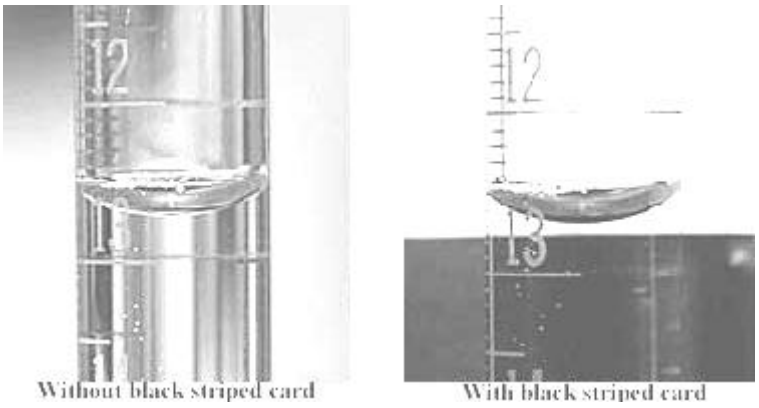
TOOLS OF VOLUMETRIC ANALYSIS

READING THE MENISCUS

Volumetric flasks, burets, pipets and graduated cylinders are calibrated to measure volumes of liquids. When a liquid is confined in a narrow tube such as a buret or a pipet, the surface is found to exhibit a marked curvature, called a **meniscus**. It is common practice to use the bottom of the meniscus in calibrating and using volumetric ware. Special care must be used in reading this meniscus. By positioning a black-striped white card behind the meniscus, which is transparent, becomes more distinct.



PROCEDURE

Location of the eyes in reading any graduated glassware is important.

1. With the eye above the meniscus, too small a volume is observed.
2. With the eye at the same level as the meniscus, the correct volume is observed.
3. With the eye below the meniscus, too large a volume is observed.

The eye must be level with the meniscus of the liquid to eliminate parallax errors. Read the top of the black part of the card with respect to the graduations on the buret.

TOOLS OF VOLUMETRIC ANALYSIS

Pipets, burets and volumetric flasks are standard volumetric equipment. Volumetric apparatus calibrated to contain a specified volume is designated **TC**, and apparatus calibrated to deliver a specified amount, **TD**.

CASE OF VOLUMETRIC EQUIPMENT

Only clean glass surfaces will support a uniform film of liquid; the presence of dirt or oil will tend to cause breaks in this film. The appearance of water breaks is a sure indication of an unclean surface. The manufacturer carefully cleans volumetric glassware before being supplied with markings, and in order for these to have meaning; the equipment must be kept equally clean when in use.

As a general rule, the heating of calibrated glass equipment should be avoided. Too rapid cooling can permanently distorted the glass and cause a change in volume.

VOLUMETRIC FLASKS

Volumetric flasks are calibrated to contain a specified volume when filled to line etched on the neck.

DIRECTIONS FOR THE USE OF A VOLUMETRIC FLASK

Before use, volumetric flasks should be washed with detergent and, if necessary, cleaning solution. Then they should be carefully and repeatedly rinsed in distilled water; only rarely need they be dried. Should drying be required, however, it is best accomplished by clamping the flasks in an inverted position and letting the flask air-dry.

VOLUMETRIC MEASUREMENT

Direct preparation of solution requires a known mass or exact volume of solute be introduced into a volumetric flask.

Fill the flask about half full with the solvent and introduce the solute as described above. Swirl to contents to achieve solution. Add more solvent and again mix well. Bring the liquid level almost to the mark, and allow time for drainage. Then use a medicine dropper to make such final addition of solvent as are necessary. Firmly stopper the flask and invert repeatedly to assure uniform mixing.

NOTES: **Start over should you overfill when completing the volume.**

PIPETS

Pipets are designed for the transfer of known volumes of liquid from one container to another. Pipets, which deliver a fixed volume, are called **volumetric or transfer** pipets. Other pipets, known as **measuring pipets**, are calibrated in convenient units so that any volume up to maximum capacity can be delivered.

DIRECTIONS FOR THE USE OF A PIPET

**** NEVER DRAW LIQUIDS INTO THE PIPET BY MOUTH, USE A PIPET PUMP ****

1. Clean pipet thoroughly with soap and rinse with distilled water.
2. Drain completely. Condition the pipet by rinsing three times with the solution to be measured.
3. Keep the tip of the pipet below the surface of the liquid.
4. Draw the liquid up beyond the calibration mark. Lift the pipet above the liquid and adjust to volume.
5. For **volumetric** pipets, transfer the pipet to the container to be used and **remove** the pipet pump from the pipet or use the dispensing bar. Allow the solution to drain completely. Remove the last drop by touching the wall of the container. The calibrated amount of liquid has been transferred. **DO NOT BLOW OUT THE PIPET!**
6. For **measuring** pipets, transfer the pipet to the container to be used. Using the pipet pump release the volume needed to transfer. If the measuring pipet is a **blowout** pipet, use the pipet pump to blow out the remaining drops. In case of color-coded measuring pipet, a frosted ring indicates complete blowout.

NOTES: Pipets should be thoroughly rinsed with distilled water after each use.

BURETS

Burets, like measuring pipets, deliver any volume up to their maximum capacity. Burets are designed to measure the volume of solutions dispensed consequently the calibration marks start at 0.00 mL and end at 50.00 mL. Fifty mL burets are calibrated so that measurements can be carried to 2 significant numbers after the decimal.

DIRECTIONS FOR THE USE OF A BURET

Before being placed in service, a buret must scrupulously clean. In addition, it must be established that the stopcock is liquid-tight.

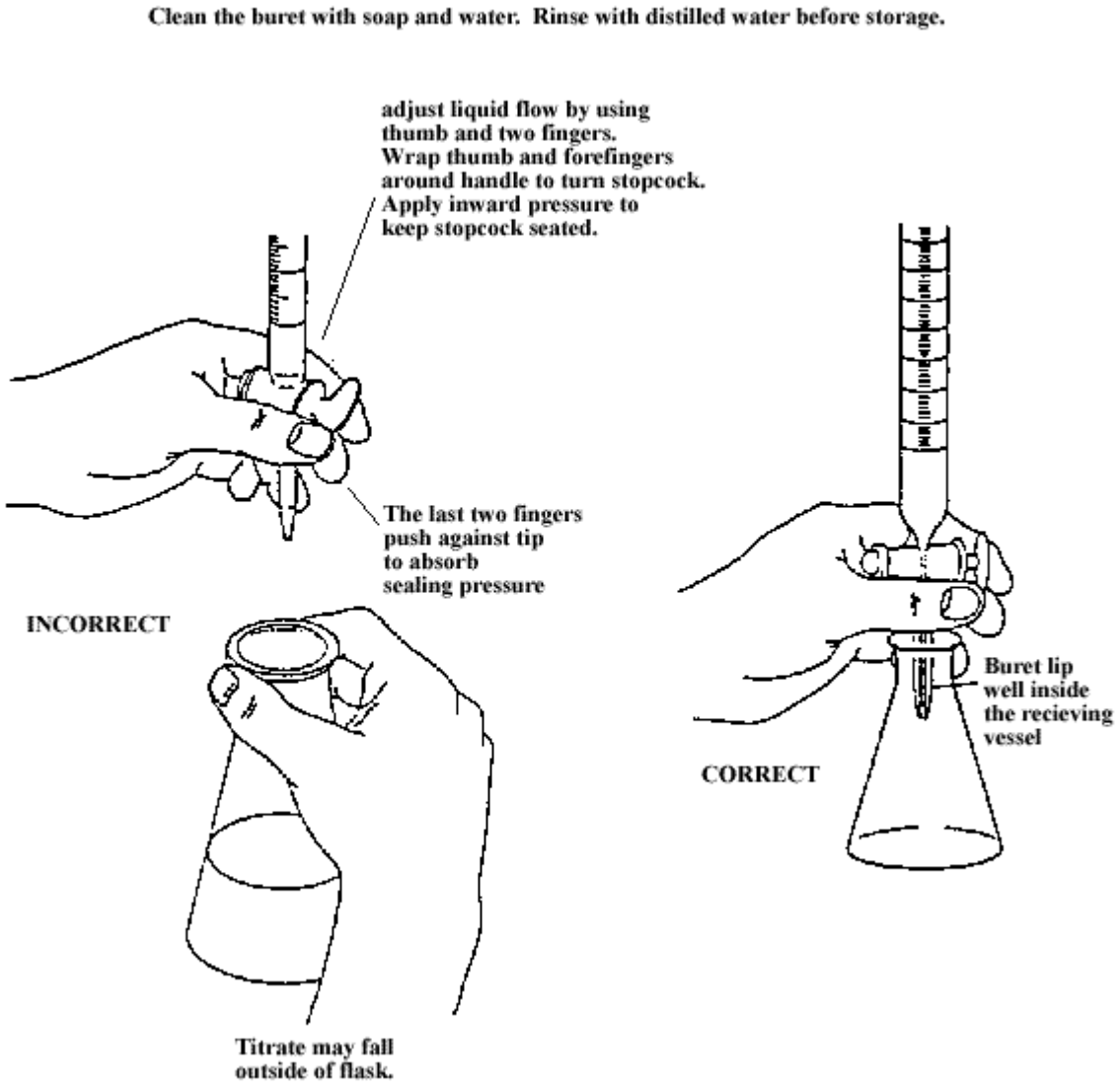
NOTE: When using the buret, **dispense the solution down from the 50.00 mL mark only**. Above the 50.00 mL mark does not have any measurement.

FILLING

Test the buret for cleanliness by clamping it in an upright position and allow it to drain. No water drops should adhere to the inner wall. If they do, clean the buret again.

Make certain that the stopcock is closed. Condition the pipet with 5 to 10 mL of solution by carefully rotating the buret to wet the wall completely; allow the liquid to drain through the tip. Repeat this procedure two more times. Then fill the buret above the zero mark. Free the tip of air bubbles by rapidly draining the solution through while gently tapping on the burette. Finally, lower the level of the solution to or somewhat below the zero mark; after allowing about a minute for drainage, take an initial volume reading. After dispensing the necessary volume from the buret take the final volume reading. The final volume minus the initial volume is the amount of solution dispensed from the buret.

Clean the buret with soup and water. Rinse with distilled water before storage.



HOLDING THE STOPCOCK

Always push the plug into the barrel while rotating the plug during a titration. A right-handed person points the handle of the stopcock to the right, operates the plug with the left hand and grasps the stopcock from the left side as shown.

GRAVITY FILTRATION

PREPARING FILTER PAPER FOR A FILTER FUNNEL

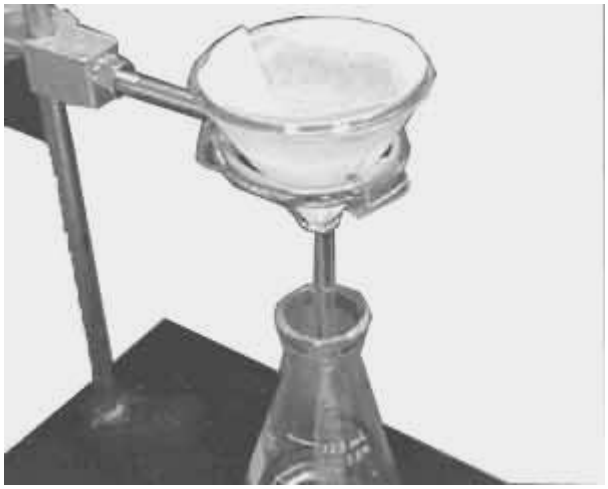
If the solid is separated from the liquid through a filtering process, then the filter paper must be properly prepared. For a gravity filtration procedure, first fold the filter paper in half, again fold the filter paper to within about 10° of a 90° fold, tear off the corner unequally, and open. The tear enables a close seal to be made across the paper's folded portion when placed in a funnel.

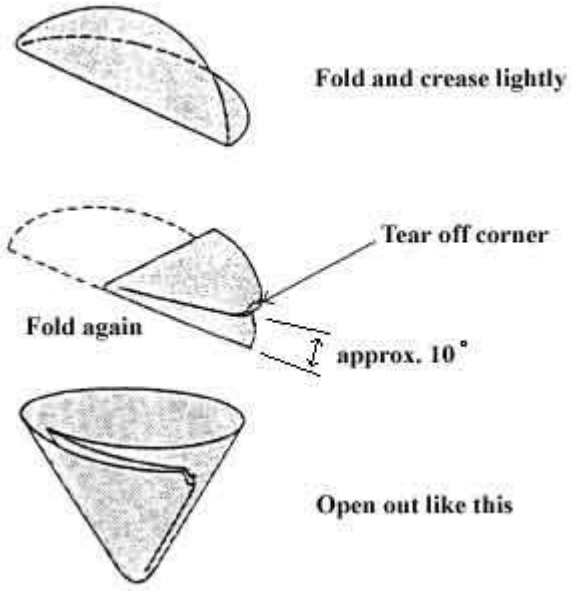
Place the folded filter paper snugly into the funnel. Moisten the filter paper with the solvent of the liquid/solid mixture being filtered (most likely this will be deionized water) and press the filter paper against the top wall of the funnel to form a seal. Support the funnel with a clamp or in a funnel rack.

TRANSFERING THE LIQUID

The tip of the funnel should touch the wall of the receiving beaker to reduce any splashing of the filtrate. Fill the bowl of the funnel until it is less than two-thirds full with the mixture. Always keep the funnel stem full with the filtrate; the weight of the filtrate creates a slight suction on the filter in the funnel, and this hastens the filtration process.

Flush a precipitate from a beaker with the mixture's solvent (usually deionized water) contained in a wash bottle, while holding the beaker over the funnel or receiving vessel.





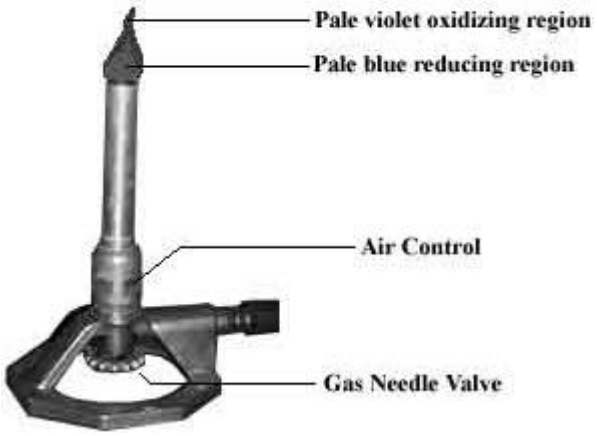
USE OF THE LABORATORY BURNER

NOTE: Before attempting to light a Bunsen burner, make sure that you are successful in generating sparks out of the striker. If you are not successful in getting the burner lit after two attempts, TURN OFF THE GAS FROM THE GAS JET.

Bunsen burners are located in the metal ware drawer. Always check the rubber tubing for holes. Most heating in your chemistry course is done with a gas burner. In this laboratory you will use a burner of the Bunsen type. The Bunsen burner has a gas needle valve at the base of the burner to control the amount of gas and in that way, the size of the flame. The burner also has an air inlet just above the gas inlet, which can be adjusted by screwing or unscrewing the barrel of the burner. This adjustment determines the amount of air mixes with the gas. The larger the air opening is the hotter the flame gets.

The fuel used for the burner is natural gas. You will find a natural gas jet at each work area. Always be sure the gas jet is shut off completely when the burner is not lit.

To light the burner, close the gas needle valve of the burner (turn counter clockwise). Adjust the barrel of the burner so that you see an air opening. Turn the gas jet 90^{o}. Adjust the gas needle valve of the burner clockwise until you hear the gas. Light the burner with a striker. Adjust the air control to get a blue, nearly transparent flame.



- If the air inlet is closed and the gas is lit, the flame will be large and luminous. The light is the radiation given off by the hot carbon particles that are burned only partially. This luminous flame is not very hot and **dangerously flimsy**. **This very cool flame type will never be used in this lab.**
- If the air control is adjusted so that air is mixed with the gas before it gets to the flame, the flame will become less luminous, and finally blue. When the air is adjusted correctly to give the hottest flame, it will look something as shown in the picture. The inner cone of the flame is pale blue, and the outer cone is pale violet. The inner cone contains the unburned gas that is hot enough to radiate light. The hottest point is just above the inner cone.