Appendix IV
Laboratory Equipment and Techniques

Laboratory Equipment

Much of the laboratory equipment you will use in this course is shown on the following pages. Use this list when you are checking into your drawer at the beginning of the quarter. Learn the names and uses for each piece of equipment.

It is important that you have some knowledge of the precision (see the following table) of the various pieces of laboratory equipment available. You will use different apparatus and techniques for measurement depending on the precision you need. For example, if you want to measure a volume of water, you should know whether it is more appropriate to use a buret, a pipet, a volumetric flask, a graduated cylinder, or a graduated beaker, or whether you should weigh the water. When planning experiments, you should use equipment and techniques that have approximately the same precision. If two measurements are to be added or subtracted, they should have the same absolute precision; if they are to be multiplied or divided, they should have the same percent precision. The overall precision of the experiment is determined by the least precise measurement you make, so you should not waste time by making other measurements more precisely.

Measuring Device	**Precision**	**% Precision (of Total Volume)**
Platform balance	±0.1 g	---
Cent-O-Gram balance	±0.01 g	---
Analytical balance	±0.0002 g	---
50-mL buret	±0.05 mL	0.1 %
5-mL transfer pipet	±0.01 mL	0.2 %
10-mL transfer pipet	±0.02 mL	0.2 %
25-mL transfer pipet	±0.03 mL	0.1 %
50-mL transfer pipet	±0.05 mL	0.1 %
100-mL transfer pipet	±0.08 mL	0.08 %
10-mL measuring pipet	±0.1 mL	1 %
25-mL volumetric flask	±0.03 mL	0.1 %
50-mL volumetric flask	±0.05 mL	0.1 %
100-mL volumetric flask	±0.08 mL	0.08 %
250-mL volumetric flask	±0.12 mL	0.05 %
500-mL volumetric flask	±0.15 mL	0.03 %
1000-mL volumetric flask	±0.3 mL	0.03 %
Graduated beaker	---	5 %
10-mL graduated cylinder	±0.1 mL	1 %
25-mL graduated cylinder	±0.2 mL	0.8 %
50-mL graduated cylinder	±0.25 mL	0.5 %
Mercury barometer	±0.5 torr	0.1 %
Thermometer (1°C graduations)	±0.2 °C	---

Laboratory Techniques

To carry out common laboratory procedures accurately and efficiently, you must use the proper techniques. Poor technique usually results in poor data and may lead to difficulty in finishing the work in the allotted time. Some of the common techniques required in this course are given below. Your instructor will assist you in learning the best way to carry out these operations. Refer to this section as necessary when planning your work in the laboratory.

Dispensing Materials

You should learn proper dispensing techniques in order to maintain the purity of the reagents. The use of proper techniques will also help to ensure your safety and the safety of others in the laboratory.

Solids: Solids should be dispensed by rolling and tilting the bottle until enough of the solid is obtained. To avoid contamination, never dip a stirring rod, pipet, dropper, or spatula into a reagent bottle. Never return unused reagent to the bottle. Conserve chemicals by taking only what you need. If you accidentally get too much chemical, see if another student can use your excess. Always replace the cap on the reagent bottle.

Liquids: Liquids will be stored in bottles with droppers or ground glass stoppers. Before using dropper bottles, make sure the dropper is in good condition. For stoppered bottles, hold the stopper in and tilt the bottle to wet the stopper. Wetting the stopper lubricates the ground glass to allow easy removal of the stopper. Moisten the inside of the neck and the lip with the wet stopper. This will prevent the first drops from gushing out when pouring. Never set the stopper down or it will become contaminated. Replace the stopper. Remove the stopper again by turning your hand over and grasping the stopper between two fingers. The neck of the bottle should touch the edge of the vessel that you are using to collect the liquid to prevent the liquid from running back down the outside of the bottle. The stopper should remain firmly held between the fingers while the liquid is poured. Replace the stopper when enough liquid has been dispensed. Do not pour excess chemical back into the reagent bottle. If you accidentally get too much chemical, see if another student can use your excess.

If you spill chemicals while dispensing them follow the suggestions given in Appendix A.

Bunsen Burner

You will use a Bunsen burner regularly as a source of heat. Methane (natural gas) is used as the fuel for the burners and is very flammable. To avoid large flame-ups, follow these steps. First, adjust the air control by closing the air adjustment valve, located at the bottom of the burner, so that a minimum of air is supplied. Hold a lighted match or striker close to the top, but to one side of the burner; then turn on the gas. Adjust the gas flow and then increase the air flow until a nonluminous blue flame is obtained. If the flame rises too high from the burner, turn down the gas. If the blue inner cone is absent or the flame has a yellow tip, open the air adjustment valve. The hottest part of the flame is just above the top of a blue inner cone, which can usually be observed in the flame.

If the burner "strikes back" and ignites at the base, turn off the gas and let the burner cool before relighting it. Striking back can be prevented by decreasing the air flow or by adjusting the gas flow.

Heating a Test Tube: Use a test tube holder. Hold the test tube at a slight angle. Wave the tube through the flame. Heat near the top of the solution. Never stopper a tube while it is being heated and never point the test tube at anyone.

Heating a Beaker or Flask: To obtain smooth boiling, use boiling chips or a stirring rod and locate the flame directly under the end of the rod. Use a ring, ring stand, and wire gauze to heat heavy glassware such as beakers and flasks. Never heat volumetric glassware (i.e. volumetric flasks) since heating will distort the glass and you will lose the accuracy of the flask. For best results, do not heat a flask that is more than half full.

Hot Plate/Stirrers

It is often preferable to use a hot plate rather than a Bunsen burner for heating because a hot plate does not involve the use of an open flame. The hot plates available to you are combination hot plate and stirrers. You may use either function or both at the same time.

Heating: Adjust the dial to the desired level of heating. The highest settings will boil water; the lowest will maintain a fairly constant temperature. Keep a close eye on your liquid. Students have the tendency to crank the dial up to "high," but at this level the thermometer can not keep up with the heating and the liquid may be much hotter than expected.

Stirring: Place a magnetic stir bar in your liquid. Adjust the speed of stirring to the desired level. The stir bar should be spinning freely in the center of your beaker/flask. If the stir bar is jumping around, stop the stirring and try again.

Balances

There are two types of balances you will be using: triple beam and digital analytical balances. Regardless of the type of balance, the following rules apply:

a) If the balance is left in an improper condition, or if there is an apparent difficulty on operating the balance, consult your instructor immediately. Do not attempt any adjustments on your own.
b) Clean up any spilled chemicals on the balance immediately.
c) During the course of the experiment, use the same balance.
d) Never weigh chemicals directly on the balance pan; use a watch glass, weighing bottle, weighing boat, or other container.
e) Weigh volatile or corrosive samples (by difference) in stoppered containers.
f) Do not weigh objects unless they are at room temperature.
g) After using the balance, return all knobs, dials, or weighs to zero.
h) Leave the balance and its area clean and free of all chemicals, containers, papers, and so forth.

Triple beam balances: These balances are like the ones found in doctors' offices and are familiar to most people. To begin, make sure the balance is reading 0.0 g with no weight on the beams. Place the sample to be weighed on the balance pan. Add weight to the beam starting with the largest weight. Add the weight one notch at a time until the beam is depressed below the zero line, then back the weight up one notch. Do this for each successively smaller weight until all you have left is the small slider. Slide this small

weight until the beam levels at the zero mark. Sum all of the weights for your reading, estimating between marks on the smallest scale. Slide all weights back to zero and remove your sample.

Digital Analytical Balance: These balances have made precision weighing very simple. Zero the balance by pressing the large bar on the front of the balance. Add the sample to be weighed to the balance pan. A few seconds will pass while the balance tries to stabilize. Use the wind shields and do not lean on the counter. These balances are surprisingly sensitive. Record all of the digits displayed by the balance.

Determining mass by taring is also very easy on digital balances. Place the container on the balance pan and press the zero button. The balance will now read 0.00 g with this container on it. Remove the container and fill it with the sample to be weighed. Return the container to the balance pan and the balance will display the mass of the sample.

Accurate Weighing on an Analytical Balance

Weighing by difference will be the preferred weighing technique in this class. To use this technique, put sufficient material for all the samples to be weighed into a vessel. **The vessel must never be handled directly: use tongs, fingercots, or other protection.** Weigh the vessel and contents and record the reading. Remove the vessel from the balance. Using a spatula, quantitatively transfer small amounts of material to a clean beaker or flask until the approximate weight is reached. Weigh the vessel precisely again. The difference between the two weighings is the weight for the first sample. The weight of the second sample is obtained in the same manner as that of the first, but since the initial weight is already known, only one additional weighing is needed. Thus, three samples require only four weighings.

Some balances, such as the Mettler balances you will use, are equipped with a built-in compensator (taring mechanism) that enables the operator to set the balance to zero with an empty vessel on the pan. In this way, a sample can be weighed directly into a beaker or flask with only one weighing (excluding the zero reading) per sample. While this is the most efficient method, the advantage of weighing by difference is that it enables hygroscopic or volatile samples to be weighed with little exposure to the atmosphere. (The cap remains on the weighing bottle except for the time it takes to rapidly transfer some to the empty flask). Furthermore, weighing by difference prevents any spillage of chemicals to ever occur **INSIDE** the balance. **Beware that TA's have been instructed to deduct points from your laboratory report grade if they ever see a student trying to use a spatula to place chemicals in a weighing container that is inside a balance.**

Potential sources of error in weighing with a constant-load balance include inaccurate weights, shifts in balance zero or sensitivity, and changes in the sample. Errors from weight changes in the sample include absorption of moisture (especially serious when a hygroscopic material is being weighed), volatilization, air currents from a hot sample or container, and static charge on containers.

Graduated Cylinders

Make sure the graduated cylinder is clean before attempting to use it. Water should not bead up on the sides of the cylinder. If it does, clean the cylinder before use. Once the cylinder is clean, pour the desired liquid in, being careful not to spill any. Read the graduated cylinder with your eye on the same level as the meniscus (curved surface of the liquid). Always read the bottom of the meniscus. When reading volumes remember to

estimate to a fraction of the distance between adjacent lines. That is, if the bottom of the meniscus is between the lines 12.0 and 12.1, and closer to 12.0, your reading might be 12.03 mL.

Buret

Make sure the buret is clean before attempting to use it. Water should not bead up on the sides of the buret. If it does, clean the buret before use. Push the stopcock in to make sure it securely placed in the buret. If it feels loose, replace the o-ring. Using a small funnel to assist in filling, rinse the clean buret with a few milliliters of the solution. Allow the buret to drain completely. Repeat the rinsing. Fill the buret to above the zero mark with the solution. Open the stopcock wide for a few seconds to remove all the air bubbles from the tip.

Refill the buret, if necessary, keeping the volume below 0.00 mL. Do not attempt to set the initial reading at 0.00 mL or at any other specific reading. Read the bottom of the meniscus, keeping your eye level with the meniscus. Estimate the volume to the nearest 0.02 mL. This volume will be your initial reading.

Allow the solution to flow from the buret. Use the stopcock to adjust the rate at which the liquid flows from the buret. Place the hand so that the thumb and two fingers span the stopcock. This enables you to maintain a slight inward pressure on the Teflon plug to prevent leakage. Swirl the liquid in the flask and wash the walls of the flask frequently with distilled water from a wash bottle. With practice, you should be able to deliver fractions of a drop to your flask. To do this, hold the buret with one hand and with the other hand rotate the stopcock rapidly 180 degrees. Keep an eye on the volume of solution remaining in the buret as you are dispensing it; you do not want the solution to end up below the graduations. Once you have delivered the appropriate amount of solution, take your final reading. The difference between the final reading and the initial reading is the volume you delivered.

Transfer Pipet

Make sure the pipet is clean before attempting to use it. Water should not bead up on the sides of the pipet. If it does, clean the pipet before use. Obtain the liquid to be measured in a beaker. Do not pipet from a reagent bottle because you risk contaminating the reagent. Rinse the pipet with a small portion of the liquid. Discard the rinse liquid in the appropriate waste container.

Use a bulb to draw the liquid above the mark on the transfer piper. *Never pipet by mouth!* Place the bulb tight against the end of the pipet to get a seal. Do not cram the bulb over the end of the pipet. If you are right-handed, use your right hand to hold the pipet and your left hand to hold the bulb. Remove the bulb and quickly place you right index finger over the end of the pipet. Wipe off the outside of the pipet with a paper towel. By adjusting finger pressure, allow the liquid to drain so the bottom of the meniscus is on the marked line. Touch off the adhering drop against the side of the vessel (or carefully wipe off the outside of the pipet with a paper towel). Remove your finger from the pipet end to drain the pipet into an appropriate vessel. After discharge, allow an extra 20 seconds for complete drainage. Touch the tip of the pipet to the side of the vessel to remove the last drop. The liquid remaining in the pipet tip should not be blown out into the sample;

however, it must be disposed of properly. The pipet is calibrated to account for this remaining amount.

Volumetric Flask

Make sure the flask is clean before attempting to use it. Water should not bead up on the sides of the flask. If it does, clean the flask before use. Use a funnel to place a calculated amount of solute (or a known volume of liquid to be diluted) in the clean volumetric flask. Rinse the funnel with distilled water (or appropriate solvent). When diluting an acid, add some water first, then add the acid. Add water and swirl to dissolve the solute. Add more water, swirling occasionally until the bottom of the meniscus of the liquid just matches the marked line on the neck of the flask. Insert the stopper and invert the flask several times. The enclosed air passing through the solution ensures complete mixing. If you see schlieren lines (swirls in the liquid), the solution is not mixed. Continue inverting the flask until no schlieren lines exist.

Filtration

Gravity filtration: Prepare a piece of filter paper by folding it in half and creasing lightly. Fold the paper in half again, then pull one layer of the paper open to form a cone. Fit the cone into a funnel and wet the paper with a few milliliters of distilled water or appropriate solvent. Press the cone against the side of the filter funnel to make it fit tightly.

Transfer the solution to the filter paper by pouring the liquid along a stirring rod. Do not fill the filter paper too full. If the paper is properly sealed to the funnel, a column of water should fill the funnel stem. The mass of this column of water hastens the filtration. If the stem of the funnel is in contact with the side of the beaker or flask, the filtrate will run down the walls of the beaker, which prevents spattering. Wash the solution from the original beaker with small amounts of distilled water or appropriate solvent.

Vacuum filtration: Place a piece of filter paper flat against a Büchner funnel bottom and moisten with distilled water to seal. Be sure the filter paper is large enough to cover the holes but small enough to easily fit flat in the funnel. Make sure the flask is securely clamped. Create a partial vacuum by connecting to a vacuum source. The vacuum source may be a house vacuum system or an aspirator. An aspirator is connected to a water faucet. Rapidly flowing water creates a vacuum as it passes a T in the aspirator. The filtration system is connected to the T.

Once the vacuum is established, pour the solution to be filtered into the funnel. The residue should be washed, if appropriate, and may be dried by pulling air through the filter. If an aspirator vacuum is used, *do not turn off the water to the aspirator until the filter flask is disconnected or the vacuum otherwise broken.* Failure to do this could lead to a water backup into your filtrate.

PLEASE REFER TO THE NEXT COUPLE PAGES FOR IMAGES OF SCIENTIFIC LABORATORY EQUIPMENT.