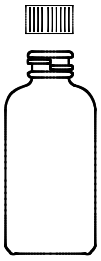
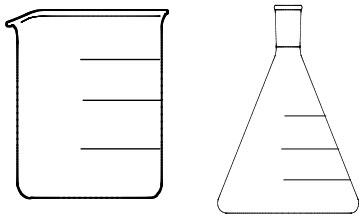
Measuring Volume

Chemists use a variety of glassware to measure a reagent's volume. The specific type of glassware used in any situation depends on how accurately or precisely you need to know the volume. In general, glassware is divided into two broad categories: glassware for approximate measurements and glassware for accurate and precise measurements.

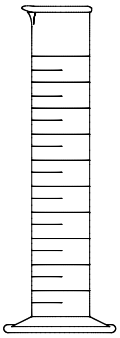
Glassware for Approximate Measurements



Five common types of glassware are used to make approximate measurements of volume: reagent bottles, beakers, Erlenmeyer flasks, graduated cylinders, and disposable pipets. A reagent bottles (shown to the right) is the least accurate as it seldom has any marks to indicate approximate volume. Adding 0.1 moles of a reagent to a 1-L bottle and adding water to the top of the bottle's rounded shoulder produces a solution that is approximately 0.1 M.



Beakers and Erlenmeyer flasks (shown to the left) usually have several graduation marks on their sides. These marks are accurate to within ±10% of the flask's maximum volume. For example, adding water to the 100 mL mark on a 250-mL beaker gives a net volume between 75 mL and 125 mL.

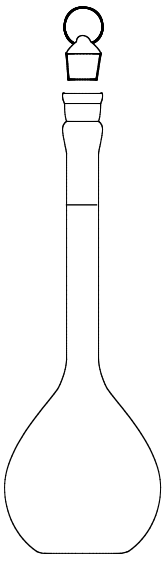


A graduated cylinder (shown to the right) provides a more accurate measure of volume than does a beaker or an Erlenmeyer flask. A typical graduated cylinder is accurate to within ±5% of the cylinder's maximum volume. When delivering 5 mL using a 10-mL graduated cylinder, for example, the actual volume is probably between 4.5 mL and 5.5 mL.

A disposable pipet (not shown) is a useful way to add a reagent whose volume is given in drops. A common estimate is that 20 drops is approximately equivalent to 1 mL.

In general, the precision for these types of glassware is better than their respective accuracies, although their precision seldom is an issue.

Glassware for Accurate and Precise Measurements



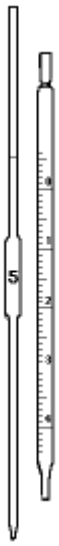
Sometimes we need to be know a reagent's exact volume. When this is the case we worry both about the volume's accuracy (How close is it to 10 mL?) and its precision (How much variation might we expect from one aliquot to the next?). Three types of glassware are commonly used when we need an accurate and a precise measurement of volume: volumetric flasks, volumetric pipets, and burets. In general, the precision of these types of glassware is better than their respective accuracies.

Volumetric Flasks. When filled to its calibration mark, a volumetric flask (shown to the right) contains a specified volume of solution, usually to within ±0.03-0.2% of the stated value, depending on the size of the volumetric flask (although accuracy can be improved through calibration, typically by determining the mass of water contained within the flask and converting to volume using water's known temperature-dependent density). A volumetric flask with a capacity of less than 100 mL generally measures volume to the hundredths of a milliliter, whereas volumetric flasks of 100 mL or greater capacity measure volumes to the tenth of a milliliter. For example, a 10-mL volumetric flask contains 10.00 mL, but a 250-mL volumetric flask holds 250.0 mL. This is an important issue to consider when keeping track of significant figures.

Note the use of the verb contain in describing a volumetric flask's properties; this description is important. Although a 100-mL volumetric flask contains exactly 100.0 mL (±0.1 mL), it cannot deliver 100.0 mL to another container. This apparent contradiction results from the fact that you can never completely transfer a liquid from one container to another; some liquid, even if it is only a few drops, remains behind.

Because a volumetric flask contains a solution of known volume, it is useful when preparing a solution whose exact concentration must be known accurately. A known amount of reagent is transferred to a clean volumetric flask and enough solvent added to dissolve the reagent. After the reagent is dissolved, additional solvent is added in several portions, mixing the solution after each addition. The final adjustment of volume to the flask's calibration mark is made dropwise using a disposable pipet or a solvent dispensing bottle. To complete the mixing process, the volumetric flask is inverted and shaken at least 10 times.

Volumetric Pipets. A volumetric pipet delivers a specified volume of solution. Several styles of volumetric pipets are available (see pictures to the right), but the most common and the most accurate is a transfer pipet. Transfer pipets consist of a long tube with a bulge in the middle and a single calibration mark (the pipet on the left, for example, is a 5-mL transfer pipet). A transfer pipet's accuracy is similar to that of a volumetric flask of equal volume; thus, for example, a 100-mL transfer pipet will deliver 100.0 mL of solution (±0.1 mL). As with a volumetric flask, accuracy is improved by calibrating with water. The other common type of volumetric pipet is a Mohr pipet, which consists of narrow tube with multiple calibration marks (the pipet on the right, for example, is a 5-mL Mohr pipet). The multiple calibration marks allow you to dispense volumes of variable size; thus a 5-mL Mohr pipet is used to deliver any specific volume between 0 mL and 5 mL. In this lab we will make exclusive use of transfer pipets.



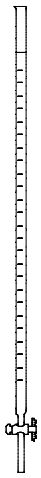
Note that a volumetric transfer pipet delivers a known volume of solution, whereas a volumetric flask contains a known volume. A transfer pipet always contains a volume greater than that delivered. When delivery is complete a small, residual amount of solution always remains behind. A transfer pipet, therefore, is always contaminated with a small amount of the last solution for which it was used.

Because a transfer pipet delivers a known volume of solution, it is an excellent way to deliver an accurate and a precise amount of reagent. To use a transfer pipet, first rinse it with deionized water to remove any traces of the last solution remaining in the pipet. Then, since water is, itself, a contaminant (it will dilute your solution), fill the pipet once with your solution and dispense it to waste. If you have a limited amount of your solution you can partially fill the pipet, seal the top and bottom and rock it back and forth to rinse the pipet's inner surfaces. Any residual amount of solution remaining in the pipet is similar enough in composition to your original solution such that dilution errors are inconsequential.

To fill a transfer pipet, use suction from a rubber bulb to pull the solution above the pipet's calibration mark (never use your mouth to suck a solution into a pipet). Remove the suction bulb and place your fingertip over the top of the pipet. While slowly twisting the pipet, allow the solution's level to drop until it reaches the calibration mark. Wipe the outside of the pipet to ensure it is dry and to remove any drop of solution clinging to the pipet's tip. Place the pipet over the container in which the solution is to be dispensed. Remove your fingertip and allow the pipet's contents to drain into the container. Touch the tip of the pipet to the container's wall to ensure the

final drop is dispensed. A small, residual amount of solution will remain in the pipet; do not try to force this into the container. Practice this technique until you confidently can use the pipet.

Burets. A buret is used to deliver a variable volume of solution. As shown in the figure on the right, a buret is a long, narrow tube with graduated markings and a stopcock on its bottom end. The topmost mark is labeled with a volume of zero, with additional markings increasing in volume as they go down the buret. The most commonly buret in the laboratory is a 50-mL buret, which has major markings for every 1-mL increment and minor markings for every 0.1-mL increment. The accuracy of a buret is approximately 0.1% of its total volume; thus, for a 50-mL buret, volumes are accurate to approximately ±0.05 mL.



To use a buret, fill it with solution to a point well above the mark for zero volume. Open the stopcock and allow the solution to flow until the portion of the buret below the stopcock is filled (without any air bubbles) and the volume is below the zero mark. Record this volume as the initial volume. Place a receiving flask below the buret and open the stopcock until the desired amount of solution has been dispensed. Record the final volume and determine the total volume delivered by difference.

Additional Important Details. Two important precautions are needed when working with volumetric pipets, volumetric flasks, and burets. First, the volume delivered by a volumetric pipet assumes the glassware is clean. Dirt and grease on a volumetric pipet's inner surface prevents liquids from draining evenly, leaving droplets of the liquid on the pipet's walls and resulting in the delivery of less liquid. For a volumetric flask, drops of liquid above the calibration mark mean the flask contains more than its specified volume.

Second, when filling a pipet or volumetric flask, set the liquid's level exactly at the calibration mark. The liquid's top surface is curved into a meniscus, the bottom of which should be exactly even with the glassware's calibration mark. To avoid parallax errors, the meniscus is adjusted with your eye at the same level as the calibration mark. If your line of sight is from above the calibration mark, for example, then you will tend to overfill the volumetric pipet or volumetric flask. When using a buret, the recorded volumes also are read using the solution's meniscus.