

Calibration of Glassware

Introduction

Glassware is commonly calibrated using a liquid of known, specific density, and an analytical balance. The procedure is to determine the mass of liquid the glassware will hold, and to divide this mass of liquid by the density of the liquid, obtaining the corresponding volume of liquid. Density is affected by temperature, so it is necessary to measure the liquid temperature and look up appropriate density values.

While the above procedure will suffice for items calibrated “TC” (to contain), it is not useful for those items calibrated “TD” (to deliver). For these items (such as pipets), the proper procedure is to fill the item with the appropriate volume of liquid, and then dispense the liquid into a previously weighed container of suitable size. The mass of the dispensed liquid is determined, and the volume calculated as before.

Note to student: before starting this experiment, read through the procedures below. This will give you a good overview of the experiment, and will allow you to plan your work.

Volumetric flasks. Figure 1 is a sketch of a typical volumetric flask.

1. Clean and rinse 3 volumetric flasks of appropriate size (25.00 or 50.00 mL). Rinse the flasks 2 or 3 times with small (~2 mL) portions of acetone, making sure that you cover all inside surfaces with the acetone rinses, and discard these rinses. Allow the residual acetone to evaporate. Clearly label each flask, using the white space on the flask and a pencil.

Note to student: this is potentially the slowest step in the experiment. Depending on the ambient temperature in the lab, and the temperature of the acetone, it may take several minutes for all of the acetone to evaporate from the volumetric flask. Be patient. You may want to calibrate pipets or your buret (see procedures below) while waiting for the volumetric flasks to dry.

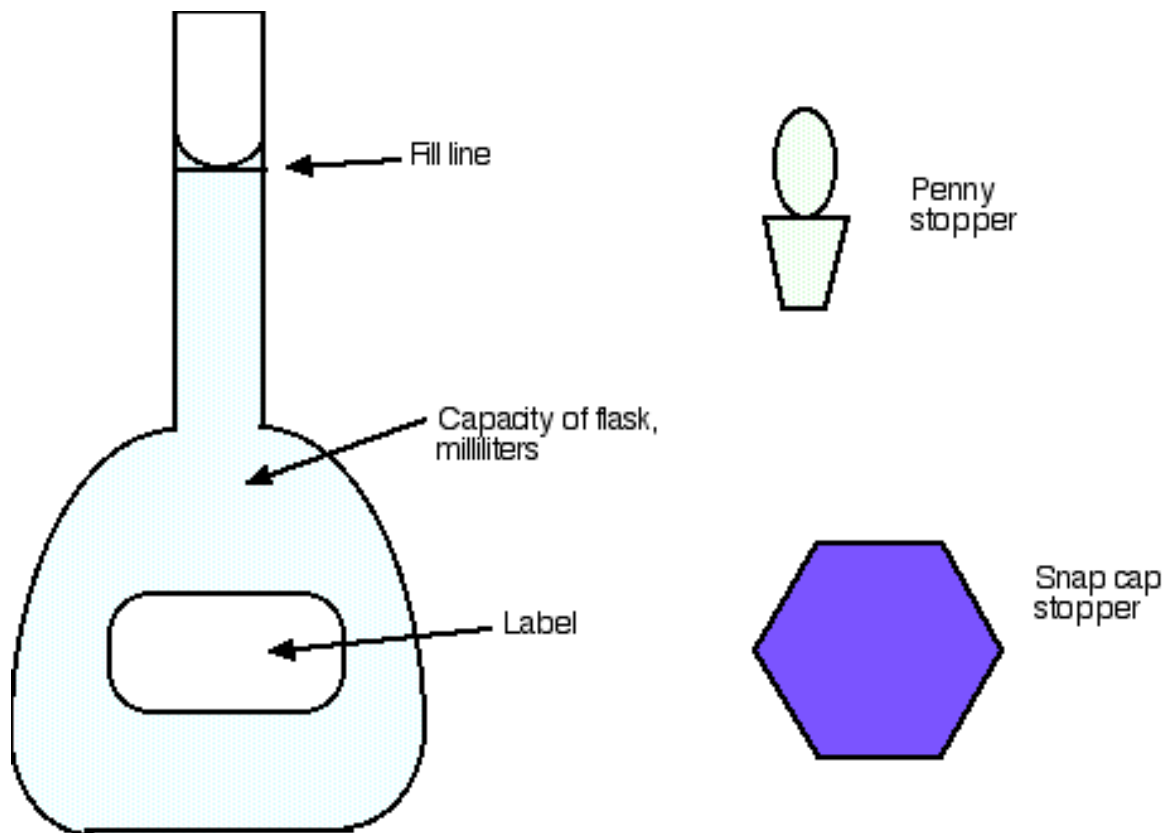


Figure 1. Volumetric flask. Typical stoppers are either the penny stopper or the plastic snap cap stopper. Penny stoppers fit flasks having ground glass necks. Snap cap stoppers fit flasks without ground glass necks.

2. After the analytical balance has been leveled and calibrated, tare the balance. Determine the mass of each flask and its stopper, and record these masses in your notebook. Make sure that you tare the balance before massing each flask.

Note to student: strictly speaking, we never weigh substances in the laboratory, for the simple reason that we are not interested in their weight. Instead, we determine the mass of a substance. If you don't have a problem with the sentence "I weighed the penny to determine its weight", then you shouldn't have a problem with "I massed the penny to determine its mass."

3. Dispense a suitable volume of deionized water into a beaker, and measure and record the temperature of the deionized water. Use the table in your textbook and the water temperature to determine the correct density of water. Fill each of the flasks with deionized water to the fill line (see Figure 1). Mass the filled flasks, making sure to record the masses of the filled flasks in your notebook.

- Return the deionized water to your beaker. Rinse the flasks 2 or 3 times with acetone as described in step 1. When the residual acetone has evaporated, repeat steps 2 and 3 until you have made triplicate measurements of the mass of water contained by each of the three flasks.
- Calculate the corresponding volumes of water contained by each of the flasks, and *calculate the mean and sample deviation for each flask*. When you are done, you should have a data table similar to that shown below.

Flask 1	1 st	2 nd	3 rd
Tare mass, g	24.3398	24.3398	24.3398
Filled mass, g	74.3634	74.3630	74.3627
Mass of water, g	50.0236	50.0232	50.0229
Volume water, mL	50.0386	50.0382	50.0379

Pipets. Figure 2 is a sketch of a typical pipet. Your instructor will demonstrate the correct procedure for filling and emptying a pipet.

- Clean and rinse 3 pipets (10.00 or 25.00 mL). There is NO need to rinse the pipets with acetone. Label the pipets so that you can tell them apart.

Note to student: pipets generally don't have a labeling space engraved on the pipet. Almost any kind of commercially available gummed label can be affixed to the pipet.

- Measure the mass of a suitably sized beaker (~50 mL) on the analytical balance, and record the mass in your notebook.
- Rinse the first pipet 3 times with deionized water by filling the pipet to the fill line, and discarding the rinse. Finally, fill the pipet to the fill line, wipe excess water from the outside of the pipet with a Kimwipe® and dispense this liquid into the beaker. Measure and record the mass of beaker and water, discard the water, and dry the beaker. Repeat this procedure until you have triplicate measurements made for each pipet. The mass of water dispensed from the pipet is found by subtracting the empty weight of the beaker from the weight of the beaker and water.
- Calculate the corresponding volume of water delivered by each pipet, and *calculate the mean and sample deviation for each pipet*.

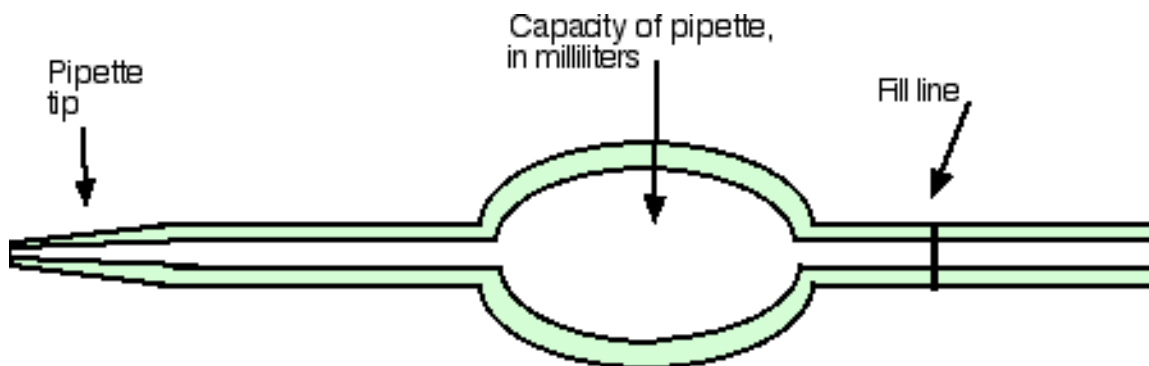


Figure 2. Typical pipet.

When you are done, you should have a data table for each pipet similar to the table shown below.

Pipet 1	1 st	2 nd	3 rd
Mass of water, g	10.0119	10.0087	10.0128
Volume, mL	10.0149	10.0117	10.0158

Buret. Figure 3 is a sketch of a typical buret. Your instructor will demonstrate the proper procedure for filling the buret, and dispensing liquid from the buret.

1. Select a suitably sized buret (25.00 or 50.00 mL) and carefully clean and rinse the buret with deionized water. There is NO need to rinse the buret with acetone.
2. Determine the empty mass of a suitably sized beaker on the analytical balance. Record this mass in your notebook.
3. Obtain a volume of deionized water (100 - 200 mL), and measure the temperature of the water. Fill the buret with deionized water to the 0.00 mL line.

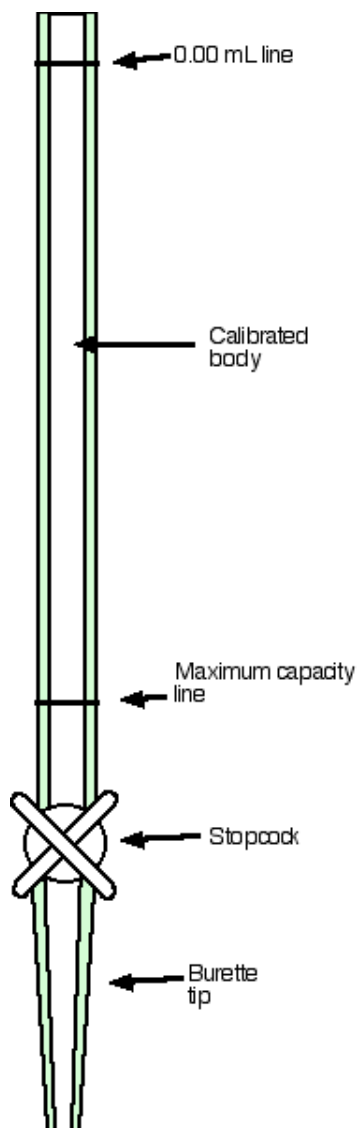


Figure 3. Typical buret. Calibration marks will be located between the 0.00 mL and the maximum capacity line.

4. Dispense a portion of deionized water from the buret into the beaker. The portion size depends on the size of the buret: ~5 mL for a 25.00 mL buret, or ~10 mL for a 50.00 mL buret. Record the indicated volume from the buret in your notebook.
5. Determine the mass of water dispensed using the analytical balance. Record this value in your notebook.
6. Repeat steps 4 and 5 until the 25.00 mL (or 50.00 mL, depending on the size of the buret) mark has been reached. For each TOTAL volume of water dispensed, record the TOTAL mass.

7. Now you can discard the water in the beaker. Refill the buret to the 0.00 mL line, and repeat the procedure (Steps 2 through 6) two more times, for a total of 3 calibrations. Do NOT try to dispense exactly the same volumes of liquid!
8. For each set of calibration data, determine the volume of water dispensed by dividing the total mass of water dispensed by the density of water at that temperature. When you have finished each calibration, you should have a data set similar to that shown below.

Indicated volume (buret), mL	Mass of water dispensed, g	Calculated volume of water, mL
5.32	5.2993	5.3009
9.97	10.0038	10.0068
15.06	15.0027	15.0072

9. Determine the difference in volumes by subtracting the calculated volume of water from the indicated volume of water. In our table above, we have a calculated volume of 5.3009 mL and an indicated volume of 5.32 mL. Performing the subtraction, we see that the difference is 0.0191 mL. The differences can be either positive or negative; do not ignore the signs.
10. Prepare a single calibration chart by plotting the differences in volume (y-axis), versus the indicated volume (x-axis).

Lab report.

There is no specific report format for this experiment.

You are to write what you believe to be a complete and thorough lab report.

You may handwrite this report, or use a word-processor.

The report may be in ink or in pencil, and it may be written on plain paper or on notebook paper.

DO NOT WRITE THE REPORT ON LABORATORY NOTEBOOK PAPER AND DO NOT REMOVE PAGES FROM THE LABORATORY NOTEBOOK.

WASTE DISPOSAL: Water and acetone can be disposed of down the sink drain. Any broken glassware is disposed of in the broken glass container.

Do not throw broken glass into the trashcan, and don't throw trash into the broken waste containers.

Tolerances for Various Types of Glassware

Class A Burets

Buret volume (mL)	Smallest Graduation (mL)	Tolerance (mL)
5	0.01	± 0.01
10	0.05 or 0.02	± 0.02
25	0.1	± 0.03
50	0.1	± 0.05
100	0.2	± 0.10

Class A volumetric flasks

Flask capacity (mL)	Tolerance (mL)
1	± 0.02
2	± 0.02
5	± 0.02
10	± 0.02
25	± 0.03
50	± 0.05
100	± 0.08
200	± 0.10
250	± 0.12
500	± 0.20
1000	± 0.30

Class A transfer pipets

Volume (mL)	Tolerance (mL)
0.5	± 0.006
1	± 0.006
2	± 0.006
3	± 0.01
4	± 0.01
5	± 0.01
10	± 0.02
15	± 0.03
20	± 0.03
25	± 0.03
50	± 0.05