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Experiment #1: Measurements involving Mass, Volume, and Statistical Analysis of Data

The error of a measurement is defined as the difference between the experimental and the true value. This is often expressed as percent (%) error, which is calculated as:

$$\text{Percent Error} \; = \; \frac{|\text{Experimental} - \text{True}|}{\text{True}} \times 100 \; \% \tag{1}$$

Note: At times a true value may not be known or possible. If two experimental values are determined and the true value is unknown, then the percent difference may be calculated. The percent difference is the absolute value of the difference between the two experimental values divided by the average value and multiplied by 100.

$$\text{Percent Difference} = \frac{|\text{Value 1} - \text{Value2}|}{(\text{Value 1} + \text{Value 2}) \, / \, 2} \times 100 \; \% \tag{2}$$

In chemical measurements, we try to eliminate errors, which can be divided into two broad types: systematic and random. *Systematic error* occurs regularly and predictably because of faulty methods, defective instrumentation, and/or incorrect assumptions. *Random error* is more difficult to define and is governed by chance. Examples include a weighing error due to air currents near a balance and line current fluctuations for electronic instrumentation. Systematic errors always affect the measured quantity in the same direction, while random errors can make the measured quantity either too large or too small.

Accuracy is the closeness of agreement between a measured value and the true (or accepted) value. True values can never be obtained by measurement. However, we accept values obtained by skilled workers using the best instrumentation as true values for purposes of calculation or for judging our own results.

Precision describes the reproducibility of our results. A series of measurements with values that are very close to one another is a sign of good precision. It is important to understand, though, that good precision does NOT guarantee accuracy!

The *standard deviation* of a series of measurements including at least 6 independent trials may be defined as follows: let x_m represent a measured value, n be the number of measurements, and $\bar{x}$ be the average or mean of the various independent trials or measurements. Then d is the average deviation:

$$d = x_m - \bar{x} \tag{2}$$

and the standard deviation, s, is defined by:

$$s = \sqrt{\frac{\sum d^2}{n-1}} \qquad\qquad (3)$$

where $\sum d^2$ refers to the sum of all the values of d^2.

The value of any measurement should include some indication of the precision of the measurement. The standard deviation is used for this purpose if a large number of measurements of the same quantity is subject to random errors only. We can understand the meaning of s if we plot the number of times a given value of x_m is obtained (on the y-axis) versus the values x_m (on the x-axis). Such a *normal distribution curve* is bell-shaped with the most frequent value being the average value $\bar{x}$.
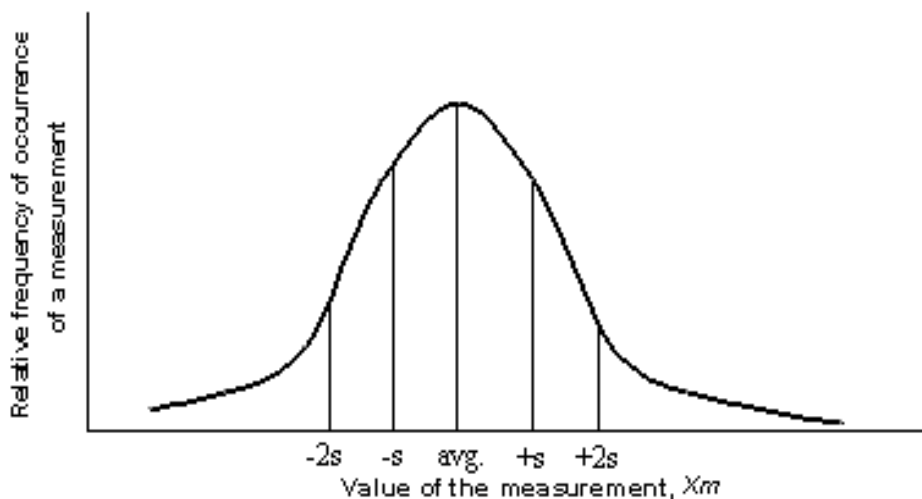
Most of the measurements result in values near $\bar{x}$. In fact, 68% of the measurements fall within the standard deviation s of $\bar{x}$ as shown in the graph above. 95% of the measured values are found within 2s of $\bar{x}$. We call the value of 2s the uncertainty of the measurement. Then, if we report our value of the measurement as $\bar{x} \pm 2s$, we are saying that $\bar{x}$ is the most probable value, and 95% of the measured values fall within this range. Consider the following example as one illustration of this statistical analysis.

<u>Example One</u>: A test tube is weighed on ten different balances, and the data below is obtained along with statistical analyses:

Balance Number	Mass (g) = x_m	d = $x_m - \bar{x}$	d^2
1	24.29	0.00	0.0000
2	24.26	–0.03	0.0009
3	24.17	–0.12	0.0144
4	24.31	0.02	0.0004
5	24.28	–0.01	0.0001
6	24.19	–0.10	0.0100
7	24.33	0.04	0.0016
8	24.50	0.21	0.0441
9	24.30	0.01	0.0001
10	24.23	–0.06	0.0036

From the data above, it is calculated that $\bar{x}$ = 242.86/10 = 24.29 g, and the standard deviation, s = $\sqrt{\dfrac{0.0752}{9}}$ = 0.0917. The range is found to be $\bar{x} \pm 2s$ = (24.29 ± 0.18) g. Therefore, the test tube weighs between 24.11 g and 24.47 g with 95% certainty. Now the values of x_m are checked against the range. We observe that the weight from balance #8 is OUTSIDE the range. This value is discarded as it is unreliable, and the values of $\bar{x}$, d, d^2, and s are thus recalculated (see Problem #1 in the Pre-lab).

For most of the experiments in this course, the standard deviation is impossible to calculate because we perform too few measurements of a particular quantity. When there are such few measured values (< 6), the *Q Test* is used to decide whether to reject suspected "bad" values as outliers.

$$Q = \frac{|\text{suspect} - \text{nearest}|}{\text{largest} - \text{smallest}} \qquad\qquad (4)$$

n (# of measurements)	3	4	5	6 or more
Q Test value (90% probability)	0.941	0.765	0.642	Do Not Use

If *Q* for a set of data is larger than those listed in the table above, the suspect value may be rejected, and the average of the other values is reported. Please note that the sign of *Q* is NOT important since we are using the absolute value. Consider the following example as one illustration of the *Q Test*.

Example Two: A student determines the concentration of a hydrochloric acid solution by titration with standardized sodium hydroxide. S/he obtains the values: 0.555 M, 0.565 M, and 0.564 M. Should the value 0.555 M be rejected? Apply the *Q Test*:

$$Q = \frac{|\text{suspect} - \text{nearest}|}{\text{largest} - \text{smallest}} = \frac{|0.555 - 0.564|}{0.565 - 0.555} = 0.90$$

Since 0.90 is LESS THAN the tabulated value of 0.94 for three measurements, we may NOT reject the suspect value, 0.555 M. If the agreement had been greater than the tabulated value of 0.94, the chemist would repeat the measurement until s/he is satisfied (see Problem #2 in the Pre-lab).

Procedure

Part 1

Use calipers to measure the inside diameter of a medium test tube. Use a ruler to measure the height of the test tube from the lip to where the bottom curves. Calculate the volume of this part of the test tube as a cylinder ($V_{cylinder} = \pi r^2 h$, where r is the radius, and h is the height in cm). Assume that the bottom of the test tube is HALF of a sphere with the same radius. Calculate its volume ($V_{sphere} = 4\pi r^3 / 3$). Divide the result by 2; add this number to the volume of the cylindrical portion. This is the total volume of the test tube.

Now fill the test tube with water. Determine the actual capacity of the test tube by pouring the water (from the test tube) into a 50 mL graduated cylinder and calculate the percent difference.

Part 2

Obtain an unknown metal cylinder from your instructor. Determine its density by weighing it on the analytical balance, measuring its dimensions with calipers, and calculating its volume. You will now confirm your calculated volume. Fill a 50 mL graduated cylinder with 25 mL of water, carefully slide your metal into your graduated cylinder, and note the volume of the displaced water. How does this value compare to your calculated volume? Now refer to the *CRC Handbook of Chemistry and Physics* in order to check your density by looking up the handbook value for the material of which your solid is made. Determine the percent error in your density.

Part 3

Obtain a buret and unknown sodium chloride (NaCl) solution from your instructor. Fill the buret with the salt solution. Allow some to drain out so as to fill the nozzle with the solution.

Weigh a stoppered Erlenmeyer flask on the analytical balance.

Record the initial buret reading containing your salt solution, and then add approximately 6 mL of solution to the flask. Carefully record the final buret reading, and weigh your stoppered flask containing the solution. Repeat this procedure FIVE more times; DO NOT EMPTY THE FLASK OR REFILL THE BURET BETWEEN WEIGHINGS! Each time, make the samples different sizes, allowing them to vary between 5 – 8 mL. Be certain to record the weight and volume of each sample as accurately as possible. At the end of the experiment, you should have SIX SAMPLES in the stoppered flask, but you have weighed each one separately and measured the volume of each one separately as well.

Calculate the six values of the density of the solution, making sure to carry out the calculation to 3 digits past the decimal. Look up the density of sodium chloride solutions in the *CRC Handbook of Chemistry and Physics*. Determine the percent sodium chloride by weight of your unknown by comparing your density with the densities in the *CRC*.

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Data and Calculations

Part 1

Diameter: _____ cm   Radius: _____ cm   Height (cylinder part): _____ cm

Volume (cylinder part): _____ cm^3 Volume (half-sphere part): _____ cm^3
  SHOW CALCULATION: SHOW CALCULATION:

Total Volume (sum): _____ cm^3 Volume (graduated cylinder): _____ mL

Average Volume: _____ mL   Percent Difference: _____ %
  SHOW CALCULATION: SHOW CALCULATION:

Part 2

Mass of Metal Cylinder _____

Diameter _____   Length _____   Volume $_{calipers}$ _____

Volume$_{water}$ _____   Volume$_{metal + water}$ _____   Volume $_{water\ displacement}$_____

Density of the Cylinder: calipers: _____   water displacement: _____

Handbook Density _____

Identity of Metal _____

% Error: calipers: _____   water displacement: _____

SHOW CALCULATIONS:

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Unknown Number _____

Mass of Flask with stopper _____   Initial Buret reading _____

Sample	Mass Flask+Stopper+Liquid (g)	Mass Liquid Only (g)	Final Buret Reading (mL)	Net Volume (mL)	Density (x_m) (g / mL) 4 sig. figs.	d ($x_m - \bar{x}$)	d^2
1							
2							
3							
4							
5							
6							
				sum of x_m:		sum of d^2:	

Show your calculation of the standard deviation, s, from d^2 below:

Mean value ($\bar{x}$): _____   Standard Deviation (s): _____

Range: _____   % NaCl from Table: _____

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Post-lab Questions

1. Calculate the density of a pure gold sphere with a diameter of 2.120 cm and a mass of 94.19 g.

2. The density of aluminum is 2.70 g/cm^3. Calculate the thickness of a rectangular sheet of aluminum foil with a width of 11.5 cm, a length of 14.0 cm, and a mass of 2.04 g.

3. Examine your results from your data table in Part 3. Do you have any values for the density of the salt solution that lie OUTSIDE the range ($\bar{x} \pm 2s$)? If so, list them here:

    Recalculate $\bar{x}$ by omitting values that lie OUTSIDE the range. This is the density value you should use to determine your experimental % NaCl.

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Pre-lab Questions

Upon reading the procedure in preparation for this experiment, you should also answer the following questions:

1. Consider Example One in the laboratory discussion. Since measurement 8 lies outside the range, it may be omitted in the calculation of the reported value. Omit measurement 8 and recalculate the mean ($\bar{x}$). Fill in the d and d^2 columns in the table, then calculate the standard deviation (s) and the range.

    Recalculated mean ($\bar{x}$), without measurement 8: _____

Balance Number	Mass (g) = x_m	$d = x_m - \bar{x}$	d^2
1	24.29		
2	24.26		
3	24.17		
4	24.31		
5	24.28		
6	24.19		
7	24.33		
8 – OMITTED	~~24.50~~		
9	24.30		
10	24.23		
sum of x_m:		sum of d^2:	

Recalculated standard deviatiation (s): _____ and range: _____

SHOW CALCULATIONS:

2. Now consider Example Two in the laboratory discussion. The student doing the titration repeated the experiment twice more. The following five values were obtained: 0.555 M, 0.565 M, 0.564 M, 0.567 M, and 0.563 M.

    A. Use the *Q Test* to demonstrate that the first value should be rejected.

    B. Recalculate the values for $\bar{x}$, omitting the value 0.555 M. Compare with the original value of $\bar{x}$.