CHEM 231

Experiment 2

Measurements of volume

In this experiment, you will learn three common ways of measuring liquid volumes in the laboratory. These are the graduated cylinder, the volumetric pipet, and the buret. You will evaluate these methods to determine their relative accuracy and precision.

Using each method, you will make multiple measurements of a specified volume of distilled water. You will compare these measurements to an "actual" value that you obtain from the mass of the water as determined on an analytical balance. You will evaluate the deviations from the true values in order to determine the accuracy and precision of the methods.

The determination of the true value of the mass relies on knowing the density of water. Although it is approximately 1 g/mL, you should use a more precise value. In advance of lab, you should look up the density of water in the temperature range 20-25°C, in 1° intervals. This information may be available in the CRC Handbook or on internet sources. You may need to extrapolate to determine the density at these intervals. You should have these values to at least 4 significant figures. At the beginning of the lab session, your instructor will tell you the temperature of the distilled water used in the experiment. This will determine the value of the density to use throughout the procedure. Actual volumes of water will be determined from the formula m/d.

Before beginning, your instructor will demonstrate the proper use of the analytical balance as well as the correct way to measure volumes using the three techniques.

Procedure

The graduated cylinder

The general procedure will be as follows. You will use the analytical balance to determine the mass of an empty 150 mL beaker and record this mass in a table such as the one shown in this example:

Beaker mass before (g)	Beaker mass after (g)	Net mass of water(g)	Actual V = m/d (mL)	Measured V (mL)
75.1234				10.0
				10.0
				10.0
				10.0
				10.0

At your lab station, measure 10 mL of water and add it to the beaker. Record this volume in the table as the measured volume. Return to the balance and weigh the beaker again. This mass should also be entered in the next row as it will be the starting point for the second determination. The table should now appear as in the following example:

Beaker mass before (g)	Beaker mass after (g)	Net mass of water(g)	Actual V = m/d (mL)	Measured V (mL)
75.1234	85.2468			10.0
85.2468				

From the graduated cylinder

Before continuing, subtract the two masses to find the net mass of the water and use the density formula to find the actual volume. Confirm that it is at least approximately the same as the measured volume before continuing. The table should now look like this:

Beaker mass before (g)	Beaker mass after (g)	Net mass of water(g)	Actual V = m/d (mL)	Measured V (mL)
75.1234	85.2468	10.1357	10.1575	10.0
85.2468				

Ordinarily the same as the final mass in the previous row

Difference of beaker masses

Calculated using density of water

Note that all of the measured volumes will be 10.0 mL.

If, during the course of the experiment, you notice a significant outlier indicating a serious mistake in your measurement, do not use that value; in that case, you may start a new line in the table with an empty beaker. Check with your instructor if you are unsure about whether to include a value. The actual volumes should all be close to 10 mL within a reasonable tolerance.

The volumetric pipet

Your instructor will show you the proper method of using a volumetric pipet. Use a 10mL pipet for this experiment. Unlike the other two devices used in this experiment, a volumetric pipet can only measure a single volume. Proceed exactly as described above for the graduated cylinder, determining 5 measured volumes and 5 corresponding true volumes. Again, all of the measured volumes will be identical. It is appropriate to express this volume as 10.00mL. Fill out a table just like that described above.

The buret

Your instructor will demonstrate the proper method of reading and using a buret. There are two important points to take note of as he/she gives you instructions. (1) Although the burets we use are calibrated in units of 0.1mL, it is always appropriate to estimate another digit, even if this digit is somewhat imprecise; and the numbers you write down should always reflect that you have done so. If the water level is exactly on a division line, then you would indicate this by writing a 0 digit. For example, write 15.20mL instead of 15.2mL. (2) You are always to determine measured volumes as a difference between two values, a starting volume and an ending volume.

The experiment with the buret should proceed as follows. Fill the buret to a level close to the top and read the initial volume **(to the nearest 0.01mL!)**. Record this number in the table as the first starting volume. Then add exactly 10.00 mL from the buret to the weighed beaker. Record the final volume which should be exactly 10.00 mL greater than the initial volume. Weigh the beaker again in order to find the net mass of added water exactly as in the previous experiments with the graduated cylinder and the volumetric pipet. If you overshoot the 10.00 mL goal volume, you will need to empty the beaker, reweigh it and start over.

Continue in the same manner until you have made 5 volume measurements of 10.00 mL each, and have determined the net mass of water corresponding to each measurement.

Incidentally, this is an unusual way to use a buret. Ordinarily, you would not make an extraordinary effort to measure a certain given volume. When the buret is used in practice, you determine where to stop on the basis of the appearance of a solution that you are titrating, not on the basis of the buret reading. However, in this case, you are to try to measure 10.00 mL each time.

Before continuing with subsequent measurements, you should let a small (random) volume of water out of the buret into a waste container. This will assure that the next measurement does not start at the same volume as the last measurement ends. This is to avoid undesirable statistical correlations between the different volume measurements.

With these considerations, the experiment should proceed exactly as described above for the other devices. Prepare a table with 5 rows for 5 volume measurements; use the buret to add 10-mL samples to a beaker; weigh the beaker after each addition; and use the masses to determine the “true” volumes. This time, however, the measured volume will be determined by a difference of two buret readings, so it will be appropriate for the table to have two additional columns.

A sample table with the first two rows filled in with sample data is shown:

Beaker mass before (g)	Beaker mass after (g)	Net mass of water(g)	Actual V = m/d (mL)	Initial buret (mL)	Final buret (mL)	Measured V (mL)
75.1234	85.2468	10.1357	10.1575	0.13	10.13	10.00
85.2468	95.1968	9.9500	9.9213	10.35	20.35	10.00

*Notice that a little bit of water has been drained so that this is **not** the same as the final reading from the last line.*

***All** buret readings are recorded to the nearest 0.01mL.*

These numbers are differences between buret readings. This should be exactly 10.00 each time

Analysis

For each of the three devices, you now have 5 measurements of a volume each of which should be 10 mL. To evaluate the accuracy and precision of each device, you should calculate the average (also called the mean) of the 5 measurements as well as the standard deviation. The mean will tell you how close the actual volumes are, on the whole, to the target value of 10 mL. A significant difference can be an indication of systematic error in the measurement.

The standard deviation is a measure of how close the measurements are to each other. A large standard deviation indicates large variation from one measurement to another. A very precise method will have a small standard deviation.

Your instructor may choose to allow you to use your calculator or a computer spreadsheet to calculate the mean and standard deviation. Or he/she may want you to do a detailed calculation yourself. This will be discussed in class.

Interpretation

In the end it is appropriate to report the standard deviation to 2 significant figures and the average deviation the same number of places as the standard deviation. Or each device, you will have a mean (x) and a standard deviation (s). It is appropriate to report the result in the form x ± s. For example, if your calculation gives the result x=10.15832712 for the mean and s=0.231567811 for the standard deviation, you should report the result for that device as 10.16 ± 0.23, rounding s to 2 significant figures.

The mean of each set of measurements should ideally be 10 mL. How close the result is to this target value is a measure of the **accuracy** of a measurement. A large deviation from the target could indicate the presence of a **bias** or a **systematic error**.

The standard deviation is a measure of how close the measured values are to each other regardless of how close they are to the "true" value. The standard deviation gives a measure of the **precision** of a method. The lower the standard deviation, the more precise is the method.

It is possible for a method to have good precision but poor accuracy. In this case, we would find a standard deviation significantly smaller than the deviation from the target value of 10. This would indicate a strong possibility of systematic error.

We would not expect to have an accuracy significantly better than the precision. If the deviation of the mean from the target value is found to be a very small number compared to the standard deviation, it is probably just an accidental coincidence.

With these considerations in mind, use the results of your experiment to evaluate the three methods of measuring volume. Which is the most precise? Which is the least? Is there any evidence of significant systematic error? If so, to what do you attribute this error? Write a short paragraph interpreting the results which answers these questions.

Whatever you do, please **do not** simply attribute anomalies to "human error." Instead you should attempt to make some reasonable speculation as to the source of the anomaly. Using "human error" as an explanation begs the question: Who was the "human" who made the error? And if this human knew that they made an error, why didn't the human go back and do the experiment right?