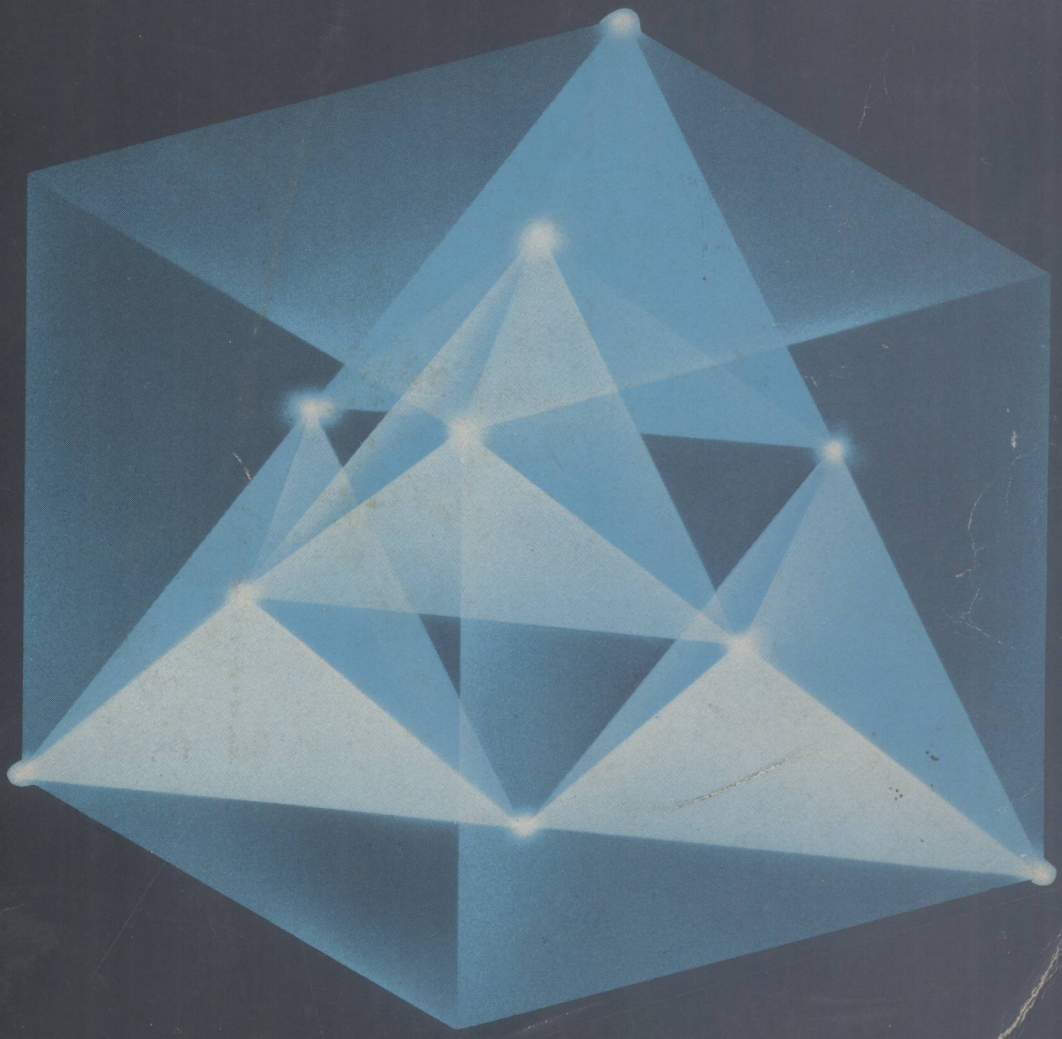
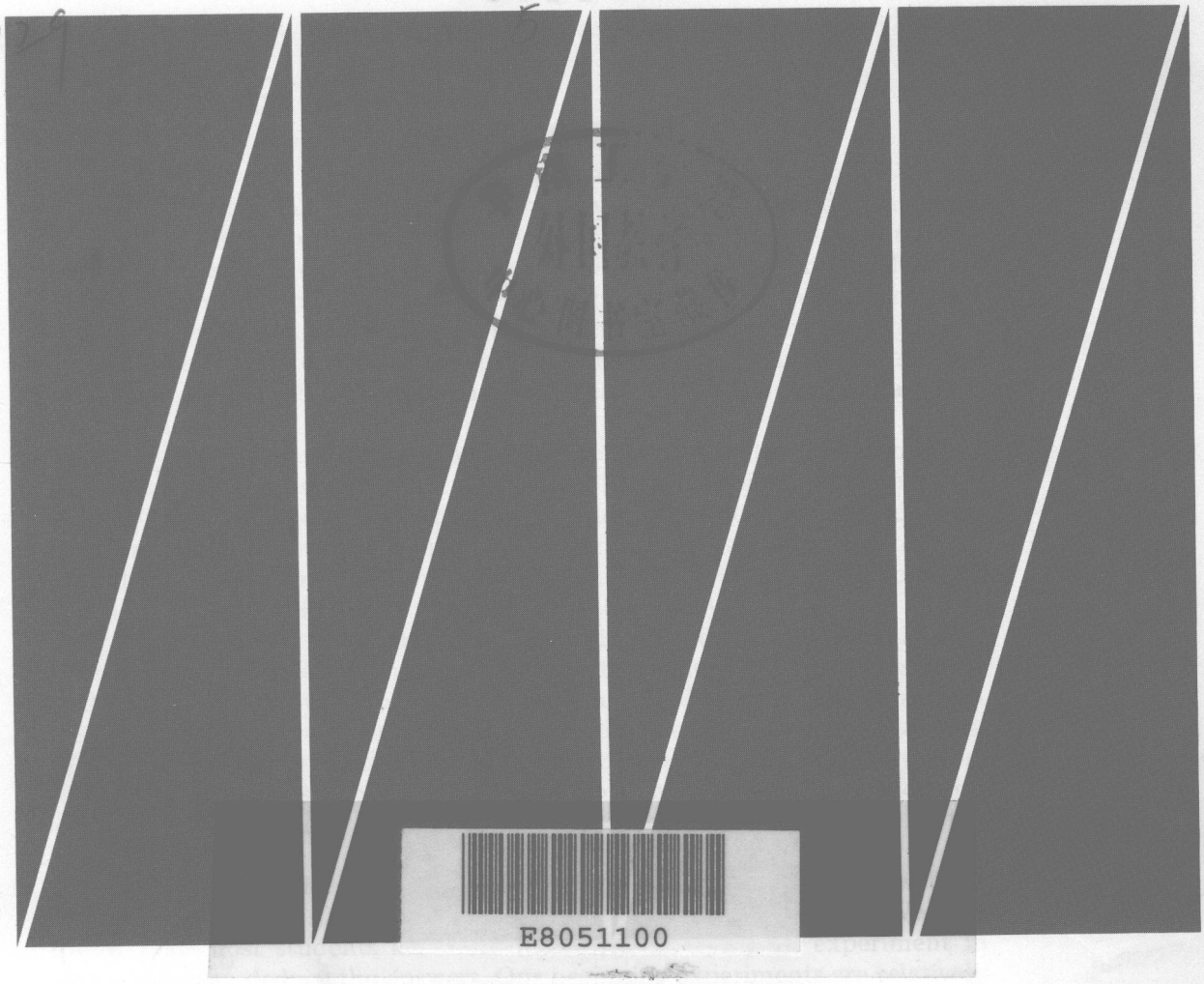
Laboratory Manual for CHEMISTRY

MACIEL · TRAFICANTE · LAVALLEE



by Mark Bacon · Clarence Josefson



LABORATORY MANUAL for Chemistry

MACIEL / TRAFICANTE / LAVALLEE

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Laboratory Manual for CHEMISTRY

Preface

Over several years we have developed a set of experiments to use in our general chemistry laboratories. Since our experimental approach matched the goals of *Chemistry* by Maciel, Traficante, and Lavallee, it seemed natural simply to revise our experiments, add some new ones, and compile all for an accompanying laboratory manual. Although written to accompany the Maciel, Traficante, and Lavellee textbook, the manual can be used in most general chemistry laboratories for majors in chemistry, engineering, and the health sciences. We devised many of the experiments; a few are standard and traditional.

An important feature of our approach is the section called Pre-Laboratory Questions included with each experiment. These questions encourage the student to think about the experiment, and where desirable, to look up facts and data before going into the laboratory. We recommend that the laboratory instructor insist that the student complete the pre-laboratory questions satisfactorily before going into the laboratory.

A section called Post-Laboratory Questions and Calculations is also provided with each experiment. These questions give students a chance to assess their understanding of what they did and to discuss the questions and calculations. The experiments are all designed to be run in two hours or less, so that the usual three-hour laboratory period should allow plenty of time for both pre- and post-laboratory sessions. Both the pre-laboratory questions and the post-laboratory questions and calculations are incorporated in the report sheets.

Instead of telling the student the answer and asking him or her to verify it, we have encouraged students to discover the answer for themselves. We have found that most students are more interested in trying an experiment in which they can deduce the answers. Our beginning experiments are relatively concrete and should give the student the basic tools for approaching new problems and methods confidently. Our experiments gradually require more initiative from the student. For example, later experiments require the student to devise a qualitative analysis scheme or to engage in a three-step synthesis of a transition metal complex and understand the purpose of each operation in the synthesis.

In keeping with our goal of encouraging students to question and to discover for themselves, we have deliberately left the report sheets unstructured. Although space is made available for data, observations, and results, the responsibility for the manner of recording this information remains with the student. A sample report sheet is included at the end of the Introduction.

We gratefully acknowledge the help and encouragement of Professors Gary E. Maciel, Daniel D. Traficante, and David Lavallee.

Mark Bacon
Clarence Josefson

Introduction

COMMON LABORATORY EQUIPMENT

When you first check into your general chemistry laboratory and open your equipment drawer, you will be confronted with an array of essential equipment, much of which may be unfamiliar. To get acquainted with your equipment, compare each piece with the items shown in Fig. 1.

WEIGHING OPERATIONS

Two types of balance are normally found in a chemistry laboratory: a simple, rugged type for rough weighings (to ±0.1 or ±0.01 g) and a more delicate, precise analytical type for precise weighings (to ±0.001 or 0.0001 g).

Platform balances and triple-beam balances (see Figs. 2 and 3) are used to weigh out approximate amounts of materials. They are the "workhorse" balances in the laboratory. Although it is very easy to use them, a few basic rules should be observed:

1. Before placing an object on the balance, arrest the balance pan (if the balance has an arrest). This precaution avoids unnecessary jarring of the mechanism.
2. Never put chemicals directly on the balance pan. If you do, both reagents and balance will become contaminated. Place the chemical in an aluminum or a plastic weighing dish or on a piece of weighing paper. These items are so light that their weight does not significantly increase the total. In some situations, weighing in a beaker is more convenient; since a beaker is itself heavy, its weight must be subtracted from the total weight to get the weight of the sample. This procedure is called weighing by difference. First weigh the dry, empty beaker (this step is called taring and the empty beaker is called the tare). Place the sample in the beaker and reweigh. You can add or subtract small amounts of sample until a predetermined, combined weight is reached, corresponding to the sample weight desired. The difference in weight of beaker-plus-sample and the tare weight is the weight of the sample.
3. Volatile, highly corrosive, or highly poisonous liquids should be weighed in stoppered containers.
4. Hot objects should be allowed to cool before being weighed.
5. When you are finished weighing, clean up any spills and arrest the balance pan. If a highly corrosive or poisonous substance is spilled in or on the balance, immediately inform the laboratory instructor.

If you are unfamiliar with the operation of the platform or triple-beam balances in your laboratory, ask your instructor for help. Practice weighing a few objects to learn how to operate these balances.

Figure 1 Illustrations of common equipment

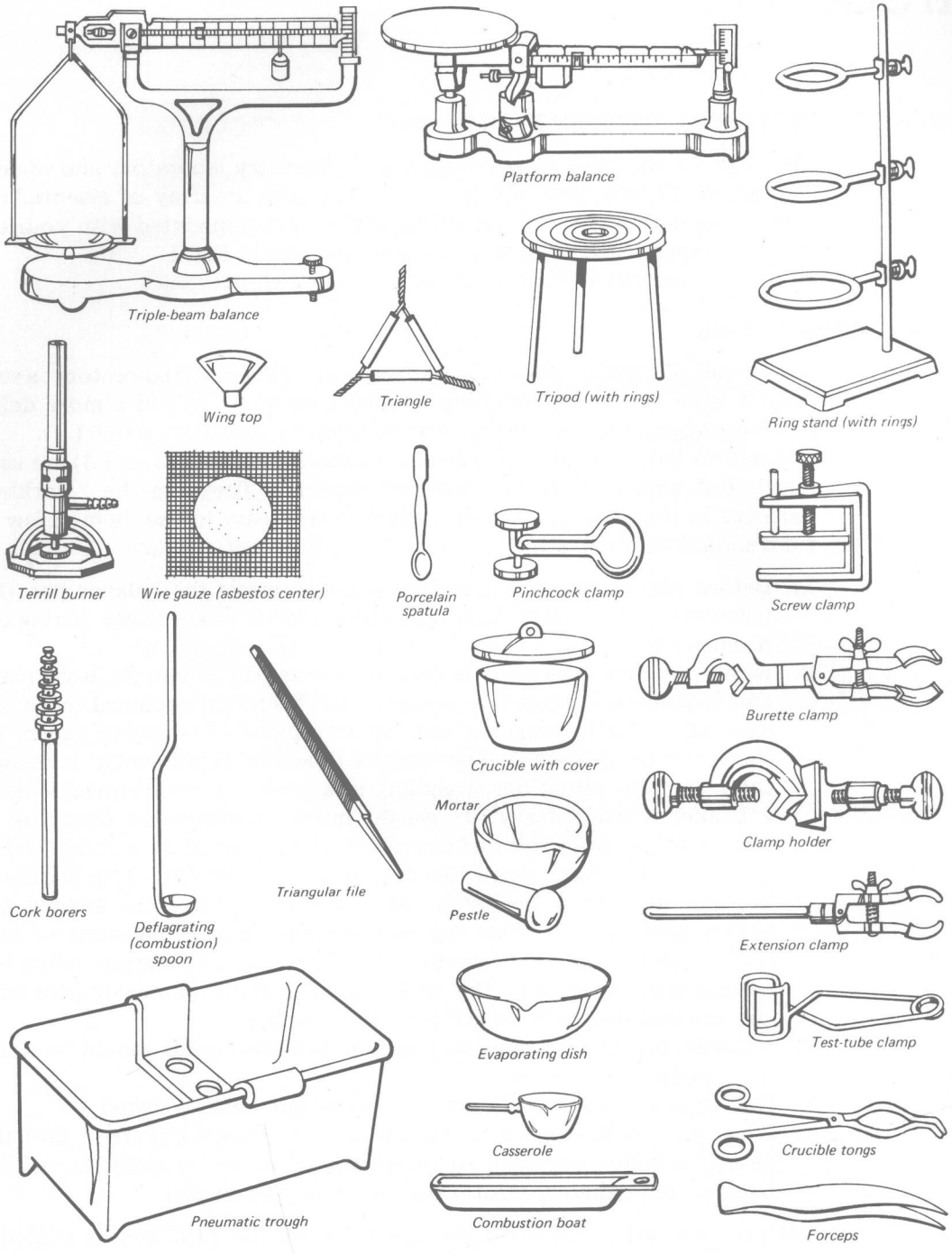
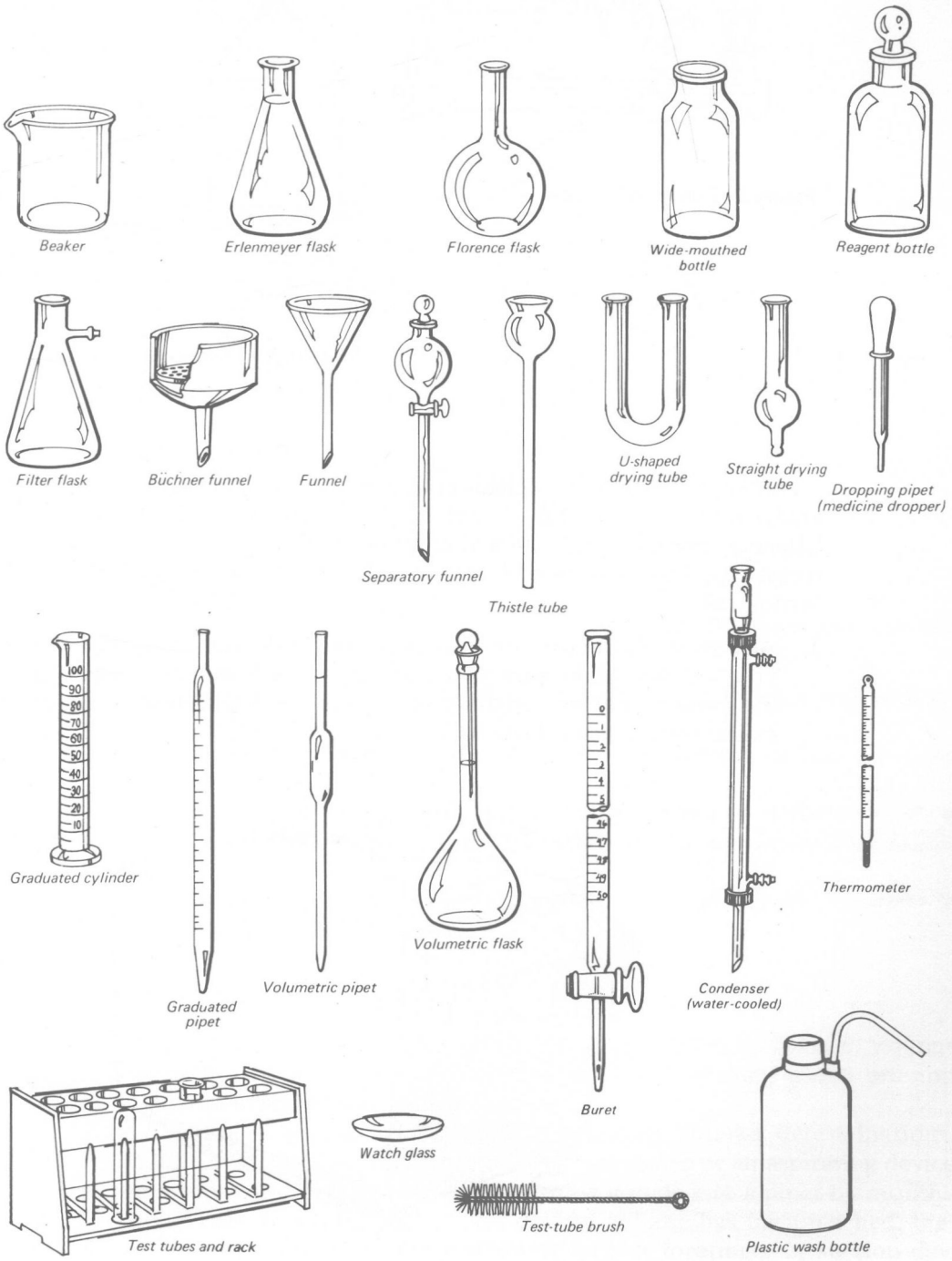


Figure 1 (continued)



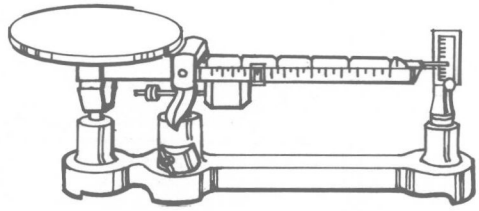


Figure 2 Platform balance

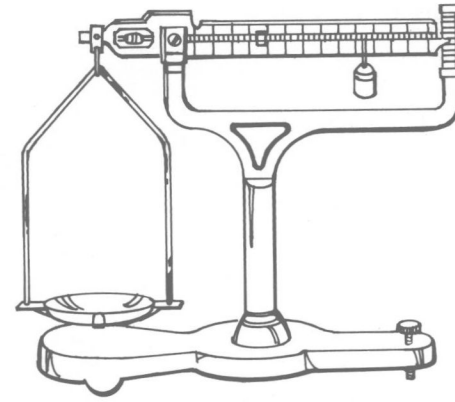


Figure 3 Triple-beam balance

Precise, quantitative weighings are made on top-loading and single-pan analytical balances (Figs. 4 and 5). These devices, especially the single-pan balances, are more delicate and more easily damaged than balances for rough weighings; therefore special care in using them is essential. Observe the following rules:

1. Ask your instructor for directions and a demonstration of the balance you will use. Make sure you understand such essential details as zeroing the balance before weighing and using the half-release position (if one exists) on the pan release knob.

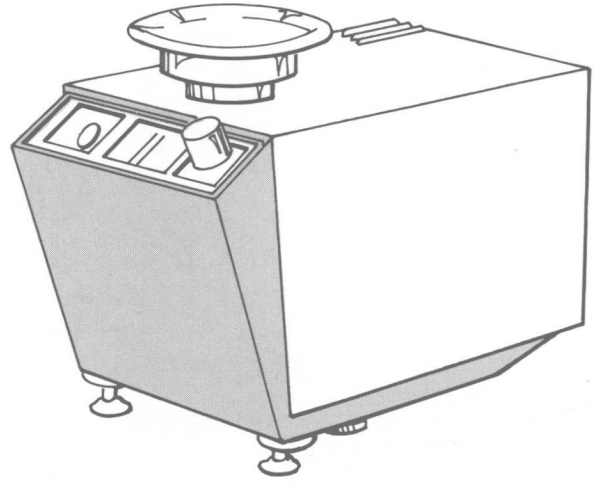


Figure 4 Top-loading balance

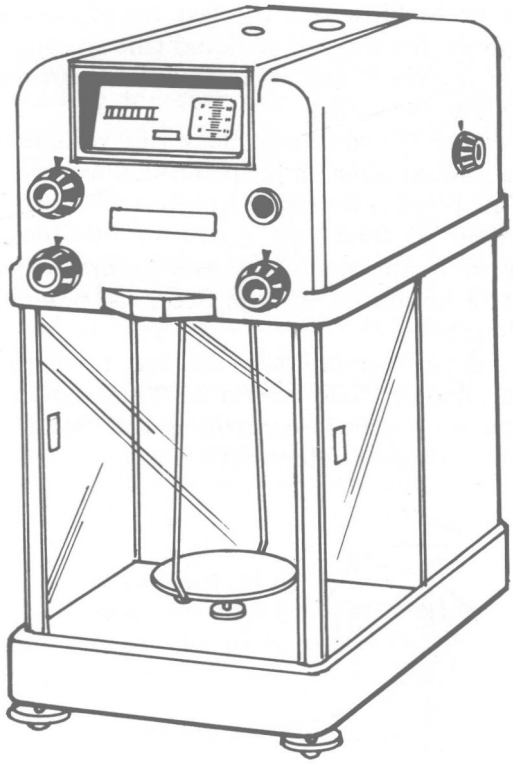


Figure 5 Single-pan analytical balance

2. Always keep the pan arrested when dialing in weights and adding or removing sample.
3. Arrest the pan and return all weight adjustments to zero when you are finished weighing.
4. Weighing on precise balances is nearly always by difference, since even the lightest weighing dish or paper weighs an appreciable fraction of a gram.
5. The rules given for approximate weighings also apply to more precise weighings.

VOLUME MEASUREMENTS

Approximate volumes are marked on many beakers and flasks. Volumes can be measured more precisely with graduated cylinders, which are normally supplied in sizes from 5 ml to 2 liters.

Calibrated pipets afford relatively precise volume determinations. The liquid is drawn into the pipet by vacuum power or an aspirating device such as a rubber bulb. CAUTION: Never draw liquids into a pipet by mouth. Most liquids are poisonous! When the desired volume has been reached, the pipet is held over the receiving container, with a forefinger or suction device in place over the top opening of the pipet, and allowed to drain by gravity when the finger or suction device is removed. Do not force air into the pipet to

make it drain faster or to blow out the last bit of liquid. Most pipets are calibrated to compensate for the bit of liquid (the so-called holdup) that remains in the tip after the pipet has drained. Your measured volumes will be too large if you force out the holdup.

The most precise device for routinely measuring volumes is the volumetric pipet. Unlikely a calibrated pipet, a particular volumetric pipet can deliver only one volume. This pipet is normally supplied with capacities of 1.00 ml, 2.00 ml, 5.00 ml, 10.00 ml, 20.00 ml, 25.00 ml, 50.00 ml, and 100.00 ml. It is filled and drained in the same way as a calibrated pipet. This type is also compensated for a holdup at the tip. See Figs. 6 and 7 for illustrations on using a volumetric pipet.

A more specialized device for precisely measuring volumes is the buret. A buret is normally supplied in 25.00-ml and 50.00-ml capacities. It has a stopcock near the bottom for precisely controlling the desired volume. Figure 8 shows how to read the indicated volume of a buret.

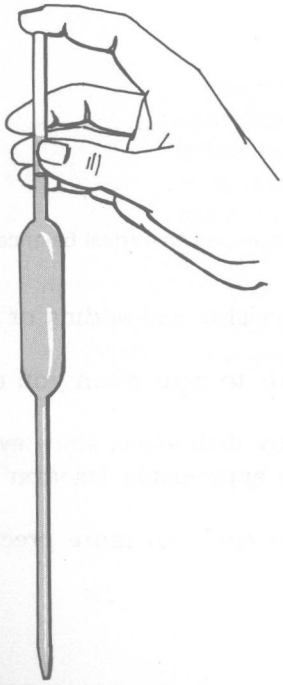


Figure 6 Retaining the liquid in a volumetric pipet

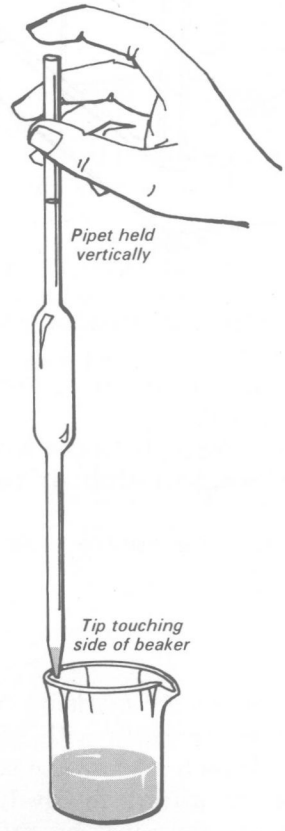


Figure 7 Draining the liquid from a pipet

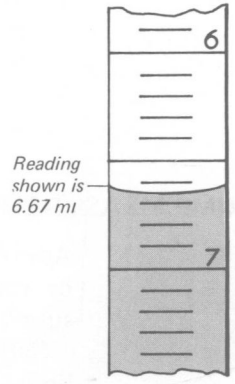


Figure 8 Close-up of buret

GLASSWORKING

Glass is a noncrystalline, transparent mixture of metal silicates. It is made by melting together silicon dioxide (sand), calcium carbonate (limestone), and sodium carbonate (washing soda); small amounts of other substances are often added. Glass is a liquid that has been supercooled to a temperature at which its viscosity (resistance to flow) is so great that it appears to be a solid. Unlike a true solid, glass does not start to melt at a definite temperature; instead, it gradually softens as it is heated—the soft glass you will work with starts to soften at about 600 °C. Soft glass is about 75% silicon dioxide.

Most of your laboratory equipment—beakers, flasks, and so on—is made of Pyrex brand heat-resistant glass. This contains aluminum oxide and boron oxide instead of calcium oxide. The result is a glass with a higher softening temperature and a very low coefficient of thermal expansion. Since this glass does not expand or contract much, it is much less likely to crack during a sudden temperature change. The advantages of glass for laboratory use are:

1. It is transparent.
2. It does not dissolve in most chemicals nor react with them.
3. When softened, it can be formed into various useful shapes.
4. It is a poor conductor of heat.

Its disadvantages include:

1. It is brittle; and when it becomes broken, its ragged edges can cut you badly.
2. It cools very slowly, so you can burn yourself with it if you are impatient.

Following are some methods of working glass. When you are finished working glass or performing experiments, remember to clean up your work area. Dispose of glass scraps in an appropriate container.

1. Cutting Glass Tubing

Obtain a length of 6-mm glass tubing from the side shelf in the lab. Cut it into 20-cm lengths as follows. Scratch the glass at the desired point with a single stroke of a triangular file or glass scratcher. Place both thumbs close together on the side of the tubing opposite the scratch and pull on the tubing lengthwise while your thumbs exert an outward pressure against the scratch. See Fig. 9. If gentle pressure does not cause the glass to break at the point of the scratch, make the scratch slightly deeper and repeat the operation. Tubing larger than 6 mm in diameter must always be held in a towel while pressure is being applied, to prevent injury to the hands.

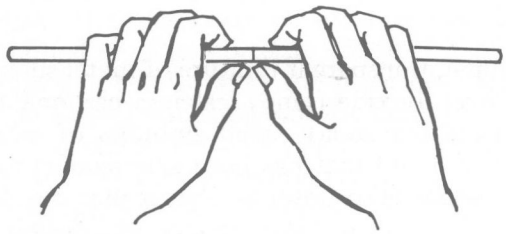


Figure 9 Correct positioning of the the fingers for breaking glass tubing

2. Fire-Polishing Glass Tubing

All edges of glass tubing must be fire-polished to avoid cutting the hands or the rubber stopper or rubber tubing into which the glass may be inserted. Hold the sharp edges of the tubing in the hot part of the flame while holding the tubing at a 45° angle. Rotate the tubing until a bright yellow color is imparted to the flame by the glass. Fire-polish each end of the tubing previously cut. (CAUTION: A heated piece of glass stays hot for some time.)

3. Bending Glass Tubing

Attach a wing top to the burner to spread the flame. This makes it possible to heat a greater length of glass tubing. Hold a piece of 6-mm tubing horizontally in the hot upper portion of this flame. Support the tube with one hand and rotate it with the other to ensure uniform heating. See Fig. 10. Continue rotation until the glass softens; then remove the tubing from the flame and bend it to the desired angle (Fig. 11). While the glass is cooling, it should be laid on a wire gauze or held until cool. Your instructor will give you specific instructions on the dimensions of bends that you are to make. Have your instructor check your bends.

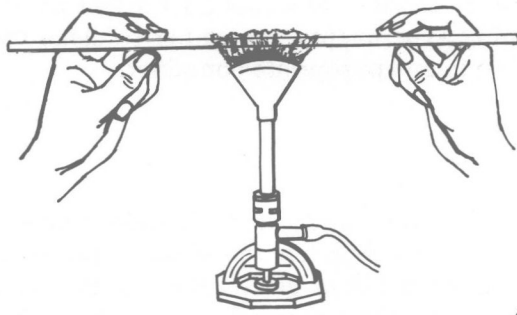


Figure 10 Method of heating glass tubing preparatory to bending. Notice the wing top on the burner.

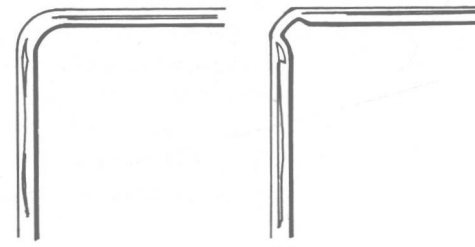


Figure 11 Glass tube bent at an angle of 90°. Notice the constriction in the bend illustrated in (b).

4. Constructing Dropping Pipets

In a burner flame without the wing top attached, rotate the middle 3-cm section of a 20-cm length of 8-mm glass tubing. When the glass becomes soft, remove it from the flame and pull slowly on the ends until a constriction 2-3 cm long forms at the point of heating. Allow this to cool; then cut at the center of the constriction. Fire-polish all sharp edges. Next, rotate the other end in the flame. When the glass is soft, remove it from the flame and press vertically down on a sheet of asbestos to form a thick ring, or bead. After the glass is cool, attach a rubber bulb. Make two pipets and store in a safe place for future use.

5. Preparation of Capillary Tubes

Rotate the middle 3-cm section of a 20-cm length of a 6-mm glass tubing in a burner flame. When the glass becomes quite soft, remove from the flame and pull quickly on the ends until your arms are completely extended. Cut sections of capillary about 10 cm long and save. Repeat if necessary, to obtain good capillary tubes.

6. Inserting Glass Tubing in a Stopper

Make sure that both ends of the tubing have been fire-polished. Wet both the tubing and the hole in the stopper with water or glycerol. Protecting your hand with a towel, introduce the tubing into the hole in the stopper with a gentle twisting motion. Never try to push the tubing through the stopper, as the tubing is almost certain to snap and inflict serious, painful cuts. Insert one of your long glass bends into a two-hole stopper.

7. Removing Glass Tubing from a Stopper

Wrap the tubing with a towel and pull it from the stopper with a gentle twisting motion. Water or glycerol introduced around the hole and on the tubing makes removal easier. In stubborn cases, a cork borer must be used as a tool. See the instructor for details.

8. Preparation of a Small Test Tube

Take the pieces of tubing left from the preparation of the capillaries and strongly heat near the end. Excess glass can be removed by touching the hot glass with a glass rod and quickly pulling away the excess glass. Heat the end of the tube while rotating the glass until it is quite hot. You can round off the end by blowing gently into the tube.

DECANTATION AND FILTRATION

Decantation and filtration are ways of separating a liquid from a solid. In some cases the liquid is the desired part of the mixture and the solid is to be discarded; in other cases, the solid is to be saved and the liquid discarded.

1. Decantation

When the solid is dense and settles readily, the easiest way of separating it from the liquid is by decantation. After the solid has settled to the bottom of the container, carefully pour the liquid from the solid, using a stirring rod to guide the stream of liquid (Fig. 12).

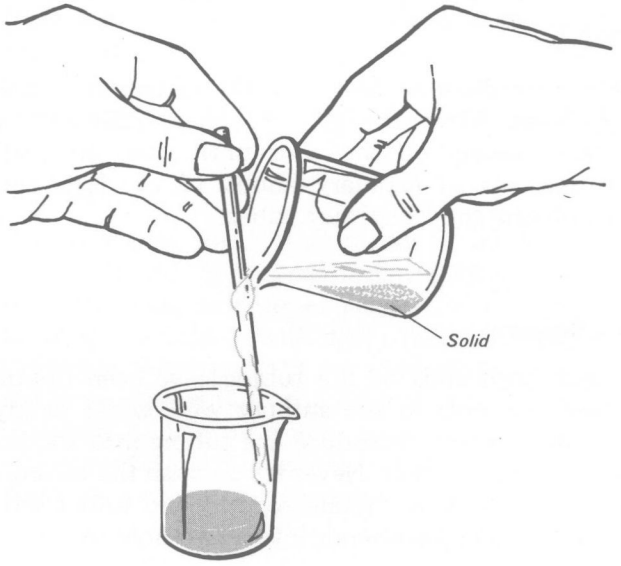


Figure 12 Decanting a liquid from a solid

2. Gravity Filtration

When the solid tends to remain suspended in the liquid without settling completely, the intervention of a piece of filter paper is necessary for adequate separation. Gravity filtration will usually provide the desired separation. A piece of qualitative or semiquantitative (relatively porous) filter paper is folded in half, then folded again, approximately but not exactly in quarters. The corner of the smaller two "quarters" is torn off so that the filter paper will properly fit the funnel (Fig. 13). Place the quartered filter paper in the funnel, unfold one of the larger "quarters" from the others to make a cone, and wet it slightly so that it will form a seal with the funnel. Support the funnel on an iron ring so that the stem touches the inside of the beaker to receive the liquid (Fig. 14). Pour the mixture to be separated into the filter paper-funnel combination, using a stirring rod to guide the stream. The filter paper should not be filled to the top, since suspended solid will tend to creep over the edge and end up in the liquid being separated (called the filtrate).

Figure 13 Steps in folding filter paper for gravity filtration

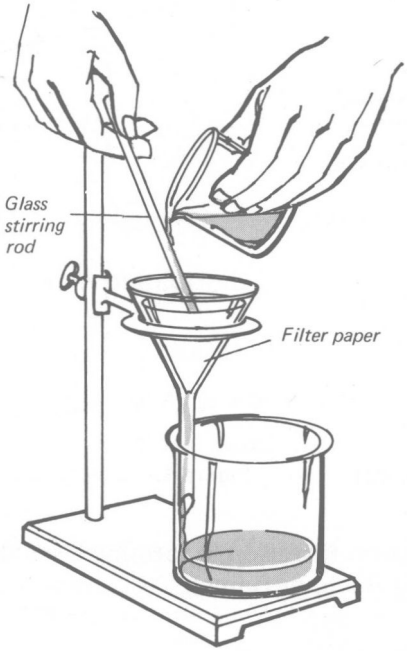


Figure 14 Correct method of gravity filtration

3. Suction Filtration

It is only slightly more tedious to set up and perform suction filtration than it is gravity filtration, yet suction filtration provides more complete separation. It is much faster. It is the method of choice for very fine precipitates (fine-porosity quantitative filter paper can be used) and for solids that must be washed free of impurities and partially dried on the filter paper.

A Büchner funnel (or a Hirsch funnel for very small volumes) is fitted with a rubber stopper and attached to a filter flask (Fig. 15). A length of heavy-walled rubber tubing is used to connect the filter flask to a water aspirator (by way of a safety trap; see Fig. 16) or other source of vacuum. Place a circle of filter paper of appropriate diameter and porosity into the funnel. If the filter paper is too small, the outer holes in the bottom of the funnel will not be covered, and if it is too large, it will bunch up along the side of the funnel. Either situation will cause the filtration to fail. Turn on the suction and pour enough solvent into the funnel to seal the filter paper to the bottom. Now pour the mixture into the funnel, using a stirring rod to guide the stream, as in gravity filtration. If you wish, the solid can be conveniently washed under suction by pouring a little pure solvent on the solid and letting it drain under suction.

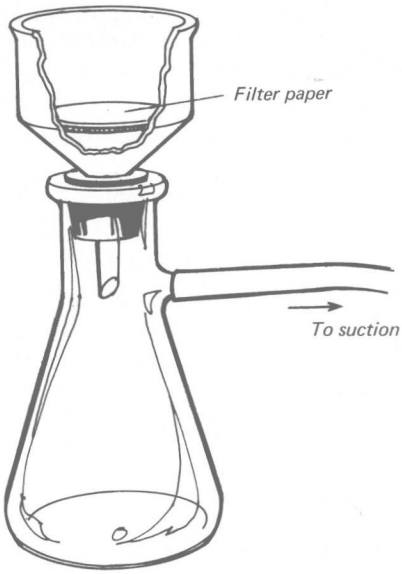


Figure 15 Using the Büchner funnel

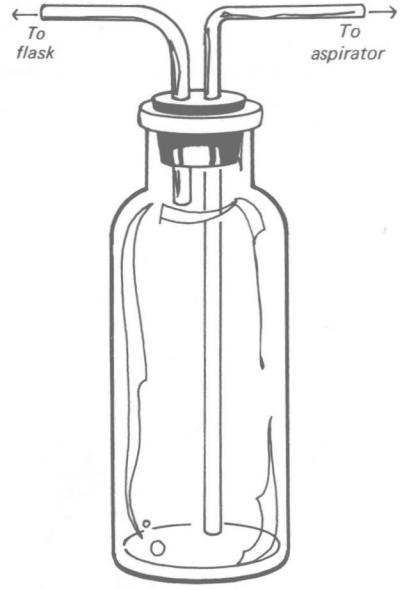


Figure 16 Safety trap for suction filtration

The only real trick to suction filtration is forming a seal between the filter paper and the bottom of the Büchner funnel. Test the seal by pouring a little solvent into the funnel under suction. If the seal is good, the liquid will quickly be sucked through the filter paper.