

Calibration of Volumetric Glassware

This set of laboratory experiments is designed to introduce you to some of the apparatus and operations you will be using during the remainder of this course, including the treatment of data using spreadsheets. While some of the procedures may seem trivial and the results obvious, this is a chance for you to develop good laboratory technique.

Required Reading

D.C. Harris, *Quantitative Chemical Analysis* (9th ed., W. H. Freeman, NY, 2016) chapter 2 (Tools of the trade)

Electronic Balances (including Weighing by Difference), *Analytical Lab Manual* (Laboratory Introduction).

Burettes, *Analytical Lab Manual* (Laboratory Introduction).

REAGENTS AND APPARATUS

- Analytical Balance
- 400 mL beaker
- 3 - 2 oz. plastic bottles with caps
- 5 mL volumetric pipet
- 100 μ L Eppendorf micropipet
- thermometer
- distilled, deionized water
- 2 , 50 mL burets

PART A - USE OF THE ANALYTICAL BALANCE

The analytical balances in the lab are probably the most precise, accurate and reliable pieces of equipment that you will use during the semester. To understand how these balances operate and

their limitations you should read the appropriate section in your textbook. Although there are inherent limits in the accuracy and precision of these balances most weighing errors are caused by incorrect handling of the sample. In the first part of this lab you will investigate several potential sources of error.

PROCEDURE

1. In this section you will determine the mass of a clean, dry weighing bottle under various conditions. Unless instructed otherwise, you should handle the bottle with your crucible tongs, gloves, or lint-free paper and measurements should be made to the nearest 0.1 mg. Begin by placing the weighing bottle and cap (with cap removed) in the oven for about 5 minutes. Remove and re-mass while warm. Follow the change in its apparent mass for several minutes, reweighing every thirty seconds. Record all masses including the final constant value.

2. After massing the weighing bottle, roll it around in your hand (handle the bottle with your fingers) and then re-mass and compare the two masses. *How should weighing bottles be handled on a regular basis?*
3. Next, wipe the bottle clean with a dry, lint-free cloth or tissue and reweigh. Record all observations.
4. Hold the weighing bottle and inch from your mouth and breathe on it several times. Re-mass and compare with previous data.
5. Discuss your results in your laboratory write-up.

PART B- CALIBRATION OF VOLUMETRIC GLASSWARE

Throughout the semester, you will be required to make accurate measurements of volume. The primary means of doing so will be by using either a volumetric pipet, a Mohr pipet, a micropipet, or a buret. Many manufacturers calibrate their glassware so that the true volume is within specified limits of the labeled volume. In cases where particularly accurate measurements are needed, it may be necessary to make a more accurate calibration of your volumetric containers. In this experiment you will calibrate a buret, a volumetric pipet, and a micropipet.

PROCEDURE

In order to prevent difficulties with access to the analytical balances, please perform all pipetting and volume measurement manipulations at your normal lab station, then transport the bottle to the analytical balance to mass it.

1. If you have any questions concerning the use of any of the volumetric glassware necessary for the experiment, be certain to refer to Chapter 2 in your textbook. Additional information can be obtained from your instructor. A summary of the calibration techniques are provided on pgs 39 (pipet) and 45 (buret).
2. Place about 250 mL of distilled, deionized water in a 400-mL beaker. Place a thermometer in the beaker containing the water and leave it in place through the remainder of the experiment. If the temperature of the water is not identical to that of the room temperature, allow the water to equilibrate to room temperature. Record the temperature of the equilibrated water. If the temperature of the water changes during the experiment, record the changed temperature. Use the temperature recorded just prior to each mass measurement to do the calculations described later in the experiment. *Why are these calculations necessary? How much effect does the temperature of the water have upon the actual delivery of the pipet or buret?*
3. Label the 4 plastic bottles, one for each different volumetric device to be calibrated, with an identifying tag for each of the pieces of glassware that you will calibrate. Cap the bottles and weigh to the nearest 0.1 milligram. Record the masses.
4. Using the 10-mL volumetric pipet, transfer a water sample to the appropriately labeled bottle. Replace the bottle's cap and reweigh the bottle to the nearest 0.1 mg.

5. Repeat step 4 with the same pipet into the same bottle until you have made at least 8 repeat deliveries. You should at this point have recorded a total of 9 masses for the bottle.

6. With your second plastic bottle do a similar exercise using the Eppendorf micropipet instead of the volumetric pipet. Ask your instructor to show you the correct way to use the micropipet. A volume size of 100 μL or greater is appropriate for this analysis.

Be certain to put approximately 10 mL of liquid in the bottle before you begin your measured deliveries. *Why is this done?*

7. The third piece of equipment that you need to calibrate are your burets. Fill the buret with distilled, deionized water so that the meniscus is above the zero mark. (Be absolutely certain that there are no air bubbles in the tip of the buret before calibrating it.) Slowly, allow approximately 5 mL of the water to drain into the bottle, wait 30 seconds for the drainage from the wall of the buret, and read the meniscus to the nearest 0.01 mL. (**Special Notes on Burets: 1. All of your buret measurements should be made to 0.01 mL. 2. You should never attempt to start the buret at 0.00 mL. Points will be deducted from your lab score whenever either of these guidelines is violated**) Touch the tip of the buret to the inner lip of the bottle, cap the bottle and reweigh. Repeat this procedure using approximately 5.00 mL additions (5.00 to 10.00, 10.00 to 15.00, etc, until you reach 50.00 mL) read accurately from the buret. In each case make sure that you carefully write down the true reading of the buret. Your instructor will find it very unlikely that you delivered exactly 5.00 mL from the buret in each step! Once you have completed calibrating your first buret, repeat the same procedure for the second buret.

CALCULATIONS

All calculations and requested graphs to be performed in this laboratory should be done using a spreadsheet program such as Microsoft Excel.

It is important to understand the role of spreadsheets in data manipulation and graphing as you will make use of these skills throughout the semester.

Use the difference in mass between each set of two consecutive mass measurements to determine the mass of water delivered by the glassware being tested. Consider the application of a correction for buoyancy errors in this mass. Once the true masses are known, find the volume delivered by making a density correction for each mass measurement obtained in your experiment (Remember that the temperature of the water at each massing is important when making this correction). *Where might you find information about the density of water as a function of temperature?*

Calculate the average volume, the standard deviation, and the percent standard deviation of the results obtained with each pipet. Compare and contrast the accuracy of the two pipets you calibrated. *How does the precision of each of the pipets compare? Does yours volumetric pipet*

meet the National Bureau of Standards requirements for class A glassware? Calculate the volume of water contained in each delivery from your buret. Compare it to the value you read from the buret scale. *How does each compare?* Summarize the net percent error in delivery from your buret and comment upon it. If it is a significant error, based on an expectation of the tolerance of your buret, you may wish to apply a correction to all future data obtained with this buret.

Include in the Report

1. Set up tables and perform your calculations (see example in text (pg 50)) in Microsoft Excel, using the volume of water corrected to 20°C (Table 2-7, page 38).
2. Using the spreadsheet, graph the calibration curve for each buret. These graphs should be similar to Figure 3-3, page 49 of Harris.
3. Calculate the average volume delivered by the 10 mL pipet, the N-1 standard deviation and the 95% confidence interval for your pipet data and state your final value.
4. Compare your buret results with the tolerances in Table 2-2, page 30 of Harris.
5. Compare your pipet results with the tolerances in Table 2-4, page 32 of Harris.
6. Assume 36.45 mL of a solution is delivered from your buret. Calculate the amount your buret actually delivered. (Do this for both burets).

Note: You need your buret calibration graphs throughout the semester to correct your buret readings so it is important to do a very careful job today. The error plotted indicates the amount that should be added to the buret reading to give the true volume delivered. Interpolation is used for readings between those corresponding to the calibration measurements.