Laboratory Activity Measurement and Density

Background:
Measurements of mass and volume are very common in the chemistry laboratory. The analytical balance is used to measure mass, and the graduated cylinder, pipette, or burette are used for the measurement of volume. All measurements are subject to error. Generally, the last decimal place reported is the one assumed to be the least accurate. If a balance produces a reading of 12.22 grams, this implies that the “true” mass is between 12.21 and 12.23 grams. With volume, the last decimal place is estimated to lie between the graduations. For example, if the volume is found to lie between 5.6 and 5.7 on a 10 ml pipette, an additional decimal place can be reported as 5.68. Refer to the appendix for rules pertaining to the correct use of significant digits.

Repeat measurements are often made to increase accuracy of measurement. If multiple measurements are made, the average of the values is often reported, along with an expression of the spread of the data. Before computers and calculators made these calculations easy, the error was often expressed as an “average deviation”; the process for calculating this is below.

Deviation for each trial = trial result – average value

Average deviation = Sum of absolute values of all deviations
Number of trials

Now, because spreadsheet software is widely available and has built in statistical functions, we almost always see the data from multiple measurements expressed as an average (mean, represented as X) ± standard error of the mean, SEM (which is the standard deviation, STDEV, divided by the square root of the number, n, of data points available). Occasionally, the spread of data is represented as the coefficient of variation CV; the percent coefficient of variation is the mean divided by the standard deviation times 100.

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X = \frac{\text{sum of all values}}{n}
\]

STDEV from Excel

\[
\text{SEM} = \frac{\text{STDEV}}{\sqrt{n}}
\]

%CV = \frac{\text{STDEV}}{X} \times 100

Although this is not a course in statistics, we will explore some simple data sets and learn how to use Excel to calculate mean, standard deviation, and standard error. Deviations should be reported to the correct number of significant figures; for standard deviation, because it is based on a sum of the deviations from the mean, the number of significant figures should be no more decimal places than the number with the fewest decimal places. (The division by an exact number does not affect the sig figs.) The underlying Excel formula for standard deviation is:

\[
\sqrt{\frac{\sum \text{Deviations}^2}{N-1}} \quad \text{where} \Sigma \text{ means sum and} \ N \text{ is the number of measurements}.
\]

The Excel file below shows the calculated mean, standard deviation, coefficient of variation, and standard error for several data sets. These values are not corrected for significant figures; these numbers are just what are shown based on the column widths in Excel. Post lab questions will refer back to these data.
For now, in order to use the built in statistical functions, you enter the data in the correct cells. All formulas start with an equals sign. The formula in cell B11 to calculate the mean of the data in the column above is entered as “=average(B1:B5)”. The formula for standard deviation in cell B12 is “=stdev(B1:B5)”. The percent coefficient of variation is “=B12/B11*100”, and the standard error is “=B12/(5^0.5)”. The denominator for standard error is obtained by taking the square root of the number of data points (5). Excel has several ways to take the square root; an easy one is to raise the number you wish to take the square root of to the 0.5 (half) power. The symbol ^ is used to raise a number. The calculated descriptive statistics for the other columns’ data is similarly obtained.

Density is a property that can be used to aid in identification of pure compounds. At a given temperature, the density of a pure substance is a constant. Density is calculated as mass/unit volume. Densities of solids and liquids are typically expressed with units of grams/ml; gases are grams/liter. Since density is obtained by a division operation, it should be reported to the number of significant digits of either the mass or volume, whichever has the fewest significant digits. For example, if you record the mass of a liquid as 8.4333 grams, and its volume as 5.66 ml, the density should be reported as 1.49 grams/ml.

Although the exercises in this lab are very simple, they will ensure that you are comfortable with basic techniques of measurement. You will measure the mass of a penny on several balances and report the average mass, average deviation, standard deviation, and standard error. You will determine the density of a solid by volume displacement, and the density of two unknown liquids. Finally, working in a group, you and your group members will produce some dilutions of a colored substance using a variety of lab equipment designed for volume measurement. By reading the absorbances of the resulting solutions and calculating the average absorbances and the deviation of the absorbances, you will be able to see how choosing the right equipment gives the best results by comparing your data to your classmates’ data.
**Procedure:**

**Part A  Mass of a Penny**

1. Obtain a penny. Using each of the four Mettler AG204 balances, obtain the weight of the penny to 4 decimal places. To do this, first tare (zero) the balance with the door shut. Place the penny on the weighing tray, close the door, wait till the reading is constant, and record the weight on the data page.
2. Repeat on all four balances.
3. Calculate the deviation, average deviation, and the standard deviation using Excel to the correct number of significant digits.

**Part B  Density of a Solid by Volume Displacement**

1. Obtain one of the solids (i.e., a large nail, rock, etc.) from the front bench.
2. Weigh as before and record the weight on the data sheet.
3. Place approximately 15 mls of tap water into a 25 or 50 ml graduated cylinder.
4. Record the volume, estimating the value of the tenths place.
5. Place the solid into the cylinder by sliding it down the side. Dislodge any air bubbles by tapping the side of the cylinder. Record the new volume on the data sheet.
6. The increase in volume is the volume of the solid.
7. Calculate the density of the solid to the correct number of significant digits.

**Part C  Density of a Liquid**

1. Obtain two liquid unknowns and record the identifying number of your unknowns.
2. Pour 25 – 50 mls of one of your unknowns into an appropriately sized graduated cylinder and record the exact volume.
3. Tare an empty beaker that will hold your liquid on an analytical balance with the door shut. Remove the beaker and pour all of the liquid from the cylinder into the beaker.
4. Reweigh on the same tared balance and record the weight of the liquid. NOTE – some of the unknowns are “volatile”, which means that they evaporate easily. If you observe the mass deceasing, it shows that the substance is evaporating; just record the mass as quickly as you can.
5. Calculate the density to the correct number of significant digits.
6. Repeat with your second liquid.

**Part D  Comparison of Equipment Precision**

Often, getting the best results in the chemistry lab requires you to choose the equipment that is right for the task at hand. We will study the precision (reproducibility, measured by calculating the deviations) of selected pieces of volumetric glassware.

**Procedure:**

1. Work in groups of 3 – 4 for this part of the lab. Your instructor will assign groups.
2. You will be assigned to one of the following groups:
   a. Group 1
      i. Make 5 duplicate dye solutions by measuring 2 mls of the stock dye (0.3 g Congo Red in 500 ml H₂O) in a 100 ml graduated cylinder and bring the volume up to 100 mls. DO NOT worry that it is difficult to
accurately measure 2 mls using a 100 ml graduated cylinder; that is the point of this exercise.

ii. After each solution is made as directed, pour into a beaker or flask that will contain at least 100 mls and mix well by stirring.

iii. Bring your 5 samples to the instructor at the spectrophotometer and record the absorbance of your solutions.

iv. Put the raw data on the board for the class to share.

b. Group 2

i. Make 5 duplicate dye solutions by measuring 2 mls of the stock dye with a 10 ml graduated cylinder. Pour this amount into a 100 ml graduated cylinder, rinse with some water and pour the rinse water into the 100 mls cylinder. Add water to the 100 ml mark.

ii. Repeat steps ii. through iv. in the directions for group 1.

c. Group 3

i. Make 5 duplicate solutions by measuring 2 mls of the stock solution using a 5 ml volumetric pipette. (Practice with water first if you have not used these before). Place the 2 mls into a 100 ml volumetric flask and fill with water to the fill line. NOTE: if you overshoot the fill line, you need to start again, so add the last bit of water with a dropper.

ii. Repeat steps ii. through iv. in the directions for group 1.

d. Group 4

i. Make 5 duplicate solutions by measuring 2 mls of the dye stock solution with a 10 ml graduated cylinder. Place the 2 mls into a 100 ml volumetric flask, rinse the 10 ml cylinder with water and add the rinse water to the 100 ml flaks, and fill with water to the fill line. NOTE: if you overshoot the fill line, you need to start again, so add the last bit of water with a dropper.

ii. Repeat steps ii. through iv. in the directions for group 1.

3. Working in your groups, calculate the average absorbance, the average deviation, the standard deviation, and the standard error for your set of data. Record your groups’ calculations on the board. Also, have your group discuss which group’s equipment and dilution procedure gave the most precise results. Refer back to the calculated deviations to support your claim; you must use the data to provide the evidence. Write a paragraph summarizing this discussion.

Write-up:

1. Before you leave, hand in your data sheet. Answer the questions following the data sheet that refer back to the data sets on page 2. Make sure your name is on your group discussion about equipment precision.
Data Sheet

Part A
Mass on balance 1 _______________

Mass on balance 2 _______________ Average Mass: _______________

Mass on balance 3 _______________

Mass on balance 4 _______________

Deviation 1 ____________

Deviation 2 ____________ Average Deviation: ____________

Deviation 3 ____________ Standard Deviation: ____________

Deviation 4 ____________ Standard Error: ____________

Part B
Mass of solid ___________

Volume in cylinder without solid ___________

Volume in cylinder with solid ___________

Volume solid ______________

Density solid _____________

Part C
Volume unknown 1 ____________

Mass unknown 1 ____________

Density unknown 1 ____________

Volume unknown 2 ____________

Mass unknown 2 ____________

Density unknown 2 ____________
Part D data

Group number: __________    Mean: ________
Reading 1: ________     Standard deviation: ________
Reading 2: ________     Standard error: ________
Reading 3: ________     Average deviation: ________
Reading 4: ________
Reading 5: ________

Questions on statistical data (page 2):

1. What is the mean (average) of the data set in column B? C? D? E?

2. Compare the range (spread) of data in columns B and C, lowest to highest.

3. How does the spread of data in these two columns compare to the standard deviation of these two columns?

4. Compare the spread of data in these two columns to the %CV and standard error as well.

5. What is the range of data in column D? How does this compare to column C? Compare the standard deviation, % CV, and standard error in columns C and D. What do you think accounts for the difference?

6. What is the range of data in column F? How does this compare to column D? Compare the standard deviation, % CV, and standard error in columns D and F. What do you think accounts for the difference? Explain, using this information, why more repeat measurements give you more confidence in your answer.
Appendix
Rules for Use of Significant Digits

1. ALL non-zero numbers (1,2,3,4,5,6,7,8,9) are ALWAYS significant.
   Example: 455.34 has five significant digits

2. ALL zeroes between non-zero numbers are ALWAYS significant.
   Example: 4.508 has four significant digits

3. ALL zeroes which are SIMULTANEOUSLY to the right of the decimal point AND
   at the end of the number are ALWAYS significant.
   Examples: 4.0 has two significant digits
             5.5080 has five significant digits

4. Zeros to the left of the nonzero digits are NOT significant; they are merely
   placeholders.
   Example: 0.00458 has three significant digits

5. Trailing zeros (at the end of a number of 10 or greater) are only significant if a
   decimal point is present. If there is no decimal point, the number of significant digits
   is ambiguous.
   Examples: 150. has three significant digits
             150 is ambiguous

Rules for Arithmetic Operations

1. In addition or subtraction, the answer should have no more decimal places than the
   number with the fewest decimal places.
   Example: 5.2 + 6.31111 equals 11.5 to the correct number of significant
digits

2. In multiplication or division, the answer should have no more significant digits than
   the number with the fewest significant digits.
   Example: 5.44 x 18.6888 equals 102 to the correct number of significant
digits

Round up or down as necessary to obtain the correct number of significant digits.