

1 – VOLUMETRIC PIPET

Topics for Study: Sec III.3b, III.3c. *Use of the pipet, care and cleaning of volumetric glassware*

Glossary: pipet, tare, buoyancy, aliquot, precision, accuracy, Archimedes' principle

ASSIGNMENT PREPARATION AND DOCUMENTATION

Refer to the Course Web Site for details regarding assignment due dates, homework study or prelab questions, modifications to the procedure, technique tutorials, and guidelines on the required components of your post-lab report.

TECHNIQUES

In this assignment you will use the following procedures:

Manipulative skills

- use an analytical balance

- use a volumetric pipet

Theoretical skills

- calculate the precision of a set of data using the relative average deviation

- calculate the precision of a set of data using the relative standard deviation of the mean

- use the critical ratio test to decide the statistical significance of an item in a set of data

SAFETY

Always wear safety glasses or goggles, a flame-resistant lab coat, and other personal protective equipment.

BACKGROUND

When quantitative measurements are required in the laboratory, a scientist must choose equipment that will give the needed accuracy and precision. When it is necessary to quantitatively transfer a volume of solution, there are many choices. For large predetermined, specific volumes (>1 mL) it is possible to deliver volumes that have an accuracy of $>0.2\%$ using a glass volumetric pipet and a pipetting bulb. When smaller volumes are required, an air-displacement pipetter is more convenient. However, the error in accuracy increases as the volumes decreases. For 1-mL volumes the error is 0.6% increasing to 5% for volumes of 1 microliter. If a high degree of precision is also necessary, you must first ensure two things: (1) that you have the skills to use the equipment reproducibly and (2) that you know the true volume delivered by the equipment at the temperature at which you will use it. Determining these two things is commonly called calibrating the equipment although technically, you are calibrating both the experimenter (for precision) and the equipment (for accuracy).

All vessels expand and contract slightly with temperature. Thus, the capacity of a particular piece of equipment depends on the temperature at which it is used. Additionally, small variations in capacity may occur during the manufacture of volumetric equipment and the actual amount of liquid delivered depends on the viscosity of the liquid and its tendency to adhere to the sides of the pipets. However, in most laboratory situations, where dilute aqueous solutions are used, a larger variation occurs through lack of reproducibility in technique of the experimenter, particularly as one learns to use the equipment. A calibration procedure thus gives information on two things: the accuracy of the manufacturer's stated calibration volume, and the reproducibility of the experimenter's technique in using the equipment. To calibrate a volumetric pipet you will weigh the amount of distilled water (at the laboratory room temperature) that you deliver from the pipet. The actual volume can be obtained from the known density of the water at that temperature. However, the apparent weight of the water will always be less than the true weight that would be obtained if the water could be weighed in a vacuum. This observation is in agreement with Archimedes' principle, which can be stated, "When an object is weighed in a fluid, the apparent weight of the object is less than the true weight, and the difference is the weight of the fluid displaced." In the particular case of weighing the water, the fluid is air. That is,

$$\text{True weight} = \text{Apparent weight} + \text{Weight of air displaced by water} \quad \text{Eq.1.1}$$

The following example illustrates a calculation involving buoyancy of air. Density data needed for the calculations are given in Table 1.1.

Example 2.1. A 10.00-mL pipet was filled to the mark with distilled water at the laboratory temperature, 22°C. The water, delivered into a tared weighing bottle was found to weigh 9.9503 g. The density of air is 0.00119 g/mL and the density of water at 22°C is 0.99780 g/mL. Use equation 1.1 to solve for true weight of water:

$$\begin{aligned} \text{True weight of water} &= 9.9503 \text{ g} + 0.00119 \text{ g/mL} \left(\frac{9.9503 \text{ g}}{0.99780 \text{ g/mL}} \right) \\ &= 9.9503 + 0.0120 \text{ g} = 9.9623 \text{ g} \end{aligned}$$

Dividing this number by the density of water gives the volume of water and the volume of the pipet.

$$\text{Volume of the pipet} = \frac{9.9623 \text{ g}}{0.99780 \text{ g/mL}} = 9.98 \text{ mL}$$

We used an approximation in calculating the weight of water in a vacuum. In order to calculate the volume of air displaced by the water, the weight of the water is required. The correction term is small compared to the apparent weight of the water by itself. Thus, a negligible error is introduced by using the apparent weight of the object, that is, 9.9503 g.

Is this a significant difference? The difference between the stated volume and the calibrated volume is 0.02 mL, or 0.2% of the stated volume of 10.00 mL. Does this volume difference matter? That is a decision that you as an experimenter must make. Sometimes it will, but most times it will not. When you are expecting a high degree of accuracy, then it does. You need to use your judgment; if it is important, then you need to calibrate the pipet, each time you choose the equipment or change the characteristics of the solution that you are using. The true "delivered volume of distilled water" of the pipet, 9.98 mL in this case, should be used in all calculations of experimental results where this pipet is used.

TABLE 1.1

Density of air = 0.0012 g mL^{-1}

Density of Water (g mL^{-1})	T($^{\circ}\text{C}$)
1.00000	4
0.99862	18
0.99843	19
0.99823	20
0.99802	21
0.99780	22
0.99756	23
0.99732	24
0.99707	25
0.99681	26
0.99654	27
0.99299	37
0.96534	90

THEORETICAL AND PRACTICAL CONCEPTS

By the end of this assignment, you should be able to

1. Define: the terms in the glossary.
2. Determine the absolute and percentage errors in additions, subtractions, multiplications and divisions.
3. Use a percentage error in a measurement to determine the number of significant figures that should be reported.
4. Calculate the percent relative average deviation and the percent standard deviation of the mean of a set of measurement.
5. Apply the critical ratio test to a set of measurements to determine if any measurement can be discarded on statistical grounds.

PROCEDURE

Calibration of a 10-mL Volumetric Pipet. At the beginning of the laboratory period, fill a wash bottle with distilled water. This will insure that the water that you will use for the calibrations will be at a constant temperature, near room temperature.

Check that your 10.00-mL volumetric pipet is clean. Clean it if necessary. Then, place about 50 mL of distilled water from the wash bottle into a beaker. Then, using pipet pump, practice transferring 10.00-milliliter aliquots of water into an Erlenmeyer flask (**refer to “Procedure 5” on page III.6 in the manual**). When you feel confident that your pipeting technique will give reproducible results, label each of three weighing bottles. Place a top on each of the bottles and weigh them on the analytical balance.

*Why do you **not** need to clean and dry the inside of these bottles? Why must the outside be dry?*

Record the weights (all digits) in your laboratory notebook. Transfer a 10.00-mL aliquot of distilled water to each of the bottles. Cover (be careful not to mix the tops) and reweigh the bottles and water. Record the temperature of the water left in the wash bottle.

ASSIGNMENT REPORTING AND DOCUMENTATION

Refer to the Course Web Site for details and guidelines on the observations, data analysis, error analysis, and conclusions sections of your report.