Experiment #2: Mass-Volume Exercise • Calibration of Volumetric Equipment

Introduction:

The outcome of any chemical analysis is unequivocally dependent on the two major factors: the professional skills of a laboratory analyst and the accuracy of the analytical equipment. Therefore, mastering the proper volume measuring techniques are essential to maximize the data quality and the data reliability. Furthermore, the hands-on skills gained from this procedure will be instrumental in building the necessary confidence in students' ability to successfully perform the subsequent laboratory experiments.

The purpose of this experiment is to gain the practical skills in measuring and delivering accurate and precise volumes using volumetric glassware. This experiment consists of three parts. In part one, a *10-mL graduated cylinder* is used to accurately transfer 10-mL portions of the distilled/deionized water into pre-weighed *100-mL volumetric flask*. In parts two and three, the same 10-mL volume of water, as in the part one, will be delivered by a *10-mL volumetric pipette* and a *50-mL burette*.

After each volumetric transfer, the accurate mass of the delivered water will be measured using a *calibrated analytical balance*. The mass of the measured water will be converted into its corresponding volume using the water's density values listed Table 5. Upon completion of this exercise, the students will perform statistical analysis to determine the *mean* (average), the *standard deviation* (precision) and the *relative error* (accuracy) on the transferred volumes generated by each volumetric device. The data from the statistical analysis will be further used to assess the students' pipetteting skills as well as to establish the most accurate and precise volumetric device.

Definition of Terms:

<u>*Accuracy*</u> assesses the closeness of the experimental measurement to its true or accepted value. In this procedure, *relative error* will be used to assess the degree of accuracy of the analytical glassware as well as a student's ability to deliver specific volumes. The relative error will be determined based on the equation stated below:

$$E_r = \frac{x_i - x_t}{x_t} \cdot 100\%$$

x_i ≡ Individual Measurement (Delivered Volume)

x_t ≡ True or Accepted Value (Theoretical Volume: 10.00 mL)

<u>*Precision*</u> assesses the degree of agreement among the individual measurements when the same procedure is applied repeatedly to a sample (*analyte*) of interest. In this procedure, standard deviation will be used to assess the precision of the analytical glassware as well as the student's ability to deliver the reproducible volumes. The standard deviation will be determined quantitatively based on the equation stated below:

Written by A. Gorbenko • Graphics & Edits by M. Danza

$$\text{Standard Deviation (s)} = \sqrt{\frac{\sum_{i=1}^{N}(x_i - \bar{x})^2}{N-1}}$$

$\bar{x}$ ≡ Sample Mean

N ≡ Number of Replicate Measurements

x_i ≡ Individual Measurement (Delivered Volume)

Equipment, Glassware and Reagents:

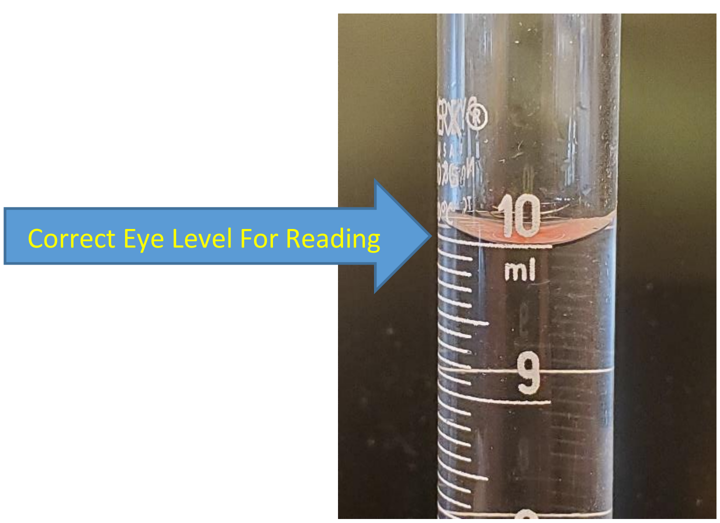
10-mL Volumetric Pipette, 10-mL Graduated Cylinder, 50-mL Burette, Three-Way-Valve Pipette Bulb, Regular Pipette Bulb, 100-mL Volumetric Flask, 100-mL Beaker, Analytical Balance, Thermometer, Transfer Pipettes, Distilled/Deionized Water.

Procedure:

Part I: Volume Measurement with 10-mL Graduated Cylinder

1. Weigh a dry 100-mL stoppered, volumetric flask. Record the mass to all significant figures displayed on the balance.
2. Fill the 10-mL graduated cylinder to the 10-mL mark. Use a transfer pipette to adjust the volume so that the bottom of the meniscus coincides with the 10-mL mark as depicted in Figure 1.
3. Transfer the water into pre-weighed 100-mL volumetric flask, close the flask with the stopper and weigh the flask containing the water recording all significant figures.

Figure 1. Volume Measurement with 10-mL Graduated Cylinder



4. Calculate the mass of the transferred water by subtracting the mass of the empty flask from mass of the flask filled with water. Record the mass of the transferred water in Table 1.
5. Repeat this procedure nine more times and record each mass in Table 1.
6. Then, subtract mass #1 from mass #2, mass #2 from mass #3, etc., to obtain individual masses of 10-mL water.
7. Calculate the **Volume of Water (mL) from Density** for each corresponding mass using the density values listed in Table 5 and report those volumes in Table 1 (sample calculation is depicted at the end of this procedure).
8. Determine the Average (Mean), Standard Deviation, and Relative Error for **Volume of Water (mL) from Density** and report those results in Table 1 (sample calculation is depicted at the end of this procedure).

Mass of the empty 100-mL Volumetric Flask: ____________g

Balance Brand and Model # ____________

Temperature of Water: ____________ºC

Density of Water (Table 5): ____________g/mL

Table 1. Volumes Measured with 10-mL Graduated Cylinder

Sample #	**Mass of Water & Volumetric Flask (g)**	**Mass of Water (g)**	**Volume of Water (mL) from Density**
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
Average (Mean)			
Standard Deviation			
Relative Error			

Theoretical Volume: 10.00-mL (for Relative Error Calculation)

Part II: Volume Measurement with 10-mL Volumetric Pipette

1. Weigh a dry 100-mL stoppered, volumetric flask. Record the mass to all significant figures displayed on the balance.

Technique #1: Operation of Three-Way-Valve Pipette Bulb

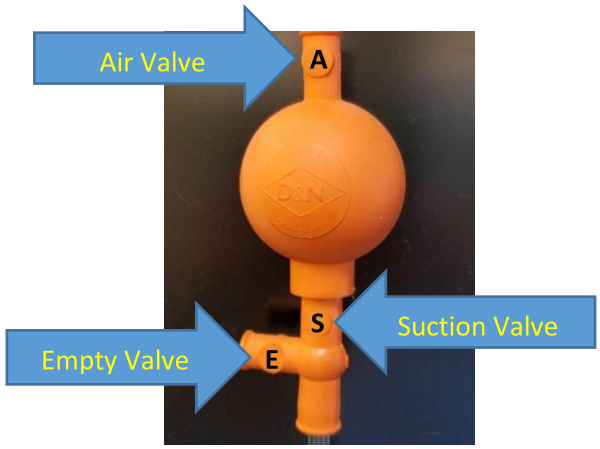
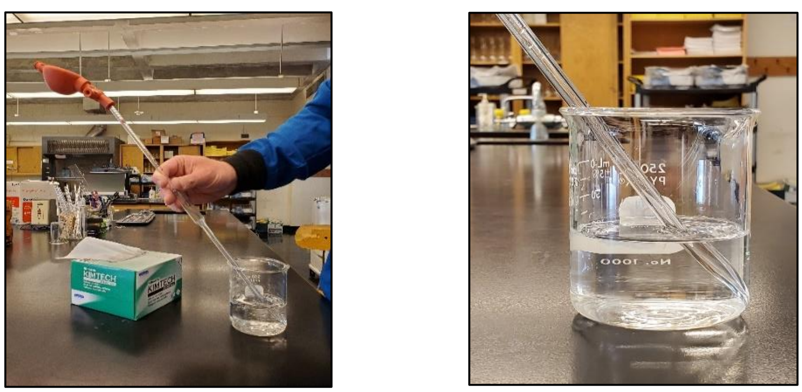


Figure 2. Three-Way-Valve Pipette Bulb

T1a. Insert the top **(about 1/4 inch)** of the 10-mL volumetric pipette into the bottom of the pipette filler. To draw the liquid into the pipette, press and hold **A** (*air valve*) and squeeze the bulb with your free hand. This action will create the necessary vacuum to draw liquid into the pipette.

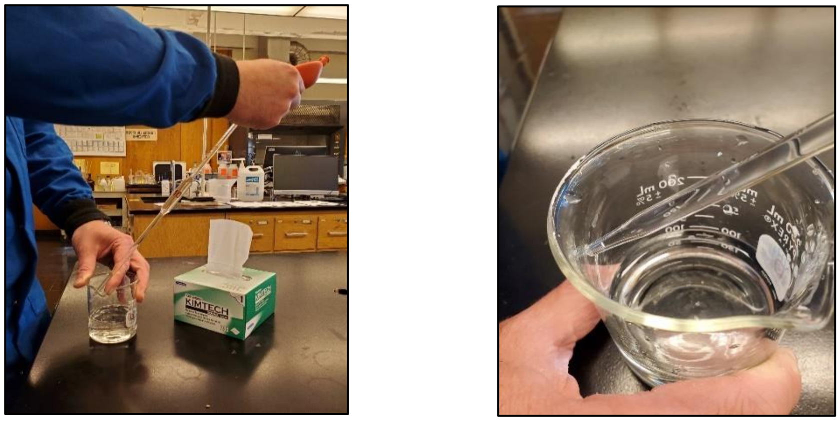
T1b. To fill the liquid into the pipette, place the pipette slightly slanted inside the beaker as shown in Figure 3. On the pipette bulb press **S** (*suction valve*) to aspirate the water to the level above the graduation mark.

Figure 3. Position of Pipette to Aspirate Liquid



T1c. Take the pipette out of the liquid and wipe its exterior with a Kimwipe tissue to remove any adhering liquid. To remove the excess liquid and to ensure that no air bubbles are transferred into the receiving vessel, slightly tilt the pipette and let the tip touch the walls of the beaker as depicted in Figure 4.

Figure 4. Pipette Position for Volume Adjustment



T1d. As the pipette is held in that position, press **E** (*empty valve*) to release the liquid into the beaker until the bottom of the meniscus coincides exactly with the graduation mark.

T1e. Bring the pipette filled with the desired volume into the pre-weighed 100-mL volumetric flask. On the bulb, press and hold **E** to release the liquid. When the transfer of the liquid is complete, rest the tip against the inner wall of the receiving vessel for about 10 seconds. Withdraw the pipette from the volumetric flask. **Don't blow out the liquid that remains in the tip of the pipette.** The volumetric pipettes are calibrated to retain that liquid.

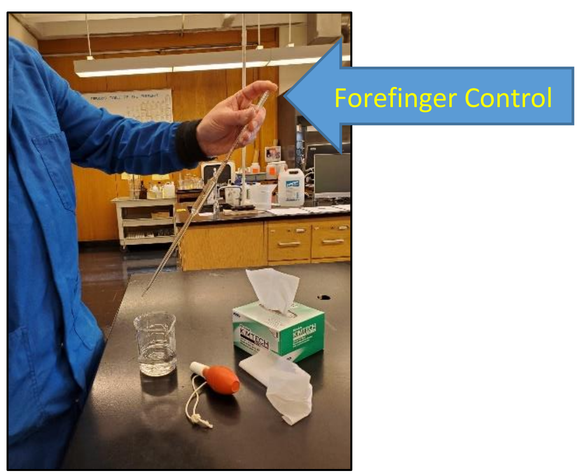
Technique #2: Operation of Regular Pipette Bulb

Figure 5. Regular Pipette Bulb

T2a. Squeeze the pipette bulb (this action will create the necessary vacuum for the liquid aspiration). Insert the top of the 10-mL volumetric pipette into the bottom of the squeezed pipette bulb. Carefully draw the liquid into the pipette above the graduation mark by slowly releasing the bulb.

T2b. Quickly replace the pipette bulb with your forefinger to stop the outflow of liquid as depicted in Figure 6.

Figure 6. Control of Liquid Outflow (Using Forefinger)



T2c. Take the pipette out of the liquid and wipe its exterior with a Kimwipe tissue to remove any adhering liquid. To remove the excess liquid and to ensure that no air bubbles are transferred into the receiving vessel, slightly tilt the pipette and let the tip touch the walls of the beaker (refer to Figure 4). As the pipette is held in that position, partially release your forefinger to allow the bottom of the meniscus to coincide exactly with the graduation mark.

T2d. Bring the pipette filled with the desired volume into the pre-weighed 100-mL volumetric flask. Remove your forefinger to release the liquid. When the transfer of the liquid is complete, rest the tip against the inner wall of the receiving vessel for about 10 seconds. Withdraw the pipette from the volumetric flask. **Don't blow out the liquid that remains in the tip of the pipette.** The volumetric pipettes are calibrated to retain that liquid.

2. Record the mass of the filled volumetric flask reporting all significant figures.
3. Repeat this procedure nine more times and record each mass in Table 2.
4. Then, subtract mass #1 from mass #2, mass #2 from mass #3, etc., to obtain individual masses of 10-mL water.
5. Calculate the **Volume of Water (mL),** Average (Mean), Standard Deviation, Relative Error as described in **Part I,** steps 7, 8 and report those values in Table 2.

Mass of the empty 100-mL Volumetric Flask: ____________**g**

Temperature of Water: ____________**ºC**

Density of Water (Table 5): ____________**g/mL**

Table 2. Volumes Measured with 10-mL Volumetric Pipette

Sample #	Mass of Water & Volumetric Flask (g)	Mass of Water (g)	Volume of Water (mL) from Density
1			
2			
3			
4			
5			
6			
7			
8			

Sample #	Mass of Water & Volumetric Flask (g)	Mass of Water (g)	Volume of Water (mL) from Density
9			
10			
Average (Mean)			
Standard Deviation			
Relative Error			

Theoretical Volume 10.00-mL (for Relative Error Calculation)

Part III: Volume Measurement with 50-mL Burette

1. Weigh a dry 100-mL stoppered, volumetric flask. Record the mass to all significant figures displayed on the balance.
2. Clean the burette with detergent and a long brush. Rinse the burette with tap water and then with distilled water.
3. Prior to fill, make sure that the stopcock is closed (stopcock handle perpendicular to the burette). Fill the burette with distilled water well above the zero mark.
4. Prime (purge) the burette from the air bubbles by rotating the stopcock to the open position (stopcock handle parallel to the burette) and **gently** tapping the lower side of the burette. Repeat this procedure several times to completely rid the burette of air bubbles.
5. Finally, lower the level of the liquid so that the bottom of the meniscus coincides with the zero graduation mark as depicted in Figure 7.

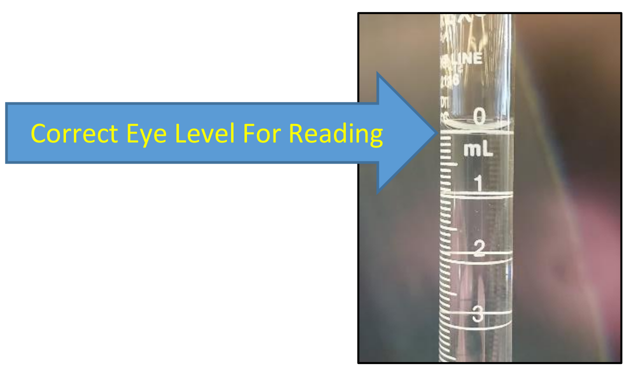
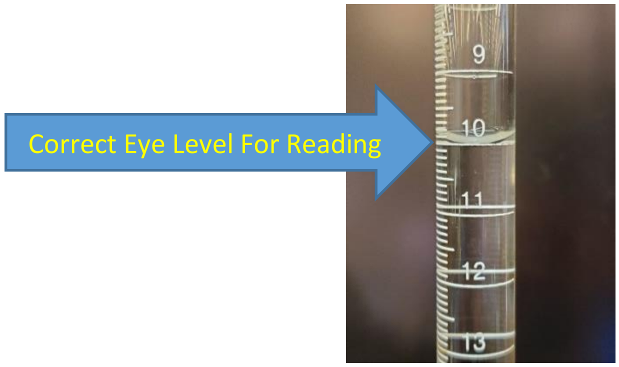


Figure 7. Initial Volume: 0.00-mL Reading

6. Using the burette, deliver 10.00-mL of the distilled water into the pre-weighed 100-mL volumetric flask. Start adding the water dropwise as you approach ten milliliter mark.

Figure 5. Final Volume: 10.00-mL Reading



7. Record the final volume to the nearest hundredth place.
8. Record the mass of the filled volumetric flask reporting all significant figures.
9. Repeat steps 5 through 8 nine more times and record each mass in Table 3
10. Then, subtract mass #1 from mass #2, mass #2 from mass #3, etc., to obtain individual masses of 10-mL water.
11. Calculate the **Volume of Water (mL),** Average (Mean), Standard Deviation, Relative Error as described in **Part I,** steps 7, 8 and report those values in Table 3.

Mass of the empty 100-mL Volumetric Flask: ____________**g**

Temperature of Water: ____________**ºC**

Density of Water (Table 5): ____________**g/mL**

Table 3. Volumes Measured with 50-mL Burette

Sample #	Mass of Water & Volumetric Flask (g)	Mass of Water (g)	Volume of Water (mL) from Density
1			
2			
3			
4			
5			
6			
7			

Sample #	**Mass of Water & Volumetric Flask (g)**	**Mass of Water (g)**	**Volume of Water (mL) from Density**
8			
9			
10			
Average (Mean)			
Standard Deviation			
Relative Error			

Theoretical Volume: 10.00-mL (for Relative Error Calculation)

Sample Calculation to Determine the Volume of Water from its Density at a Specific Temperature:

Calculation of Volume of Water (mL) for Sample #1 from its mass (g) using the density value obtained from Table 5:

Density of Water at 25.0 ºC (from Table 5): 0.997044 g/mL

Mass of Water and Volumetric Flask (g): **78.4294** g

Mass of Empty 100-mL Volumetric Flask (g): – **68.3996** g

Mass of Water (g): = **10.0298** g

$$\textbf{Density} = \frac{\text{Mass}}{\text{Volume}} \qquad \textbf{Volume} = \frac{\text{Mass}}{\text{Density}}$$

$$\textbf{Volume} = \frac{10.0298\text{g}}{0.997044\text{g/mL}} = 10.0595 \text{ mL (First Entry in Table 4)}$$

Sample Calculation of Standard Deviation (Precision):

$$\text{Standard Deviation (s)} = \sqrt{\frac{\sum_{i=1}^{N}(x_i - \bar{x})^2}{N-1}}$$

Table 4. Calculation of Standard Deviation and Relative Error

Sample #	Volume of Water (mL)	Deviation from the Mean $(x_i - \bar{x})$	$(\text{Deviation})^2$ $(x_i - \bar{x})^2$	$(\text{Deviation})^2$ $(x_i - \bar{x})^2$
1	10.0595	$+0.0735_2$	$5.40_5 \times 10^{-3}$	$5.40_5 \times 10^{-3}$
2	9.9502	-0.0357_8	$1.28_0 \times 10^{-3}$	$1.28_0 \times 10^{-3}$
3	10.1002	$+0.1142_2$	$1.304_6 \times 10^{-2}$	$13.04_6 \times 10^{-3}$
4	9.9234	-0.0625_8	$3.91_6 \times 10^{-3}$	$3.91_6 \times 10^{-3}$
5	10.0256	$+0.0396_2$	$1.57_0 \times 10^{-3}$	$1.57_0 \times 10^{-3}$
6	9.9042	-0.0817_8	$6.68_8 \times 10^{-3}$	$6.68_8 \times 10^{-3}$
7	9.8999	-0.0860_8	$7.41_0 \times 10^{-3}$	$7.41_0 \times 10^{-3}$
8	10.0389	$+0.0529_2$	$2.80_1 \times 10^{-3}$	$2.80_1 \times 10^{-3}$
9	10.0050	$+0.0190_2$	$3.61_8 \times 10^{-4}$	$0.361_8 \times 10^{-3}$
10	9.9529	-0.0330_8	$1.09_4 \times 10^{-3}$	$1.09_4 \times 10^{-3}$
Average(Mean)	9.9859_8			
Summation				$43.57_2 \times 10^{-3}$
Standard Deviation	0.0695_8			
Relative Error	0.1%			

$\bar{x}$ ≡ Sample Mean

N ≡ Number of Replicate Measurements

x_i ≡ Individual Measurement (Delivered Volume)

$$s = \sqrt{\frac{43.57_2 \times 10^{-3}}{(10-1 \text{ trials})}} = 0.0695_8$$

Sample Calculation of Relative Error (Accuracy):

$$E_r = \frac{x_i - x_t}{x_t} \cdot 100\%$$

x_i ≡ Individual Measurement (Delivered Volume)

x_t ≡ True or Accepted Value (Theoretical Volume: 10.00-mL)

$$E_r = \frac{9.9859_8 - 10.00}{10.00} \cdot 100\% = 0.1\%$$

Table 5. Density of Water at Various Temperatures

Temperature (ºC)	**Density (g/mL)**	**Temperature (ºC)**	**Density (g/mL)**	**Temperature (ºC)**	**Density (g/mL)**
19.0	0.998405	21.6	0.997860	24.2	0.997246
19.2	0.998365	21.8	0.997815	24.4	0.997196
19.4	0.998325	22.0	0.997770	24.6	0.997146
19.6	0.998285	22.2	0.997724	24.8	0.997095
19.8	0.998265	22.4	0.997678	25.0	0.997044
20.0	0.998203	22.6	0.997632	25.2	0.996992
20.2	0.998162	22.8	0.997585	25.4	0.996941
20.4	0.998120	23.0	0.997538	25.6	0.996888
20.6	0.998078	23.2	0.997490	25.8	0.996836
20.8	0.998035	23.4	0.997442	26.0	0.996783
21.0	0.997922	23.6	0.997394	26.2	0.996729
21.2	0.997948	23.8	0.997345	26.4	0.996703
21.4	0.997904	24.0	0.997296		

Post-Lab Questions:

1. Rank the glassware devices from least accurate to most accurate? Explain your reasoning.

____
____
____
____
____
____
____
____

2. Rank the glassware devices from the least precise to the most precise? Explain your reasoning.

____
____
____
____
____
____
____
____