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Laboratory 1
(SCM)

Scientific Measurements

Objectives

- **Explore:** Practice using laboratory balances and glassware to understand the uses and limitations of each and be able to choose appropriate lab equipment.
- **Explain:** Analyze the results and explain variation in measurements by using the concepts of precision and accuracy, significant figures, and determinate and indeterminate errors.
- **Apply:** Justify your choice of different laboratory resources, like glassware and balances, for use in future experiments.

Introduction

Two chemistry students are in a heated argument. Pete feels that "scientific experiments are done in a controlled manner and are reproducible if done correctly. Science cannot progress if measurements give different results every time." But Claire responds that "scientists should take multiple measurements. Results are not always the same and it is wrong to simply perform one test and report the answer."

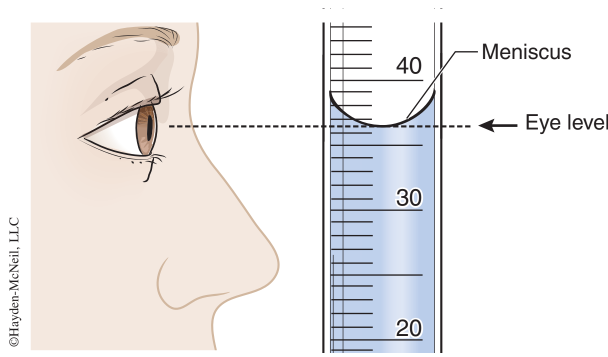
Who do you think is right? If measured values are always the same, then why take more than one measurement? If measured values are different, does that mean that science is not reproducible? Or are "true" values never truly known?

In this experiment, you will learn more about scientific measurements by investigating the density of water at room temperature and (perhaps) resolve this dilemma. In this process you will also gain familiarity and proficiency with several important laboratory resources including laboratory glassware and balances.

Table 1.1 Summary of properties measured in this experiment.

Property	What Is Being Measured?	How Is It Measured?	Additional Information
Mass	The amount of matter in a sample.	Top-loading balance Analytical balance	Common units are grams (g) and milligrams (mg). Our top-loading balance has a maximum load of 610 g and a precision of +/– 0.01 g. The analytical balance has a smaller maximum load (120 g) and is used for more careful measurements; the precision is +/– 0.0001 g.
Volume	The amount of space an object occupies.	Measuring the dimensions of a solid (e.g., with a ruler). Water displacement for irregularly shaped solids. Liquids are measured in glassware.	Common units are cubic centimeters (cm^3), liters (L), and milliliters (mL). By definition, 1 L = 1000 mL = 1000 cm^3. For precise measurements, burets and pipets are used. Graduated cylinders and beakers are useful when a careful measurement is **not** necessary.
Temperature	The average kinetic energy of the particles (atoms, molecules) comprising matter.	Thermometer	When something becomes warmer, the kinetic energy of its submicroscopic particles increases. As it cools, the kinetic energy decreases. For nearly all materials, an increase in temperature (and hence motion) causes the material to expand. The temperature tells how warm or cold an object is relative to a standard. In the lab we report temperature on the Celsius (°C) or Kelvin (K) scales.
Density	The ratio of an object's mass to its volume.	Calculated from mass and volume data.	Usually reported in g/cm^3 or g/mL.

Reading Scale Values



The volume of a liquid is measured in graduated (marked) glassware by recording the position of the bottom of the meniscus (the curved surface of a liquid in a tube or cylinder). As shown in the figure, it is important to align your eye perpendicular to both the glassware and the bottom of the meniscus in order to obtain an accurate reading. In this example a reading of 36.5 mL is appropriate. **When reading a scale value the number you should record includes all of the digits that are known, plus one that is estimated.** In this example, the bottom of the meniscus is clearly greater than 36.0 and less than 37.0; a reasonable estimate is 36.5 mL. Graduated laboratory glassware, like burets, graduated cylinders, pipets, and thermometers are read in this manner.

Calculations

Dimensional analysis is a common problem-solving approach and in this experiment it can be used for density calculations. Conversion factors are used to ensure that the answers to problems are in the correct units. In this method both the numbers and the units are multiplied together, divided into each other, or canceled out. The end result will be a number and a unit.

Example: A student weighed a marble to obtain a mass of 4352.4 mg and measured a volume, by displacement, of 2.31 mL. What is the density in g/cm³ of the marble?

The question is asking for density, which is mass divided by volume, in units of g/cm^3. The measurements give us the mass and volume, but not in the units we need. We can use conversion factors with dimensional analysis to solve this problem:

$$\text{Density} = \frac{\text{Mass}}{\text{Volume}} = \frac{4352.4\ \cancel{\text{mg}}}{2.31\ \cancel{\text{mL}}} \times \frac{1\ \text{g}}{1000\ \cancel{\text{mg}}} \times \frac{1\ \cancel{\text{mL}}}{1\ \text{cm}^3} = 1.88\ \text{g/cm}^3$$

Question 1.1: The student then decided to cool the same marble and see if the density changed. After analysis, the density of the colder marble was determined to be 1.91 g/cm^3. What are the mass and volume of the cold marble?

Experimental Measurements

Precision and accuracy are commonly mentioned in discussions of experimental measurements. ***Precision*** refers to how closely individual measurements agree with one another and ***accuracy*** refers to how closely a measured value agrees with the accepted value.

Question 1.2: A chemist analyzed a liquid sample multiple times by two different methods. The sample has an accepted density of 0.9855 g/mL.

Measurement #	Method A	Method B
1	0.9834	0.9855
2	0.9844	0.9754
3	0.9838	0.9847
4	0.9843	0.9799

Which method has better precision?

Which method has better accuracy?

The chemist decided that Method A was better than Method B (do you agree?) and reported an average value of 0.983975 g/mL. This may seem like a good idea, but clearly their laboratory equipment can only determine the density to the 0.000$\underline{1}$ place. It does not make sense to report an average value 0.983975 g/mL because it implies a much more precise measurement than the one possible with the available equipment. It would be possible to determine a density value to this level of precision, but it would require more sophisticated (and expensive) laboratory resources.

It is important to properly use laboratory resources to obtain and report the maximum number of **significant figures**, which are the digits that indicate the **accuracy** of the instrument used to make the measurement. For example, a mass reading on an analytical balance of 12.0409 g has six significant figures. This number tells you the mass from this balance is known exactly to the 3rd decimal place and the 4th decimal place is an estimate. Measuring the same object on a top-loading balance in our labs would result in only four significant figures. For example, a mass of 12.03 g, which would indicate it's known exactly to the first decimal place (the tenths place) and the "3" in the 2nd decimal place (the hundredths place) is an estimate. This tells us this balance can measure a mass exactly to the tenths place and is estimating the mass in the hundredths place. You should be able to explain this based on the accuracy of the instruments as listed in the table above. Your textbook has more information on the calculation of significant figures.

It should be recognized that the reported number of significant digits encompasses all measurements in a given procedure. Often, a single piece of glassware or instrumentation will limit the number of significant digits that can be obtained.

Returning to the above example, you will notice with Method A 1) the measured density value is consistently less than the accepted value, and 2) repeated measurements produced different density values. The idea of **errors in measurement** may be used to describe both of these.

Errors in measurement fall into two categories: determinate errors and indeterminate errors. A **determinate error** is one that has an identifiable source and can be corrected for. Examples of determinate errors include using a meter stick that is actually 1.020 meters long or reading the top of the meniscus instead of the bottom. Both of these errors can be identified and corrected. Note that determinate errors will have a directional bias. Using a "meter" stick that is 1.020 meters long will always result in an error that understates the actual length. It is likely that Method A has a determinate error that leads to a result consistently less than this value.

Indeterminate errors are ones that *cannot* be identified and corrected. For example, in our effort to obtain the maximum number of significant figures we estimate the last digit when reading a scale value. Sometimes the estimate might be too high, other times too low. In either event, it is not possible to estimate the last value in a reproducible way every time. This results in unavoidable variation in the reported value. Even when a value is recorded from a digital instrument (like with a pH meter or analytical balance) there is unavoidable uncertainty in the last digit. This means that, even if determinate errors are absent and the procedure followed flawlessly, there will be indeterminate errors that lead to variation in the measured values. Indeterminate errors will always lead to a range of measured values and they will not display the directional bias of a determinate error. If only indeterminate error is present, taking multiple measurements and averaging the results is a good way to approximate the true value. The fact that Method A did not always provide the same measured value is consistent with the unavoidable presence of indeterminate errors.

Inherent errors are errors due to the way the experiment is being performed or the equipment provided. These are errors which are generally beyond your control. These can be either indeterminate or determinate errors.

Finally, please note that a scientist's treatment of error differs from how others view error. For a scientist, "error" is not forgetting to close the door or writing down the wrong number; if these mistakes occur, the experiment should be started over! Instead, errors are an inevitable part of measurement. To have the very best precision and accuracy it is important to identify and remove determinate errors and reduce the magnitude of indeterminate errors.

Discussion

Pure water has a well-established **density** (mass/volume ratio) that depends on its temperature; the accepted value for the density being 1.000 g/cm^3 at 4 °C. For the range of temperatures that are typically associated with "room temperature" (about 20 °C to 30 °C) the density of pure water is given by the following equation, determined experimentally:

$$density\left(\frac{g}{cm^3}\right) = \left(-0.00030\frac{g}{^{\circ}C\ cm^3} \times temperature\ (^{\circ}\mathrm{C})\right) + 1.0042\frac{g}{cm^3} \qquad (1)$$

Suppose a chemist wishes to determine which type of glassware would be the most accurate and with which they could be the most precise in delivering specific volumes of liquids. Using water, they can measure the volume of the water with various types of glassware and its corresponding mass. If they know the temperature at which the volume is measured, they can determine the density of water and compare it to its actual density using the equation above. Whichever glassware gives the better density should be the best to use when delivering a liquid to a vessel.[1]

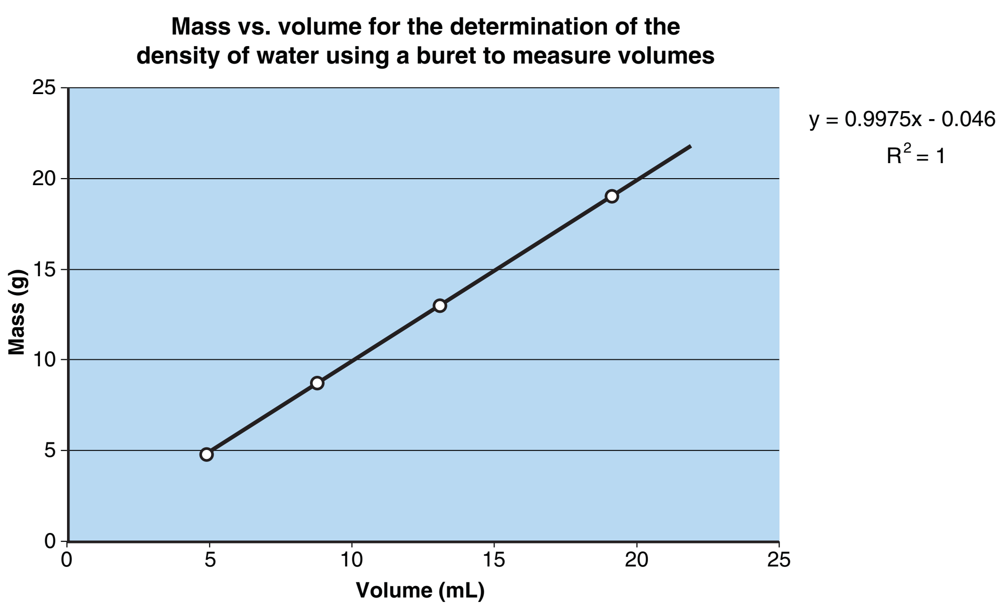
With this information, you can compare the experimental density of water determined using the laboratory glassware to the actual density calculated from the equation. You can calculate the **percent error** associated with these measurements using the following equation:

$$percent\ error(\%) = \frac{\textbf{\textit{measured}}\ density\ (\mathrm{g/cm^3}) - \textbf{\textit{actual}}\ density\ (\mathrm{g/cm^3})}{\textbf{\textit{actual}}\ density\ (\mathrm{g/cm^3})} \times 100\% \quad (2)$$

Remember, the actual density is determined by the equation given previously, and the measured density is that measured by the chemist using the different types of glassware. You will also make graphs to determine the measured density and see how systematic and random errors present themselves in a graph. A rough sample graph is shown below. For additional resources to help you determine the correct number of significant figures to report for your calculations, see Appendix F: Treatment of Numerical Data.

Experimental Design (Procedure)

In this experiment, your task is to prepare plots of mass/volume for distilled water at room temperature using four different pieces of laboratory glassware (see Appendix D about how to use a pipet and buret). As seen in the following figure, each mass/volume plot should have **four different measured values spaced out at about 5-mL intervals between 5 mL and 20 mL** (except for the beaker, which has volumes in mL of around 20, 25, 30, 35). The mass will be measured using an analytical balance (see Appendix E about using the balances).



All data must be recorded directly into your notebook, not just summarized in the report table.

[1] This assumes the mass measurement is at least as accurate as the volume measurements, which, in this case, is an appropriate assumption given the analytical balances provided in the lab.

1. At the start of the experiment the Teaching Assistant may demonstrate the correct use of the 25 mL buret, the graduated cylinder, the pipet, and the beaker. They will also communicate the correct operation of the laboratory balance and thermometer.

2. As mentioned above, a liquid's density will be affected by temperature. Impurities in a liquid may also affect the density. For these reasons begin the experiment by adding ~ 240 mL of distilled water to a 250 mL beaker and letting it reach room temperature. You will use this water in subsequent steps. Record the temperature of the water before using each type of glassware. Average these four temperatures and use it as the water temperature in the data table and in the calculation of the actual density using equation (1) on page 11.

3. Use an analytical balance to determine and record the mass of a 100 mL beaker. This is the "weighing beaker." Remember to tare the balance before the measurement. *In your notebook* record all digits; they are all significant.

4. Fill the 25 mL buret with the water from step 2 and *in your notebook* record the initial reading to the correct number of significant figures. Remember, for a buret this is reading and estimating to the hundredths place, e.g., 1.03 mL or 0.07 mL.

5. Transfer at least 5 mL of water to the weighing beaker. Record the buret's final reading in your notebook to the correct number of significant figures. The final and initial readings will be used to calculate the volume transferred. Also, record the temperature of the water. Liquid must NOT be transferred in the balance room.

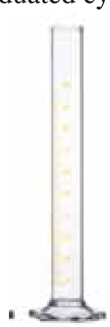
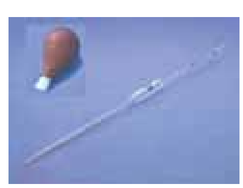
6. Return to the balance room and record the new mass of the weighing beaker with water *in your notebook*. Use the same balance for steps 3 and 5, remembering to tare the balance before each measurement. The mass of the empty beaker and beaker + water will be used to calculate the mass of water transferred.

7. Repeat this procedure until you have four different values that are between 5 mL and 20 mL for the buret. Your results will be similar to those shown in the figure.

8. Move on to the next piece of glassware and repeat steps 3 through 6. After finishing with the buret, you should move on to the graduated cylinder, then the pipet, and finally the beaker (use volumes of 20, 25, 30, and 35 mL for the beaker).

9. After completing data collection, in your notebook reproduce the data table (shown on page 15) and begin filling it in. It is important to use the correct number of significant figures in the data table! Be sure to have your TA check your work at this point. You may need to collect additional data if your results are anomalous.

Glassware to use:

25-mL buret	Graduated cylinder	Volumetric pipet	Beaker
			

Balances available in lab: **Thermometer:**

Top-loading balance	Analytical balance	
		

Materials Provided

Equipment
2–100-mL beakers
1–250-mL beaker
1–400-mL beaker
1–600-mL beaker
2–125-mL Erlenmeyer flasks
3–250-mL Erlenmeyer flasks
1–25-mL buret and buret clamp
1–10-mL graduated cylinder
1–100-mL graduated cylinder
1–5-mL volumetric pipet
1–10-mL volumetric pipet
rubber pipet bulb
1–thermometer (–20 °C–100 °C)

Common Equipment
analytical balances
Parafilm (covers glassware)

Chemicals
distilled water, H_2O

Cautions

Goggles must be worn in the laboratory.

Procedure

You are responsible for recording your procedure in your notebook. You should provide a sufficient level of detail such that another scientist could repeat your experiment.

Waste Disposal

All solutions may be rinsed down the drain.

Scientific Measurements[1]

Average Water temperature (°C): ______________

Water, accepted density at room temperature (calculated)[2] (g/cm³): ____________

Mass of Beaker (g): ______________

Glassware	Mass of Beaker + Water (g)	Mass of Water (g)	Volume of Water (mL)	Measured Density (g/mL)	Error[3] (g/mL)	% Error (g/mL)
Buret						
Buret						
Buret						
Buret						
Average	—	—	—			
Graduated Cylinder						
Graduated Cylinder						
Graduated Cylinder						
Graduated Cylinder						
Average	—	—	—			
Pipet						
Pipet						
Pipet						
Pipet						
Average	—	—	—			
Beaker						
Beaker						
Beaker						
Beaker						
Average	—	—	—			

[1] Use the correct number of significant figures throughout.

[2] At 4 °C water's density is 1.000 g/cm³. For the range of temperatures associated with "room temperature" (from about 20 °C to 30 °C), the density of pure water is: Density (g/cm³) = [–0.00030 (g/°C · cm³) × Temperature (°C)] + 1.0042 (g/cm³)

[3] **Error** = experimental value – true value. The **relative error** = error/true value. The % error = relative error × 100%. Include the sign for error and % error. To get the average values for error and % error, use the absolute values.

Report Details

1. Reproduce your completed data table. Be sure to use the correct number of significant figures.

2. Prepare and submit four graphs of mass/volume. Each graph should have a descriptive title that includes what's being graphed and the glassware employed, the axes should be properly labeled (including units and proper significant figures), and a linear best-fit line (y = mx + b format) and the R^2 value (correlation coefficient—gives an idea about the quality of the fit of the line to the data points) reported on the graph. Use Excel (available free to all students from the university) for preparing the graphs. A best-fit line averages out the random errors in the data.

Report Questions (give concise explanations)

1. (2 pts) Suppose an experiment called for the transfer of 17.50 mL of a liquid, and precision and accuracy of the volume is important. Which piece of glassware would be the most appropriate to use? Explain your reasoning and use supporting data from this experiment.

2. (3 pts) When using a pipet, a student repeatedly makes several transfers with the top of the meniscus level with the mark instead of the bottom of the meniscus. As a result, all of the volume transfers are 0.05 mL smaller than the actual volume.

    a. Is this a determinate or indeterminate error? (Give a short explanation.)

    b. Would the actual density be greater or less than the calculated density? (Give a short explanation.)

    c. Does this affect the precision, accuracy, both, or neither? (Give a short explanation.)

3. (2 pts) In this investigation you used a buret, graduated cylinder, beaker, and pipet to transfer water. Based on your percent errors, answer the following questions. (Give a short explanation for your answers.)

    a. Which of these resulted in the most precise measurement?

    b. Which of these resulted in the least accurate measurement?

4. (2 pts) For a particular type of glassware, if all the errors had the same sign, would this likely indicate a determinate or indeterminate error? (Give a short explanation.)

5. (6 pts) Based on the graphs, answer the following questions. (Give a short explanation for your answers.)

    a. How might indeterminate errors appear?

    b. What should be the numeric value of the y-intercept?

    c. The mass of water should be plotted on the y-axis. If instead one plotted the total mass of beaker and water, would this affect the slope, y-intercept, or both?

    d. Is the error in part (c) a determinate or indeterminate error?

e. Would the error in (c) affect the density determined from the graph?

f. Which type of glassware gives the most accurate result for the density, and does this agree with the most accurate type of glassware from the calculations in the table?

6. (3 pts) Understanding how errors could affect your results is important. (Give a short explanation for your answers.)

   a. Would not wiping off any liquid on the outside of the tip of a buret or pipet be an inherent or human error?

   b. If the top-loading balance had been used to measure the mass would this be an inherent or human error?

   c. Would using a 5-mL pipet twice to measure 10-mL of liquid produce more or less error than using a 10-mL pipet once?

Scientific Measurements

Answers to In-Report Questions

Question 1.1: A student weighed a marble to obtain a mass of 4352.4 mg and measured a volume, by displacement, of 4.31 mL. The resulting density was 1.88 g/cm^3. The student then decided to cool the same marble and see if the density changed. After analysis the density of the colder marble was determined to be 1.91 g/cm^3. **What are the mass and volume of the cold marble?**

Answer

The density = 1.91 g/cm^3. This is a ratio of mass/volume. In the original question the mass = 4352.4 mg and the volume = 2.31 mL. With a change in temperature will the mass change or the volume? It must be the volume because the amount of matter in the marble has not changed, only the amount of space it takes up.

Mass = 4352.4 mg

$$\frac{\text{Mass}}{\text{Density}} = \text{Volume} = \frac{4352.4 \text{ mg}}{1.91 \text{ g/cm}^3} \times \frac{1.000 \text{ g}}{1000 \text{ mg}} = 2.27 \text{ cm}^3$$

Question 1.2: A chemist analyzed a liquid sample multiple times by two different methods. The sample has a true density of 0.9855 g/mL.

Measurement #	Method A	Method B
1	0.9834	0.9855
2	0.9844	0.9754
3	0.9838	0.9847
4	0.9843	0.9799

Which method has better precision?

Answer

The range of values for method A is much narrower. Method A has better precision.

Which method has better accuracy?

Answer

The true value = 0.9855.

The average for method A = 0.9840. The average for method B = 0.9814.

Method A has better accuracy. Note: It is difficult to determine the accuracy if the precision is poor as the results will be widely scattered. Simply calculating an average may be misleading under these circumstances.