Basic Laboratory Techniques

Measuring Volume

1. **Medicine Droppers**.
   A satisfactory, but often rough, method of estimating volumes, the dropper is calibrated by counting the number of drops it produces to make up a milliliter.
2. **Beakers and Flasks**.
   The volume on the side of the beaker or flask is only a rough approximation at best. Use this method only for crude estimates of volume.
3. **Volumetric Glassware**.
   This is the most precise and accurate method of transferring and delivering liquids. It is important that the volumetric glassware be extremely clean before use, as dirt and other chemicals will not only reduce precision due to improper draining but also can contaminate the experiment. The best time to clean glassware is immediately after its use. Special cleaning solutions are available, but soap, warm water, and a brush, followed by thorough rinsing-first with tap water and then with small amounts of distilled water-is often satisfactory. No drops of distilled water should adhere to the surface of clean glassware. To dry, the glassware is inverted onto a paper towel. Do not wipe or air-blow dry due to possible contamination.
   In determining the volume of all volumetric glassware, it is important to understand how to read the *meniscus*. The meniscus is the apparent downward curvature of the liquid mainly due to surface tension. It is necessary to read the bottom of the meniscus with the eye horizontal to this surface. If it is not read at eye level, error in the reading will result. Proper lighting is important to see the meniscus clearly. To enhance the meniscus, a small white card with a 1 x 1.5Ó black rectangle in the lower one-third of the card is placed behind the glassware. The card is slowly raised until the reflection of the black rectangle on the meniscus is seen. The bottom of the meniscus is now readily visible against the white background.

   a. *Graduated cylinders* are the most commonplace measuring instruments in the laboratory. A tall cylinder with a small diameter will be more accurate than a short one with a large diameter.
   b. *Burets* are constructed so that it is possible to measure and deliver accurate volumes. To clean a buret, clean with a soap solution. If a buret brush is used be sure not to scrape the sides of the buret wall with the metal handle. Rinse the soap solution from the buret several times through the stopcock, first with tap water and then with several small portions of distilled water. Be sure to roll the buret in a near horizontal position to thoroughly wet the entire surface of the glass.
   To *activate* the buret, close the stopcock and add a small portion of titrant (3-5 mL). Tilt the buret to a nearly horizontal position and roll the buret so that the rinse comes in contact with the entire inner surface. Drain this material through the buret tip into a waste beaker. Repeat this procedure three times.
   To fill and operate a buret, close the stopcock and, with a funnel, fill the buret to just above the zero mark. Be sure the buret is vertical and does not slant. Open the stopcock briefly to remove any air bubbles and

drain the titrant to some point below the zero mark. Record the starting volume and perform the titration. Be sure to record the final volume. When the titration is finished drain the excess titrant and rinse the buret with a small amount of distilled water two to three times. Clean it if necessary.

c. *Volumetric pipets*, properly manipulated, can deliver volumes reliable to one part per thousand. *Graduated pipets* are not capable of such precision. **In pipetting a liquid, oral suction must never be used.** Keeping the pipet tip under the surface of the liquid and, using a pipet bulb, draw the liquid above the graduation mark. Slowly squeeze the release button to level off the amount. Any drops adhering to the bottom of the tip should be removed before delivering the volume. Hold the pipet vertically and let the liquid drain for 20 seconds after the liquid has been delivered; the tip is then touched to the wall of the receiver. The liquid in the tip of the pipet must not be removed; calibration of the pipet has allowed for this.

d. *Volumetric flasks* are used to prepare solutions of a specified concentration. In making a solution the solute is placed in the flask first. Then a small amount of the solvent is added and the flask is vigorously shaken to dissolve or mix the solutions. Care should be taken as the flask and solution may get very warm. **Never** use your fingers to cover the opening at the top. There will be an appropriate sized stopper to use. Add more solvent to just below the fudicial mark, stopper, and shake again. Let the flask come to room temperature and then fill the flask to the fudicial mark and stopper. Invert the stoppered flask several times to thoroughly mix contents. Clean the glassware when finished.

Glass working

1. **Cutting Glass Tubing.** Make a scratch across the glass tubing at the desired location with a single stroke of a triangular file. Place a drop of water on the scratch with your finger. The tubing must always be held in a towel while pressure is being applied to prevent injury to the hands. Place both thumbs close together on the side of the tubing opposite the scratch, and snap the tubing in a direction away from you and others against the pressure of the thumbs.
2. **Fire Polishing Glass Tubing.** All edges of glass tubing must be fire-polished to round off the sharp edges. Hold the sharp edges of the tubing at an angle in the flame and rotate the tubing until a bright yellow color is imparted to the flame. Be sure to avoid over heating the glass tubing.
3. **Bending Glass Tubing.** Attach a wing top to the burner to spread the flame. Hold a piece of tubing horizontally in the upper portion of the flame and slowly rotate to ensure uniform heating. Continue rotation of the tubing until it becomes soft. Smoothly bend the glass tubing and allow it to cool on a fire resistant surface.

Heating Liquids

1. **Test Tube.** The test tube should be no more than 1/3 full. Place a gentle flame at the top of the liquid not at its base. Move the test tube in and out of the flame while gently swirling its contents. **Never point the test tube toward anyone.**

2. **A Hot Water Bath.** A small quantity of solution that needs to be heated to a constant temperature can be place in a hot water bath. If the solution is in a beaker than place it in a larger beaker filled _ to _ with water, and heat to the desired temperature.

Separating Liquids and Solids

1. **Decanting.** Allow the solid to settle in the beaker or test tube. Then transfer the liquid, or supernatant, with the aid of a stirring rod. Hold the stirring rod against the lip of the beaker and pour the liquid down the rod, which is touching the inner wall of the receiving vessel. Do this slowly so as not to disturb the settled solid.
2. **Gravity Filtration.** Fold a piece of filter paper in half; refold to within 10^{O} of a 90^{O} fold; tear off the corner unequally, and open. Place the folded filter paper snuggly into the funnel. Moisten the filter paper with the solvent being used and press the filter paper against the funnelÕs top wall to form a seal. Hold a stirring rod against the lip of the beaker and pour the liquid down the rod, which is touching the inside of the funnel. The funnels tip should touch the inside wall of the receiving vessel to reduce splashing. **Never** fill the funnel more than two-thirds full.
3. **Vacuum Filtration.** A Buchner funnel fitted with a rubber stopper is inserted into a suction flask. The side arm of the flask is connected to a safety trap, which in turn is connected to the water aspirator by a short piece of pressure tubing. Filter paper slightly smaller than the funnel diameter is placed over the holes and moistened with the solvent. Turn the aspirator on full.
4. **Centrifugation.** Whenever the centrifuge is used it must be balanced or it may become damaged. To do this fill, an identical tube with the same level of water in the position opposite the mixture to be separated. Close the cover and set the machine in motion. **Keep the cover closed and your hands away from the top of the centrifuge while it is rotating.**

Transferring Liquids

When the reagent is being transferred from a reagent bottle, remove the glass stopper and hold it between the fingers of the hand used to grasp the reagent bottle. **Never** lay the stopper on the desktop; impurities may be picked up and contaminate the solution. Hold the stirring rod against the lip of the reagent bottle and pour the liquid down the rod, which is touching the inner wall of the receiving vessel. This prevents splashing and losses of reagent down the side of the bottle. **Never** transfer more liquid than is necessary and **never** return unused portions to the reagent bottle.

Testing Gases for Odor

An educated nose is an important and useful asset to have in the laboratory. It must be used with caution because some gases are toxic or just irritant. **Never** hold your nose directly over the vessel from which the gas is coming from. Rather, fan some of the vapors toward your nose.

Testing with Litmus

To test the acidity/basicity of a solution with litmus paper, insert a stirring rod into the solution, withdraw it, and touch it to a litmus paper that is resting on a

clean, inverted watchglass. **Never** place the litmus paper directly into the solution.

Keeping Samples Dry

Materials may absorb water if left exposed to the air. This is to be avoided, especially if the sample of material is to be weighed precisely. The desiccator is the container to keep samples dry. It contains Drierite as a drying agent; blue Drierite turns pink when it is no longer effective.

Measuring Mass

To accurately determine the mass of a sample an analytical balance is used. Before use, the pan must be clean and dry. Zero the instrument by pressing the tare bar on the panel. Nothing can be on the pan while this is being done and the sliding glass panes must be shut. Begin the weighing by opening the glass pane and placing the object to be massed on the pan. **Never** place any liquid or solid directly on the pan. Either use a piece of weighing paper or weigh an empty weighing bottle then place the desired substance into the bottle and weigh again. Subtract to determine the actual mass of the sample. It also may be possible to tare a weighing vessel before adding the material whose mass you are trying to determine.