Laboratory Manual for Chem 260 (Spring 2020)

1. Data Analysis Workshop
2. Preliminary Experiments
    - Making Measurements and Preparing Solutions
        - Experiment
        - Example Lab Notebook Entry
        - Accuracy, Precision, and Analytical Measurements
        - The Art of Measuring Mass
        - The Art of Measuring Volume
    - Newton's Law of Cooling
        - Experiment
        - Sample Results and Conclusions Section
        - How to Design a Table and a Figure
        - Mathematical Modeling of Data
    - Determining the Amount of Acetic Acid in Vinegar
        - Experiment
        - How to Craft a Procedure Section
        - Titrimetry
        - Potentiometry
    - Characterizing an Oscillating Reaction
        - Experiment
        - Crafting an Introduction
        - Visible Absorbance Spectra and Beer's Law
3. Project Experiments
    - Thermodynamics of Hydrogen Peroxide's Decomposition
    - Thermodynamics and Solubility of Calcium Hydroxide
    - Determining the Acid Dissociation Constant for an Organic Dye
    - Kinetics of the Bleaching of Dyes
4. Useful Odds and Ends
    - Guidelines for Writing Formal Reports
    - Sample Formal Report
    - Rubric for Evaluating Formal Reports
    - Preparing a Poster
    - Working Safely in the Chem 260 Lab
    - Tips for Working as a Group
    - Maintaining an Electronic Laboratory Notebook

Data Analysis Workshop

During the semester you will learnhow to collect and analyze data, and how to use that data to answer questions about the thermodynamic, equilibrium, and kinetic behavior of reactions. In the second preliminary experiment, for example, you will learn how use a theoertical relationship—Newton's Law of Cooling—to model how a stainless steel temperature probe cools in a laboratory environment. Later, in the second project-based experiment, you will use measurements on the concentration of OH^- to determine the thermodynamic and equilibrium properties that characterize the solubility of $Ca(OH)_2$ in water.

The data we collect in lab, by itself, rarely provides a direct answer to the questions we are seeking to answer. Instead, we usually have to manipulate the data by calculating values for other variables, by summarizing these results statistically, by organizing the data and results in useful tables, and by visualizing graphically the data and results. The goal of this workshop is to learn how to use a spreadsheet to work with data, and to begin thinking about how to present that data to others using well-designed tables and figures.

The data we will use in this workshop comes from an experiment to determine the composition of United States pennies, which are made from Cu and Zn. Prior to 1982, pennies were made using an alloy of 95% Cu and 5% Zn; after 1982, pennies were made using a core of pure Zn with a coating of Cu, giving a final composition of 97.5% Zn and 2.5% Cu. The data we will use is in the file pennydata.xlsx, which you can download from the course's website; you can open the file in either Excel or Google Sheets. We will work together to process, summarize, evaluate, tabulate, visualize, and analyze this data.

Making Measurements and Preparing Solutions

One of the most important laboratory skills is the ability to prepare a solution accurately and precisely. In your previous laboratory experiences most solutions were prepared for you, or the solutions you made did not require an exact or a reproducible concentration. This semester, however, you will prepare many solutions and you will need to do so with appropriate accuracy and precision. This is not a difficult task; it just requires patience and attention to detail.

One of this experiment's goals is to prepare three solutions, cleverly labeled Solution A, Solution B, and Solution C. To do so you will need to complete some stoichiometric calculations, carefully measure out several reagents using a balance, a volumetric pipet, or a graduated cylinder, and bring these reagents to known final volumes in volumetric flasks. After preparing these solutions you will combine them with a fourth solution (which is provided to you) and observe the resulting reaction. If you prepare your solutions carefully, you will see an interesting result. Before preparing these solutions you first will evaluate the accuracy and the precision of various methods for measuring volume.

Skills Emphasized In This Lab

By completing this lab you will become more comfortable with the following skills:

- measuring volume with appropriate accuracy and precision
- summarizing data using basic statistics, such as means, standard deviations, and confidence intervals
- performing the stoichiometric calculations associated with preparing solutions
- preparing solutions
- maintaining an electronic record of your work

Preparing for Lab

Before your come to lab read the following essays, which are available on the course website: "Accuracy, Precision and Analytical Measurements," "The Art of Measuring Mass," and "The Art of Measuring Volume." In addition, read the instructions for preparing the three solutions and complete all necessary calculations. In addition, complete the appropriate sections of your electronic laboratory notebook, being sure to document your calculations.

Part A. Evaluating the Accuracy and Precision of Glassware for Measuring Volume

As noted in the essay on "Measuring Volume," there are many ways to measure the volume of a liquid reagent, each with its own inherent accuracy and precision. Generally, the more accurately or more precisely you need to know the volume, the more time it takes to make the measurement. For this reason you should carefully think about your need for accuracy and precision when selecting glassware.

In the first part of today's lab you will investigate the accuracy and the precision of the equipment available to you by dispensing 5 mL of water, measuring its mass, and converting that mass to its corresponding volume using water's density, the value for which is a function of temperature. Working with your partners, determine the accuracy and the precision of the following approaches to dispensing 5 mL of water: a disposable dropper, a 10-mL graduated cylinder, and a 5-mL volumetric pipet. The general procedure is described here for using a 10-mL graduated cylinder:

1. Obtain approximately 500 mL of deionized water, record its temperature, and use the table below to find its density at this temperature.
2. Dry a 100-mL beaker and record its exact weight in grams.

3. Dispense 5 mL of water into the beaker using a 10-mL graduated cylinder. Weigh the beaker and the water, and determine the mass of water dispensed by difference.
4. Repeat Step 3 for a total of at least five trials, adding each new aliquot of water to your beaker.
5. Calculate the mean, the standard deviation, the relative standard deviation, and the percent error (assuming a true value of 5 mL). Be sure to think about the number of significant figures that you can reasonably report.

Density of Water as a Function of Temperature

Temperature (^oC)	Density(g/mL)
15	0.9991026
16	0.9989460
17	0.9987779
18	0.9985986
19	0.9984082
20	0.9982071
21	0.9979955
22	0.9977735
23	0.9975415
24	0.9972995
25	0.9970479
26	0.9967867
27	0.9965162
28	0.9962365
29	0.9959478
30	0.9956502

Repeat this procedure for the disposable dropper and the 5-mL volumetric pipet, beginning each with an empty beaker. When using the disposable dropper, assume the "rule of thumb" that 1 mL is equivalent to 20 drops. Create a spreadsheet to record your data and to handle the necessary calculations, including finding means, standard deviations, relative standard deviations, confidence intervals, and percent errors. Be sure to save this file in your shared folder and to identify in your lab notebook the spreadsheet's name. Include a brief analysis of your results in your notebook. As a part of this analysis, identify the equipment that was the most accurate, the least accurate, the most precise, and the least precise.

Part B. Preparing the Solutions

Solution A is 50 mL of 0.23 M $KBrO_3$, also known as potassium bromate. Weigh out the correct amount of $KBrO_3$, transfer it to a 50-mL volumetric flask, and dilute to volume with deionized water. Transfer the solution to a clean, dry, and labeled beaker. Cover the beaker and save it for later.

Solution B is 50 mL of a mixture that contains 0.31 M $CH_2(COOH)_2$, also known as malonic acid, and 0.059 M KBr, which is potassium bromide. Using the general approach used to prepare Solution A, add both solids to the volumetric flask and dilute to volume with deionized water. Transfer the solution to a clean, dry, and labeled beaker. Cover the beaker and save it for later.

Solution C is 50 mL of a mixture that consists of 0.019 M cerium (IV) and 2.7 M H_2SO_4, or sulfuric acid. Begin by preparing 100 mL of 2.7 M H_2SO_4 using the available solution of 42.3% w/w H_2SO_4. This concentration unit, which may be less familiar to you, is a weight-to-weight percent (100.0 g of the solution contains 42.3 g of H_2SO_4). The density of 42.3% w/w H_2SO_4 is 1.25 g solution/mL solution. Using a graduated cylinder, measure out the correct volume of 42.3% w/w H_2SO_4 and slowly add it to a 100-mL volumetric flask that already contains approximately 25 mL of deionized water. Dilute to volume with deionized water and mix thoroughly.

Cerium is obtained using cerium (IV) ammonium nitrate, $Ce(NH_4)_2(NO_3)_6$, which is available as a solid. Using the general approach used to prepare Solution A, use your solution of 2.7 M H_2SO_4 to transfer the $Ce(NH_4)_2(NO_3)_6$ to the 50-mL volumetric flask and dilute to volume using your 2.7 M H_2SO_4. When complete, transfer the solution to a clean, dry, and labeled beaker. Cover the beaker and save it for later.

Did You Prepare the Solutions Carefully?

Now comes the moment of truth! Obtain a clean 250-mL beaker, a magnetic stirrer, and a stir bar. Place the stir bar in the beaker and place the beaker on the magnetic stirrer. Pour all of *Solution A* and all of *Solution B* into the beaker and adjust the stir rate to produce a small vortex (A little solution tornado!). If the solution develops an initial amber color, then wait until it turns colorless before proceeding; this is not a common occurrence. When the solution is colorless, add, at the same time, all of *Solution C* and 3 mL of the already prepared *Solution D* (measured using a graduated cylinder). Examine the resulting reaction for several minutes, observing the interesting and spectacular results. You will have no trouble deciding if your solutions were prepared carefully. Be sure to have your instructor verify your success and to record your observations in your notebook.

Cautions

There are no cautions for this lab other than the normal respect for chemicals.

Waste Disposal

The reaction mixture can be flushed down the drain with plenty of water.

Lab Report

Your electronic laboratory notebook and associated data files serve as your report for this lab. Be sure to place copies of both in your group's shared folder. These documents are due at the end of today's lab. See the sample lab notebook entry for an example of how to document your work. You will find additional guidelines for maintaining a lab notebook in the last section of the lab manual.

Example of Lab Notebook Entry

This is an example of a useful set of entries for an experiment to determine the densities of pre-1982 and post-1982 pennies. The first two entries are completed before coming to lab, the next three entries are completed in lab, and the last entry is completed either in lab or, later, outside of lab. Note that, with the exception of two initial volumes, the data itself is not recorded here; instead, a note indicates that the data is stored in a spreadsheet file.

Statement of Purpose

To determine the density of pre-1982 and post-1982 US pennies. The composition of the penny is mostly copper before 1982 and mostly zinc after 1982.

Experimental Planning

We found two ways to determine the density of pennies. Method 1 is to measure the mass of a penny and then determine its volume by measuring its thickness, t, and diameter, d, where volume $= t \times \pi \times (d/2)^2$. Method 2 is to measure the mass of a penny and then determine its volume by the volume of water it displaces. We've decided to use Method 2 because it seems simpler. To ensure that the change in volume is easier to measure, we will use samples of five pennies each.

Equipment and Reagents

Pennies are taken from a large supply of pre-1982 and post-1982 pennies. A three-digit balance was used to measure mass and a 50-mL graduated cylinder used to measure volumes.

Procedure

Six samples each of five pennies were obtained from the supply of pre-1982 and post-1982 pennies. We confirmed that each penny had an appropriate date, but did not record the individual dates.

To determine the density of pre-1982 pennies, we added approximately 25 mL of water to a 50-mL graduated cylinder. We next placed the cylinder on a balance and tared the balance. We then added five pennies to the graduated cylinder, recording the mass and the volume, repeating this process for six trials.

We repeated this procedure for the six samples of post-1982 pennies.

Experimental Data

Our data is stored in the file pennydata.csv.

The initial volume for the pre-1982 pennies was 25.4 mL, and the initial volume for the post-1982 pennies was 24.9 mL.

Analysis of Data

We found that the density of pre-1982 pennies averaged 8.8 g/mL and that the denisty of post-1982 pennies averaged 7.3 g/mL. These results are reasonable given that pure Cu has a density of 8.96 g/mL and that pure Zn has a density of 7.13 g/mL.

Accuracy, Precision and Analytical Measurements

What are accuracy and precision?

Accuracy is how close a measurement is to its desired or theoretical value. For example, if we need to dispense 25.0 mL of dilute HCl, then dispensing 24.9 mL is more accurate then dispensing 25.7 mL. We usually report accuracy as a percent error

$$\% \text{ error} = \frac{\text{actual value} - \text{expected value}}{\text{expected value}} \times 100$$

which, for the two examples cited above, are $\frac{24.9-25.0}{25.0} \times 100 = -0.4\%$ and $\frac{25.7-25.0}{25.0} \times 100 = +2.8\%$. Note that an error that affects accuracy is either positive or negative.

Precision is the reproducibility of a set of measurements. Three identically prepared solutions with pH values of 6.76, 6.73, and 6.78, for example, are more precise than a duplicate set with pH values of 6.76, 6.54, and 6.92. We usually report precision as a standard deviation, s, which we define as

$$s = \sqrt{\frac{\sum(x_i - \bar{x})^2}{n-1}}$$

where $\bar{x}$ is the average, or mean result, and x_i is one of the n different results. If you closely examine this equation you will see that a standard deviation essentially is the "average" deviation of the individual measurements from their mean value.[1] Note, as well, that squaring the term in the numerator guarantees that the standard deviation is always positive. As an example, the mean pH for the measurements 6.76, 6.73, and 6.78 is

$$\frac{6.76 + 6.73 + 6.78}{3} = 6.757$$

and the standard deviation is

$$s = \sqrt{\frac{(6.76 - 6.757)^2 + (6.73 - 6.757)^2 + (6.78 - 6.757)^2}{3-1}} = 0.0252$$

Alternatively, we can express the standard deviation as a percent relative standard deviation, s_r, or rsd, by dividing s by $\bar{X}$ and multiplying by 100

$$s_r = \frac{s}{\bar{x}} \times 100$$

For the example above, the relative standard deviation is $\frac{0.0252}{6.757} \times 100 = 0.373\%$.[2]

[1] Note that the standard deviation is not a true average because we divide the numerator by $n-1$ instead of by n. The reason for this is not important to us at this time.

[2] Although you can calculate the mean and the standard deviation by hand, it is inconvenient to do so for a large data set. All scientific calculators and spreadsheets have the ability to calculate the mean and the standard deviation; learn how to do so with your calculator and/or favorite spreadsheet.

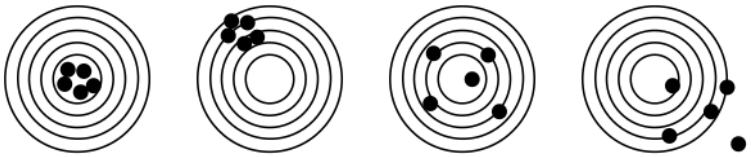


Figure 1: Clusters of five rifle shots illustrating the difference between accuracy and precision.

Is a pH of 6.76 both more accurate and more precise than a pH of 6.8?

Good question. It is tempting to say that a pH of 6.76 is more accurate than a pH of 6.8 because it contains more significant figures, but this is not necessarily correct. In fact, if the instrument used to measure the pH is not calibrated, then neither pH reading is accurate.

Regardless of its accuracy, we can say that a pH of 6.76 is known more precisely to us than a pH of 6.8 because the absolute uncertainty for the first measurement is ± 0.01 while that for second is ± 0.1.[3] If the pH meter is calibrated properly, then a more precise measurement can lead to a smaller percentage error and, consequently, to better accuracy.

If a measurement is accurate, must it also be precise?

Interestingly, the answer to this question is no. As we see in Figure 1, there are four possible combinations of accuracy and precision.

The target at the far left shows both accuracy and precision as the shots are clustered together (they are precise) in the target's center-most ring (they are accurate). The next example shows results that are precise, due to a tight clustering of the shots, but inaccurate because they are at the target's outer edge instead of its center. The third example is considered accurate because the five shots cluster around the target's center, but they are not precise because the individual shots are quite far apart from each other. The final example shows a dispersion of shots that is both inaccurate and imprecise. Note that the average for a set of measurements may be accurate even if the individual measurements deviate significantly from the desired or theoretical value.

What factors affect accuracy and precision?

Three main factors affect the accuracy and the precision of a measurement: the quality of the equipment we use to make the measurement, our ability to calibrate the equipment, and our skill using the equipment. These factors are considered further in this section.

We cannot make an accurate measurement if our equipment is not calibrated properly. To calibrate equipment we analyze a system where the response is known to us and either adjust the equipment to give that response or to determine the mathematical relationship between the measured result and its known value. The two examples of accurate target shooting in Figure 1—the target on the far left and the target second from the right—require that you calibrate the rifle's scope so that an accurate result is possible. In addition, the potential accuracy of any individual measurement is greater with better quality equipment or instrumentation; the better the scope, the closer each shot is to the target's center.

Precision, on the other hand, is influenced by both the quality of the equipment and the skill of the person using it. The importance of the user's skill is obvious; when shooting a rifle, for example, you must have a steady hand to achieve a tight, precise pattern of shots. Although less obvious, the quality of the equipment

[3] We also can think about this in terms of relative uncertainty where 6.76 has a relative uncertainty of 1 part in 676, or 0.148%, and where 6.8 has a relative uncertainty of 1 part in 68, or 1.5%.

is equally important. The smooth bored muskets used during the Revolutionary War, for example, produced less precise shot patterns than those of a modern rifle because they lacked grooved bores.

Shouldn't I always strive for the best possible accuracy and precision?

Surprisingly, the answer to this question is a resounding NO. Improving accuracy and precision almost always comes at the expense of time and money. Calibrating an instrument, for example, takes time and the better the quality of the instrument, the more the instrument costs and the less likely it is to be freely available for your use because fewer units are available. You can save a lot of time and aggravation in lab if you learn to make the most accurate and precise measurement only when it is absolutely necessary.

So, how do I decide whether a measurement needs to be accurate or precise?

The simplest answer is this: if the result of the measurement is used in a calculation, then you should try to make the measurement with a suitable level of accuracy and precision. Note the use of the adjective suitable. If you must know the final result to within $\pm 1\%$, then the requirements you place on your individual measurements are more stringent than if your final accuracy must be within $\pm 10\%$. The same observation holds true for precision.

A useful method to determine how accurate or precise to make a measurement is to use significant figures as a guide. For example, if a procedure calls for a 1-L solution of 0.1 M NaCl, then you do not need an accurate or a precise measurement of the mass of NaCl or the volume of water. You can simply measure approximately 5.8 g of NaCl and dissolve it in a 1-L reagent bottle and know the molarity is 0.1 to within one significant figure. Or, if a procedure requires that you add approximately 0.01 g of a reagent to each sample, weigh out the first portion to judge the amount and simply add a similar portion to the remaining samples; there is no need to weigh out and record each addition. On the other hand, if a procedure calls for you to make 1.000-L of a 0.1000 M solution of NaCl, then it is necessary to weigh out 5.844 g of NaCl and to use a 1-L volumetric flask to prepare the solution.

When you use significant figures as a guide to determining a measurement's appropriate accuracy and precision, be sure to consider how the value is used in a calculation. If a procedure calls for a solution of NaCl with a nominal concentration of 0.1 M but the exact concentration is essential when you analyze your results, then you must obtain an accurate and precise mass of NaCl and dilute to volume in a volumetric flask; the exact concentration is then calculated. Alternatively, you can prepare the solution without regard to accuracy and precision and determine the concentration of NaCl experimentally, a process we call standardization. Let the procedure from which you are working guide in you making such decisions.

The Art of Measuring Mass

To weigh out a portion of a reagent—typically a solid, but, on occasion, a liquid—we use an analytical balance. Although there are several types of balances, the most common is the electronic pan balance. The sample is placed on a pan, which displaces the pan downward due to the force of gravity acting on the sample. The balance's circuitry detects this downward motion and supplies an opposing electromagnetic force to counterbalance that from the sample. The magnitude of this force is proportional to the sample's mass. If the balance is calibrated, then an accurate measurement of mass is possible.

Electronic pan balances are available with a variety of precisions, which we define here as the number of decimal points to which we can weigh the sample. For most samples a three-digit balance (an uncertainty of ± 0.001 g) is sufficient, although a four-digit balance (± 0.0001 g) is needed in some cases.

Balances are susceptible to air currents that produce small deflections in the balance's pan and, as a result, produce fluctuations in the recorded mass. This is particularly true for four-digit balances, which is why the balance pan is enclosed within a housing with sliding glass doors that remain closed when you record the mass.

For a reagent that is not hydroscopic—that is, a reagent that does not absorb water—the sample is weighed directly into a suitable container. Because many items of glassware have small openings that make it hard to directly add a solid reagent or have a mass greater than the balance's capacity, solids often are dispensed onto weighing paper or a weighing boat. In either case, the weighing paper or weighing boat is placed on the balance pan and the balanced is tared so that it registers a mass of 0.000 g. A spatula or scoopula is used to transfer the desired amount of reagent to the paper or boat. This is done carefully to avoid spilling reagent on the balance pan as this results in an inaccurate reading (and may damage the balance as well). The solid reagent is transferred to another container using a small stream of solvent.

If a reagent is hydroscopic or it cannot be transferred using a solvent, then samples are obtained in a different manner. First, we place a portion of the reagent greater than we need into a small, closed weighing bottle. Weigh the bottle and then transfer a portion of the sample to the appropriate container. Reweigh the bottle and determine the sample's mass by difference.

The Art of Measuring Volume

Chemists use glassware to measure a reagent's volume with the choice of glassware determined by the required accuracy and precision. In general, we divide glassware into two broad categories: glassware for approximate measurements and glassware for accurate and precise measurements.

Glassware for Approximate Measurements

Five common types of glassware are used to make approximate measurements of volume: reagent bottles, beakers, Erlenmeyer flasks, graduated cylinders, and disposable pipets.

- A *reagent bottle* is the least accurate as it seldom has any marks to indicate even an approximate volume. Adding 0.1 moles of a reagent to a 1-L bottle and adding water to the top of the bottle's rounded shoulder produces a solution that is approximately 0.1 M.
- A *beaker* or an *Erlenmeyer flask* has several graduation marks on its side. These marks are accurate to within approximately ±10% of the flask's maximum volume. For example, adding water to the 100 mL mark on a 250-mL beaker generally gives a net volume between 75 mL and 125 mL.
- A *graduated cylinder* provides a more accurate measurement of volume than a beaker or an Erlenmeyer flask. A typical graduated cylinder is accurate to within ±5% of the cylinder's maximum volume. When delivering 5 mL using a 10-mL graduated cylinder, for example, the actual volume is probably between 4.5 mL and 5.5 mL.
- A *disposable pipet* is a useful way to add a reagent whose volume is given in drops. A common estimate is that 20 drops is approximately equivalent to 1 mL, although this varies greatly from brand-to-brand.

In general, the precision for these types of glassware is better than their respective accuracies, although their precision seldom is an issue.

Glassware for Accurate and Precise Measurements

Sometimes we need to be know a reagent's exact volume. When this is the case we worry both about accuracy (How close is it to 10 mL?) and precision (How much variation might we expect from one aliquot to the next?). Three types of glassware are common options when we need an accurate and a precise measurement of volume: volumetric flasks, volumetric pipets, and burets. In general, the precision of these types of glassware is better than their respective accuracies.

Volumetric Flask. When filled to its calibration mark, a volumetric flask contains a specified volume of solution, usually to within $\pm 0.03 - 0.2\%$ of the stated value, depending on the size of the volumetric flask (although accuracy is improved by determining the mass of water contained within the flask and converting to volume using water's known temperature-dependent density, a process called a calibration). A volumetric flask with a capacity of less than 100 mL generally measures volume to the hundredths of a milliliter, whereas a volumetric flask of 100 mL or greater capacity measure volume to the tenth of a milliliter. For example, a 10-mL volumetric flask contains 10.00 mL, but a 250-mL volumetric flask contains 250.0 mL. This is an important issue to consider when keeping track of significant figures.

Note the use of the verb *contain* in describing a volumetric flask's properties; this description is important. Although a 100-mL volumetric flask contains exactly 100.0 mL (±0.1 mL) when filled to its calibration mark, it cannot deliver 100.0 mL to another container because you can never completely transfer a liquid from one container to another; some liquid, even if it is only a few drops, remains behind.

Because a volumetric flask contains a solution of known volume, it is useful when you need to prepare a solution with an exact concentration. A known amount of reagent is transferred to a clean volumetric flask and enough solvent added to dissolve the reagent. After the reagent is dissolved, additional solvent is added in several portions, mixing the solution after each addition. The final adjustment of volume to the flask's

calibration mark is made dropwise using a disposable pipet or a solvent dispensing bottle. To complete the mixing process, the volumetric flask is capped, and then inverted and shaken at least 10 times.

Volumetric Pipets. A volumetric pipet delivers a specified volume of solution. Several styles of volumetric pipets are available, but the most common and the most accurate is a transfer pipet. Transfer pipets consist of a long tube with a bulge in the middle and a single calibration mark. A transfer pipet's accuracy is similar to that of a volumetric flask of equal volume; thus, for example, a 100-mL transfer pipet will deliver 100.0 mL of solution (±0.1 mL). As with a volumetric flask, accuracy is improved by calibrating with water. The other common type of volumetric pipet is a Mohr pipet, which is a narrow tube with multiple calibration marks that allows you to dispense volumes of variable size; thus a 5-mL Mohr pipet is used to deliver any specific volume between 0 mL and 5 mL. In this lab we will make exclusive use of transfer pipets.

Note that a volumetric transfer pipet *delivers* a known volume of solution, whereas a volumetric flask *contains* a known volume. When filled to its calibration mark, a transfer pipet always contains a volume greater than that delivered. When delivery is complete a small amount of solution remains behind. A transfer pipet, therefore, is always contaminated with a small amount of the last solution for which it was used.

Because a transfer pipet delivers a known volume of solution, it is an excellent way to deliver an accurate and a precise amount of reagent. To use a transfer pipet, first rinse it with deionized water to remove any traces of the last solution remaining in the pipet. Then, since water is, itself, a contaminant (it will dilute your solution), fill the pipet once with your solution and dispense it to waste. If you have a limited amount of your solution you can partially fill the pipet, seal the top and bottom and rock it back and forth to rinse the pipet's inner surfaces. Any residual amount of solution remaining in the pipet is similar enough in composition to your original solution such that dilution errors are inconsequential.

To fill a transfer pipet, use suction from a rubber bulb to pull the solution above the pipet's calibration mark (never use your mouth to suck a solution into a pipet). Remove the suction bulb and place your fingertip over the top of the pipet. While slowly twisting the pipet, allow the solution's level to drop until it reaches the calibration mark. Wipe the outside of the pipet to ensure it is dry and, if necessary, remove any solution that clings to the pipet's tip. Place the pipet over the container in which the solution is to be dispensed. Remove your fingertip and allow the pipet's contents to drain into the container. Touch the tip of the pipet to the container's wall to ensure the final drop is dispensed. A small, residual amount of solution will remain in the pipet; do not try to force this into the container. Practice this technique until you confidently can use the pipet.

Burets. A buret is a tube with graduated markings and a stopcock on its bottom end. Although a volumetric pipet can deliver only the one specific volume for which it is designed, a buret can deliver any volume up to its maximum capacity. The accuracy of the burets available to you is approximately ±0.05 mL.

To use a buret, fill it with your solution and fill the buret's tip by briefly opening the stopcock. Place a receiving flask below the buret, open the stopcock until the desired amount of solution is dispensed, and record the volume delivered.

Additional Important Details

Two important precautions are needed when you work with volumetric pipets, volumetric flasks, and burets. First, the volume delivered by a volumetric pipet assumes the glassware is clean. Dirt and grease on the inner surface of a volumetric pipet will prevent it from draining evenly, leaving drops of the reagent on the pipet's walls and delivering less reagent than expected. For a volumetric flask, if drops of reagent remain above the calibration mark, then the flask will contain more than its specified volume.

Second, when you fill a pipet or a volumetric flask, set the liquid's level exactly at the calibration mark. The liquid's top surface is curved into a meniscus, the bottom of which should be exactly even with the glassware's calibration mark. To avoid parallax errors, the meniscus is adjusted with your eye at the same level as the calibration mark. If your line of sight is from above the calibration mark, for example, then you will tend to overfill the volumetric pipet or volumetric flask.

Newton's Law of Cooling

Introduction

A hot object in contact with a cooler environment loses heat by forced convection. The rate at which heat is lost depends on a number of factors, including the difference between the object's temperature and that of its surroundings. Mathematically, this relationship is known as Newton's Law of Cooling and is expressed as

$$\frac{dT(t)}{dt} = -\kappa \left[T(t) - T_s\right]$$

where $T(t)$ is the object's temperature at time t, T_s is the temperature of the surroundings, and κ is a constant whose value depends upon the object's properties.

This form of Newton's law is hard to interpret as generally humans are not particularly adept at visualizing a differential equation. If we integrate the equation, however, we see easily that temperature decreases exponentially with time

$$T(t) = T_s + (T_0 - T_s)\, e^{-\kappa t}$$

where T_0 is the object's original temperature. In this experiment you will examine the validity of Newton's law for the cooling of a metallic temperature probe.

Skills Emphasized In This Lab

By completing this lab you will become more comfortable with:

- using the LabQuest Mini interface and LoggerPro software to collect and analyze data
- using a regression analysis to fit a theoretical equation to experimental data
- preparing useful figures and tables for reporting data
- communicating the results and conclusions of your work to others through a written report

Preparing for Lab

Review the essays "How to Design a Table and Figure," and "The Mathematical Modeling of Experimental Data," which are available on the course's website, and complete the appropriate sections of your electronic notebook before coming to lab.

Procedure

Begin by heating approximately 500 mL of deionized water to a temperature between 50°C and 100°C using a hotplote; the actual volume of water is not important. Connect the LabQuest Mini interface to a computer and attach two temperature probes to analog ports. Set your data acquisition parameters for a time-based experiment lasting ten minutes with a sampling rate of 20 points per minute.

When the temperature of the water in your beaker is within the desired range, place your two temperature probes in the water and allow them to equilibrate for at least one minute. Remove the probes, wipe off any residual water with a Kimwipe, and suspend the probes in the air so that they are not close to your hotplate and so that they are not close to each other. Once your probes are positioned, initiate data collection. When data collection is complete be sure to store your data before you continue with the next trial. Repeat

this procedure for a minimum of five trials. ***Do not try to begin these trials at the same initial temperature***; in fact, it is best if you have a range of initial temperatures between 50°C and 100°C.

Data Analysis

Analyze the data for each trial and for each probe by fitting a suitable equation to the data, determining values for T_0, for T_s, and for κ.

Cautions

There are no serious cautions for this lab other than using care when handling hot water.

Waste Disposal

This is easy – it's just water!

Lab Report

For this report, focus on the ***results and conclusions section only***, paying particular attention to developing a narrative that uses tables and figures to summarize your data, that clearly analyzes your results, and that reaches clearly stated conclusions.

Of particular interest is your determination of values for T_0, T_s, and κ. At a minimum, your report should address the following questions:

- What are the expected (theoretical) values for these variables or are their expected values unknown?
- If expected values for T_0, T_s, and κ are known, how accurate are your experimental results?
- How reproducible are your results for each variable and is this reproducibility (or lack or reproducibility) expected?
- How similar are your results for the two probes and is this similarity (or lack or similarity) expected?
- Is Newton's Law obeyed for the cooling of a hot probe and, if not, can you explain why?

It might help to do some research on Newton's law, particularly with respect to its limitations and/or assumptions. Be sure to define Newton's law and its variables at the beginning of your report. Limit this report to approximately 3–5 pages of double-spaced text, tables, and figures. At a minimum, you should have at least one well-designed table that presents your results for T_0, T_s, and κ, and at least one figure that provides visual evidence that your data follows (or does not follow) Newton's law.

Working together, prepare a draft of your report and then, after receiving feedback on this draft, prepare a final report. Deadlines are listed on the course's website. See the sample results and conclusions for an example of how to prepare this part of a formal report. You will find additional guidelines for writing reports in the last section of the lab manual.

Sample Results and Conclusions Section

Shown here is an example of how to present your results and conclusions for an experiment, drawing on data from the data analysis workshop. Note that the data sits within a narrative that points the reader to the data and tells the reader what the data means, and that the narrative ends with a clear conclusion. Note, also, that subheadings are used to organize the presentation of the data.

Results and Conclusions

In pubishing their method for determining the composition of bimetallic coins, Curtis and Znesky did not take into consideration how the relative abundance of the two metals might affect the accuracy and precision of an analysis. Pennies manufactured in the United States prior to 1982, which have a known composition that is 95% Cu and 5% Zn, make a useful test of Curtis and Znesky's method.

Determining the Density of Pre-1982 Pennies

Table 1 provides a summary of our results for six trials, each consisting of a single sample of five pennies. As expected, the mean density of 8.81 g/mL falls between the density of 8.96 g/mL for pure copper and of 7.14 g/mL for pure zinc. It also is much closer to the density of pure copper, consistent with our expectation that pre-1982 pennies contain significantly more copper than zinc.

Table 1: Determination of Density for Pre-1982 US Pennies

mass (g)	volume (mL)	density (g/ml)
15.674	1.8	8.71
15.452	1.7	9.09
15.611	1.8	8.67
15.374	1.7	9.04
15.629	1.8	8.68
15.542	1.8	8.63
Summary Statistics		
mean		8.81
standard deviation		0.204
confidence interval		0.163
upper limit		8.97
lowerlimit		8.81

Pennies (five per sample) were used as is without removing surface tarnish or rejecting those with obvious flaws.

Determining the Composition of Pre-1982 Pennies

To determine the composition of a penny from its density, we use the following equation

$$d_{\text{penny}} = d_{\text{Cu}} \times x + d_{\text{Zn}} \times (1 - x)$$

where the density of copper, d_{Cu}, is 8.96 g/mL, the density of zinc, d_{Zn}, is 7.14 g/mL, and x and $1 - x$ are the fractions of copper and zinc in the penny. Using the data from Table 1 gives the fraction of copper and

fraction of zinc shown in Table 2. The mean composition of pre-1982 pennies, using this data, is 91.5 % Cu and 8.5 % Zn, which is in modest agreement with the expected composition of 95% Cu and 5% Zn.

Table 2: Composition of Pre-1982 US Pennies

mass	volume	density	fraction Cu	fraction Zn
15.674	1.8	8.71	0.861	0.139
15.452	1.7	9.09	1.071	-0.071
15.611	1.8	8.67	0.842	0.158
15.374	1.7	9.04	1.046	-0.046
15.629	1.8	8.68	0.848	0.152
15.542	1.8	8.63	0.821	0.179

Evaluating the Utility of Curtis and Znesky's Analytical Method

Although there is modest agreement between the experimentally determined mean composition of pre-1982 United States pennies and the expected composition as reported by the United States Mint, the data here is troubling. In particular, the six samples of pennies in Table 2 seem to fall into two groups, one that averages 84% Cu and 16% Zn (trials 1, 3, 5, and 6) and one that averages 106% Cu and -6% Zn (trials 2 and 4). The result for the latter group is, of course, unreasonable as the composition of Cu cannot excede 100% and the concentration of Zn cannot be less than 0%. The problem here is a well-known limitation of analyzing a sample that consists of just two species where the concentration of one species is close to the upper limit. The problem is compounded by the limited number of significant figures, two, associated with the measurement of volume. The relative uncertainty in the volume of slightly more than 1 part in 20 (5%) is simply too large to yield accurate and precise results. Future work will focus on exploring ways to improve the precision of the measurement of volume.

How To Design a Table and a Figure

When you prepare a table or a figure you have two goals: to convey information to your reader **and** to make it easy for the reader to see that information. This "How to. . . " document will help you prepare more effective tables and figures. It uses the penny data we analyzed during our first lab.

How to Design an Effective Table

We read a document from left-to-right and from top-to-bottom because that is the standard for written English. The dominate motion is left-to-right, which explains two important features of a good table: we usually place our variables (the things we measured) in columns and we usually place our samples (the things we made measurements on) in rows, and we organize the columns so the most important variable (our result) is on the far right. Here is a good organization for our pre-1982 penny data where mass and volume are our measurements and density is the result.

mass	volume	density
15.674	1.8	8.71
15.452	1.7	9.09
15.611	1.8	8.67
15.374	1.7	9.04
15.629	1.8	8.68
15.542	1.8	8.63

This is nicely organized information, but it fails to provide the reader with enough information to understand what the table represents. To provide this information, we add units to our variables, and add a short, descriptive title, placing it above the table so that it is the first thing the reader sees. Footnotes are another way to provide additional information. *Note that a table does not include a caption; instead, your must guide the reader to the table in your narrative and then tell your reader what important information they will find there.*

Table 1: Determination of Density for Pre-1982 US Pennies

mass (g)	volume (mL)	density (g/ml)
15.674	1.8	8.71
15.452	1.7	9.09
15.611	1.8	8.67
15.374	1.7	9.04
15.629	1.8	8.68
15.542	1.8	8.63

Pennies (five per sample) were used as is without removing surface tarnish or rejecting those with obvious flaws.

When appropriate, we include a statistical summary of our results, as seen at the bottom of Table 2.

As a final check of our table, ask yourself this question: Can your reader confirm the results (in this case, the reported densities) using only the measurements in your table (in this case, the reported masses and volumes) and, perhaps, a small number of constants available elsewhere in your report. If the answer is no, then you need to rethink your table's design!

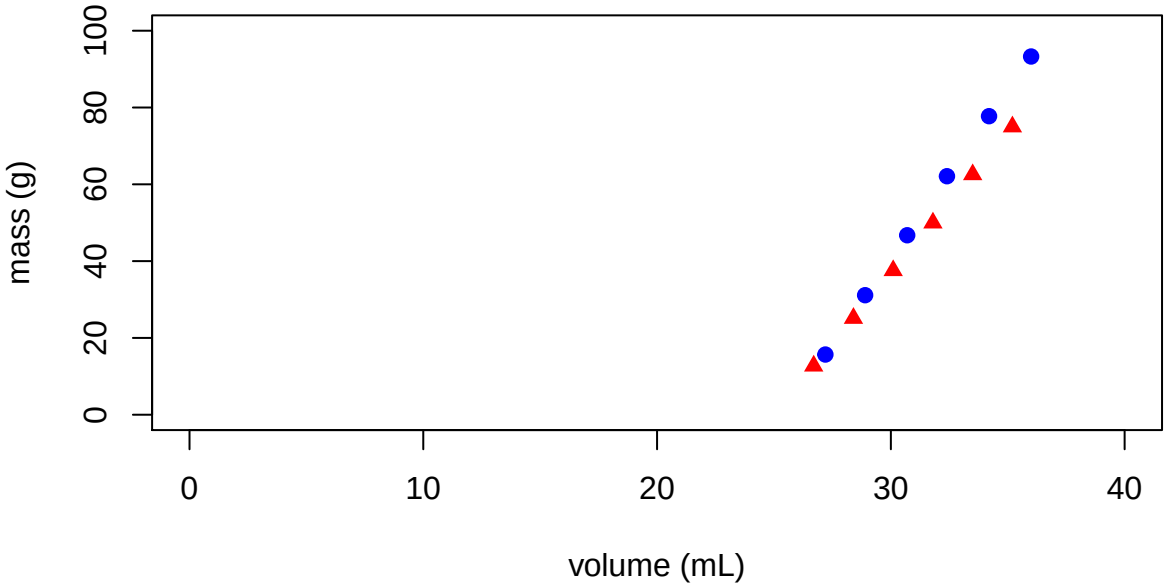
Table 2: Determination of Density for Pre-1982 US Pennies

mass (g)	volume (mL)	density (g/ml)
15.674	1.8	8.71
15.452	1.7	9.09
15.611	1.8	8.67
15.374	1.7	9.04
15.629	1.8	8.68
15.542	1.8	8.63
Summary Statistics		
mean		8.81
standard deviation		0.204
confidence interval		0.163
upper limit		8.97
lowerlimit		8.81

Pennies (five per sample) were used as is without removing surface tarnish or rejecting those with obvious flaws.

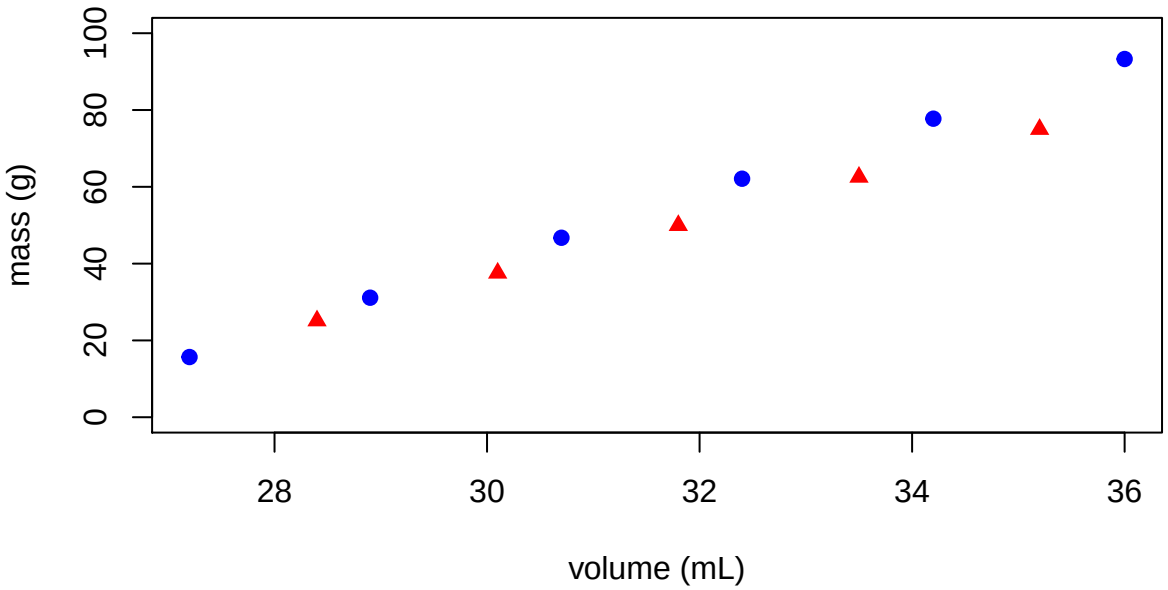
How to Design an Effective Figure

When we look at a painting, our eyes move to its most salient features before we move outwards, searching for additional information. When we look at a figure, our eyes do the same thing, locating the data first and then searching for information that gives addtional meaning to the data. A well-designed figure, therefore, places the data at or near the center and distributes clues around the data in the form of labels and legends. In a museum, a detailed description of a painting, which provides important information about the artist and painting's context, is placed to its side where it does not intrude on our experience of the image itself. For a figure we include this information in a descriptive caption placed **below** the figure where the viewer can find it when they are ready (if we place it above the figure, the viewer will read the caption before they examine the figure; we want the viewer to see the figure before they read about it). Here is a first draft of a figure that shows how the cumulative mass and the cumulative volume change for pre-1982 pennies and post-1982 pennies using the typical default parameters of many plotting programs.



Because our volumes are offset by an initial volumes of 25.4 mL for the pre-1982 pennies and of 24.9 mL for

the post-1982 pennies, the data is shifted to the right side of the figure, which makes it more difficult for the viewer to take in the information on the y-axis and to study the differences between the data sets. We want the most important information at the figure's center, which we accomplish by rescaling the x-axis.



Adding regression lines, a legend, and a detailed figure caption leaves us with an effective figure. The close proximity of the figure and the figure caption allows the two to convey to the reader a complete story.

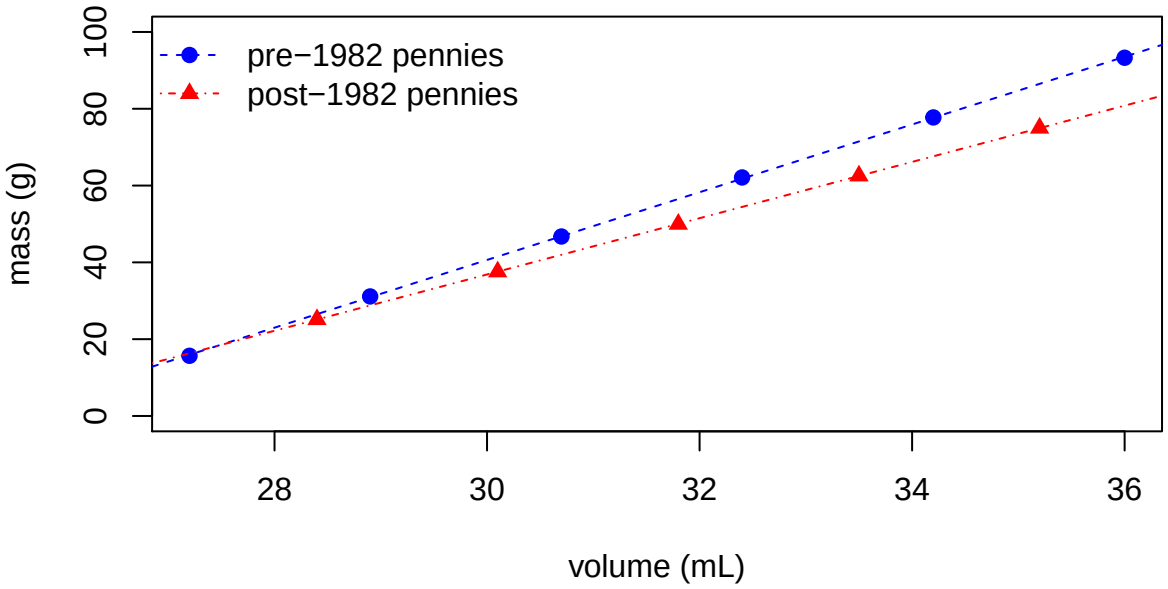


Figure 1: One way to find the density of pennies is to plot the cumulative mass as a function of the cumulative volume. The slope of the resulting regression lines give the densities in g/mL. For pre-1982 pennies the density is 8.82 g/mL and for the post-1982 pennies the density is 7.33 g/mL. The difference in the two densities reflects the change in the composition of a penny. Prior to 1982, pennies were minted using an alloy of 95% Cu and 5% Zn. After 1982, the composition was changed to a pure Zn core with a thin overlayer of Cu, giving a penny that is 97.5% Zn and 2.5% Zn. Note that the volumes on the x-axis are the combined volume of the pennies and the water initially present in the graduated cylinder.

Mathematical Modeling of Data

In an experiment we usually measure a response for one variable as a function of a second variable whose value is directly under our control. In the language of experimental design, the variable under our control is the *independent variable* and the variable whose value we measure is the *dependent variable*. For example, consider the following hypothetical data for an experiment designed to determine the effect on a Princess' sleep (the dependent variable) of placing peas (the independent variable) under her mattress.

number of peas	average hours of sleep
1	8.72
2	7.86
3	6.29
4	5.68
5	4.22

A quick scan of this data suggests there is an inverse linear relationship between the number of peas and the average hours of sleep: fewer peas results in more sleep. Seeing this relationship we might ask questions such as "What is the mathematical relationship between the average hours of sleep and the number of peas placed under the mattress?" or "If we place seven peas under the mattress, how many hours might the Princess sleep?"

Regression Analysis

To answer questions such as those suggested above requires a mathematical equation that models the data. This is the realm of a regression analysis. For a straight-line relationship the model equation is

$$y = \beta_0 + \beta_1 x \tag{1}$$

where y (the dependent variable) is the average hours of sleep, x (the independent variable) is the number of peas placed under the mattress, β_0 is the average hours of sleep in the absence of any peas (the value of y when x is zero, or the y-intercept) and β_1 is the average hours of sleep lost per pea (which also is the slope of the line or the rate of change of y relative to x; that is, $\frac{\Delta y}{\Delta x}$). The terms β_0 and β_1 are adjustable fitting parameters of the model. The goal of a regression analysis is to find the best values for β_0 and for β_1 such that the net difference between the experimental values of y and those values predicted by the model is as small as possible. The mathematical details of how this is accomplished are too involved for this course; nevertheless, both Excel and LoggerPro have functions that will complete a regression analysis and add the model to a plot of your data, as shown in Figure 1, where the mathematical model is

$$\text{average hours sleep} = -1.12 \times \text{ number of peas} + 9.91 \tag{2}$$

Is My Regression Model A Good Model?

Of course we can fit a straight-line to any set of data, even if the data clearly are not linear. For this reason it is important to examine the results of a regression analysis and determine whether the model is reasonable. One common method to evaluate what often is called the model's "goodness of fit" is to look at the correlation coefficient, R, or the coefficient of determination, R^2. A value of R close to $+1$ or to -1 (or an R^2 close to $+1$) suggests the model does a good job of explaining its data and a value for R or for R^2 close to 0 suggests that the model is inappropriate. For the data in Figure 1, the values of R and R^2 are 0.986 and 0.981, respectively.

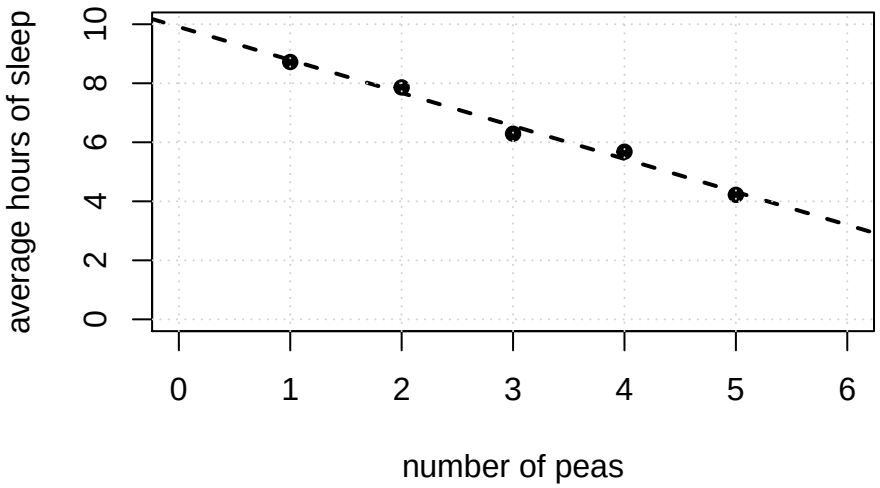


Figure 1: Average hours of sleep of the Princess as a function of the number of peas placed under her mattress. Each point is the average of three consecutive nights. The dashed line is equation 2 is the result of fitting equation 1 to the data.

Another common measure of a model's goodness of fit is the root-mean-square error, which is defined as

$$\text{RMSE} = \sqrt{\frac{\sum(y_i - \hat{y}_i)^2}{n}}$$

where y_i is the experimental value for the i^{th} value of x, $\hat{y}_i$, which is read as "y-hat," is the model's predicted value for y_i, and n is the number of measurements. The smaller the root-mean-square error, which essentially is the average difference between the data and the model—the better the fit between the model and the data. For the data in Figure 1, the RMSE is 0.080.

By themselves, the correlation coefficient, the coefficient of determination, and the root-mean-square error are not always useful measures of a model's suitability. Large values for R or for R^2, or a small RMSE may falsely lead you to assume that a model provides an accurate description of the data. Before you accept the result of any mathematical model, prepare a plot of the data and the predicted model and examine them critically. If the model is a good model then the regression line should closely fit the data with individual data points scattered randomly around the model's predicted curve. Note that although the regression model in Figure 2 has a favorable value for R^2, the data clearly show evidence of curvature with values of y for low and for high values of x falling below the model's curve and values of y for intermediate values of x falling above the model's curve. A quadratic model of the form $y = \beta_0 + \beta_1 x + \beta_0 x^2$ might be a better choice for this data.

Interpolating and Extrapolating From a Model

The reason for developing a regression model is to predict the value of the independent variable (or to predict the value of dependent variable) for a sample where its value is unknown. Recall that our model for the data in Figure 1 is

$$\text{average hours sleep} = -1.12 \times \text{ number of peas} + 9.91$$

We can use this model in two ways. If we know how many peas we plan to place under her mattress, we can predict how long the Princess will sleep. Alternatively, if we measure the number of hours the Princess sleeps on a given night, we can predict how many peas were placed under her mattress. These are powerful and useful applications of a model; however, when when we use a model to a make prediction we need to be

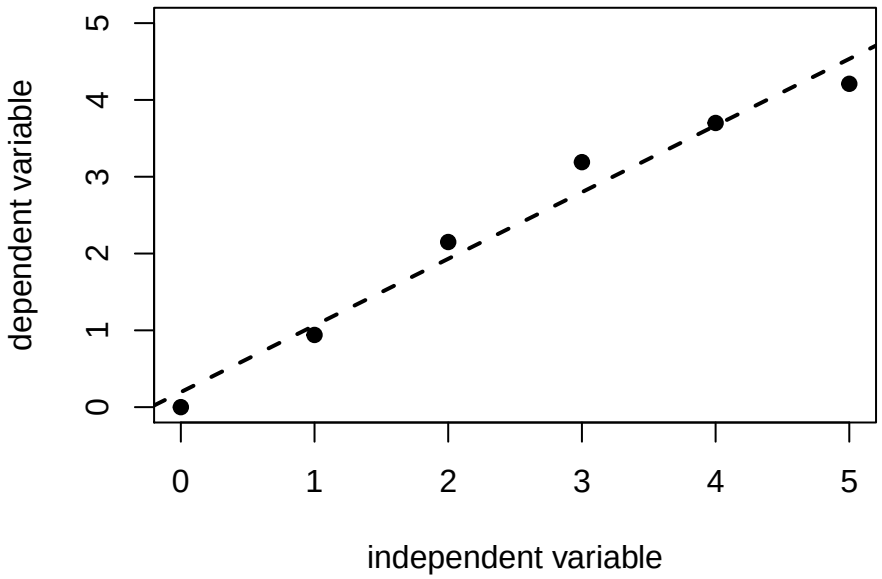


Figure 2: Example of data where a straight-line regression model provides a poor fit to the data even though the values for R and for R^2 are, respectively, 0.973 and 0.967.

careful how we interpret the result. Here we need to make an important distinction between interpolation and extrapolation.

To develop the model in Figure 1 we used samples of 1, 2, 3, 4, and 5 peas. Based on our analysis of this data, we have every confidence that the mathematical model works well for this range of peas and hours of sleep. If we limit the model to making a prediction within this range, a process called interpolation, then our confidence in our prediction's accuracy is high. For example, if we determine that the Princess slept 6.0 hours last night, then we can predict that there were 3.5 peas under her mattress and be confident in this prediction.

Extending our model to values of the dependent variable and the independent variable that we did not study, a process called extrapolation, is possible but it is more susceptible to uncertainty. If the Princess sleeps 10 hours our mathematical model suggests there probably were no peas placed under her mattress. This extrapolation of our model to smaller values of the independent variable seems reasonable as there is no reason to believe that the linear behavior between 1 and 5 peas does not hold between 0 and 1 peas.

Can we safely extrapolate the model to larger values of the dependent variable or the independent variable? What is our prediction, for example, if we place 10 peas under the Princess's mattress? Using our model, we predict that the Princess will sleep for -1.29 hours, a result that is impossible. We clearly cannot extrapolate our model this far. Given this contradiction, it is tempting to modify our model by assuming that it is valid until the dependent variable reaches zero. Such an assumption, however, is still fraught with potential uncertainty. Suppose the Princess sleeps for 2.0 hours. Extrapolating our model leads us to predict that there are 7 peas under the mattress; however, it also is possible that the Princess will sleep a minimum of two hours regardless of the number of peas under the mattress. If true, then we cannot extrapolate the model to 2.0 hours or less of sleep.

When you build a mathematical model it is important to consider how you plan to use the model and, if it is possible and practicable, to ensure that the range of values for the independent variable spans the range of values you wish to model. In this way your predictions rely on interpolations and not extrapolations. If an extrapolation is necessary, be sure to consider its limitations.

Determining the Amount of Acetic Acid in Vinegar

Introduction

Vinegar is a dilute solution of acetic acid, CH_3COOH, that, according to the Food and Drug Administration, should contain at least 4.0 g of acetic acid per 100 mL of solution. Because acetic acid is a weak acid, its concentration in any solution, including vinegar, is easy to determine by titrating a sample of known volume using a strong base such as NaOH.

$$CH_3COOH(aq) + OH^-(aq) \longrightarrow H_2O(l) + CH_3COO^-(aq)$$

Because the reaction's stoichiometry is 1:1, the moles of NaOH used is equivalent to the moles of acetic acid in the sample of vinegar. The concentration of acetic acid in vinegar is then easily calculated.

Skills Emphasized In This Lab

By completing this lab you will become more comfortable with:

- calibrating and using a pH electrode
- performing an automated titration using a drop counter
- standardizing a solution by an acid-base titration
- learning to find a titration curve's equivalence point
- writing a succinct procedure that provides an *experienced* scientist with enough information to duplicate your work

Preparing for Lab

Before coming to lab, review the essays on "Titrimetry" and "Potentiometry," and complete the appropriate sections of your electronic notebook.

Procedure

Begin by preparing approximately 300 mL of nominally 0.1 M NaOH by transferring an appropriate amount of NaOH into a beaker and dissolving it in 300 mL of deionized water. Because NaOH is not available in a pure form, we cannot calculate the exact concentration of this solution from the mass of NaOH taken and the volume of solution used. Instead, you will determine the solution's concentration by titrating it against the weak acid potassium hydrogen phthalate, $C_8H_5O_4K$, also known as KHP, which is available in a pure form. This process is called a standardization, the reaction for which is

$$OH^-(aq) + C_8H_5O_4^-(aq) \longrightarrow H_2O(l) + C_8H_4O_4^{2-}(aq)$$

To complete the titration, set up and calibrate the drop counter. Fill the reservoir with your solution of nominally 0.1 M NaOH. Transfer an approximately 0.3 g portion of KHP into a small beaker and dissolve it in approximately 50 mL of deionized water. If this is not sufficient to cover the tip of the pH probe, then add additional water. Calibrate your pH electrode and suspend it in the solution of KHP. Add a small stir bar and gently stir the solution. Begin the titration and continue until you obtain a complete titration curve. Locate the equivalence point and calculate the molarity of your NaOH solution.

To analyze vinegar for its concentration of acetic acid, pipet 2.00 mL of vinegar into a small beaker and add enough deionized water to cover the pH electrode's sensing bulb when it is suspended in the beaker. Gently

stir the solution and titrate the sample with your NaOH until you obtain a complete titration curve. Locate the equivalence point and calculate the %w/v acetic acid in the vinegar.

To ensure you have sufficient data for both the standardization of NaOH and the analysis of vinegar, alternate between these two titrations. Gather as much data as you can by the end of the lab period.

Caution

There are no cautions for this lab other than the normal respect for chemicals.

Waste Disposal

All solutions may be disposed of down the drain with copious amounts of water.

Lab Report

For this report, focus your draft on the ***procedure section only***, limiting yourself to a single double-spaced page of text (if you find yourself needing more space then this, then you are including too much detail!). You may find it useful to make a list of everything you did and every measurement you recorded in your notebook and then divide these items into two groups: those that you would include in your results and conclusions section (if you were writing this section) and those that you will include in your procedure.

Working together, prepare a draft of your report and then, after receiving feedback on this draft, prepare a final report. When you submit your final report, please include a two well-constructed tables: one that reports results for the standardization of your NaOH solution and one that reports results for your analysis for the %w/v of acetic acid in vinegar. Deadlines are listed on the course's website. See the sample procedure section for how to identify what is important to include in your procedure. You will find additional guidelines for maintaining a lab notebook in the last section of the lab manual.

How to Craft a Good Procedure Section

Writing a procedure is surprisingly difficult as you will find yourself tempted to include far more detail than is needed or appropriate. Science writing is concise, so generally we try to communicate each fact about our work just once, often placing them in our introduction section or in our results and conclusions sections. What is left over after we write the introduction and the results and conclusions, finds its ways into the procedure. This ensures that the procedure, even though important to the reader, is of secondary importance. As an exercise, consider all the things you did during the Newton's law experiment:

- You made a hot-water bath using 500 mL of water, a 600-mL beaker, and a hotplate.
- You immersed two Vernier digital temperature probes in the hot-water bath and connected them to a Vernier MiniQuest data interface.
- You set up your data collection to follow temperature as a function of time, setting the length of the experiment to 10 minutes and the sampling rate to 20 samples per minute.
- You removed the two probes from the water, wiped them to remove residual water, suspended them in air in a way that would minimize interference from the hot plate, and recorded their cooling curves.
- You repeated this process for a total of five trials for each of the two probes.
- You used LoggerPro to fit your data to Newton's law using a natural exponential function.
- You calculated values for T_0, T_s, and K based on the results of your curve-fitting, which were given in terms of the fitting parameters A, B, and C.
- You measured the temperature of the room one or more times during the experiment.

Here is a good procedure section for this experiment:

> *Vernier temperature probes were used as is without further calibration. Probes were immersed in a hot-water bath whose temperature was maintained using a hotplate. The probes were allowed to equilibrate for several minutes, removed from the hot-water bath, wiped dry with a Kimwipe, and suspended in air at a distance of at least 0.5 m from the hotplate. Temperature-time curves were recorded at a rate of 20 points per minute using a Vernier MiniQuest data interface and analyzed using Vernier LoggerPro software. Room temperature was recorded at the beginning and at the end of data collection using an alcohol thermometer.*

Note the details from our list above that are missing from this procedure.

- The volume of water and the beaker used as a hot-water bath are not cited here as these are not particularly important choices. You could use more or less water, or a larger or smaller volume beaker without affecting the results; the choice simply is not important. This is an example of a detail that will not appear anywhere in the final report.
- The number of trials and the collection time are not mentioned both because this information will be clear from the tables and figures that appear in the results and conclusion section and because the exact values are not crucial; a longer or shorter experiment, for example, will not affect your ability to analyze the data and evaluate Newton's Law.
- How you analyzed the data is at the center of the results and conclusions section, so you do not need to include details about it in the procedure.

Now Its Your Turn

Make a list that outlines everything you did in completing thus lab. Go through your list and place each item into one of three categories:

- items that will appear in the introduction (if you wrote it)
- items that will appear in the results and conclusions (if you wrote it)
- items that are too trivial to mention anywhere

What remains from your list are the details to highlight in your procedure section.

Titrimetry

This brief tutorial describes the basic procedure for conducting a titration in the Chem 260 lab. Although the tutorial uses acid-base chemistry as an example, the discussion also applies to other types of reactions.

What is an Acid-Base Titration?

An acid–base titration is an technique in which a solution that contains an acid (or a base) is added dropwise to a solution that contains a base (or an acid). The solution added dropwise is called the titrant and the solution to which the titrant is added is called the sample. The sample normally is placed in a flask whose size is sufficient to contain both the sample and the added titrant, and that has sufficient room to swirl the solution without having it slosh out of the flask. The titrant is placed in a buret allowing for its controlled addition to the sample.

How is a Titration Used to Determine the Concentration of an Acid or Base?

Suppose, for example, that we have a monoprotic acid, HA, of unknown concentration and a monoprotic base, B, whose concentration is known. When we mix them together, the acid and base react according to the following stoichiometry

$$\mathrm{HA}(aq) + \mathrm{B}(aq) \longrightarrow \mathrm{HB^+}(aq) + \mathrm{A^-}(aq)$$

Let's assume, as well, that the reaction essentially proceeds to completion. If we titrate a solution of HA with B until they mix in an exact stoichiometric ratio, then we know that

$$\text{moles B} = \text{moles HA} \tag{1}$$

because the reaction's stoichiometry is 1:1. The moles of HA and the moles of B are equal to the product of their respective molarities, M, and volumes, V; thus

$$M_{\mathrm{HA}} \times V_{\mathrm{HA}} = M_{\mathrm{B}} \times V_{\mathrm{B}} \tag{2}$$

As an example, if 36.42 mL of a 0.116 M solution of B completely reacts with 25.00 mL of HA, then the concentration of HA is

$$M_{\mathrm{HA}} = \frac{M_{\mathrm{B}} \times V_{\mathrm{B}}}{V_{\mathrm{HA}}} = \frac{(0.116\text{ M})(36.42\text{ mL})}{25.00\text{ mL}} = 0.169\text{ M HA}$$

If the acid is diprotic, $\mathrm{H_2A}$, and we react it with sufficient B such that both protons are completely consumed, then

$$\mathrm{H_2A}(aq) + 2\mathrm{B}(aq) \longrightarrow 2\mathrm{HB^+}(aq) + \mathrm{A^{2-}}(aq)$$

and

$$2 \times M_{\mathrm{H_2A}} \times V_{\mathrm{H_2A}} = M_{\mathrm{B}} \times V_{\mathrm{B}} \tag{3}$$

Obviously, there are many other possibilities (diprotic bases, triprotic acids, etc.), but the details for such cases follow easily from this description.

Sometimes the acid or base is obtained as a solid. Suppose, for example, you need to determine the concentration of a monobasic strong base, B, by titrating it against a known mass of a solid monoprotic weak acid, HA. In this case we obtain the concentration of the base using the following equation

$$\mathrm{B} = M_{\mathrm{B}} \times V_{\mathrm{B}} = \frac{\text{g HA}}{MM_{\mathrm{HA}}} = \mathrm{HA} \tag{4}$$

where MM_{HA} is the molar mass of HA.

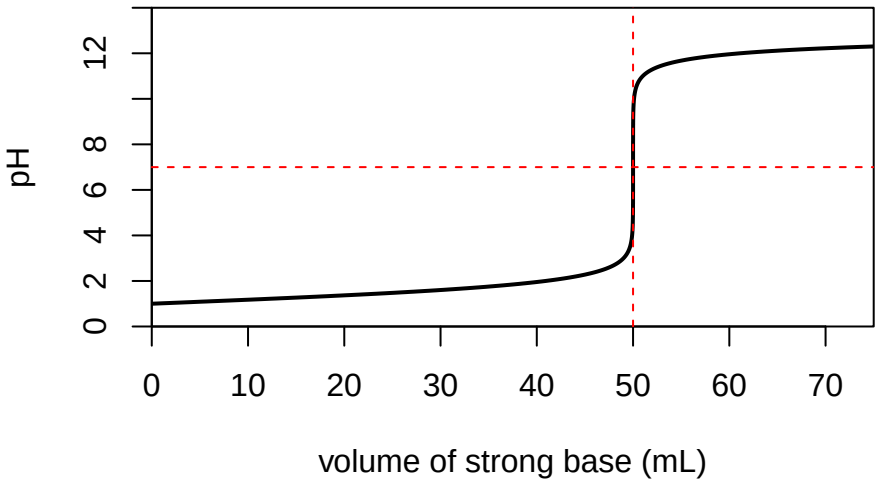


Figure 1: Titration curve for a strong acid using a strong base as a titrant.

How Do We Know When an Acid and Base Have Been Mixed Stoichiometrically?

When the titrant and the sample are mixed in an exact stoichiometric ratio, then the titration has reached its *equivalence point*. Finding this equivalence point is the key to any titration and there are two general approaches for accomplishing this: use a visual indicator that changes color at the equivalence point, or measure the sample's pH as the titrant is added. Using an indicator to find the equivalence point is straightforward and needs no detailed discussion (you just add the titrant dropwise until the indicator changes color, recording the total amount of titrant needed to reach the equivalence point).[1]

Suppose the sample is a strong acid and the titrant is a strong base. Before we add the titrant the sample's pH depends on the strong acid's concentration. Adding titrant causes a slow increase in pH as the strong base neutralizes the strong acid. The rate at which the pH changes becomes greater as we approach the equivalence point, reaching its maximum rate of change at the equivalence point, which in this case occurs when the pH is 7.00. After the equivalence point, the rate of change in pH becomes smaller, resulting in a slow, gradual rise in pH. The resulting titration curve looks something like Figure 1 where the pH at the equivalence point is indicated by the horizontal dashed line and the volume at the equivalence point is indicated by the vertical dashed line.

Titration curves for other samples are similar in shape. If the sample is a strong base and the titrant is a strong acid, for example, then the titration curve begins at a more basic pH and ends at a more acidic pH; the general shape, as shown in Figure 2, is the same. Note that the equivalence point in this case also occurs at a pH of 7.00.

Although the equivalence points in Figure and Figure 2 are both 7.00, this is not always the case. If we titrate a weak acid, HA, with a strong base, for example, the pH at the equivalence point is basic because the reaction

$$\mathrm{HA}(aq) + \mathrm{OH}^-(aq) \longrightarrow \mathrm{H_2O}(aq) + \mathrm{A}^-(aq)$$

produces a solution of the weak acid's conjugate weak base, A^-, at the equivalence point; the pH, therefore, is greater than 7.00. Titrating a weak base with a strong acid, of course, gives an equivalence point that is less than 7.00.

[1] To be exact, a visual indicator signals an endpoint, not an equivalence point. If we choose an appropriate indicator, then the difference between the endpoint and the equivalence point is of no consequence. Selecting an appropriate indicator is a topic that we will not explore in this course.

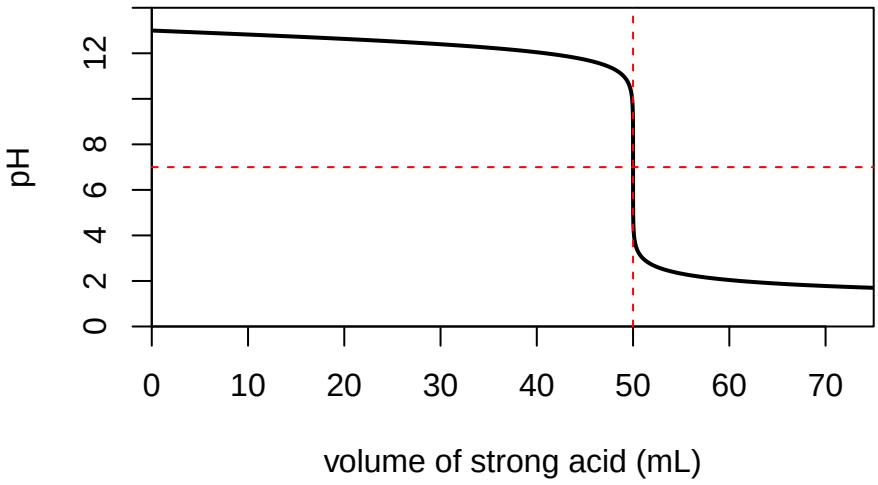


Figure 2: Titration curve for a strong base using a strong acid as a titrant

Automated Titrations

The most common equipment for a titration is a manual buret in which the analyst (that's you!) opens and closes the stopcock, recording the pH after each addition of titrant. This is time-consuming and tedious. A more convenient method for recording a titration curve is to use an automated titrator that records both the volume of titrant added and the pH as a function of time. In this case the titrant is allowed to stream into the sample, usually at a slow rate, and the pH is monitored continuously. In the Chem 260 lab this is accomplished using the Vernier Drop Counter. Further details on its use is available on the course's website.

Titrations Based on Other Types of Reactions

Although this tutorial uses acid-base reactions to explain titrimetry, any chemical reaction can serve as the basis of a titration provided that the reaction is favorable, that it occurs rapidly, and that there is a suitable means to identify the equivalence point. The shape of such titration curves are similar to an acid-base titration curve.

Potentiometry

To record an acid-base titration curve it is necessary to monitor the sample's pH while we add the titrant. The most common way to measure pH is to use an electrochemical sensor that develops a measurable potential whose value is a function of pH. Such sensors are known as potentiometric electrodes and the technique is known as potentiometry.

The Basis of Potentiometry

In potentiometry we measure the electrochemical potential, E, of a redox system under conditions in which we do not allow the reaction to proceed; that is, essentially no current flows during the measurement. Electrochemical potential is related to free energy ($\Delta G = -nFE$) and provides a measure of a reaction's thermodynamic favorability. Consider, for example, the reduction of Ag^+ by Zn

$$Zn(s) + 2Ag^+(aq) \longrightarrow 2Ag(s) + Zn^{2+}(aq) \tag{1}$$

in which Zn undergoes a two election oxidation and each of the two Ag^+ ions undergoes a one-electron reduction. As in any redox reaction, a conservation of electrons requires that the number of electrons gained by the two Ag^+ ions equal the number of electrons lost by the Zn.

If we isolate the oxidation and the reduction half-reactions in separate cells, as is the case in Figure 1, then we can force the electrons produced by the oxidation of Zn to travel through a potentiometer where we measure the potential. This potential is given by the Nernst equation

$$E = E^o + \frac{RT}{nF}\ln Q_r \tag{2}$$

where E^o is the redox reaction's standard state potential, R is the gas constant, T is the temperature in Kelvin, n is the number of electrons in the redox reaction, F is Faraday's constant, and Q_r is the reaction quotient, which, in this case, is

$$Q_r = \frac{[Zn^{2+}]}{[Ag^+]^2} \tag{3}$$

If E is positive, then ΔG is negative and the reaction is favorable as written.

Finding Concentration from Potential

When we use a potentiometric electrode we usually are interested in determining the concentration of one ion in solution. For example, suppose that in the cell shown above the concentration of Ag^+ is 0.100 M and the

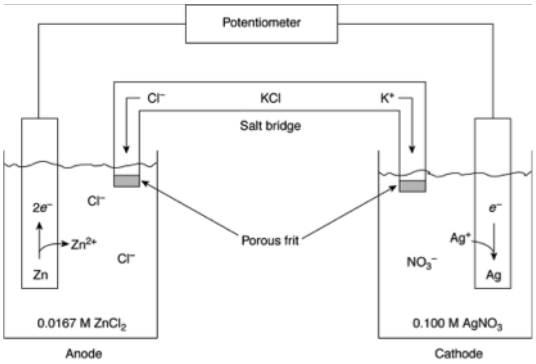


Figure 1: Example of a potentiometric electrochemical cell.

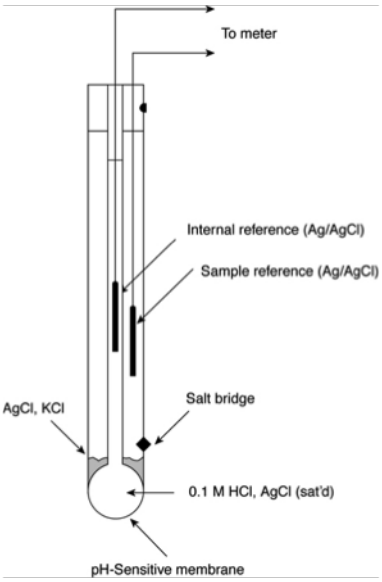


Figure 2: Schematic diagram of a typical pH electrode.

concentration of Zn^{2+} is unknown to us. In this case the Nernst equation is

$$E = E^o + \frac{RT}{2F} \ln \frac{[Zn^{2+}]}{[0.100]^2} \tag{4}$$

In principle the terms in the Nernst equation are well-defined and known so that we can calculate the concentration of Zn^{2+} from a measured potential, E; however, because of a variety of non-ideal behaviors, which we will not consider here, we generally treat this equation as taking the following form

$$E = \beta_0 + \beta_1 \ln \frac{[Zn^{2+}]}{[0.100]^2} \tag{5}$$

where β_0 and β_1 are constants whose values we determine by measuring the potential for two (or more) solutions that contain known concentrations of Zn^{2+}.

The pH Probe

A pH probe (see Figure 2) consists of a thin pH-sensitive membrane. This membrane is a specially manufactured glass that strongly binds H_3O^+. Because the concentration of H_3O^+ on the member's inner surface (typically a solution of 0.1 M HCl) is different from that on the membrane's outer surface (the sample solution), the two sides of the membrane differ in charge and a potential develops. It is this potential that is related to the concentration of H_3O^+ in the sample. Before using the electrode it is calibrated using two buffers of known pH. One buffer generally has a pH value near 7 and the other is more acidic or more basic depending upon whether the samples are acidic or basic.

Characterizing an Oscillating Reaction

Introduction

In the first experiment you prepared three solutions, combined them with a fourth solution, and observed the resulting reaction. This reaction, which is known as the cerium-catalyzed Belousov-Zhabotinsky reaction, is one of a class of reactions that oscillate between different chemical states.

Chemical reactions usually proceed smoothly from reactants-to-products with the concentrations of reactants decreasing and the concentrations of products increasing. In an oscillating reaction, however, some of the species participate in two distinct sets of reactions: in one set the concentrations of these species decrease and in the other set their concentrations increase. Which set of reactions is most important at any time depends on the relative concentrations of these species.

The cerium-catalyzed Belousov-Zhabotinsky reaction is complex, consisting of as many as 80 individual steps that involve 26 different species! At its simplest level, the net reaction is the bromination of malonic acid; thus, the reaction's overall stoichiometry is

$$2H^+(aq) + 2BrO_3^-(aq) + 3CH_2(COOH)_2(aq) \longrightarrow 2BrCH(COOH)_2(aq) + 4H_2O(l) + 3CO_2(g)$$

Although the concentrations of malonic acid and bromate decrease throughout the reaction, the concentration of some intermediate species—those present only during the reaction—and the oxidation state of the cerium catalyst undergo oscillations that continue until the reaction's limiting reagent is depleted.

Skills Emphasized In This Lab

By completing this lab you will become more comfortable with:

- using a spectrometer
- preparing a set of calibration standards and verifying Beer's law
- writing an introduction that provides a background to your work and outlines the goals of your work

Preparing for Lab

Before coming to lab, read the essays "Visible Absorbance Spectra and Beer's Law" and complete the appropriate sections of your electronic notebook.

Procedure

The goal of this week's lab is to characterize the oscillations in the cerium-catalyzed Belousov-Zhabotinsky reaction using a computer-interfaced spectrometer. Because the reaction changes color, we can follow its progress by monitoring its absorbance as a function of time. Properties of interest include the magnitude and period of the oscillations and how each changes as the reaction progresses, and whether there is a difference in the oscillations at different wavelengths.

To save time, all solutions are prepared for you. For this lab, the standard run is 25 mL each of solutions A, B, and C, and 1.5 mL of ferroin (Solution D). Use graduated cylinders to measure the appropriate volumes of solutions A, B, and C, and a disposable plastic pipet for solution D. Be sure to follow the correct order for mixing the reagents: first combine solutions A and B and, when the solution is clear, add simultaneously solutions C and D.

Before monitoring the reaction, you will first verify that Beer's law applies to ferroin, which is red due to the presence of Fe(*o*-phenanthroline)$_3^{2+}$. Begin by adding 1.5 mL of ferroin to 75 mL of deionized water,

using graduated cylinders to measure volume. This *working solution* has a concentration of ferroin similar to that at the beginning of the reaction. Set up the spectrometer and record the spectrum of your working solution using deionized water as a reference. Adjust the signal acquisition parameters to obtain a reasonably smooth spectrum over the available range of wavelengths. When you are satisfied with your spectrum, select a wavelength where the solution's absorbance is strong, but not noisy, and record the absorbance. If the absorbance exceeds 1.0, dilute your working solution and reacquire the spectrum.

Next, prepare a series of standard solutions of ferroin by pipetting 5, 10, 15, and 20 mL of your working solution into separate 25-mL volumetric flasks and dilute each to volume using deionized water. Measure the absorbance of these additional solutions at your previously selected wavelength. Verify Beer's law by constructing a plot of absorbance vs. the concentration of ferroin; you should have a total of five points on this graph, one for each standard. Because you do not know the concentration of the original stock solution of ferroin, treat your *working solution* as having a concentration of 1—unit concentration—and calculate the concentrations of your other ferroin solutions relative to this.

After verifying Beer's law for ferroin, initiate the oscillating reaction, transfer a portion of the reaction mixture to a cuvette and follow the reaction's changing colors using the spectrometer. For your first trial, simply observe the changes in the solution's spectrum as the reaction progresses and identify two to four wavelengths where there is an oscillation in absorbance. Try to select wavelengths that correspond to each of the oscillating reaction's most obvious colors. After selecting your wavelengths, discard the reaction mixture and initiate a new oscillating reaction. This time use the software's kinetics mode to monitor the reaction at the wavelengths identified above. Set the sampling rate to one point per second and monitor the reaction for 15 minutes or until the oscillations stop, whichever is shorter.

Cautions

There are no cautions for this lab other than the normal respect for chemicals.

Waste Disposal

Pour any remaining solutions together and flush them down the drain with plenty of water.

Lab Report

For this report, focus on the ***introduction only***. Research has a purpose and its context forms the basis for your introduction. This context includes what others have done and what question(s) you are addressing. Your introduction should review the history of B-Z reaction—see Winfree, A. T., "The Prehistory of the Belousov-Zhabotisky Oscillator," *J. Chem. Educ.* **1984**, *61*, 661-663—and use the following prompt as your central question:

> It is 1967, and although the scientific community now accepts the B-Z reaction is real and that oscillations are possible, its mechanism remains controversial. You believe the oscillating colors are due to Ce(IV), and the oxidized and reduced forms of ferroin, Fe(*o*-phenanthroline)$_3^{3+}$ and Fe(*o*-phenanthroline)$_3^{2+}$, and decide to study this spectrophotometrically.

Limit your introduction to 2-3 pages of double-spaced text and include a minimum of three references.

Working together, prepare a draft of your report and then, after receiving feedback on this draft, prepare a final report. When you submit your final report, please append two well-constructed figures, one that shows the calibration curve for Fe(*o*-phenanthroline)$_3^{2+}$ and one that shows the reaction's oscillations as a function of time. Deadlines are listed on the course's website. See the sample introduction section for an example of how to prepare your introduction. You will find additional guidelines for maintaining a lab notebook in the last section of the lab manual.

Crafting an Introduction

The goal of an introduction is to provide a context for the work being reported, including why the results are of interest to others. For our purposes, the context you develop in your introduction provides you a way to focus your results and conclusions section. As shown in the example below, the context need not be real; feel free to use your imagination. Aim to end your introduction with a strong transitional sentence, such as "Here we report the first investigation of . . . " or, as in the example below, "Here we present the results of initial studies that aim to identify possible limitations to the applicability of Newton's equations in the laboratory. In particular, we report preliminary results on the cooling of metallic temperature probes in an active laboratory environment."

Introduction

An object whose temperature is greater than ambient temperature loses heat to its environment, decreasing in temperature until it reaches thermal equilibrium with its environment. Understanding the rate of this cooling, and thus the how the object's temperature changes with time, is critical in process chemistry where precise control of the temperature in large scale reaction tanks is essential to ensure that reactions proceed to completion and to prevent the formation of unwanted side products.

Recently, Isaac Newton presented a theoretical model that describes how an object cools over time under conditions of forced convection[1]. In this model, the rate at which an object cools is expressed as

$$\frac{dT(t)}{dt} = -\kappa\,[T(t) - T_s]$$

where $T(t)$ is the object's temperature at time t, T_s is the temperature of the surroundings, and κ is a constant whose value depends upon the object's properties. Rearranging and integrating this equation shows that cooling follows an inverse exponential function

$$T(t) = T_s + (T_0 - T_s)\,e^{-\kappa t}$$

where T_0 is the object's original temperature.

Joule[2], Faraday[3], and Nernst[4] have published detailed theoretical treatments of Newton's equations and Java[5] explored the applicability of Newton's equations to the cooling of coffee in a closed container. No studies, however, have examined Newton's equations under typical laboratory conditions in which forced convection may not be the only mechanism responsible for the dissipation of heat. Here we present the results of initial studies that aim to identify possible limitations to the applicability of Newton's equations in the laboratory. In particular, we report preliminary results on the cooling of metallic temperature probes in an active laboratory environment.

Comments

Note that this introduction presents a clear context—determining whether Newton's law applies in a typical laboratory environment—but the context itself is my invention. An introduction always has references; for an invented context such as this, the references would be inventions as well, such as Java, J. "Initial Studies on the Cooling of Coffee," *Intl. J. Caffinated Beverages* **2020**, *15*, 45-60.

Visible Absorbance Spectra and Beer's Law

Why is cranberry juice red? The simple answer is that one or more components of cranberry juice absorb visible light such that the light passing through or reflecting off the juice appears red; that is, the sample absorbs light whose color is the complement of red We can take advantage of this phenomenon to study molecules, atoms, and ions by measuring their ability to absorb electromagnetic radiation. Sometimes this information is qualitative (What compound is this?) and other times it is quantitative (How much of this compound is present?); in the context of Chem 260, our interest is in the quantitative application of visible spectroscopy.

Spectrometers

A sample's ability to absorb light is measured using a spectrometer. The simplest visible spectrometer has five parts: (a) a place to put the sample; (b) a source of visible light; (c) a detector that measures the amount of radiation that passes through the sample; (d) a means of dispersing the light, typically a prism or a diffraction grating, so that we can analyze at each wavelength the light's interaction with the sample; and (e) a signal processor, such as a meter or a computer, to manipulate and display the resulting measurements. A simple schematic diagram of the spectrometer used in Chem 260 is shown in Figure 1. This instrument uses a diffraction grating to disperse the light over a series of individual detectors, each of which monitors absorbance simultaneously over a narrow band of wavelengths.

Transmittance vs. Absorbance

At any wavelength, λ, the fraction of light not absorbed by a sample is defined as its transmittance, T_λ

$$T_\lambda = \left(\frac{P_T}{P_0}\right)_\lambda \tag{1}$$

where P_T is the intensity of light transmitted by the sample and P_0 is the intensity of light from the source. Frequently the transmittance is expressed as a percentage, $\%T_\lambda$, where

$$\%T_\lambda = T_\lambda \times 100 \tag{2}$$

A little thought should convince you that values for the transmittance must fall within a range from 0 to 1 and that values for the percent transmittance must fall within a range from 0% to 100%.

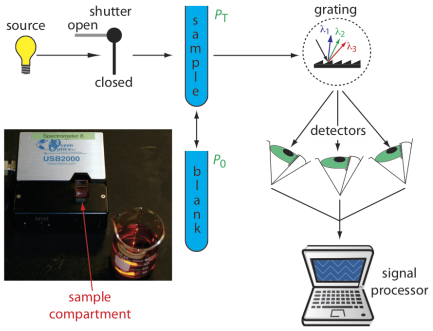


Figure 1: Schematic diagram of a simple diode array spectrometer.

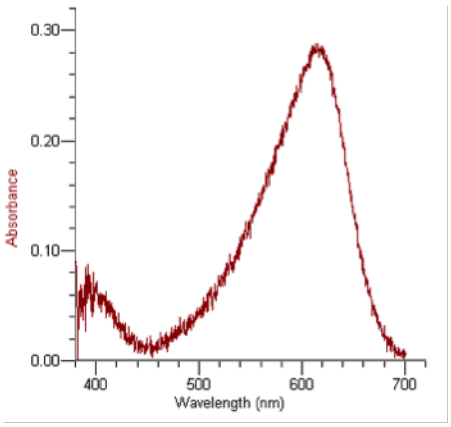


Figure 2: Typical visible absorbance spectrum.

Because the relationship between transmittance and concentration is logarithmic (for reasons we will not consider here), it is more common to report absorbance, A_λ, instead of the transmittance, where

$$A_\lambda = -\log(T_\lambda) = 2 - \log(\%T_\lambda) \tag{3}$$

Visible Absorbance Spectra

A spectrometer monitors absorbance (or transmittance) over a range of wavelengths. A plot of absorbance as a function of wavelength is called an absorbance spectrum, a typical example of which is shown in Figure 2. Note that in this example the sample absorbs strongly at a wavelength of 618 nm.

Beer's Law

One of the most important applications of visible spectrometry is determining the concentration of the species absorbing light. As you might expect, a solution with a higher concentration of the absorbing species transmits less light and has a smaller percent transmittance and a greater absorbance. Although the relationship between $\%T_\lambda$ and concentration is logarithmic, the relationship between concentration and absorbance is linear. The exact relationship between absorbance and concentration is known as Beer's law

$$A_\lambda = \epsilon_\lambda b C \tag{4}$$

where A_λ is the sample's absorbance at the wavelength λ, ϵ_λ is the molar absorptivity at that wavelength, a constant whose value depends on the absorbing species and the selected wavelength, b is the distance the light travels through the sample, and C is the molar concentration of the absorbing species.

Values for ϵ_λ rarely are known with sufficient accuracy, so they are determined by measuring the absorbance of a solution of known concentration. Usually we just combine the value for b with the value of ϵ_λ and drop the stipulation that concentration is expressed with units molarity. Under these conditions, Beer's law reduces to

$$A_\lambda = k_\lambda C \tag{5}$$

where k_λ is a calibration constant and C is any concentration unit.

To determine the calibration constant we prepare several solutions that contain known concentrations of the absorbing species, measure the absorbance of each, plot A_λ vs. C, and use linear regression to find the best straight-line through the data. The equation of this line is used to calculate the concentration of the

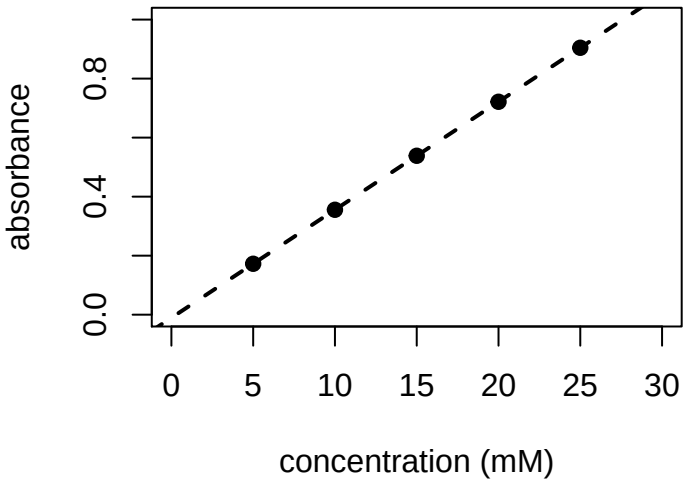


Figure 3: Example of a Beer's law calibration curve.

absorbing species given its absorbance. Figure 3 shows an example of a Beer's law calibration curve where a regression analysis gives a calibration curve of

$$A_\lambda = -0.00840 + 0.0366C \tag{6}$$

The equation for the calibration curve allows us to calculate the concentration of analyte in a sample. For example, if a sample has an absorbance of 0.372, then the analyte's concentration is

$$A_\lambda = 0.372 = -0.00840 + 0.0366C$$

$$C = 10.4 \text{ mM}$$

Thermodynamics of Hydrogen Peroxide's Decomposition

At the beginning of the term we discussed and observed the exothermic decomposition of hydrogen peroxide in the presence of Fe^{3+}. Although present as a reactant, the reaction's stoichiometry does not include Fe^{3+}

$$2H_2O_2(aq) \rightarrow 2H_2O(l) + O_2(g)$$

which means it serves as a catalyst. The purpose of this two-week, open-ended project is to design and carry out suitable experiments that will help you answer the following three questions:

- Can you demonstrate experimentally that Fe^{3+} serves as a catalyst for the decomposition of hydrogen peroxide?

- Does the absolute amount of heat produced during the decomposition of hydrogen peroxide depend on the quantity of H_2O_2 and/or the quantity of Fe^{3+} used?
- What is the value of ΔH for the decomposition of hydrogen peroxide?

Preparing for Lab

Planning for this lab is critical to your success. Be sure to complete the relevant sections of your notebook before each week's lab session. As you develop strategies to explore and answer the questions posed above, keep the following in mind:

- The easiest way to demonstrate a relationship between two variables is to ensure that all other variables remain fixed.
- A calorimeter is not a perfect insulator; thus, you need to determine if the amount of heat your calorimeter absorbs is significant and, if so, determine how to correct for this when you report ΔH. Hint: Search for information about calorimeter constants, but be forewarned that many on-line sources do not follow the standard convention for reporting a change in temperature.
- Be sure to investigate the properties of a catalyst and to consider how you can verify that Fe^{3+} behaves as a catalyst for this reaction.
- To determine a value for ΔH you must make some assumptions. Identify these assumptions and, where possible, consider how you can minimize them. When an assumption is necessary, consider how it might affect your results. Not all assumptions are reasonable and unreasonable assumptions can lead to poor results.
- Compare your experimental result for ΔH with its theoretical values and identify possible source(s) of error that are consistent with their difference.

Procedure

Stock solutions of H_2O_2 and $Fe(NO_3)_3$ are available in lab (the exact concentrations are provided on the bottles). You may dilute these solutions as needed. Calorimeters also are provided.

Cautions

The decomposition of hydrogen peroxide is reasonably exothermic and, depending on your experimental conditions, can produce solutions with temperatures near or above 60°C. Additionally, the rapid formation of O_2 can produce enough pressure to shoot the hot contents out through the small holes in the calorimeter's lid. It is a good idea to first test any reaction without placing a lid on your calorimeter, observing the approximate change in temperature and noting how vigorous the reaction is. Be careful and wear your safety glasses at all times.

Waste Disposal

You may dispose of all solutions by rinsing them down the drain with copious amounts of water.

Lab Report

For the first three project-based labs, each group member will take on one of the following three roles, each of which defines responsibilities and the form of the final report. Each group member will serve once in each role; the assigning of roles is left to you. *Note: For a group with two members, you will need to share the responsibilities assigned to the chemist and the technician during all three experiments and share the responsibilities of lab manager for one experiment.*

Role	Final Product	Responsibilities
Manager	formal report	organizes all aspects of the group's work both in and out of lab; makes all final decisions on experimental design; determines when sufficient work is complete
Chemist	short report	prepares solutions; weighs out samples; carries out the experiment
Technician	oral report	sets up, calibrates, and optimizes the group's equipment; maintains the group's electronic laboratory notebook

All group members must contribute to planning the experiment and to the analysis of data, and are responsible for understanding how to convert the experiment's data into results. Here are some details on the different types of reports; deadlines are listed on the course's website:

- For the **formal** report you will present the results of your experimental work in the form of a journal article. For more details on the format of formal reports, review the document "Some Guidelines for Preparing a Formal Report," "Sample Report," and "Rubric for Evaluating Formal Reports" available at the course website. Although I will not formally review a draft of your report, I do encourage you to bring a draft of your report to my office with specific questions you wish to discuss.
- For the **short** report you will receive a set of data similar to that collected in lab along with some specific questions to answer using this data.
- For the **oral** report we will meet to discuss the experiment, considering topics, such as how to intrepret the data you collected and the effect of possible errors on your results. To prepare for this meeting, review your group's experimental plan, your group's data, and your group's analysis of that data. We will schedule 30 minutes for our conversation.

Thermodynamics and Solubility of Calcium Hydroxide

We usually view thermodynamics and equilibrium chemistry as providing different information about a chemical reaction. For example, with thermodynamics we ask *Is this reaction likely to occur?*, calculate ΔG, and make a prediction; with equilibrium chemistry, however, we ask *What is the composition of this reaction mixture at equilibrium?*, and use the reaction's equilibrium constant, K_{eq} and initial composition to make a prediction. What is hidden in these questions is the mathematical relationship $\Delta G^o = -RT\ln K_{eq}$ that ties together these two views of chemical reactivity; thus, we can use a reaction's equilibrium constant and the reaction mixture's reactant quotient, Q_r, to predict the favorable direction for a reaction, and we can use the free energy at any time during the reaction's progress to determine the reaction's composition at that moment. The purpose of this two-week, open-ended project is to study the solubility reaction $\mathrm{Ca(OH)_2}(s) \rightleftharpoons \mathrm{Ca^{2+}}(aq) + 2\mathrm{OH^-}(aq)$ for which the equilibrium constant is $K_{sp} = [\mathrm{Ca^{2+}}][\mathrm{OH^-}]^2$. More specifically, you should design and carry out suitable experiments that can provide answers to the following questions:

- How does the solubility of $\mathrm{Ca(OH)_2}$ change with temperature?
- What are the values of ΔG^o, ΔH^o, and ΔS^o for the solubility of $\mathrm{Ca(OH)_2}$?

Preparing for Lab

Planning for this lab is critical to your success. Be sure to complete the relevant sections of your notebook before each week's lab session. As you develop strategies for determining values ΔG^o, ΔH^o, and ΔS^o, keep the following in mind:

- Because the solubility reaction releases a base, $\mathrm{OH^-}$, you can use an acid-base titration to determine the concentration of $\mathrm{OH^-}$ in a saturated solution of $\mathrm{Ca(OH)_2}$.
- To determine the concentration of $\mathrm{OH^-}$ in a saturated solution of $\mathrm{Ca(OH)_2}$ you must first remove any undissolved $\mathrm{Ca(OH)_2}$ by filtering. Consider why this is necessary and what complications might arise if you don't successfully remove all the $\mathrm{Ca(OH)_2}$.
- To determine the concentration of $\mathrm{OH^-}$ in a saturated solution of $\mathrm{Ca(OH)_2}$ you will need a solution of HCl with a nominal concentration of 0.01 M. You will need to determine how to prepare this solution using the available stock solution of 1 M HCl. You will determine this solution's concentration by titrating it against the standard weak base tris(hydroxymethyl)aminomethane, $(\mathrm{HOCH_2})_3\mathrm{CNH_2}$, which also is called TRIS or THAM; you may take the reaction to be

$$\mathrm{TRIS}(aq) + \mathrm{HCl}(aq) \rightarrow \mathrm{TRISH^+}(aq) + \mathrm{Cl^-}(aq)$$

- Review your preparation and standardization of a standard solution of NaOH in the third preliminary experiment and adapt that procedure to this lab. Plan titrations that require ≈ 15 mL of titrant to reach the equivalence point. If your first titration requires significantly less than or significantly more than 15 mL, then adjust your procedure. To determine how much HCl to make, consider how many total titrations you will make in two weeks and assume each will require 30 mL of HCl; double this total and you will be fine.
- You need to decide how many mL of filtered $\mathrm{Ca(OH)_2}$ to use in your titrations. As a "rule of thumb," a room-temperature titration should use approximately 15 mL of nominally 0.01 M HCl to reach the equivalence point. You might find it helpful to know that the solubility of $\mathrm{Ca(OH)_2}$ at 25°C is reported as one gram per liter of water. If your first titration requires significantly less than or significantly more than 15 mL, then adjust your procedure.
- You need to decide how to calculate ΔG^o using data from your titrations. Be sure you consider this before you begin gathering data so that you know the accuracy and precision needed for different measurements.
- It is possible to determine values for ΔH^o and ΔS^o by studying the solubility of $\mathrm{Ca(OH)_2}$ as a function of temperature; give some thought to how this is done.
- Experimentally determined values should be compared to their expected theoretical values.

Procedure

Your first task is to prepare and standardize your solution of HCl. Once this is completed you may begin to analyze samples. During the first week a room-temperature saturated solution of $Ca(OH)_2$ is available to you, which you can filter using gravity filtration. During the second week you will have access to a saturated solution of $Ca(OH)_2$ at elevated temperatures, which you can filter by syringe filtration.

Cautions

There are no cautions for this lab other than the normal respect for chemicals and hot solutions.

Waste Disposal

You may dispose of all solutions by rinsing them down the drain with copious amounts of water.

Lab Report

Remember to switch roles for this lab.

Role	Final Product	Responsibilities
Manager	formal report	organizes all aspects of the group's work both in and out of lab; makes all final decisions on experimental design; determines when sufficient work is complete
Chemist	short report	prepares solutions; weighs out samples; carries out the experiment
Technician	oral report	sets up, calibrates, and optimizes the group's equipment; maintains the group's electronic laboratory notebook

All group members must contribute to planning the experiment and to the analysis of data, and are responsible for understanding how to convert the experiment's data into results. Here are some details on the different types of reports; deadlines are listed on the course's website:

- For the **formal** report you will present the results of your experimental work in the form of a journal article. For more details on the format of formal reports, review the document "Some Guidelines for Preparing a Formal Report," "Sample Report," and "Rubric for Evaluating Formal Reports" available at the course website. Although I will not formally review a draft of your report, I do encourage you to bring a draft of your report to my office with specific questions you wish to discuss.
- For the **short** report you will receive a set of data similar to that collected in lab along with some specific questions to answer using this data.
- For the **oral** report we will meet to discuss the experiment, considering topics, such as how to intrepret the data you collected and the effect of possible errors on your results. To prepare for this meeting, review your group's experimental plan, your group's data, and your group's analysis of that data. We will schedule 30 minutes for our conversation.

Determining the Acid Dissociation Constant for an Organic Dye

One of the simplest ways to estimate a solution's pH is to add a small amount of an organic dye and observe the solution's color. For example, a solution of bromothymol blue—one of several sulfonophthalein dyes—is yellow when the pH is less than 6 and blue when the pH is greater than 8. Figure 1 shows the structure of bromothymol blue in its weak acid and weak base forms. When the pH of a solution of bromothymol blues is adjusted from 6 to 8, its color transitions from yellow to blue by passing through various shades of yellow-green, green, and green-blue, each of which correlates to within a few tenths of a pH unit.

This relationship between the change in color and pH, is easy to appreciate from the basics of mixing color. Less obvious is the mathematical relationship between absorbance and pH

$$\log\left(\frac{A - A_{\text{In}}}{A_{\text{HIn}} - A}\right) = \text{p}K_a - \text{pH}$$

where A is the absorbance at a particular pH and A_{In} and A_{HIn} are the absorbance values for the weak base and weak acid forms, respectively. Note that this equation suggests it is possible to determine a dye's pK_a by monitoring its absorbance as a function of pH. The purpose of this two-week, open-ended project is to design and carry out suitable experiments that can provide the pK_a value for two organic dyes: bromocresol green and neutral red.

Preparing for Lab

Planning for this lab is critical to your success. Read the following paper as it provides useful information for determining the pK_a values for organic dyes; a copy of the paper is in your group's shared folder.

> Patterson, G. S. "A Simplified Method for Finding the pK_a of an Acid–Base Indicator by Spectrophotometry," *J. Chem. Educ.* **1999**, *76*, 395-398.

Be sure to complete the relevant sections of your notebook before each week's lab session. As you develop strategies for determining the pK_a values for these dyes, keep the following in mind:

- The paper provides complete experimental details for determining the pK_a of bromophenol blue and sufficient information to help you design a procedure for determining the pK_a of bromocresol green. You will need to adapt the procedure to determine the pK_a for neutral red.
- The work described in the paper uses a different instrument for acquiring absorbance spectra and for measuring absorbance; you will need to modify the published procedure to take advantage of the spectrometer available to you.
- One shortcoming of the paper is that the author did not verify Beer's law, which means they provide no evidence that absorbance is a linear function of concentration. Review how you did this in an earlier lab and adapt that approach to this lab.

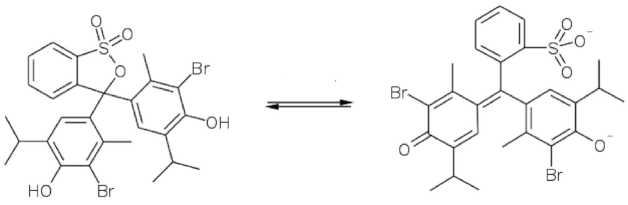


Figure 1: Structure of bromothymol blue in its weak acid form (left) and its weak base form (right); modified from https://commons.wikimedia.org/wiki/File:Bromothymol_blue_protolysis.svg.

- You will need to complete some research on each dye. Among the information you may find useful are structures, the expected color of the weak acid and weak base forms, and approximate pK_a values.
- Any experimentally determined values should be compared to their expected theoretical values.

Procedure

During the first week you should plan to work on determining the pK_a value for bromocresol green. During second week you will determine the pK_a for neutral red.

Cautions

There are no cautions for this lab other than the normal respect for chemicals and hot solutions.

Waste Disposal

You may dispose of all solutions by rinsing them down the drain with copious amounts of water.

Lab Report

Remember to switch roles for this lab.

Role	Final Product	Responsibilities
Manager	formal report	organizes all aspects of the group's work both in and out of lab; makes all final decisions on experimental design; determines when sufficient work is complete
Chemist	short report	prepares solutions; weighs out samples; carries out the experiment
Technician	oral report	sets up, calibrates, and optimizes the group's equipment; maintains the group's electronic laboratory notebook

All group members must contribute to planning the experiment and to the analysis of data, and are responsible for understanding how to convert the experiment's data into results. Here are some details on the different types of reports; deadlines are listed on the course's website:

- For the **formal** report you will present the results of your experimental work in the form of a journal article. For more details on the format of formal reports, review the document "Some Guidelines for Preparing a Formal Report," "Sample Report," and "Rubric for Evaluating Formal Reports" available at the course website. Although I will not formally review a draft of your report, I do encourage you to bring a draft of your report to my office with specific questions you wish to discuss.
- For the **short** report you will receive a set of data similar to that collected in lab along with some specific questions to answer using this data.
- For the **oral** report we will meet to discuss the experiment, considering topics, such as how to intrepret the data you collected and the effect of possible errors on your results. To prepare for this meeting, review your group's experimental plan, your group's data, and your group's analysis of that data. We will schedule 30 minutes for our conversation.

Kinetics of the Bleaching of Dyes

Thermodynamics allows us to predict whether a reaction is favorable, but it does not tell us if the reaction will occur in a reasonable amount of time. To know something about the conditions that favor a timely reaction we must study the reaction's kinetics. The relationship between a reaction's rate and the concentrations of species that affect the rate is given by a rate law, which takes the general form

$$R = k[\mathrm{A}]^a[\mathrm{B}]^b \cdots [\mathrm{E}]^e$$

where k is the rate constant and A, B, and E are species whose concentration might affect the reaction's rate. The superscripts associated with the concentration terms are called reaction orders and may be positive or negative integers; they may even be zero or fractional.

In this two-week, open-ended experiment you will investigate the kinetics of the reaction responsible for the effectiveness of bleach. Commercial bleaches are solutions that contain one or more oxidizing agents. One of the most common oxidizing agents in bleach is the hypochlorite ion, $\mathrm{OCl^-}$, which usually is added in the form of its sodium salt, NaOCl, typically at a concentration of 5–6 % w/v.

Bleaches work by oxidizing stains, converting the compound responsible for the stain to a colorless product. Most stains are just colored organic dyes; thus, we will use blue food coloring as a model stain compound. The overall reaction, then, simplifies to

$$\mathrm{dye}(aq) + \mathrm{OCl^-} \rightarrow \text{colorless product}$$

and the rate law for the reaction can, as a first estimate, is

$$R = k[\mathrm{dye}]^\alpha[\mathrm{OCl^-}]^\beta$$

The goal of this study is to characterize the factors that affect the reaction's rate; more specifically, you will

- determine the rate law for the reaction, including the reaction orders and the rate constant
- study the effect of pH on the rate law

Preparing for Lab

Planning for this lab is critical to your success. Be sure to complete the relevant sections of your notebook before each week's lab session. As you develop strategies for determining the reaction's rate law keep the following in mind:

- As the reaction proceeds the solution will fade from its initial blue color to a colorless solution. You can follow the reaction's kinetics, therefore, by monitoring the solution's absorbance as a function of time. To use absorbance in place of concentration, however, you must establish that Beer's law applies to your solution of dye. You may wish to review your work from earlier labs to see how you accomplished this. In addition, to obtain smooth kinetic data you need to minimize the noise in your absorbance spectra.
- To study a reaction's kinetics you must ensure that the concentration of only one reactant is changing; that is, you will study the reaction under pseudo-order conditions in which the initial concentration of dye is significantly smaller than the initial concentration of $\mathrm{OCl^-}$. Be sure to review how pseudo-order kinetics work and verify that the rate law under these conditions is $R = k_{obs}[\mathrm{dye}]^\alpha$ with an observed rate constant, k_{obs}, that is equal to $k[\mathrm{OCl^-}]$.
- You need to verify that the initial concentrations of dye and $\mathrm{OCl^-}$ are suitable for a pseudo-order kinetic study. The dye's molar absorptivity is 1.38×10^5 $\mathrm{M^{-1}\ cm^{-1}}$ at a wavelength of 630 nm. To determine the concentration of $\mathrm{OCl^-}$ in bleach you may adapt the following general redox titrimetric approach: Pipet 1 mL of bleach into a suitable flask and add approximately 50 mL of deionized water and 2 g of KI. Swirl the solution to dissolve the KI and then add approximately 3 mL of 3 M $\mathrm{H_2SO_4}$. The resulting solution will be brown due to the formation of $\mathrm{I_2}$, as shown by the following reaction

$$\mathrm{OCl^-}(aq) + 2\mathrm{I^-}(aq) + 2\mathrm{H_3O^+} \rightarrow \mathrm{I_2}(aq) + \mathrm{Cl^-}(aq) + 3\mathrm{H_2O}(l)$$

Determine the amount of I_2 produced by titrating with a solution of $Na_2S_2O_3$ of known concentration using the procedure described in lab. The titration reaction is

$$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow S_4O_6^{2-}(aq) + 2I^-(aq)$$

- You need to determine how kinetic data consisting of absorbance as a function of time can be used to determine the reaction order for the dye and the reaction's observed rate constant.
- You need to think about how kinetic runs using different initial concentrations of OCl^- can be used to extract information about its reaction order and the reaction's true rate constant, k. Consider, as well, how you might prepare solutions of OCl^- with different concentrations.
- Because OCl^- is a weak base it is reasonable to expect that pH might affect the reaction's rate. Give some thought to reasonable pH levels to investigate and how you might set up a suitable kinetic experiment. Note: the reported pK_a values for blue food coloring are 5.63 and 6.85.

Procedure

To prepare your dye solution, try adding 18 drops of dye to a liter of deionized water and then adjust the concentration so that the solution's maximum absorbance is between 0.8 and 1.0. All kinetic runs should consist of 25 mL of a dye solution and 1 mL of a bleach solution. Be sure to begin data acquisition the moment you add the bleach and then transfer a portion of the solution to the spectrometer as soon as possible. Collect data until the absorbance reaches zero or a steady-state value.

Cautions

There are no serious cautions for this lab other than the normal respect for chemicals.

Waste Disposal

If you have any unused bleach solution, place it in the beaker located in the hood nearest to where solutions are stored. All other solutions can be disposed of down the drain, flushing with copious amounts of water.

Lab Report

As a group, you will prepare a report for this lab in the form of a scientific poster. Instructions on preparing a poster are provided elsewhere in this lab manual and a template is available from the course's archive page. Your group's poster is due by the last day of final exams.

Guidelines for Writing Formal Reports

The results of research generally are shared with other scientists in the form of an article published in a scientific journal. Although scientists often present oral or poster papers at professional meetings, such work usually is preliminary and tentative. When research reaches the point where you can tell a convincing story, then publication in a suitable journal is the appropriate way to share your results.

If you look at several scientific journals, particularly journals from different disciplines, you will see that there are many ways to prepare a research article. Each journal, however, remains true to its format, requiring authors to follow specific guidelines. For this course we will use the structure listed below when preparing formal reports.

- a title
- an abstract
- an introduction
- a procedure
- a discussion of results and conclusions
- notes
- references
- supporting materials

The remainder of this document provides some suggestions for each section of a lab report.[1] *Note: Your electronic notebook and a formal report serve different purposes. In general, copying and pasting the contents of your notebook into your formal report is a poor idea.*

Writing the Title

A good title is brief (15 words or less) and clearly identifies the problem you investigated. Don't use the experiment's title as the title for your report.

Writing an Informative Abstract

A good abstract provides a brief (3-6 sentence) descriptive summary of your report's major finding.[2] At a minimum it should clearly state the goal(s) of your work and provide a summary of your key result(s), including the expected result(s) when known. Because the abstract summarizes your work, you should prepare it after you complete the remainder of your report. For a formal report the abstract is the first thing I read as it provides me with a sense of where to focus my attention. For example, if your results are clearly in error then I will give extra attention to your data, calculations, and experimental design.

Writing a Thorough Introduction

The introduction to your formal report needs to accomplish three things: it should clearly explain, as appropriate, how your experiment fits within the broader context of the course's three main topics (i.e. thermodynamics, equilibria, and kinetics); it should explain why your experimental approach is suitable and briefly outline any relevant theory; and it should clearly state the goal(s) of your experiment. Many of these topics are, of course, outlined in the experiment's handout and you may use this as a starting point as

[1] There are many good texts that cover technical writing. The suggestions compiled here were culled from Porush, D. *A Short Guide to Writing About Science*, Harper and Collins: New York, 1995.

[2] The abstract for the the paper "Can Apparent Superluminal Neutrino Speeds be Explained as a Quantum Weak Measurement?" by M. V. Berry, et. al., and published in the *Journal of Physics A: Mathematical and Theoretical*, is particularly short: it simply states "Probably Not." You will want a longer abstract than this!

you draft your introduction. A good introduction, however, does not simply repeat information from the experiment's handout; instead, it extends and contextualizes this information. Use the introduction to define terms, to explain basic theory, and to convince me that you understand the experiment's goals. Searching for additional information in the library or on the internet is appropriate and desirable.

Writing a Useful Procedure

This often is the hardest part of a report to write well. Surprisingly, your initial draft will inevitably provide too much detail instead of too little detail! Here are some useful guidelines to consider:

- A procedure is not a list of what you did in lab; it is, instead, a well-written narrative. Avoid using phrases such as "First we. . . " or "Next we. . . ".
- A procedure does not describe the specific things you did in lab; it provides, instead, a general guideline to what you did. For example, you do not need to state that "We evaluated the cooling rate of porridge using initial temperatures of 60°C, 55°C, 50°C, and 45°C," because you will include these temperatures as data in your results and conclusions section. You should state, however, that "The porridge samples used to evaluate cooling rates were heated using a gas stove while stirring with a wooden spoon." Or, you do not need to mention that "We prepared a dilute solution of 2.00×10^{-3} M hemlock by adding 5.00 mL of a 0.100 M stock solution to a 250-mL volumetric flask and diluting to volume with spring water." What is important is the concentration of your final solution of hemlock, not its volume. It is more appropriate to write that "A solution of 2.00×10^{-3} M hemlock was prepared from the available 0.100 M stock solution."
- A procedure does include information on the reagents used in the experiment. Be sure to list all reagents provided for your use or prepared by you (e.g. "Solutions of 6.00 M hemlock and reagent-grade powdered toadstools were used as supplied." or "A solution of 0.10 M hemlock was prepared using the available 6.00 M stock solution and a 5.0 % w/v solution of toadstools was prepared using reagent-grade powdered toadstools.").
- A procedure does include information on the major equipment used during the experiment. Be sure to identify the make and model of the instrumentation and software used to collect and analyze data. Where specific operating conditions are used, be sure to state them (e.g. "Cooling curves for porridge samples were measured using a CelFar thermometer interfaced to a Merlin Microcomputer equipped with a NEWTon data interface. Temperature measurements were made every five seconds until the porridge's temperature reached 30°C.").
- A procedure might mention the type of glassware and minor equipment used in the experiment, but only if the choice is crucial or unusual. For example, if it essential to collect samples of witch's brew using gold-lined sampling bottles, then say so. In general, there is no need to specify the type of glassware as this is made evident by the proper use of significant figures. If your procedure states that you prepared a nominally 0.1 M solution of hemlock, then it is clear that you measured at least one volume using less accurate and less precise glassware, such as a beaker or bottle. On the other hand, if your procedure states that you prepared a 0.100 M solution of hemlock, then it is clear that you used volumetric glassware.
- If your procedure closely follows a previously published procedure, then you may simply make a reference to it and note any significant modifications. Thus, you might write that "The number of plums in a pie was determined using the method of Jack Horner (*13*) with the modification that individual plums were removed using a fork instead of the thumb."

Presenting Your Results and Conclusions

This is the heart of your report so it deserves your greatest attention. The most important requirement of this section is that it is a well-written narrative that clearly guides the reader through a presentation of your data and your analysis of that data. Use tables and figures to organize your data and to enhance its presentation. Be sure that you refer in your narrative to each table and figure and guide the reader to the specific point(s) of information contained within each. Remember that your goal is to make a convincing

argument about the analysis of your data and to arrive at specific conclusions that are well-supported by your data. Don't leave this to your reader! Finally, be sure to evaluate the reasonableness of your results. If you know the expected results for the experiment, then compare them to your conclusions and discuss possible sources of error. *When discussing sources of error do not cite "human error" as you may assume that you correctly used your equipment*; instead, consider other sources of errors that might reasonably account for the magnitude and the direction of your error.

Making Using of Notes

There are two important aspects of your report—derivations and calculations—that you may wish to include, but whose presence detracts from a smoothly written narrative. To include this material without distracting the reader, place it in a note appended to the back of the report. For example, you might write the following: "Assuming that breaking a standard hand mirror causes seven years of bad luck, we know that Sleeping Beauty's step-mother can expect an additional 23.6 yrs of bad luck (Note 1)." In the note you can then work through the relevant calculation.

Referencing Other Works

Every discipline has its rules for preparing references. In most chemistry journals references are cited in the text using italicized numbers listed within parentheses—for example, citing the first reference as (*1*)—or using superscripts. References are placed either at the bottom of the page where the reference is made or collected, in numerical order, at the end of the report. Use the following standard formats:

- Journal articles: Green, A.; Scarlet, R., "Preliminary Measurements on the Strength of Huts Made Using Bricks: Can They Withstand the Huffs and Puffs of Wolves?" *Folk Tales Sci.*, **2003**, *45*, 313-315.
- Books: Blue, V. *A Brief History of Magic Potions*, Merlin Press: Salem, MA, 1999.
- Chapter or article in book with editor: Rumpelstiltskin, T., "Seeking a New Means for Spinning Straw into Gold" in *Studies in Alchemy*, Grimm, J.; Grimm, W., eds., Merlin Press: Salem, MA, 2001.
- Internet sites: http://www.goldilocks.com/mattresshardness/ (accessed August 2014).

Supporting Materials

Inevitably you will gather more data than you need to include in your report. Use this section to list additional tables and/or figures that support your work and that you wish me to examine (as needed). Place these materials in a folder labeled "Appendix" and place it in the experiment's folder included within your group's Dropbox folder. Include in your lab report a brief outline of what supporting information is in the appendix.

Stylistic Considerations for Scientific Writing

Grammar, spelling and formatting matter, as does well-written prose. Expectations for the quality of your writing for this course are no different than that for courses in the humanities or social sciences. A few specific suggestions are provided here:

- Be concise. Use simple words. Write short sentences. Thermodynamics, equilibria, and kinetics are complicated enough; there is no need to make them more complicated by writing confusing, wordy sentences.
- Remember the basic rules for writing a good essay. Introduce a paragraph's main idea with a topic sentence and develop the idea throughout the remainder of the paragraph. Link your paragraphs together with smooth transitions.
- Words have specific meanings. This is particularly important in scientific or technical writing. Although rate and energy, for example, have many definitions, in the context of this course their meanings are very specific. Be sure you use terms correctly.

- Numbers have significant figures and units. You know this, so use them properly. In scientific writing the use of significant figures carries meaning. When you say that "A 0.127 g portion of dried toadstools was reacted with. . . ," providing three significant figures tells the reader that you measured the mass using a balance with three decimal places. If the same statement simply said "0.1 g of dried toadstools" the reader will assume you simply used an approximate means to measure out the sample, such as the amount that fits on the tip of a spatula. While on the subject of numbers, a decimal point is placed between numbers; the decimal equivalent of $\frac{1}{2}$ mL is 0.5 mL, not .5 mL. Finally, if you must begin a sentence with a number, write out the number; thus, write write "Five liters of hemlock were obtained." instead of "5-L of hemlock were obtained."
- Use captions, legends, and footnotes to explain the contents of figures and tables. Even though you will discuss a figure or table in your narrative, a caption helps focus the reader's attention. Figures that contain more than a single set of data must include a legend that identifies the data sets, which you can incorporate into the caption or embed in the figure. Make use of footnotes in tables to add helpful annotations.
- Sequentially number equations included in your narrative. The appropriate format is to center the equation on its own line (rather than including the equation in the middle of a line of text) and place a numerical label at the right margin; thus

$$PV = nRT \tag{1}$$

Rather than retyping the equation later, you can simply refer to it by its number. For example, "Rewriting equation (1) as. . . ".

I'm Suffering From Writer's Block! How Do I Get Started?

Preparing a formal lab report is a daunting task. Here are some suggestions on how to start. Begin by thoroughly organizing and analyzing your data. As you do this, you likely will create a variety of tables and figures; cull through these and select those that most efficiently summarize your data and results and that are most crucial to your conclusions. Next, write the narrative for the results and conclusions section, building it around your tables and figures. Write the procedure section after you complete the results and conclusions section. As you write your procedure, focus on ensuring that another student in Chem 260 could explain how the data presented in the results section were obtained. Finally, write your abstract at the very end.

What about the introduction? You can write this at any point as it is independent of your procedure, data, and results (although it does define the problem on which you were working). You might even (gasp!) want to begin working on your introduction while you are preparing for your work in lab.

A Thermodynamic Investigation of the Reaction Between Hypochlorite and Iodide

Abstract[1]

The method of continuous variations and a calorimetric determination of enthalpy are used to verify the stoichiometry for the reaction between hypochlorite, OCl^-, and iodide, I^-. Stoichiometric mixing occurred with a 3:1 mole ratio between OCl^- and I^-, suggesting that the reaction is

$$3OCl^-(aq) + I^-(aq) \rightarrow IO_3^-(aq) + 3Cl^-(aq)$$

The experimentally determined value of –341 kJ/mol_{rxn} for ΔH compares favorably with the expected value of –346.4 kJ/mol_{rxn}, providing further evidence that the proposed stoichiometry is correct.

Introduction[2]

Hypochlorite ion, OCl^-, is a potent oxidizing agent used in many household bleaches. Although hypochlorite's reactivity with a wide variety of organic molecules is well known (*1*), particularly for organic dyes, its reactivity with inorganic ions has been studied less frequently. This is somewhat surprising given the significant release of OCl^- into the environment from household use (*2*).

One of the challenges in studying any reaction is determining the products. This is a particular problem for an oxidation-reduction reaction when many oxidation states are available to the oxidizing agent or to the reducing agent. For example, the chlorine in OCl^-, which has an oxidation state of +1, can be reduced to an oxidation state of 0 in Cl_2 or to an oxidation state of –1 in Cl^-, or it can be oxidized to an oxidation state of +3 in ClO_2^-, to an oxidation state of +5 in ClO_3^-, or to an oxidation state of +7 in ClO_4^-. Although it is possible to identify the products by using a standard qualitative analysis scheme, this often requires that we first isolate an individual species before we use an identifying chemical test.

The method of continuous variations (MCV), first described by Job (*3*) for the stoichiometric analysis of metal–ligand complexes, provides a simple method for determining the stoichiometry of an oxidation-reduction reaction. The basis of MCV involves combining solutions of two reactants, A and B, such that their combined moles, n_{tot}, remains constant in all experiments; thus

$$n_A + n_B = n_{tot} \tag{1}$$

where n_A and n_B are, respectively, the moles of A and B. The relative amount of each reactant is expressed as its mole fraction, X_A or X_B

$$X_A = \frac{n_A}{n_{tot}} \tag{2}$$

$$X_B = 1 - X_A = \frac{n_B}{n_{tot}} \tag{3}$$

[1] There are four things to note about this abstract: it states the experiment's purpose; it summarizes the experiment's two main conclusions; it compares one of the experiment's results to a theoretical value; and, at just three sentences in length, it is concise. As you prepare your abstract, try to accomplish these four things.

[2] An introduction places your work within a context that defines the experiment's goals. For this fictitious report, assume that students are told that OCl^- is the active ingredient in bleach, that it is an oxidizing agent, and that its release into the environment poses a concern. Assume, as well, that the goal of the experiment is to determine the stoichiometry of the reaction between OCl^- and I^-. To help students in their planning, they are provided with a copy of Job's paper on the method of continuous variations for metal–ligand complexes and asked to adapt that procedure to this experiment. In addition, because they had just studied calorimetry, the students are asked to use calorimetry in this experiment. Note that this introduction accomplishes three things: it provides additional context for the experiment by citing references to the chemistry of OCl^- and by discussing the difficulty of determining the stoichiometry of oxidation-reduction reactions; it explains Job's method; and it clearly states the experiment's goals. Note that the introduction's first paragraph begins with a "hook" that tells the reader why s/he should be interested in this research, and note, as well, that the introduction's final paragraph provides a transition to the rest of the report, telling the reader what is to come.

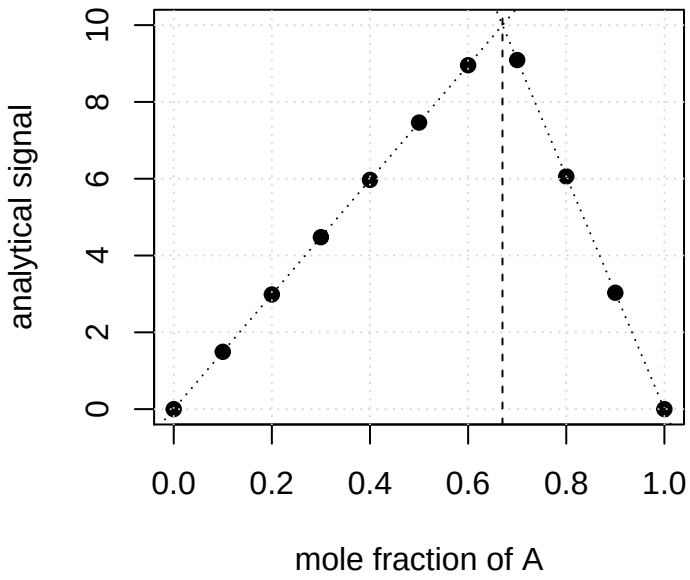


Figure 1: Hypothetical MCV plot for the reaction of A and B showing a stoichiometric mixing when the mole ratio of A to B is 2:1. The vertical dashed line shows the stoichiometric mixture.

In a reaction between A and B, the extent of the reaction is determined by the limiting reagent. If an analytical signal is proportional to the limiting reagent, then a plot of that signal as a function of X_A consists of two straight lines that intersect at a mole fraction for A that corresponds to the stoichiometric mixing of A and B. The mole ratio of A and B at this point is given by

$$\frac{n_A}{n_B} = \frac{X_A}{X_B} = \frac{X_A}{1 - X_A} \tag{4}$$

Figure 1 shows an MCV plot for a hypothetical reaction between the reactants A and B. The intersection at $X_A = 0.67$ indicates that two moles of A react stoichiometrically with one mole of B, from which we conclude that the reaction is

$$2\text{A} + \text{B} \rightarrow \text{Products}$$

As noted by Job (*3*), there are three important limitations to the MCV: the reaction must involve only two reactants, there must be only one possible reaction, and the free energy for the reaction must be highly favorable.

To evaluate the utility of the MCV as a method for studying oxidation-reduction chemistry, we present results for an analysis of the reaction between hypochlorite and iodide. A determination of the reaction's enthalpy provides further confirmation of the MCV results.

Procedure[3]

All reactions were carried out in a locally-designed cup calorimeter. Two small 8 oz. Styrofoam cups were nestled together and trimmed such that the inner cup did not protrude above the outer cup. The bottom of a 20 oz. Styrofoam cup was removed and used to fashion a top. Small holes were punched in the top to accommodate a digital thermometer, which was connected to a LabQuest data interface (Vernier), and to accommodate a small glass funnel for adding reagents. Data were analyzed using LoggerPro (Vernier).

[3]The most striking observation about this procedure is the relative absence of numerical information. Note that the procedure makes no attempt to describe in detail each individual experiment. For example, examine carefully the first two sentences in the procedure's second paragraph and contrast the general information provided there with the more specific details included in the results and conclusions section.

The calorimeter's suitability for calorimetry experiments was tested by measuring the change in temperature when reacting 1.00 M solutions of HCl (Fisher Scientific) and NaOH (Fisher Scientific). The calorimeter's constant was determined using the method of Joule (*4*), which is based on the thermal transfer of heat from hot water to cold water. In all calorimetry experiments, the temperature was monitored for several minutes both before and after adding the second solution to ensure that ΔT could be determined accurately by extrapolation.

Solutions of 0.213 M OCl^- and 0.213 M I^- were prepared from a commercial bleach (Kroger Brand) and reagent-grade KI (Fisher Scientific), respectively. The solution of KI was 0.1 M in NaOH (Fisher Scientific). Calorimetry experiments for reactions involving OCl^- and I^- were limited to a combined volume of 80 mL. The reagent with the larger volume was placed in the calorimeter and the reagent with the smaller volume was then added. For those experiments used to establish the reaction's stoichiometry only the solution volumes are reported; the mass of each solution was measured in experiments used to determine ΔH.

Results and Conclusions[4]

When an object with a known mass, m, and a known specific heat, S, experiences a change in temperature, ΔT, the heat, q, absorbed or released is given by

$$q = mS\Delta T \tag{5}$$

where q is negative when the object loses heat and is positive when the object gains heat. Equation 5 is the fundamental equation of calorimetry.

Evaluating the Calorimeter. An ideal calorimeter is a perfect insulator that allows a reaction to take place without exchanging energy with either the calorimeter itself or with the external environment. To evaluate the suitability of our calorimeter, we measured the change in temperature upon mixing 40.0 mL of 1.00 M HCl with 40.0 mL of 1.00 M NaOH. As seen in Figure 2, the calorimeter maintains a relatively constant temperature once the reaction is complete, suggesting that there is a minimal loss of heat to the laboratory environment.

To determine whether the calorimeter absorbs heat, we note that the theoretical ΔH^o for the reaction

$$H_3O^+(aq) + OH^-(aq) \rightleftharpoons 2H_2O(l)$$

is –55.836 kJ/mol_{rxn}, which gives an expected change in temperature for the data in Figure 2 of approximately 6.7°C (Note 1). As the actual change in temperature is 6.3°C, the calorimeter itself clearly absorbs some of the energy released by the reaction, a contribution that we can correct for by measuring the calorimeter's heat capacity.

Determining the Calorimeter's Heat Capacity. To accurately measure an exothermic reaction's ΔH by calorimetry we must know how much heat the calorimeter absorbs, q_{cal}, which is equivalent to

$$q_{cal} = C_{cal}\Delta T_{cal} \tag{6}$$

where C_{cal}, which is equivalent to mS, is the calorimeter's heat capacity and ΔT is the calorimeter's change in temperature. In Joule's method for determining a calorimeter's constant we combine a known mass of hot water, m_h, with a known mass of room temperature water, m_{rt}, and observe the change in temperature for both (*4*). A conservation of energy requires that the heat lost by the hot water is absorbed by the room temperature water and by the calorimeter; thus

$$-q_h = q_{rt} + q_{cal} \tag{7}$$

[4]There are five things to note here. First, the results and conclusions are organized so that the reader can follow the logic of the data analysis; note, in particular, the use of subheadings to focus the reader's attention. Second, figures are used to present representative data and tables are used to present results across a range of conditions. Third, where appropriate, results are summarized using means and standard deviations. Fourth, the main conclusions for each part of the experiment are stated clearly. Fifth, and finally, an analysis of error in included.

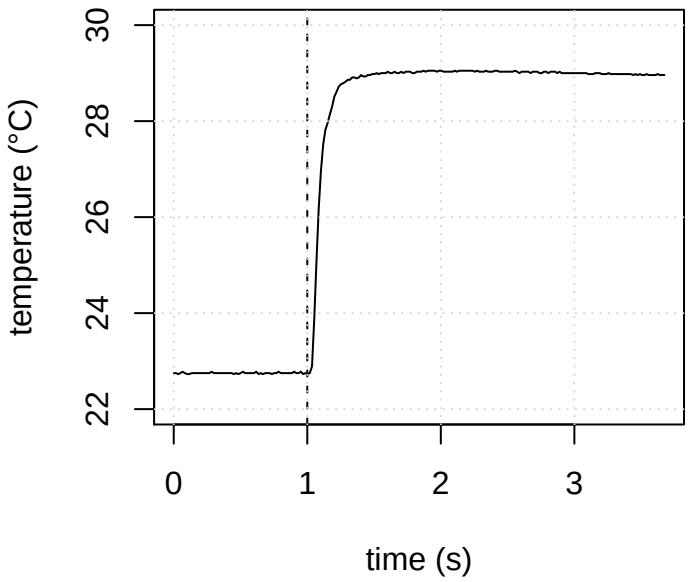


Figure 2: Temperature vs. time curve for the reaction of 40.0 mL of 1.00 M HCl with 40.0 mL of 1.00 M NaOH. The sample of HCl was added to the calorimeter first with the NaOH added at the time indicated by the dashded vertical line. The nearly constant temperature after the reaction is complete indicates that the rate at which heat is lost to the external environment is minimal.

Substituting equation 5 and equation 6 into equation 7 gives

$$-m_h S\Delta T_h = m_{rt} S\Delta T_{rt} + C_{cal}\Delta T_{cal} \tag{8}$$

where m is the mass of water and S is the specific heat of water (4.184 J/g°C). In a typical experiment, a final temperature of 41.3°C was achieved upon mixing 40.133 g of room temperature water at an initial temperature of 24.7°C with 39.168 g of hot water at an initial temperature of 61.5°C, which gives a C_{cal} of 31.5 J/°C (Note 2). The average C_{cal} for three trials was 31.4 J/°C with a standard deviation of 0.56 J/°C.

Establishing Stoichiometry by the Method of Continuous Variations. To establish the reaction's stoichiometry by the method of continuous variations, we carried out a series of calorimetry experiments in which we changed the relative amounts of OCl^- and of I^- while maintaining a constant combined volume of 80.0 mL. Temperature-time curves for these experiments were similar to that shown in Figure 2 and are not included here. Figure 3, which is a variation of the traditional MCV plot, shows that a stoichiometric mixing occurs for the reaction of 60.0 mL of 0.213 M OCl^- with 20.0 mL of 0.213 M I^-. This corresponds to 1.28×10^{-2} mol OCl^- and 4.26×10^{-3} mol I^-, or a 3:1 molar ratio.

Because iodine in I^- is in its most negative oxidation state, it must undergo oxidation. The chlorine in OCl^- must be reduced to either Cl_2 or to Cl^-. If Cl_2 is the product, then a total of three electrons are needed to reduce the chlorine in three OCl^- ions from oxidation states of +1 to 0. This requires the single iodine in I^- to experience a three electron oxidation from its initial oxidation state of –1 to a final oxidation state of +2; however, no such oxidation state exists for iodine. If Cl^- is the reduction product, then a total of six electrons are needed to reduce the chlorine in three OCl^- ions from oxidation states of +1 to –1. Oxidation of I^- to IO_3^- provides the necessary electrons. The proposed balanced reaction for this system is

$$3OCl^-(aq) + I^-(aq) \rightarrow IO_3^-(aq) + 3Cl^-(aq)$$

Determining ΔH for the Reaction. To determine ΔH for the reaction between OCl^- and I^- we completed additional calorimetry experiments using 60.00 mL of 0.213 M OCl^- and 20.00 mL of 0.213 M I^-. Table 1 summarizes the results of these experiments where

$$\Delta H = \frac{q_{rxn}}{n_{LR}} \times \frac{\nu_{LR}}{\text{mol}_{\text{rxn}}} = \frac{-(q_{soln} + q_{cal})}{n_{LR}} \times \frac{\nu_{LR}}{\text{mol}_{\text{rxn}}} = \frac{-(mS\Delta T + C\Delta T)}{n_{LR}} \times \frac{\nu_{LR}}{\text{mol}_{\text{rxn}}} \tag{9}$$

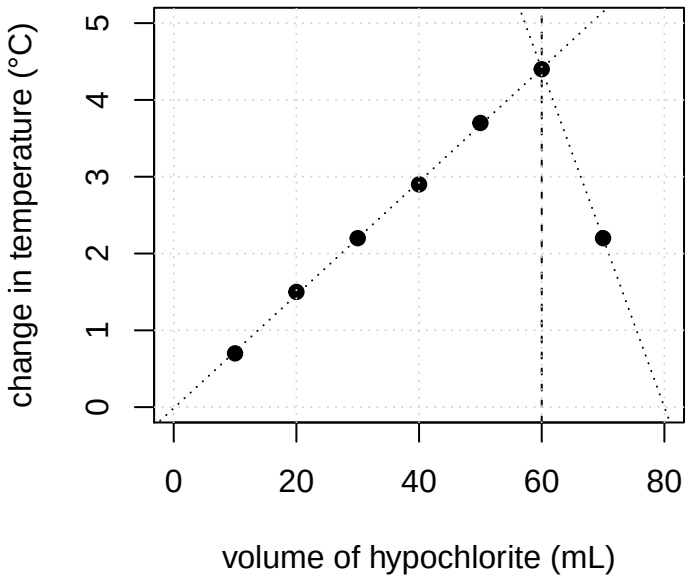


Figure 3: MCV plot showing that stoichiometric mixing occurs when 60.0 mL of 0.213 M hypochlorite react with 20.0 mL of 0.213 M iodide.

and where q_{rxn} is the heat released by the reaction, q_{soln} is the heat absorbed by the reaction mixture, q_{cal}, is the heat absorbed by the calorimeter, m is the total mass of the reaction mixture, S is the reaction mixture's specific heat (which we assume is equivalent to that for water), C, is the calorimeter's constant, n_{LR} is the moles of the limiting reagent, and ν_{LR} is the limiting reagent's stoichiometric coefficient in the balanced reaction (Note 3).

Table 1: Calorimetry Results for the Reaction of OCl^- with I^-

	total mass (g)	ΔT (^oC)	ΔH (kJ/mol$_{\text{rxn}}$)
1	82.409	3.9	-344
2	82.616	3.8	-336
3	82.195	3.9	-344

The mean and the standard deviation for the results in Table 1 are –341 kJ/mol$_{\text{rxn}}$ and 4.62 kJ/mol$_{\text{rxn}}$, respectively. The theoretical value for ΔH is –346.4 kJ/mol$_{\text{rxn}}$, which gives an error of –1.8%. Because ΔT is known to just two significant figures, the expected uncertainty in ΔH is ±10 kJ/mol$_{\text{rxn}}$, or a relative uncertainty of ±2.8%. Our experimental uncertainty of –1.8%, therefore, is reasonable and suggests that there are no significant problems with the experiment.

Notes[5]

Note 1. The value of 6.7°C was determined as follows:

- Because equal moles of HCl and of NaOH are mixed—in this case, 0.0400 mol of HCl or NaOH—we can assign either reagent as the limiting reagent.
- The amount of heat released, q_{rxn}, is

$$q_{rxn} = \frac{-55.836 \text{ kJ}}{\text{mol}_{\text{rxn}}} \times 0.0400 \text{ mol HCl} \times \frac{1000 \text{ J}}{\text{kJ}} = -2233 \text{ J}$$

[5]Use notes to present sample calculations or to include additional explanations. The purpose of this section is to present important material whose presence in the main body of the report might disrupt your reader's ability to engage with your report's narrative.

- The amount of heat absorbed by the calorimeter, q_{cal}, is $-q_{rxn}$, or +2233 J.
- The change in temperature is determined using the equation $q_{cal} = mS\Delta T$. Assuming the combined solution has a density of 1.0 g/mL, gives the solution's mass as 80 g; thus

$$+2233 \text{ J} = (80 \text{ g})(4.184 \text{ J/g°C})\Delta T$$

  or a ΔT of 6.7°C.

Note 2. Substituting into equation 7 the results for this trial

$$-(39.168 \text{ g})(4.184 \text{ J/g°C})(41.3 - 61.5 \text{ °C}) = (40.133 \text{ g})(4.184 \text{ J/g°C})(41.3 - 24.7 \text{ °C}) + C_{cal}(41.3 - 24.7 \text{ °C})$$

and solving for C_{cal} gives a result of 31.5 J/°C.

Note 3. Outlined here is the calculation for the first row in Table 1. Applying a conservation of energy requires that

$$-q_{rxn} = q_{soln} + q_{cal}$$

where q_{rxn} is the heat released by the exothermic reaction, q_{soln} is the heat absorbed by the reaction mixture, and q_{cal} is the heat absorbed by the calorimeter. Substituting in equation 5 and equation 6 gives

$$-q_{rxn} = m_{soln}S_{soln}\Delta T_{soln} + C_{cal}\Delta T_{cal}$$

$$-q_{rxn} = (82.409 \text{ g})(4.184 \text{ J/g°C})(3.9 \text{ °C}) + (31.4 \text{ J/°C})(3.9 \text{ °C}) = -1467 \text{ J}$$

Because this is a stoichiometric mixture, we can calculate ΔH using either OCl^- or I^-; here we use I^- as the limiting reagent

$$\Delta H = \frac{q_{rxn}}{n_{LR}} \times \frac{\nu_{LR}}{\text{mol}_{\text{rxn}}} = \frac{-1467 \text{ J}}{(0.213 \text{ M})(0.02000 \text{ L})} \times \frac{1 \text{ mol } I^-}{\text{mol}_{\text{rxn}}} \times \frac{1 \text{ kJ}}{1000 \text{ J}} = -344 \text{ kJ/mol}_{\text{rxn}}$$

References[6]

1. Ajax, V. *The Chemistry of Bleach*, Comet Press: New York, 1999.
2. "The Effects of Chlorine Bleach on the Environment", http://www.livestrong.com/article/217675-the-effects-of-chlorine-bleach-on-the-environment/ (accessed January 2017)
3. Job, P. "The Method of Continuous Variations for Studying Metal-Ligand Complexation," *Ann. Chim.* **1928**, *9*, 113.
4. Joule, K. J. "Establishing the Calorimeter Constant by the Thermal Transfer of Energy from Hot to Cold Water," *J. Therm.* **1945**, *8*, 134.

Supporting Materials[7]

The following supplemental information is available on Dropbox:

- two additional temperature-time curves used to validate the calorimeter's performance
- the three temperature-time curves used to determine the calorimeter's heat capacity
- the three temperature-time curves used to determine the reaction's ΔH value

[6]The second and the third references are real; the first and the last references are fictitious entries created for this sample report. Note that books are identified by their title, information about the publisher, and the date of publication; journal articles are identified by their title, the journal, the year of publication, the volume, and the page; websites are identified by their title, the url, and the date the url was accessed.

[7]Use the appendix to summarize the data files available to the reader. All data that appears in your report—whether directly in the form of a figure or a table—or indirectly in the discussion of your results must be accessible to your reader.

Rubric for Evaluating Formal Reports

The following list provides a general set of questions that I use to evaluate formal reports. You should be able to confidently answer "**Yes!**" to each of these questions.

Abstract: Does your abstract...

- state the purpose of your experiment?
- summarize your key experimental results?
- where possible, compare your experimental results to theoretical results?

Introduction: Does your introduction...

- provide a context for your experiment?
- clearly explain how your experiment fits within the broader context of one or more of the course's three main topics (i.e. thermodynamics, equilibria, and kinetics)?
- explain why your experimental approach is suitable and briefly outline any relevant theory?
- clearly state your experiment's goal(s)?

Procedure: Does your procedure...

- avoid reading as though it is a list or a timeline?
- provide only essential details?
- omit numerical information that is not critical or that us included with your results and conclusions?

Results and Conclusion: Are your results and conclusions...

- presented in a logically structured manner?
- supported by well-designed tables and figures, as appropriate?
- clearly stated and, where appropriate, compared to theoretical or expected results?
- supported by an analysis of reasonable experimental errors?

Miscellaneous Questions

- Is each each table and each figure discussed in the report?
- Is the report free from spelling and grammatical errors?
- Is there sufficient information in the report to verify the results and conclusions?
- Are measurements and results reported with units and with attention to significant figures?
- Is the work of others properly referenced?

Figures: Does each figure...

- include an informative caption?
- use plotting symbols of sufficient size so that the data points are not obscured by a regression line?
- use appropriate scales for each axis?
- have properly labeled axes (including units)?
- include a legend, when necessary, to ensure that multiple data sets are clearly identified?
- remove connecting lines between data points, unless such lines are essential?

Tables: Does each table...

- include an informative title?
- include all relevant measurements and final results?
- exclude unnecessary information, such as intermediate calculations?
- properly label the rows and/or columns (including units)?
- use appropriate significant figures?

Preparing a Poster

In addition to reports and presentations, posters are a means that scientists use to disseminate information to each other. At a conference or a meeting, scientists often choose to present work that is still in progress through the vehicle of a poster.

Posters typically are hung side by side (sometimes by the hundreds) in large rooms. Over the course of a meeting scientists mosey around these posters on their own time or they choose to study the poster during dedicated "poster session" times when the communicating author is expected to stand by his or her poster to explain their work and to answer any questions. In either case, you can imagine that the poster should contain detailed, clear information that is easily visible and visually pleasing to ensure that it is easy to read and understand.

In place of a traditional, written report for your last experiment, your group will prepare a poster that describes the results of your experiment. You will not actually present these posters at a poster session; instead you will provide me with a pdf version that I can evaluate.

All sections normally present in a written report are included in your poster including an abstract, introduction, procedure, results/discussion, figures, tables, and references. In a written lab report your figures and tables support your narrative; in a poster, however, this role is reversed and your figures and tables become the focal point. In a poster, your goal is to share your work using figures and tables, annotating each with a succinct but meaningful description, often in the form of an informative caption (note: this is the one place where tables often have captions instead of titles). It is also possible to incorporate your procedural information in the captions and titles that accompany your figures and tables.

There is a PowerPoint template for a poster on the course website that you choose to use. Note that the template has places for a title and a list of authors, as well as three columns that you can use to present information about your experiment. There is a sample figure from a poster in the middle column to give you an idea of how you might format your work. You will also find posters hanging in the hallways of Julian and Olin that you can review.

The template will make a poster that is 44 inches by 36 inches. Do not print a full version of your poster. If you want to see your poster on paper, open "Print Preview," and select "Options" and "Scale to Fit to Paper" before printing. This will print a copy of your poster on a single sheet of 8.5 $\times$ 11 paper. Before submitting your final poster, be sure that it is legible at this scale.

Finally, a poster has a limited amount of space, which means you likely will have to makes some tough decisions about what to include in and what to leave out of your poster. Don't worry about making these decisions before you begin working on your poster; if you run out of space before you are able to add your final, crucial figure or table, then this is the time to sit back and determine what is the least critical element in your poster.

Working Safely in the Chem 260 Lab

Although a chemistry or biochemistry laboratory is equipped with chemicals and equipment that can result in injuries, there is no reason that working in a laboratory inherently is less safe than working in other environments, such as a kitchen, where one is exposed to caustic materials (Drano!), sharp objects (knives!), and hot items (gas burners!). Just as you can work safely in a kitchen if you pay careful attention to what you are doing, you can work safely in the laboratory if you pay careful attention to how you dress for lab, how you prepare for lab, how you work while in the lab, and how you clean up at the end of lab. Although all items in this document are important, those in **bold** merit your particular attention.

Dressing for Lab

Spills can and do happen in lab, so careful attention to how you dress for lab is important.

- The chemical reagents used in lab are hard on both the natural and the synthetic fabrics used to make clothing. Some reagents bleach out colors and others break down fibers leaving holes or weakened threads; for this reason, you should wear clothes that you will not mind discarding if damaged by a spill or by exposure to the laboratory environment.
- How you dress from the waist down is particularly important as this is where you are most vulnerable to spills; for this reason, you must:
  - **wear shoes with completely closed toes**; sandals and other shoes that leave your feet exposed are not acceptable; sneakers or boots are preferred
  - wear pants or skirts that end at or below your knees; long pants are preferred; avoid long dresses or other clothing that restricts your movement
  - shirts and blouses must extend below your waist
- If you have long hair, tie it back or place it up so that it does not get in your way as you work in lab.
- The metal in jewelry can react with some chemicals. If you choose to wear jewelry to lab then you should:
  - wear long necklaces inside your shirt, dress, or blouse so that they do not get in your way as you work in lab
  - avoid wearing loose fitting bracelets as they may become tangled with lab equipment

##Preparing for Lab

Safety in the lab begins before you enter the room and begin work. In addition to any pre-lab assignments, pay careful attention to the following items that will better prepare you for a safe experience in lab.

- **Read through the lab handout at least once** to familiarize yourself with the experiment's procedure, and make a list of questions regarding any parts of the procedure you find confusing.
- Make careful note in your lab notebook of any warnings regarding specific hazards and the proper disposal of hazardous waste.

Working in Lab

Despite our best efforts, accidents happen: beakers tip over; test tubes break, solutions splash, and reactions run more vigorously than expected. Paying attention to the following will decrease the likelihood of an accident and, more important, minimize your chance of injury if an accident does happen:

- Your eyes are precious and easily damaged by caustic chemicals; thus, **you must wear approved safety glasses whenever you are in lab** regardless of whether or not you are engaged in an experiment. Be sure you **know the location of the eye wash fountains and that you know how to use them**.

- To avoid the accidental ingestion of toxic chemicals, **food, drinks, chewing gum, and tobacco are not allowed in lab**. If you bring these items to lab, be sure to store them in the study area.
- **Never use mouth suction** to fill a pipet or to siphon a reagent from one bottle to another bottle.
- Because many chemicals are flammable, **open flames are not allowed in lab unless approved by the instructor**. Be sure you **know the location of the fire extinguishers and the fire blanket and that you know how to use them**.
- If you get a chemical on your skin, rinse the exposed area using copious amounts of water. Be sure you **know the location of the lab's overhead shower and know how to use it**. Gloves are available in lab, which you may choose to use if you are particularly sensitive to chemicals.
- Particularly foul-smelling chemicals and volatile reagents are stored in one of the lab's fume hoods; **pay particular attention to directions that require you to complete one or more steps in a fume hood** and do not remove materials from the fume hood unless the procedure indicates that it is safe to do so.
- Be sure you **know the location of all exits from the laboratory** and the location of the closest red emergency phone, which provides a direct link to campus security.
- **If you are injured while working in lab, immediately report the injury to your instructor**.

To minimize the chance of an accident, pay attention to the following:

- Keep you lab bench clean, organized, and uncluttered.
- Use labels to identify the contents of beakers, test tubes, and flasks.
- When you are finished using a piece of equipment, return it to your lab drawer or its storage bin.
- **Do not engage in horseplay, pranks, or other similar acts, including unauthorized experiments and altering the quantities of reagents used in an experiment**.
- **You may not work in lab unless another person is in lab**.
- **If an accident happens, immediately inform the instructor** who will assist you in cleaning up.

Cleaning Up After Lab

When you are finished with your work, be sure to complete the following important tasks that protect you, your classmates, and the environment:

- **Dispose of all chemical wastes as directed**. Chemical wastes generally are collected in waste bottle located in one of the labs hoods; be sure to place wastes in their proper container as mixing waste streams can result in an unwanted chemical reaction. Do not dispose of chemicals in a sink unless the procedure specifically indicates that this is the proper method of disposal.
- **Do not return unused chemicals to their original container**; instead, treat them as chemical waste and dispose of them as described above (or share with others who are need of the reagent).
- **Place glass waste and sharps (needles, etc.) in the proper waste container**.
- **If you are unsure of how to dispose of a reagent properly, ask your instructor**.
- Clean your lab bench using a sponge and water.

Acknowledging the Importance of Lab Safety

After reading these safety guidelines, sign and date the form acknowledging that you read and understand the guidelines, that you had an opportunity to ask clarifying questions, and that you agree to abide by these guidelines.

Tips for Working as a Group

Working with other students as part of a small research team is a rewarding experience. There is an abundance of evidence in the educational literature that the process of discussing an experiment with others leads to a deeper understanding of both the specific experiment and the broader science underlying the experiment. In addition, working as part of a group is a valuable skill that is of increasing importance to employers, to graduate programs, and to the health professions. Indeed, you will spend most of your professional career working closely with others. An effective group, however, does not happen without some effort on your part. The following tips will help you get more out of this experience.

Forming Groups and Assigning Responsibilities. Whenever possible, each group has three members (with groups of two only when absolutely necessary). Assignments to groups are made by the instructor to ensure that groups are balanced in terms of academic backgrounds (e.g. prior coursework, major vs. non-major, prior research experience, etc.).

Working as a group can be a chaotic experience if no one knows who is responsible for completing tasks: Who is responsible for searching the library or internet for the information needed to make decisions?; Who is responsible for gathering together equipment?; and, Who ensures that the electronic laboratory notebook is up-to-date?

Groups work best when each member has a specific set of responsibilities. For the first three project-based labs, each of you will take on one of these three roles, which will, in turn, define your final product for the lab:

Role	Final Product	Responsibilities
Manager	formal lab report	organizes all aspects of the group's work both in and out of lab; makes all final decisions on experimental design; determines when sufficient work is complete
Chemist	short report	prepares solutions; weighs out samples; carries out the experiment
Technician	oral report	sets up, calibrates, and optimizes the group's equipment; maintains the group's electronic laboratory notebook

For the last project-based lab, you may divide up the work as you see fit. Because the preliminary experiments are scripted and do not require advanced planning on your part, specific roles are not assigned, although each of you will take the lead in preparing the first draft for one of the group's written reports.

Speak Up When You are Confused and Listen to Each Other. A critical part of working together is ensuring that each group member understands the experiment's goals and understands how your individual efforts help accomplish those goals. If you don't understand something, no matter how trivial it seems, then speak up and ask questions. If one member of the group asks a question, then the remaining group members should ensure that the question is answered satisfactorily before continuing; never sacrifice one group member's understanding for the sake of expediency.

Be Responsible. By participating in a group you assume responsibility for each other. Remember that your effort affects not just yourself, but it also affects others in your group. When each member of a group lives up to his or her responsibilities, the group's work inevitably is better.

Respect Each Other. Even the best group has disagreements. If a disagreement occurs, take a break to cool down and, as a group, try to talk through the problem. Remember to respect and listen to each other.

Maintaining an Electronic Laboratory Notebook

Because each group member needs routine access to a shared lab notebook, a traditional bound notebook is, at best, cumbersome. Instead, you will maintain an electronic notebook using a shared folder on a Google Drive. As you work on an experiment—whether that work takes place in the lab or out of the lab—be sure that you place the following items in the experiment's folder: a text file that documents your work; data files from the LoggerPro software used to collect data; and, any spreadsheet files or other files you create while processing your data. You are free to use Google Docs to create text files and spreadsheet files; however, you must maintain a current copy of these files in your group's shared folder.

As with any laboratory notebook, an electronic laboratory notebook must provide an accurate record of your work. At a minimum, the text file you create for this purpose must contain the following items:

- **A Statement of Purpose**. This is a brief statement of the theory or the hypothesis your will test (e.g. "The purpose of this experiment is to verify Chicken Little's claim that 'The sky is falling!'.") or the piece of information you will seek (e.g. "The purpose of this experiment is to determine the average number of spots on a Dalmatian."). In addition, you may wish to summarize the expected result or to indicate how an unexpected result will lead to a different conclusion (e.g. "If we find that the egg's density is not 19.3 g/cm^3, then we will know that the goose did not lay a golden egg."). *Complete this section before you come to lab.*
- **A Description of Your Planning for the Experiment**. This is a summary of your group's planning for the experiment. Use this section to document your library or internet research, and to outline the procedure you plan to follow in lab. The purpose of this section is to ensure that your time in lab is more productive. Note that this section does not contain data. Thus, you might note that you will need to obtain approximately 0.25 g of dried toadstools, but you will not record here the actual value obtained in lab (which you will enter in a later section). In addition, this section should contain a summary of how you will process your data. *Complete this section before you come to lab.*
- **A List of the Materials and Reagents Used During the Experiment**. This is a brief summary of the equipment and chemicals used during the experiment, which you may write in either a narrative form or as a list. Under the subheading of materials list the equipment used, including make and model, and its role in the experiment (e.g. "Soap bubbles were produced using B&B's Big Bubble Blower.") and any hardware and software used to collect and process data. Under the subheading of chemicals, list all reagents provided to you (e.g. "A stock solution of 0.10 M Hemlock and freeze-dried Newt's Blood were provided."), but do not list solutions prepared by you as these belong with your experimental data and results. You do not need to list commonly available lab supplies, the contents of your lab drawers or distilled water. *Complete this section during lab.*
- **A Comprehensive Record of Experimental Data**. This is the heart of your electronic laboratory notebook as it contains the data collected during the experiment. You may choose to write this as a narrative annotated with data or as a list (e.g. Trial 1. . . , Trial 2. . .). Be sure to record all relevant data in an appropriate manner: data for a single analysis is entered directly; data for a series of related analyses are best entered in a table; store LoggerPro files and spreadsheet files separately, but list the filenames in your notebook along with a brief description of the file's contents. Clarity of presentation and good organization are important here as the quality of your written and oral reports depends on your ability to remember what you did in lab. *Complete this section as you do the experiment.*
- **An Analysis of Your Data**. In the process of preparing your group's lab report you will need to analyze your data. For example, if you average the results of several trials to obtain a single value, then record this information in your notebook so you have a record of it (e.g. "The average for the five temperature measurements of Baby Bear's porridge is 48.9°C."). For data that is analyzed using a software program, store the resulting data file in the appropriate folder and identify its filename in this section of your notebook. Feel free to use this section to speculate on the meaning of your data. *Complete this section as you analyze your data.*

Note: You cannot include too much detail or too much information in your electronic notebook. No one has ever had to repeat an experiment because they recorded too much information in a laboratory notebook!