This section is intended as a reference for general information and specific techniques. Some of this material is already familiar to you (or should be) but in a few cases you will not have performed the actual technique in your first year of Chemistry due to time constraints. While not the most exciting reading, this segment of the lab text *is* important. Poor skills carried over from previous work will haunt a student. Therefore, even if you know it ALL, reading this will not kill you (whereas the consequences of *not* reading it cannot be assured).

CHEMICALS

Each experiment in this text includes information on the chemicals to be used, including relevant cautions. As a general rule you know to avoid contact with chemicals (other than water) as much as is possible. Washing your hands thoroughly immediately after accidental contact is the next best thing.

Your previous contact with organic reagents has been limited to a few simple hydrocarbons (e.g., hexane), some alcohols and acetone. Although every attempt has been made to select organic materials which are relatively benign, added caution is warranted when handling organic compounds. **Gloves are suggested on occasion in the text and you should not hesitate to ask for gloves whenever you feel it is in your best interest.** Many organic reagents have unpleasant odors and quite a few have high vapor pressures. Spills must be cleaned up immediately and wastes containing these compounds should always go in the containers provided in the fume hood, not into the general troughs.

You have worked with acids and bases before and know the general hazards associated with them. Even 6 M acid can leave holes in clothing and certainly will sting if splashed on the skin. A number of the experiments in this course involve the use of ***concentrated*** acids. That obvious term carries with it a great caution. Concentrated acids should not be carried out into the open lab, but confined to fume hoods. Transfers of these reagents should occur within the hood. All spills should be appropriately neutralized and cleaned up immediately, flushing any waste water down the hood sink with plenty of water.

Concentrated acids should NEVER be poured directly from a stock bottle into a reaction mixture or container holding any other substance, *including water*. The possible resulting reaction might startle you and cause you to drop the bottle. The amount you think is needed should be measured out first and then added cautiously from the graduated cylinder or beaker.

NEVER add water to concentrated acids! A highly exothermic reaction is likely to occur, perhaps with explosive violence. When adding acid to water, caution is the key, and plenty of stirring. The addition of concentrated sulfuric acid to water is particularly hazardous and is best done by freezing an appropriate amount of distilled water in ice cubes and adding the acid slowly to the ice cubes (with stirring, of course).

The only concentrated base you will use in the course is aqueous ammonia. In addition to its corrosive properties (which it shares with concentrated sodium hydroxide solutions) ammonia is hazardous due to the high partial pressure of gaseous ammonia over the solution. Concentrated ammonia solutions MUST be dispensed in the fume hood and solutions which are taken into the open lab should be covered.

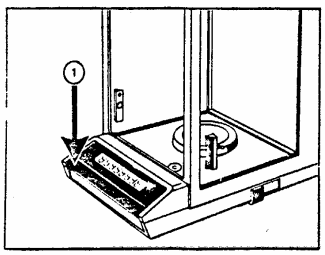
Adapted from: Chemical Principles in the Laboratory, 3rd ed., Robert F. Bryan, Robert S. Boikess
Experimental General Chemistry with Problems, James D. McCullough, Hosmer W. Stone

THE ANALYTICAL BALANCE

The maximum load to be placed on the analytical balance is 150 g.

OPERATION: How to switch the balance on, and how to tare

The balance is tared by pressing the single control bar; this bar also turns the display on and off. When switching the balance off by means of the control bar, only the display is turned off. The electronic components are on as long as the power cable is connected (standby). This allows the balance to be operational at all times and eliminates the need for a warm-up time.



Switching the balance on/off

Switching on:

- Briefly press control bar (1).

All display elements light up for several seconds:

8.8.8.8.8.8.8.

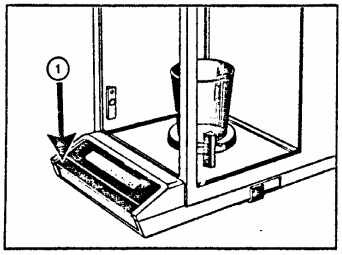
This permits a functional check of the display.

Then, 0.0000 is displayed.

Switching off:

- Briefly lift the control bar.

If the balance displays *OFF* the control bar must be pressed again briefly.



Taring

- Place a container on the pan.

Weight is displayed.

- Briefly press control bar (1).

Display is blanked out, then

0.0000 appears.

The container weight is now tared out.

The weighing range is now available for weighing-in, minus the tared-out container weight.

Some general comments

Analytical balances designed to mass to the nearest milligram or better are very sensitive instruments, costing several thousand dollars each. Treat the fine balances with the greatest respect, carefully following the operating directions and never forcing any of the controls. **Never add chemicals or reagents to any container at the fine balance.** Spills of such materials could find their way into the inner workings of the balance and lead to corrosion or mechanical damage.

The rough balances in the lab (top loading—***maximum capacity 400 g***) are designed to measure to the nearest 10 mg (0.01 g). These balances should be used when massing chemicals. If only an approximate mass is required, these balances will suffice. If greater precision is desired, measure out the approximate amount on the rough balance and then repeat the measurement on the fine balance.

The typical sequence for obtaining a chemical mass on the analytical balance would be to first measure the mass of an empty container on the balance. This container should then be placed on a rough balance and tared. The chemical is carefully added, approximating the desired mass as closely as possible. The container and chemical are then placed on the analytical balance for a final mass. The mass of the chemical on the analytical balance can be determined by difference.

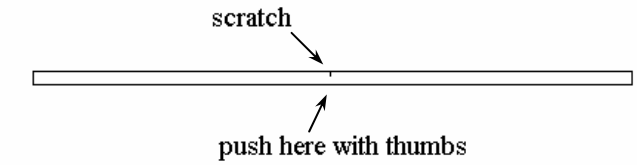
Working with glass tubing

Cutting tubing

Glass tubing of various sizes may be cut easily with a tubing cutter. The cutter consists of a diamond wheel at the bottom of a V-shaped channel. The tubing is seated in the channel and light pressure is applied as the tubing is turned or the cutter is moved. A small scratch results.

Alternatively, the tubing is laid flat on the bench and the edge of a triangular file is drawn across it to scratch where the tubing is to be cut.

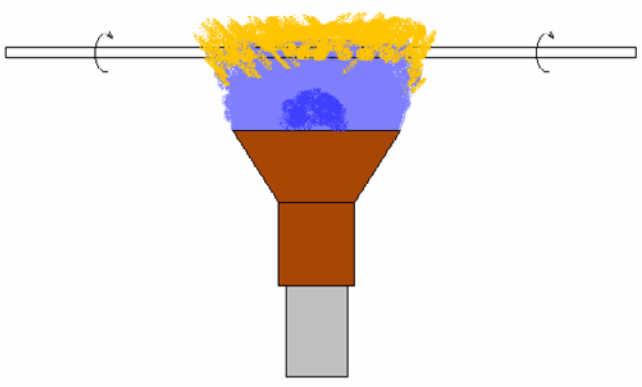
In either case, the tubing is then gripped in both hands, with the scratch pointing away from you and your thumbs directly behind it. Pushing with your thumbs should cause the tubing to break cleanly at the scratch:



If the glass does not break readily, repeat the scratching process. If the ends are jagged, discard the glass or try another break farther down the tubing. It is unusual for tubing to shatter during this procedure, but that's why accidents are called accidents. If you are nervous about the possibility, wrap the tubing loosely in a layer or two of paper toweling before attempting the break (but be sure the scratch and your thumbs are properly positioned!).

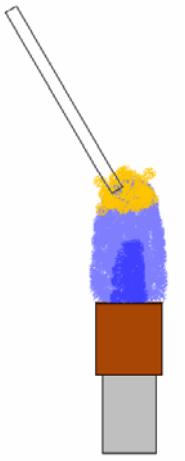
Bending tubing

Fit a wing-top attachment to your burner. This brass fitting is designed to spread out the flame (it is often called a "flame spreader") so that a longer segment of tubing can be heated. Light the burner and adjust for a flame about 3 cm high. The flame should be as hot as possible and remain stable. Grip one end of the tubing to be bent in each hand and place its center in the flame. The tubing should be parallel to the wing-top, not perpendicular. The object is to soften a significant length of the glass so that a smooth bend can be made. Heating only one spot will cause the tubing to pinch when it is bent. Holding the tubing at the very upper edge of the flame, twirl the tube around its long axis to heat it evenly. The glass should begin to color the flame due to the sodium ions in it:



Continue heating until the glass begins to soften (it will suddenly feel "sloppy" in your hands), remove it from the flame and quickly bring the ends together to obtain the angle you want. Set the tubing aside on a heat-resistant surface to cool. **Hot glass looks like cool glass.**

Fire-polishing



The tube ends produced by cutting should be clean, and they will also be SHARP. Before using the cut tubing, you must first "fire-polish" the sharp edges to round them off, reducing the risk of cuts and making it easier to fit the tubing into stoppers, etc. To do this place the end of the tube in the burner flame and rotate the tubing until the softening of the glass removes any sharp edge. Do not overheat the ends, as they may collapse. Set the tubing aside on a heat-resistant surface to cool. **Hot glass looks like cool glass.**

Inserting glass tubing into rubber stoppers

Incredibly, this may be the most hazardous procedure that you ever attempt in the lab! (I speak from personal experience....) Serious cuts may result if you are careless when inserting glass tubing, funnels, thermometers, etc. into rubber stoppers or tubing. Manufacturers generally make the holes in stoppers fairly small to accommodate many different diameters of tubing. However, it is rare that the holes are even close to the size you need.

Two methods are usually suggested. In both the stopper and glass are lubricated--in one case with water and in the other with glycerol. Holding the glass close to the point of insertion, and keeping the stopper between thumb and forefinger in the other hand, the glass is gently twisted and pressed through the hole. The secret of success is partly luck and holding everything so that in case of breakage, no glass ends up in one's hand. Lots of lubrication helps. Glycerol is water soluble and easy to remove.

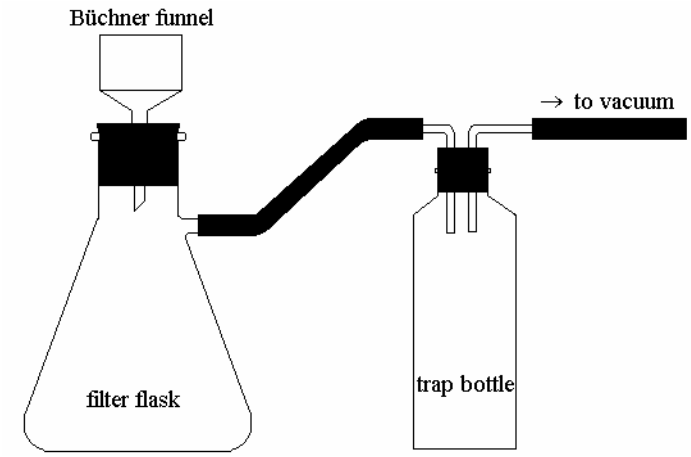
I have used a third method with some success (after a number of stitches...) owing to the elasticity of rubber and the difficulty of enlarging holes with a cork borer. Since cork borers were designed to cut cork, they do a poor job on rubber. However, they are good at temporarily stretching a hole. If a borer is chosen so that the glass to be inserted in the stopper just fits inside the borer, then the borer can be inserted in the stopper, the glass placed through the metal tube, and the borer carefully withdrawn, leaving the glass behind. It sounds a little strange, but is quite effective. The opposite procedure--though trickier--may be used to REMOVE glass from a stopper.

There are thus two things to consider when glass must be inserted into a rubber stopper: 1) if you are having trouble, DON'T FORCE glass, call the instructor; and 2) make sure that you only put together those things you really need. It is an art to master.

Oh, and **hot glass looks like cool glass**.

Suction Filtration

You are familiar with gravity filtration and what you probably remember most about it is that it is sometimes quite *slow*. The process can be hastened by the use of suction and a special funnel. A "trap" is placed between the vacuum source and the filter flask as shown below:



The Büchner funnel pictured above is specially designed for only one size of filter paper. The paper is laid into the funnel flat on a perforated surface, and the solvent is used to seat it, usually with suction applied. The entire apparatus should always be secured with clamps. For successful use, the following points should be observed:

1. the suction must be <u>on</u> before filtration begins
2. the filter paper must be seated and sealed and not allowed to dry out during filtering
3. liquids poured into the funnel should be run down a stirring rod to the center of the paper where they will not accidentally unseat the paper
4. if partial drying is desired, the suction may be left on for a time after filtering is complete
5. when not in use the vacuum valve should be closed to maximize the amount of vacuum available to others

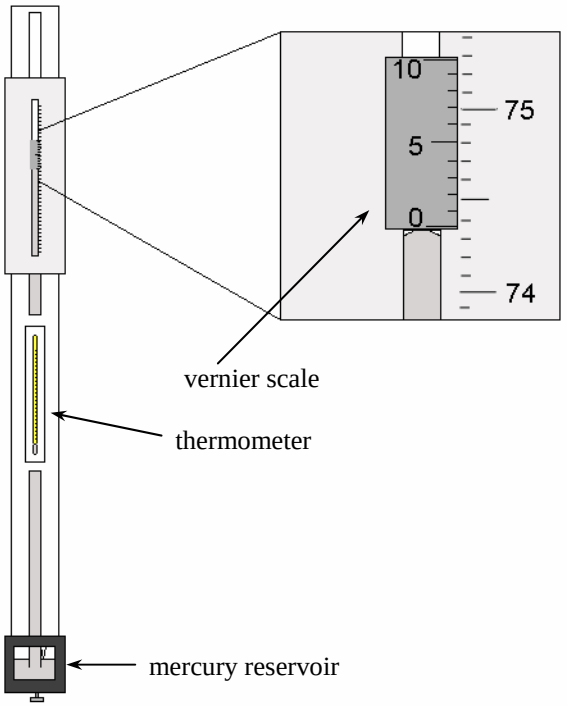
Burets and pipets

Both burets and volumetric pipets are familiar to you already. What may not be familiar is the careful use of both for accurate and precise quantitative work. Cleanliness of the glass is essential and is also a detail that is typically out of your control (i.e., the dirty glassware is spirited away and returns magically cleaned). Nonetheless, it is important to get into the habit of rinsing the inside of either device with a small amount of the solution that you are going to dispense [this should <u>not</u> be done from the original container---a small amount of the solution should be poured into a beaker and then into the buret, or drawn up into the pipet]. The rinse is discarded, allowing some to run through the tip.

Remember that air bubbles in buret tips will cause errors in the total volume measurement and that most of the pipets used in our lab are designated "TD", i.e., "<u>T</u>o <u>D</u>eliver". The tip of the pipet should be touched against the side of the container into which the solution is dispensed but the small residual amount of solution left in the pipet tip should <u>not</u> be blown, shaken or otherwise forced out.

The Barometer

A barometer is a special type of manometer, a device for measuring gas pressure. In a barometer, the force of the air pushing down on a reservoir of mercury supports a column of mercury in a long, evacuated glass tube, the open end of which is immersed in the mercury reservoir. The height of the mercury column is thus a measure of the force exerted by the atmosphere or "atmospheric pressure". It is usually measured in the convenient units of mm Hg (the height of the column itself), which are sometimes called *torrs* (after Torricelli).



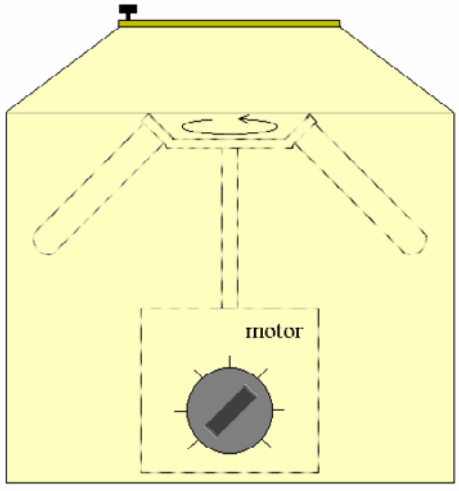
A stationary scale is attached to the glass tube which falls in the range of realistic atmospheric pressures. A sliding or *vernier* scale is attached to the fixed scale to facilitate precise reading of the fixed scale. Many mercury barometers also have a thermometer attached.

At the bottom of the barometer is the reservoir of mercury. This is open to the atmosphere via a small pinhole at the top of the protective metal casing. If you look into the reservoir chamber you will notice a small cone-shaped peg, made of ivory, which is used to "zero" or calibrate the barometer. The thumb screw on the reservoir chamber is used to adjust the level of the ivory peg until it just touches the surface of the mercury. At this point, the column is adjusted for whatever elevation the barometer may be at.

The air pressure may now be read by moving the sliding scale so that the bottom edge (0) coincides with the top of the mercury meniscus in the column (AT EYE-LEVEL!!). The pressure is read at the "0" mark on the vernier (sliding scale) from the fixed scale. This scale is calibrated in centimetres (who knows why!) but should be read in mm. This will give a three digit reading. On the diagram above this reading is 743 [**you should check to be sure you agree!**]. A reading in the tenths place may be obtained by examining the vernier (sliding) scale. Whichever line on *that* scale lines up exactly with a line on the *fixed* scale gives the decimal reading. Referring again to the diagram above, it looks as if the 4 (i.e., one tick mark below the printed 5) on the vernier scale matches the mark on the stationary scale. Thus the pressure to the nearest tenth is 743.4 mm Hg.

The centrifuge

The centrifuge is a device for quickly separating liquids from solids in the laboratory. It consists of two parts: the motor, which turns a shaft at high speed; and the head, which holds test tubes that contain the mixture to be separated.

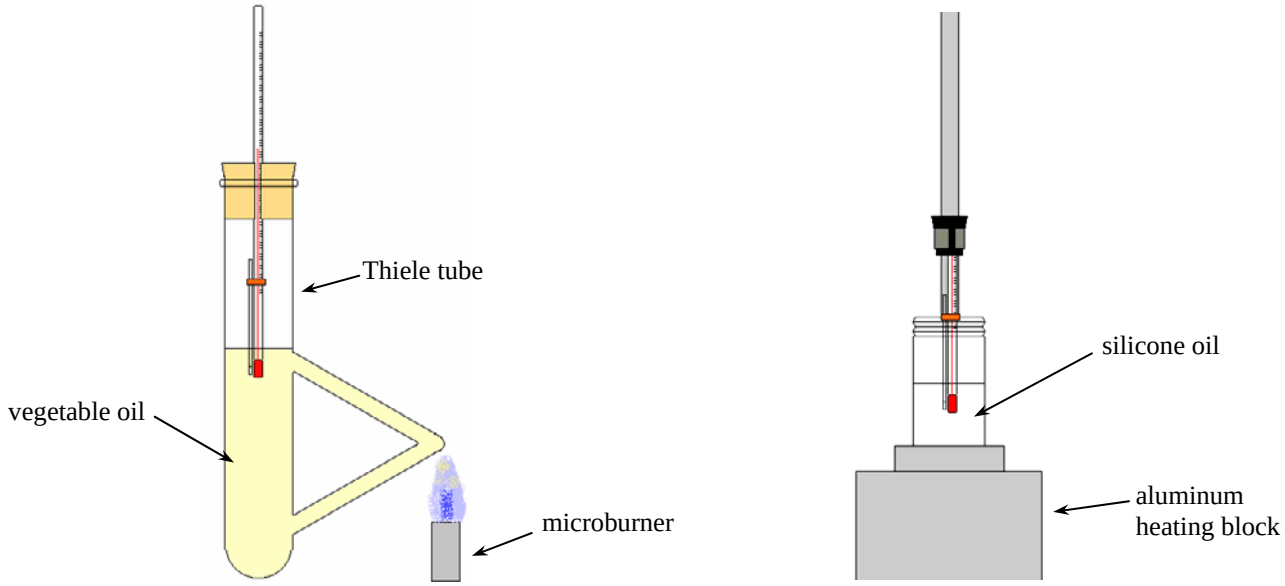


Although all centrifuges are very much alike, there are small differences with regards to starting, stopping and speed adjustment. The instructor will demonstrate these.

One rule is common to all machines: if you are doing an odd number of samples, you must place "balance tubes" filled with water opposite your samples in the centrifuge head. Otherwise the machine is off-balance and may be damaged. NEVER leave the centrifuge running and unattended. Even the best-balanced machines have a tendency to "walk" and may fall off counters. They are not inexpensive.

Melting point determination

The purity of low-melting solids is frequently checked in the lab by means of melting points. There are now machines (expensive machines) which do melting point determinations, but in the typical laboratory method a small amount of sample is packed in a capillary tube (closed at one end) and the tube is placed in an oil bath. The classic heating vessel is the Thiele tube, shown on the left below. The Micro-Combo Still™ equipment we use in other experiments can also be used to determine melting points (shown on the right).



The easiest way to fill the capillary is to place a bit of the sample on a watch glass and tap the open end of the tube into the solid a few times. The solid may be "packed" into the closed end by tapping the inverted tube briskly on a solid surface a few times. Particularly light solids may be packed by dropping the capillary through a long piece of glass tubing several times. There should not be more than 2-3 mm of compacted sample. The sample should be positioned so that it is against the thermometer bulb. The rubber band should be adjusted so that even when the warm oil has expanded, the band will not be immersed. The cork which holds the thermometer in place in the Thiele tube must have a channel cut in it. This serves two purposes: it "opens" the heated system (one should NEVER heat a closed system) and it allows reading the thermometer scale in the region of the cork.

Heat is applied to the Thiele tube with a microburner at the rate of about 2^{o}C per minute. In the alternate apparatus heat comes from a hot plate (setting #3) and is distributed via the aluminum heating block. A very small magnetic stirring "flea" is used to help circulate the oil in the vial. In the Thiele tube convection currents circulate the oil if the burner is positioned as shown in the diagram. The temperature at which the solid begins to melt is noted. Generally, a sample should not be frozen and re-melted.

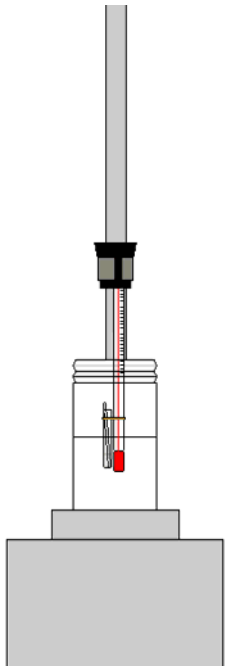
Obviously, one could spend a long time simply raising the temperature with a high-melting solid. If a high melting point is anticipated, it may be advantageous to prepare two samples and estimate the melting point quickly with one. The heating fluid is then allowed to cool 10-15^{o}C below the estimated melting point and the second tube inserted.

The observed melting point obtained in these ways is dependent on a number of factors: sample size, state of subdivision of the sample, heating rate, and the purity and identity of the sample. The first three cause the melting point to differ from the actual melting point due to a time lag in heat transfer. Also, if heating is too fast the thermometer will lag behind the actual temperature of the heating fluid. For accurate work the standard thermometers should be calibrated with a well-behaved solid of a known melting point.

Boiling point determination

It is possible to measure the boiling point of a small amount of liquid by the following method:

Place a small amount of the liquid (1 mL or less) into a micro test tube. Break a melting point capillary tube to obtain a segment slightly longer than the micro test tube. Insert the capillary (closed end up) into the liquid. Fasten the test tube to a thermometer with a rubber band as shown at the right and suspend the entire assembly in the Micro-Combo Still™ oil bath. Be sure the thermometer bulb is completely immersed. Stirring the oil bath with a magnetic "flea", set the hot plate on #3 and watch for bubbles to emerge from the submerged open end of the capillary. As soon as a steady stream of bubbles is forming, raise the assembly until the thermometer bulb and test tube are just above the oil.



Watch as the stream slows and finally stops. When liquid is just drawn into the capillary, record the temperature as the boiling point.

The method requires patience and practice. It is a good idea to check the boiling point of one or two known liquids. A second trial is advisable if time and sample amount permits, but you need a clean, dry capillary tube for each trial. Mixtures should not be reboiled as their compositions change during boiling.