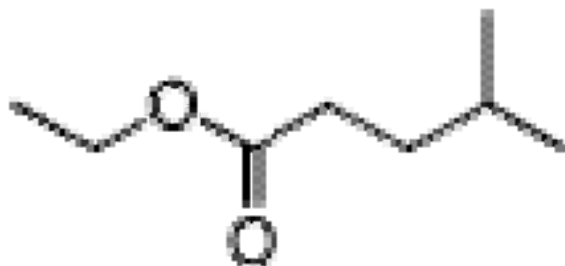# AP Chemistry
Laboratory Manual

Mr. Davis
Auburn Riverside HS

This manual contains the laboratory experiments you will be completing during the course of the school year. It is an ambitious schedule, but one that the College Board deems necessary for you to get the best Advanced Placement Chemistry experience. The College Board publishes a list of required laboratory topics and this compilation of labs is designed to meet those requirements. The experiments are in order according to the schedule included in your syllabus. The laboratories in this manual are taken from the following sources:

Nelson, John H and Kemp, Kenneth C. Laboratory Experiments. Upper Saddle River, NJ: Prentice Hall, Inc, 2000

Hollenberg, J Leland, et al. Chemistry in the Laboratory. New York: Freeman Press, 2000

Flinn Scientific Spectrophotometer Laboratory Manual. Flinn Scientific, 1994

To achieve the ideal lab experience, you will need to be here for all of the experiments and will need to complete the post lab write up. You will have adequate time to complete everything and labs will be discussed in class before and after the experiment is run.

SAFETY: The chemistry laboratory is a place where you will be using compounds that may have adverse reactions to your skin, a place where you will be working with flames, hot glassware, and vigorous reactions. **To ensure your safety, it is absolutely required that you wear safety glasses during every experiment. Failure to do so will result in a grade of "F" or no credit for the lab and no chance of making it up.** Your eyes are far too valuable to you and your future and glasses are provided to protect them. You will also have the option to use aprons and, for certain experiments, protective gloves. You will need to know where the eyewash station, the shower, and the fire extinguisher are located and how to use them.

Food and Drink: Washington State law says that no food or drink may be consumed in the chemistry laboratory. Do NOT bring any food into the classroom. Drinks (your own container) must be left at your desk and not consumed during the laboratory period.

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This is by order, not by page

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29. Oxidation-Reduction Titrations I: Determination of Oxalate
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PERIODIC TABLE OF THE ELEMENTS

Table of Selected Radioactive Isotopes

KEY

- DENSITY at 300 K (g/cm³)
- BOILING POINT, K
- MELTING POINT, K
- ATOMIC NUMBER
- NAME
- SYMBOL
- ATOMIC WEIGHT (2)
- OXIDATION STATES (Bold most stable)
- ELECTRON CONFIGURATION

Example:
- 30 Zn Zinc — 65.409 (2) — [Ar]3d¹⁰4s² — 7.13 / 1180 / 692.73

Estimated Values

NOTES:

(1) Black — solid. Red — gas. Blue — liquid. Outline — synthetically prepared.

(2) Based upon carbon-12. ( ) indicates most stable or best known isotope.

(3) Entries marked with daggers refer to the gaseous state at 273 K and 1 atm and are given in units of g/l.

The A & B subgroup designations, are those recommended by the International Union of Pure and Applied Chemistry.

Selected Radioactive Isotopes

Naturally occurring radioactive isotopes are designated by a mass number in blue (although some are also manufactured). Letter m indicates an isomer of another isotope of the same mass number. Half-lives shown in parentheses, where s, min, h, d, and y stand respectively for seconds, minutes, hours, days, and years. The table includes mainly the longer-lived radioactive isotopes; many others have been prepared. Isotopes known to be radioactive but with half-lives exceeding 10¹⁵ y have not been included. Symbols describing the principal mode (or modes) of decay are as follows (these processes are generally accompanied by gamma radiation):

- α: alpha particle emission
- β⁻: beta particle (electron) emission
- β⁺: positron emission
- EC: orbital electron capture
- IT: isomeric transition from upper to lower isomeric state
- SF: spontaneous fission

GROUP 1/IA ... 18/VIII

TABLE OF PERIODIC PROPERTIES OF THE ELEMENTS

Percent Ionic Character of a Single Chemical Bond

Difference in electronegativity	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0	1.1	1.2	1.3	1.4	1.5	1.6	1.7	1.8	1.9	2.0	2.1	2.2	2.3	2.4	2.5	2.6	2.7	2.8	2.9	3.0	3.1	3.2
Percent ionic character %	0.5	1	2	4	6	9	12	15	19	22	26	30	34	39	43	47	51	55	59	63	67	70	74	76	79	82	84	86	88	89	91	92

DATA CONCERNING THE MORE STABLE ELEMENTARY (SUBATOMIC) PARTICLES

Symbol	n	p	e⁻ (e⁺)	Neutrino*	Photon
	Neutron	Proton	Electron*	Neutrino*	Photon
Rest mass (kg)	1.67493×10⁻²⁷	1.67265×10⁻²⁷	9.1095×10⁻³¹	~0	0
Relative atomic mass, (¹²C=12)	1.008665	1.007276	5.48580×10⁻⁴	~0	0
Charge (C)	0	1.60219×10⁻¹⁹	-1.60219×10⁻¹⁹	~0	0
Radius (m)	8×10⁻¹⁶	8×10⁻¹⁶	<1×10⁻¹⁶	~0	0
Spin quantum number	1/2	1/2	1/2	1/2	1
Magnetic Moment†	-1.913 μ_N	2.793 μ_N	1.001 μ_B	0	0

NOTES: (1) For representative oxides (higher valence) of group. Oxide is acidic if color is red, basic if color is blue and amphoteric if both colors are shown. Intensity of color indicates relative strength.

(2) □ Cubic, face centered; ⬡ hexagonal; ◇ rhombohedral; ▱ cubic, body centered; ▭ tetragonal; ◊ orthorhombic; ⬠ monoclinic; □ cubic;

(3) At 300 K (27°C)
(4) At boiling point
(5) At melting point
(6) Generally at 293 K (20°C) for liquid for polycrystalline material
(7) Quantum mechanical value for free atom
(8) From density at 300 K (27°C) for liquid and solid elements; values for gaseous elements refer to liquid state at boiling point

The A & B subgroup designations, are those recommended by the International Union of Pure and Applied Chemistry.

*ESTIMATED VALUES

Catalog Number WLS-18806

Sargent-Welch
VWR INTERNATIONAL
P.O. Box 4130 • Buffalo, NY 14217
1-800-727-4368 • FAX 1-800-676-2540
www.sargentwelch.com

SIDE 2

Positive Ions – Cations

1+		2+		3+		4+	
Ammonium	NH_4^+	Barium	Ba^{2+}	Aluminum	Al^{3+}	Carbon	C^{4+}
Cesium	Cs^+	Beryllium	Be^{2+}	Antimony (III)	Sb^{3+}	Lead (IV)	Pb^{4+}
Copper (I)	Cu^+	Cadmium (II)	Cd^{2+}	Bismuth (III)	Bi^{3+}	Silicon	Si^{4+}
Gold (I)	Au^+	Calcium	Ca^{2+}	Chromium (III)	Cr^{3+}	Tin (IV)	Sn^{4+}
Hydrogen	H^+	Chromium (II)	Cr^{2+}	Cobalt (III)	Co^{3+}		
Lithium	Li^+	Cobalt (II)	Co^{2+}	Gallium	Ga^{3+}	5+	
Potassium	K^+	Copper (II)	Cu^{2+}	Gold (III)	Au^{3+}	Antimony (V)	Sb^{5+}
Rubidium	Rb^+	Iron (II)	Fe^{2+}	Iron (III)	Fe^{3+}	Bismuth (V)	Bi^{5+}
Silver	Ag^+	Lead (II)	Pb^{2+}	Manganese (III)	Mn^{3+}		
Sodium	Na^+	Magnesium	Mg^{2+}	Nickel (III)	Ni^{3+}		
		Manganese (II)	Mn^{2+}				
		Mercury (I)	Hg_2^{2+}				
		Mercury (II)	Hg^{2+}				
		Nickel (II)	Ni^{2+}				
		Strontium	Sr^{2+}				
		Tin (II)	Sn^{2+}				
		Zinc	Zn^{2+}				

Negative Ions – Anions

| -1		-2		-3		-4	
Acetate	CH_3COO^-	Carbonate	CO_3^{2-}	A. Arsenide	As^{3-}	A. Carbide	C^{4-}
Bromide	Br^-	Chromate	CrO_4^{2-}	B. Nitride	N^{3-}		
Chlorate	ClO_3^-	Dichromate	$Cr_2O_7^{2-}$	C. Phosphate	PO_4^{3-}		
Chloride	Cl^-	Monohydrogen Phosphate	HPO_4^{2-}	Phosphide	P^{3-}		
Chlorite	ClO_2^-	Oxalate	$C_2O_4^{2-}$	Phosphite	PO_3^{3-}		
Cyanide	CN^-	Oxide	O^{2-}				
Dihydrogen phosphate	$H_2PO_4^-$	Peroxide	O_2^{2-}				
Fluoride	F^-	Selenide	Se^{2-}				
Hydride	H^-	Silicate	SiO_3^{2-}				
Hydrogen carbonate	HCO_3^-	Sulfate	SO_4^{2-}				
Hydrogen sulfate	HSO_4^-	Sulfide	S^{2-}				
Hydrogen sulfide	HS^-	Sulfite	SO_3^{2-}				
Hydrogen sulfite	HSO_3^-	Telluride	Te^{2-}				
Hydroxide	OH^-	Thiosulfate	$S_2O_3^{2-}$				
Hypochlorite	OCl^-						
Iodate	IO_3^-						
Iodide	I^-						
Nitrate	NO_3^-						
Nitrite	NO_2^-						
Perchlorate	ClO_4^-						
Permanganate	MnO_4^-						
Thiocyanate	SCN^-						

The Solubility Rules

1. The nitrates, nitrites, chlorates, and acetates of all metals are soluble in water.
   a. Silver acetate, silver nitrite, and potassium perchlorate are sparingly soluble.

2. All sodium, potassium, and ammonium salts are soluble in water.

3. The chlorides, bromides, and iodides of all metals EXCEPT lead, silver, and mercury (I) are soluble in water.
   a. $HgBr_2$ is moderately soluble.
   b. $PbCl_2$, $PbBr_2$, and PbI_2 are soluble in hot water.
   c. The water insoluble chlorides, bromides, and iodides are also insoluble in dilute acids.

4. The sulfates of all metals except lead, strontium, mercury (I), and barium are soluble in water.
   a. Silver sulfate and calcium sulfate are slightly soluble.
   b. The water insoluble sulfates are also insoluble in dilute acids.

5. The carbonates, phosphates, borates, sulfites, chromates, and arsenates of all metals EXCEPT sodium, potassium, and ammonium are insoluble in water, but soluble in dilute acids.
   a. $MgCrO_4$ is soluble in water.
   b. $MgSO_3$ is slightly soluble in water.

6. The sulfides of all metals except lithium, barium, calcium, magnesium, potassium, sodium, and ammonium are insoluble in water.
   a. BaS, CaS, and MgS are sparingly soluble.

7. The hydroxides of lithium, potassium, sodium, and ammonium are very soluble in water.
   a. The oxides and hydroxides of calcium, strontium, and barium are moderately soluble.
   b. The oxides and hydroxides of all other metals are insoluble.

Laboratory Safety and Work Instructions

Attention Student! Read the following carefully because your instructor may give you a quiz on this material.

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 (including splash guards) at all times while in the laboratory. (*No one will be admitted without it.*) 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.


Approved eye protection

Typical eyewash

PUSH
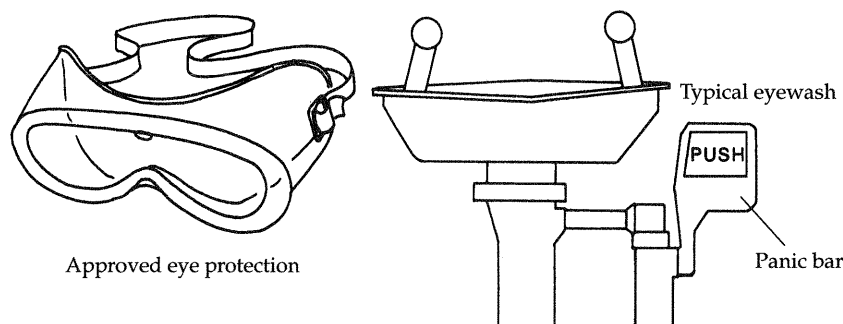
Panic bar

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. Wear shoes at all times. (*No one will be admitted without them.*)

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

4. Know where to find and how to use all safety and first-aid equipment (see the first page of this book).

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

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

7. 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. Chemicals are not to be used to obtain a "high" or clear your sinuses.

Waft toward
your nose
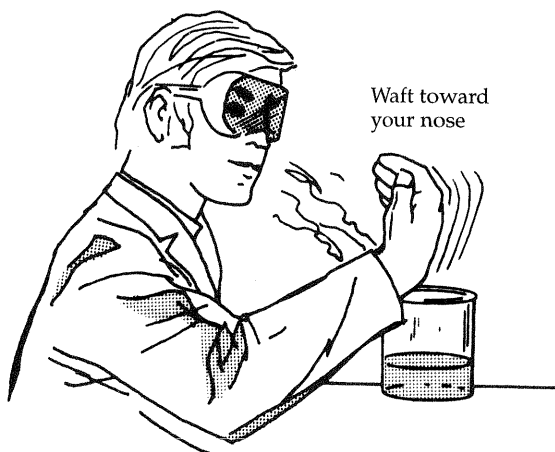
8. Perform in the fume exhaust hood any reactions involving skin-irritating or dangerous chemicals, or unpleasant odors. This is a typical fume exhaust hood. Exhaust hoods have fans to exhaust fumes out of the
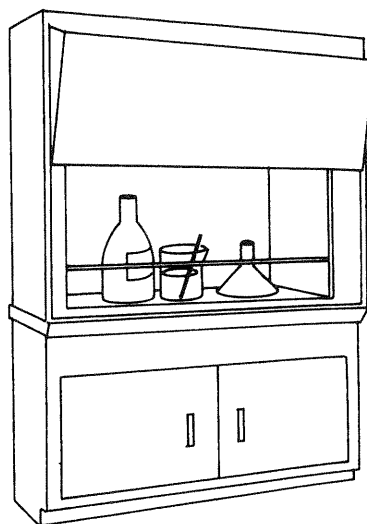
hood and away from the user. The hood should be used when you are studying noxious, hazardous, and flammable materials. It also has a shatterproof glass window, which may be used as a shield to protect you from minor explosions. Reagents that evolve toxic fumes are stored in the hood. Return these reagents to the hood after their use.

9. Never point a test tube that you are heating at yourself or your neighbor—it may erupt like a geyser.

10. Do not perform *any* unauthorized 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. "Do as you oughter, pour acid into water."

13. Avoid rubbing your eyes unless you *know* that your hands are clean.

14. When inserting glass tubing or thermometers into stoppers, *lubricate the tubing and the hole in the stopper with glycerol or water.* Wrap the rod in a towel and grasp it as close to the end being inserted as possible. Slide the glass into the rubber stopper with a twisting motion. Do not push. Finally, remove the excess lubricant by wiping with a towel. Keep your hands as close together as possible in order to reduce leverage.
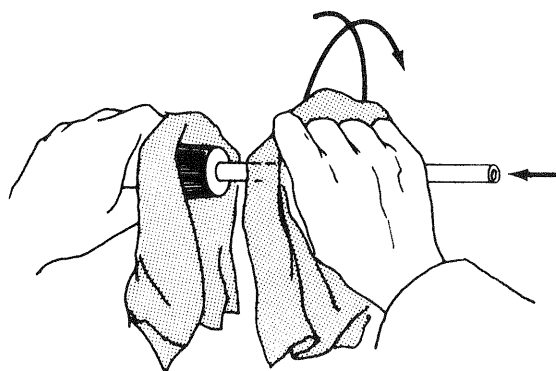
15.   For safety purposes, always place the ring stand as far back on the laboratory bench as comfortable, with the long edges of the base perpendicular to the front of the bench.
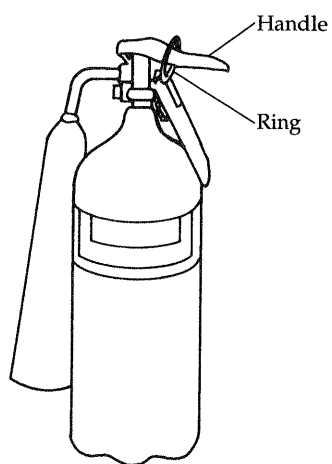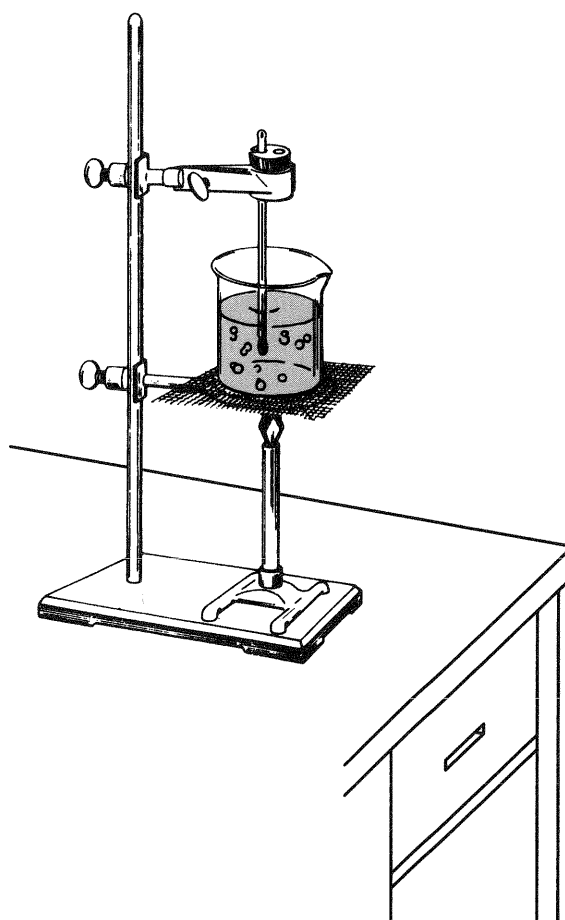
16. NOTIFY THE INSTRUCTOR IMMEDIATELY IN CASE OF AN ACCIDENT.

17. Many common reagents—for example, alcohols, acetone, and especially ether—are highly flammable. *Do not use them anywhere near open flames.*

18. Observe all special precautions mentioned in experiments.

19. Learn the location and operation of fire-protection devices.

   In the unlikely event that a large chemical fire occurs, carbon dioxide fire extinguishers are available in the lab (usually mounted near one of the exits in the room). A typical carbon dioxide fire extinguisher is shown below.

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

One of the most frightening and potentially most serious accidents is the ignition of one's clothing. 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 neighbors 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:
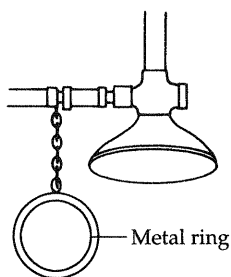
In case someone's clothing or hair is on fire, immediately lead the person to the shower and pull the metal ring. Safety showers generally dump 40 to 50 gallons of water, which should extinguish the flames. These showers generally cannot be shut off once the metal ring has been pulled. Therefore, the shower cannot be demonstrated. (Showers are checked for proper operation on a regular basis, however.)

20. Whenever possible, use hot plates in place of Bunsen burners.

BASIC INSTRUCTIONS FOR LABORATORY WORK

1. Read the assignment *before* coming to the laboratory.
2. Work independently unless instructed to do otherwise.
3. Record your results directly onto your report sheet or notebook. DO NOT RECOPY FROM ANOTHER PIECE OF PAPER.
4. Work conscientiously to avoid accidents.
5. Dispose of excess reagents as instructed by your instructor. NEVER RETURN REAGENTS TO THE REAGENT BOTTLE.
6. Do not place reagent-bottle stoppers on the desk; hold them in your hand. Your laboratory instructor will show you how to do this. Replace the stopper on the same bottle, never on a different one.
7. Leave reagent bottles on the shelf where you found them.
8. Use only the amount of reagent called for; avoid excesses.
9. Whenever instructed to use water in these experiments, use distilled water unless instructed to do otherwise.
10. Keep your area clean.
11. Do not borrow apparatus from other desks. If you need extra equipment, obtain it from the stockroom.
12. When weighing, do not place chemicals directly on the balance.
13. Do not weigh hot or warm objects. Objects should be at room temperature.
14. Do not put hot objects on the desktop. Place them on a wire gauze or heat-resistant pad.

"I have read and understand these instructions as well as the laboratory safety and work instructions"
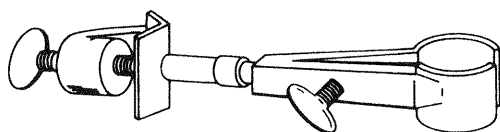
_____   _____
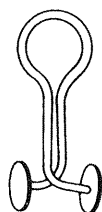Student Signature   Date

COMMON LABORATORY APPARATUS
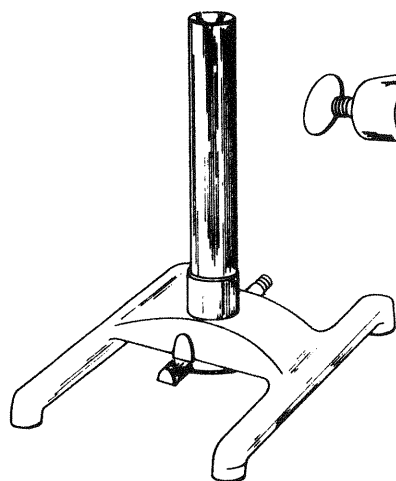
Utility clamp

Test tube

Bunsen burner
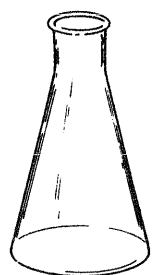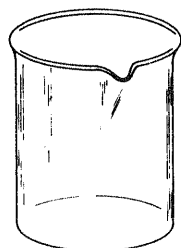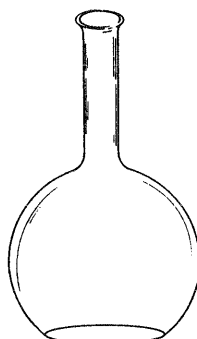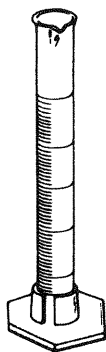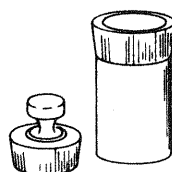
Pinchclamp

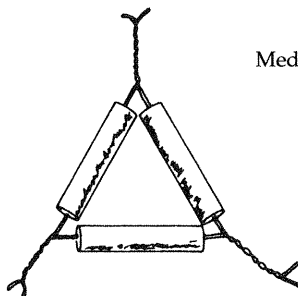Watch glass

Erlenmeyer flask

Beaker

Florence flask

Graduated cylinder
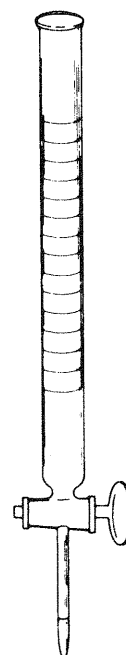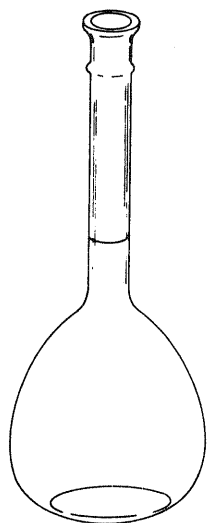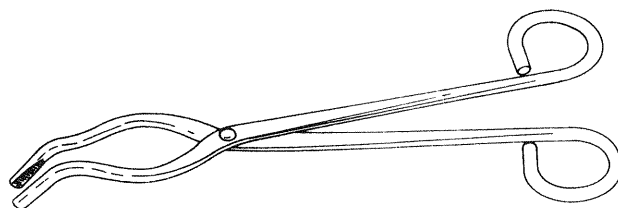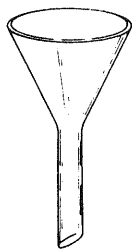
Weighing bottle

Medicine dropper

Buret

Clay triangle

Crucible tongs
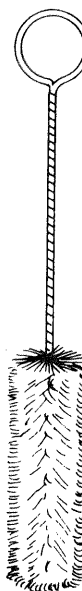
Volumetric flask

Funnel

Test tube brush

Evaporating dish

Test tube holder

Deflagrating spoon

Pipet

Wire gauze

Triangular file

Crucible and cover
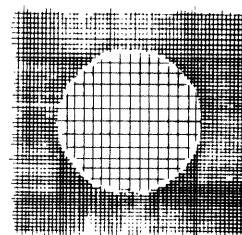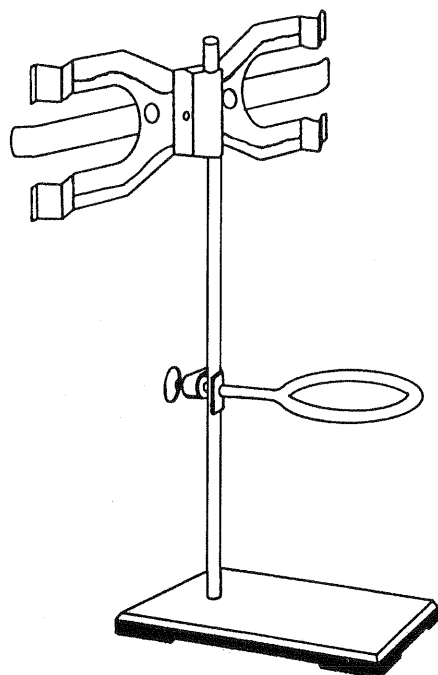
Stirring rod

Ring stand, iron ring, and double buret clamp
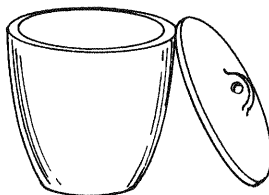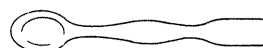
Spatulas

Basic Laboratory Techniques

To learn the use of common, simple laboratory equipment.

Apparatus

balance	Bunsen burner and hose
150- and 250-mL beakers	meter stick, or ruler
50- and 125-mL Erlenmeyer flasks	10-mL pipet
50- or 100-mL graduated cylinder	rubber bulb for pipet
barometer	ring stand and iron ring
clamp	thermometer
large test tube	

Chemicals

ice	antifreeze (ethylene glycol)

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 and precision of the device you use to make the measurement. Appendix A of this manual contains a section on significant figures and measurements that you may find helpful in performing this experiment. 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 prefixes given in Table 1.2 and you use dimensional analysis.

In 1960 international agreement was reached, specifying a particular choice of metric 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.

In Table 1.1, the prefix *means* the power of 10. For example, 5.4 *centi*meters means 5.4×10^{-2} meter; *centi-* has the same meaning as $\times 10^{-2}$.
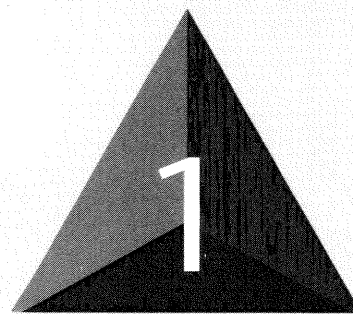
TABLE 1.1 Units of Measurement in the Metric System

Measurement	Unit and definition
Mass or weight	Gram (g) = mass of 1 cubic centimeter (cm^3) of water at 4°C and 760 mm Hg Mass = quantity of material Weight = mass × gravitational force
Length	Meter (m) = 100 cm = 1000 millimeters (mm) = 39.37 in.
Volume	Liter (L) = volume of 1 kilogram (kg) of water at 4°C
Temperature	°C, measures heat intensity: $°C = \frac{5}{9}(°F - 32)$ or $°F = \frac{9}{5}°C + 32$
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, g/L for gases, and g/cm^3 for solids $d = \dfrac{mass}{unit\ volume}$
Specific gravity	sp gr, dimensionless: $sp\ gr = \dfrac{density\ of\ a\ substance}{density\ of\ a\ reference\ substance}$

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 between metric and English units
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 1 L = 10^{-3} m^3 1 L = 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)	0K = −273.15°C $°C = \frac{5}{9}(°F - 32)$ $°F = \frac{9}{5}°C + 32$

*A mL is the same volume as a cubic centimeter: 1 mL = 1 cm^3.
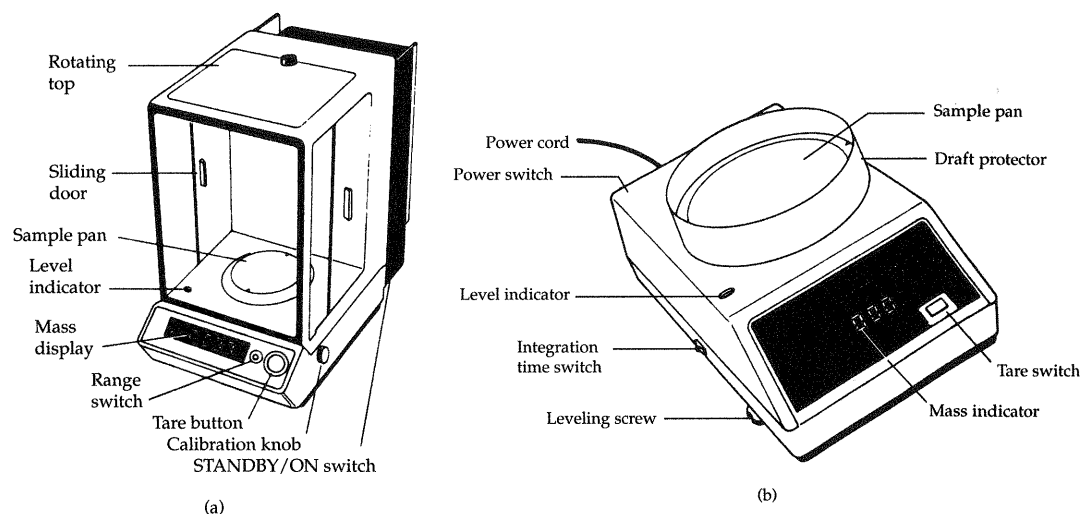
▲ **FIGURE 1.1** Digital electronic balances. The balance gives the mass directly when an object to be weighed is placed on the pan. (a) Analytical balance. (b) Top loader.

EXAMPLE 1.1

Convert 6.7 nanograms to milligrams.

SOLUTION:

$$(6.7 \text{ ng})\left(\frac{10^{-9} \text{ g}}{1 \text{ ng}}\right)\left(\frac{1 \text{ mg}}{10^{-3} \text{ g}}\right) = 6.7 \times 10^{-6} \text{ mg}$$

Notice that the conversion factors have no effect on the magnitude (only the power of 10) of the mass measurement.

The quantities presented in Table 1.1 are measured with the aid of various pieces of apparatus. A brief description of some measuring devices follows.

Laboratory Balance

A laboratory balance is used to obtain the mass of various objects. There are several varieties of balances, with various limits on their accuracy. Two common kinds of balances are depicted in Figure 1.1. These single-pan balances are found in most modern laboratories. Generally they are simple to use, but they are very *delicate* and *expensive*. The amount of material to be weighed and the accuracy required determine which balance you should use.

Meter Rule

The standard unit of length is the meter (m), which is 39.37 in. in length. A metric rule, or meterstick, is divided into centimeters (1 cm = 0.01 m; 1 m = 100 cm) and millimeters (1 mm = 0.001 m; 1 m = 1000 mm). It follows that 1 in. is 2.54 cm. (Convince yourself of this, because it is a good exercise in dimensional analysis.)

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 have 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.

Thermometers

Most thermometers are based upon the principle that liquids expand when heated. Most common thermometers use mercury or colored alcohol as the liquid. These thermometers are constructed so that a uniform-diameter capillary tube surmounts a liquid 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 subdivided 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. 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 (see the section on significant figures in Appendix A). For example, a mass obtained from an analytical balance should be observed and recorded to the nearest 0.0001 g, or 0.1 mg. If the same object were weighed on a top-loading balance, its mass is recorded to the nearest 0.001 g. This is illustrated in Table 1.5.

PROCEDURE | ## A. The Meterstick

Examine the meterstick and observe that one side is ruled in inches, whereas the other is ruled in centimeters. Measure and record the length and width of your lab book in both units. Mathematically convert the two measurements to show that they are equivalent.

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 *meniscus* (curved surface of the water) is concave (Figure 1.3).

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*1 mm Hg is also called 1 torr.
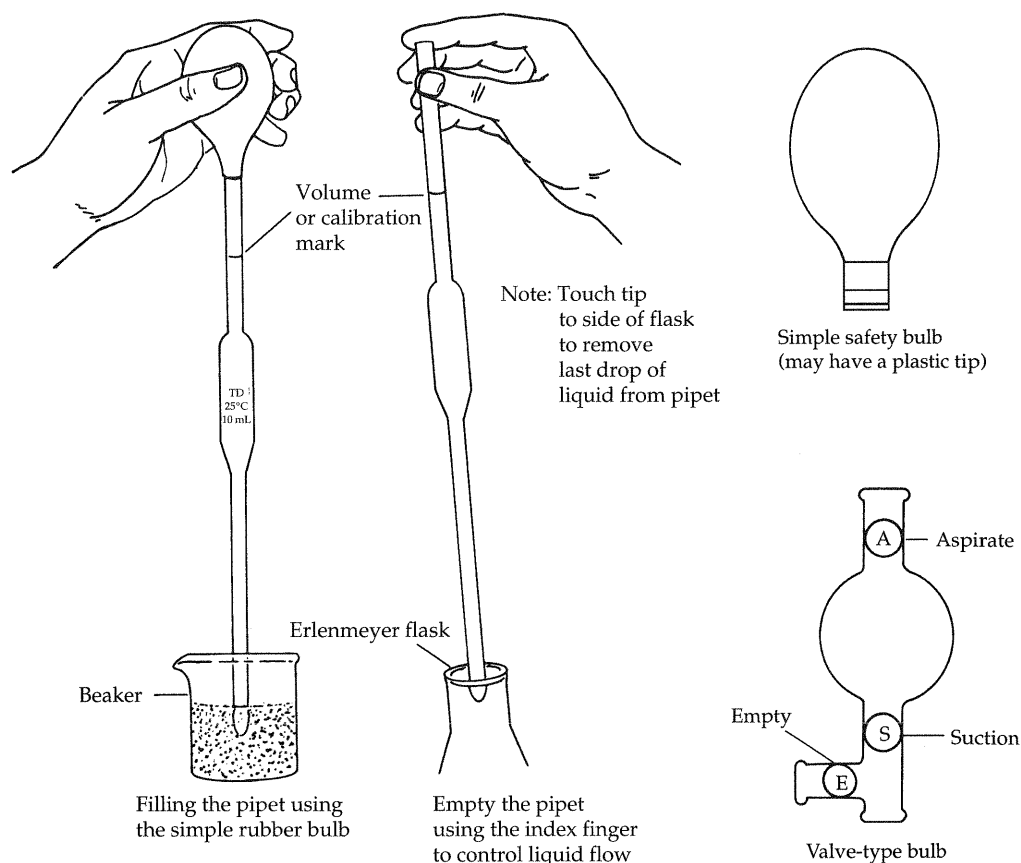
▲ FIGURE 1.2 A typical volumetric pipet, rubber bulbs, and the pipet-filling technique.

TABLE 1.4 Equipment Accuracy

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

TABLE 1.5 Significant Figures Used in Recording Mass

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)	

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 largest test tube will hold. Record this volume.

C. The Thermometer and Its Calibration

This part of the experiment is performed to check the accuracy of your thermometer. These measurements will show how measured temperatures (read from thermometer) compare with true temperatures (the boiling and freezing points of water). The freezing point of water is 0°C; the boiling point
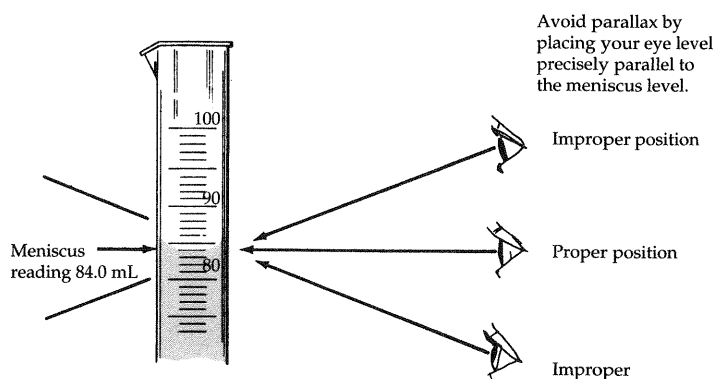
▲ FIGURE 1.3 Proper eye position for taking volume readings.

depends upon atmospheric pressure and is calculated as shown in Example 1.2. Place approximately 50 mL of ice in a 250-mL beaker and cover the ice with distilled water. Allow about 15 min for the mixture to come to equilibrium and then measure and record the temperature of the mixture. *Theoretically, this temperature is 0°C.* Now, set up a 250-mL beaker on a wire gauze and iron ring as shown in Figure 1.4. Fill the beaker about half full with distilled water. Adjust your burner to give maximum heating and begin heating the water. *(Time can be saved if the water is heated while other parts of the experiment are being conducted.)* Periodically determine the temperature of the water with the thermometer, but be careful not to touch the walls of the beaker with the thermometer bulb. Record the boiling point (b.p.) of the water. Using the data given in Example 1.2, determine the *true boiling point at the observed*
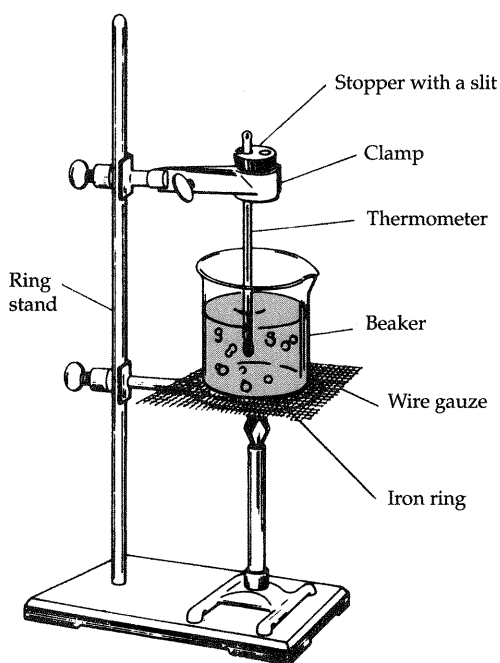
▲ FIGURE 1.4 Apparatus setup for thermometer calibration.

atmospheric pressure. Obtain the atmospheric pressure from your laboratory instructor. Determine the temperature correction to be applied to your thermometer readings.

EXAMPLE 1.2

Determine the boiling point of water at 642 mm Hg.

SOLUTION: Temperature corrections to the boiling point of water are calculated using the following formula:

b.p. correction = (760 mm Hg − atmospheric pressure) × (0.037°C/mm)

The correction at 642 mm Hg is therefore

b.p. correction = (760 mm Hg − 642 mm Hg) × (0.037°C/mm) = 4.4°C

The true boiling point is thus

$$100.0°C - 4.4°C = 95.6°C$$

Using the graph paper provided, construct a thermometer-calibration curve like the one shown in Figure 1.5 by plotting observed temperatures versus true temperatures for the boiling and freezing points of water.

D. 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 balances 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.
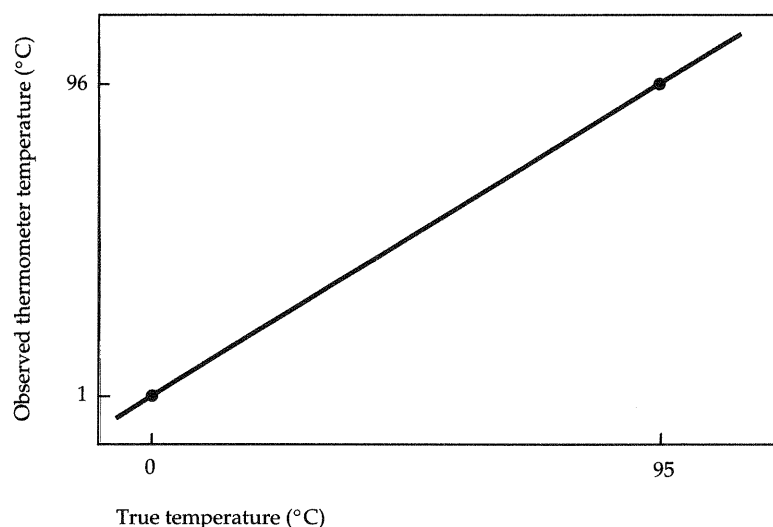
▲ **FIGURE 1.5** Typical thermometer-calibration curve.

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 mass of an empty, dry 50-mL Erlenmeyer flask (tare) to the nearest 0.1 mg. Measure and record the temperature of the water. Using your pipet and rubber bulb, pipet exactly 10 mL of water into this flask and weigh the flask with the water in it (gross) to the nearest 0.1 mg. Obtain the mass 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.

$$\text{Density} = \frac{\text{mass}}{\text{volume}} \qquad d = \frac{m}{V}$$

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 mL of water three separate times.

EXAMPLE 1.3

Using the procedure given above, a mass of 10.0025 g was obtained for the water delivered by one 10-mL pipet at 22°C. What is the volume delivered by the pipet?

SOLUTION: From the density equation given above, we know that

$$V = \frac{m}{d}$$

For mass we substitute our value of 10.0025 g. For the density, consult Table 1.6. At 22°C, the density is 0.997770 g/mL. The calculation is

$$V = \frac{10.0025 \text{ g}}{0.997770 \text{ g/mL}} = 10.0249 \text{ mL}$$

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

TABLE 1.6 **Density of Pure Water at Various Temperatures**

T (°C)	d (g/mL)	T (°C)	d (g/mL)
15	0.999099	22	0.997770
16	0.998943	23	0.997538
17	0.998774	24	0.997296
18	0.998595	25	0.997044
19	0.998405	26	0.996783
20	0.998203	27	0.996512
21	0.997992	28	0.996232

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 1.4 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 1.4

The following volumes were obtained for the calibration of a 10-mL pipet: 10.15, 10.12, and 10.00 mL. Calculate the mean value and the average deviation from the mean.

SOLUTION:

$$\text{Mean} = \frac{10.15 + 10.12 + 10.00}{3} = 10.09$$

Deviations from the mean: $|\text{value} - \text{mean}|$

$$|10.15 - 10.09| = 0.06$$
$$|10.12 - 10.09| = 0.03$$
$$|10.00 - 10.09| = 0.09$$

Average deviation from the mean

$$= \frac{0.06 + 0.03 + 0.09}{3} = 0.06$$

The reported value is therefore 10.09 ± 0.06 mL.

E. Measuring the Density of Antifreeze

Weigh a dry 50-mL flask to the nearest 0.1 mg, and record its mass. Using your pipet, measure a 10-mL sample of antifreeze solution into the 50-mL flask, weigh the flask and its contents, and record this mass. Repeat these measurements two more times to give you an indication of the precision of your measurements. Use the measured mass and volume to calculate the density of the antifreeze for each measurement. Using the three values for the density, calculate the mean density and the average deviation from the mean for your determinations.

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

PRE LAB QUESTIONS

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

2. What decimal power do the following abbreviations represent: (a) M (b) k (c) m (d) n (e) μ?

3. What is the number of significant figures in each of the following measured quantities? (a) 351 g; (b) 0.0100 mL; (c) 1.010 mL; (d) 3.72×10^{-3} cm.

4. What is the length of a crystal of copper sulfate in millimeters that is 0.250 inches long?

5. Perform the following conversions: (a) 72.3 mg to g; (b) 6.0×10^{-10} m to mm; (c) 325 mm to μm.

6. DNA is approximately 2.5 nm in length. If an average man is 6 ft tall, how many DNA molecules could be stacked end to end in an average man?

7. A liquid has a volume of 3.70 liters. What is its volume in mL? In cm^3?

8. Why should you never weigh a hot object?

9. Why is it necessary to calibrate a thermometer and volumetric glassware?

10. What is precision?

11. What is the density of an object with a mass of 1.663 g and a volume of 0.2009 mL?

12. Weighing an object three times gave the following results: 9.2 g, 9.1 g, and 9.3 g. Find the mean mass and the average deviation from the mean.

13. Normal body temperature is 37.0°C. What is the corresponding Fahrenheit temperature?

14. What is the mass in kilograms of 750 mL of a substance that has a density of 0.930 g/mL?

15. An object weighs exactly 5 g on an analytical balance that has an accuracy of 0.1 mg. To how many significant figures should this mass be recorded?

Name _____ Desk _____

Date _____ Laboratory Instructor _____

Basic Laboratory Techniques | 1

A. The Meterstick

Length of this lab book   _____ in. _____ cm _____ mm _____ m

Width of this lab book   _____ in. _____ cm _____ mm _____ m

Using an equation (including units), show that the above measurements are equivalent.

Area of this lab book (show calculations)   _____ cm^2

B. The Graduated Cylinder

Volume of water in graduated cylinder   _____ mL

Volume of water contained in largest test tube   _____ mL (depends upon test tube)

C. The Thermometer and Its Calibration

Observed temperature of water-and-ice mixture   _____ °C

Temperature of boiling water   _____ °C

Observed atmospheric pressure   _____ mm Hg

True (corrected) temperature of boiling water   _____ °C

Thermometer correction   _____ °C

D. Using the Balance to Calibrate Your 10-mL Pipet

Temperature of water used in pipet   _____ °C

Corrected temperature   _____ °C

	Trial 1	Trial 2	Trial 3
Mass of Erlenmeyer plus ~10 mL H$_2$O (gross mass)	_____	_____	_____ g
Mass of Erlenmeyer (tare mass)	_____	_____	_____ g
Mass of ~10 mL of H$_2$O (net mass)	_____	_____	_____ g

Volume delivered by 10-mL   _____   _____   _____ mL
pipet (show calculations)

Mean volume delivered by 10-mL pipet (show calculations)   _____ mL

	Trial 1	Trial 2	Trial 3
Individual deviations from the mean	_____	_____	_____

Average deviation from the mean (show calculations)   _____ mL

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

E. Measuring the Density of Antifreeze

Temperature of antifreeze   _____ °C

	Trial 1	Trial 2	Trial 3
Mass of flask + antifreeze	_____	_____	_____ g
Mass of empty flask	_____	_____	_____ g
Mass of antifreeze	_____	_____	_____ g
Density of antifreeze (show calculation below)	_____	_____	_____ g

Mean (average) density

Average deviation from the mean
(show calculation below)

QUESTIONS

1. Identify each of the following as measurements of length, area, volume, mass, density, time, or temperature: (a) ns; (b) 10.0 kg/m^3; (c) 1.2 pm; (d) 750 km^2; (e) 83K; (f) 4.0 mm^3.

2. Carry out the following operations, and express the answer with the appropriate number of significant figures and units. (a) (5.231 mm)(6.1 mm); (b) 72.3 g/1.5 mL; (c) 12.21 g + 0.0132 g; (d) 31.03 g + 12 mg.

3. Drug medications are often prescribed on the basis of body weight. The adult dosage of Elixophyllin, a drug used to treat asthma, is 6 mg/kg of body mass. Calculate the dose in milligrams for a 150-lb person.

4. A man who is 5 ft 8 in. tall weighs 160 lb. What is his height in centimeters and his mass in kilograms?

5. Determine the boiling point of water at 620 mm Hg.

6. A pipet delivers 9.98 g of water at 20°C. What volume does the pipet deliver?

7. A pipet delivers 10.4, 10.2, 10.8, and 10.6 mL in consecutive trials. Find the mean volume and the average deviation from the mean.

8. A 141-mg sample was placed on a watch glass that weighed 9.203 g. What is the weight of the watch glass and sample in grams?

9. (a) Using the defined freezing and boiling points of water, make a plot of degrees Fahrenheit versus degrees Celsius on the graph paper provided.
   (b) Determine the Celsius equivalent of 40°F using your graph. The relationship between these two temperature scales is linear (i.e., it is of the form $y = mx + b$). Consult Appendix B regarding linear relationships and determine the equation that relates degrees Fahrenheit to degrees Celsius.
   (c) Compute the Celsius equivalent of 40°F using this relationship.

Thermometer Calibration Curve

Observed thermometer temperature, °C

True temperature, °C
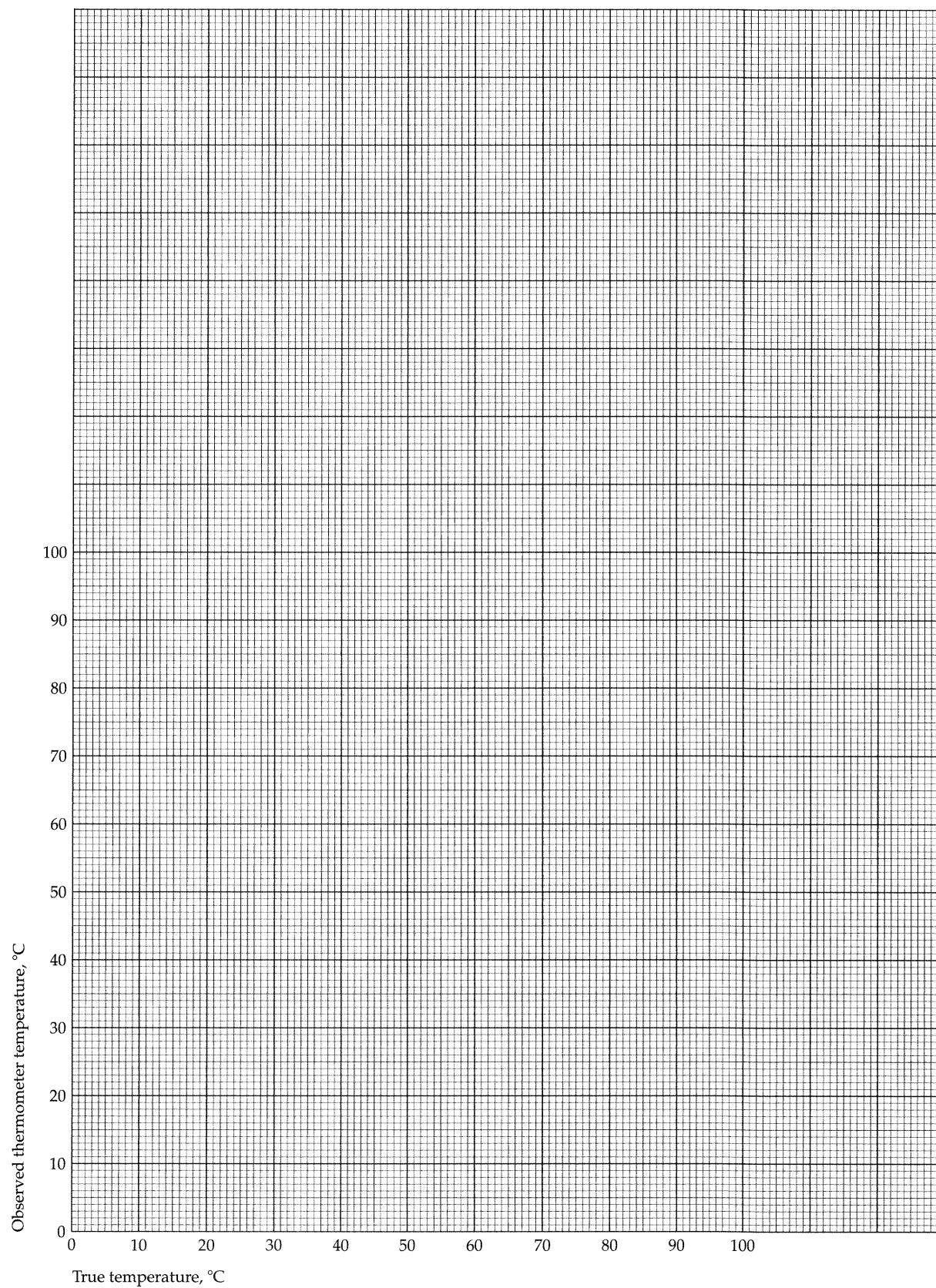
Fahrenheit—Celsius Graph

Temperature, °F

Temperature, °C
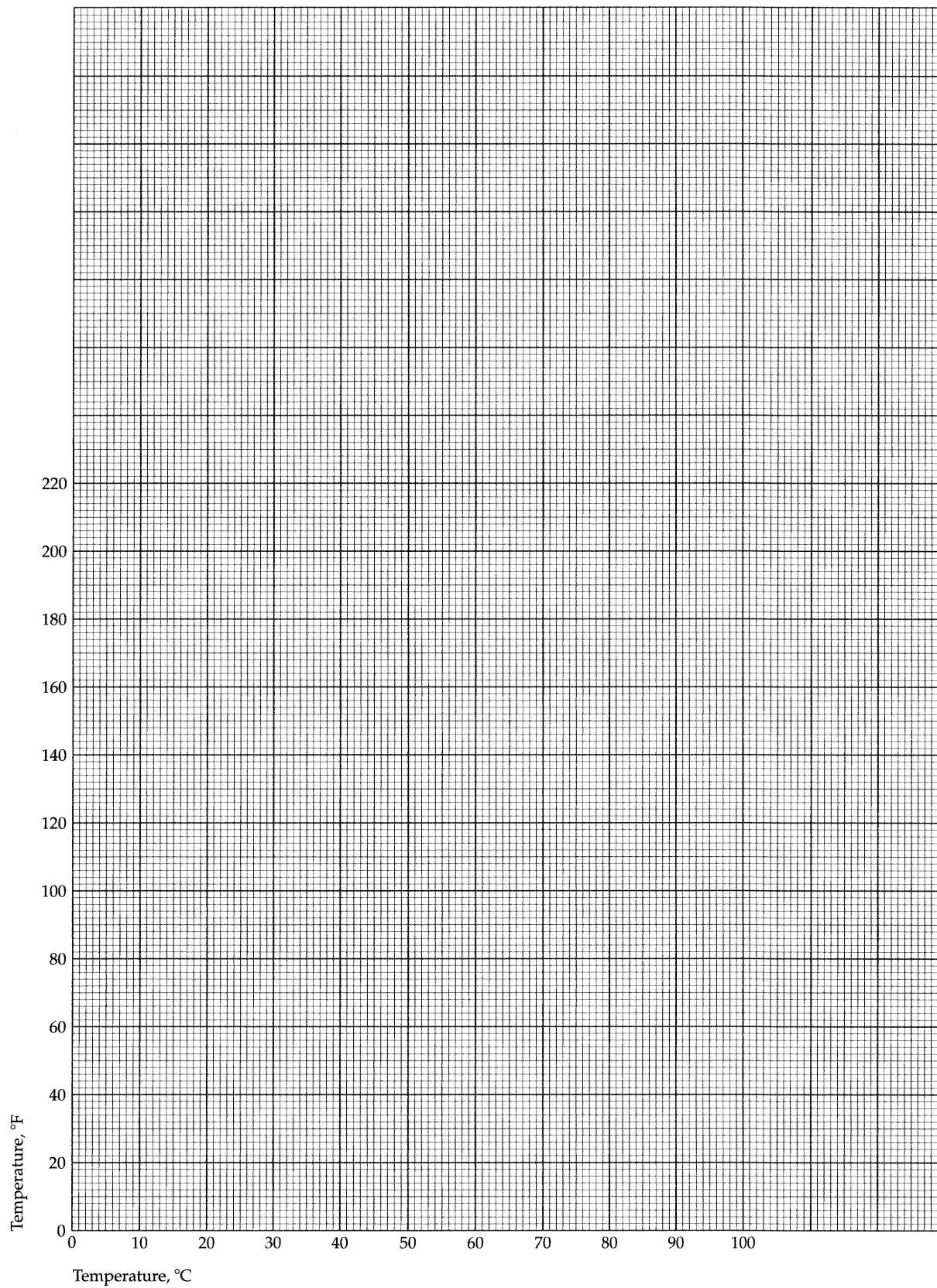
Identification of Substances by Physical Properties

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

OBJECTIVE

Apparatus

**APPARATUS
AND CHEMICALS**

balance	Bunsen burner and hose
50-mL beakers (2)	stirring rod
250-mL beaker	dropper
50-mL Erlenmeyer flask	boiling chips
10-mL graduated cylinder	thermometer
large test tubes (2)	spatula
small test tubes (6)	small watch glass
test-tube rack	capillary tubes (5)
10-mL pipet	tubing with right-angle bend
ring stand and ring	utility clamp
wire gauze	two-hole stopper
no. 3 two-hole stopper	small rubber bands (or small
with one of the holes slit	sections of $1/4$-in. rubber tubing)
to the side or a buret clamp	rubber bulb for pipet

Chemicals

ethyl alcohol	cyclohexane
toluene	naphthalene
soap solution	two unknowns (one liquid;
	one solid)

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.

DISCUSSION

    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. Many
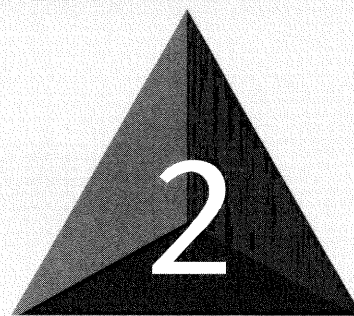
scientists, most notably several German scientists during the latter part of the nineteenth century and earlier part of the twentieth, spent their entire lives gathering data of this sort. Two of the most complete references of this type that are readily available today are The Chemical Rubber Company's *Handbook of Chemistry and Physics* and N. A. 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, melting point, 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 mL or 1000 mL) of a solvent. It is tabulated in handbooks in terms of grams per 100 mL of solvent; the solvent is usually water.

Density is an important physical property and is defined as the mass per unit volume:

$$d = \frac{m}{V}$$

Melting or freezing points correspond to the temperature at which the liquid and solid states of a substance are in equilibrium. These terms refer to the *same* temperature but differ slightly in their meaning. The *freezing point* is the equilibrium temperature when approached from the liquid phase—that is, when solid begins to appear in the liquid. The *melting point* is the equilibrium temperature when approached from the solid phase—that is, when liquid begins to appear in the solid.

A liquid is said to boil when bubbles of vapor form 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 a lower temperature at a lower pressure. The temperature at which a liquid boils at 760 mm Hg 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. A *nomograph* is a set of scales for connected variables (see Figure 2.5 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.

PROCEDURE | A. Solubility

Qualitatively determine the solubility of naphthalene (mothballs) in three solvents: water, cyclohexane, and ethyl alcohol. (**CAUTION:** *Cyclohexane is highly flammable and must be kept away from open flames.*) Determine the solubility by adding two or three small crystals of naphthalene to 2 to 3 mL (it is not necessary to measure either the solute weight or solvent volume) of each of these three solvents in separate, clean, *dry* test tubes. Make an attempt to keep the amount of naphthalene and solvent the same in each case. Place a cork in each test tube and shake briefly. Cloudiness indicates insolubility. Record your conclusions on the report sheet using the abbreviations s (soluble), sp (sparingly soluble), and i (insoluble). Into each of three more

clean, *dry* test tubes place 2 or 3 mL of these same solvents and add 4 or 5 drops of toluene in place of naphthalene. Record your observations. The formation of two layers indicates immiscibility (lack of solubility). Now repeat these experiments using each of the three solvents (water, cyclohexane, and ethyl alcohol) with your solid and liquid unknowns and record your observations.

SAVE your solid and liquid unknowns for Parts B, C, and D, but dispose of the other chemicals in the marked refuse container. Do not dispose of them in the sink.

B. Density

Determine the densities of your two unknowns in the following manner.

The Density of a Solid   Weigh about 1.5 g of your solid unknown to the nearest 0.001 g and record the mass. Using a pipet or a wash bottle, half fill a *clean, dry* 10-mL graduated cylinder with a solvent in which your unknown is *insoluble*. Be *careful* not to get the liquid on the inside walls, because you do not want your solid to adhere to the cylinder walls when you add it in a subsequent step. Read and record this volume to the nearest 0.1 mL. Add the weighed solid to the liquid in the cylinder, being careful not to lose any of the material in the transfer process and ensuring that all of the solid is beneath the surface of the liquid. Carefully tapping the sides of the cylinder with your fingers will help settle the material to the bottom. Do not be concerned about a few crystals that do not settle, but if a large quantity of the solid resists settling, add one or two drops of a soap solution and continue tapping the cylinder with your fingers. Now read the new volume to the nearest 0.1 mL. The difference in these two volumes is the volume of your solid (Figure 2.1). Calculate the density of your solid unknown.

You may recall that by measuring the density of metals in this way, Archimedes proved to the king that the charlatan alchemists had in fact not transmuted lead into gold. He did this after observing that he weighed less in the bathtub than he did normally by an amount equal to the weight of the fluid displaced. According to legend, upon making his discovery, Archimedes emerged from his bath and ran naked through the streets shouting *Eureka!* (I have found it).

The difference between these two readings is the volume of the solid.

Solid material settled on bottom

EUREKA!

THINK TANK
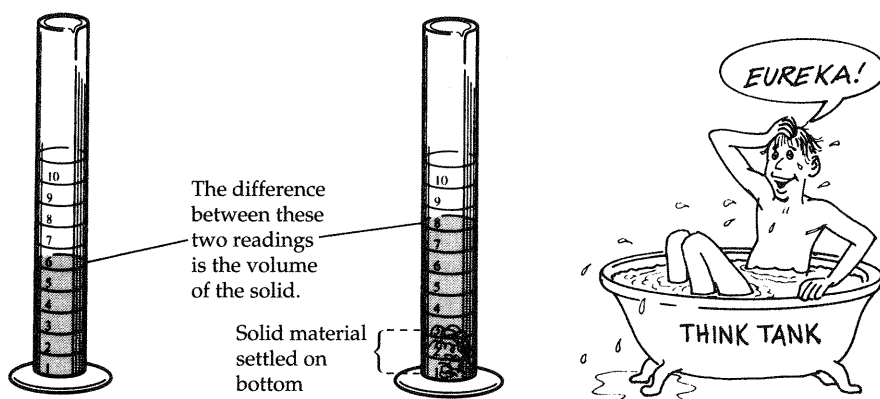
▲ FIGURE 2.1

The Density of a Liquid   Weigh a clean, *dry* 50-mL Erlenmeyer flask to the nearest 0.0001 g. Obtain at least 15 mL of the unknown liquid in a clean, *dry* test tube. Using a 10-mL pipet, pipet exactly 10 mL of the unknown liquid into the 50-mL Erlenmeyer flask and quickly weigh the flask containing the 10 mL of unknown to the nearest 0.0001 g. Using the calibration value for your pipet, if you calibrated it, and 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. Melting Point of Solid Unknown

Obtain a capillary tube and a small rubber band. Seal one end of the capillary tube by carefully heating the end in the edge of the flame of a Bunsen burner until the end *completely* closes. Rotating the tube during heating will help you to avoid burning yourself (Figure 2.2).

Pulverize a small portion of your solid-unknown sample with the end of a test tube on a clean watch glass. Partially fill the capillary with your unknown by gently tapping the pulverized sample with the open end of the capillary to force some of the sample inside. Drop the capillary into a glass tube about 38 to 50 cm in length, with the sealed end down to pack the sample into the bottom of the capillary tube. Repeat this procedure until the sample column is roughly 5 mm in height. Now set up a melting-point apparatus as illustrated in Figure 2.3.

Place the rubber band about 5 cm above the bulb on the thermometer and out of the liquid. Carefully insert the capillary tube under the rubber band with the closed end at the bottom. Place the thermometer with attached capillary into the beaker of water so that the sample is covered by water, the thermometer does not touch the bottom of the beaker, and the open end of the capillary tube is above the surface of the water. Heat the water slowly while gently agitating the water with a stirring rod. Observe the sample in the capillary tube while you are doing this. At the moment that the solid
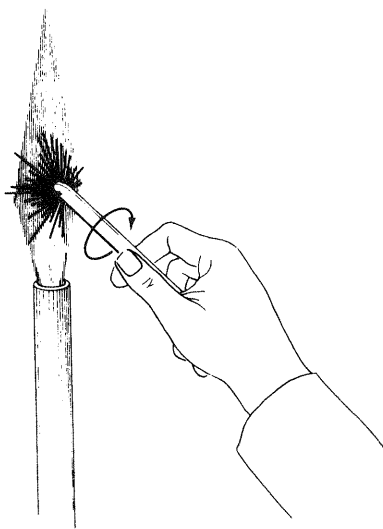
▲ FIGURE 2.2 Sealing one end of a capillary tube.

No. 3 two-hole
rubber stopper
with slit (stopper
and clamp may be
replaced by a
buret clamp)

Thermometer

250-mL beaker
with water

Capillary melting-point tube

Rubber band

Wire gauze

Place the capillary tube and
thermometer bulb at the same
elevation.
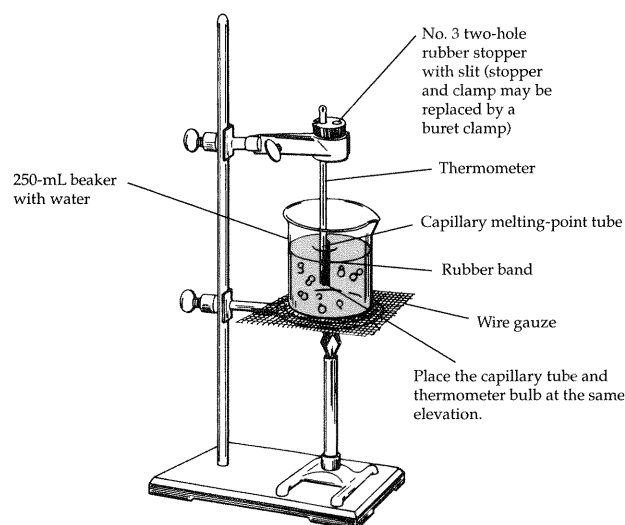
▲ **FIGURE 2.3** Apparatus for melting-point determination.

melts, record the temperature. Also record the melting-point range, which is
the temperature range between the temperature at which the sample begins
to melt and the temperature at which all of the sample has melted. Using
your thermometer-calibration curve (from Experiment 1), correct these tem-
peratures to the true temperatures and record the melting point and melting-
point range. These temperatures may differ by only 1°C or less.

D. Boiling Point of Liquid Unknown

To determine the boiling point of your liquid unknown, put about 3 mL of
the material you used to determine the density into a clean, dry test tube.
Fit the test tube with a two-hole rubber stopper that has one slit; insert your
thermometer into the hole with the slit and one of your right-angle-bend
glass tubes into the other hole, as shown in Figure 2.4. Add one or two small

Thermometer

Plug slit with paper

Rubber stopper with a slit for
the thermometer

Clamp

Rubber tubing
to drain (avoid
constrictions)

250-mL beaker with water

Wire gauze

Ring

Ring stand

Bunsen burner
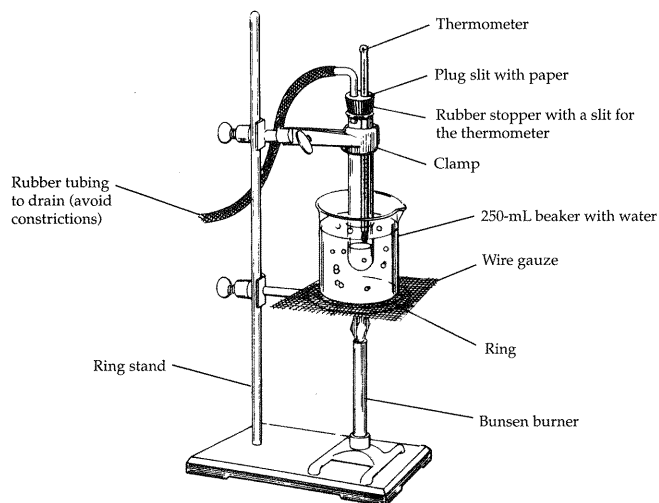
▲ **FIGURE 2.4** Apparatus for boiling-point determination.
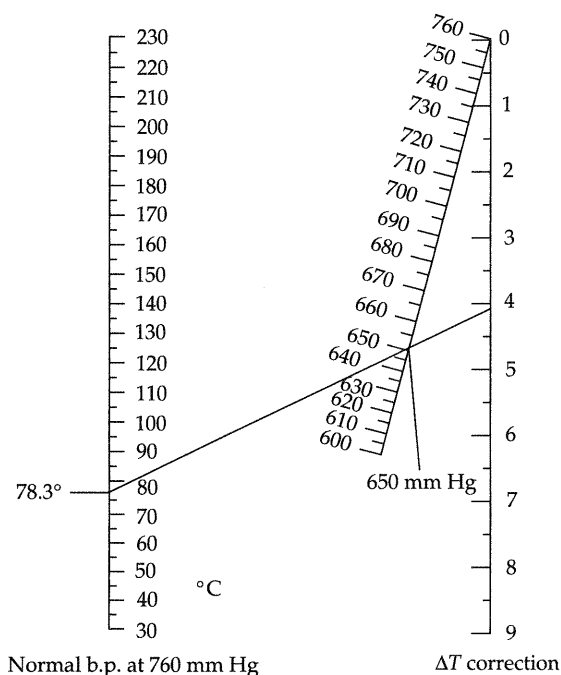
▲ **FIGURE 2.5** Use of nomograph for boiling-point correction for ethyl alcohol to 650 mm Hg.

boiling chips to the test tube to ensure even boiling of your sample. Position the thermometer so that it is about 1 cm above the surface of the unknown liquid. Clamp the test tube in the ring stand and connect to the right-angle-bend tubing a length of rubber tubing that reaches to the sink. Assemble your apparatus as shown in Figure 2.4. (**CAUTION:** *Be certain that there are no constrictions in the rubber tubing. Your sample is flammable. Keep it away from open flames.*)

Heat the water gradually and watch for changes in temperature. The temperature will become constant at the boiling point of the liquid. Record the observed boiling point. Correct the observed boiling point to the true boiling point at room atmospheric pressure using your thermometer-calibration curve. The normal boiling point (b.p. at 1 atm = 760 mm Hg) can now be calculated (see Example 2.1, below) using the nomograph provided in Figure 2.5. Your boiling-point correction should not be more than +5°C.

Example 2.1

What will be the boiling point of ethyl alcohol at 650 mm Hg when its normal boiling point at 760 mm Hg is known to be 78.3°C?

SOLUTION: The answer is easily found by consulting the nomograph in Figure 2.5. A straight line drawn from 78.3°C on the left scale of normal boiling points through 650 mm Hg on the pressure scale intersects the temperature correction scale at 4°C. Therefore,

$$\text{normal b.p.} - \text{correction} = \text{observed b.p.}$$
$$78.3°C - 4.0°C = 74.3°C$$

TABLE 2.1 Physical Properties of Pure Substances

Substance	Density (g/mL)	Melting point (°C)	Boiling point (°C)	Water	Cyclohexane	Ethyl alcohol
				Solubility[a] in		
Acetanilide	1.22	114	304	sp	sp	s
Acetone	0.79	−95	56	s	s	s
Benzophenone	1.15	48	306	i	s	s
Bromoform	2.89	8	150	i	s	s
2,3-Butanedione	0.98	−2.4	88	s	s	s
t-Butyl alcohol	0.79	25	83	s	s	s
Cadmium nitrate · 4H$_2$O	2.46	59	132	s	i	s
Chloroform [b]	1.49	−63.5	61	i	s	s
Cyclohexane	0.78	6.5	81.4	i	s	s
p-Dibromobenzene	1.83	86.9	219	i	s	s
p-Dichlorobenzene	1.46	53	174	i	s	s
m-Dinitrobenzene	1.58	90	291	i	s	s
Diphenyl	0.99	70	255	i	s	s
Diphenylamine	1.16	53	302	i	s	s
Diphenylmethane	1.00	27	265	i	s	s
Ether, ethyl propyl	1.37	−79	64	s	s	s
Hexane	0.66	−94	69	i	s	s
Isopropyl alcohol	0.79	−98	83	s	s	s
Lauric acid	0.88	43	225	i	s	s
Magnesium nitrate · 6H$_2$O	1.63	89	330[c]	s	i	s
Methyl alcohol	0.79	−98	65	s	i	s
Methylene chloride[b]	1.34	−97	40.1	i	s	s
Naphthalene	1.15	80	218	i	s	sp
α-Naphthol	1.10	94	288	i	i	s
Phenyl benzoate	1.23	71	314	i	s	s
Propionaldehyde	0.81	−81	48.8	s	i	s
Sodium acetate · 3H$_2$O	1.45	58	123	s	i	sp
Stearic acid	0.85	70	291	i	s	sp
Thymol	0.97	52	232	sp	s	s
Toluene	0.87	−95	111	i	s	s
p-Toluidine	0.97	45	200	sp	s	s
Zinc chloride	2.91	283	732	s	i	s

[a] s = soluble; sp = sparingly soluble; i = insoluble.
[b] Toxic. Most organic compounds used in the lab are toxic.
[c] Boils with decomposition.

Similar calculations could be done for the compounds in Table 2.1 at any pressure listed on the nomograph in Figure 2.5. In this experiment you will observe a boiling point at a pressure other than at 760 mm Hg, and you wish to know its normal boiling point. In order to estimate its normal boiling point, assume that, for example, your observed boiling point is 57.0°C and the observed pressure is 650 mm Hg. Use your observed boiling point of 57.0°C as if it were the normal boiling point and find the correction for a pressure of 650 mm Hg. Using the nomograph, you can see that the correction is 3.8°C. You would then add this correction to your observed boiling point to obtain an approximate normal boiling point:

$$57°C + 3.8°C = 60.8°C, \text{ or } 61°C$$

By consulting Table 2.1, you can find the compound that best fits your data; in this example, the data are for chloroform.

E. Unknown Identification

Your unknowns are substances contained in Table 2.1. Compare the properties that you have determined for your unknowns with those in the table. Identify your unknowns and record your results.

Dispose of your unknowns in the appropriate marked refuse containers.

PRE LAB QUESTIONS

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

1. List five physical properties.

2. A 1.22 g sample of an unknown has a volume of 1.73 cm^3. What is the density of the unknown?

3. Are the substances lauric acid and stearic acid solids or liquids at room temperature? (See Table 2.1.)

4. Could you determine the density of zinc chloride using water? Why or why not? (See Table 2.1.)

5. What would be the boiling point of hexane at 670 mm Hg?

6. Why do we calibrate thermometers and pipets?

7. Is toluene miscible with water? With ethyl alcohol? (See Table 2.1.)

8. When water and bromoform are mixed, two layers form. Is the bottom layer water or bromoform? (See Table 2.1.)

9. What solvent would you use to determine the density of zinc chloride?

10. The density of a solid with a melting point of 42° to 44°C was determined to be 0.87 ± 0.02 g/mL. What is the solid?

11. The density of a liquid whose boiling point is 55° to 57°C was determined to be 0.77 ± 0.05 g/mL. What is the liquid?

12. Which has the greater volume, 10 g of hexane or 10 g of toluene? What is the volume of each?

Name _____ Desk _____

Date _____ Laboratory Instructor _____

Liquid unknown no. _____

Solid unknown no. _____

REPORT SHEET | EXPERIMENT

Identification of Substances by Physical Properties | 2

A. Solubility

	Water	Cyclohexane	Ethyl alcohol
Naphthalene	_____	_____	_____
Toluene	_____	_____	_____
Liquid unknown	_____	_____	_____
Solid unknown	_____	_____	_____

B. Density

Solid

Final volume of liquid in cylinder	_____ mL	
Initial volume of liquid in cylinder	_____ mL	Diphenylamine data
Volume of solid	_____ mL	
Mass of solid	_____ g	Density of solid _____ g/mL
		(show calculations)

Liquid

Volume of liquid	_____ mL	
Volume of liquid corrected for the pipet correction	_____ mL	
Mass of 50-mL Erlenmeyer plus 10 mL of unknown	_____ g	Isopropyl alcohol data
Mass of 50-mL Erlenmeyer	_____ g	
Mass of liquid	_____ g	Density _____ g/mL
		(show calculations)

C. Melting Point of Solid Unknown

Observed melting point   _____ °C

Corrected (apply thermometer
correction to obtain)   _____ °C

Observed melting-point range   _____ °C

Corrected (apply thermometer
correction to obtain)   _____ °C

D. Boiling Point of Liquid Unknown

Barometric pressure   _____ mm Hg

Observed   _____ °C

Corrected (apply thermometer
correction to obtain)   _____ °C

Estimated true (normal) b.p. (apply
pressure correction to obtain)   _____ °C

E. Unknown Identification

Solid unknown   _____
Liquid unknown   _____

QUESTIONS

1. Is p-dibromobenzene a solid or a liquid at room temperature?

2. What solvent would you use to measure the density of napthalene acid?

3. Convert your densities to kg/L and compare these values with those in g/mL.

4. If air bubbles were trapped in your solid beneath the liquid level in your density determination, what error would result in the volume measurement, and what would be the effect of this error on the calculated density?

5. A liquid unknown was found to be insoluble in water and soluble in cyclohexane and alcohol; the unknown was found to have a boiling point of 58°C at 670 mm Hg. What is the substance? What could you do to confirm your answer?

6.   A liquid that has a density of 0.80 ± 0.01 g/mL is soluble in cyclohexane. What liquids may this be?

7.   What is the boiling point of cyclohexane at 620 mm Hg?

Consult a handbook or the internet for the following questions and specify the source used.

_____

8.   Osmium is the densest element known. What are its density and melting point?

9.   What are the colors of $CoCl_2$ and $CoCl_2 \cdot 6H_2O$?

10.   What are the formula, molar mass, and color of potassium permanganate?

---

*Until recently, much confusion existed in the literature as to which is the densest metal, osmium or iridium. The currently accepted densities are 22.588 ± 0.015 g/mL and 22.562 g/mL respectively. See: *Platinum Metal Rev.*, 1995, *39*, 164.

NOTES AND CALCULATIONS

The experiment is designed to familiarize you with some standard chemical techniques and to encourage careful work in separating and weighing chemicals.

In this experiment you will separate a mixture of three substances, sodium chloride (NaCl), benzoic acid (C_6H_5COOH), and silicon dioxide (SiO_2) , into pure substances based on their solubility in water. The amount of a substance that will dissolve in water depends on the nature of the substance and on the temperature. Water is a polar substance so other polar substances will dissolve in water - ("Likes dissolve Likes"). Sodium chloride is an ionic compound that is quite soluble in water. Silicon dioxide (sand) is a large macromolecule that is essentially insoluble in water. Benzoic acid is polar, but is much less polar than water so it dissolves in water only to a limited extent. The solubility of these three substances in water is given below. The solubility of each is expressed in terms of grams of the solid that dissolves in 100 g of water. It is evident that each of these has a different solubility/temperature relationship.

Solubility (g/100g H₂O) as a Function of Temperature

				Temperature		
Substance	**0**	**20**	**40**	**60**	**80**	**100**
NaCl	36.00	36.00	37.00	37.00	38.00	40.00
C_6H_5COOH	0.17	0.29	0.42	1.20	2.80	7.00
SiO_2	0	0	0	0	0	0

In this experiment you will use decantation and two types of filtration. Decantation is a process of separating a liquid (called supernatant) from a solid residue by gently pouring off the liquid from the solid. It is easier to decant when you pour the liquid down a stirring rod as shown in the procedure section. Filtration separates a solid and a liquid using a porous material such as filter paper which allows the liquid to pass but retains the solid. Gravity filtration uses the force of gravity to achieve separation. Vacuum filtration uses the suction created by an aspirator to speed up the separation.

Materials List: 250 mL beakers   mixture   wire screen   ring stand
Burner   stirring rod   ice bath   drying oven
Buchner funnel   boiling chips spatula   watch glass

Procedure:

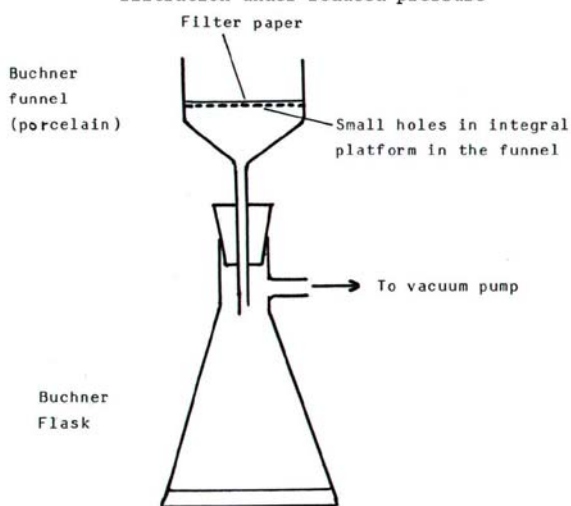1. Carefully weigh a clean dry 250-ml beaker (beaker 1) to the nearest .001 gram. Using a spatula, transfer about 5 to 6 grams of the mixture, which contains sodium chloride, benzoic acid, and silicon dioxide into beaker 1. Be sure to record which unknown you have. Weigh beaker 1 containing the mixture to the nearest 0.001 g. From the two weights, obtain the weight of the mixture. Record your results on the data

sheet.

2. Add about 50 ml of distilled water to beaker. Place beaker 1 with its contents on a wire screen, which is resting on an iron ring connected to a ring stand. See the diagram below:

3. Heat the mixture to the boiling point (stop heating when boiling is observed), and stir the mixture with a stirring rod to make sure that all soluble material is dissolved. At the boiling point temperature, all benzoic acid and sodium chloride should be in solution. Thus, they have been separated from sand. (This is an extraction process.) Take about 15 ml of distilled water in a beaker and bring it to a boil. This boiling water will be used in step 5.

4. Decant the liquid while it is hot into another 250 ml beaker (beaker 2). Use Hot Hands to hold the hot beaker. **Do not** let the sand get into beaker 2 or wait too long before decanting. Use a glass rod to aid the decantation process. See figure below:

5. To dissolve as much benzoic acid and sodium chloride as possible, wash the sand in beaker 1 with about 10 ml or less of boiling water and decant the washing into beaker 2.

6. Place beaker 2 in an ice bath to let it cool. Observe carefully how the benzoic acid crystallizes out of the solution. Set beaker 2 aside to ensure maximum crystallization.
7. Place your beaker of sand in the drying oven so that it can dry completely before weighing. Make sure you mark the beaker with a grease pencil so you can identify it with your initials or some other mark. Leave the beaker in the oven until you have finished the rest of the lab to insure it is fully dried. When you take it out of the oven let it cool, and weigh. You should move on to the next step and come back to weigh the beaker at the end of the experiment.
8. Assemble the Buchner funnel with a filter flask. See the figure below.

FIG 23.4. Buchner funnel and flask for
         filtration under reduced pressure

Filter paper

Buchner
funnel
(porcelain)   Small holes in integral
                               platform in the funnel

                    To vacuum pump

Buchner
Flask

9. Connect a piece of vacuum rubber tubing from the water aspirator to the filter flask. Vacuum rubber tubing is used so that it will not collapse when the pressure is reduced.

10. Place the proper size of filter paper in the Buchner funnel and wet it with distilled water using a wash bottle. This allows the filter paper to sit down on the grating. **Make sure to weigh your filter paper before doing so.**

11. Turn on the water aspirator. Make sure to turn it on just enough to have proper suction occurring.

12. Pour the contents of beaker 2 (benzoic acid crystals) into the Buchner funnel.
13. Wash the solid (benzoic acid) with about 5ml or less of distilled ice water to ensure that the benzoic acid is free of sodium chloride.

14. Continue to suction until the liquid no longer drips from the funnel.

15. Disconnect the rubber tubing from the filter flask before turning off the water aspirator. This prevents the water from backing up into the filter flask. The filtrate (the liquid collected) is often the desired material.

16. Using a spatula transfer the suction dried benzoic acid onto a pre-weighed marked watch glass. Place the filter paper and watch glass into the drying oven for a few minutes to dry. When dry, take it out and let it cool before weighing.

17. To a large beaker add three or four pieces of black boiling chips. Then weigh the beaker to the nearest 0.001g.

18. Transfer the filtrate (liquid) from the filter flask into the large beaker you just weighed in #17. Using a wash bottle rinse the filter flask with as little water as possible. Add the washings to the beaker. Place the beaker (which now contains sodium chloride and water) on a wire screen, which is resting on a iron ring. Start to heat the solution gently with a burner so that the liquid does not boil over. Later, reduce the flame to avoid overheating, which may cause splattering of solid sodium chloride or shatter your beaker. When the water is evaporated entirely, let the sodium chloride dry, and the beaker cool before weighing. Weigh the beaker and its contents to the nearest .001 g and calculate the weight of sodium chloride.

19. Calculate the percentage of each substance in the mixture by using the following formula:

%component = <u>grams of component (salt or benzoic acid or sand)</u> x 100
Total grams of mixture

Name: _____

Lab Partner's Name _____

Date: _____

**REPORT SHEET: SEPARATION OF A MIXTURE**

A. Record Identity of unknown _____

1. Weight of beaker 1 _____

2. Weight of beaker 1 and mixture _____

3. Weight of mixture _____

4. Weight of beaker 1 and dry sand _____

5. Weight of dry sand _____

6. Weight of filter paper _____

7. Weight of watch glass _____

8. Weight of watch glass, filter paper, and benzoic acid. _____

9. Weight of benzoic acid _____

10. Weight of large beaker and boiling chips _____

11. Weight of beaker, boiling chips and sodium chloride _____

12. Weight of sodium chloride _____

13. Percentage of sand in mixture _____

14. Percentage of benzoic acid in mixture _____

15. Percentage of sodium chloride _____

16. Total percentage of sample recovered _____

QUESTIONS:

1. Based on the data provided in the introduction, graph the solubility of benzoic acid as it changes with temperature.

2. From your graph, determine the number of grams of benzoic acid that will dissolve in 100 grams of water at 25 degrees C.

3. If a solution is made up by mixing 6.0 g of benzoic acid in 1.00 L of water at 42 degrees C, would the solution be saturated? Would you expect to see solid on the bottom of the container?

4. From an 11.65 g sample containing sodium chloride, benzoic acid, and silicon dioxide, 3.64 g of NaCl, 1.56 g of C_6H_5COOH and 5.92 g of SiO_2, were recovered. Calculate the percentage of sodium chloride in the sample.

Paper Chromatography: Separation of Cations and Dyes

OBJECTIVE
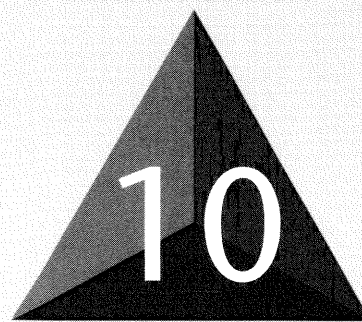
To become acquainted with chromatographic techniques as a method of separation (purification) and identification of substances.

APPARATUS AND CHEMICALS

Apparatus

petri or evaporating dishes (2)	4-in. watch glasses (2)
paper chromatography strips	12.5-cm Whatman #1 filter paper scissors
600-mL beaker	50- or 100-mL graduated cylinder
capillary pipets (6)	sample vials (3)
6-in. glass stirring rod	paper clips (3)
metric ruler	

Chemicals

0.5 M Cu(NO$_3$)$_2$	0.5 M Fe(NO$_3$)$_3$
0.5 M Ni(NO$_3$)$_2$	solvent (90% acetone, 10%
15 M NH$_3$	6 M HCl, freshly prepared)
1% dimethylglyoxime in ethanol	isopropyl alcohol
black felt tip pen	unknown solutions

DISCUSSION

Chromatography (from the Greek *chrōma*, for color, and *graphein*, to write) is a technique often used by chemists to separate components of a mixture. In 1906, the Russian botanist Mikhail Tsvett separated color pigments present in leaves by allowing a solution of these pigments to flow down a column packed with an insoluble material such as starch, alumina (Al$_2$O$_3$), or silica (SiO$_2$). Because different color bands appeared along the column, he called the procedure chromatography. Color is not a requisite property to achieve separation of compounds by this procedure. Colorless compounds can be made visible by being allowed to react with other reagents, or they can be detected by physical means. Consequently, because of its simplicity and efficiency, this technique has wide applicability for separating and identifying compounds such as drugs and natural products. Chromatography is also widely used in analyses such as detecting the use of steroids by competitive athletes or by forensic scientists involved in criminal investigations.

The basis of chromatography is the *partitioning* (that is, separation arising from differences in solubility) of compounds between a stationary phase and a moving phase. Stationary phases such as alumina (Al$_2$O$_3$), silica (SiO$_2$), and paper (cellulose) have enormous surface areas. The molecules or ions of the substances to be separated are continuously being adsorbed and then released (desorbed) into the solvent flowing over the surface of the stationary phase. This brings about a separation of the components—that is, they will travel with different speeds in the moving solvent because there are generally different attractions between these components and the stationary phase.
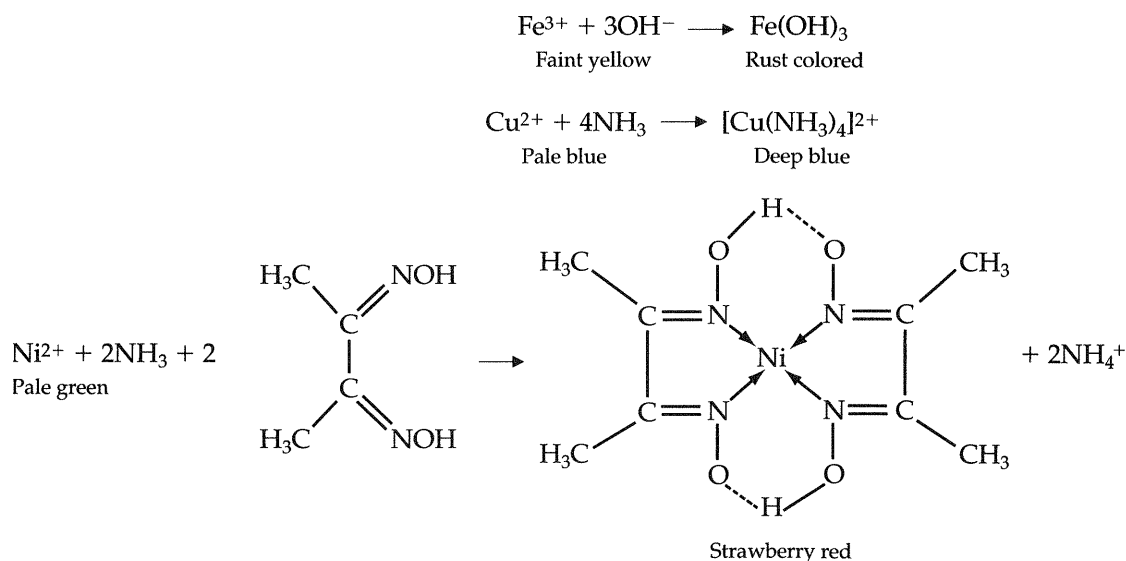
97

In paper chromatography, a small spot of the mixture to be separated is placed at one end of a strip of paper, and solvent is allowed to move up the paper, through the spot, by capillary action. The solvent and various components of the mixture each travel at different speeds along the paper. In this experiment, Fe^{3+}, Cu^{2+}, and Ni^{2+} will be separated with a solvent that consists of a mixture of acetone, water, and hydrochloric acid. A diagram of a portion of a typical chromatogram strip is shown in Figure 10.1.
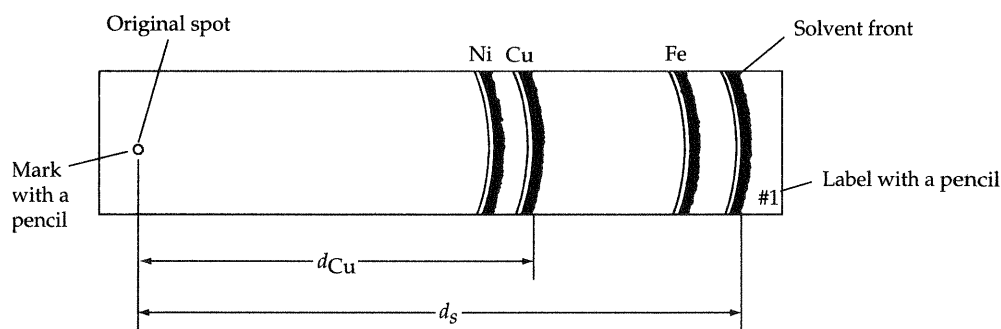
The identity of components in a mixture can be deduced by comparing a chromatogram of the unknown with chromatograms of mixtures of components suspected to be present in the unknown. An additional aid in identification of a compound is its R_f value, which is defined as the ratio of the distance traveled by a compound to the distance traveled by the solvent. For example, for Cu^{2+} (see Figure 10.1),

$$R_f(\text{Cu}) = \frac{d_{\text{Cu}}}{d_s} \qquad [1]$$

The R_f value of a compound is a characteristic of the compound, the support, and the solvent used, and it serves to identify the constituents of a mixture. An estimate of the relative amount of each of the constituents in a mixture can be made from the relative intensities and sizes of the various bands in a chromatogram.

The three ions studied in this experiment will be discerned in the following manner: Fe^{3+} in water imparts a red-brown or rust color and thus will produce a rust-colored band on the paper. Although Cu^{2+} is blue, the color is faint and not easily detected, especially if copper is present in small amounts. In aqueous solution, however, Cu^{2+} reacts with NH_3 (from ammonium hydroxide) to form a complex ion, $[Cu(NH_3)_4]^{2+}$, which is deep blue and therefore readily observed. Finally, Ni^{2+} reacts with an organic reagent, dimethylglyoxime, to produce a strawberry-red color. The following reactions will occur during the development process on the paper:

$$Fe^{3+} + 3OH^- \longrightarrow Fe(OH)_3$$
Faint yellow   Rust colored

$$Cu^{2+} + 4NH_3 \longrightarrow [Cu(NH_3)_4]^{2+}$$
Pale blue   Deep blue

Strawberry red

▲ FIGURE 10.1

There are two simple ways of performing this experiment: either by circular horizontal chromatography or by ascending chromatography using paper strips. One half of the class should perform this experiment using one technique, and the other half should use the other technique. The two techniques can then be compared in terms of time required, separation, and ease of identification.

Make six capillary pipets (2 in. long) by drawing out 6-mm glass tubing. Your instructor will demonstrate how this is done, or you can obtain the capillary tubes from your instructor.

Obtain an unknown; you will analyze this at the same time you are conducting the experiment on the known solutions. Your unknown may contain one, two, or three of the ions being studied. Record the colors of the *known solutions* on your report sheet (1).

Do not pour the chemicals down the drain. Dispose of them in the designated receptacles.

A. Horizontal Circular Technique

Cut a wick about 1 cm wide on a piece of 12.5-cm Whatman # 1 filter paper, as shown in Figure 10.2. Avoid touching the filter paper with oily hands. Trim about 1 cm from the end of the wick so that the filter paper will fit into the petri dish. Mark the center of the paper with a pencil dot (do not use a
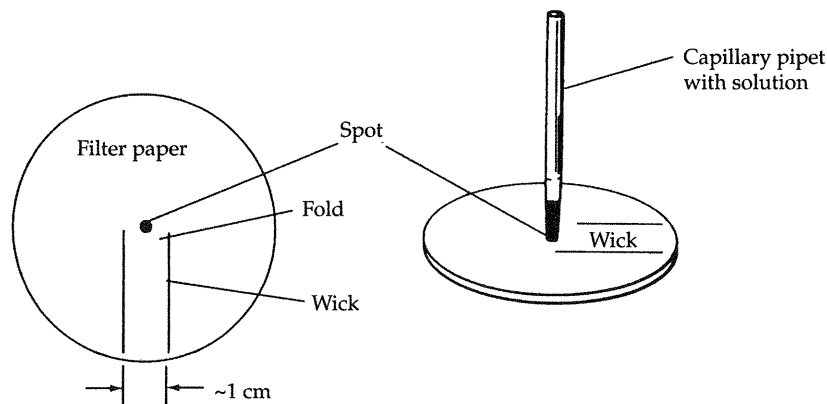
PROCEDURE

▲ FIGURE 10.2

pen). Place the cut filter paper on top of another piece of filter paper or paper towel, which will serve as an absorbing pad. Using the following technique, "spot" the filter paper at the center directly on the pencil dot sequentially with 1 drop of each of the known solutions of Cu^{2+}, Ni^{2+}, and Fe^{3+} (let the spot dry completely between applications). Pour a small quantity (only 1 to 2 mL) of Cu^{2+} solution into a clean sample vial and fill your capillary pipet by dipping the end into the solution. Allow the solution to rise by capillary action. Withdraw the pipet and touch the inside of the vial with the tip of the pipet to remove the hanging drop. Spot the filter paper on the pencil dot by touching it with the capillary held perpendicular to the paper. Allow the solution to flow out of the capillary until a spot having about a 5- to 7-mm diameter is obtained. Dry the filter paper completely by waving it in the air. In the same way, apply a single drop of the Ni^{2+} and then the Fe^{3+} solution to the same spot; be certain that the paper is dry before making each application. Spot a second piece of filter paper that you have prepared for chromatography with 1 drop of your unknown solution in the same manner.

Place two petri (or evaporating) dishes on your desktop away from direct sunlight or heat. Fill the dishes with the solvent mixture to a depth of 5 to 7 mm. (**CAUTION:** *Acetone is highly flammable! Keep it away from open flames.*) Carefully place the (dry) spotted filter papers on the rims of the petri dishes with the wicks bent down into the solvent (Figure 10.3). Cautiously place a 4-in. watch glass on top of each filter paper; be careful not to push the papers into the solvent. The purpose of the cover is to prevent uneven evaporation of the solvent from the paper by providing an enclosed atmosphere that is saturated with solvent vapor. These conditions permit the solvent to travel across the paper in a more uniform manner; this is necessary for effective separation of the ions. Do not disturb the systems while the chromatograms are developing. When the solvent front has nearly reached the edge of each dish (15 to 25 min), carefully remove the watch glass and filter paper. Immediately mark the position of the solvent front with a pencil. Because the solvent evaporates quickly, this marking must be made as soon as possible. Allow each paper to dry by fanning the air with it.

Which of the known ions do you detect without resorting to the use of any other reagents (or development) (2)? Is there any difference between the ring front of this ion and the solvent front (3)? Mark the ring front of this ion.

Developing Reactions   IN THE HOOD, pour about 5 mL of 15 M ammonia into a clean, shallow dish and rest the filter paper containing the knowns on top of the dish. Do not permit the paper to dip into the solution. What color develops (4)? What ion does this indicate (5)? Mark the ring front.

To detect the third ion, dip a new piece of filter paper into a 1% solution of dimethylglyoxime; then, using the new piece as a brush, paint the test filter paper. As an alternative procedure, you may spray the paper with a

Wick   Filter paper   Watch glass   Solvent   Petri dish
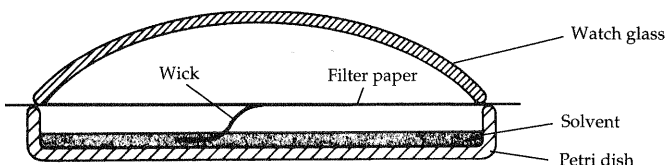
▲ FIGURE 10.3

dimethylglyoxime-containing aerosol. What color develops (6)? What ion does this indicate (7)? Mark the ring front.

Measure the distance to each of the ring fronts in millimeters (8). Calculate the R_f values for the three known ions (9) using Equation [1].

Repeat the above sequence of developing reactions with paper containing your unknown. What ions are present in your unknown (10)? Record the distances in millimeters to the ring fronts (11) and calculate the R_f values (12).
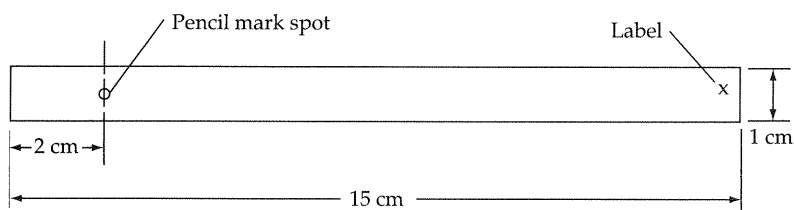
Relative amounts of the components of a mixture can be determined by using the methods employed in this experiment. Prepare a mixture of these ions in the following manner: Mix together 5 mL of the Fe^{3+} and 5 mL of the Ni^{2+} solutions, and then add 2 or 3 drops of the Cu^{2+} solution as a trace contaminant. Apply 1 drop of this mixture to a prepared piece of filter paper. Run and develop the chromatogram. Record your observations (13). Compare your results with those of someone who performed the experiment using the other technique (14).

Dyes You may believe that food coloring consists of one substance or that the black ink in felt-tip pens is a single substance. Spot a piece of filter paper heavily with a black felt-tip pen (such as a Flair pen). Make the spot quite dark to be sure that enough ink has been transferred to the paper. Obtain a chromatogram, using as the solvent a solution prepared by diluting 10 mL of isopropyl alcohol with 5 mL of water.

Attach all the dry, developed chromatograms to your report sheet.

B. Ascending Strip Technique

Obtain a piece of chromatography paper strip about 50 cm long. Cut this into 15-cm strips and mark each strip with a pencil dot. Label as diagrammed in Figure 10.4. You will be running three chromatograms simultaneously by this technique: the known, the unknown, and the trace Cu^{2+}. Place the developing solvent to a depth of 10 to 12 mm in the bottom of a 600-mL beaker (Figure 10.5). Spot the strips as described above for the circles: one strip with the three knowns, one strip with the unknown solution, and one strip with the solution containing a trace of Cu^{2+}. Attach the labeled ends of the strips to a 6-in. glass rod by folding the ends over the rod and clipping the paper together using paper clips (Figure 10.5). Place the rod with the three strips attached across the top of the beaker. Be certain that the spots on the strips are completely dry before you place the strips in the beaker. Make sure that the strips touch neither one another nor the walls of the beaker and that the bottom of each strip is resting in the solution. Cover the beaker carefully with a watch glass. Do not disturb the beaker while the chromatograms are developing. When the solvent front has nearly reached the union of the folded part of the paper, carefully remove the watch glass and the glass rod with the

Pencil mark spot   Label

2 cm   15 cm   1 cm

▲ FIGURE 10.4

REPORT SHEET | EXPERIMENT

Paper Chromatography: Separation of Cations and Dyes

10

1. Color of solutions: Cu^{2+} _____ Fe^{3+} _____ Ni^{2+} _____

2. Ion requiring no development _____

3. Yes _____ No _____

4. Ammonia develops a _____ color.

5. The ion that causes the color of (4) is _____

6. Dimethylglyoxime develops a _____ color with (7) _____ ion.

8. Ring front distances for knowns in mm are: solvent _____ mm;

   Fe^{3+} _____ mm; Cu^{2+} _____ mm; Ni^{2+} _____ mm.

9. R_f values (show calculations at end of report): Fe^{3+} _____ ; Cu^{2+} _____ ;

   Ni^{2+} _____

10. Ions present in unknown _____

11. Ring front distances for unknown _____

    _____

12. R_f values for unknown _____

13. Observation on relative amounts _____

    _____

14. Comment on the relative merits of the two techniques.

Calculations for R_f's (9) and R_f's (12):

ATTACH CHROMATOGRAMS

NOTES AND CALCULATIONS

Analysis of Analgesics

Analgesics are drugs used for pain relief. A common example is aspirin. Since these drugs are so readily available, it is not uncommon for persons to suffer overdoses (either intentional or unintentional.) When a person is admitted to the hospital emergency room with a drug overdose, it is necessary to identify the drug in order to provide appropriate treatment. This analysis can be done on a blood sample. The same technique can be used to study the drug directly, as we will do in this experiment.

Analgesic tablets of many different types are available. These may contain one or more of the following compounds: aspirin (acetylsalicylic acid), phenacetin (p-acetophenotidin), acetaminophen (4-acetamidophenol) and caffeine. The following table shows the ingredients of several different types of analgesic tablets.

Ingredients of Some Analgesic Tablets

Brand	Aspirin	Phenacetin	Acetominophen	Caffeine
Anacin	+	-	-	+
Anacin-3	-	-	+	+
Bayer Aspirin	+	-	-	-
Revco APC	+	+	-	+
Excedrin	+	-	+	+
Tylenol	-	-	+	-

Since different types of analgesics contain different ingredients, it is possible to identify a tablet by determining its active constituents. This can be fairly easily done by using the technique of thin-layer chromatography. (TLC) This technique can be used in many other applications as well. Like all chromatographic techniques, TLC involves a mobile phase and a stationary phase. The stationary phase in this case is a plate coated with silica gel. When one end of this plate is placed in a solvent, (the mobile phase), the solvent travels up the plate (in the same manner that water is drawn up a piece of paper by capillary action). In TLC, a small amount of the sample is placed on the plate and the solvent is allowed to travel up the plate. As the solvent moves, it carries the constituents of the spot with it, but carries some of them further than others. In general, the more attracted one constituent is to the solvent, the further it will move.

When the TLC plate is placed in the solvent, it is important to have the spots above the level of the solvent. It is also necessary to remove the plate from the solvent before the solvent reaches the top of the plate.

If TLC is performed on colored substances, colored spots appear on the chromatogram. The compounds that you are studying in this experiment are all colorless so a slightly different technique is used. The stationary phase is coated with silica gel that has been impregnated with a fluorescent indicator. When your finished chromatogram is viewed under UV light it will be colored everywhere except where the constituents of the analgesic tablets are present.

For this analysis you will compare the components of your tablet with standard solutions (0.1 g/ml) of aspirin, phenacetin, acetaminophen, and caffeine. Each of these solutions will be spotted on your plate along with a solution of your tablet. When the

plate is developed, each compound will have traveled a different distance and will be separated on the plate. If there is a spot from your tablet at the same distance from the bottom as the spot for acetaminophen, for example, you can tell that acetaminophen is present in your analgesic tablet.

Procedure:

Obtain a TLC plate that is about 5 cm by 12 cm.

Prior to putting your sample on the TLC plate it is necessary to separate the analgesics and caffeine from the binders and inorganic components of the tablet. Using a clean mortar and pestle, grind the tablet into a fine powder. Transfer the powder to a piece of weighing paper. Loosely block the narrow end of a Pasteur pipet with a small amount of glass wool. Pour the powder into the pipet. Slowly add 5 ml of methanol, dropwise to the top of the pipet and collect the solvent as it flows through the pipet.

Place small spots of your tablet sample and of the four standard solutions on your TLC plate as demonstrated by your instructor. It is important to keep the spots of sample as small as possible. With a pencil, mark five equal spaces about 5 mm from the bottom of the plate. Be sure to record in your lab notebook which spot is which. When all five spots have been placed on the TLC plate, view the plate under UV light. All five spots should appear dark. If they do not, add more solution to those spots.

An 800 ml beaker will be used as a developing tank. The developing solvent in this experiment is ethyl acetate. Place enough solvent in the tank so that the depth is about 3 mm. Place the TLC plate in the developing tank with the dull side facing up. Make sure that the solvent does not cover the sample spots. Let the plate remain in the tank until the solvent level nearly reaches the top of the plate. At this time, remove the plate and mark the position of the solvent front. This must be done right away as the solvent will dry very quickly and the line will disappear.

Let the developed plate dry for several minutes and then view the plate under UV light. The compounds will appear as dark spots on a light background. With a pencil, draw a circle around each spot. Compare the position of the components of the unknown sample to the position of the spots for the standards to determine which compounds are in the unknown.

On your data sheet, draw a representation of your developed TLC plate and label the spots. Once you have identified the components of your tablet, determine the brand of the analgesic tablet.

Report Sheet: Name: _____

Analysis of Analgesics   Partner's Name: _____

                                        Date: _____

1. What is the identity of your unknown analgesic?

2. Write out the structures of the four possible compounds and identify the
   functional groups present in each:

   a. aspirin (acetylsalicylic acid acetate)

   b. phenacetin (p-acetophenotidin)

   c. acetaminophen (4-acetamidophenol)

   d. caffeine

3. Using the Merck Index, or other appropriate resources, determine the LD_{50} of each compound in question 2 (if more than one type of animal is given, chose one and label the value accordingly).

4. What is meant by LD_{50}?

5. Assuming that the LD_{50} for humans for aspirin is the same as that given in the Merck Index for mice, calculate how many 325 mg aspirin tablets would be necessary to reach the LD_{50} for someone who weighs 150 pounds.

Chemical Formulas

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

Apparatus

balance	250-mL beaker
Bunsen burner and hose	evaporating dish
50-mL graduated cylinder	ring stand and ring
wire gauze	stirring rod
crucible and cover	clay triangle
carborundum boiling chips or	
glass beads	

Chemicals

granular zinc	copper wire
powdered sulfur	6 M HCl

Chemists use an abbreviated notation to indicate the exact chemical composition of compounds (chemical formulas). We then use these chemical formulas to indicate how new compounds are formed by chemical combinations of other compounds (chemical reactions). However, before we can learn how chemical formulas are written, we must first acquaint ourselves with the symbols used to denote the elements from which these compounds are formed.

Symbols and Formulas

We use one or two letters (with the first letter capitalized) to denote a chemical element. These symbols are derived, as a rule, from the first two letters or first syllable of the element's name.

Many elements are found in nature in molecular form; that is, two or more of the same type of atom are tightly bound together. The resultant "package" of atoms, or *molecule*, as it is termed, behaves in many ways as a single distinct object or unit. For example, the oxygen normally found in air consists of molecules that contain two oxygen atoms. We represent this molecular form of oxygen by the chemical formula O_2. The subscript in the formula tells us that two oxygen atoms are present in each oxygen molecule.

Compounds that are composed of molecules are called *molecular compounds*, and they may contain more than one type of atom. For example, a molecule of water consists of two hydrogen atoms and one oxygen atom and is represented by the chemical formula H_2O. The absence of a subscript on the O implies there is one oxygen atom per water molecule. Another compound composed of these same elements, but in different proportions, is
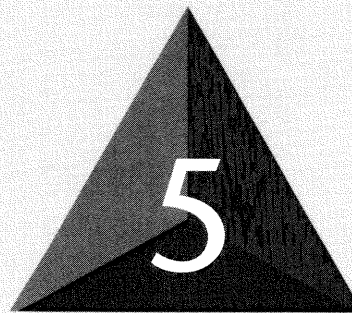
hydrogen peroxide, H_2O_2. The physical and chemical properties of these two compounds are very different. We shouldn't be surprised, for they are two different substances.

Chemical formulas that indicate the *actual* numbers and types of atoms in a molecule are called *molecular formulas*, whereas chemical formulas that indicate only the *relative* numbers of atoms of a type in a molecule are called *empirical formulas*. The subscripts in an empirical formula are always the smallest whole-number ratios. For example, the molecular formula for hydrogen peroxide is H_2O_2, whereas its empirical formula is HO. The molecular formula for glucose is $C_6H_{12}O_6$; its empirical formula is CH_2O. For many substances the molecular formula and empirical formula are identical, as is the case for water, H_2O, and sulfuric acid, H_2SO_4.

Atomic Weights

It is important to know something about masses of atoms and molecules. With a mass spectrometer we can measure the masses of individual atoms with a high degree of accuracy. We know, for example, that the hydrogen-1 atom has a mass of 1.6735×10^{-24} g and the oxygen-16 atom has a mass of 1.674×10^{-24} g. Because it is cumbersome to express such small masses in grams, we use a unit called the *atomic mass unit*, or amu. An amu equals 1.66054×10^{-24} g. Most elements occur as mixtures of isotopes. The average atomic mass of each element expressed in amu is also known as its *atomic weight*. The atomic weights of the elements listed both in the table of elements and in the periodic table inside the front and back covers of this book, respectively, are in amu.

Formula and Molecular Weights

The *formula weight* of a substance is merely the sum of the atomic weights of all atoms in its chemical formula. For example, nitric acid, HNO_3, has a formula weight of 63.0 amu.

$$FW = (AW \text{ of } H) + (AW \text{ of } N) + 3(AW \text{ of } O)$$
$$= 1.0 \text{ amu} + 14.0 \text{ amu} + 3(16.0 \text{ amu})$$
$$= 63.0 \text{ amu}$$

If the chemical formula of a substance is its molecular formula, then the formula weight is also called the *molecular weight*. For example, the molecular formula for formaldehyde is CH_2O. The molecular weight of formaldehyde is therefore

$$MW = 12 \text{ amu} + 2(1.0 \text{ amu}) + 16.0 \text{ amu}$$
$$= 30.0 \text{ amu}$$

For ionic substances such as NaCl that exist as three-dimensional arrays of ions, it is not appropriate to speak of molecules. Similarly, the terms molecular weight and molecular formula are inappropriate for these ionic substances. It is correct to speak of their formula weight, however. Thus, the formula weight of NaCl is

$$FW = 23.0 \text{ amu} + 35.5 \text{ amu}$$
$$= 58.5 \text{ amu}$$

Percentage Composition from Formulas

New compounds are made in laboratories every day, and the formulas of these compounds must be determined. The compounds are often analyzed for their *percentage composition*, i.e., the percentage by mass of each element present in the compound. The percentage composition is useful information in establishing the formula for the substance. If the formula of a compound is known, calculating its percentage composition is a straightforward matter. In general, the percentage of an element in a compound is given by

$$\frac{(\text{Number of atoms of element})(\text{AW})}{\text{FW of compound}} \times 100$$

If we want to know the percentage composition of formaldehyde, CH_2O, whose formula weight is 30.0 amu, we proceed as follows:

$$\%C = \frac{12.0 \text{ amu}}{30.0 \text{ amu}} \times 100 = 40.0\%$$

$$\%H = \frac{2(1.0 \text{ amu})}{30.0 \text{ amu}} \times 100 = 6.7\%$$

$$\%O = \frac{16.0 \text{ amu}}{30.0 \text{ amu}} \times 100 = 53.3\%$$

The Mole

Even the smallest samples we use in the laboratory contain an enormous number of atoms. A drop of water contains about 2×10^{21} water molecules! The unit that the chemist uses for dealing with such a large number of atoms, ions, or molecules is the *mole*, abbreviated mol. Just as the unit dozen refers to 12 objects, the mole refers to a collection of 6.02×10^{23} objects. This number is called Avogadro's number. Thus, a mole of water molecules contains 6.02×10^{23} H_2O molecules, and a mol of sodium contains 6.02×10^{23} Na atoms. The mass (in grams) of 1 mol of a substance is called its *molar mass*. The molar mass (in grams) of any substance is numerically equal to its formula weight.

Thus:

One CH_2O molecule has a mass of 30.0 amu; 1 mol CH_2O has a mass of 30.0 g
and contains 6.02×10^{23} CH_2O molecules

One Na atom has a mass of 23.0 amu; 1 mol Na has a mass of 23.0 g
and contains 6.02×10^{23} Na atoms

It is a simple matter to calculate the number of moles of any substance whose mass and formula we know. For example, suppose we have one quart of rubbing alcohol (generally isopropyl alcohol) and know its density to be 0.785 g/mL and we want to know how many moles this is. First we need to convert the volume to our system of units. Since 1 qt is 0.946 L and 1 L contains 1000 mL, our quart of isopropyl alcohol is

$$(1 \text{ qt})\left(\frac{0.946 \text{ L}}{\text{qt}}\right)\left(\frac{1000 \text{ mL}}{\text{L}}\right) = 946 \text{ mL}$$

We can now calculate the mass:

$$946 \text{ mL} \times 0.785 \text{ g/mL} = 743 \text{ g}$$

We next need the chemical formula for isopropyl alcohol. This is C_3H_7OH. The molecular weight is, therefore,

Weight carbon	$3 \times 12.0 = 36.0$ amu
Weight hydrogen	$8 \times 1.0 = 8.0$ amu
Weight oxygen	$1 \times 16.0 = 16.0$ amu
Molecular weight	$C_3H_7OH = 60.0$ amu

Hence,

$$\text{moles of } C_3H_7OH = (743 \text{ g } C_3H_7OH)\left(\frac{1 \text{ mol } C_3H_7OH}{60.0 \text{ g } C_3H_7OH}\right) = 12.4 \text{ mol}$$

Thus our quart of rubbing alcohol contains 12.4 mol of isopropyl alcohol. It should now be apparent to you how much information is contained in a chemical formula.

Empirical Formulas from Analyses

The empirical formula for a substance tells us the relative number of atoms of each element in the substance. Thus, the formula H_2O indicates that water contains two hydrogen atoms for each oxygen atom. This ratio applies on the molar level as well; thus, 1 mol of H_2O contains two mol of H atoms and 1 mol of O atoms. Conversely, the ratio of the number of moles of each element in a compound gives the subscripts in a compound's empirical formula. Thus, the mole concept provides a way of calculating the empirical formula of a chemical substance. This is shown in the following example.

EXAMPLE 5.1

While you are working in a hospital laboratory, a patient complaining of severe stomach cramps and labored respiration dies within minutes of being admitted. Relatives of the patient later tell you that he may have ingested some rat poison. You therefore have his stomach pumped to verify this and also 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% potassium, 18.5% carbon, and 21.5% nitrogen. What is the chemical formula for this compound?

SOLUTION: One simple and direct way of making the necessary calculations is as follows. Assume you had 100 g of the compound. This 100 g would contain

$$(100 \text{ g})(0.600) = 60.0 \text{ g potassium}$$
$$(100 \text{ g})(0.185) = 18.5 \text{ g carbon}$$
$$(100 \text{ g})(0.215) = 21.5 \text{ g nitrogen}$$

Divide each of these masses by the appropriate atomic weight to obtain the number of moles of each element in the 100 g:

$$60.0 \text{ g K}\left(\frac{1 \text{ mol K}}{39.0 \text{ g K}}\right) = 1.54 \text{ mol K}$$

$$18.5 \text{ g C} \left(\frac{1 \text{ mol C}}{12.0 \text{ g C}} \right) = 1.54 \text{ mol C}$$

$$21.5 \text{ g N} \left(\frac{1 \text{ mol N}}{14.0 \text{ g N}} \right) = 1.54 \text{ mol N}$$

Then divide each number by 1.54 to determine the simplest whole-number ratio of moles of each element. (In general, after determining the number of moles of each element, we determine the simplest whole-number ratio by dividing each number of moles by the smallest number of moles. In this example all the numbers are the same.)

$$K = \frac{1.54}{1.54} = 1.00$$

$$C = \frac{1.54}{1.54} = 1.00$$

$$N = \frac{1.54}{1.54} = 1.00$$

The ratio obtained in this case is 1.00, and we conclude that the formula is KCN. This is the simplest, or empirical, formula because it uses as subscripts the smallest set of integers to express the correct ratios of atoms present. Because 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.

Molecular Formulas from Empirical Formulas

The formula obtained from percentage composition is *always* the empirical formula. We can obtain the molecular formula from the empirical formula if we know the molecular weight of the compound. *The subscripts in the molecular formula of a substance are always a whole-number multiple of the corresponding subscripts in its empirical formula.* The multiple is found by comparing the formula weight of the empirical formula with the molecular weight. For example, suppose we determined the empirical formula of a compound to be CH_2O. Its formula weight is

$$FW = 12.0 \text{ amu} + 2(1.0 \text{ amu}) + 16.0 \text{ amu} = 30.0 \text{ amu}$$

Suppose the experimentally determined molecular weight is 180. Then, the molecule has six times the mass ($180/30.0 = 6.00$) and must, therefore, have six times as many atoms as the empirical formula. The subscripts in the empirical formula must be multiplied by 6 to obtain the molecular formula: $C_6H_{12}O_6$.

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:

$$x\text{Cu}(s) + y\text{S}(s) \longrightarrow \text{Cu}_x\text{S}_y(s)$$
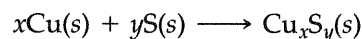
The other is zinc chloride, which you will prepare according to the chemical reaction

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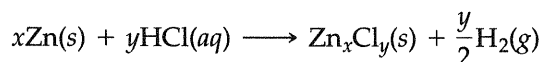
The objective is to determine the combining ratios of the elements (that is, to determine x and y) and to balance the chemical equations given above.

PROCEDURE | ## A. Zinc Chloride

Clean and dry your evaporating dish and place it on the wire gauze resting on the iron ring. Heat the dish with your Bunsen burner, gently at first, and then more strongly, until all of the condensed moisture has been driven off. This should require heating for about 5 min. Allow the dish to cool to room temperature on a wire gauze (do not place the hot dish on the countertop) and weigh it. Record the mass of the empty evaporating dish to the nearest 0.01 g.

Obtain a sample of granular zinc from your laboratory instructor and add about 0.5 g of it to the weighed evaporating dish. Weigh the evaporating dish containing the zinc and record the total mass to the nearest 0.01 g. Calculate the mass of the zinc.

Slowly, and with constant swirling, 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. (**CAUTION:** *No flames are permitted in the laboratory while this reaction is taking place, because hydrogen gas is very explosive.*) If any undissolved zinc remains after the reaction ceases, add an additional 5 mL of acid. Continue to add 5-mL portions of acid as needed until all the zinc has dissolved. **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.*

Set up a steam bath as illustrated in Figure 5.1 using a 250-mL beaker, and place the evaporating dish on the steam bath. Heat the evaporating dish very carefully on the steam bath until most of the liquid has disappeared. Then remove the steam bath and heat the dish on the wire gauze. During this last stage of heating, the flame must be carefully controlled or there will be spattering, and some loss of product will occur. (**CAUTION:** *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 mass. After this first weighing, heat the dish again very gently. Cool it and

Boiling chips
in water
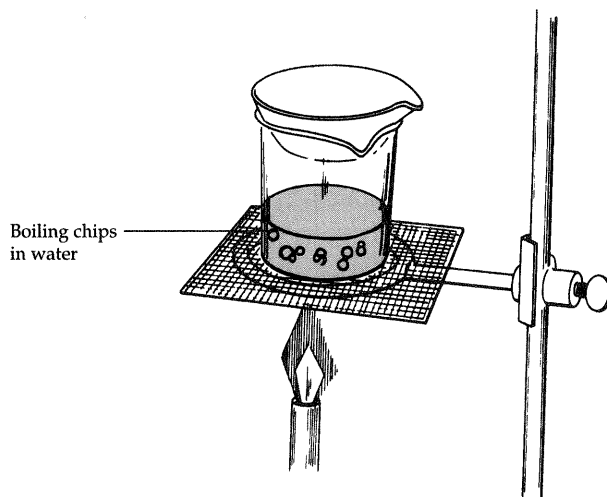
▲ **FIGURE 5.1** Steam bath.

reweigh it. If these weighings do not agree within 0.02 g, repeat the heating and weighing until two successive weighings agree. This is known as *drying to constant mass* and is the only way to be certain that all the moisture is driven off. Zinc chloride is very deliquescent (rapidly absorbs moisture from the air) and so should be weighed as soon as possible after cooling.

Calculate the mass of zinc chloride. The difference in mass between the zinc and zinc chloride is the mass of chlorine. Calculate the mass 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.
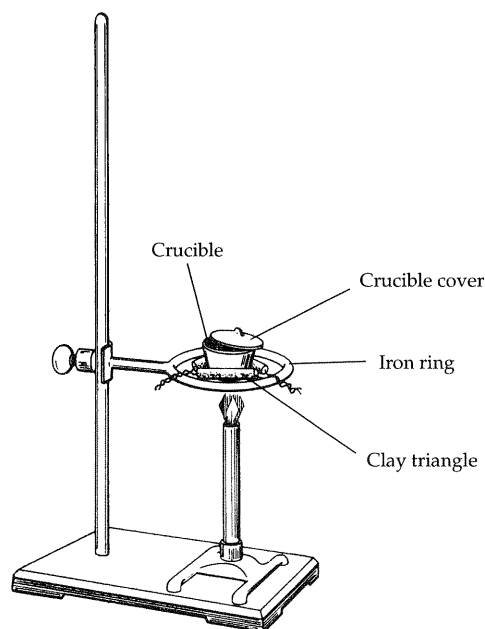
B. Copper Sulfide

Support a clean, dry porcelain crucible and cover on a clay triangle and dry by heating to a dull red in a Bunsen flame, as illustrated in Figure 5.2. Allow the crucible and cover to cool to room temperature and weigh them. Record the mass to the nearest 0.01 g.

Place 1.5 to 2.0 g of tightly wound copper wire or copper turnings in the crucible and weigh the copper, crucible, and lid to the nearest 0.01 g and record your results. Calculate the mass of copper.

In the hood, add sufficient sulfur to cover the copper, place the crucible with cover in place on the triangle, and heat the crucible gently until sulfur ceases to burn (blue flame) at the end of the cover. Do not remove the cover while the crucible is hot. 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 mass. Again cover the

▲ **FIGURE 5.2** Setup for copper sulfide determination.

contents of the crucible with sulfur and repeat the heating procedure. Allow the crucible to cool and reweigh it. Record the mass. 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 mass is obtained.

Calculate the mass of copper sulfide obtained. The difference in mass between the copper sulfide and copper is the mass of sulfur in copper sulfide. Calculate this mass. 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.

Waste Disposal Instructions   All chemicals must be disposed of in the appropriately labeled containers.

PRE LAB QUESTIONS

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

1. Give the chemical symbols for the following elements: (a) lead, (b) iron, (c) fluorine, (e) potassium.

2. What are the formula weights of: (a) KBr, (b) $NiCO_3$?

3. Define the term *compound*.

4. The molecular formula for cyclohexane is C_6H_{12}. What is its empirical formula?

5. Balance the following equation: $Al(s) + Cl_2(g) \longrightarrow AlCl_3(s)$?

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

7. A substance was found by analysis to contain 45.57% tin and 54.43% chlorine. What is the empirical formula for the substance?

8. What is the law of definite proportions?

9. How do empirical and molecular formulas differ?

10. What is the mass in grams of 1.73 moles CaH_2?

11. Air bags in automobiles inflate when sodium azide, NaN_3, rapidly decomposes to its component elements: $2NaN_3(s) \longrightarrow 2Na(s) + 3N_2(g)$

    (a) How many moles of N_2 are formed by the decomposition of 2.50 moles of NaN_3?

    (b) How many grams of NaN_3 are required to form 6.00 g of N_2 gas?

12. A 5.325 g sample of methyl benzoate, a compound found in perfumes, was found to contain 3.758 g of carbon, 0.316 g of hydrogen, and 1.251 g of oxygen. What is the empirical formula? If its molar mass is about 130 g/mol, what is its molecular formula?

13. An analysis of an oxide of nitrogen with a molecular weight of 92.02 amu gave 69.57% oxygen and 30.43% nitrogen. What are the empirical and molecular formulas for this nitrogen oxide? Complete and balance the equation for its formation from the elements nitrogen and oxygen.

14. How many sodium atoms are present in 0.2310 g of sodium?

REPORT SHEET | EXPERIMENT

Chemical Formulas | 5

A. Zinc Chloride

1. Mass of evaporating dish and zinc _____ g
2. Mass of evaporating dish _____ g
3. Mass of zinc _____ g
4. Mass of evaporating dish and zinc chloride:

                  first weighing _____ g

                  second weighing _____ g

                  third weighing _____ g

5. Mass of zinc chloride _____ g
6. Mass of chlorine in zinc chloride _____ g

7. Empirical formula for zinc chloride
   (show calculations) _____

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

B. Copper Sulfide

1. Mass of crucible, cover, and copper _____ g
2. Mass of crucible and cover _____ g
3. Mass of copper _____ g
4. Mass of crucible, cover, and copper sulfide:

                  first weighing _____ g

                  second weighing _____ g

                  third weighing _____ g

5. Mass of copper sulfide _____ g

6. Mass of sulfur in copper sulfide   _____ g

7. Empirical formula for copper sulfide   _____
    (show calculations)

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

QUESTIONS

1. Can you determine the molecular formula of a substance from its percent composition?

2. Given that zinc chloride has a formula weight of 136.28 amu, what is its formula?

3. Can you determine the atomic weights of zinc or copper by the methods used in this experiment? How?
    What additional information is necessary in order to do this?

4. How many grams of zinc chloride could be formed from the reaction of 3.57 g of zinc with excess HCl?

5. Aluminum sulfide reacts with water to form aluminum hydroxide and hydrogen sulfide. (a) Write a balanced equation for this reaction. (b) How many grams of aluminum hydroxide can be obtained from 10.5g of aluminum sulfide?

6. If copper(I) sulfide is partially roasted in air (reaction with O_2), copper(I) sulfite is first formed. Subsequently, upon heating, the copper sulfite thermally decomposes to copper(I) oxide and sulfur dioxide. Write balanced chemical equations for these two reactions.

NOTES AND CALCULATIONS

Chemical Reactions

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

OBJECTIVE

Apparatus

Bunsen burner	crucible and cover
6-in. test tube	glass tubing
thistle tube or long-stem funnel	ring stand, ring, wire triangle
droppers (2)	tongs

APPARATUS AND CHEMICALS

Chemicals

0.1 M sodium oxalate, $Na_2C_2O_4$	0.1 M $KMnO_4$
10 M NaOH	0.1 M $Pb(NO_3)_2$
1 M K_2CrO_4	6 M HCl
mossy zinc	6 M H_2SO_4
6 M $NH_3(aq)$*	3 M $(NH_4)_2CO_3$
conc. HNO_3	$KMnO_4$
0.1 M $NaHSO_3$ (freshly prepared)	Na_2CO_3
2-in. length of copper wire	Na_2SO_3
2 1-in. lengths of copper wire	ZnS
(14, 16, or 18 gauge)	
0.1 M $BaCl_2$	
powdered sulfur	

Chemical equations represent what transpires in a chemical reaction. For example, the equation

DISCUSSION

$$2KClO_3(s) \xrightarrow{\Delta} 2KCl(s) + 3O_2(g)$$
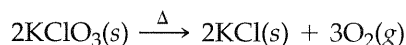
means that potassium chlorate, $KClO_3$, decomposes on heating (Δ is the symbol used for heat) to yield potassium chloride, KCl, and oxygen, O_2. *Before* an equation can be written for a reaction, someone must establish what the products are. How does one decide what these products are? Products are identified by their chemical and physical properties as well as by analyses. That oxygen rather than 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, consult Table 4.1, which lists some of the properties of the substances that could be formed in these reactions.

*Reagent bottle may be labeled 6 M NH_4OH.

TABLE 4.1 Properties of Reaction Products

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

PROCEDURE | **A. A Reaction Between the Elements Copper and Sulfur**

PERFORM THIS EXPERIMENT IN THE HOOD WITH A PARTNER. Obtain about a 5-cm (2-in.) length of copper wire and note its properties. Observe that its surface is shiny, that it can be easily bent, and that it has a characteristic color. Make a small coil of the wire by wrapping it around your pencil, and place the wire coil in a crucible. Add sufficient powdered sulfur to the crucible. Cover and place it on a clay triangle on an iron ring for heating. THIS APPARATUS MUST BE SET UP IN THE HOOD, because some sulfur will burn to form noxious sulfur dioxide. Heat the crucible with a Bunsen burner initially with low heat on all sides and then use the hottest flame to heat the bottom of the crucible to red heat. CONTINUE heating until no more smoking occurs, indicating that all the sulfur is burned off. Using the crucible tongs, remove the crucible from the clay triangle without removing the cover, and place it on a heat-resistant pad or wire gauze, *not on the desktop*, to cool. After the crucible has cooled, remove the cover and inspect the substance. Note its properties. Be sure to record your answers on the Report Sheet.

1. Does the substance resemble copper?
2. Is it possible to bend the substance without breaking it?
3. What color is it?
4. Has a reaction occurred?

Copper(II) sulfide, CuS, is insoluble in aqueous ammonia, NH_3, (that is, it does not react with NH_3), whereas 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 Bunsen burner.

5. Does your product react with NH_3?
6. Suggest a possible formula for the product.
7. Write a reaction showing the formation of your proposed product:

$$Cu(s) + S_8(s) \longrightarrow ?$$
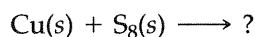
Waste Disposal Instruction The copper compounds and the acids are toxic and should be handled with care. Avoid spilling any solution, and immediately clean up (using paper towels) any spills that occur. If you spill any solution on your hands, wash them immediately. After completing each series of reactions and before moving on to the next series, dispose of the contents of your test tubes in the designated receptacles. Do not wash the contents down the sink.

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 with nitric acid, HNO_3; 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.

8. Record your observations.
9. 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.

10. Record your observations.
11. 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.

12. Record your observations.
13. Is the gas colored?
14. Suggest a formula for the gas.
15. After the reaction has proceeded for 5 min, carefully add 5 mL of water. Based on 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. Place 1 mL of 0.1 M sodium oxalate, $Na_2C_2O_4$, in a clean test tube. Add 10 drops of 6 M sulfuric acid. Mix thoroughly. To the resulting solution add 1 to 2 drops of 0.1 M $KMnO_4$ and stir. If there is no obvious indication that a reaction has occurred, warm the test tube gently in a hot water bath.

16. Record your observations. Was the $KMnO_4$ reduced to Mn^{2+}?

Place 3 mL of 0.1 M sodium hydrogen sulfite, $NaHSO_3$, solution in a test tube. Add 1 mL of 10 M sodium hydroxide, NaOH, solution and stir. To the mixture in the test tube add 1 drop of 0.1 M $KMnO_4$ solution.

17. Record your observations. Was the $KMnO_4$ reduced? Identify the manganese compound formed.

Add additional 0.1 M $KMnO_4$ solution, one drop at a time, and observe the effect of each drop until 10 drops have been added.

18. Record your observations.
19. Suggest why the effect of additional potassium permanganate changes as more is added.

WHILE HOLDING A TEST TUBE IN THE FUME HOOD, add one or two crystals of potassium permanganate, $KMnO_4$, to 1 mL of 6 M HCl.
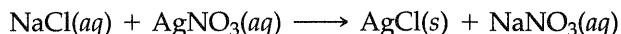
20. Record your observations.
21. Note the color of the gas evolved.
22. Based on the color of the gas, what is the gas?

C. Metathesis Reactions

Additional observations are needed before equations can be written for the reactions 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:

$$AB + CD \longrightarrow AD + CB$$
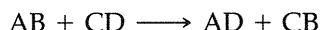
These are called double-decomposition, or *metathesis*, reactions. This type of reaction involves the exchange of atoms or groups of atoms between interacting substances. The following is a specific example:

$$NaCl(aq) + AgNO_3(aq) \longrightarrow AgCl(s) + NaNO_3(aq)$$

Place a small sample of sodium carbonate, Na_2CO_3, in a test tube and add several drops of 6 M HCl.

23. Record your observations.
24. Note the odor and color of the gas that forms. (See safety instructions.)
25. What is the evolved gas?
26. Write an equation for the reaction $HCl(aq) + Na_2CO_3(s) \longrightarrow$? (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 and H_2SO_3 readily decompose as follows:

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

$$H_2SO_3(aq) \longrightarrow H_2O(l) + SO_2(g)$$
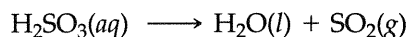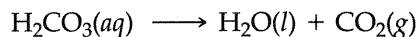
IN THE HOOD, repeat the same test with sodium sulfite, Na_2SO_3.

27. Record your observations.
28. What is the gas?
29. Write an equation for the following reaction (note the similarity to the equation above): $HCl(aq) + Na_2SO_3(s) \longrightarrow$?

IN THE HOOD, repeat this test with zinc sulfide, ZnS.

30. Record your observations.
31. What is the gas?
32. Write an equation for the reaction: $HCl(aq) + ZnS(s) \longrightarrow$?

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.

33. Record your observations.
34. What is the precipitate?
35. Write an equation for the reaction $Pb(NO_3)_2(aq) + HCl(aq) \longrightarrow$?

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

36. Record your observations.
37. What is the precipitate?
38. 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.

39. What is the precipitate?
40. Write an equation for the reaction $(NH_4)_2CO_3(aq) + BaCl_2(aq) \longrightarrow$?

After the precipitate has settled somewhat, carefully decant (that is, pour off) the excess liquid. Add 1 mL of water to the test tube, shake it, allow the precipitate to settle, and again carefully pour off the liquid. To the remaining solid, add several drops of 6 M HCl.
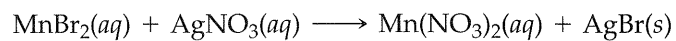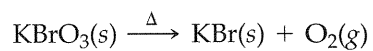
41. Record your observations.
42. Note the odor.
43. What is the evolved gas? (Recall the reaction in step 26 of this experiment.)

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

PRE LAB QUESTIONS

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

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

3. How could you distinguish between NH_4Cl and $AgCl$?

4. Define metathesis reactions. Give an example.

5. What is a precipitate?

6. Balance these equations:

$$KBrO_3(s) \xrightarrow{\Delta} KBr(s) + O_2(g)$$

$$MnBr_2(aq) + AgNO_3(aq) \longrightarrow Mn(NO_3)_2(aq) + AgBr(s)$$

7. How could you distinguish between the gases H_2 and NO_2?

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 $H_2SO_3(aq)$.

REPORT SHEET | EXPERIMENT
Chemical Reactions | 4

A. A Reaction Between the Elements Copper and Sulfur

1. _____

2. _____

3. _____

4. _____

5. _____

6. _____

7. $Cu(s) + S_8(s) \longrightarrow$ _____

B. Oxidation-Reduction Reactions

8. _____

9. $Zn(s) + HCl(aq) \longrightarrow$ _____

10. _____

11. _____

12. _____

13. _____
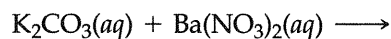
14. _____

15. _____
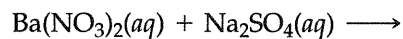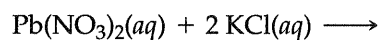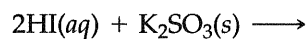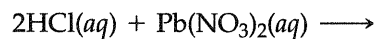
16. _____

17. _____

18. _____

19. _____

20. _____

21. _____

22. _____

C. Metathesis Reactions

23. _____

24. _____

25. _____

26. $HCl(aq) + Na_2CO_3(s) \longrightarrow$ _____

27. _____

28. _____

29. $HCl(aq) + Na_2SO_3(s) \longrightarrow$ _____

30. _____

31. _____

32. $HCl(aq) + ZnS(s) \longrightarrow$ _____

33. _____

34. _____

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

36. _____

37. _____

38. $BaCl_2(aq) + K_2CrO_4(aq) \longrightarrow$ _____

39. _____

40. $(NH_4)_2CO_3(aq) + BaCl_2(aq) \longrightarrow$ _____

41. _____

42. _____

43. _____

QUESTIONS:

1. Complete and balance the following chemical reactions:

   $2HCl(aq) + Pb(NO_3)_2(aq) \longrightarrow$

   $2HI(aq) + K_2SO_3(s) \longrightarrow$

   $Pb(NO_3)_2(aq) + 2\,KCl(aq) \longrightarrow$

   $Ba(NO_3)_2(aq) + Na_2SO_4(aq) \longrightarrow$

   $K_2CO_3(aq) + Ba(NO_3)_2(aq) \longrightarrow$

   $HCl(aq) + AgNO_3(aq) \longrightarrow$

2. How could you separate gold from a mixture of zinc and gold?

3. How could you determine whether a white powder was zinc sulfide or silver nitrate using a hydrochloric acid solution? Write balanced equations.

NOTES AND CALCULATIONS

Water of Crystallization and Formula of a Hydrate

Process Objectives

♦To determine that a reaction is complete by heating a sample to constant mass.

♦To calculate the moles of the anhydrous substance and the water of crystallization used in the experiment.

Learning Objectives

♦To determine the empirical formula of a hydrate when the formula of the anhydrous compound is known.

Hypothesis

♦Predict the percent accuracy of hydrate (number of waters) you determine relation to the actual number.

Apparatus   Materials

Balance, centigram   sparker   C.P. hydrate
crucible and cover   iron ring   crystals
tongs   pipestem triangle   magnesium sulfate
ring stand   spatula
burner and tubing

Pre-Lab Questions

1. Throughout the experiment, handle the crucible and cover with clean crucible tongs only. Why?

2. Why is preheating the crucible and cover necessary for 2 to 3 minutes?

3. (a) Why is it important to have a lid on the crucible?
   (b) Why do you need an opening between the lid and crucible?

4. Why is procedure #5 necessary?

5. How does this experiment exemplify the law of definite composition?

6. The following results were obtained when a solid was heated by three different pairs of students. In each case the student observed that when they began to heat the solid, drops of a liquid formed on the sides of the test tube.

Student Number	Mass before Heating	Mass after Heating
1	1.48g	1.26g
2	1.64g	1.40g
3	2.08g	1.78g

    (a) Could the solid be a hydrate? What is your evidence?

    (b) If the solid after heating has a molar mass of 208 g/mole and a formula of XY, what is the formula of the hydrate?

7. Some cracker tins include a glass vial of drying material in the lid. This is often a mixture of magnesium sulfate and cobalt chloride indicator. As the magnesium sulfate absorbs moisture($MgSo_4 \cdot 1 H_2O \rightarrow MgSO_4 \cdot 7H_2O$) the indicator changes color from blue to pink ($CoCl_2 \cdot 4H_2O \rightarrow CoCl_2 \cdot 6H_2O$). When this drying mixture becomes totally pink, it can be restored be heating in the oven. What two changes are caused by the heating?

8. Study the specific properties of the compounds listed in the following table to determine which lend themselves to this method of determining percentage hydration. Which do not? Why?

| Name of compound	Loses mass when heated	Description of Residue	Is it a hydrate?
Sodium carbonate	yes	white powder	
sodium sulfate	yes	white powder	
sodium aluminum sulfate	yes	white powder	
potassium chloride	no	unchanged	
magnesium chloride	yes	white powder	
Copper sulfate	yes	white powder	

Procedures

Strategy for Determining
It is very difficult to determine that a reaction is complete by observation of the compounds. Only when the mass has reached a constant value has all of the water been removed. Then constant mass can be determined by repeating the heat-cool weigh cycle until the mass does not change significantly.

Strategy for Calculating
Carefully, quantitative techniques will improve the accuracy of your calculations. The same balance should be used for all weighings and the crucible should not be touched with the hands. Be sure to cool the crucible and the contents sufficiently as heat may affect the operation of the balance.

Crucible

Clay triangle

Figure 10-1
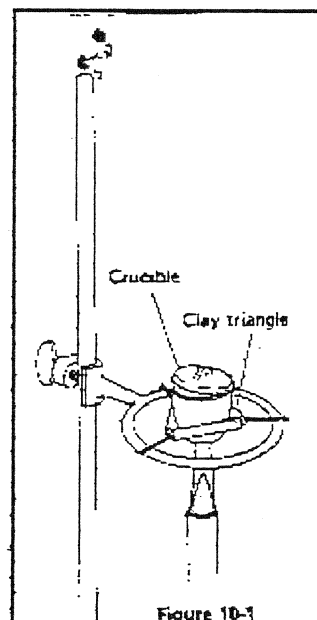
1. Place the crucible and cover on the triangle to be heated. Position the cover slightly tipped, leaving only a small opening for any gases to   escape. Preheat the crucible and cover to redness.

    CAUTION: Before lighting the burner, remember to confine loose clothing and long hair, remember to handle the crucible and cover only with tongs. THE crucible and cover are very hot after each heating.

    Allow them to cool 5 Minutes. Never place a hot crucible on a balance. When cool, determine the mass of the crucible and cover to the nearest 0.01g. Record this mass in your Data Table.

2. Using a spatula, add approximately 5g of fine magnesium sulfate crystals to the crucible. Determine the mass of the covered crucible and crystals to the nearest 0.01g. Record this mass in your Data Table.

3. Place the crucible with the hydrate on the triangle and again position the cover so there is only a small opening. Too large an opening may allow the hydrate to splatter out of the crucible. Heat the crucible very gently on a low flame to avoid splattering any of the hydrate. Increase the temperature gradually for 2 to 3 minutes. Then heat strongly (red-hot) for at least 5 minutes. Allow the crucible, cover, and contents to cool for 5 minutes and then determine their mass. Enter all masses, properly labeled, in your Data Table.

4. Heat the covered crucible and contents again to redness for 5 minutes. Allow the crucible, cover, and contents to cool and then determine their mass. If the last two mass determination differ be no more than 0.01g, you may assume all that the water has been driven off. Other wise repeat the process, until the mass no longer changes. This means all the water has been evaporated. Record this constant mass in your Data Table. The dehydrated compound left in the crucible should be returned to your instructor, since it can be used in the preparation of solutions.

5. At the end of the experiment, clean all apparatus. Check to see that the gas valve is completely shut off before leaving the laboratory, and remember to wash your hands.

Data Table

Mass of empty crucible and cover		g
Mass of crucible, cover, and hydrate		g
Mass of crucible, cover, and anhydrous MgSo$_4$ after 1st heating		g
Mass of crucible, cover, and anhydrous MgSo$_4$ after second heating		g

Calculations

Show your computations in the spaces provided below. Place your answers in the Calculations Table.

1. Calculate the <u>mass</u> of the anhydrous magnesium sulfate (the residue that remains after driving off the water).

2. Calculate the <u>moles</u> of anhydrous Magnesium Sulfate.

3. Calculate the mass of water driven from the hydrate.

4. Calculate the moles of water driven from the hydrate.

5. The reaction for this experiment is: magnesium sulfate

$$MgSo_4 \cdot nH_2O(s) \rightarrow MgSO_4 + nH_2O$$
   hydrate   anhydrous
                      hydrate
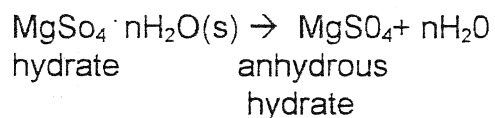
n= the number of moles of water driven off per mole of anhydrous barium chloride. Determine this mole ratio to the nearest whole number, and write the empirical formula for the Magnesium sulfate.