# 2-MEASUREMENT: VOLUME, MASS, AND DENSITY

 The purpose of this exercise is to measure volume and mass, to evaluate precision of the measurements, and to use the data to calculate the density.

## Definitions

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

% error = 

You know from lecture discussion that all measurements have **uncertainty** in the last reported digit of the measurement. This is a result of the estimation of that last digit and of the random differences in reading from trial to trial. Uncertainty is inevitable and unavoidable in measurement and must always be accepted as a part of the recording and analysis of data. Uncertainty is NOT an error in data gathering. However, errors can also occur.

In chemical measurements we try to eliminate errors. **Errors** may be divided into two broad types, **systematic** and **random**. **Systematic** errors occur regularly and predictably because of faulty methods or defective instruments, or even because of incorrect assumptions (for example, a reagent bottle that has a missing or incorrect label). Random errors are more difficult to define. An example is a weighing error due to air currents near the balance. Line current fluctuations for electronic instruments also lead to random errors. Random errors can make the measured quantity either too large or too small and are governed by chance. Systematic errors always affect the measured quantity in the same direction.

 **Accuracy** is the closeness of agreement between a measured value and the **true value**. True values can never be obtained by measurement. However, we accept values obtained by skilled workers using the best instruments 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 (i.e. with little variation in the uncertain digit) is a sign of good precision. It is important to understand, though, that good precision does not guarantee accuracy.

It is common in scientific experiments to repeat the same process with different parameters or by different experimenters to confirm results. Uncertainty in measurement produces some variation in results and inaccuracies in the experiment may produce results that seem inconsistent. To test the reliability of a series of results, scientists and mathematicians use a statistical tool called **standard deviation**. This tool tests whether a measured value or calculated result is sufficiently different from others to be considered unreliable (an **outlier**) and whether it can be discarded as likely erroneous.

## Standard Deviation

The **standard deviation** of a series of measurements, which includes at least 6 independent trials, may be defined as follows. If we let **x**m be a measured value, **N** be the number of measurements, <**x**> be the average or **mean** of all the measurements, then **d** is the **deviation** of a value from the average:

 **d** = **x**m-<**x**>

and the standard deviation, **s**, is defined by:



where Σ**d**2 means “sum of all the values of **d**2.”

The **value of the measurement** should include some indication of the precision of the measurement. Standard deviation is used for this purpose if a large number of measurements of the same quantity is subject to random errors only (so measured values are equally likely to be greater than or less than the true value). We can understand the meaning of **s** if we plot on the y-axis the number of times a given value of **x**m is obtained, against the values, **x**m,on the x-axis. The “normal distribution curve” is bell-shaped, with the most frequent value being the average value, <**x**>.

 

 *Figure 3: Distribution of Values of a Measurement*

Most of the measurements give values near <**x**>. In fact, 68% of the measurements fall within the standard deviation **s** of <**x**> (see graph). 95% of the measured values are found within 2**s** of <**x**>. We call the value of **2s** the uncertainty of the measurement, **u**. Then, if we report our value of the measurement as <**x**>**±u**, we are saying that <**x**> is the most probable value and 95% of the measured values fall within this range.

**Measured values are reported as (or assumed to represent) <x>±2s or, in other words, <x>±u**. The uncertainty may not always be reported but is always understood to be a factor in interpreting a reported value.

The next example shows how the standard deviation can be used to evaluate the data.

 **Example 1. Weight of a test tube on 10 different balances**

| Balance Number | Weight (g)=xm | d=xm - <x> | d2 |
| --- | --- | --- | --- |
| 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 |
|  | Σxm=242.86 |  | Σd2=0.0752 |



So, the test tube weighs between 24.11 and 24.47 g, with 95% certainty.

Now each value of **x**m is checked against the range. We observe that the weight from balance 8 is outside the range; we should discard it as unreliable and recalculate <**x**>, **d**, **d**2 and **s**. (See problem 1 in the Pre-Laboratory Assignment.)

## The Q Test

For most of the experiments in this course, the standard deviation, **s**, is inappropriate to calculate because we perform too few measurements of the given quantity. When there are few measured values (<6), the **Q test** is used to decide whether to reject suspected "bad" values.

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If Q for a set of data is larger than those listed in the table below, the suspect value may be rejected and the average of the other values is reported. (Note that the sign of Q is not important since we use the absolute value.)

Q test values for 3 to 5 measurements, 90% probability

| N (# of measurements) | 3 | 4 | 5 |
| --- | --- | --- | --- |
| Q | 0.94 | 0.76 | 0.64 |

Note that you need at least 3 trials to apply the Q test. If you have only 2 trials and do not know of any error for either, you have no reason to trust one more than the other. All you can do is to average the two values in hopes that the averaging process reduces the random error. The average of the trials is taken as your experimental value. (And no good scientist ever relies on a single determination!)

**Example II. The Q test**

 A student determines the molarity of a hydrochloric acid solution by titration with standard NaOH. He obtains the values 0.555, 0.565 and 0.562 M. Should the value 0.555 M be rejected, because the other two values are very close to each other? Apply the Q test:

$$Q=\frac{|0.555-0.562|}{|0.565-0.555|}=\frac{0.007}{0.010}=0.70$$

Since 0.70 is less than the tabulated value of 0.94 for 3 measurements we may **not** reject the suspect value, 0.555. We report the average value <**x**> with the average deviation <**d**>:

| Value, xm | d=|xm-<x>| |
| --- | --- |
| 0.555 | 0.006 |
| 0.565 | 0.004 |
| 0.562 | 0.0013 |
| ---------- | ---------- |
| Σxm=1.682 | Σd=0.0113 |
| <x>=0.561 | <d>=0.004 |

So for this series, we report the molarity as 0.561 M ±0.004 M.

If agreement is bad, the chemist will repeat the measurement until he is satisfied. (See Problem 2 in the Pre-Laboratory assignment.)

## PROCEDURE

Set up your notebook; enter data as shown in the Report for Procedures I and II.

### I. Use of measurements to determine a physical property

 Objective: Learn how to use the analytical balance and compare measuring tools.

 Observe the instructor’s demonstration of how to read a Vernier scale, such as on our calipers. Pay attention to the estimation of the last (uncertain) significant figure.

 Obtain an “unknown solid” from the instructor. It will most likely be a metal cylinder. Determine its **density** by weighing it on the analytical balance, measuring its dimensions with calipers, and calculating its volume:

 

Then redo the measurement of its dimensions using a ruler instead of calipers.

Next, measure the density using the *Archimedes Method*. Fill a plastic graduated cylinder with just enough water to cover your unknown solid. Record that initial volume. Tilt the graduated cylinder so you can carefully slide your solid down the graduated cylinder and do not displace or spill any of the water inside. Place the graduated cylinder plus water plus solid on a level table and record the final volume. Calculate the net volume of the solid then the density of the solid.

For most solids, the density is given in g/cm3. Lastly, check your density by looking up your density value for the solid in a reference source such as the CRC Handbook of Chemistry and Physics. Collect a list of possible elements and/or alloys that have that density then use deductive reasoning to assess which material best fits your sample. Using the handbook value as the ***“true”*** value, calculate the percent error in your density.

### II. Standard Deviation

 Check out a buret from the stockroom. Examine the calibration marks and compare them with the graduated cylinder. Make sure that you read the buret to the limit of its precision. This means that we estimate to 0.01 mL, since we report all the **certain** digits of a measurement, followed by one **uncertain** digit. For example, the buret reading 24.92 mL has four significant digits, the uncertain or estimated one is the 2 in the hundredths place.

 Clean the buret if necessary. (Be sure to return it clean and well rinsed at the end of the experiment so the next student does not have to clean it before use.)

 Obtain an unknown salt solution from the instructor. Rinse the buret three times using about 4 mL of the salt solution each time. Use this opportunity to confirm that the stopcock does not leak and that you can control the buret by smoothly opening and closing off the flow of liquid. Then fill the buret with the salt solution to somewhere just below the topmost (0.00 mL) marking. Weigh a stoppered Erlenmeyer flask on the analytical balance.

 Record the initial buret reading and then add about 6 mL of unknown salt solution to the flask. Record the final buret reading. Weigh the flask, solution and stopper. Repeat this procedure at least five more times. **Do not empty the flask between weighings**. Make the samples different sizes, letting them vary between 5 and 8 mL. Obtain at least six samples without refilling the buret, and record the weight and volume of each sample as accurately as possible. At the end of the experiment, you will have **all six (or more) samples** in the stoppered flask, but you have weighed each one separately and measured the volume of each one separately.

 Calculate the 6 (or more) values of the density of the solution. **CARRY OUT THE CALCULATION TO 3 DIGITS PAST THE DECIMAL** (four significant digits). Check the validity of all values by calculating the standard deviation and the uncertainty in your experimental density value. (See Report Sheet.)

 Look up the density of salt solutions in the CRC Handbook of Chemistry and Physics. Determine the percent salt by weight of your unknown, by comparing your density with the densities in the Handbook.

Report: Complete the report sheet and turn it in together with the Pre & Post Questions.

#  2-MEASUREMENT: VOLUME, MASS, AND DENSITY

Section \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ Name \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

##  Report Sheet

**Part I**

Table I. Mass, Volume, and Density of a Solid Unknown number \_\_\_\_\_\_\_

| method | color | shape | mass | Diameter or width | length | volume | density |
| --- | --- | --- | --- | --- | --- | --- | --- |
| calipers |  |  |  |  |  |  |  |
| ruler |  X |  X |  |  |  |  |  |
| *Archimedes* |  X |  X |  |  X |  X |  |  |

Identity of metal \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ Literature density \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

Page & year of Handbook/source \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

% error \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ (show sample calculations below)

Which method produced the most accurate density & why?

**Part II**. Unknown number \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

 Weight of flask and cork = \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

Table II. Data for Burette Procedure (at least 6 independent trials)

| Sample | Weight | Net weight | Buret, init. | Buret, final | Net volume | Density, xm | d=xm-<x> | d2 |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 1 |  |  |  |  |  |  |  |  |
| 2 |  |  |  |  |  |  |  |  |
| 3 |  |  |  |  |  |  |  |  |
| 4 |  |  |  |  |  |  |  |  |
| 5 |  |  |  |  |  |  |  |  |
| 6 |  |  |  |  |  |  |  |  |
| 7 |  |  |  |  |  |  |  |  |
| 8 |  |  |  |  |  |  |  |  |

Either show your calculation of the standard deviation, **s**, from **d**2 below or upload your excel file into Canvas as described by your instructor.

Reported density value and range: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

% NaCl, from Handbook \_\_\_\_\_\_\_\_\_\_\_\_ Page and year or source: \_\_\_\_\_\_\_\_\_\_\_\_\_\_

# 2-MEASUREMENT: VOLUME, MASS AND DENSITY

Section\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ Name\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

##  Post - Questions

1. We estimate to 1/10 or 2/10 of the smallest division on any instrument to obtain the final significant digit in a measurement. State the magnitude of the smallest division, and give the units, for the following instruments:

|  **Instrument** | **Magnitude of smallest division** |
| --- | --- |
| Ruler |  |
| Calipers |  |
| Graduated cylinder |  |
| Buret |  |
| Centigram balance |  |
| Analytical balance |  |

2. The volume of a sphere is (4/3)πr3. Calculate the density of a pure gold sphere with a diameter of 2.120 cm and a mass of 94.19 g.

3. The density of aluminum is 2.70 g/cm3. 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.

4. Do you have any values in your Table III for the density of the salt solution that lie outside the range (<**x**m> ± 2**s**)? If so, list them here: Recalculate <**x**m>, omitting values that lie outside the range. (This is the density value you should use to find your experimental % NaCl.)

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Section\_\_\_\_\_\_\_\_\_\_\_\_\_\_ Name\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

##  Pre-Laboratory Assignment

**1. Standard Deviation.** Since measurement 8 in Example I lies outside the range, it may be omitted in calculating the reported value. Make a table or entries in an Excel spreadsheet as in Example I for the calculation of <**x**>, **s** and the range. Use measurements 1-7, 9 and 10. (Note that **d** must be recalculated. **SHOW WORK**. The correct value to report is 24.26 ± 0.11 g.)

**2. The Q Test.** A student doing the titrations in Example II repeated her experiment twice more. She now has obtained five values: 0.555, 0.565, 0.564, 0.567, 0.563 M.

a. Use the Q test to show that the first value should be rejected as a probable error (use the tabulated Q for 5 values).

b. Recalculate the values for <**x**> and <**d**> omitting the erroneous value 0.555. **SHOW WORK**. (The correct value to report is 0.565 ± 0.001 M.)