USE OF LABORATORY EQUIPEMENT

A. Laboratory Burners

Almost all laboratory burners used today are modifications of a design by the German chemist Robert Bunsen. In Bunsen's fundamental design, also widely used in domestic and industrial gas burners, gas and air are premixed by admitting the gas at a relatively high velocity from a jet in the base of the burner. This rapidly moving stream of gas causes air to be drawn into the barrel from side ports and to mix with the gas before entering the combustion zone at the top of the burner.

The burner is connected to a gas cock by a short length of rubber or plastic tubing. With some burners the gas cock is turned to the **fully on** position when the burner is in use, and the amount of gas admitted to the burner is controlled by adjusting a needle valve in the base of the burner. In burners that do not have this needle valve, the gas flow is regulated by partly opening or closing the gas cock. With either type of burner **the gas should always be turned off at the gas cock when the burner is not in use** (to avoid possible dangerous gas leakage from the needle valve or old tubing).

Operation of the Burner. Examine the construction of your burner and familiarize yourself with its operation. A burner is usually lighted with the air inlet ports nearly closed. The ports are closed by rotating the barrel of the burner in a clockwise direction. After the gas has been turned on and lighted, the size and quality of the flame is adjusted by admitting air and regulating the flow of gas. Air is admitted by rotating the barrel; gas is regulated with the needle valve, if present, or the gas cock. Insufficient air will cause a luminous yellow, smoky flame; too much air will cause the flame to be noisy and possibly blow out. A Bunson burner flame that is satisfactory for most purpose should be blue, such a flame is said to be "nonluminous." Note that the hottest region is immediately above the bright blue cone of a well-adjusted flame.

B. Laboratory Balances

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 the centigram (0.00) and the analytical (0.0000). 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. These balances are not necessarily cross-calibrated so if you start with one particular balance, stay with that same balance! Remember to "TARE" the balance prior to any weighing. To "TARE" means to zero out the balance. Below are some general rules about using a digital balance.

MEASURING MASS - WEIGHING

You will no doubt receive specific instructions on the use of chemical balances in your laboratory. No attempt will be made to duplicate those instructions here. Instead, comments will be limited to some general suggestions, plus identification of a term that has special meaning throughout this course.

Chemicals are *never* weighed directly on the pan of a laboratory balance. Instead, the mass is determined by a process known as ***weighing by difference*****.** A suitable container - a small beaker, or weighing boat, or perhaps a test tube that is to be used in the experiment - is weighed empty on the balance. The desired chemical is added to the container, and the total mass of the combination is determined. Then by subtracting the mass of the empty container from the mass of the container plus chemical, you find the mass of the chemical.

Throughout this course the word **container** is used to include any and all objects that pass through the entire experiment unchanged in mass. In addition to a test tube, for example, you might include in the mass of the **"container"** which could be either a test-tube holder by which the test tube is suspended on a balance during weighing, or the mass of a beaker in which the test tube is held for weighing. For example in chemistry 101, in

one experiment the mass of a liquid is measured in a Erlenmeyer flask that is covered with a Styrofoam cup. The cup is weighed with the empty flask, and their combined masses make up the mass of the **"container."** In the various experiments where you see the "container" identified, the word has the meaning given in this paragraph.

Sometimes students use containers that are not actually part of the experiment in taking samples of solid chemicals. Most common is the practice of placing a piece of weighing paper on the pan of a balance, transferring the required quantity of chemical to the paper, and then transferring it to the vessel to be used in the experiment. If you use this technique to obtain a measure mass of the chemical, your first weighing should be of the paper with the chemical on it. Then transfer the chemical, and bring the paper back for a second weighing. This way your difference will be the mass of the chemical actually transferred, unaffected by any chemical that may have remained on the paper unnoticed. In this method you should use a hard, smooth paper - waxed weighing paper is best - rather than coarse paper, such as paper towel, which is certain to trap powders and tiny crystals.

Laboratory balances are subject to corrosion. Both the balances and the balance area should be kept clean, and spilled chemicals should be cleaned up immediately.

Here are a few miscellaneous pointers on proper balance operation, given as a series of "do's and don'ts," with some items in both lists for emphasis:

DO: **Allow hot objects to cool to room temperature before weighing.**
Close the side doors or hood of a milligram balance while weighing.
Record all digits allowed by the accuracy of the balance used, even if the last digit happens to be a zero on the right side of the decimal point.
Tare the balance prior to any weighing made.

DON'T : **Weigh objects that are warm or hot.**
Weigh objects that are wet (evaporation of water will change the mass).
Weigh volatile liquids in uncovered vessels.
Touch the object with your hand if you are using a milligram or analytical balance; your fingerprints have weight, too!
Forget to check the zero on a milligram balance after weighing.
Forget to record the mass to as many digits as the accuracy of the balance allows - and no more.

General "how to" directions for using the balances.

Your instructor will give specific directions on how to use the balance, but the following precautions should be observed:

1. The balance should always be "zeroed" before anything is placed on the balance pan. On an electronic digital balance, this is done with the "tare" or "T" button. Balances without this feature should be adjusted by the instructor.
2. Never place chemicals directly on the balance pan; first place them on a weighing paper, weighing "boat", or in a container. Clean up any materials you spill on or around the balance.
3. Before moving objects on and off the pan, be sure the balance is in the "arrest" position. When you leave the balance, return the balance to the "arrest" or standby position.
4. Never try to make adjustments on a balance. If it seems out of order, tell your instructor.

<u>SIGNIFICANT FIQURES in relationship to Balances</u>

Precise Quantities versus Approximate Quantities

In conducting an experiment it is often unnecessary to measure an exact quantity of material. For instance, the directions might state, "Weigh about 2 g of sodium sulfite." This instruction indicates that the measured quantity of salt should be 2 g plus or minus a small quantity. In this example 1.8 to 2.2 g will satisfy these requirements. To weigh exactly 2.00g or 2.000 g wastes time since the directions call for approximately 2 g.

Sometimes it is necessary to measure an amount of material precisely within a stated quantity range. Suppose the directions read, "Weigh about 2 g of sodium sulfite to the nearest 0.001g." This instruction does not imply that the amount is 2.000 g but that it should be between 1.8 and 2.2 g and measured and recorded to three

decimal places. Therefore, four different students might weigh their samples and obtain 2.141 g, 2.034 g, 1.812 g, 1.937 g, respectively, and each would have satisfactorily followed the directions.

Significant Figures in Calculations

The result of multiplication, division, or other mathematical manipulation cannot be more precise than the least precise measurement used in the calculation. For instance, suppose we have an object that weighs 3.62 lb. and we want to calculate the mass in grams (3.62 lb.) ($\frac{453.6g}{1lb}$) = 1,642.032 when done by a calculator. To report 1,642.032 g as the mass is absurd, for it implies a precision far beyond that of the original measurement. Although the conversion factor has four significant figures, the mass in pounds has only three significant figures. Therefore the answer should have only three significant figures; that is, 1,640 g. In this case the zero cannot be considered significant. This value can be more properly expressed as 1.64 x 10^4 g.

C. Laboratory 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 as 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 tam of pressure (1 tam = 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.

°F = 1.8 °C + 32

The Thermometer and Its Calibration

This section describes the proper technique for checking 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 depends upon atmospheric pressure but at sea level it is 100°C. Option 1: 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. Option 2: Set up a 250-mL beaker on a wire gauze and iron ring. Fill the beaker about half full with distilled water. 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.

Significant figures in relationship to Temperature

The simple act of measuring a temperature with a thermometer can easily involve errors. Not only does the calibration of the scale on the thermometer limit the precision of the measurement, but the improper placement of the thermometer bulb in the material being measured introduces a common source of human error. When measuring the temperature of a liquid, one can minimize this type of error by observing the following procedures:

1. Hold the thermometer away from the walls of the container
2. Allow sufficient time for the thermometer to reach equilibrium with the liquid.
3. Be sure the liquid is adequately mixed.

When converting from degrees Celsius to Fahrenheit or vice versa, we make us of the following formulas:

$°C = \left(\frac{°F-32}{1.8}\right)$ of °F = (1.8 x °C) + 32

Example Problem: Convert 70.0°F to degrees Celsius:

$°C = \left(\frac{70.0°F-32}{1.8}\right) = \frac{38.0}{1.8}$ = 21.11°C rounded to 21.1°C

This example shows not only how the formula is used by also a typical setup of the way chemistry problems should be written. It shows how the numbers are used, but does not show the multiplication and division, which should be worked out by calculator. The answer was changed from 21.11 °C to 21.1 °C because the initial temperature, 70.0 °F, has only three significant figures. The 1.8 and 32 in the formulas are exact numbers and have no effect on the number of significant figures.

D. Filtration in the Laboratory

The process of separating suspended insoluble solids from liquids by means of filters is called **filtration.** Insoluble solids, called **precipitates,** are formed during some chemical reactions. In the laboratory these precipitates are generally separated from the solutions by filtering them out on a paper filter. The liquid that passes through the filter paper is the **filtrate;** the solid precipitate remaining on the filter paper is the **residue.**

Filtering a Product.

(a) Fold a circle of filter paper in half. Fold in half again and open out into a cone. Tear off one corner of the outside folded edge. The top edge of the cone which is to touch the glass funnel should not be torn.

(b) Fit the opened cone into a short-stemmed funnel, placing the torn edge next to the glass. Wet with distilled water and press the top edge of the paper against the funnel, forming a seal. Use an iron ring clamp with a clay triangle on top, all attached to a ring stand for supporting the funnel. Then, stir the mixture of products in the small beaker with stirring rod and slowly pour it down the stirring rod into the filter paper in the funnel. Do not overfill the paper filter cone.

(c) *Decantation.* This is the process of separating a liquid from a solid (sediment) by gently pouring the liquid from the solid so as not to disturb the solid.

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

(e) *Extraction.* This is the separation of a substance from a mixture by preferentially dissolving that substance in a suitable solvent. By this process a soluble compound is usually separated from an insoluble compound.

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

E. Laboratory Volumetric Measurements

READING VOLUMETRIC GLASSWARE

When a liquid is placed into a glass container it forms a **meniscus**, a curved surface that is lower in the middle than at the edge. Volumetric laboratory equipment is calibrate to measure volume by sighting to the *bottom* of the meniscus. Notice that it is essential that the line of sight be perpendicular to the calibrated vessel if you are to read it accurately. It is also important that you hold the vessel vertically or, for more precise measurement, place it on a level surface.

Four types of calibrated glassware are used in this laboratory. The most accurately calibrated are volumetric pipets and flasks. Next are the burets. Most of your volume measurements will be made in graduated cylinders. Their main purpose is to measure volumes and they are designed and calibrated accordingly. Beakers and Erlenmeyer flasks made by some manufactures are also “calibrated,” even though the function of these items has nothing to do with measuring volume. The calibrations on beakers are flasks give only ***very rough*** indications

of volume up to a certain level in the vessel. Volumes estimated by these calibrations should ***never*** be used in calculations.

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.

Pipets

Pipets are glass vessels that are constructed and calibrated so as to deliver a precisely known volume of liquid at a given temperature. *Always* use a rubber bulb to fill a pipet. NEVER USE YOUR MOUTH! NO pipet should not be blown empty. Some of the pipets used have divisions of 0.01 mL, while others (transfer or plastic) pipets have no graduates and must be calibrated via the drop method (count the number of drops it takes to reach 1-mL. Keep in mind this is still an estimate.). Check each pipet divisions prior to using. 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.

Burets

Burets are a piece of volumetric glassware, usually graduated in 0.1-mL intervals, that is used to deliver solutions to be used in titrations in a quantitative (dropwise) manner.

Reading a Buret: All liquids, when placed in a buret, form a curved meniscus at their upper surfaces. In the case of water or water solutions, this meniscus is concave, and the most accurate buret readings are obtained by observing the position of the lowest point on the meniscus on the graduated scales. To avoid parallax errors when taking readings, the eye must be on a level with the meniscus. Wrap a strip of paper around the buret and hold the top edges of the strip evenly together. Adjust the strip so that the front and back edges are in line with the lowest part of the meniscus and take the reading by estimating to the nearest tenth of a marked division (0.01 mL).

Preparation of a Buret for use: Clean a 50-mL buret with soap solution and thoroughly rinse with tap water, at least 3 times. Then rinse with at least three 15-mL portions of distilled water. The water must run freely from the buret without leaving any drops adhering to the sides. Make sure that the buret does not leak and that the stopcock turns freely.

BURET & TITRATION: Definition of some common terms used

Titration is the controlled addition of a solution into a reaction vessel from a buret. By means of titration, the volume of solution used may be determined quite precisely. The titration process is used in many analytical determinations, including those involving acid-base reactions.

An **indicator** is a substance used to signal when the titration arrives at the point at which the reactants are stoichiometrically (or chemically) equal, as defined by the reaction equation. For example, in an acid-base titration between sodium hydroxide and hydrochloric acid,

$$NaOH\ (aq) + HCl\ (aq) \rightarrow 2\ H^2O(l) + NaCl\ (aq)$$

the indicator should tell when the numbers of moles of NaOH and HCl are exactly equal, matching the 1:1 ratio in the equation. This point of chemical equality is called the ***equivalence point*** of the titration. A suitable indicator changes colors when equivalent amounts of acid and base are present. The color change is termed the ***end point*** of the titration. Indicators change colors at different pH values. Phenolphthalein, for example, changes from colorless to pink at pH of about 9. In slightly more acidic solutions it is colorless, whereas in more alkaline

solutions it is pink. Acid-base indicators send their signal by changing color at or very near the equivalence point of the titration.

A **standard solution** is a solution with a precisely determined concentration. Initially the concentration of a standard solution is determined from a weighed quantity of a **primary standard,** a highly purified reference chemical. A standard solution may be prepared in either of two ways:

1. A primary standard is carefully weighed, dissolved, and diluted accurately to a known volume. Its concentrations can be calculated from the data.
2. A solution is made to an approximate concentration and then standardized by titrating an accurately weighed quantity of a primary standard.

Once a solution has been standardized in one reaction, it may be used as a standard solution in subsequent reactions. Thus the standard solution prepared in Experiment 12A (chemistry 101) will be used in the reaction of Experiment 12B to determine the concentration of an unknown acid.

F. Qualitative-Analysis Techniques in the Laboratory

MIXING SOLUTIONS AND PRECIPITATION

When one solution is added to another in a small test tube, it is important that the two be thoroughly mixed. Mixing can be accomplished by using a *clean* stirring rod, or it can be achieved by holding the test tube at the top in one hand and tapping or "tickling" it with the fingers of the other hand. When precipitation reagents are added to solutions in the test tube and it is believed that precipitation is complete, centrifuge the sample. Always balance the centrifuge by placing a test tube filled with water to about the same level as your sample test tube directly *across* the centrifuge head from your sample. It usually requires only about 30- 45 sec of centrifugation for the precipitate to settle to the bottom of the test tube. ***Caution: Do not slow down the centrifuge head with your hands. Instead, allow the centrifuge head to come to rest on its own accord.***

DECANTATION AND WASHING OF PRECIPITATES

The liquid above the precipitate is the *supernatant liquid,* or the *decantate*. The best way to remove this liquid without disturbing the precipitate is to withdraw it by means of a capillary pipet. We loosely refer to this operation as *decantation.* Because the precipitate separated from the supernatant liquid by this technique will be wet with decantate, it is necessary to *wash* the precipitate free of contaminating ions. Washing is usually accomplished by adding about 10 drops of distilled water to the precipitate, stirring with a stirring rod, and repeating the centrifuging and decanting.

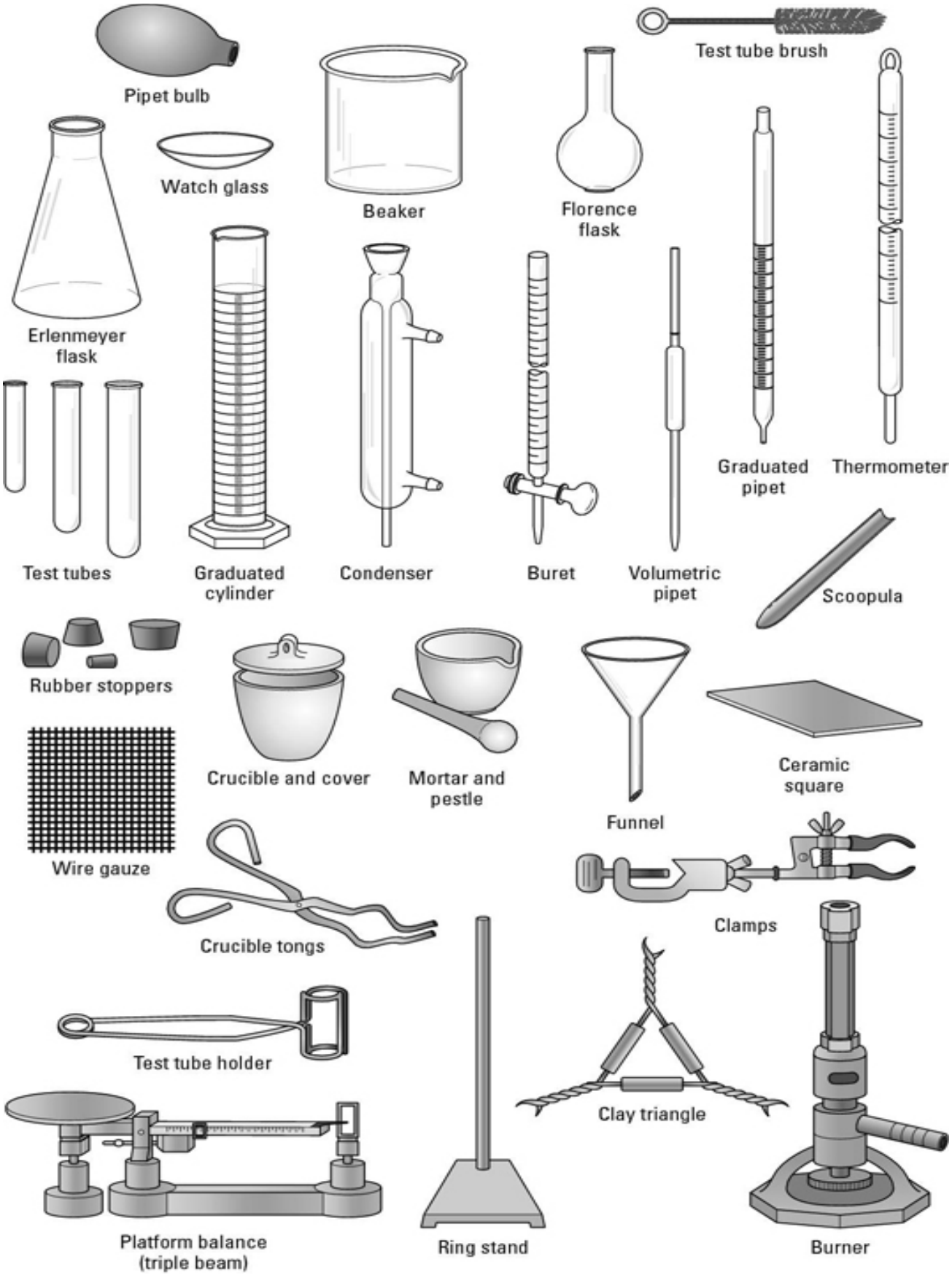
TESTING ACIDITY

Instructions sometimes require making a solution acidic or basic to litmus by adding acid or base. Always be sure that the solution is thoroughly mixed after adding the acid or base; then, by means of a clean stirring rod, remove a drop of the solution and apply it to litmus paper. Do not dip the litmus paper directly into the solution. Remember, just because you have added acid (or base) to a solution does not ensure that it is acidic (or alkaline).

HEATING SOLUTIONS IN SMALL TEST TUBES

The safest way to heat solutions in small test tubes is by means of a water bath. Two test tube holders wrapped around the test tube serves as a convenient handle for placing the tube into or removing it from the bath.

COMMON LABORATORY EQUIPMENT



Laboratory Equipment