USE OF LABORATORY EQUIPEMENT

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.