Experiment 1

Calibration of Glassware

Calibration and determination of error in volumetric glassware

Objective

Students will calibrate a beaker, buret, and volumetric pipette and also determine the error associated with measuring volumes using a micropipette.

Logistics

The duration is two weeks, and students will work individually. The spreadsheet with calculations for error analysis must be submitted to Canvas within one week after you complete the lab. Before lab, prepare your notebook according to the Lab Policies course document on Canvas.

Introduction

When we calibrate a piece of volumetric glassware, we are looking to verify the agreement between the claimed volume and the actual volume. If agreement is lacking, a correction is determined. A calibration consists of determining the mass of a liquid of known density (water in this case) that is contained in (or delivered by) a piece of volumetric glassware. From the relationship for density *(d* = mass/volume), we can determine the exact volume occupied by the liquid. Because the density of a substance varies with temperature, we must measure the temperature of the liquid to correct the value of *d* used. Table 1 contains densities at various temperatures.

Temperature (°C)	Density (g/mL)
16	0.9989460
17	0.9987779
18	0.9985986
19	0.9984082
20	0.9982071
21	0.9979955
22	0.9977735
23	0.9975415
24	0.9972995
25	0.9970479

Table 1 The density of water at various temperatures. From "Quantitative Chemical Analysis" by Daniel C. Harris, 2010

In this experiment, we will calibrate three pieces of glassware: A) a 50 mL beaker; B) a 10 mL Class A volumetric pipet; and C) a 50 mL buret. We will also learn to use and check the calibration on an adjustable 100 - 1000 µL micropipette. This is an applied laboratory skill that is worth spending time becoming proficient at.

We will check the calibration of the micropipette by calculating the inaccuracy and imprecision. Inaccuracy (A) compares the average volume measured ($\overline{V}$) to the desired volume being dispensed (V_0). Inaccuracy is a measure of systematic error. It can be calculated as the absolute value, A, and as a relative value, A%, as shown below.

$$A = \overline{V} - V_0$$

$$A\% = \frac{A}{V_0} \times 100\%$$

Imprecision is a measure of random error and helps us understand the repeatability of pipettings. Imprecision can be calculated as standard deviation (S) and as the relative value called the coefficient of variation (CV%) as shown below.

$$S = \sqrt{\frac{\sum_{i=1}^{n} \left(V_i - \overline{V}\right)^2}{n-1}}$$

$$CV\% = \frac{S}{\overline{V}} \times 100\%$$

Procedure

Read before beginning any part of this lab:

Obtain some deionized water in a large clean beaker to be used for all parts. Record the temperature of the water to the correct number of significant figures. Use the data in Table 1 to create a linear trendline, and use the equation of the line to find the density at your exact temperature. Note: you should add significant figures to the equation of the line before using it to calculate the density. You will need 6 decimal places.

Obtain a 100 mL beaker, a 50 mL buret, and a 10 mL Class A volumetric pipet. Check these items for cleanliness; water should drain uniformly while maintaining a "wetted" appearance after draining. Any breaks or non-wetting signifies that residues are present which will decrease the precision and worsen the performance of any calibration. If this is the case, you should thoroughly wash the glassware with soap and rinse several times.

A. 100 mL Beaker calibration

You will determine the accuracy at which a 100 mL beaker is able to measure **10 mL of water**. For a total of three trials, record the initial and final mass of the beaker before and after the addition of 10 mL of water.

Measure the temperature of your water to the correct number of significant figures. Use the density from your trendline to determine the corrected volume of water at each of the three masses that you determined. Then calculate the average, standard deviation, % RSD, and 95 % confidence interval. Be sure these calculations are formatted with the appropriate equation in Excel.

B. Buret calibration

You will calibrate the buret at the following volumes: 10, 20, 30, 40, and 50 mL. Additions will be performed in increments of approximately 10 mL and should be reported with the correct number of significant figures. Liquid dispensed from the buret will be collected in a 100 mL beaker.
For each of the volumes, you should record the **initial and final volumes** read from the buret as well as the **initial and final masses**. Do not empty the flask between additions!
Repeat so that you have data for two trials.
Use the masses measured for each addition and the density to determine the volume of water (V_{H_2O}). Then calculate the correction from the difference of the measured volume (V_{trial}) and the volume calculated from the density (V_{H_2O}). Consider the sample data in Table 2 for the first four additions.

Trial	V_i	V_f	V_{trial}	M_i	M_f	M_{H2O}	V_{H2O}	Correction
1	0.00	10.00	10.00	58.77	68.69	9.9134	9.9312	0.07
2	10.00	20.00	10.00	68.69	78.62	9.9330	9.9508	0.05
3	20.00	29.90	9.90	78.62	88.54	9.9224	9.9402	-0.04
4	29.90	40.00	10.10	88.54	98.54	10.0009	10.0189	0.08

Table 2. Example data from the buret calibration. The correction is calculated from the difference in volumes V_{trial} - V_{H2O}.

Prepare a calibration curve by plotting the correction vs. total delivered volume for both trials. To obtain the x-axis values, you will need to create an additional column that calculates the running total for the volume. For example, the third data point for Trial A appears at 29.90 mL, not 30 mL.

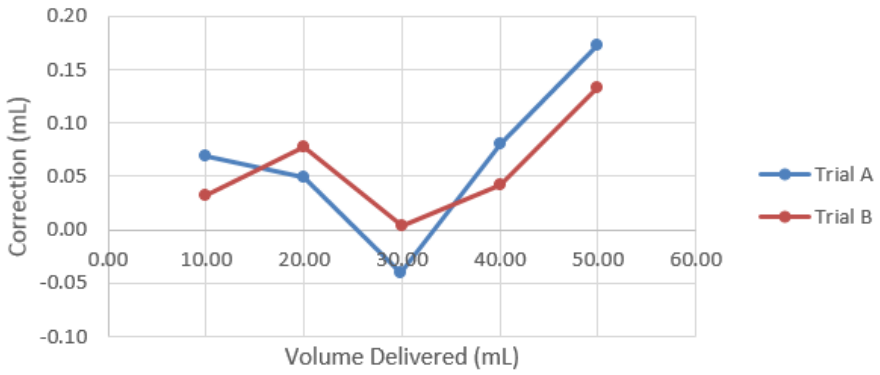

Figure 1. Plot of the error in predicting the volume dispensed as a function of the total volume.

C. Volumetric pipette calibration

On the top-loading balance, record the mass of the 50 mL beaker that you will use to weigh your dispensed volume of water. Use the same balance as often as possible. Dispense the 10 mL delivered volume into the beaker, and record the mass. Repeat for a total of three trials, making sure to discard the water between trials and to record a new initial mass each time.

Use the temperature of your water along with data in Table 1 to determine the corrected volume of water at each of the three masses that you determined. Then calculate the average, standard deviation, % RSD, and 95% confidence interval. Be sure these calculations are formatted with the appropriate equation in Excel.

D. Micropipette calibration

Obtain about 100 mL of nanopure water and record the temperature to the correct number of significant figures. Determine the density for water at that temperature using your trendline from the data in Table 1. Check out a micropipette from the instructor/ TA and write the serial number in your notebook.
Dial the pipette to 1000 µL and transfer one pipetting to waste in order to wet your pipette. Then take the mass of the next pipetting to four decimal places. Repeat this for a total of ten trials, making sure to use the technique demoed by the instructor. Check with the instructor to make sure your masses look correct, then repeat this process for the 100 µL volume. Convert each mass to volume in units of µL using the density you determined.
Use Excel to calculate the A%, standard deviation, and CV% for the 1000 µL and 100 µL volumes. Compare your values to the ISO standards in Tables 2 and 3. Do your values match the required criteria? You must have the following tables in your notebook (in addition to your data tables). If your tolerances are not within these specifications, you should consult the instructor.

Table 2. Relative Inaccuracy %A

Volume	Tolerance	Your value	Is the condition met?
1000 µL	±0.8		
100 µL	±8.0		

Table 3. Coefficient of Variation %CV

Volume	Tolerance	Your value	Is the condition met?
1000 µL	±0.3		
100 µL	±3.0		

Spreadsheet Assignment

Download the spreadsheet template from Canvas. Each piece of glassware should have a separate sheet (one for the beaker, one for the buret, and one for the volumetric pipette). The completed spreadsheet should be submitted to Canvas **before** the start of the following lab period. You should also have a sheet to determine the %A and CV% for each volume tested.

Conclusion

Your conclusion should be printed and submitted to Canvas before the next lab period. Format your typed conclusion to tell the purpose of the lab and list your results. Be sure to discuss the following.

- Discuss the volume of greatest uncertainty for the buret. Did you have the most uncertainty when trying to dispense 10, 20, 30, 40, or 50 mL? For example, in Figure 1, the data suggest this buret is very inaccurate at dispensing 50 mL but is accurate at dispensing 30 mL.
- Give the measured volumes for the beaker and volumetric pipette (report as average ± standard deviation). Which had the lowest %RSD and does this make sense?
- Summarize your micropipette calibration results. Do your results conform to the manufacturer's specifications? You may wish to copy Tables 2 and 3 from above to summarize your results.
- Discuss your error by comparing your results with the tolerances listed in the tables in Chapter 2 of the Harris textbook for the **volumetric pipette** and **buret**.

References should be cited at the end of your conclusion using ACS specifications.