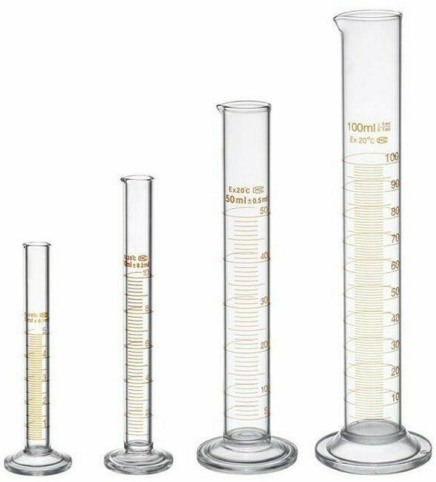
Introduction:

The purpose of this experiment is to measure different volumes of water using a number of volumetric glassware and repeated measurements in order to determine the deviation (precision) of our measurement using standard deviation and percent standard error.

Making precise and accurate measurements is an important goal of any scientist whether working in the laboratory or the field. Measurements provide the raw data that we use to calculate important quantities that we base our decisions on. For example, an environmental scientist working in the field measures the mercury level of a natural lake and based on that measurement the lake water may be considered safe for further treatment and use for drinking water or considered unsafe for such use. Reliable measuring devices must be calibrated to reflect the true value to within the precision of the measuring device. Several different volumetric glassware (different sizes and precision) are shown below.

Figure 1. From left to right, volumetric pipets, volumetric flasks, a 50-mL buret (graduated), 10-mL Mohr pipet (graduated)



Note: A graduated glassware's precision is determined to be "half" of its smallest graduation. To get this precision (uncertainty) value, determine the value of the smallest graduation mark (also known as a tick mark) and divide it by 2 and express the result to 1 significant figure. This is the estimated (uncertain) digit or the last significant figure of a measurement. Due to the small tick size and our eye sight limitation, we cannot read the estimated digit better than the "half-way" estimated point using our naked eye.

Figure 2. Some common sizes of graduated cylinders, 10,25,50,100-mL.

Figure 2. An analytical scale (left) and a centigram scale (right)

Mass measurements are often among the most precise of our lab measurements. A digital scale is used for this purpose. Scales (balances) are placed on a level surface and calibrated for accuracy using a known standard mass. There are two types of balances a centigram and an analytical balance. Centigram scale measures down to 0.01 g (1 centigram) and an analytical balance measures masses down to 0.0001 g (0.1 milligram). When working with these scales follow these steps:

1. Never spill any liquid chemical on a scale. Small quantities of spilled solid powder sometimes happen and should be immediately cleaned with a brush.
2. Never move a scale. They are leveled and calibrated. If the scale is moved it will lose its calibration (and accuracy).
3. Never weight objects directly on the pan. Always use a clean and dry plastic weigh-boat or a small beaker.
4. *Pay attention to the significant figure of the quantity you are instructed to measure. For example, if the instructions call for measuring 1.42 g on an analytical scale any mass between 1.415-1.424 g will do since they all round to 1.42 g. Pouring too much chemical into the weigh boat and then trying to remove some is not an acceptable practice and likely to cause errors in your mass measurements. It is very difficult and a waste of time (and likely to produce errors) if one tries to weigh a mass equal to 1.4200 g.*
5. *Be super careful not to spill any liquids on the scale when weighing liquid chemicals using a glassware container.*
6. *You can use the tare feature of a scale to electronically "zero" the mass of the container if you are instructed to weigh a certain mass of a chemical. After taring the empty mass of a weight-boat or a small beaker, the mass of the chemical added is directly displayed on the digital scale.*
7. *When working with an analytical scale, close all the sliding glasses with the empty container on the pan to tare the mass. Next open one of the sliding glasses and carefully transfer the chemical to the weigh-boat and then <u>close</u> the glass door and record the displayed mass.*
8. *Be sure to use a clean and dry spatula to transfer a powder (solid) sample and avoid any contamination of the original chemical bottle.*
9. *Use the same scale throughout your experiment.*

<u>Precision and accuracy errors in our measured and calculated data</u>

We will measure different volumes of water using a number of volumetric glassware and repeated measurements in order to determine the deviation (precision) of our measurement using standard deviation and percent standard error. In order to determine a precise measure of the volume we just measured we will

use the density of water (at the measured water temperature) and mass of water to calculate the true value of the measured volume and determine percent error of our volume measurement using the following equation in which the answer is generally expressed in 2 significant figures.

$$\%\ Error = \frac{|experimental\ value - true\ value\ |}{true\ value} \times 100$$

Repeated measurements must have an acceptable level of deviation which impacts the precision of the measuring device. We will determine the precision error of the measuring device using a percent relative standard deviation calculation. What is standard deviation?

Standard deviation (SD) is an important statistical parameter that predicts the chances of repeating a measurement that falls within a specified deviation from the mean value shown as μ in Figure 1. For example, statistical analysis shows that for normal distribution of measurements the following standard deviation criteria applies. There is 1 68.27% chance that repeated measurements of the same property fall within 1 sigma (σ) of the mean value. We use the formula shown to calculate standard deviation.

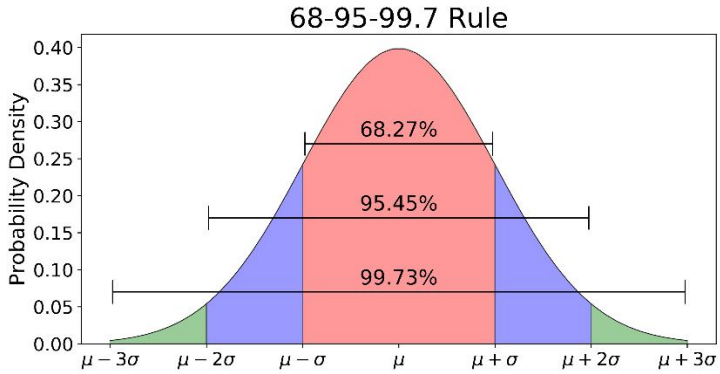


Figure 1: Standard deviation and normal distribution of measured data

$$\boldsymbol{\sigma} = \sqrt{\frac{\sum(\boldsymbol{x} - \boldsymbol{\mu})^2}{\boldsymbol{N} - \mathbf{1}}}$$

Where the fraction inside the square root represents the "sum of squared deviation" of each data, $\boldsymbol{x}$ from the mean (average) value $\boldsymbol{\mu}$, divided by the number of data sets $\boldsymbol{N}$, minus 1. You can do the calculation by hand, use the statistical feature of your scientific calculator or use a built-in function to calculate standard deviation in Excel by entering it as: "=STDEV(data range)". Commonly, we report the calculated 1 sigma (1 $\boldsymbol{\sigma}$) deviation interval as ± 1 $\boldsymbol{\sigma}$**.** Generally, standard deviation is expressed in 1 significant figure which represents the last meaningfully precise digit of the average value of a measurement. Another common method of reporting deviation is to report it as percent relative standard deviation (% RSD). We use the following formula in which the answer is generally expressed in 2 significant figures.

$$\%\ RSD = \frac{\sigma}{\mu} \times 100$$

If a repeated data set has a high % accuracy error and low precision error, the situation points to a "one directional" or **systematic error** caused by the measuring equipment lacking proper calibration. For example, a scale may be consistently reporting a deviation from the true value of mass because it lacks proper calibration. A pipet that has permanently expanded due to heat will deliver inaccurate but consistent volumes (on the high side!). Such situations can be fixed by recalibrating the measuring device if we deal with digital electronic devices. Volumetric glassware cannot be recalibrated and should be kept away from hot and cold environments. Every measurement involves a degree of operational (ex. a human operator) or **random errors**. Random errors can be minimized by careful execution of lab techniques. A high degree of random errors creates a poor precision error and makes the measurement and calculated data unreliable and non-repeatable.

Equipment, Glassware & Chemicals

- Deionized (DI) water
- 10-mL graduated cylinder
- 50-mL graduated cylinder
- 10-mL pipet
- 5-mL Mohr pipet
- 25-mL buret
- Pipet bulb or pump
- Wash bottle
- Thermometer

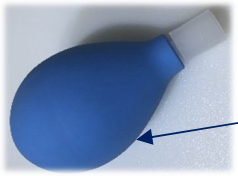
Important Notes:

- Examine your thermometer. If the colored liquid gaps in it do not use the thermometer and contact your instructor. The colored liquid should be continuous.
- Work individually
- All measurements should be read and recorded in your data tables to the full precision of the measuring equipment/glassware that you are instructed to use.

General instructions for proper use of a pipet:

Note: Never use a pipet for cold or hot liquids since pipets are calibrated at a specific temperature near room temperature (see the label on a pipet) and expansion or contraction of the glass upon heating or cooling will alter its volume and destroy the factory calibration.

1) Use a pipet pump or bulb to create the necessary suction to draw the liquid inside the pipet.
2) Rinse the pipet 3 times with DI water and discard.
3) Rinse the pipet 3 times with the liquid you are using and discard in the waste container.
4) If you are using a volumetric pipet, fill the pipet to the mark. If you are using a pump, push on the release level to allow the liquid to drain or if you are using a bulb, remove the bulb and let the liquid drain.
5) If you are using a Mohr (graduated) pipet fill the pipet to the zero mark and release the pressure to let the liquid drain to a labelled volume. Do not let the pipet drain completely because the volume beyond the last graduated tick mark is not reliable.
6) Be sure to touch the tip of pipet to the inside glass walls once <u>after</u> drawing the desired amount of liquid and once <u>after</u> delivering it in the receiving container. This will dislodge a hanging drop (that is accounted for) into the transfer container.

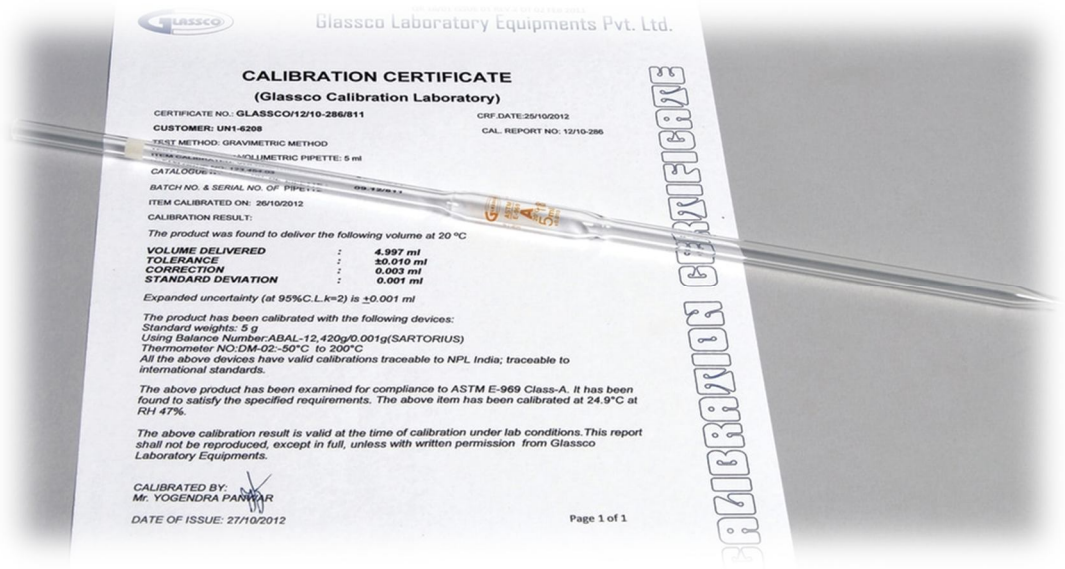


A pipet bulb works by squeezing the bulb to create suction for the liquid draw.

A Pipet pump works by turning this wheel to create suction.

Press here to equalize pressures and let the liquid in the pipet flow out completely (volumetric pipet) or to a specific volume (Mohr Pipet).

5-mL Mohr pipet



Above is a 5-mL volumetric pipet that has been calibrated at the factory with the following specifications stated on its calibration certificate:

Calibration temperature: 24.9 °C but calibration data is corrected to 20 °C
Volume delivered: 4.997 mL (based on mass measurement and true value of density)
Tolerance: ± 0.010 mL (uncertainty in measuring the 5-mL)
Correction: 0.003 mL
Standard Deviation: 0.001 mL

Blank page

Procedure

Note: Read and understand every step completely before executing the step. Record all measured data into your laboratory notebook in pen!
NOTE: Use an analytical balance (0.1 mg precision)

1. Check out a 25-mL buret, a 10-mL Mohr pipet and a pipet bulb or pipet pump whichever pump style you prefer. A pipet pump is easier to work with.
2. Weigh a clean and dry 125-mL Erlenmeyer flask. Note that the outside of the flask must be dried using a paper towel before weighing flask. Check to see if the balance pan is also dry. If you see any water on the balance pan notify your instructor immediately.
3. Mount the 25-mL buret on a buret stand securely.
4. Empty and fill a squeeze wash bottle with DI water. Measure the temperature of the water by inserting a clean thermometer in it.
5. Use the wash bottle to fill the buret to about ¼ of its volume. Drain completely. Repeat this process 2 more times to clean your buret.
6. Fill your buret to near the top. Place a waste liquid 50-mL beaker under the tip. Remove an air gap that is often caught at the stopcock by opening the valve completely (and quickly) for 1-2 seconds. Now set the volume of the water in the buret to 0.00 mL. Remove any hanging drop from the tip by touching the tip of buret to the side of a 50-mL beaker.
7. Transfer a volume of water between 4-5 mL to your pre-weighed 125-mL flask by opening the buret valve and closing it when water level is somewhere between 4-5 mL <u>randomly</u>. Don't try to get the water level to represent a particular volume reading. Final buret volumes should be read precisely after each trial. Also, after dispensing a volume of water, touch the tip of the buret to the side of the flask to dislodge any portion of a drop that may be hanging at the tip of the buret.
8. Read the meniscus level (lowest point) of water level inside the buret to the precision of buret. (don't forget to record all your measurements!)
9. Weigh the 125-mL flask using the same balance. Continue using the same flask including the water from previous trial(s) for the following steps. Weighing by difference will determine the mass of the next water sample. Please note that the final mass of the flask for one trial is the initial mass for the following trial.
10. Repeat steps 7-9 three more times to obtain 4 volume and mass readings.
11. **Next use a 50-mL graduated cylinder**. Rinse this measuring device with small portions of DI water 3 times to clean it. Obtain the precision of this measuring device.
12. Empty the 125-mL flask, dry the outside of the flask and obtain and record its initial mass.
13. Fill the graduated cylinder to a level between 5-10 mL and record the volume.
14. Transfer the measured water into your 125-mL flask and reweigh the flask.
15. Repeat steps 13-14 three more times.
16. **Next use a 10-mL graduated cylinder** and rinse it 3 times with water.
17. You will be following the same procedure as you did with the previous graduated cylinder. Repeat steps 12-14 for a total of 4 times.
18. Review the general procedures for use of a pipet as provided in the introduction.
19. **You will be using a 5-mL Mohr pipet.** Obtain the precision of this measuring device
20. Rinse the pipet 3 times with water by using a pipet pump or bulb. <u>Make sure that no water gets into the bulb or pump</u>.

21. Empty the 125-mL flask, dry the outside of the flask and obtain and record its initial mass.
22. Use your pipet to measure a volume between 4-5 mL. When you are ready to transfer the water from the pipet into the flask, make sure that you do not let the water drain past the last graduation of the Mohr pipet causing a volume error. Be sure to touch the tip of pipet to the inside glass walls once after drawing the desired amount of liquid and once after delivering it in the receiving container. Don't forget to record the flask mass and pipet volume.
23. Repeat step 22 to obtain mass-volume data for 4 trials.
24. Empty the 125-mL flask, dry the outside of the flask and obtain and record its initial mass.
25. **You will be using a 10-mL volumetric pipet**. Obtain the precision of this measuring device
26. Use your pipet to transfer 10 mL of water into your 125-mL flask each time. When you dispense 10-mL let the water drain completely by gravity. To equalize pressures and let the water flow, remove the bulb or press the release lever on the pipet pump while touching the tip of pipet to the inside top glass wall of your 125-mL flask.
27. Repeat step 26 for a total of 4 mass-volume measurements.
28. You have now completed the experiment. Start your data analysis.

Data Analysis

1. Calculate masses of water for each trial and each measuring glassware used.
2. Look up the expected density of water at the temperature of experiment and record the value in your table.
3. Calculate the density of water for each trial and each measuring glassware. Pat attention to the significant figures that the data should be reported to.
4. Calculate average densities, standard deviation and % relative standard error for densities using each measuring device.
5. Examine your data summary table values.
    a. Order your measuring devices from least accurate to most accurate.
    b. Order your measuring devices from the least precise to most precise.
    c. Do you have a density data situation in which a precise volumetric glassware exhibits a high precision error? Examine the calculated uncertainty (standard deviation) and compare to the precision of the measuring device.
6. Fill out the following table expressing the precision error of each measuring device as an uncertainty range involving 1 standard deviation (1 sigma or 68% probability range). State whether using each of these measuring devices you could accurately state the density of water to within its precise range.

Table 5. Data Summary (1 sigma precision analysis Precision range

Density Data Trial ------->	Avg	1 sigma	Low	High	True value	Within range?
25-mL buret						
50-mL grad Cylinder						
10-mL grad Cylinder						
5-mL Mohr pipet						
10-mL vol. pipet						

Table 1. Accepted Density

Water Temperature, °C:		C
Accepted Density of Water:		g/mL

Table 2. Mass data (grams)

	Initial	Final	Initial	Final	Initial	Final	Initial	Final
Mass data, Trial------->	1	1	2	2	3	3	4	4
25-mL buret								
mass, g (calculated)								
50-mL grad Cylinder								
mass, g (calculated)								
10-mL grad Cylinder								
mass, g (calculated)								
5-mL Mohr pipet								
mass, g (calculated)								
10-mL vol. pipet								
mass, g (calculated)								

Table 3. Volume data (milliliters)

	Initial	Final	Initial	Final	Initial	Final	Initial	Final
Volume data, Trial------->	1	1	2	2	3	3	4	4
25-mL buret	0							
volume, mL (calculated)								
50-mL grad Cylinder	0		0		0		0	
volume, mL (calculated)								
10-mL grad Cylinder	0		0		0		0	
volume, mL (calculated)								
5-mL Mohr pipet	0		0		0		0	
volume, mL (calculated)								
10-mL vol. pipet	0		0		0		0	
volume, mL (calculated)								

Table 4. Data Summary Table (Density: g/mL)

					Accuracy		Precision	
Density Data Trial ------->	1	2	3	4	Avg	% Error	Std Dev	%RSD
25-mL buret								
50-mL grad Cylinder								
10-mL grad Cylinder								
5-mL Mohr pipet								
10-mL vol. pipet								

Blank page

Pre-laboratory Assigment (Show all work!)

1) Describe the difference between accuracy and precision.
2) A student obtains the following data for repeated density measurements of liquid ethanol. (accepted value for density of ethanol is 0.78 g/mL)
   (g/mL: 0.73 , 0.75, 0.72, 0.75)
   a) Calculate the average density.

   b) Calculate the % accuracy error.

   c) Calculate the standard deviation (1 sigma).

   d) Calculate the percent precision error (%RSD).

   e) Does the true value fall within 1 standard (1 sigma) deviation of the mean?

   f) Are we dealing with a significant case of systematic or random error? Explain.

Post Laboratory Problems

1) In a certain situation repeated measurements of a property are within the experimental precision of the measuring device used, but results suggest a high percentage of error. What could have gone wrong?

2) Explain the potential cause of each data scenario listed:
    a. A low percent accuracy error and a high percent precision error.

    b. A low percent precision error and a high percent accuracy error.

3) Which one of the situations listed in the previous problem can be fixed? How do we fix it?