The Art of Measuring Volume

Chemists use glassware to measure a reagent's volume with the choice of glassware determined by the required accuracy and precision. In general, we divide glassware 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 bottle* 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.
- A *beaker* or an *Erlenmeyer flask* has several graduation marks on its side. These marks are accurate to within approximately ±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* provides a more accurate measurement of volume than 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* 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, although this varies greatly from brand-to-brand.

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 accuracy (How close is it to 10 mL?) and precision (How much variation might we expect from one aliquot to the next?). Three types of glassware are common options 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 Flask. When filled to its calibration mark, a volumetric flask contains a specified volume of solution, usually to within $\pm 0.03 - 0.2\%$ of the stated value, depending on the size of the volumetric flask (although accuracy is improved by determining the mass of water contained within the flask and converting to volume using water's known temperature-dependent density, a process called a calibration). A volumetric flask with a capacity of less than 100 mL generally measures volume to the hundredths of a milliliter, whereas a volumetric flask of 100 mL or greater capacity measure volume to the tenth of a milliliter. For example, a 10-mL volumetric flask contains 10.00 mL, but a 250-mL volumetric flask contains 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 because 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 you need to prepare a solution with an exact concentration. 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 capped, and then 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, 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. 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 is a narrow tube with multiple calibration marks that allows 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 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, if necessary, remove any solution that clings 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 a tube with graduated markings and a stopcock on its bottom end. Although a volumetric pipet can deliver only the one specific volume for which it is designed, a buret can deliver any volume up to its maximum capacity. The accuracy of the burets available to you is approximately ±0.05 mL.

To use a buret, fill it with your solution and fill the buret's tip by briefly opening the stopcock. Place a receiving flask below the buret, open the stopcock until the desired amount of solution is dispensed, and record the volume delivered.

Additional Important Details

Two important precautions are needed when you work with volumetric pipets, volumetric flasks, and burets. First, the volume delivered by a volumetric pipet assumes the glassware is clean. Dirt and grease on the inner surface of a volumetric pipet will prevent it from draining evenly, leaving drops of the reagent on the pipet's walls and delivering less reagent than expected. For a volumetric flask, if drops of reagent remain above the calibration mark, then the flask will contain more than its specified volume.

Second, when you fill a pipet or a 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.