Protocol | Precision, Accuracy, and Precipitation Reactions

Precision, Accuracy, and Precipitation Reactions

Bring a USB drive with you to lab this week.

Introduction

Watch the introductory video Links to an external site.for this experiment.

In this experiment, we will become familiar with essential operations in the chemistry laboratory, including weighing solids, pipetting liquids, and filtering mixtures under vacuum. In the first part of the experiment, we will use systematic observations of precipitation reactions to identify the cation and anion in an unknown solution of an ionic salt. In the second part of this experiment, we will use simple measurements of liquids to assess the accuracy and precision of common laboratory glassware. In the third part of the experiment, we will become familiar with the LabQuest data acquisition platform by measuring the heating rate of a hot plate over time.

Throughout the experiment, careful technique is required to obtain accurate and precise results. Take some time in the laboratory to become comfortable with the tools at your disposal. Developing good habits now will make you more accurate, precise, and efficient in future experiments.

Measurements, Units, and Significant Digits

We will make use of the SI system of units throughout CHEM 1310L. The seven fundamental quantities in this system are listed in the table below along with the *base units*.

Physical Quantity	Unit	Abbreviation
mass	kilogram	kg
length	meter	m
time	second	s
electric current	ampere	A
temperature	Kelvin	K
amount of substance	mole	mol
luminous intensity	candela	cd

The base units can be combined to form *derived units* with different names, such as the Newton (1 kilogram-meter per second squared) and Coulomb (1 ampere-second). Prefixes are added before the name of a base or derived unit to represent scaling by a power of ten. Familiarize yourself with the abbreviations and scaling factors associated with each prefix. When making measurements in the laboratory, carefully note the units of each instrument or piece of glassware that you use.

Prefix	Abbreviation	Scaling Factor
femto	f	10^{-15}
pico	p	10^{-12}
nano	n	10^{-9}
micro	μ	10^{-6}
milli	m	10^{-3}
centi	c	10^{-2}
deci	d	10^{-1}
kilo	k	10^{3}
mega	M	10^{6}
giga	G	10^{9}
tera	T	10^{12}

Occasionally, you will need to convert a measurement from one unit to another. This can happen, for example, when you have a physical constant in one unit but measurements were made in an incompatible unit. Unit conversions may also be necessary when comparing values (for instance, in the assessment of accuracy). *Conversion factors* are ratios that enable the transformation of a measurement from one set of units to another. The magnitude of a conversion factor depends on the relative sizes of the two units involved.

In making measurements and performing calculations based on these measurements, you should also carefully consider *significant digits*. Measuring instruments can only provide a finite level of precision; at some point, the size of a measurement becomes imprecise or "fuzzy." When using a digital measuring instrument such as the LabQuest, record all of the digits that the instrument gives you, estimating the last digit if some "wobble" or drift is observed. Digits after the smallest place reported on the device are uncertain and cannot be estimated. All of the digits in this measurement are considered significant, even trailing zeroes.

Analog instruments and glassware are similarly limited in their precision. When we refer to the precision of a piece of glassware or instrument, we mean the smallest amount that can be measured with a reasonable amount of certainty. For laboratory glassware, it is possible to systematically determine precision using the markings on the glassware. The general rule here is that one and only one digit can be estimated; other digits come from numbers printed on the glassware. Again, all of the digits in these measurements are considered significant, but it will be up to you to record the measurement with the correct number of significant digits. If you think a measurement is sitting right on a marking, add a trailing zero!

When performing calculations using measurements, it is important to avoid adding false precision to calculated values. Such false precision could lull a reader into a false sense of security about the quality of the measurements. Be sure to apply the rules for mathematical operations with significant digits in all calculations in CHEM 1310 laboratory. With practice, the rules should become highly intuitive and you'll be able to breeze through complex calculations while keeping a mental inventory of significant digits.

Accuracy and Precision

Empirical measurements represent the basis of everything that chemists know. More generally, any scientific understanding of the world is grounded in measurements. Measurement theory clarifies the *limitations* of scientific measurement and gives us tools to evaluate the *quality* of data. You'll find these tools useful not just in your time as a student in CHEM 1310 laboratory, but also throughout your career as a scientist or engineer.

Data quality has two independent dimensions: precision and accuracy. *Accuracy* is defined as the closeness of a measurement or set of measurements to a true, theoretical, or previously measured value. Although naively we might quantify accuracy as the difference between the mean measured value and the true value, this difference doesn't mean much without information about the magnitude of the true value. (Is 3 grams a lot? If the true value is 4 grams, yes; if the true value is 500 grams, no.) Thus, we divide this difference by the true value and multiply by 100% to obtain the key measure of accuracy, *percent error*.

$$\mathrm{PE} = \frac{x_{meas} - x_{true}}{x_{true}} \times 100\%$$

Precision is defined as the spread within a set of equivalent measurements. Measurements that are very similar to one another are said to be precise; measurements that are spread out over a large range are said to be imprecise. The key measure of precision is *standard deviation (σ)*. Defining as the mean of the data and *N* as the number of measurements,

$$\sigma = \sqrt{\frac{1}{N-1} \sum_i^N \left(x_i - \overline{x}\right)^2}$$

Keep in mind that precision applies only to a set of equivalent measurements, as when the same experiment is run multiple times. Accuracy, on the other hand, can be evaluated for any number of measurements (to evaluate the accuracy of a set of equivalent measurements, the mean of the set is used).

Solubility and Precipitation Reactions

When placed in water, many ionic solids or salts dissolve and undergo dissociation. On the microscopic level, *dissolution* involves the surrounding of the ionic compound by water molecules, which interact with the ions through electrostatic forces.
During *dissociation*, the cations and anions in the ionic solid separate as they are individually surrounded by water molecules. Under the right circumstances, dissolution can happen in reverse: cations and anions can form ionic bonds and leave solution, forming an ionic solid (*precipitation*). Precipitation occurs in any solution containing a greater concentration of ions than the maximum solubility of the salt. We can separate salts roughly into three groups depending on the magnitude of their solubility: *soluble* salts have very large solubility, *sparingly soluble* salts have moderate solubility, and *insoluble* salts have very small solubility. When the cationic and anionic components of an insoluble salt find themselves in the same solution, they will precipitate spontaneously.

For example, when an aqueous solution of lead(II) nitrate, $Pb(NO_3)_2$, is combined with an aqueous solution of potassium iodide, KI, a precipitate forms that consists of lead(II) iodide, PbI_2. The $Pb(NO_3)_2$ and KI solutions are prepared by dissolving the corresponding ionic solids in water, as both salts are soluble. However, when the solutions are combined, Pb^{2+} and I^- ions find themselves in the same solution, and PbI_2 is insoluble in water. As a result, PbI_2 forms spontaneously in the mixture:

$Pb^{2+}(aq) + 2\ I^-(aq) \rightarrow PbI_2(s)$

The *solubility rules* are qualitative guidelines that enable us to predict the solubility of a salt from its component cation(s) and anion(s). The rules list groups of salts that are soluble or insoluble, with exceptions associated with specific cations or anions.

1. Salts of group 1 cations (Li^+, Na^+, and K^+) are soluble.
2. All common acetates ($CH_3CO_2^-$) and nitrates (NO_3^-) are soluble.
3. All chlorides, bromides, and iodides are soluble with the exception of salts of silver, lead(II), and mercury(I).
4. Sulfates (SO_4^{2-}) are soluble with the exception of salts of barium, strontium, lead(II), calcium, silver, and mercury(I).
5. Carbonates (CO_3^{2-}), hydroxides (OH^-), oxides (O^{2-}), and phosphates (PO_4^{3-}) are insoluble, except for salts of group 1 cations.

In this experiment, we will determine the identity of a salt solution using small-scale precipitation tests. The unknown solution will be mixed systematically with known solutions and observations of the precipitate formed (if any) will be recorded. Combining the observations with the solubility rules allows us to deduce the cation and anion in the unknown solution. The cation may be barium, silver, sodium, or copper(II) and the anion may be chloride, nitrate, or sulfate.

Safety and Materials

In this experiment, you'll work with chemical glassware for the first time. Remember that glass is brittle and is liable to break when dropped. Keep glassware away from the edges of your workspace and lift glassware whenever possible to make measurements. Don't lower your head to make a measurement—this leaves you more vulnerable to spills.

The ionic salt solutions, particularly those of barium and lead(**II**), are associated with environmental hazards. To minimize the risks of these hazards, we must manage waste from the precipitation tests responsibly. Make sure to discard the liquid waste from this experiment in the designated waste bottle. ***First*****,** check the liquid level of the bottle to ensure that it isn't more than 75% full (if it is, let your teaching assistant know that a waste pickup is needed). Then, lift the red lid of the safety funnel and pour the waste into the bottle. Finally, replace the lid, which minimizes the escape of vapors from the bottle.

The following reagents will be available:

- 0.1 M sodium sulfate (Na_2SO_4) solutionLinks to an external site.
- 0.1 M lead(II) nitrate ($Pb(NO_3)_2$) solutionLinks to an external site.
- 0.1 M sodium hydroxide (NaOH) solutionLinks to an external site.
- 0.1 M sodium chloride (NaCl) solutionLinks to an external site.
- 0.1 M barium nitrate ($Ba(NO_3)_2$) solutionLinks to an external site.
- 0.1 M unknown solutions **A – D** (salts of Cu^{2+}, Ba^{2+}, Ag^{+}, or Na^{+} along with Cl^{-}, NO_3^{-}, or SO_4^{2-})

Relevant Experimental Techniques

- Weighing Solids
- Pipetting Liquids
- Measuring Temperature with a LabQuest 2

Procedures

A. Identifying an Unknown Ion by Precipitation

1. Unknowns can be found in dropper bottles at each bench. Select an **unknown solution** and record its letter in your notebook. Prepare a table in your notebook with columns for observations of reactions of your unknown with the five known solutions.
2. Add a few drops of **sodium sulfate**, **lead(II) nitrate**, **sodium hydroxide**, **barium nitrate**, and **sodium chloride** solutions to separate wells in a ceramic spot plate

or 24-well plate. Use a paper towel or your notebook to label the wells, keeping track of which known solution is in each well.

3. Add a few drops of your unknown solution to each well. Record your observations, noting in particular whether a precipitate is observed in each case and if so, the color of the precipitate.
4. Working together with your lab partner, infer the chemical formulas of any precipitates formed and record them in your notebook underneath the corresponding observations.
5. Carefully pour the reaction mixtures into a waste beaker, using your wash bottle with water to rinse off any precipitates. Ensure that there is enough water in the beaker to transfer the solid. We will vacuum filter this mixture to separate the solid and liquid phases.
6. Set up a vacuum filtration apparatus by connecting a Buchner funnel to a filter flask. Use a ring stand and clamp to hold the flask in place, then use vacuum tubing to connect the nozzle of the flask to a vacuum tap at your bench.
7. Weigh a piece of filter paper, record its mass, and add the filter paper to the Buchner funnel.
8. Gently pour the mixture of solid and liquid over the filter paper. Once all of the liquid has been pulled through, allow the vacuum to pull air through the solid for 5 additional minutes.
9. Weigh the filter paper with the solid on it and record the paper + solid mass. Use subtraction to determine the mass of solid obtained.
10. Rinse the liquid in the filter flask into the waste bottle in the hood. Remember to lift the red lid of the funnel before discarding waste!
11. Use a scoopula to scrape the solid off of the filter paper and into the solid waste container. Discard the filter paper in the trash.
12. ***Concept Check!*** In your lab notebook, draw a large box. In this box, draw a molecular-level picture of a solution of barium nitrate (2 molecules) and sodium sulfate (5 molecules) in water *before* precipitation has taken place. In a second box, draw a molecular-level picture of the mixture *after* precipitation has taken place. Use simple circles to represent ions.

B. Precision and Accuracy of Glassware for Measuring Liquids

1. Over the next few steps, we will determine the accuracy and precision of various devices for measuring liquid volumes. Obtain a 10 mL serological pipet, 10 mL graduated cylinder, 10 mL volumetric pipet, and blue pipet bulb.
2. Measure and record the mass of a clean, dry 50 mL beaker. Take the beaker to the balance, press the “Tare/Zero” button on the balance, and place the beaker on the balance. Once the mass reading stabilizes, record all displayed digits in your lab notebook. If necessary, estimate the last digit.
3. Using the serological pipet, deliver 10 mL of **deionized water** into the beaker and re-weigh. Calculate the mass of water delivered.

4. Repeat the previous step twice more to obtain three measurements of mass of water delivered using a 10 mL serological pipet. Each measurement should be roughly 10 grams.
5. Repeat steps 3 and 4 using the volumetric pipet to obtain three masses of water delivered that are approximately 10 grams each.
6. Repeat steps 3 and 4 using the graduated cylinder to obtain three masses of water delivered that are approximately 10 grams each.
7. Calculate the mean and standard deviation of the mass of water delivered for each instrument. The mean should be roughly 10 grams and the standard deviation should be less than 1 gram.

C. Using a LabQuest Data Acquisition Device to Measure Temperature

1. Obtain a hot plate and a 250 mL beaker. Add 50 mL of water to the beaker (aiming for the 50 mL line) and place it on the hot plate.
2. ***Alternative device option:*** Use a mobile device or iPad with the "Graphical Analysis GW" App to collect temperature data and easily export it. Instructions for using the app can be found in the Guide to Collecting Digital Data Download Guide to Collecting Digital Data.
3. Remove a LabQuest from the charging station in your lab and, using the charge cord in the box at your bench, plug it in to an outlet at your bench. Obtain a wireless temperature probe. Connect the probe by tapping *Sensors* → *Wireless Device Setup* → *Go Wireless*. Tap the name of your probe in the list. Ensure that your probe is properly connected by using your hand to gently warm it. Do you observe a temperature increase?
4. Place the probe in the beaker on the hot plate.
5. Tap on the *Mode* box and set up the LabQuest to collect data every 5 seconds for 10 minutes (600 seconds). Turn the knob on the hot plate about halfway and record the “set” temperature in your notebook. Start data collection on the LabQuest by pressing the play button. As the temperature of the water increases, you should observe an upward-sloping line on the LabQuest screen.
6. When data collection is complete, use one of the following methods to export and save the data.
    1. *Export the data onto a USB drive.* Insert a USB drive into the USB port on the LabQuest. Select *File* → *Export* to export the data as a TXT file readable by any text editor.
    2. Select the USB logo on the following screen and give the data a name ending in “.txt.”
    3. Unplug the USB drive and use the lab computer to verify that the data was faithfully copied. All six runs are stored in one text file.
    4. Discard any excess sodium hydroxide solution down the drain with copious water. Discard all reaction mixtures and excess crystal violet solutions in the waste container and wash test tubes, pipets,

and any other glassware used with soap and water. Return the glassware to your lab drawers.

5. *Share the data to a mobile device or computer using the Internet.* Tap the wireless connection icon at the bottom right of the LabQuest screen. On the screen that appears, tap the gear icon at the upper right and select "eduroam" as the network to connect to (if your LabQuest is already connected to eduroam, you can skip this step).
6. Enter your Georgia Tech email address (including "@gatech.edu") as the username and your password, then click *Connect* and *OK*. The wireless network icon will appear orange if you connected successfully.
7. Use your mobile device to scan the QR code that appears *or* visit the URL at the bottom left of the screen. A graph of your data will appear. Click the download icon in the upper right and select *Download data.* Give the file a descriptive name and tap *Download CSV*.
8. *Record the data by hand.* If the LabQuest cannot connect to the Internet and you do not have a USB drive, use the Table view on the LabQuest to record the data by hand in your notebook. Record at least 20 points, evenly spaced across the collected data.

Post-lab Calculations and Data Workup | SpreadsheetDownload Spreadsheet

Carefully consider your results from Part A to determine the identity of the cation and anion in your unknown solution. Tabulate your results and conclusions (Table 1).

Calculate the percent error of each mean mass of water delivered in Part B, using 10.00 grams as the “true” value. Calculate a standard deviation of the mass of water delivered for each piece of glassware. Determine the most accurate and most precise glassware using your data (Table 2).

Plot your temperature-time data from Part C in the template spreadsheet (Figure 1). Ensure that your plot includes clear axes labels with units and plot the independent variable on the *x*-axis and the dependent variable on the *y*-axis. Add a line of best fit (trend line) to the graph and include the equation of the line and the correlation coefficient R^2. Assess the precision of the data. Does the hot plate appear to cause a linear increase in temperature over time?