	black	**CO_2**	colorless, odorless
$BaCrO_4$	yellow	**Cl_2**	pale yellow-green
$BaCO_3$	white	**SO_2**	colorless, choking
$PbCl_2$	white	**H_2S**	colorless, rotten- egg odor (TOXIC)
MnO_2	black or brown		

TABLE 5.2   Activity series

Li → Li$^+$ + e-
K → K$^+$ + e-
Ba → Ba^{2+} + 2 e-
Sr → Sr^{2+} + 2 e-
Ca → Ca^{2+} + 2 e-
Na → Na$^+$ + e-
Mg → Mg^{2+} + 2 e-
Be → Be^{2+} + 2 e-
Al → Al^{3+} + 3 e-
Mn → Mn^{2+} + 2 e-
Zn → Zn^{2+} + 2 e-
Cr → Cr^{3+} + 3 e-
Fe → Fe^{2+} + 2 e-
Cd → Cd^{2+} + 2 e-
Co → Co^{2+} + 2 e-
Ni → Ni^{2+} + 2 e-
Sn → Sn^{2+} + 2 e-
Pb → Pb^{2+} + 2 e-
H$_2$ → 2 H$^+$ + 2e-
Cu → Cu^{2+} + 2e-
Ag → Ag$^+$ + e-

NO_2:
Brown gas, pungent odor

NO_2 + H$_2$O → NO$_3^-$ + 2 H$^+$ + e-

NO:
Colorless gas, pleasant odor

NO + 2 H$_2$O → NO$_3^-$ + 4 H$^+$ + 3 e-
Au → Au^{3+} + 3 e-
Pt → Pt^{2+} + 2 e-

Increase in ease of oxidation

PROCEDURE

A. Reaction between the elements Copper and Sulfur

Samples of copper (I) sulfide and copper (II) sulfide synthesized in the previous lab experiment will be provided to you.

Copper (II) sulfide, CuS, is insoluble in aqueous ammonia, NH_3 (that is, it does not react with NH_3), while copper (I) sulfide, Cu_2S, dissolves (that is, reacts) to give a blue solution with NH_3. Place a small portion of your product in a test tube and add 2 mL of 6 M NH_3 in the hood. Heat gently with a hotplate.

1. Does your product react with NH_3?
2. Suggest a possible formula for the product.
3. Write a reaction showing the formation of your proposed product:

$$Cu\ (s) + S_8\ (s) \longrightarrow\ ?$$
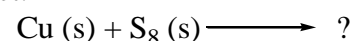
B. Oxidation-Reduction Reactions

Many metals react with acids to liberate hydrogen and form the metal salt of the acid. The noble metals do not react with acids to produce hydrogen. Some of the "unreactive" metals do react though with nitric acid, HNO_3 (see **Table 5.2**); however, in these cases gases that are oxides of nitrogen are formed rather than hydrogen.

Add a small piece of zinc to a test tube containing 2 mL of 6 M HCl and note what happens.
4. Record your observations.
5. Suggest possible products for the observed reaction: $Zn\ (s) + HCl\ (aq) \longrightarrow\ ?$
    Place a 1-in. piece of copper wire in a clean test tube; add 2 mL of 6 M HCl and note if a reaction occurs.
6. Record your observations.
7. Is Cu an active or an inactive metal?

WHILE HOLDING A CLEAN TEST TUBE IN THE HOOD, place a 1-in. piece of copper wire in it and add 1 mL of concentrated nitric acid, HNO_3.
8. Record your observations.
9. Is the gas colored?
10. Suggest a formula for the gas.
11. After the reaction has proceeded for 5 min, carefully add 5 mL of water. Based upon the color of the solution what substance is present in solution?

Potassium permanganate, $KMnO_4$, is an excellent oxidizing agent in acidic media. The permanganate ion is purple and is reduced to the manganous ion, Mn^{2+}, which has a very faint, pink color.
WHILE HOLDING A TEST TUBE IN THE FUME HOOD, add one or two drops of potassium permanganate, $KMnO_4$, solution to 1 mL of 6 M HCl.
12. Record your observations.
13. Note the color of the gas evolved.
14. Based on the color and familiar odor of the gas, what is the gas?

C. Metathesis Reactions

Additional observations are needed before equations can be written for the reaction above, but we see that we can identify some of the products. The remaining reactions are simple, and you

will be able, from available information, not only to identify products but also to write equations. A number of reactions may be represented by equations of the following type:

$$\mathbf{AB} + \mathbf{CD} \longrightarrow \mathbf{AD} + \mathbf{CB}$$

These are called double-decomposition, or *metathesis,* reactions. This type of reaction involves the exchange of atoms or groups of atoms between interacting substances. The following is a specific example:

$$NaCl\ (aq) + AgNO_3\ (aq) \longrightarrow AgCl\ (s) + NaNO_3\ (aq)$$
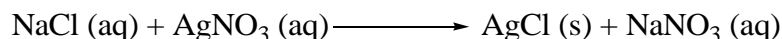
Place a small sample of sodium carbonate, Na_2CO_3, in a test tube and add several drops of 6 M HCl.
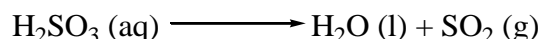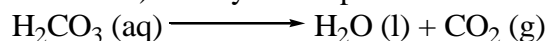
15. Record your observations.

16. Note the odor and color of the gas that forms.

17. What is the evolved gas?

18. Write an equation for the observed reaction: $HCl\ (aq) + Na_2CO_3\ (aq) \longrightarrow$

(NOTE: In this reaction the products must have H, Cl, Na, and O atoms in some new combinations, but no other elements can be present)

Note that H_2CO_3 (carbonic acid) and H_2SO_3 (sulfurous acid) readily decompose as follows:

$$H_2CO_3\ (aq) \longrightarrow H_2O\ (l) + CO_2\ (g)$$
$$H_2SO_3\ (aq) \longrightarrow H_2O\ (l) + SO_2\ (g)$$

To 1 mL of 0.1 M lead nitrate, $Pb(NO_3)_2$, solution in a clean test tube, add a few drops of 6 M HCl.

19. Record your observations.

20. What is the precipitate?

21. Write an equation for the reaction $HCl\ (aq) + Pb(NO_3)_2\ (aq) \longrightarrow$?

To 1 mL of 0.1 M barium chloride ($BaCl_2$) solution, add 2 drops of 1 M potassium chromate (K_2CrO_4) solution.

22. Record your observations.

23. What is the precipitate?

24. Write an equation for the reaction $BaCl_2\ (aq) + K_2CrO_4\ (aq) \longrightarrow$?

To 1 mL of 0.1 M barium chloride, $BaCl_2$, solution add several drops of 3 M ammonium carbonate, $(NH_4)_2CO_3$, solution in a test tube.

25. What is the precipitate?

26. Write an equation for the reaction $(NH_4)_2CO_3\ (aq) + BaCl_2\ (aq) \longrightarrow$?

Use a centrifuge to separate quickly the precipitate from the liquid phase and decant (that is, pour off) the excess liquid. Add 1 mL of water to the test tube, shake it, and isolate the solid again by using the centrifuge. Pour off the excess liquid. Finally, to the remaining solid, add several drops of 6 M HCl.

27. Record your observations.

28. Note the odor.

29. What is the evolved gas? (recall the reaction in step 26 of this experiment)

30. Write the equation of this reaction.

REVIEW QUESTIONS

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

1. Before a chemical equation can be written, what must you know?

2. What observations might you make that suggest a chemical reaction has occurred?

3. How could you distinguish between NO_2 and NO?

4. Define metathesis reactions. Give an example.

5. What is a precipitate?

6. Balance these equations:

$$KBrO_3 \text{ (s)} \longrightarrow KBr \text{ (s)} + O_2 \text{ (g)}$$

$$MnBr_2 \text{ (aq)} + AgNO_3 \text{ (aq)} \longrightarrow Mn(NO_3)_2 \text{ (aq)} + AgBr \text{ (s)}$$
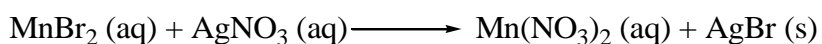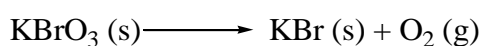
7. How could you distinguish between the gases H_2 and H_2S?

8. Using water, how could you distinguish between the white solids KCl and $PbCl_2$?

9. Write equations for the decomposition of H_2CO_3 (*aq*) and H_2SO_3 (*aq*).

Experiment 5

Chemical Reactions

Name(s) _____

Date _____   Laboratory Instructor _____

REPORT SHEET

A. Reaction between the elements Copper and Sulfur

1._____

2._____

3. $Cu\ (s) + S_8\ (s) \longrightarrow$

B. Oxidation-Reduction Reactions

4._____

5. $Zn\ (s) + HCl\ (aq) \longrightarrow$

6._____

7._____

8._____

9._____

10._____

11._____

12._____

13._____

14._____

C. Metathesis Reactions

15_____

16._____

17._____

18. $HCl\ (aq) + Na_2CO_3\ (aq) \longrightarrow$

19._____

20._____

21. $HCl\ (aq) + Pb(NO_3)_2\ (aq) \longrightarrow$

22._____

23. _____

24. $BaCl_2\ (aq) + K_2CrO_4\ (aq) \longrightarrow$

25._____

26. $(NH_4)_2CO_3\ (aq) + BaCl_2\ (aq) \longrightarrow$

27._____

28._____

29. _____

30. _____

EXPERIMENT 6

Vinegar Analysis

OBJECTIVE
To determine the percentage by mass of acetic acid in vinegar.

Relates to chapter 4 of "Chemistry the Central Science, 12th Ed.".

APPARATUS AND CHEMICALS

50 mL beaker	Ring stand and buret clamp
100 mL beaker	0.200 M NaOH
2 Erlenmeyer flasks	Red and white vinegar
Funnel	Phenolphthalein
25 mL graduated cylinder	

INTRODUCTION
Household vinegar is 4-5% (by mass) acetic acid, CH_3COOH, solution. Generally, caramel flavoring and coloring are also added to make the product esthetically more appealing. This experiment compares the acetic acid concentrations in at least two vinegars.
A titrimetric analysis is used to determine the percent by mass of acetic acid in vinegar. A measured mass of vinegar is titrated to the phenolphtalein end point with a measured volume of a standardized sodium hydroxide solution. As the volume and molar concentration of the standardized NaOH solution are known, the moles of NaOH used for the analysis are also known.
The moles of CH_3COOH are calculated from the balanced equation:

$$CH_3COOH \text{ (aq)} + NaOH \text{ (aq)} \longrightarrow CH_3COONa \text{ (aq)} + H_2O \text{ (l)} \tag{7.1}$$
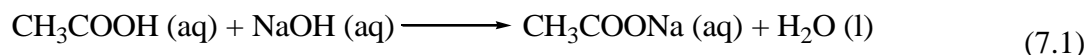
The mass of CH_3COOH in the vinegar is calculated from the measured moles of CH_3COOH and its molar mass, 60.05 g /mol:

$$mass_{CH_3COOH} = n_{CH_3COOH} \times M_{CH_3COOH} \tag{7.2}$$

Finally, the percent by mass of CH_3COOH in vinegar is calculated:

$$\% \ by \ mass_{CH_3COOH} = \frac{mass_{CH_3COOH} \ (g)}{mass_{vinegar} \ (g)} \times 100 \tag{7.3}$$

EXPERIMENTAL PROCEDURE
Procedure Overview: Samples of two vinegars are analysed for the amount of acetic acid in the sample. A titration set up is used for the analysis, using standardized NaOH solution as the titrant and phenolphtalein as the indicator. Stoichiometry calculations determine the percent of acetic acid in the vinegars.

Two samples of two vinegars are to be analyzed. Obtain about 150 mL of a standardized NaOH solution prepared by the lab technician. Record the *exact* molar concentration of the NaOH solution. Your instructor will advise you.

Clean at least two 125- or 250-mL Erlenmeyer flasks. When dry, label each flask and measure its mass (±0.01 g).

1. Volume of vinegar.

The volumes of the red and white vinegars that should be analyzed are 5 mL and 3 mL respectively.

2. Prepare the vinegar Sample. Add the volume of one brand of vinegar to a clean flask with a previously measured mass (±0.01 g). Measure the combined mass of the flask and sample. Add 2 drops of phenolphthalein to the solution and rinse the wall of the flask with 20 mL of deionized water.

3. Prepare the buret and titration setup. Rinse a clean 50-mL buret with the standardized NaOH solution, making certain no drops cling to the inside wall. Fill the buret with the standardized NaOH solution, eliminate all air bubbles in the buret tip, and, after 30 seconds, read (use the proper technique) and record (±0.02 mL) the initial volume. Place a white sheet of paper beneath the flask containing the vinegar sample.

4. Titrate the Vinegar Sample. Slowly add the NaOH solution from the buret to the acid, swirling the flask (with the proper hand) after each addition. Occasionally, rinse the wall of the flask with water from your wash bottle. Continue addition of the NaOH titrant until the endpoint is reached. The endpoint in the titration should be within one-half drop of a slight pink colour. Be careful not to surpass the endpoint. The colour should persist for 30 seconds. After 30 seconds, read (±0.02 mL) and record the final volume of NaOH titrant in the buret.

5. Repeat with the Same Vinegar. Refill the buret and repeat the titration *at least* once more with another sample of the same vinegar.

6. Repeat with Another Vinegar. Perform two analyses of the other vinegar (white or red) and determine its acetic acid content.

Disposal: All test solutions and the NaOH solution in the buret can be discarded in the sink, followed by a generous flow of water.

Experiment 6

Vinegar analysis

Name(s) _____

Date _____ Laboratory Instructor _____

REPORT SHEET

Molar concentration of NaOH: _____mol/L

	Red Vinegar		White Vinegar	
	Trial 1	*Trial 2*	*Trial 1*	*Trial 2*
Data				
1. Volume of vinegar	~ 5 mL	~ 5 mL	~ 3 mL	~ 3 mL
2. Mass of flask (g)	_____	_____	_____	_____
3. Mass of flask and vinegar (g)	_____	_____	_____	_____
4. Mass of vinegar (g)	_____	_____	_____	_____
5. Buret reading of NaOH, *initial* (mL)	0	0	0	0
6. Buret reading of NaOH, *final* (mL)	_____	_____	_____	_____
7. Volume of NaOH used (mL)	_____	_____	_____	_____
Calculations				
1. Amount of NaOH added (mol)	_____	_____	_____	_____
2. Amount of CH_3COOH in vinegar (mol)	_____	_____	_____	_____
3. Mass of CH_3COOH in vinegar (g)	_____	_____	_____	_____
4. Percent by mass of CH_3COOH in vinegar	_____	_____	_____	_____
5. Average percent by mass of CH_3COOH in vinegar	_____		_____	

Ⴅ *show calculations for "White vinegar – Trial 1" overleaf*

EXPERIMENT 7

Reactions in Aqueous Solutions:
Metathesis Reactions and Net Ionic Equations

OBJECTIVE

To become familiar with writing equations for metathesis reactions, including net ionic equations.

Relates to chapter 4 of "Chemistry the Central Science, 12th Ed.".

APPARATUS AND CHEMICALS

Small test tubes (12)

1.0 M sodium acetate

0.1 M potassium chloride

1.0 M sodium carbonate

0.1 M sodium phosphate

1.0 M hydrochloric acid

0.1 M nickel chloride

1.0 M ammonium chloride

0.1 M lead nitrate

0.1 M copper (II) sulfate

0.1 M barium chloride

1.0 M sulfuric acid

0.1 M silver nitrate

0.1 M cadmium chloride

1.0 M sodium hydroxide

0.1 M sodium nitrate

- ALL SOLUTIONS SHOULD BE PROVIDED IN DROPPER BOTTLES -

DISCUSSION

In Experiment 4 we briefly encountered metathesis, or double decomposition, reactions. We will now examine these reactions in more detail, recognising the ionic character of the species in solution. You may recall that metathesis reactions have the general form

$$AB + CD \longrightarrow AD + CB \qquad [1]$$

This kind of reaction is fairly common, especially in aqueous solution, where the cations and anions of the substances involved exchange partners. The reaction of barium chloride with silver nitrate is a typical example:

$$BaCl_2 \text{ (aq)} + 2\ AgNO_3 \text{ (aq)} \longrightarrow Ba(NO_3)_2 \text{ (aq)} + 2\ AgCl \text{ (s)} \qquad [2]$$
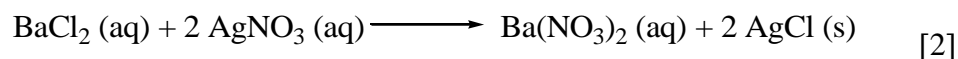
This form of the equation for this reaction is referred to as the *molecular equation*. Because we know that the salts $BaCl_2$ $AgNO_3$, and $Ba(NO_3)_2$ are strong electrolytes and are completely dissociated in solution, we can more realistically write the equation as follows:

$$Ba^{2+} \text{ (aq)} + 2\ Cl^- \text{ (aq)} + 2\ Ag^+ \text{ (aq)} + 2\ NO_3^- \text{ (aq)} \longrightarrow Ba^{2+} \text{ (aq)} + 2\ NO_3^- \text{ (aq)} + 2\ AgCl \text{ (s)} \qquad [3]$$
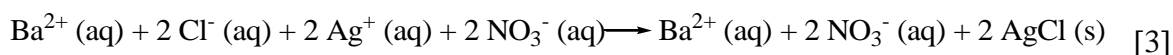
This form is known as the *ionic equation*. Reaction [2] occurs because the insoluble substance AgCl precipitates out of solution. The other product, barium nitrate, is soluble in water and

remains in solution. We see that Ba^{2+} and NO_3^- ions appear on both sides of the equation and thus do not enter into the reaction. Such ions are called
spectator ions. If we eliminate or omit them from both sides, we obtain the net ionic equation

$$Ag^+ \text{ (aq)} + Cl^- \text{ (aq)} \longrightarrow AgCl \text{ (s)} \qquad [4]$$

This equation focuses our attention on the salient feature of the reaction: the formation of the precipitate AgCl. It tells us that solutions of any soluble Ag^+ salt and any soluble Cl^- salt, when mixed, will form insoluble AgCl. When writing net ionic equations, remember that only *strong electrolytes* are written in the ionic form. Solids, gases, nonelectrolytes, and weak electrolytes are written in the molecul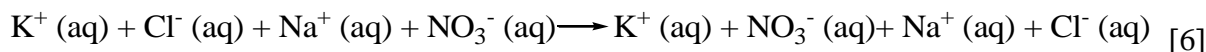ar form. Frequently the symbol *(aq)* is omitted from ionic equations. The symbols *(g)* for gas and *(s)* for solid should not be omitted. Thus equation [4] can be written

$$Ag^+ + Cl^- \longrightarrow AgCl \text{ (s)} \qquad [5]$$

Consider mixing solutions of KCl and $NaNO_3$. The ionic equation for the reaction is

$$K^+ \text{ (aq)} + Cl^- \text{ (aq)} + Na^+ \text{ (aq)} + NO_3^- \text{ (aq)} \longrightarrow K^+ \text{ (aq)} + NO_3^- \text{ (aq)} + Na^+ \text{ (aq)} + Cl^- \text{ (aq)} \qquad [6]$$

Because all the compounds are water-soluble and are strong electrolytes, they have been written in the ionic form. They completely dissolve in water. If we eliminate spectator ions from the equation, nothing remains. Hence, there is no reaction:

$$K^+ \text{ (aq)} + Cl^- \text{ (aq)} + Na^+ \text{ (aq)} + NO_3^- \text{ (aq)} \longrightarrow \text{ no reaction} \qquad [7]$$
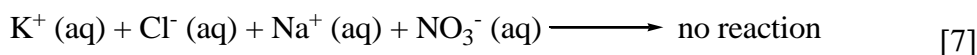
Metathesis reactions occur when a precipitate, a gas, a weak electrolyte, or a non-electrolyte is formed. The following equations are further illustrations of such processes.

• **FORMATION OF A GAS**

Molecular equation:

$$2 \text{ HCl (aq)} + Na_2S \text{ (aq)} \longrightarrow 2 \text{ NaCl (aq)} + H_2S \text{ (g)}$$

Ionic equation:

$$2 H^+ \text{ (aq)} + 2 Cl^- \text{ (aq)} + 2 Na^+ \text{ (aq)} + S^{2-} \text{ (aq)} \longrightarrow 2 Na^+ \text{ (aq)} + 2 Cl^- \text{ (aq)} + H_2S \text{ (g)}$$

Net ionic equation:

$$2 H^+ \text{ (aq)} + S^{2-} \text{ (aq)} \longrightarrow H_2S \text{ (g)} \quad \text{or} \quad 2 H^+ + S^{2-} \longrightarrow H_2S \text{ (g)}$$
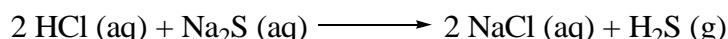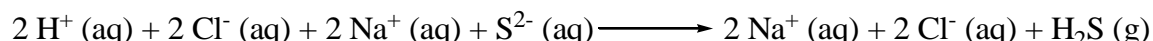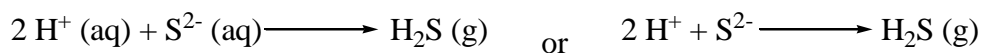
• **FORMATION OF WEAK ELECTROLYTE**

Molecular equation:

$$HNO_3 \text{ (aq)} + NaOH \text{ (aq)} \longrightarrow H_2O \text{ (l)} + NaNO_3 \text{ (aq)}$$

Ionic equation:

$$H^+ \text{ (aq)} + NO_3^- \text{ (aq)} + Na^+ \text{ (aq)} + OH^- \text{ (aq)} \longrightarrow H_2O \text{ (l)} + Na^+ \text{ (aq)} + NO_3^- \text{ (aq)}$$

Net ionic equation:

$$H^+ (aq) + OH^- (aq) \longrightarrow H_2O (l)$$
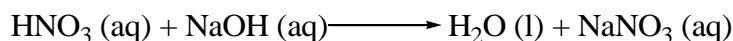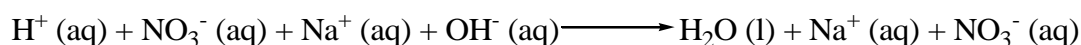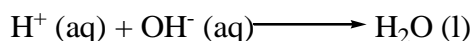
In order to decide if a reaction occurs, we need to be able to determine whether or not a precipitate, a gas, a non-electrolyte or a weak electrolyte will be formed. The following brief discussion is intended to aid you in this regard. **Table 8.1** summarizes solubility rules and should be consulted while performing this experiment.

TABLE 8.1 Solubility Rules

Water-soluble salts

Na^+, K^+, NH_4^+	All sodium, potassium, and ammonium salts are soluble
NO_3^-, ClO_3^-, CH_3COO^-	All nitrates, chlorates, and acetates are soluble.
Cl^-	All chlorides are soluble except $AgCl$, Hg_2Cl_2, and $PbCl_2{}^a$.
Br^-	All bromides are soluble except $AgBr$, Hg_2Br_2, $PbBr_2{}^a$ and $HgBr_2{}^a$.
I^-	All iodides are soluble except AgI, Hg_2I_2, PbI_2, and HgI_2.
$SO_4{}^{2-}$	All sulfates are soluble except $CaSO_4{}^a$, $SrSO_4$, $BaSO_4$, Hg_2SO_4, $PbSO_4$, and Ag_2SO_4.

Water-insoluble salts

$CO_3{}^{2-}$, $SO_3{}^{2-}$, $PO_4{}^{3-}$, $CrO_4{}^{2-}$	All carbonates, sulfites, phosphates, and chromates are insoluble except those of alkali metals and NH_4^+.
OH^-	All hydroxides are insoluble except those of alkali metals and $Ca(OH)_2{}^a$, $Sr(OH)_2{}^a$, and $Ba(OH)_2$
S^{2-}	All sulfides are insoluble except those of the alkali metals, alkaline earths, and NH_4^+.

(a) slightly soluble.

The common gases are CO_2, SO_2, H_2S, and NH_3. Carbon dioxide and sulfur dioxide may be regarded as resulting from the decomposition of their corresponding weak acids, which are initially formed when carbonate and sulfite salts are treated with acid:
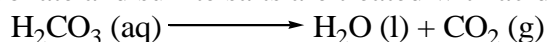
$$H_2CO_3 (aq) \longrightarrow H_2O (l) + CO_2 (g)$$

and

$$H_2SO_3 (aq) \longrightarrow H_2O (l) + SO_2 (g)$$
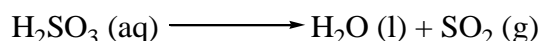
Ammonium salts form NH_3 when they are treated with strong bases:

$$NH_4^+ (aq) + OH^- (aq) \longrightarrow NH_3 (g) + H_2O (l)$$
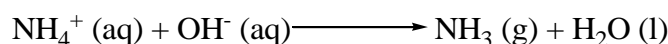
Which are the weak electrolytes? The easiest way of answering this question is to identify all of the strong electrolytes, and if the substance does not fall in that category it is a weak electrolyte. Note that water is a very weak electrolyte. Strong electrolytes are summarized in **Table 8.2**.

In this experiment, you will study some metathesis reactions. In some instances it will be very evident that a reaction has occurred, while in others it will not be so apparent. In the doubtful case, use the guidelines above to decide whether or not a reaction has taken place. You will be

given the names of the compounds to use but not their formulas. This is being done deliberately to give practice in writing formulas from names.

TABLE 8.2 Strong Electrolytes

Salts	All common soluble salts
Acids	$HClO_4$, HCl, HBr, HI, HNO_3, and H_2SO_4, are strong electrolytes. All others are weak.
Bases	Alkali metal hydroxides, $Ca(OH)_2$, $Sr(OH)_2$, and $Ba(OH)_2$ are strong electrolytes. All others are weak.

PROCEDURE

The report sheet lists 16 pairs of chemicals that are to be mixed. Use about 1 mL of the reagents to be combined as indicated on the report sheet. Mix the solutions in small test tubes and record your observations on the report sheet. If there is no reaction, write *N.R.*

The reactions need not be carried out in the order listed. Congestion at the reagent shelf can be avoided if everyone does not start with reagents for reaction 1.

REVIEW QUESTIONS

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

1. Write molecular, ionic, and net ionic equations for the reactions that occur, if any, when solutions of the following substances are mixed:
   (a)   nitric acid and barium carbonate
   (b)   zinc chloride and lead nitrate
   (c)   acetic acid and sodium hydroxide
   (d)   calcium nitrate and sodium carbonate
   (e)   ammonium chloride and potassium hydroxide

2. Which of the following are not water-soluble: $Ba(NO_3)_2$, $FeCl_3$, $CuCO_3$, $CuSO_4$, ZnS, $ZnSO_4$?

3. Which of the following are strong electrolytes: $BaCl_2$, $AgNO_3$, HCl, HNO_3, CH_3COOH?

4. Which of the following aqueous solutions are weak electrolytes: HNO_3, HF, HCl, NH_3, NaOH?

5. For each of the following water-soluble compounds, indicate the ions formed in an aqueous solution: NaI, K_2SO_4, NaCN, $Ba(OH)_2$, $(NH_4)_2SO_4$.

6. Write a balanced chemical equation showing how you could prepare each of the following salts from an acid-base reaction: $NaNO_3$, KCl, $BaSO_4$.

Experiment 7

Reactions in Aqueous Solutions:
Metathesis Reactions and Net Ionic Equations

Name(s) _____

Date _____ Laboratory Instructor _____

REPORT SHEET

---

1. Copper (II) sulfate + sodium carbonate

Observations_____

Molecular equation_____

Ionic equation_____

Net ionic equation_____

Copper (II) : Cu^{2+}
Sodium : Na^+
Sulfate : SO_4^{2-}
Carbonate : CO_3^{2-}

---

2. Copper (II) sulfate + barium chloride

Observations_____

Molecular equation_____

Ionic equation_____

Net ionic equation_____

Copper (II) : Cu^{2+}
Barium : Ba^{2+}
Sulfate : SO_4^{2-}
Chloride : Cl^-

---

3. Copper (II) sulfate + sodium phosphate

Observations_____

Molecular equation_____

Ionic equation_____

Net ionic equation_____

Copper (II) : Cu^{2+}
Sodium : Na^+
Sulfate : SO_4^{2-}
Phosphate : PO_4^{3-}

---

4. Sodium carbonate + sulfuric acid

Observations_____

Molecular equation_____

Ionic equation_____

Net ionic equation_____

Sodium : Na^+
Hydrogen : H^+
Carbonate : CO_3^{2-}
Sulfate : SO_4^{2-}

---

5. Sodium carbonate + hydrochloric acid

Observations_____

Molecular equation_____

Ionic equation_____

Net ionic equation_____

Sodium : Na$^+$
Hydrogen : H$^+$
Carbonate : CO$_3$$^{2-}$
Chloride : Cl$^-$

6. Cadmium chloride + sodium hydroxide

Observations_____

Molecular equation_____

Ionic equation_____

Net ionic equation_____

Cadmium : Cd^{2+}
Sodium : Na$^+$
Chloride : Cl$^-$
Hydroxyde : OH$^-$

7. Nickel chloride + silver nitrate

Observations_____

Molecular equation_____

Ionic equation_____

Net ionic equation_____

Nickel : Ni^{2+}
Silver: Ag$^+$
Chloride : Cl$^-$
Nitrate : NO$_3$$^-$

8. Nickel chloride + sodium carbonate

Observations_____

Molecular equation_____

Ionic equation_____

Net ionic equation_____

Nickel : Ni^{2+}
Sodium: Na$^+$
Chloride : Cl$^-$
Carbonate : CO$_3$$^{2-}$

9. Hydrochloric acid + sodium hydroxide

Observations_____

Molecular equation_____

Ionic equation_____

Net ionic equation_____

Hydrogen : H^+
Sodium: Na^+
Chloride : Cl^-
Hydroxyde : OH^-

---

10. Ammonium chloride + sodium hydroxide

Observations_____

Molecular equation_____

Ionic equation_____

Net ionic equation_____

Ammonium : NH_4^+
Sodium: Na^+
Chloride : Cl^-
Hydroxyde : OH^-

---

11. Sodium acetate + hydrochloric acid

Observations_____

Molecular equation_____

Ionic equation_____

Net ionic equation_____

Sodium : Na^+
Hydrogen : H^+
Acetate : CH_3COO^-
Chloride : Cl^-

---

12. Lead nitrate + sulfuric acid

Observations_____

Molecular equation_____

Ionic equation_____

Net ionic equation_____

Lead: Pb^{2+}
Hydrogen: H^+
Nitrate : NO_3^-
Sulfate : SO_4^{2-}

13. Potassium chloride + sodium nitrate

Observations_____

Molecular equation_____

Ionic equation_____

Net ionic equation_____

Potassium:	K^+
Sodium:	Na^+
Chloride :	Cl^-
Nitrate :	NO_3^-

14. Sodium sulfide + hydrochloric acid

Observations__*DO NOT PERFORM THIS REACTION (sodium sulfide: foul-smelling chemical)*___

Molecular equation_____

Ionic equation_____

Net ionic equation_____

Sodium:	Na^+
Hydrogen :	H^+
Sulfide :	S^{2-}
Chloride :	Cl^-

15. Sodium sulfide + cadmium chloride

Observations__*DO NOT PERFORM THIS REACTION (sodium sulfide: foul-smelling chemical)*___

Molecular equation_____

Ionic equation_____

Net ionic equation_____

Cadmium :	Cd^{2+}
Sodium :	Na^+
Chloride :	Cl^-
Sulfide :	S^{2-}

16. Sodium sulfide + lead nitrate

Observations__*DO NOT PERFORM THIS REACTION (sodium sulfide: foul-smelling chemical)*___

Molecular equation_____

Ionic equation_____

Net ionic equation_____

Lead:	Pb^{2+}
Sodium:	Na^+
Nitrate :	NO_3^-
Sulfide :	S^{2-}

EXPERIMENT 8

Oxidation Reduction Titrations:
Analysis of Bleach

OBJECTIVE

To show how redox reactions can be used to determine the amount of hypochlorite in household bleach.

Relates to chapter 4 of "Chemistry the Central Science, 12th Ed.".
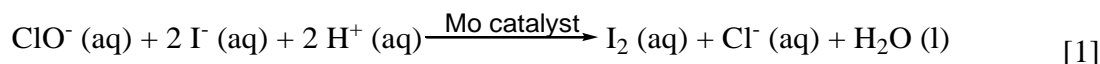
APPARATUS AND CHEMICALS

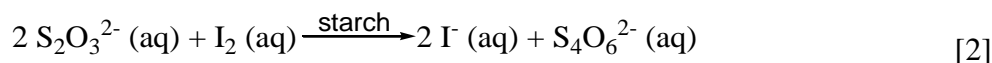|	
500-mL Erlenmeyer flask	100-mL graduated cylinder
50-mL burets (2)	3 M H$_2$SO$_4$
250-mL Erlenmeyer flasks (3)	3 M KI
buret clamp	0.05 M Na$_2$S$_2$O$_3$
ring stand	starch indicator (freshly prepared)
balance	3% ammonium molybdate
wash bottle	bleach unknown
6-in. test tubes (3)	

DISCUSSION

Laundering white clothes to remove dirt and stains is an everyday chore and is usually accomplished with the aid of bleach. The effectiveness of a bleach to whiten and remove stains is related to its oxidizing (bleaching) strength. The use of detergents helps to remove grease by an emulsification action, while agitation helps loosen grime and dirt. Most liquid "chlorine" bleaches such as Javel contains the hypochlorite ion, ClO$^-$ as the oxidizing agent. Hypochlorite is generally present as the sodium salt, NaOCl or the calcium salt, Ca(OCl)$_2$. Nonchlorine bleaches that are used in washing colored as well as white fabrics utilize the oxidative properties of hydrogen peroxide.

This experiment illustrates how redox reactions can be used to quantitatively determine the amount of oxidizing agent in liquid hypochlorite bleaches.

Two redox reactions are involved in this experiment in the analysis of the oxidizing capacity of a liquid bleach. Initially you will add an excess of potassium iodide solution to the bleach. The iodide ions, I$^-$, are oxidized to iodine I$_2$ after the solution has been acidified:

$$ClO^- \text{ (aq)} + 2\ I^- \text{ (aq)} + 2\ H^+ \text{ (aq)} \xrightarrow{\text{Mo catalyst}} I_2 \text{ (aq)} + Cl^- \text{ (aq)} + H_2O \text{ (l)} \qquad [1]$$

(NOTE: In the presence of a strong acid the hypochlorite ion is converted to hypochlorous acid. Why?) The iodine that is formed is then titrated with a standardized sodium thiosulfate, Na$_2$S$_2$O$_3$ solution that quantitatively reduces the iodine to iodide as follows:

$$2\ S_2O_3^{2-} \text{ (aq)} + I_2 \text{ (aq)} \xrightarrow{\text{starch}} 2\ I^- \text{ (aq)} + S_4O_6^{2-} \text{ (aq)} \qquad [2]$$

Starch is used as the indicator for this reaction; the starch solution is not added until the dark brownish color, due to the iodine, has changed to a pale yellow. When the starch is added the yellow color changes to a blue-black. The end point in the titration is reached when a drop of

the thiosulfate causes the solution to become colorless. Starch and iodine (actually the triodide ion, I_3^-) form a blue-black complex and if the starch is added too soon in the titration, the formation of the blue-black complex is not easily reversed making the end point very slow and difficult to detect.

$$I_3^- + \text{starch} \longrightarrow \text{starch} - I_3^- \text{ complex} \qquad \text{(blue/black)} \qquad [3]$$

❶ Dark brownish → **❷** Pale yellow — starch → **❸** Blue-black → **❹** Transparent blue → **❺** Colorless

❶ Dark brownish	❷ Pale yellow	❸ Blue-black	❹ Transparent blue	❺ Colorless
Only I_2 is present	I_2 and I_3^- are present	The I_3^-/starch is present	end point is close	end point

Adding Equations [1] and [2] yields Equation [4] that shows for every mol of hypochlorite, two moles of thiosulfate are required. Thus, from the volume of standardized thiosulfate that is required to react with the liberated iodine and the weight of bleach we can calculate the percentage oxidizing agent, by mass. We will assume the oxidizing agent to be NaOCl. This is illustred in Example 9.1.

$$\text{HOCl (aq)} + 2\,S_2O_3^{2-} \text{(aq)} + H^+ \text{(aq)} \longrightarrow Cl^- \text{(aq)} + S_4O_6^{2-} \text{(aq)} + H_2O \text{(l)} \qquad [4]$$

EXAMPLE 9.1

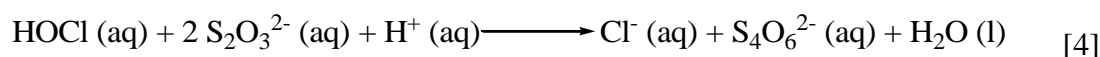A 0.501 g sample of bleach was treated with an excess of KI. The iodine liberated required 10.21 mL of 0.0692 M $Na_2S_2O_3$ for titration. What is the percentage NaOCl in the bleach?

SOLUTION: The number of moles of $Na_2S_2O_3$ used in the titration is twice the number of moles of hypochlorite; or alternatively, the number of moles of hypochlorite that react is one half the number of moles as $Na_2S_2O_3$.

$$n_{NaOCl} = \frac{1}{2} \times n_{Na_2S_2O_3} = \frac{1}{2} \times C_{Na_2S_2O_3} \times V_{Na_2S_2O_3} = \frac{1}{2} \times 0.692 \times 0.01021 = 3.53 \times 10^{-4}\, mol$$
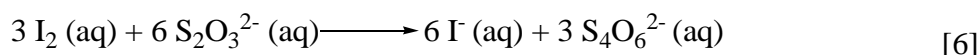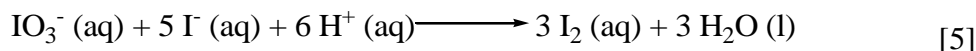
Changing this number of moles to grams:

$$m_{NaOCl} = n_{NaOCl} \times M_{NaOCl} = \left(3.53 \times 10^{-4}\right) \times 74.5 = 0.0263\, g$$

And, the percentage of NaOCl is: $\quad \% NaOCl = \dfrac{0.0263}{0.501} \times 100 = 5.25\,\%$
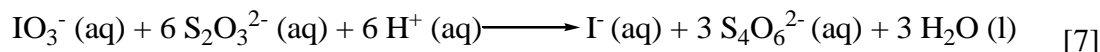
TITRATION OF SODIUM THIOSULFATE

In this experiment you will use an already standardized sodium thiosulfate solution (0.050 M) to analyze a liquid bleach.

Your $Na_2S_2O_3$ solution used to titrate the I_2 formed according to Equation [1] can be standardized using potassium iodate KIO_3 as the primary standard. As in the analysis of the bleach solution described above, tow redox reactions, Equations [5] and [6] are involved in the standardization. Notice in Equation [5] that IO_3^- plays an analogous oxidative role to HOCl in Equation [1].

$$IO_3^- \text{ (aq)} + 5\ I^- \text{ (aq)} + 6\ H^+ \text{ (aq)} \longrightarrow 3\ I_2 \text{ (aq)} + 3\ H_2O \text{ (l)} \qquad [5]$$

$$3\ I_2 \text{ (aq)} + 6\ S_2O_3^{2-} \text{ (aq)} \longrightarrow 6\ I^- \text{ (aq)} + 3\ S_4O_6^{2-} \text{ (aq)} \qquad [6]$$

The standardization procedure is similar to the procedure described above for the analysis of bleach. An excess of KI is allowed to react with a known amount of KIO_3 in acidic solution. The iodine formed according to Equation [5] will be titrated with your $Na_2S_2O_3$ solution using starch as the indicator. Once again, the starch is not added until the iodine solution has turned a pale yellow. The end point in the titration is signaled by the disappearance of the blue-black color.
The stoichiometric relation between the primary standard KIO_3 and $Na_2S_2O_3$ can easily be seen by adding Equations [5] and [6] to give Equation [7]. (NOTE: Equation [6] is obtained by multiplying Equation [2] by the factor 3):

$$IO_3^- \text{ (aq)} + 6\ S_2O_3^{2-} \text{ (aq)} + 6\ H^+ \text{ (aq)} \longrightarrow I^- \text{ (aq)} + 3\ S_4O_6^{2-} \text{ (aq)} + 3\ H_2O \text{ (l)} \qquad [7]$$

Equation [7] shows that for each mole of KIO_3 used in the titration 6 moles of $Na_2S_2O_3$ are required. Example 9.2 illustrates how the concentration of a $Na_2S_2O_3$ solution can be determined using KIO_3 as the primary standard.

EXAMPLE 9.2
What is the concentration of a $Na_2S_2O_3$ solution if 21.21 mL of the solution was required to titrate the iodine formed from 20.95 mL of 0.0100 *M* KIO_3 and excess KI?

SOLUTION: First determine the number of moles of KIO_3 that react:

$$n_{KIO_3} = C_{KIO_3} \times V_{KIO_3} = 0.02095 \times 0.0100 = 2.10 \times 10^{-4}\ mol$$

According to Equation [7], the number of moles of $Na_2S_2O_3$ that react is six times the number of moles of KIO_3:

$$n_{Na_2S_2O_3} = 6 \times n_{KIO_3} = 6 \times 2.10 \times 10^{-4} = 1.26 \times 10^{-3}\ mol$$

Hence, the molarity of the $Na_2S_2O_3$ solution is:

$$C_{Na_2S_2O_3} = \frac{n_{Na_2S_2O_3}}{V_{Na_2S_2O_3}} = \frac{1.26 \times 10^{-3}}{0.02121} = 0.0594\ M$$
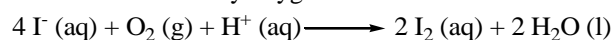
PROCEDURE

It should be noted that solid iodine crystals may form during each titration, as the iodine concentration exceeds the solubility product. All of the iodine must be redissolved and reacted before the titration is complete. As the titration proceeds and the solution becomes pale yellow, the flask should be swirled until all of the iodine is dissolved. Over time, the iodine will clump together and become more and more difficult to dissolve. For this reason, the titrations should be performed as rapidly as practical.

Determination of the Oxidizing Capacity of an Unknown Liquid Bleach

> Liquid bleaches containing sodium hypochlorite are corrosive to the skin. Take care when handling the liquid bleach not to get any on yourself. *If you do, immediately wash the area with a copious amount of water.*

The oxidizing power of an unknown liquid bleach will be determined in the following way: Place a clean test tube into a 100 mL beaker and weigh and record the weight. Add about 0.5 mL bleach to the test tube. Re-weigh and record the weight. The total mass of bleach should be 0.4 - 0.6 g. Pour the bleach into a 250 mL titration flask and rinse the test tube several times with distilled water to ensure that all of the bleach is transferred (the total volume of water added to the flask should be about 25 mL, including the water used to rinse the test tube). Add 3 mL of 3 M KI to the flask and swirl the flask. Add 2 mL of 3 M H_2SO_4 and swirl the flask. Add 5 drops of 3% ammonium molybdate catalyst immediately after the acid is added. The molybdate ion catalyses the reaction between the iodide and oxidizing agent. A deep brown color should appear, indicating the presence of iodine. Fill a buret with 0.05 M sodium thiosulfate. Record the initial and final reading of the buret to the nearest 0.02 mL. After recording the initial buret reading, immediately* begin titration with the sodium thiosulfate solution. As titrant is added, the color of the solution in the flask will fade to light brown and then light yellow. When the solution has reached a light yellow, add 0.5 mL of starch indicator and mix. The solution will turn a dark blue-black color. Slowly continue to add titrant drop by drop. The dark-blue black color will fade and when the solution becomes a transparent bright blue the end point is generally a drop or two away. The end point is the transition from a blue to a colorless solution. Record the final buret reading. Perform two more titrations. From the concentration and volume of the added sodium thiosulfate solution used to titrate the bleach, calculate the weight of sodium hypochlorite, NaOCl, present. For each titration, calculate the strength of the bleach as the effective percentage mass of the bleach that is sodium hypochlorite. Report the average percentage mass of sodium hypochlorite in the bleach. This is the oxidizing capacity of the bleach.

---

*Iodide is oxidized by oxygen in air:

$4\,I^- (aq) + O_2 (g) + H^+ (aq) \longrightarrow 2\,I_2 (aq) + 2\,H_2O (l)$

The reaction is slow in neutral solution but is faster in acid and is accelerated by sunlight. After the solution has been acidified it must be titrated immediately. Moreover, after reaching the end point the solution may darken on standing for an extended length of time.

REVIEW QUESTIONS

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

1. Write the formula for the salts that are oxidizing agents in chlorine bleaches.

2. Complete and balance the following equations

(a) $HOCl + I^- + H^+ \longrightarrow$

(b) $S_2O_3^{2-} + I_2 \longrightarrow$

3. Is the iodate ion, IO_3^- an oxidizing or reducing agent?

4. What is the colour of the iodate-iodide solution being titrated with $Na_2S_2O_3$,
    (a) before the starch solution is added?
    (b) after the starch solution is added?
    (c) at the end point?

5. How many moles of $Na_2S_2O_3$ react with each mole of KIO_3 used in the standardization?

6. If 16.92 mL of a $Na_2S_2O_3$ solution were required to titrated the iodine formed from 20.95 mL of a 0.0111 *M* KIO_3 solution and excess KI, what is the molarity of the $Na_2S_2O_3$ solution?

7. What is meant by the term standard solution?

8. What are the oxidation states of
    (a) chlorine in ClO^-?
    (b) sulfur in $Na_2S_2O_3$?

Experiment 8

Oxidation Reduction Titration: Analysis of Bleach
Determination of the Oxidizing Capacity of an Unknown Liquid Bleach

Name(s) _____

Date _____ Laboratory Instructor _____

Unknown n^o _____

REPORT SHEET

Molarity of $Na_2S_2O_3$: 0.050 M

	Trial 1	**Trial 2**	**Trial 3**
Mass of beaker and test tube (g)	_____	_____	_____
Mass of beaker, test tube and bleach (g)	_____	_____	_____
Mass of unknown bleach (g)	_____	_____	_____
Volume of $Na_2S_2O_3$ (mL) ➔ Final buret reading	_____	_____	_____
➔ Initial buret reading	0	0	0
Mass of NaOCl (g)	_____	_____	_____
Percent NaOC1	_____	_____	_____

⚗ *show calculations for Trial 1:*

EXPERIMENT 9

Bomb calorimetry

OBJECTIVES

To understand what a bomb calorimeter is, how to use it, and why it is useful.

To determine the heat capacity of the calorimeter.

To determine the enthalpy of combustion of an oil sample.

Relates to Chapter 5, "Thermochemistry", of "Chemistry the Central Science, 12th Ed.".

APPARATUS AND CHEMICALS

Parr 1341 oxygen bomb calorimeter

Parr 1108 oxygen bomb

Digital thermometer

Parr 45C10 nickel-chromium fuse wire

Oxygen cylinder, medical grade (99.99%)

Benzoic acid pellets, calorific grade

19V firing mechanism

Amperemeter/Ohmmeter

5kg balance

Iron ring and stand

DISCUSSION

Heat released in a chemical reaction can be determined experimentally by using an adiabatic calorimeter. The reaction must proceed without any side reactions and sufficiently fast that the heat exchange with the surroundings is negligible. In an oxygen bomb calorimeter, the combustion reaction occurs in a closed container under constant volume ("bomb"). The bomb is immersed in a weighed quantity of water and surrounded by an adiabatic shield that serves as a heat insulator (see **Figure 9.1**).

The bomb and the water bath, which are in direct thermal contact, constitute an adiabatic bomb calorimeter. Continuous stirring ensures that heat is distributed evenly in the system. In this experiment an oil sample is combusted and its heat of combustion is determined.

Figure 9.1 Adiabatic bomb calorimeter

The Parr bomb calorimeter (see **Figure 9.2**) is a self-contained instrument used in determination of heats of combustion of certain fuels and pure organic substances. The results obtained are sufficiently precise to make them of extreme importance in most commercial and laboratory procedures concerned with heats of combustion.

Figure 9.2   Parts of the Parr 1341 oxygen bomb calorimeter

The combustion bomb, made of corrosion-resistant metal, holds the sample whose heat of combustion is to be measured. The sample is held in a cup and an ignition wire, used to start the combustion reaction, is attached to the electrodes (see **Figure 9.3**).

      (a)   (b)   (c)

Figure 9.3   (a)   Interior of the bomb vessel.
**   (b)   Schematic of the sample support stand.**
**   (c)   Attachment of the nickel-chrome ignition wire.**

After the sample and the wire have been properly placed in the bomb, it is charged with oxygen gas from a commercial cylinder to the pressure of about 25 atm.

The assembled bomb is then placed in a bucket containing a specified quantity of water (2000.00g). The temperature rise accompanying the combustion is read from a digital thermometer. A stirrer insures an even distribution of the heat in water. The bucket in turn is surrounded by an insulating air space, which prevents, as far as possible, heat leakage to the surroundings.

First, it is necessary to obtain the heat capacity of the calorimeter system (C_{cal}). This is the number of calories necessary to raise the temperature of the entire calorimeter system by 1 °C. This is found by burning a sample material of known heat of combustion. Benzoic acid of high purity is usually employed. The temperature rise due to the sample is noted, and the number of joules of heat released in the combustion is calculated. These two values enable one to calculate the heat capacity of the calorimeter system C_{cal}, in kJ/°C or kcal/°C.

kJ
kJ/°C   °C
$$\Delta H_{comb}(benzoic\ acid) = -C_{cal} \times \Delta T$$

kJ/g
kJ/°C   °C
$$\Delta H_{comb}(benzoic\ acid) = \frac{-C_{cal} \times \Delta T}{m}$$
g

$$\Rightarrow\quad C_{cal} = -\Delta H_{comb}(benzoic\ acid) \times \frac{m}{\Delta T}$$

- 26.434 kJ/g
÷ 4.184
= - 6.318 kcal/g

Knowing that $q_{cal} = C_{cal} \times \Delta T_{cal}$, then we can calculate the heat of combustion of the assigned material.

Example:
In an experiment, 1.50 g of sucrose ($C_{12}H_{22}O_{11}$) is burned and causes the temperature of the bomb calorimeter to rise from 25.00 °C to 27.88 °C. If the heat capacity of the bomb calorimeter C_{cal} is 8.57 kJ/°C, calculate the enthalpy of combustion ΔH_{comb}, expressed in kJ/mol.

kJ/mol
kJ/°C   °C
$$\Delta H_{comb}(sucrose) = \frac{-C_{cal} \times \Delta T}{n}$$
mol

$$\Delta H_{comb}(sucrose) = \frac{-\left(C_{cal} \times \Delta T_{cal}\right)}{\left(\dfrac{m_{sample}}{M_{sample}}\right)} = \frac{-\left(8.57 \times \left(27.88 - 25.00\right)\right)}{\left(\dfrac{1.50}{342.34}\right)} = -5.63 \times 10^3 \; kJ/mol$$

$$\Downarrow \div 4.184$$

$$= -1.35 \times 10^3 \, kcal/mol$$

PROCEDURE

Experiment 1 – Determination of C_{cal}:

You will find an oxygen bomb calorimeter charged with 1.00 g benzoic acid pellet under an oxygen atmosphere (P ≈ 25atm). When ready, stand back and let your instructor fire the bomb; start recording the temperature at 30s intervals by reading the digital thermometer.
The bomb is fired by plugging the 19V battery for 5 seconds. A typical temperature increase during the experiment will be in the range of 4°C.
Record the difference between the highest and lowest temperatures reached, ΔT.
Compute the heat capacity of the calorimeter C_{cal}.

Experiment 2 – Determination of ΔH_{comb} of an oil sample:

Then, the oxygen bomb calorimeter is emptied and charged with 1.00 g of oil sample (M_{oil} = 872.3 g/mol) under an oxygen atmosphere (P ≈ 25atm). When ready, stand back and fire the bomb under the supervision of your lab instructor; start recording the temperature at 30s intervals by reading the digital thermometer.
A typical temperature increase during the experiment will be in the range of 4°C.
Record the difference between the highest and lowest temperatures reached, ΔT.
Compute ΔH_{comb} of this oil by using the value of C_{cal} previously determined in experiment 1.

Note:
In this experiment, several approximations are made to simplify the calculations of C_{cal} and ΔH_{comb}. In a more rigorous procedure, several corrections would have to be applied:
- correction of the temperature rise ΔT
- correction for the heat of formation of HNO_3 and H_2SO_4
- correction for the heat of combustion of the Nickel-chrome fuse wire
Moreover, it is recommended to perform 7 trials to get a representative mean value.

Experiment 9

Bomb calorimetry

Name(s) _____

Date _____ Laboratory Instructor _____

REPORT SHEET

1 - Determination of C_{cal}

mass of benzoic acid pellet =   1.00   g

$T_{initial}$ =   _____ °C

T_{final} =   _____ °C

ΔT =   _____ °C

$\Delta H_{comb}(benzoic\ acid) =$ - 26.434 kJ/g

C_{cal} =   _____ kJ /°C

                    = _____ kcal /°C

⚕ *show calculations overleaf*

2 - Determination of ΔH_{comb} of an oil sample

mass of oil sample =   1.00   g

MW of oil sample =   872.3 g/mol

n of oil sample   =   _____ mol

$T_{initial}$ =   _____ °C

T_{final} =   _____ °C

ΔT =   _____ °C

C_{cal} =   _____ kJ /°C

$\Delta H_{comb}(oil) =$ - _____ kJ/mol

                    = - _____ kcal /mol

⚕ *show calculations overleaf*

EXPERIMENT 10

Flame Test

OBJECTIVE

Identify unknown metal salts by means of the flame test.

Relates to chapter 6 of "Chemistry the Central Science, 12th Ed.".

What is a flame test?

The flame test is used to visually determine the identity of an unknown metal of an ionic salt based on the characteristic color emitted from the salt when exposed to a flame.

How is the test performed?

A small amount of a metal salt is placed on the tip of a spatula previously rinsed with HCl and dried over the flame of a Bunsen burner. Then the spatula is exposed to the blue flame of the Bunsen burner. Students will be able to see the brilliant characteristic colors of each metal ions.

What metals do colors indicate ?

Color	Metal
Red	Lithium compounds.
yellow	Yellowish-Orange: Sodium compounds, even in trace amounts.
White	White-Green: Zinc compounds
Green	Green: Copper compounds
Blue	Azure: Lead compounds Greenish Blue: $CuBr_2$
Violet	Potassium compounds
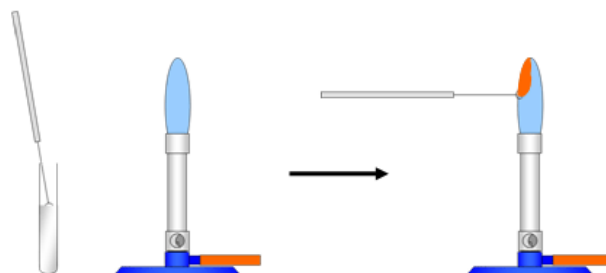
N.B. The value of the flame test is limited by interference from other brighter colors and by ambiguities where certain different metals cause the same flame color. Sodium, in particular, is present in most compounds and will color the flame. Sometimes a colored glass is used to filter out light from one metal. Cobalt glass is often used to filter out the yellow of sodium

PROCEDURE

1. Observe the colors emitted when you ignite a little amount of a metal salt held on a spatula over a Bunsen burner.
   The salts tests contain the following metals: calcium (Ca), sodium (Na), barium (Ba), strontium (Sr), potassium (K), copper (Cu) and zink (Zn).

2. Identify the unknown salts

Experiment 10

Flame Test

Name(s) _____

Date _____ Laboratory Instructor _____

REPORT SHEET

1. What is the color emitted from each of the following substances?

	Metal salt	Color emitted
a)	Calcium	_____
b)	Sodium	_____
c)	Barium	_____
d)	Strontium	_____
e)	Potassium	_____
f)	Copper	_____
g)	Zink	_____

2. Identify the unknown salts (A, B, and C)

A- _____

B- _____

C- _____

3. Is the flame test enough to identify a given metal salt? Explain.

APPENDIX I

Physical Properties of Some Common Laboratory Chemicals

Compound	Density (g/cm^3)	Melting point (^{o}C)	Boiling point (^{o}C)	Solubility			
				Water	Ethanol	Acetone	Cyclohexane
Acetone	0.79	-95	56	s	s	▨	s
Acetamide	1.00	82.3	221	s	s	-	-
Acetanilide	1.22	114	304	-	s	s	s
Anthracene	1.28	216	-	-	-	s	s
Benzamide	1.08	132	290	s	s	-	s
Benzoic acid	1.07	122	249	-	s	s	s
Benzoin	1.31	137	344	-	s	s	-
2-Butanone	0.81	-86	80	s	s	s	s
Cyclohexane	0.79	6.5	81	-	s	s	▨
Cyclohexene	0.81	-104	83	-	s	s	s
Ethanol	0.79	-117	79	s	▨	s	s
Ethyl acetate	0.90	-84	77	s	s	s	s
Heptane	0.68	-91	98	-	s	s	s
n-Hexane	0.66	-95	69	-	s	-	-
Methanol	0.79	-94	65	s	s	s	s
Naphthalene	0.96	80.5	218	-	s	s	s
1-Propanol	0.80	-127	97	s	s	s	s
2-Propanol	0.79	-90	82	s	s	s	s

APPENDIX II

Common polyatomic ions

acetate	$C_2H_3O_2^-$		AsO_3^{3-}	arsenite
ammonium	NH_4^+		AsO_4^{3-}	arsenate
arsenate	AsO_4^{3-}		BO_3^{3-}	borate
arsenite	AsO_3^{3-}		BrO_3^-	bromate
azide	N_3^-		$C_2H_3O_2^-$	acetate
benzoate	$C_7H_5O_2^-$		$C_2O_4^{2-}$	oxalate
borate	BO_3^{3-}		$C_4H_4O_6^{2-}$	tartrate
bromate	BrO_3^-		$C_7H_5O_2^-$	benzoate
carbonate	CO_3^{2-}		ClO^-	hypochlorite
chlorate	ClO_3^-		ClO_2^-	chlorite
chlorite	ClO_2^-		ClO_3^-	chlorate
chromate	CrO_4^{2-}		ClO_4^-	perchlorate
cyanide	CN^-		CN^-	cyanide
dichromate	$Cr_2O_7^{2-}$		CO_3^{2-}	carbonate
dihydrogen phosphate	$H_2PO_4^-$		$Cr_2O_7^{2-}$	dichromate
dihydrogen phosphite	$H_2PO_3^-$		CrO_4^{2-}	chromate
hydrogen carbonate	HCO_3^-		$H_2PO_3^-$	dihydrogen phosphite
hydrogen phosphate	HPO_4^{2-}		$H_2PO_4^-$	dihydrogen phosphate
hydrogen phosphite	HPO_3^{2-}		HCO_3^-	hydrogen carbonate
hydrogen sulfate	HSO_4^-		HPO_3^{2-}	hydrogen phosphite
hydrogen sulfide	HS^-		HPO_4^{2-}	hydrogen phosphate
hydrogen sulfite	HSO_3^-		HS^-	hydrogen sulfide
hydroxide	OH^-		HSO_3^-	hydrogen sulfite
hypochlorite	ClO^-		HSO_4^-	hydrogen sulfate
iodate	IO_3^-		IO_3^-	iodate
manganate	MnO_4^{2-}		MnO_4^-	permanganate
nitrate	NO_3^-		MnO_4^{2-}	manganate
nitrite	NO_2^-		N_3^-	azide
oxalate	$C_2O_4^{2-}$		NH_4^+	ammonium
perchlorate	ClO_4^-		NO_2^-	nitrite
permanganate	MnO_4^-		NO_3^-	nitrate
peroxide	O_2^{2-}		O_2^{2-}	peroxide
phosphate	PO_4^{3-}		OH^-	hydroxide
phosphite	PO_3^{3-}		PO_3^{3-}	phosphite
silicate	SiO_3^{2-}		PO_4^{3-}	phosphate
sulfate	SO_4^{2-}		$S_2O_3^{2-}$	thiosulfate
sulfite	SO_3^{2-}		SCN^-	thiocyanate
tartrate	$C_4H_4O_6^{2-}$		SiO_3^{2-}	silicate
thiocyanate	SCN^-		SO_3^{2-}	sulfite
thiosulfate	$S_2O_3^{2-}$		SO_4^{2-}	sulfate

Common ions

Symbols	Names
Ag^+	silver ion
Al^{3+}	aluminum ion
As^{3-}	arsenide
AsO_4^{3-}	arsenate
Au^+	gold(I) / aurous
Au^{3+}	gold(III) / auric
Ba^{2+}	barium ion
Be^{2+}	beryllium ion
BO_3^{3-}	Borate
Br^-	Bromide
BrO_3^-	Bromate
BrO^-	Hypobromite
CH_3COO^-	Acetate
$C_2O_4^{2-}$	Oxalate
Ca^{2+}	calcium ion
Cl^-	Chloride
ClO_2^-	Chlorite
ClO_3^-	Chlorate
ClO_4^-	Perchlorate
ClO^-	Hypochlorite
CN^-	Cyanide
CO_3^{2-}	Carbonate
Co^{2+}	cobalt(II) / cobaltous
Co^{3+}	cobalt(III) / cobaltic
$Cr_2O_7^{2-}$	Dichromate
Cr^{2+}	chromium(II) / chromous
Cr^{3+}	chromium(III) / chromic
CrO_4^{2-}	Chromate
Cu^+	copper(I) / cuprous
Cu^{2+}	copper(II) / cupric
F^-	Fluoride
Fe^{2+}	iron(II) / ferrous
Fe^{3+}	iron(III) / ferric
$H_2PO_4^-$	dihydrogen phosphate
H^+	hydrogen ion
H^-	Hydride
$HC_2O_4^-$	hydrogen oxalate (binoxalate)
HCO_3^-	hydrogen carbonate (bicarbonate)
Hg_2^{2+}	mercury(I) / mercurous

Hg^{2+}	mercury(II) / mercuric
HPO_4^{2-}	hydrogen phosphate
HS^-	hydrogen sulfide
HSO_3^-	hydrogen sulfite (bisulfite)
HSO_4^-	hydrogen sulfate (bisulfate)
I^-	Iodide
IO_3^-	Iodate
IO_4^-	Periodate
IO^-	Hypoiodite
Mn^{2+}	manganese(II) / manganous
Mn^{3+}	manganese(III) / manganic
MnO_4^-	Permanganate
N^{3-}	Nitride
NH_2^-	Amide
NH_4^+	ammonium ion
Ni^{2+}	nickel ion
NO_2^-	Nitrite
NO_3^-	Nitrate
O_2^{2-}	Peroxide
O^{2-}	Oxide
OH^-	Hydroxide
P^{3-}	Phosphide
Pb^{2+}	lead(II) / plumbous
Pb^{4+}	lead(IV) / plumbic
PO_3^{3-}	Phosphite
PO_4^{3-}	Phosphate
$S_2O_3^{2-}$	Thiosulfate
S^{2-}	Sulfide
SCN^-	Thiocyanate
Se^{2-}	Selenide
SeO_4^{2-}	Selenate
SiF_6^{2-}	Hexafluorosilicate
SiO_3^{2-}	Silicate
Sn^{2+}	tin(II) / stannous
Sn^{4+}	tin(IV) / stannic
SO_3^{2-}	sulfite
SO_4^{2-}	sulfate
Te^{2-}	telluride
Zn^{2+}	zinc ion

The Periodic Table of the Elements

1																	2
H Hydrogen 1.0079																	**He** Helium 4.003
3 **Li** Lithium 6.941	4 **Be** Beryllium 9.012											5 **B** Boron 10.81	6 **C** Carbon 12.01	7 **N** Nitrogen 14.01	8 **O** Oxygen 16.00	9 **F** Fluorine 19.00	10 **Ne** Neon 20.18
11 **Na** Sodium 22.99	12 **Mg** Magnesium 24.31											13 **Al** Aluminum 26.98	14 **Si** Silicon 28.09	15 **P** Phosphorus 30.97	16 **S** Sulfur 32.06	17 **Cl** Chlorine 35.45	18 **Ar** Argon 39.95
19 **K** Potassium 39.10	20 **Ca** Calcium 40.08	21 **Sc** Scandium 44.96	22 **Ti** Titanium 47.90	23 **V** Vanadium 50.94	24 **Cr** Chromium 52.00	25 **Mn** Manganese 54.94	26 **Fe** Iron 55.85	27 **Co** Cobalt 58.93	28 **Ni** Nickel 58.70	29 **Cu** Copper 63.55	30 **Zn** Zinc 65.38	31 **Ga** Gallium 69.72	32 **Ge** Germanium 72.59	33 **As** Arsenic 74.92	34 **Se** Selenium 78.96	35 **Br** Bromine 79.90	36 **Kr** Krypton 83.80
37 **Rb** Rubidium 85.74	38 **Sr** Strontium 87.62	39 **Y** Yttrium 88.91	40 **Zr** Zirconium 91.22	41 **Nb** Niobium 92.91	42 **Mo** Molybdenum 95.94	43 **Tc** Technetium [98]	44 **Ru** Ruthenium 101.1	45 **Rh** Rhodium 102.9	46 **Pd** Palladium 106.4	47 **Ag** Silver 107.9	48 **Cd** Cadmium 112.4	49 **In** Indium 114.8	50 **Sn** Tin 118.7	51 **Sb** Antimony 121.8	52 **Te** Tellurium 127.6	53 **I** Iodine 126.9	54 **Xe** Xenon 131.3
55 **Cs** Cesium 132.9	56 **Ba** Barium 137.3	57 **La** Lanthanum 138.9	72 **Hf** Hafnium 178.5	73 **Ta** Tantulum 180.9	74 **W** Tungsten 183.9	75 **Re** Rhenium 186.2	76 **Os** Osmium 190.2	77 **Ir** Iridium 192.2	78 **Pt** Platinum 195.1	79 **Au** Gold 197.0	80 **Hg** Mercury 200.6	81 **Tl** Thallium 204.4	82 **Pb** Lead 207.2	83 **Bi** Bismuth 209.0	84 **Po** Polonium [209]	85 **At** Astatine [210]	86 **Rn** Radon [222]
87 **Fr** Francium [223]	88 **Ra** Radium [226]	89 **Ac** Actinium [227]	104 **Rf** Rutherfordium [261]	105 **Db** Dubnium [262]	106 **Sg** Seaborgium [266]	107 **Bh** Bohrium [264]	108 **Hs** Hassium [269]	109 **Mt** Meitnerium [268]	110 **Uun** Ununillium [269]	111 **Uuu** Unununium [272]	112 **Uub** Ununbium [277]	113 **Uut** Ununtrium Unk.	114 **Uuq** Ununquadium [285]	115 **Uup** Ununpentium Unk.	116 **Uuh** Ununhexium [289]	117 **Uus** Ununseptium Unk.	118 **Uuo** Ununoctium [293]

58 **Ce** Cerium 140.1	59 **Pr** Praseodymium 140.9	60 **Nd** Neodymium 144.2	61 **Pm** Promethium [145]	62 **Sm** Samarium 150.4	63 **Eu** Europium 152.0	64 **Gd** Gadolinium 157.3	65 **Tb** Terbium 158.9	66 **Dy** Dysprosium 162.5	67 **Ho** Holmium 164.9	68 **Er** Erbium 167.3	69 **Tm** Thulium 168.9	70 **Yb** Ytterbium 173.0	71 **Lu** Lutetium 175.0
90 **Th** Thorium 232.0	91 **Pa** Protactinium [231]	92 **U** Uranium 238.0	93 **Np** Neptunium [244]	94 **Pu** Plutonium [242]	95 **Am** Americium [243]	96 **Cm** Curium [247]	97 **Bk** Berkelium [247]	98 **Cf** Californium [251]	99 **Es** Einsteinium [252]	100 **Fm** Fermium [257]	101 **Md** Mendelevium [258]	102 **No** Nobelium [259]	103 **Lr** Lawrencium [260]