

Calibration of Burets and Pipets

I. Introduction

According to the National Institute of Standards and Technology (NIST), the error of a 25-mL Class A buret should not exceed 0.03 mL, i.e., the allowable error is about one (1) part per thousand (ppth). For careful analytical work, each buret should be calibrated to ensure that the buret markings conform to these standards. Similarly, the tolerance of a 2-mL, class A, transfer pipet is ± 0.006 mL (or 3 ppth). The 2-mL pipet should also be calibrated.

Before calibration the buret should be cleaned and rinsed thoroughly with deionized water. The buret must be checked for satisfactory performance; the stopcock must not leak. To check it, fill the buret with water, place a paper towel beneath the tip, and observe it over a period of time. If a drop appears, the stopcock may need to be tightened or cleaned. If the problem persists, the buret should be replaced. In order to ensure that your volumetric glassware is clean and ready for calibration, fill with deionized water and allow it to drain completely. If clean, the water should drain without beading on the inner walls. If the buret or pipet is in need of cleaning, check with your lab instructor about soaking the pipet in cleaning solution or obtaining some cleaning solution to clean the buret.

II. Procedure

A. Buret Calibration

Obtain about 200 mL of deionized water and allow it to come to room temperature. Clean a 60-mL conical weighing flask with ground glass stopper; the flask should be dry on the outside and in the inner neck region, but not necessarily on the lower inside. (Why?) Weigh the stoppered flask. Fill the clean buret with the room temperature deionized water (measure and record the temperature of the water). **Ensure that there is no air bubble in the tip of the buret.** Open the stopcock until the meniscus is at the zero mark, allowing the water to flow into any convenient vessel. Touch the tip of the buret to the inside of the vessel to remove the suspended droplet. Read the meniscus level to the nearest 0.01 mL. Now remove the stopper of the weighed flask. Place the flask on a clean piece of white paper under the buret, with the buret low enough so that the buret tip extends approximately 3/4" into the flask neck, and allow the water to flow into the flask until the meniscus is at the 5-mL mark. Read the meniscus level exactly. Use your plastic magnifying lens for easier location of the exact meniscus level. Touch the tip of the buret to the flask to remove the suspended drop and stopper the flask. Try to keep the stopper dry, since any water on it can evaporate when it is out of the flask. Weigh the flask again with its contents. Without emptying the flask or refilling the buret, again allow water to flow into the flask until the reading is 10 mL. Remove the suspended drop and weigh the flask and contents. Repeat this process at 5-mL intervals until the 25-mL mark is reached. After the final weighing, determine the temperature of the water. The initial and final temperatures should agree within about 1°C. (Why?)

While we only require an accuracy of 0.01 g in the mass of the water - since 0.01 g of water corresponds to about 0.01 mL which is the limiting precision in reading the buret - it is desirable and easy to determine the mass of the water and flask to the nearest milligram and then round off the final value for the buret correction.

Tabulate the data and results as indicated below.

Typical Data Obtained in the Calibration of a Buret

For the calibration below, assume that the temperature of the water is 23°C (your water temperature may be different). At this temperature, the conversion factor from mass of water to volume, corrected for buoyancy and glass expansion, is 1.0035 mL/g.

Buret reading (mL)	Apparent mass of flask and contents (g)	Apparent mass of water (g)	True volume (mL)	Buret correction (mL)
0.02*	41.153	0.000	0.00	0.00
5.01	46.160	5.007	5.02	+0.03*
10.00	51.136	9.983	10.02	+0.04
15.03	56.125	14.972	15.02	+0.01
20.01	61.096	19.943	20.01	+0.02
24.98	66.023	24.870	24.96	0.00

* Note that the initial buret reading is not zero and must be subtracted from subsequent readings.

The calculations are performed as follows: Columns 1, 2, and 3 of the above table are self-evident. Each value in Column 4 is obtained by multiplying the respective value in Column 3 by 1.0035, the true volume of one apparent gram of water. The values in Column 5 are obtained by subtracting the volume delivered (from Column 1 data) from the corresponding values in Column 4. Each correction represents the quantity that must be added algebraically to a particular buret reading to obtain the true volume delivered.

Repeat the calibration. The corrections for each reading should agree within approximately 0.03 mL. If the results do not agree, repeat the calibration process until they do. Average the results and prepare a plot of the mean correction against the buret readings (rounded to 5-mL intervals) using the EXCEL spreadsheet software in the computer lab (see Appendix A). Hand in a summary sheet of your results (see p. 6).

B. Pipet Calibration

Similarly, pipets can be calibrated by weighing the water delivered from them. The same stoppered flask used for the buret calibration may be employed. In order to obtain meaningful results, proper techniques for delivering the water must be followed.

1. Transfer Pipets

Using a bulb, draw up deionized water until the calibrated volume is full and the water level is slightly above the calibration mark. With a finger over the upper end of the pipet, lift the pipet out of the water, tilt the pipet almost to the horizontal and wipe any residual water from the outside walls. Turn the pipet upright and allow the water to drain slowly until the meniscus is exactly at the calibration line. Remove the hanging drop by touching it to the side of a glass vessel. Hold the pipet vertically and allow the water to drain - with the pipet tip against the wall of the flask - into the pre-weighed flask and, making sure to remove the suspended drop, stopper the flask and weigh it to the nearest 0.1 mg. **DO NOT BLOW OUT OR SHAKE OUT THE WATER REMAINING IN THE TIP - THE PIPET IS CALIBRATED TO ALLOW FOR THIS.** Repeat the process several times until consistent results are obtained for at least 5-6 measurements. From the mass of the water for each calibration measurement, **CALCULATE THE TRUE VOLUME DELIVERED BY YOUR PIPET.** Calculate and report (see p. 6) the mean volume delivered by your pipet and the relative mean deviation (RMD) for your series of measurements.

2. Autopipets

Autopipets are conveniently used to deliver small (μL to mL) volumes. Although autopipets are easy to use, care must be taken or the volume delivered can be very different from what is expected. **YOUR INSTRUCTOR WILL DEMONSTRATE THE PROPER USE OF THE AUTOPIPETS AVAILABLE IN THE LABORATORY.** (If the autopipets are adjustable, one must check that the proper volume is selected before **every** use of the pipet.) Calibrate the 1-mL autopipet that you've been assigned by weighing water samples as was done for the transfer pipet. Calculate and report (see p. 6) the mean volume delivered by your pipet and the RMD for your series of measurements.