EXPERIMENT 1

Basic Laboratory Techniques

OBJECTIVE

To learn the use of common, simple laboratory equipment.

APPARATUS AND CHEMICALS

balance	iron ring and ring stand
150-mL beaker	meterstick
250-mL beaker	10-mL pipet
Bunsen burner and hose	rubber bulb
clamp	thermometer
50-mL Erlenmeyer flask	wing tip
125-mL Erlenmeyer flask	ice
50- or 100-mL graduated cylinder	barometer

DISCUSSION

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

Because every measurement made in the laboratory is really an approximation, it is important that the numbers you record reflect the accuracy of the device you use to make the measurement. 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.

Recently, scientists have started to use a briefer version of the metric system of units in which the basic units for length, mass, and time are the meter, the kilogram, and the second. This system of units, known as the International System of Units, is commonly referred to as the SI system and is preferred in scientific work. A comparison of some common SI, metric, and English units is presented in Table 1.3.

Conversions within the metric system are quite easy if you remember the definitions for the prefixes and use dimensional analysis in problem solving.

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) = weight 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 H_2O 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 and g/L for gases: $d = \frac{\text{mass}}{\text{unit volume}}$
Specific gravity	sp gr, dimensionless: $\text{sp gr} = \frac{\text{density of a substance}}{\text{density of a reference substance}}$

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
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 $°C = \frac{5}{9}(°F - 32)$ $°F = \frac{5}{9}°C + 32$

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

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 different varieties of balances, with various limits on their accuracy. Three of these balances are pictured in Figures 1.1 and 1.2. Most modern laboratories possess single-pan balances with only two knife edges. These are the most accurate balances; generally, they are also the simplest to use and are the most delicate and expensive. A comparison of the basic operation of a single-pan balance and that of a double-pan balance is given in Figure 1.3. 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, since 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

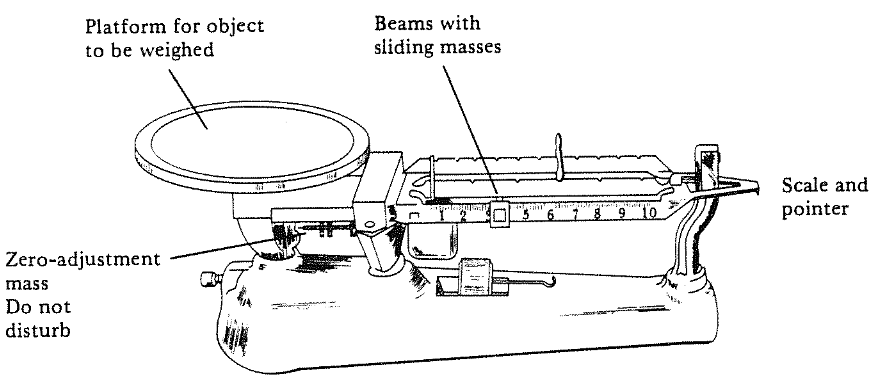
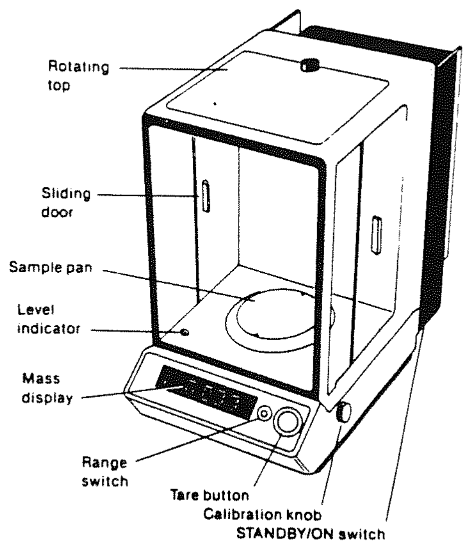


Figure 1.1 A laboratory platform balance (one knife edge) for crude weighing (0.1 g).



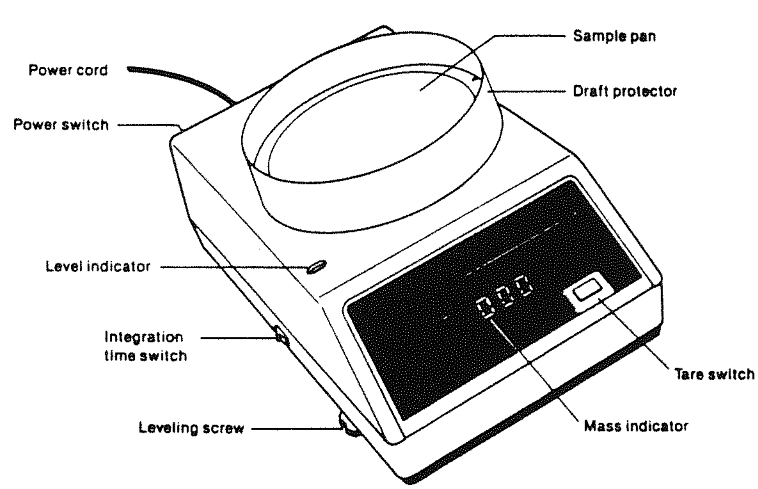


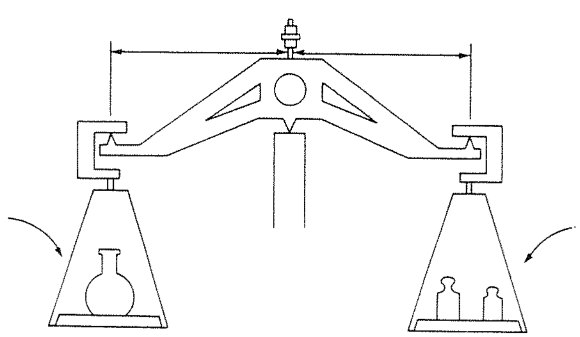
Figure 1.2 Digital electronic balances. The balance gives the mass directly when an object to be weighed is placed on the pan (0.0001 to 1 g depending upon the balance range).

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, 1 L = 1000 mL, and 1 L = 1.06 qt.

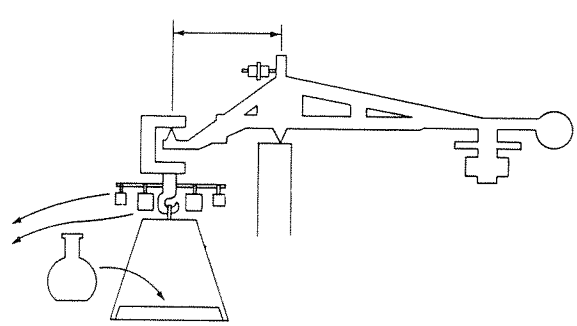
Thermometers

Most thermometers are based upon the principle that liquids expand when heated. Most common thermometers use mercury as the liquid. These thermometers are constructed so that a uniform-diameter capillary tube surmounts

Figure 1.3 A comparison of the essential features of the 2-pan and the 1-pan analytical balances. Substituting the flask for the two equivalent weights maintains a constant sensitivity on the 1-pan type, and also eliminates errors due to unequal lever arms.



Double arm 2-pan balance with 3 knife edges



Substitution 1-pan balance with only 2 knife edges a mercury reservoir. To calibrate a thermometer, one defines two reference points, normally the freezing point of water (0°C, 32°F) and the boiling point of water (100°C, 212°F) at 1 atm of pressure (1 atm = 760 mm Hg).* Once these points are marked on the capillary, its length is then 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 1.4 signify that this pipet was calibrated to deliver (TD) 10.0 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. This is illustrated in Table 1.5.

PROCEDURE

A. The Meterstick

Examine the meterstick and observe that one side is ruled in inches, while 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 Bunsen Burner and Operations with Glass Tubing

The Bunsen burner is a convenient source of heat in the laboratory. Although there are several varieties, their principle of operation is the same and is similar to that of the common gas stove. The Bunsen burner requires gas and air, which it mixes in various proportions. The amount of air and gas mixed in the chamber is varied by use of the two adjustments illustrated in Figure 1.5. The relative proportions of gas and air determine the temperature of the flame.

* 1 mm Hg is also called 1 torr.

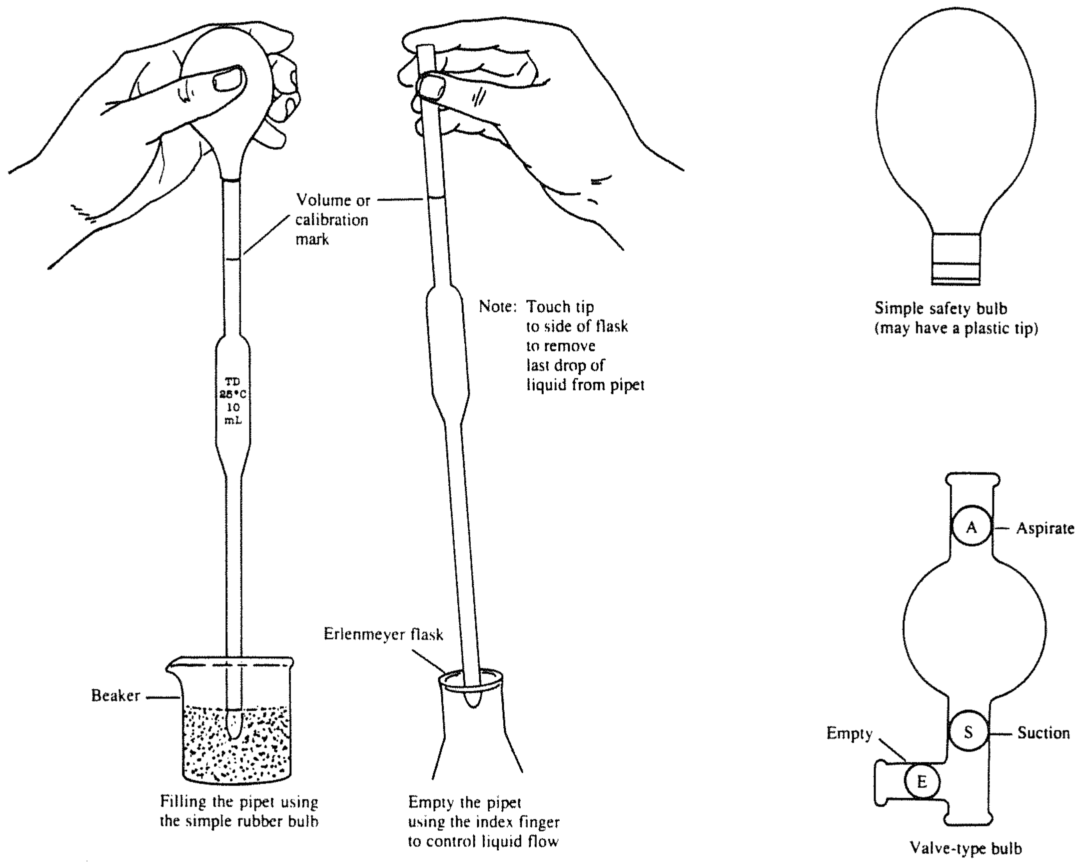


Figure 1.4 A typical volumetric pipet, rubber bulbs, and the pipet filling technique.

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	±0.2°C

Table 1.5 Obtaining Significant Figures

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

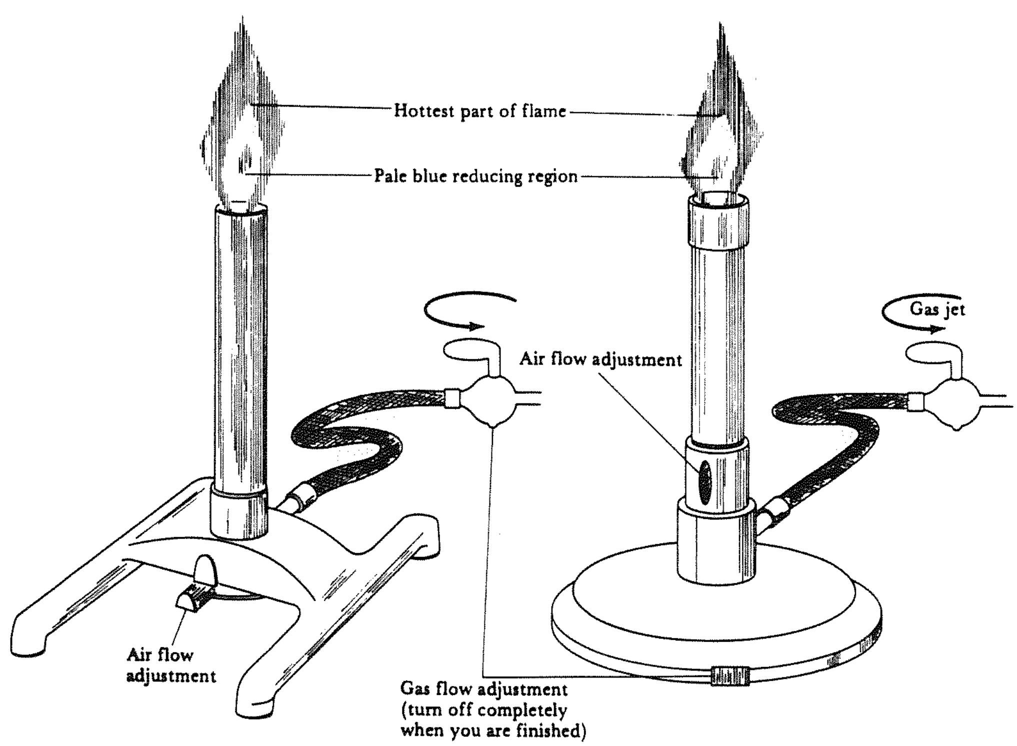
Examine your burner and locate the gas and air flow adjustments (valves) (see Figure 1.5). Determine how each valve operates before connecting the burner to the gas outlet. Close both valves; connect a rubber hose to the gas outlet on the burner and the desk; then open the desk valve about two-thirds of the way. Strike a match or use a gas lighter. Hold the lighted match to the side and just below the top of the barrel of the burner while gradually opening the gas valve on the burner to obtain a flame about 3 or 4 in. high. Gradually open and adjust the air valve until you obtain a pale blue flame with an inner cone as shown in Figure 1.5.

Glass is not a crystalline solid, but rather a supercooled liquid. Thus when it is heated it softens, flows, and can be worked. Crystalline solids melt rather than soften when heated.

Soda-lime glass (or soft glass) is made by heating a mixture of sodium carbonate, Na_2CO_3; calcium carbonate, $CaCO_3$; and silicon dioxide, SiO_2. It softens in the region of 300–400°C. It can easily be worked using a Bunsen burner, but because of its high temperature coefficient of expansion, it must be heated and cooled gradually to avoid undue strain or breakage. *Annealing* by a mild reheating and uniform, slow cooling is often wise. Such glass must not be laid on a cold surface while it is hot, because this introduces strains, and the glass will crack or even shatter.

Borosilicate glass (such as Pyrex or Kimax) does not soften much below 700–800°C and must be worked in an oxygen–natural gas flame or blowtorch.

Figure 1.5 Typical burners.



Because it has a low temperature coefficient of expansion, objects made from it can withstand sudden temperature changes. For this reason, among others, most of today's laboratory glassware is made from borosilicate glass. However, care must be exercised with it also. Uneven or rapid heating or cooling will cause the glass to shatter.

Study the illustrations in Figures 1.6 and 1.7 thoroughly and observe your instructor's demonstrations of how to work the glass. *Construct a right-angle bend and a 60° bend and two dropper tips for use in future experiments.* Use a wing tip on your Bunsen burner for the construction of the angle bends.

C. 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 (see Figure 1.8).

The *lowest* point on the curve is always read as the volume, never the upper level. Avoid errors due to parallax; different and erroneous readings are obtained if the eye is not perpendicular to the scale. Read the volume of water to the nearest 0.1 mL. Record this volume. Measure the maximum amount of water that your 125-mL Erlenmeyer flask will hold. Record this volume.

D. Relationship Between Temperature Scales

Both the Fahrenheit (°F) and Celsius (°C) temperature scales are linear scales that are based upon the physical properties of water. On the Celsius scale, water freezes at 0°C and boils at 100°C, whereas on the Fahrenheit scale, water freezes at 32°F and boils at 212°F. Using these two values, make a plot of degrees Fahrenheit versus degrees Celsius on the graph paper provided. Determine the Celsius equivalent of 40°F using your graph. The relationship between these two temperature scales is linear, that is, 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; then compute the Celsius equivalent of 40°F using this relationship.

E. 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 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 1.9. 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*

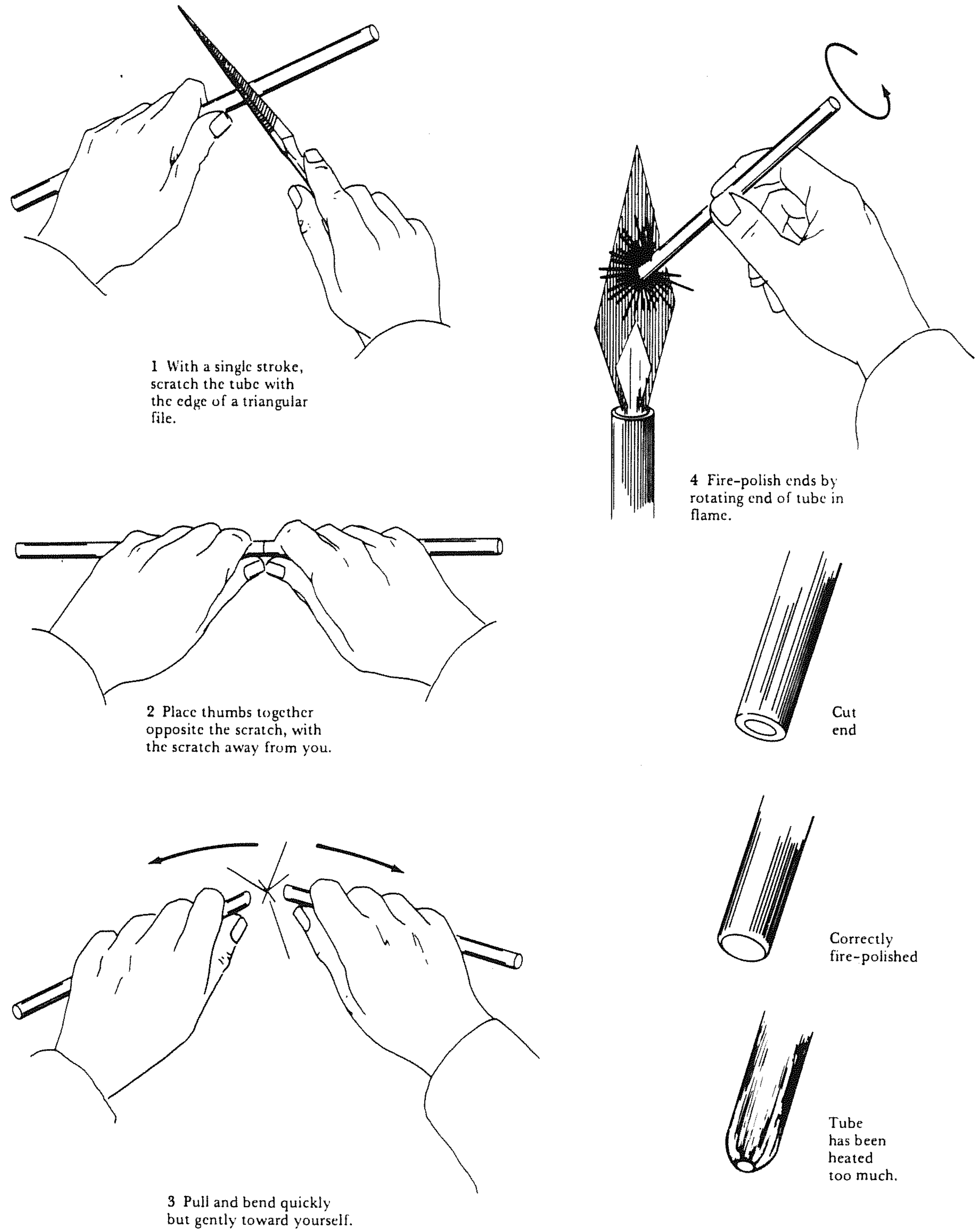


Figure 1.6 Cutting and fire-polishing a glass tube.

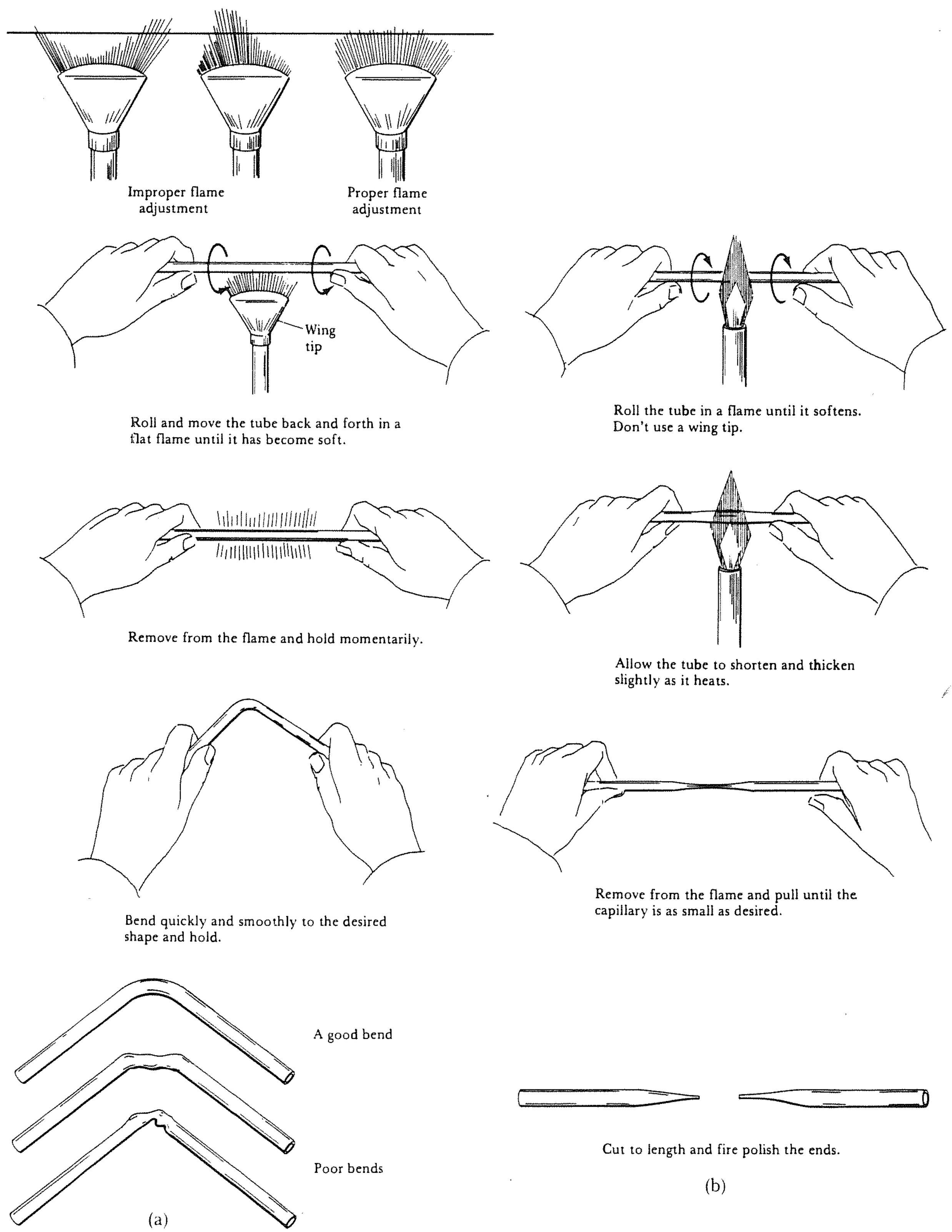


Figure 1.7 (a) Bending a glass tube and (b) drawing a capillary.

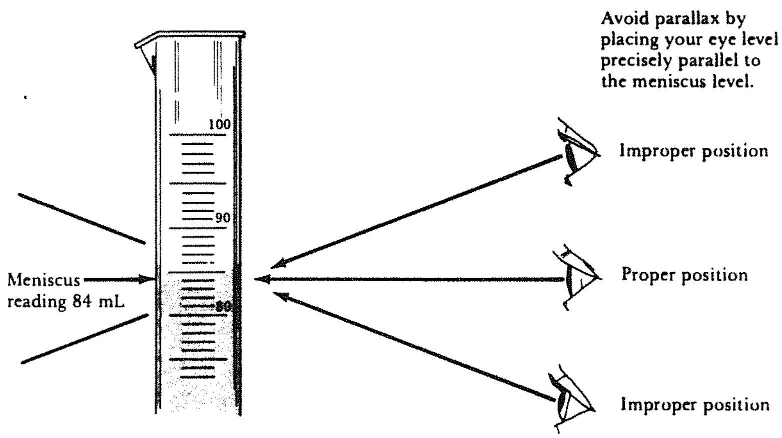


Figure 1.8 Proper eye position for taking volume readings.

atmospheric pressure. Obtain the atmospheric pressure from your laboratory instructor.

EXAMPLE 1.2

Determine the boiling point of water at 659.3 mm Hg.

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

$$\text{b.p. correction} = (760 \text{ mm Hg} - \text{atmospheric pressure}) \times (0.037°\text{C/mm})$$

The correction at 659.3 mm Hg is therefore

$$\text{b.p. correction} = (760 \text{ mm Hg} - 659.3 \text{ mm Hg}) \times (0.037°\text{C/mm}) = 3.7°\text{C}$$

The true boiling point is thus

$$100.0°\text{C} - 3.7°\text{C} = 96.3°\text{C}$$

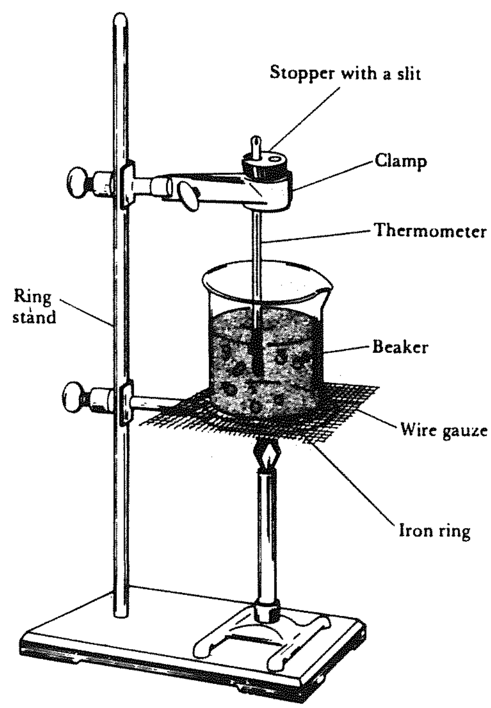


Figure 1.9 Apparatus setup for thermometer calibration.

Using the graph paper provided, construct a thermometer-calibration curve like the one shown in Figure 1.10 by plotting observed temperatures versus true temperatures for the boiling and freezing points of water.

F. 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.
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.

Weigh a penny and record its mass.

The following method is used to calibrate a pipet or other volumetric glassware. Obtain about 40 mL of distilled water in a 150-mL beaker. Allow the water to sit on the desk while you weigh and record the weight of an empty, dry 50-mL Erlenmeyer flask (tare) to the nearest 0.1 mg. Measure and record the temperature of the water. Using your pipet, pipet exactly 10 mL of water into this flask and weigh the flask with the water in it (gross) to the

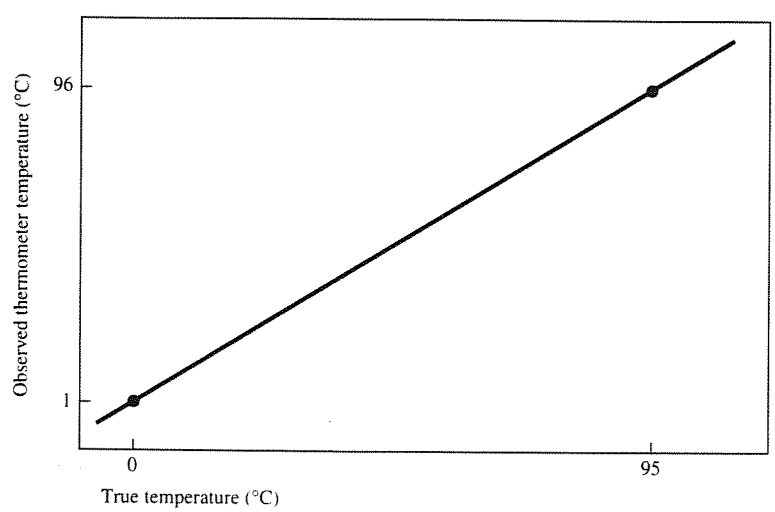


Figure 1.10 Typical thermometer-calibration curve.

nearest 0.1 mg. Obtain the weight of the water by subtraction (gross − tare = net). Using the equation below and the data given in Table 1.6, obtain the volume of water delivered and therefore the volume of your pipet.

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

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.

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

EXAMPLE 1.3

Using the procedure given above, a weight of 10.0025 g was obtained as the weight of the water delivered by one 10-mL pipet at 22°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 precision can be determined only to within ±0.02 mL.

The precision of a measurement is a statement about the internal agreement among repeated results; it is a measure of the reproducibility of a given set of results. The arithmetic mean (average) of the results is usually taken as the "best" value. The simplest measure of precision is the *average deviation from the mean*. The average deviation is calculated by first determining the mean of the measurements, then calculating the deviation of each individual measurement from the mean and, finally, averaging the deviations (treating each as a positive quantity). Study Example 1.4 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 values were obtained for the calibration of a 10-mL pipet: 10.10, 9.98, and 10.00 mL. Calculate the mean value and the average deviation from the mean.

Solution:

$$\text{Mean} = \frac{10.10 + 9.98 + 10.00}{3} = 10.03$$

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

$$|10.10 - 10.03| = 0.07$$
$$|9.98 - 10.03| = 0.05$$
$$|10.00 - 10.03| = 0.03$$

Average deviation from the mean

$$= \frac{0.07 + 0.05 + 0.03}{3} = 0.05$$

The reported value is therefore 10.03 ± 0.05 mL.

REVIEW QUESTIONS

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

1. What are the basic units of length, mass, volume, and temperature in the SI system?
2. A liquid has a volume of 1.25 liters. What is its volume in mL? in cm^3?
3. If an object weighs 1.52 g, what is its weight in mg?
4. Why should you never weigh a hot object?
5. Why is it necessary to calibrate a thermometer and volumetric glassware?
6. What is precision?
7. What is the definition of density? Can it be determined from a single measurement?
8. What is the density of an object with a mass of 9.67 g and a volume of 0.2236 mL?
9. Weighing an object three times gave the following results: 10.4 g, 10.1 g, and 10.2 g. Find the mean weight and the average deviation from the mean.

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

11. What is the weight in kilograms of 1000 mL of a substance that has a density of 1.349 g/mL?

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