Cégep de la Gaspésie et des Îles
CAMPUS DE GASPÉ

EXPERIMENT 1
BASIC LABORATORY TECHNIQUES

INTRODUCTION, SAFETY, EXPERIMENTAL, STANDARD DEVIATION, PERCENTAGE ERROR & PRELAB QUESTIONS

General Chemistry 202-NYA-05

Introduction

Chemistry is an experimental science. It depends upon careful observation and the use of good laboratory techniques. In this experiment, you will become familiar with some basic operations that will help you throughout this course. Thus, the objective of this laboratory experiment is to learn to use common and simple laboratory equipment. 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. 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.

Table 1.1 Units of Measurement in the Metric System

Measurement	Unit, Definition, Conversion Factors Between Metric and English Units
Mass and 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 1 kilogram (kg) = 10^3 g = 2.205 lb 1 lb = 453.6 g 1 milligram (mg) = 0.001 g
Length	Meter (m) = 100 cm = 1000 millimeters (mm) = 39.37 in. 1 in. = 2.54 cm
Volume	Liter (L) = volume of 1 kg of water at 4°C 1 L = 10^3 cm^3 = 10^{-3} m^3 = 1.06 qt 1 milliliter (mL) = 1 cm^3 = 0.001 L
Temperature	Kelvin (K); 0K = –273.15°C °C = 5/9 (°F – 32) °F = 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* = mass / volume

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.

Table 1.2 The Meaning of Prefixes in the Metric System

Factor	prefix	Symbol
10^{9}	giga	G
10^{6}	mega	M
10^{3}	kilo	k
10^{2}	hecto	h
10^{1}	deka	da
10^{-1}	deci	d
10^{-2}	centi	c
10^{-3}	milli	m
10^{-6}	micro	μ
10^{-9}	nano	n
10^{-12}	pico	p
10^{-15}	femto	f
10^{-18}	atto	a

In 1960, an 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, respectively. 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 metric and English units is presented in Table 1.1.

In Table 1.2, the prefix means the power of 10. For example, 5.4 centimeters means 5.4×10^{-2} meters; *centi-* has the same meaning as $\times 10^{-2}$.

Example 1.1

Convert 6.7 nanograms to milligrams using dimensional analysis

Solution:

(6.7 ng) (10^{-9}g / 1 ng) (1 mg / 10^{-3} g) = 6.7×10^{-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 digital electronic balances are the *analytical balance* and the *top-loading balance*. These single-pan balances are found in most modern laboratories, and will be routinely used in your laboratory experiments. 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 (Figure 1.1). 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 such 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 = 1 torr). 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 (Figure 1.1). The markings on the top area of the pipet signify that the pipet was calibrated to deliver (TD) a specific amount 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 device. The approximate accuracy of some of the equipment you will use in this course is given in Table 1.3.

Table 1.3 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)
Buret	±0.02 mL

Not only should you obtain a measurement to the highest degree of accuracy that the device or instrument permits, but you should also record that reading or measurement in a manner that reflects the accuracy of the instrument. For example, a mass obtained from an analytical balance should be observed and recorded to the nearest 0.0001 g. This is illustrated in Table 1.4.

Table 1.4 Obtaining Significant Figures	
Analytical Balance	**Top-loading Balance**
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)	

Safety

Carefully examine all glassware for cracks or chips. Ask for a replacement if you find any. Handle thermometers with extreme care. They contain mercury!!!

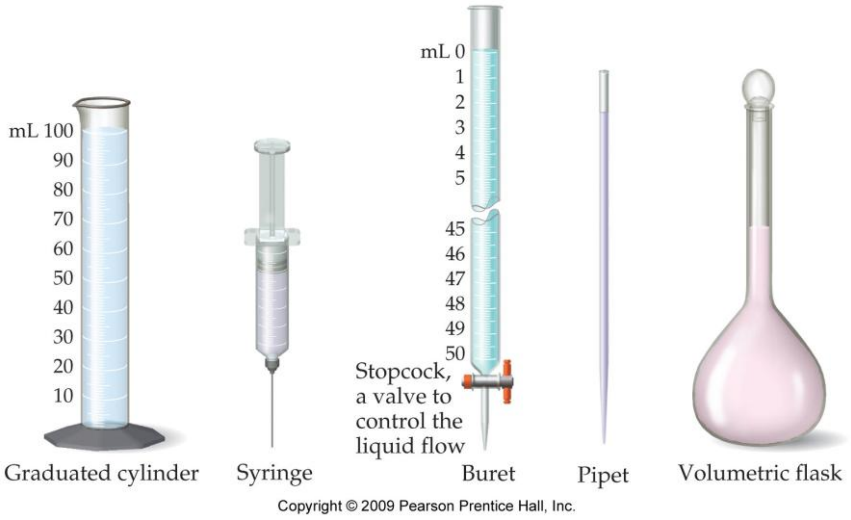


Figure 1.1. Common volumetric glassware. The graduated cylinder, syringe, and burette are used in laboratories to deliver variable volumes of liquid; the pipet is used to deliver a specific volume of liquid. The volumetric flask contains a specific volume of liquid when filled to the mark.

Experimental

Part 1: The meter stick

Procedure

Examine the meter stick 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 centimeters. Record the measurements. Convert these measurements into meters and record the results.

Part 2: The graduated cylinder

Procedure

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 1.2).

In Figure 1.2, 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 the volume.

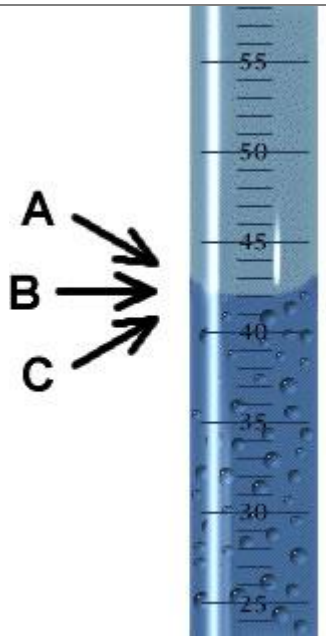


Figure 1.2. Proper eye position for taking volume readings from a graduated cylinder. A and C are improper eye positions. B is the correct eye position. Meniscus reading is 42.0 mL.

(Image taken from: Mr. Murray's science (Internet). Grapevine (TX): C. Stephen Murray; c2003-2009. [Image], Reading the meniscus; [cited 2009 Aug 17]. Available from: http://www.cstephenmurray.com/onlinequizes/chemistry/measuring/readingthemeniscus.htm)

Part 3: The thermometer and its calibration

This part of the experiment is performed to check the accuracy of your thermometer, and to calculate the corrected boiling point of water from the experimental value (also called observed value) obtained in the laboratory.

These measurements will show how experimental temperatures (read from thermometers in the laboratory) compare with normal or true temperatures: the ***normal (true) freezing point*** and ***normal***

(true) boiling point of water are the boiling point and the freezing point at standard atmospheric pressure (760.0 mmHg).

The normal freezing point of water is 0°C at 760.0 mmHg. The normal boiling point is the boiling point of water is 100°C at 760.0 mmHg. However, the boiling point depends upon atmospheric pressure. So, if you are measuring the boiling point of water in the laboratory on a day when the atmospheric pressure differs from the standard value of 760.0 mmHg, you must correct your experimental value in order to be able to compare the boiling point you obtained by experiment (experimental or observed) with the normal boiling point of water (100°C at 760.0 mmHg). Example 1.2 below shows you how to calculate the ***temperature correction*** for a boiling point obtained at an atmospheric pressure other than the standard value of 760.0 mmHg, and how to calculate the ***corrected boiling point*** (the boiling point that has been corrected to standard atmospheric pressure).

Example 1.2

Determine the temperature correction and the corrected boiling point of water, if the experimental boiling point at 730.3 mmHg is 98.2°C.

Solution:

Boiling point temperature corrections are calculated using the following formula:

bp temperature correction = (760.0 mmHg – atmospheric pressure in the lab) × (0.037°C / mmHg)

The bp temperature correction at 730.3 mmHg is therefore:
bp correction = (760.0 mm Hg – 730.3 mmHg) × (0.037°C / mmHg) = 1.0989°C

The corrected boiling point of water at 760 mmHg is thus:
98.2 + 1.0989 = 99.2989°C = 99.3 °C (1 place after the decimal; correct number of significant figures = 3)

Procedure

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 hotplate. Fill the beaker about half full with distilled water, add a stirring bar, adjust the stirring speed, and begin heating the water. Secure a thermometer with a clamp so that its tip is immersed in the water, but not touching the stirring bar. (*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 experimental boiling point (bp) of the water.

Following Example 1.2, determine the temperature correction and the corrected boiling point. Obtain the atmospheric pressure from your laboratory instructor.

Whether it be for the melting or boiling point, the difference between the experimental and the theoretical values provides the ***temperature adjustment*** that must be applied to future temperature readings.

Calculate the percentage error, which is a measure of the accuracy of your results.

Prelab questions (answer these questions in your prelab)

Answer the following questions:

1. What are the basic units of length, mass, volume, and temperature in the SI system?
2. A liquid has a volume of 1.35 liters. What is it volume in mL? In cm^3?
3. If an object weighs 1.47 g, what is its mass in mg?

Errors

Please refer to *Chemistry Guide* for a description of what should be included in the *Errors* section.

Standard deviation – A measure of precision

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. In other words, it gives an idea of how closely individual measurements agree with one another. 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. However, a better measure of the precision of your results is calculated using *standard deviation*. This value gives one a feel for the scatter about the mean, or the deviations from the mean. The smaller the deviations from the mean, the more precise the measurements.

To calculate standard deviation (*s*), the arithmetic mean or average ($\bar{x}$; "x-bar") of the results (a set of data) is obtained using the formula:

$$\bar{x} = \frac{\sum x_i}{n}$$

The deviations of each individual measurement from the mean is then calculated using:

Deviation from the mean = value – mean = x_i **-** $\bar{x}$

Standard deviation (*s*) is calculated using the formula:

***s* = [sum of the squares of deviations from the mean /(number of observations – 1)]^{½}**

$$s = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n-1}}$$

where x_i = an individual measurement of the set of data, $\bar{x}$ = the mean or average of the set of data, and n = number of measurements in the set of data. The symbol $\sum_i$ means to sum all of the individual measurements of the set of data.

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 standard deviation.

Solution:

Mean = $\bar{x}$ = (10.10 + 9.98 + 10.00) / 3 = 10.03 mL

Deviations from the mean = x_i - $\bar{x}$

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

Standard deviation:

$$s = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n-1}}$$

$\{[(0.07)^2 + (-0.05)^2 + (-0.03)^2] / 3\text{-}1\}^{½}$
$[0.0083 / 2]^{½} = 0.0644205$

The volume of the pipet is then reported as: 10.03 ± 0.064 mL (standard deviation).

Note that the standard error is always written to one more decimal place than the volume mean value.

The relative standard deviation is then:

$(s / \bar{x}) * 100 = 0.064 / 10.03 = 0.0064 = 0.64$ %

The volume of the pipet is then reported as: 10.03 mL ± 0.64% (relative deviation).

Using your own experimental results, calculate the mean volume delivered by your 10-mL pipet. Also, for your three trials, calculate the individual deviations from the mean and then the standard deviation. State your mean pipet volume with its standard deviation and with its relative deviation.

Percentage error – A measure of accuracy

Error is the difference between the experimental (synonyms include: measured, observed value and the true (synonyms include: literature, accepted, theoretical, reported value) of the thing being measured.

To determine the accuracy of your results calculate percent (%) error.

$$\% \text{ error} = [(\text{experimental value} - \text{theoretical value}) / \text{theoretical value}] * 100$$

Note that a negative % error indicates that your experimental value is lower than the true value. Similarly, a positive % error indicates that your experimental value is higher than the true value.

Calculate the % error of the freezing and boiling points of water.