EXPERIMENT 1

Basic Laboratory Techniques

Objectives:

1. To light and properly adjust a Bunsen burner.
2. To develop skills for properly operating a laboratory balance.
3. To develop skills for measuring volumes of liquids and solids.
4. To determine the density of a metal and an unknown liquid.

Introduction:

All chemical principles, tools, and techniques are developed in the laboratory .The experience of observing a chemical phenomenon and then rationalizing its behavior is one that simply cannot be gained by reading a textbook or by listening to a lecturer. ***It is here, in the laboratory, that chemistry comes alive, and it is here that chemical principles can be learned and applied to the vast natural "chemistry laboratory" of our everyday lives.*** The objectives of laboratory work are to design and build apparatus, develop techniques, record data and deduce rational theories so that the real world of science can be better understood.

In this laboratory you use common equipment in designing experiments that provide data. You record the observed data and try to rationalize or theorize "why" such data are being observed based upon sound chemical principles. A good scientist is a thinking scientist, try to account for contradictory data, cultivate self-reliance and confidence in your work even if "it doesn't look right"-this is how breakthroughs in science occur.

In the first few laboratory periods you will encounter some basic rules, equipment and techniques and some situations where you use them. These include laboratory safety rules, the Bunsen burner and the top-load balance. Additional laboratory techniques are gained in techniques of volume measurement of solids and liquids and in separation techniques such as filtration and decantation.

A. THE BUNSEN BURNER

Laboratory burners come in many shapes and sizes, but all accomplish one main purpose: producing a combustible gas-air mixture that yields a hot, efficient flame. Because Robert Bunsen (1811- 1899) was the first to perfect and use this burner, his name commonly given to all simpler burners of this type.

The natural gas used in most laboratories is composed primarily of the hydrocarbon methane, CH_4. If sufficient oxygen is supplied, methane burns with a blue, nonluminous flame producing carbon dioxide and water. With insufficient oxygen, small carbon particles are produced which, heated to incandescence, produce a yellow, luminous flame.

- ***Lighting the burner***.

Attach the burner's tubing to the gas outlet on the lab bench. Turn off the burner's gas control and fully turn on the gas valve at the outlet. Close the air holes at the base of the burner and open the gas control slightly. Bring a lighted match or striker up the outside of the burner tube until the escaping gas at the top ignites. After it ignites, adjust the gas control until the flame is pale blue and has two or more distinct cones. Opening of the air control valve produces a slight buzzing sound indicating hottest flame, the addition of air may blow the flame out. ***The best adjustment is when you see 3 distinct cones.***

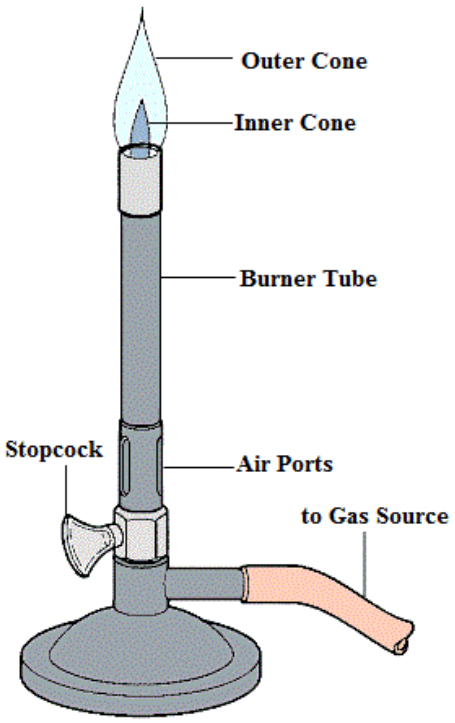

Figure 1: Bunsen burner

B.THE LABORATORY BALANCE

Several types of balances may be found in the general chemistry laboratory. The triple beam and the top-loading balances are the most common. As suggested on the Report Sheet, weigh several objects. Use the top-loading balance only after the instructor explains its operation. Be sure to record the weight of the objects to the precision that the balance allows: ± 0.01 g for both the triple beam balances and the top-loading balances.

Figure 2: Top-load balance

C. VOLUMETRIC GLASSWARE

Graduated cylinders, transfer pipets, Mohr pipets, and burets are used to make volumetric measurements. This glassware must be scrupulously clean. Liquids must drain without leaving drops adhering to the inner walls of the glassware.

- ***Graduated Cylinders***

A graduated cylinder is used to measure an approximate volume of a liquid. When water or an aqueous solution (a solution containing water) is added, the upper surface of the liquid in the graduated cylinder will be concave. This concave surface is called a ***meniscus***. **<u>The bottom of the meniscus is used for all measurements</u>**. To avoid error, your eye should always be level with the meniscus when you are measuring the volume.

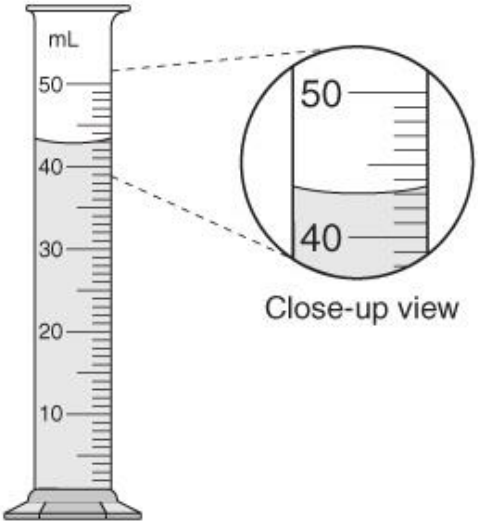

Figure 3: Graduated Cylinder

Graduated cylinders come in many sizes, but **10**-mL, **25**-mL, **50**-mL, and **100**-mL graduated cylinders ate often found in general chemistry laboratories.

- ***Pipets***

Transfer and Mohr pipets are required by some of the experiments in this laboratory manual. A transfer pipet is calibrated to deliver ***(TD) one and only one volume***, whereas a ***Mohr pipet is***

graduated so that it can deliver any volume (usually to the nearest tenth of a milliliter) up to its maximum volume.

Transfer pipets come in many sizes, but **5**-mL, **10**-mL,**20**- mL and **25**-mL pipets are usually found in general chemistry laboratories. Mohr pipets are commonly restricted to **5**-mL,**10**-mL and **25**-mL volumes.

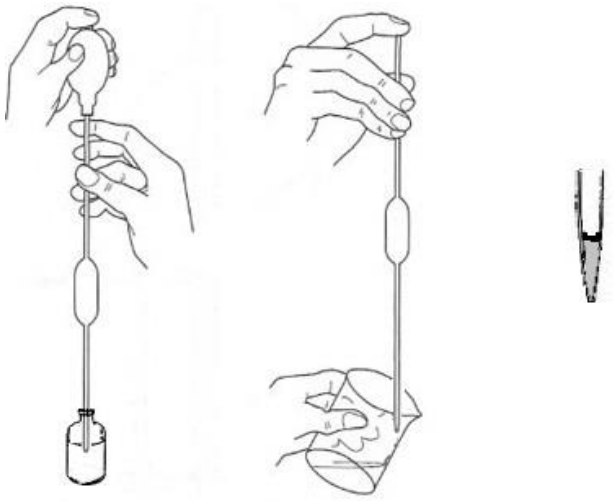

Figure 4: Proper Pipetting Technique

The correct use of a pipet requires considerable manipulator skill. This is not an innate skill, but one that will come only with practice. ***Remember that you are not allowed to use your mouth for suction even if you are filling the pipet with water!***

- ***Burets***

The principal use of the buret is for titrations. ***Precise titrations require burets that drain freely, are very clean, and do not leak around the stopcock***. The following three steps will help you to have a buret that operates as it should.

1. The capillary tip of the buret should be dean and free of foreign objects. A thin wire can sometimes be used successfully to dislodge grease or dirt that prevents draining.
2. If water droplets are left on the inner walls of the buret after draining, the buret needs a thorough cleaning. It should be cleaned with hot water, detergent, and a buret brush, then it should be rinsed with tap water. Finally, it should be rinsed with deionized water.

3. Some maintenance is required if the stopcock leaks while the buret is draining or if drops form on the capillary tip when the stopcock is turned off. Glass stopcocks must be lubricated to prevent both kinds of leaking. Lubrication will also allow the stopcock to turn easily.

The clean, properly operating buret should be held in place by a clamp, preferably a buret damp, which is attached to a ring stand. Before you fill the buret, you should rinse it several times with the solution that will eventually be in it. (Demonstration by the instructor).

The buret will show the volume of a liquid that has been delivered rather than the volume that remains. That's why the data should show the initial and final reading of the buret. The spacing between these lines will allow you to estimate the volume to the nearest 0.01 mL. Thus, typical buret readings would be 9.34 mL or 17.60 mL. Readings such as 9.3 mL or 17.6 mL are not acceptable.

D. FILTRATION

Filtration through special paper (called filter paper) is the simplest method of separating a solid from a liquid. Filter paper is available with a variety of porosities. A finely porous paper should be used for solids with very small particles, but filtration will be slow. A coarser, more porous paper can be used with solids whose particles are larger. Filtration will then be more rapid.

- ***Gravity Filtration***

This technique requires a **conical filter funnel** with a hollow stem and a **glass stirring rod**. For this method of filtration, the filter paper is folded as paper cone, placed in the filter funnel. Distilled water is added to wet the paper thoroughly. Pour off the excess water and place the funnel in its support. The support can be an iron ring, a clay triangle on an iron ring, a wooden board with a circular hole, or the mouth of an Erlenmeyer flask. If you are filtering into a beaker, you can minimize splashing by putting the stem of the filter funnel against the inner wall of the beaker.

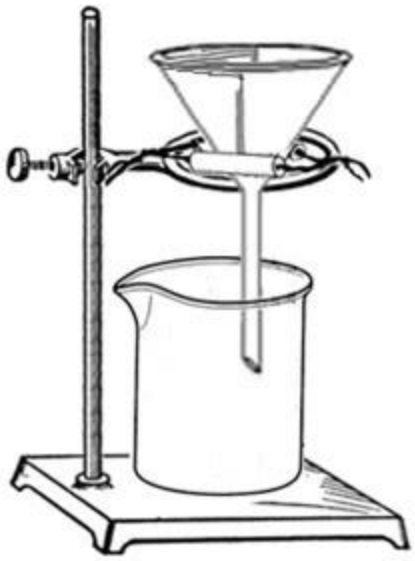

Figure 5: Gravity filtration

The mixture to be filtered should be poured ***(decanted***) along the stirring rod to direct the flow of the liquid into the filter paper. Never fill the filter paper to more than two-thirds of its volume. Most of the solid should be transferred to the filter during this stage. If you have a rubber policeman, use it to remove a solid that adheres stubbornly to the walls of the container. When all of the liquid has been transferred to the filter, use a stream of distilled water from a plastic wash bottle to rinse the remainder of the solid into the paper. When the entire solid has been transferred, rinse the stirring rod in such a way that the distilled water is also directed onto the paper. Finally, wash the solid with two small portions of distilled water. ***A quantitative transfer of the solid will now have occurred***.

- ***Suction Filtration***

This type of filtration is much faster than gravity filtration, ***but quantitative recovery of a solid is rarely achieved***.

Suction filtration requires a **Buchner funnel**, a **suction flask**, a rubber stopper or **rubber ring** to hold the funnel tightly in the flask, a **glass stirring rod**, **heavy rubber tubing**, and a **water aspirator**. In addition, some type of safety trap is advised to prevent water from the aspirator from backing up into the suction flask.

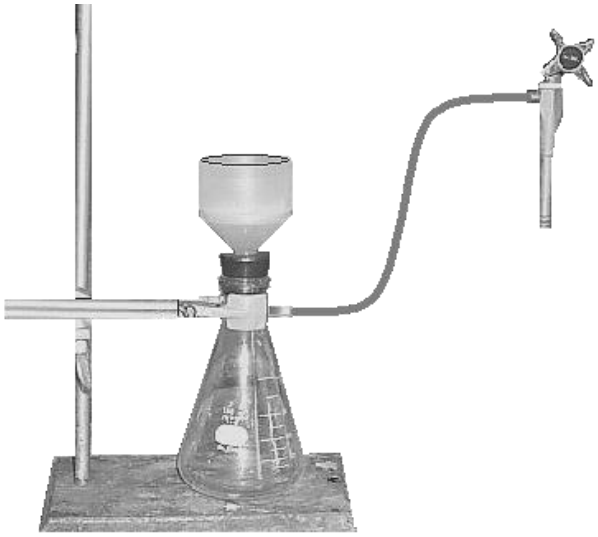

Figure 6: Suction filtration

E. DENSITY

Each pure substance exhibits its own set of characteristic properties. One is ***density: the mass of a substance per unit volume.*** We say that the density of water is 1.0 g/mL or 1.0 g/cm^3.By measuring an object's mass and volume; we can determine its density. In this experiment ,we will determine the density of a water-insoluble metal by its displacement of water and the density of an unknown liquid.

Procedure:

1.Solid

1. Measure the mass using the balance assigned to you, as accurately as possible.
2. Half-fill the 50-mL graduated cylinder with water and record its volume to the nearest 0.2 mL.
3. Add the weighed unknown to it and roll the solid around in the cylinder, removing any air bubbles that are trapped or that adhere to it.
4. Record the new water level. The solid's volume is the difference between the two water levels.

2.Liquid

1. Clean and dry a 100-mL beaker.
2. Using the top loading balance, precisely measure and record its mass.
3. Pipet 10 mL of water to the beaker.
4. Reweigh the beaker and contents.
5. Dry the beaker and the pipet.
6. Ask the instructor for an unknown liquid and repeat the measurement substituting the unknown for the water.

Report Sheet

Density of Solids and Liquids

Student Name_____

Student No._____

Density

1. Solid Unknown

a. Mass of solid (g) _____
b. Volume of water (mL) _____
c. Volume of water and solid (mL) _____
d. Volume of solid (mL) _____
e. Density of solid (g/ mL) _____

2. Liquid

*Wate*r

a. Mass of beaker (g) _____
b. Mass of beaker and water (g) _____
c. Mass of water (g) _____
d. Volume of water (ml) _____
e. Density of water (g/mL) _____

Unknown liquid

a. Mass of beaker (g) _____
b. Mass of beaker and unknown liquid (g) _____
c. Mass of unknown liquid (g) _____
d. Volume of unknown liquid (ml) _____
e. Density of unknown liquid (g/mL) _____

Post Laboratory Questions

1) Name two checks you should make before using a balance.

2) Two general chemistry students determined the density of lead following the procedure given in this experiment. Give one reasons why their experimental values may differ.

3) If an air bubble adheres to the metal's surface when submerged in water, how does this affect the metal's experimental density? Explain.

4) If several drops of unknown liquid cling to the pipet's inner walls (due to a dirty pipet), will its reported density be higher or lower than its actual density? Explain.