

# 1. The Use of Volumetric Glassware

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## Objective

Familiarity with the various instruments used for making physical measurements in the laboratory is essential to the study of experimental chemistry. In this experiment, you will investigate the uses and limits of the various types of volumetric glassware.

## Introduction

Most of the glassware in your laboratory locker has been marked by the manufacturer to indicate the volume contained by the glassware when filled to a certain level. The graduations etched or painted onto the glassware by the manufacturer differ greatly in the precision they indicate, depending on the type of glassware and its intended use. For example, beakers and Erlenmeyer flasks are marked with very approximate volumes, which serve merely as a rough guide to the volume of liquid in the container. Other pieces of glassware, notably burets, pipets, and graduated cylinders, are marked much more precisely by the manufacturer to indicate volumes. It is important to distinguish when a *precise* volume determination is necessary and appropriate for an experiment and when only a *rough* determination of volume is needed.

Glassware that is intended to contain or to deliver specific precise volumes is generally marked by the manufacturer with the letters “TC” (to contain) or “TD” (to deliver). For example, a flask that has been calibrated by the manufacturer to contain exactly 500 mL of liquid at 20°C would have the legend “TC 20 500 mL” stamped on the flask. A pipet that is intended to deliver a precise 10.00 mL sample of liquid at 20°C would be stamped with “TD 20 10 mL.” It is important not to confuse “TC” and “TD” glassware: such glassware may not be used interchangeably. The temperature (usually 20°C) is specified with volumetric glassware since the volume of a liquid changes with temperature, which causes the density of the liquid to change. While a given pipet will contain or deliver the same *volume* at any temperature, the *mass* (amount of substance present in that volume) will vary with temperature.

### A. Graduated Cylinders

The most common apparatus for routine determination of liquid volumes is the graduated cylinder. Although a graduated cylinder does not permit as precise a determination of volume as do other volumetric devices, for many applications the precision of the graduated cylinder is sufficient. Figures 1-1 and 1-2 show typical graduated cylinders. In Figure 1-2, notice the plastic safety ring, which helps to keep the graduated cylinder from breaking if it is tipped over. In Figure 1-1, compare the difference in graduations shown for the 10-mL and 100-mL cylinders. Examine the graduated cylinders in your lab locker, and determine the smallest graduation of volume that can be determined with each cylinder.

When water (or an aqueous solution) is contained in a narrow glass container such as a graduated cylinder, the liquid surface is not flat as might be ex-

FIGURE 1-1  
Expanded view of 10-mL and 100-mL cylinders. Greater precision is possible with the 10-mL cylinder, since each numbered scale division represents 1 mL.

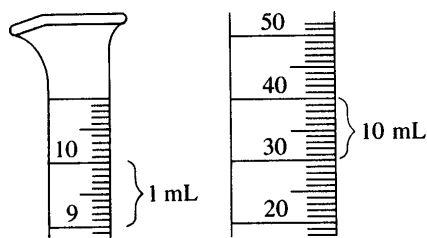
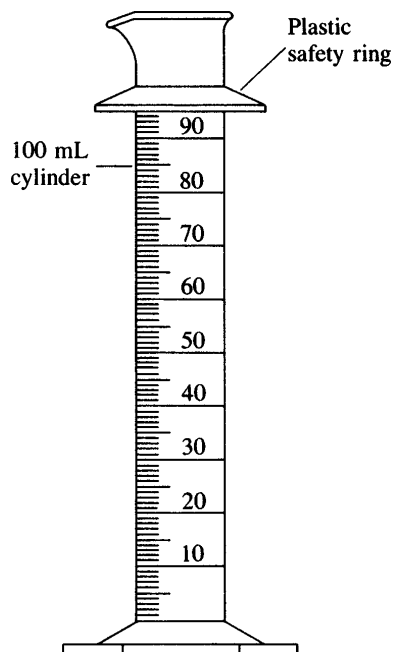
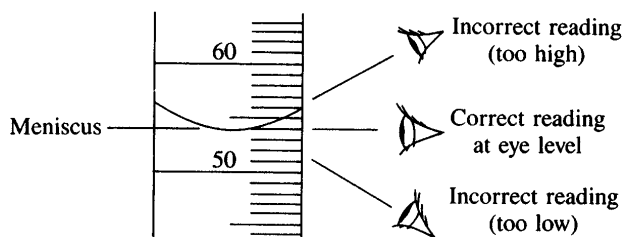


FIGURE 1-2  
A 100-mL graduated cylinder with a plastic safety ring.



pected. Rather, the liquid surface is curved downward (see Figure 1-3). This curved surface is called a **meniscus**, and is caused by an interaction between the water molecules and the molecules of the glass container wall. When reading the volume of a liquid that makes a meniscus, hold the graduated cylinder so that the meniscus is at eye level, and read the liquid level at the *bottom* of the curved surface.

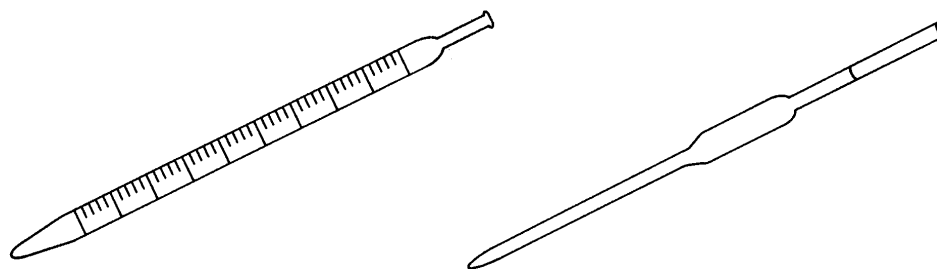
FIGURE 1-3  
Reading a meniscus. Read the bottom of the meniscus while holding at eye level.



## B. Pipets

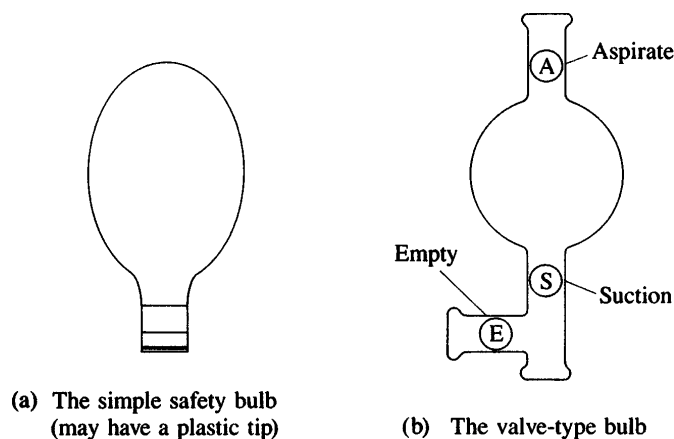
When a more precise determination of liquid volume is needed than can be provided by a graduated cylinder, a transfer pipet may be used. Pipets are especially useful if several measurements of the same volume are needed (such as in preparing similar-sized samples of a liquid substance). Two types of pipet are commonly available, as indicated in Figure 1-4. The Mohr pipet is calibrated at least at each milliliter and can be used to deliver any size sample (up to the capacity of the pipet). The volumetric transfer pipet can deliver only one size sample (as stamped on the barrel of the pipet), but generally it is easier to use and is more reproducible.

FIGURE 1-4  
A Mohr pipet  
(left) and a  
volumetric  
pipet (right).



Pipets are filled using a rubber safety bulb to supply the suction needed to draw liquid into the pipet. *It is absolutely forbidden to pipet by mouth in the chemistry laboratory.* Two common types of rubber safety bulb are shown in Figure 1-5.

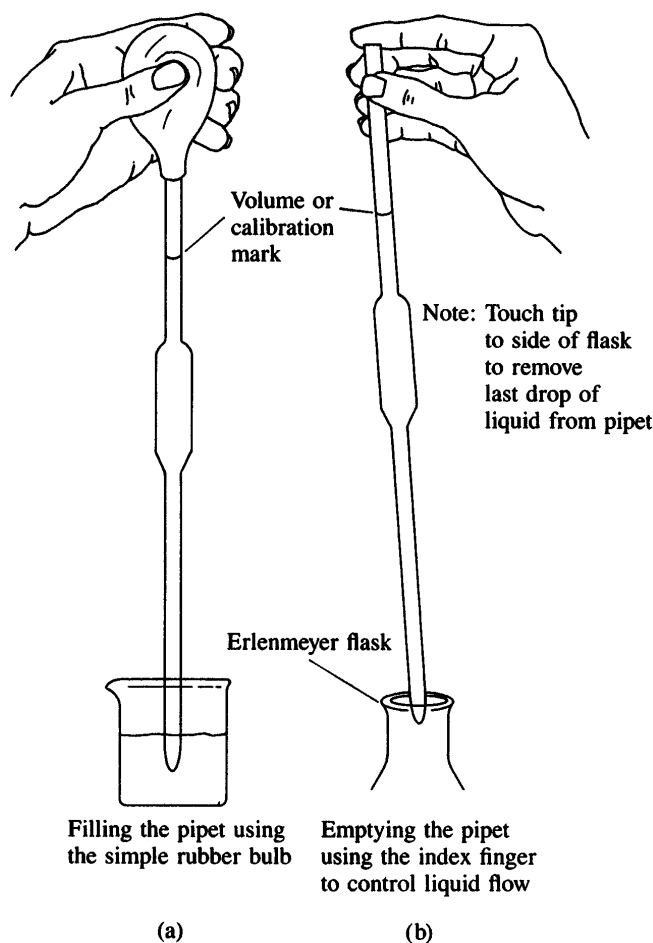
FIGURE 1-5  
Pipet safety  
bulbs. Never  
pipet by  
mouth.



The simple bulb should *not* actually be placed onto the barrel of the pipet. This would most likely cause the liquid being measured to be sucked into the bulb itself. Rather, squeeze the bulb, and just *gently* press the opening of the bulb against the opening in the barrel of the pipet to apply the suction force, keeping the tip of the pipet under the surface of the liquid being sampled. Do not force the pipet into the plastic tip of the safety bulb, or the pipet may break. Allow the suction to draw liquid into the pipet until the liquid level is 1 or 2 inches above the calibration mark on the barrel of the pipet. At this point, quickly place your index finger over the opening at the top of the pipet to prevent the liquid level from falling. By gently releasing the pressure of your index finger, the liquid level can be allowed to fall until it reaches the calibration

mark of the pipet. The tip of the pipet may then be inserted into the container that is to receive the sample and the pressure of the finger removed to allow the liquid to flow from the barrel of the pipet. (See Figure 1-6.)

FIGURE 1-6  
Filling  
technique for  
volumetric  
pipet.



To use the more expensive valve-type bulb (see Figure 1-5b), squeeze the valve of the bulb marked *A*, and simultaneously squeeze the large portion of the rubber bulb itself to expel air from the bulb. Press valve *A* a second time, release the pressure on the bulb, and attach the bulb to the top of the pipet. Insert the tip of the pipet under the surface of the liquid to be measured, and squeeze the valve marked *S* on the bulb, which will cause liquid to begin to be sucked into the pipet. When the liquid level has risen to an inch or two above the calibration mark of the pipet, stop squeezing valve *S* to stop the suction. Transfer the pipet to the vessel to receive the liquid, and press valve *E* to empty the pipet. The use of this sort of bulb generally requires considerable practice to develop proficiency.

When using either type of pipet, observe the following rules:

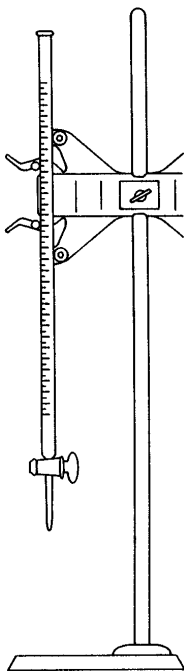
1. The pipet must be scrupulously clean before use: wash with soap and water, rinse with tap water, and then with distilled water. If the pipet is clean enough for use, water will not bead up anywhere on the inside of the barrel.

2. To remove rinse water from the pipet (to prevent dilution of the solution to be measured), rinse the pipet with several small portions of the solution to be measured, discarding the rinsings in a waste beaker for disposal. It is not necessary to completely fill the pipet with the solution for rinsing.
3. The tip of the pipet must be kept under the surface of the liquid being measured out during the entire time suction is being applied, or air will be sucked into the pipet.
4. Allow the pipet to drain for at least a minute when emptying to make certain the full capacity of the pipet has been delivered. Remove any droplets of liquid adhering to the tip of the pipet by touching the tip of the pipet to the side of the vessel that is receiving the sample.
5. If you are using the same pipet to measure out several different liquids, you should rinse the pipet with distilled water between liquids, and follow with a rinse of several small portions of the next liquid to be measured.

### C. Burets

When samples of various sizes must be dispensed or measured precisely, a buret may be used. The buret consists of a tall, narrow calibrated glass tube, fitted at the bottom with a valve for controlling the flow of liquid. The valve is more commonly called a **stopcock**. (See Figure 1-7.)

FIGURE 1-7  
A volumetric buret.  
Typically, 50-mL burets are used in chemistry labs.



Like a pipet, a buret must be scrupulously clean before use. The precision permitted in reading a buret is 0.02 mL, but if the buret is not completely clean, this level of precision is not attainable. To clean the buret, first use soap and water, using a special long-handled buret brush to scrub the interior of the glass. Then rinse the buret with tap water, followed by several rinsings with distilled water.

Before use, the buret should be rinsed with several small portions of the solution to be used in the buret. The buret should be tilted and rotated during the rinsings, to make sure that all rinse water is washed from it. Discard the rinsings. After use, the buret should again be rinsed with distilled water. Many of the reagent solutions used in burets may attack the glass of the buret if they are not removed. This would destroy the calibration. To speed up the cleaning of a buret in future experiments, the buret may be left filled with distilled water during storage between experiments (if your locker is large enough to permit this).

A common mistake made by beginning students is to fill the buret with the reagent solution to be dispensed to exactly the 0.00 mark. This is not necessary or desirable in most experiments, and wastes time. The buret should be filled to a level that is comfortable for you to read (based on your height). The precise initial liquid level reading of the buret should be taken before the solution is dispensed and again after the liquid is dispensed. The readings should be made to the nearest 0.02 mL. The volume of liquid dispensed is then obtained by simple subtraction of the two volume readings.

### **SAFETY PRECAUTIONS**

- **Wear safety glasses at all times while in the laboratory.**
- **When using a pipet, use a rubber safety bulb to apply the suction force. *Never pipet by mouth.***
- **Rinse the buret carefully. Do not attempt to admit water directly to the buret from the cold water tap. Fill a beaker with tap water, and pour from the beaker into the buret.**

### **Apparatus/Reagents Required**

Graduated cylinders, pipets and safety bulb, buret and clamp, beakers, distilled water

### **Procedure**

Record all data and observations directly in your notebook in ink.

#### ***A. The Graduated Cylinder***

Your instructor will set up a display of several graduated cylinders filled with different amounts of colored water. Several sizes of cylinder are available (10-mL, 25-mL, 50-mL, 100-mL). Examine each cylinder, paying particular attention to the marked scale divisions on the cylinder. For each graduated cylinder, to what fractional unit of volume does the smallest scale mark correspond?

Read the volume of liquid contained in each graduated cylinder and record. Make your readings to the level of precision permitted by each of the cylinders.

Check your readings of the liquid levels with the instructor before proceeding, and ask for assistance if your readings differ from those provided by the instructor.

Clean and wipe dry your 25-mL graduated cylinder and a 50-mL beaker (a rolled-up paper towel will enable you to dry the interior of the graduate). Weigh the graduated cylinder and beaker and record the mass of each to the nearest milligram (0.001 gram).

Obtain about 100 mL of distilled water in a clean Erlenmeyer flask. Determine and record the temperature of the distilled water.

Fill the graduated cylinder with distilled water so that the meniscus of the water level lines up with the 25-mL calibration mark of the cylinder. Place distilled water in the 50-mL beaker up to the 25-mL mark.

Weigh the graduated cylinder and the 50-mL beaker to the nearest milligram (0.001 gram) and calculate the mass of water each contains.

Using the Density of Water table from Appendix: Density of Water at Various Temperatures, calculate the volume of water present in the graduated cylinder and beaker from the exact mass of water present in each.

Compare the *calculated* volume of water (based on the mass of water) to the observed volumes of water determined from the calibration marks on the cylinder and beaker. Calculate the percentage difference between the calculated volume and the observed volume from the calibration marks. Why are the calibration marks on laboratory beakers taken only to be an approximate guide to volume?

## ***B. The Pipet***

Obtain a 25-mL pipet and rubber safety bulb. Clean the pipet with soap and water. Rinse the pipet with tap water, and then with small portions of distilled water. Practice filling and dispensing distilled water from the pipet until you feel comfortable with the technique. Ask your instructor for assistance if you have any difficulties in the manipulation.

Clean and wipe dry a 150-mL beaker. Weigh the beaker to the nearest milligram (0.001 gram) and record.

Obtain about 100 mL of distilled water in a clean Erlenmeyer flask. Determine and record the temperature of the water.

Pipet 25 mL of the distilled water from the flask into the clean beaker you have weighed. Reweigh the beaker containing the 25 mL of water. Determine the weight of water transferred by the pipet.

Using the Density of Water table from Appendix: Density of Water at Various Temperatures, calculate the *volume* of water transferred by the pipet from the *mass* of water transferred. Compare this calculated volume to the volume of the pipet as specified by the manufacturer. Any significant difference in these two volumes is an indication that you need additional practice in pipeting. Consult with your instructor for help.

How does the volume dispensed by the pipet compare to the volumes as determined in Part A using a graduated cylinder or beaker?

### ***C. The Buret***

Obtain a buret and set it up in a clamp on your lab bench.

Fill the buret with tap water, and check to make sure that there are no leaks from the stopcock before proceeding. If the stopcock leaks, have the instructor examine the stopcock to make sure that all the appropriate washers are present. If the stopcock cannot be made leakproof, replace the buret.

Clean the buret with soap and water, using a long-handled buret brush to scrub the inner surface of the buret. Rinse all soap from the buret with tap water, being sure to flush water through the stopcock as well. Rinse the buret with several small portions of distilled water, then fill the buret to above the zero mark with distilled water.

Open the stopcock of the buret and allow the distilled water to run from the buret into a beaker or flask. Examine the buret while the water is running from it. If the buret is clean enough for use, water will flow in sheets down the inside surface of the buret without beading up anywhere. If the buret is not clean, repeat the scrubbing with soap and water.

Once the buret is clean, refill it with distilled water to a point somewhat below the zero mark. Determine the precise liquid level in the buret to the nearest 0.02 mL.

With a paper towel, clean and wipe dry a 150-mL beaker. Weigh the beaker to the nearest milligram (0.001 g).

Place the weighed beaker beneath the stopcock of the buret. Open the stopcock of the buret and run water into the beaker until approximately 25 mL of water have been dispensed. Determine the precise liquid level in the buret to the nearest 0.02 mL. Calculate the volume of water that has been dispensed from the buret by subtraction of the two liquid levels.

Reweigh the beaker containing the water dispensed from the buret to the nearest milligram, and determine the mass of water transferred to the beaker from the buret.

Use the Density of Water table from Appendix: Density of Water at Various Temperatures to calculate the volume of water transferred from the mass of the water. Compare the volume of water transferred (as determined by reading the buret) with the calculated volume of water (from the mass determinations). If there is any significant difference between the two volumes, most likely you need additional practice in the operation and reading of the buret.

How does the volume dispensed by the buret compare to the volumes as determined in Part A using a graduated cylinder or beaker? How does the volume dispensed by the buret compare to that dispensed using a pipet in Part B?

# *The Use of Volumetric Glassware*

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Date: ..... Student name: .....  
Course: ..... Team members: .....  
Section: .....  
Instructor: .....

## **Prelaboratory Questions**

1. Pipets used for the transfer of samples of aqueous solutions are always *rinsed* with a small portion of the solution to be used before the sample is taken. Calculate the percentage error arising in an experiment if 1-mL, 5-mL, and 10-mL pipets are used for transfer and each pipet contains 5 drops of water adhering to the inside of the barrel. A single drop of water has an approximate volume of 0.05 mL.
  
  
  
  
  
  
  
  
  
  
2. It is important to make certain that there is no air bubble in the tip of the buret below the stopcock *before* the initial reading of the liquid level in the buret is taken. If a 0.5-mL air bubble is present in the tip of a buret, what percent error in 10-mL, 20-mL, and 40-mL samples will result if the air bubble is dislodged during the dispensing of the samples?



# The Use of Volumetric Glassware

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Date: ..... Student name: .....  
Course: ..... Team members: .....  
Section: .....  
Instructor: .....

## Results/Observations

### A. The Graduated Cylinder

Identifying color of cylinder water	Volume contained in cylinder
.....	.....
.....	.....
.....	.....

Mass of *empty* 25-mL graduated cylinder .....

Mass of cylinder plus water sample .....

Mass of water in cylinder .....

Temperature of water sample .....

Density of water at this temperature .....

Calculated volume of water transferred .....

Difference between observed volume and calculated volume .....

Mass of empty 50-mL beaker .....

Mass of beaker plus water sample .....

Mass of water in beaker .....

Temperature of water sample .....

Density of water at this temperature .....

Calculated volume of water transferred .....

Difference between observed volume and calculated volume .....

### **B. The Pipet**

Mass of empty 150-mL beaker .....

Mass of beaker plus water sample .....

Mass of water transferred .....

Temperature of water sample .....

Density of water at this temperature .....

Calculated volume of water transferred .....

Difference between actual and calculated volumes transferred .....

### **C. The Buret**

Initial liquid level in buret .....

Final liquid level in buret .....

Volume of liquid transferred .....

Mass of empty beaker .....

Mass of beaker plus water .....

Mass of water transferred .....

Temperature of water .....

Density at this temperature .....

Calculated volume of water transferred .....

Difference between actual and calculated volumes transferred .....

### **Question**

Based on your experience in this experiment, briefly discuss the relative precision permitted by a graduated cylinder, a pipet, and a buret. Give several circumstances under which you would use each instrument, in preference to the other two.