

Introduction to the Analytical Balance & 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.

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
- 50 mL buret

Special Notes on Burets:

1. *All of your buret measurements should be made to two decimal points (0.01 mL.)*
2. *You should never attempt to start the buret at 0.00 mL.*

Points will be deducted whenever either of these guidelines is violated

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, nylon 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 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. Do not mass liquids on a balance unless they are in a closed container!

1. If you have any questions concerning the use of any of the volumetric glassware necessary for the experiment, refer to Chapter 2 in your textbook. Additional information can be obtained from your instructor.
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 3 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 5-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 is your buret.

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. 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, 15.00 to 20.00, and 20.00 to 25.00 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.

CALCULATIONS

All calculations and requested graphs in this experiment should be done using a spreadsheet program such as Microsoft Excel. If you are unfamiliar with spreadsheets, see the “Data Analysis” section on chemlab.truman.edu for a tutorial. 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.

1. For your weighing bottle measurements from Part A, plot the mass of the weighing bottle as a function of the time that the bottle was out of the oven. Often this will be noted as “mass as f(time)” where mass is the dependent variable and is plotted on the y-axis and time is the independent variable and is plotted on the x-axis. Based on your plot, how long did it take for the mass of the bottle to stabilize? What does this mean in terms of making mass measurements of items removed from the oven?
2. For the volumetric pipet and micropipette measurements, correct each of your mass measurements for buoyancy using the information in Table 1. 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. Once the true masses are known, find the volume delivered by using the densities in Table 1 (Remember that the temperature of the water at each massing is important when making this correction).

	Temperature (°C)	Density (g/mL)
$m = \frac{m'[1 - (d_a/d_w)]}{1 - (d_a/d)}$	20	0.9982071
m = corrected mass	21	0.9979955
m' = measured mass	22	0.9977735
d_a = density of air (0.00120 g/mL)	23	0.9975415
d_w = density of balance weights (8.00 /mL)	24	0.9972995
d = density of object being weighed	25	0.9970479
	26	0.9967867
	27	0.9965162
	28	0.9962365
	29	0.9959478

Table 1. Information for buoyancy and density corrections.

Once you have corrected for buoyancy and density, calculate the average, the standard deviation, and the percent relative standard deviation of the volumes obtained with each pipet. Compare the accuracy and precision of the two pipets you calibrated. *Does yours volumetric pipet meet the National Bureau of Standards requirements for class A glassware?*

3. Using the masses of each addition and the density of water, 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.