Experiment 1

Basic Laboratory Techniques

OBJECTIVE

To learn the use of common, simple laboratory equipment.

APPARATUS AND CHEMICALS

Apparatus

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

Chemicals

ice
antifreeze (ethylene glycol)

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 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 $d = \frac{\text{mass}}{\text{unit volume}}$
Specific gravity	sp gr, dimensionless: $\text{sp gr} = \frac{\text{density of a substance}}{\text{density of a reference substance}}$

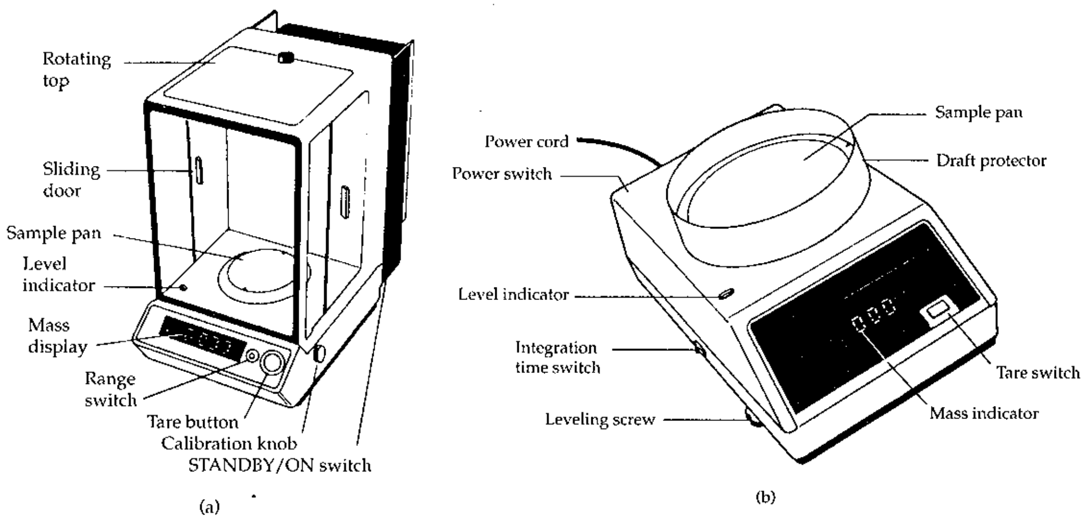
TABLE 1.2 The Meaning of Prefixes in the Metric System

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 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:

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

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, 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 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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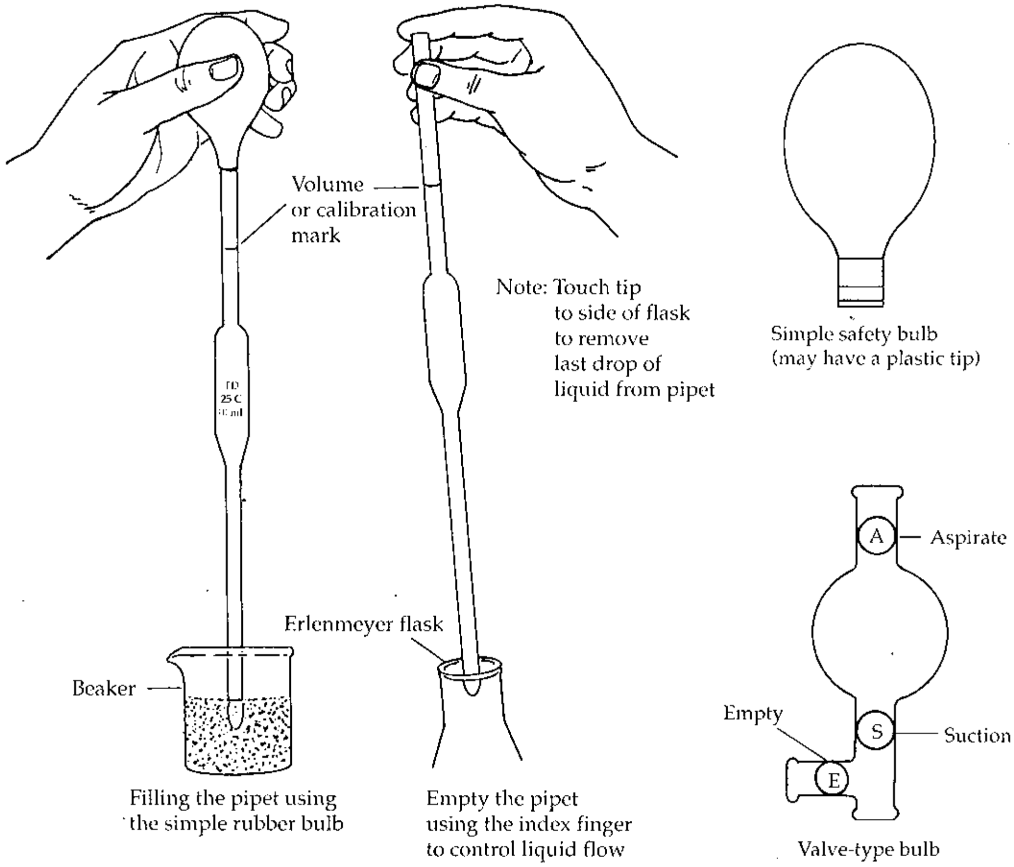
*1 mm Hg is also called 1 torr.



▲ **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	±0.2°C

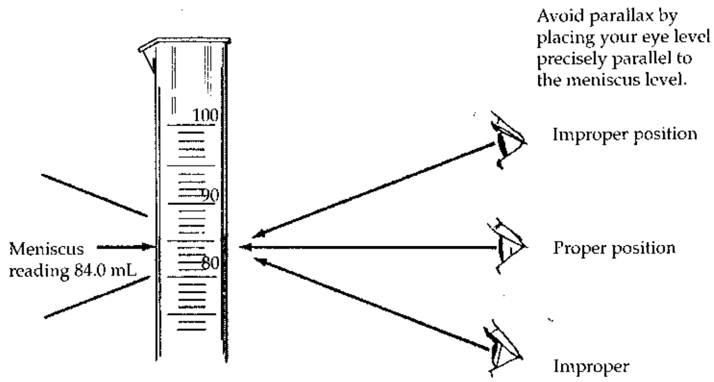
TABLE 1.5 Significant Figures Used In Recording Mass

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.

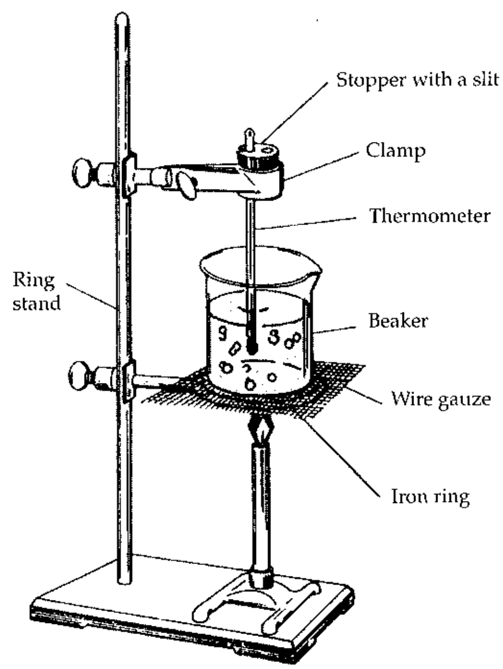
C. The Thermometer and Its Calibration

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 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

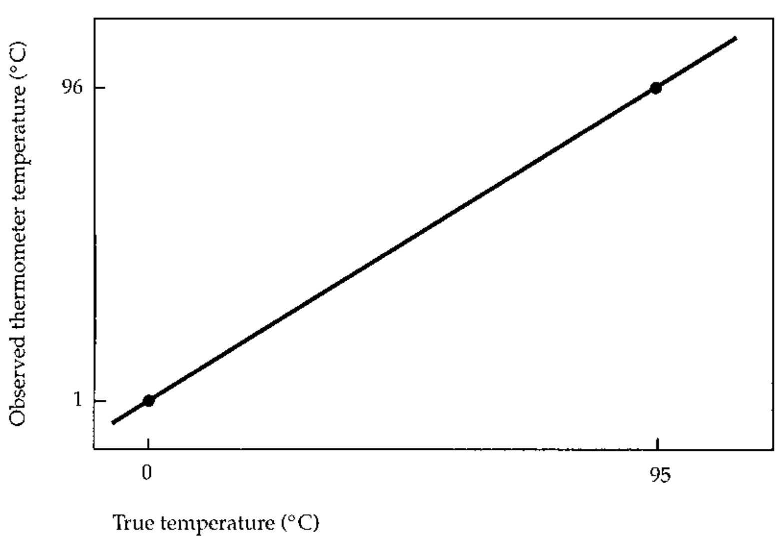
$$100.0°\text{C} - 3.7°\text{C} = 96.3°\text{C}$$

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: 10.10, 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.

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.

PRE LAB 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. What is the number of significant figures in each of the following measured quantaties? (a) 2578 g; (b) 0.010 mL; (c) 1.010 mL; (d) 3.72×10^{-3} cm.
3. What is the length of a crystal of copper sulfate in centimeters that is 0.125 inches long?

4. 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?

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

6. If an object weighs 0.092 g, what is its mass in mg?

7. Why should you never weigh a hot object?

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

9. What is precision?

10. Define the term *density*. Can it be determined from a single measurement?

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 ______________________

REPORT SHEET | EXPERIMENT

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

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_2O (gross mass)	______	______	______	g
Mass of Erlenmeyer (tare mass)	______	______	______	g
Mass of ~10 mL of H_2O (net mass)	______	______	______	g

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

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. What is the number of significant figures in each of the following measured quantities? (a) 2513 kg; (b) 0.0034 g; (c) 5.060 mL; (d) 0.01060 cm; (e) 1.245×10^{-6} L.

2. Carry out the following operations, and express the answer with the appropriate number of significant figures and units. (a) (5.231 mm)(7.1 mm); (b) 72.3 g/1.2 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 10 in. tall weighs 170 lb. What is his height in centimeters and his mass in kilograms?

5. Determine the boiling point of water at 698.5 mm Hg.

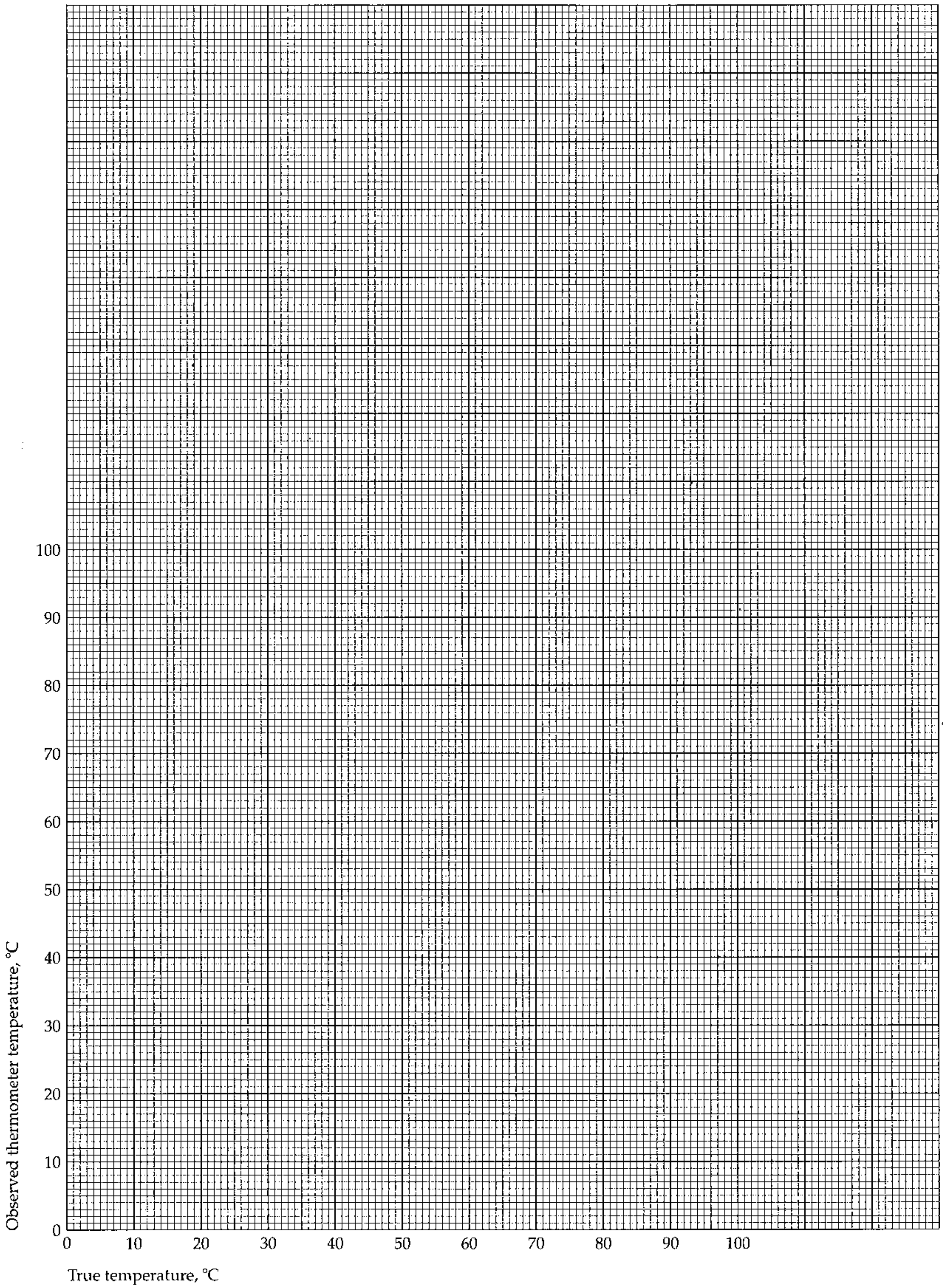
6. A pipet delivers 9.98 g of water at 20°C. What volume does the pipet deliver?

7. A pipet delivers 9.04, 9.02, 9.08, and 9.06 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



Fahrenheit—Celsius Graph