GENERAL CHEMISTRY LAB



Karsten Theis
Westfield State University

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This text was compiled on 10/21/2026

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1.1: Lab 1 check-in and measuring matter

Lab 1: Volume and Mass

Chemistry is the study of matter. Matter has mass and takes up space, so measuring mass and volumes is something we do all the time in chemistry. This lab introduces balances (to measure mass) and lab glassware (to handle and measure the volume of liquids). At the end of the lab, we hope you can answer questions such as:

- How do I use a balance?
- What are the items in my drawer called, and what are they used for?
- How can I mix a liquid, and why would I?
- How do we measure small volumes (less than a teaspoon)?
- When would I use a graduated cylinder, and when would I use a buret instead?

Safety and hazardous waste

This lab has no specific safety concerns. The substances used are water and food coloring. We are not generating any hazardous waste today. However, for EVERY experiment we conduct in the chemistry lab, there are general safety concerns to be aware of, including tripping hazards and broken glass. Whenever you or anyone in the chemistry lab is conducting an experiment, you must wear your safety goggles and other required Personal Protection Equipment (PPE) to protect yourself.

Organization

Use your lab notebook to record measurements and observations, and to show your work for calculations. There is a summary sheet you will hand in. This lab has 4 parts **that can be done in any order**. For part 1, you will receive a time slot to avoid waiting time at the analytical balances (we have four, and there are up to 18 students in the lab). Part 3 uses a buret which is not part of your drawer equipment. You will work individually on parts 1 and 2, and in pairs on parts 3 and 4.

Average and standard deviation

For some tasks, you are asked to calculate and average (also called mean) and a standard deviation of repeated measurements. While you can quickly calculate an average by hand, it takes a bit longer to write out the calculation for standard deviation. To quickly calculate standard deviation, you may either use a spreadsheet (Excel or Google Sheets) OR you can also use an online calculator such as the one linked in the QR code.

Part 1: Using an analytical balance

An analytical balance is pretty amazing. It can measure masses down to a milligram, i.e. 0.001 g. To get the full power of the analytical balance, you have to learn how to use it and to take good care of it.

1a) Precision: mass of a penny

The mass (m) of a penny is easy to measure. Set the balance to zero while all doors are closed. Open one of the doors, place

the penny in the middle of the weighing pan, close the doors, and read off the mass once it has stabilized. There might be some fluctuations in the last digit even after waiting quite some time. The mass shown will be in grams (g). Test the balance for consistency by measuring your penny on two other balances. Record all measurements in your notebook. Then calculate average and standard deviation of your three measurements of the penny. In your summary sheet, record this as "m = (average +/- standard deviation) g."

1b) Mass of a powdered solid

To measure a powdered solid, you have to use weighing paper, a weighing dish or some other container. Your sample has a mass ("net") and your container has a mass ("tare"), but the balance records the sum ("gross"). To get the net mass of your sample, either measure the mass of the empty container and write it down, or press the zero button while the empty container is on the balance ("taring the balance"). For the latter, you have to be sure than you can complete the measurement before someone else uses the balance.

When you use a weighing paper, it is good practice to make a crease in it before use. This makes it easier to transfer the sample off the paper later. Measure the mass of a "spatula tip" of baking soda using weighing paper and record it. Then, tare the balance with a fresh weighing paper, transfer the baking soda onto the fresh weighing paper, measure the mass and record it. How much do the two measurements of the powder differ? Why might they be slightly off?

1c) Mass of a liquid

The maximal mass for an analytical balance is often 110 g. Make sure the container you choose for measuring the mass of a liquid is not too heavy for the balance. In this experiment, we will test how consistent the mass of 10 mL in a graduated cylinder is, and how close to the expected mass (at room temperature, the mass of 10 mL of distilled water is about 9.9820 grams). Measure the mass of a dry empty 10 mL graduated cylinder. Then, fill it to 10 mL, measure the gross mass, calculate the net mass and record it. Remove some water with a transfer pipette, fill to 10 mL again, and record the mass. Record a third mass the same way. Then, calculate the average and the standard deviation. Compare your result with the claim on the graduate cylinder. The easiest way to do that is the convert the average mass and the standard deviation into volumes by dividing by the density of 0.99820 g/mL.

1d) Cleaning the balance

Before moving on to the next part, check the weighing pan, the bottom of the balance and the surrounding of the balance whether there is any powder or liquid you spilled. It is important that you clean up after using the balance because you are the one that knows which substances you used. If there is a major spill, inform your instructor.

Part 2: Drops as volume measurement

There are expensive instruments to dispense or measure tiny volumes, such as a Hamilton syringe, an adjustable micropipette, or the head of an inkjet printer. The cheapest way to dispense a small volume is to count drops (like you would do for eye drops, for example). Here, we will estimate the volume of drops made in different ways, and explore how consistent they are.

2a) Volume of a drop of water

Fill a 10 mL graduated cylinder to the 6 mL mark with water. Then, using three different ways to make drops (transfer pipette, glass dropper, beaker), count how many drops it takes to add 1 mL. Then, calculate the average drop size in µL (1 mL = 1000 µL) and record it on the summary sheet.

2b) How consistent are drops?

To explore how consistent the drop volumes are, you will make a green solution from a yellow and a blue solution. In the first recipe, take two drops of yellow and one drop of blue, dilute to 5 mL with water, mix and transfer to a medium test tube. Do this in triplicate. Now, take ten drops of yellow and five drops of blue, dilute to 25 mL with water, mix, and transfer some of it to a medium test tube to the same height as the 5 mL samples for good visual comparison (discarding the remainder). Do this in triplicate as well. Compare the color of the solutions. How much variation is there within the triplicates, and between the two different recipes? Look around for another set of green solution, and compare yours with theirs.

Part 3: Volumetric glassware

For this part, **you will work in pairs**, but write your own words into the summary sheet.

We use volumetric glassware such as graduated cylinders and burets to measure the volume of liquids. There are other glass containers in your drawer (beaker, Erlenmeyer flask, filter flask) that are not designed to measure volume.

We will use a buret today as our most accurate and precise way of measuring volumes. We will practice to read volumes to 0.01 mL, so please always quote measurements to two decimal places, i.e. if the liquid level is *exactly* at the 1 mL line, you record this value as 1.00 mL. Note that the scale goes from 0.00 mL at the top (filled) to 50.00 mL at the bottom (empty). If you are outside of this range before or after dispensing, you do not know how much you dispensed. For instructions how to read off the buret, watch this 4-minute video.



We can figure out how much liquid we dispensed with a buret by writing down the liquid level before and after dispensing, and taking the difference. For example, if the liquid level was at 0.15 mL before dispensing and at 36.72 mL after, you dispensed V = 36.72 mL - 0.15 mL = 36.57 mL.

$$V_{\text{before}} = 0.15 \text{ mL}$$

$$V_{\text{after}} = 36.72 \text{ mL}$$

$$V_{\text{dispensed}} = V_{\text{after}} - V_{\text{before}} = 36.57 \text{ mL}$$

→ explore live on PQcalc

Before you can start, you have to set up the buret and fill it with water so that the water level is in between the 0 mL and the 1 mL mark and there is no air in the spout. Ask your instructor to check the setup before you start.

3a) Accuracy and precision of measuring 10 mL

We will compare and contrast two graduated cylinder sizes (10 mL and 100 mL) as well as a 50 mL beaker to explore which is best to measure 10 mL of water. Starting with a dry empty container and a buret with known starting volume, dispense water from the buret into the container to the 10 mL line. As you get closer to the line, you should adjust the dispensing speed to a slow drip. Once you reached the line, read off the buret, calculate the dispensed volume and record it in your notebook. For each item of glassware, do this in triplicate and calculate the average and the standard deviation. With your partner, you can do two measurements before you have to empty and dry your containers for the third measurement. Copy your results into the lab summary sheet.

3b) To deliver or to contain

For the set of experiments above, you used the buret “to deliver” and the graduate cylinder “to contain”, the way it is intended. Measurements “to contain” are useful in making a solution or reaction mixture where the final volume in the container is critical. Measurements “to deliver” are useful in titrations, where you add solution until you observe a color change or other “endpoint”, and you need to know how much you added. We will encounter burets again in future labs.

Just to experience using the buret and the graduate cylinder in the unintended way, take a graduated cylinder filled with 10 mL water and try to deliver the liquid to the buret, recording the beginning and the final volume. Make sure your first buret reading is larger than 10 mL, otherwise you will overfill the buret. How much water did you add to the buret according to the two readings? Is it more or is it less than you expected? What are the problems you encounter trying this?

Part 4: Mixing liquids and dissolving solids

For this part, **you will work in pairs**, but write your own words into the summary sheet.

How will samples mix depends on their scale. In a small volume of a liquid (such as the cytosol of a bacterial cell), everything mixes rapidly because of diffusion, i.e. particles moving all the time. The larger the volume, the more effort you have to put into mixing if you want a homogenous mixture (a solution). Quoting from Wikipedia, “modern industrial processing almost always involves some form of mixing”. We will explore mixing of liquid volumes typically used in the GenChem lab.

4a) Order of addition

Take two beakers of the same size, one from each of your drawers. In the first beaker, fill to the highest line with water and then add one drop of red solution. To the second beaker, add one drop of read solution and then fill to the highest line with water. Observe them for 20 seconds and describe the difference.

4b) Mixing by swirling

Fill a beaker, an Erlenmeyer flask and a graduated cylinder with water. Then, add a drop of red solution to each. Try to mix each by swirling. Compare your results, in terms of efficiency of mixing and in terms of splashes, if any.

4c) Mixing with a stir bar, a stirring rod or inverting

Repeat the setup from 4b) but without swirling. Use a stirring rod to stir the contents of the beaker. Use a magnetic stir bar and a magnetic stirrer for the Erlenmeyer flask. Use parafilm to cover the top of the graduated cylinder and invert to mix. Which of the methods did you like the best? Which do you think resulted in the largest loss of solution (clinging to the stir bar, to the stirring rod, or to the parafilm)?

4d) Dissolving some salt (thought experiment)

Using what you just learned, make a plan to dissolve a tablespoon of salt in water, giving a total volume of 50 mL. Which container would you use to dissolve the salt in water? How would you mix it? Which container would you use to make the solution up to 50 mL with water? How would you minimize losses of transfer?

Cleanup

Before you clean up, show your summary sheet to your instructor to get an exit stamp on it. Once you received permission to wrap things up, empty your buret (no need to rinse because it held water only) and clamp it in an inverted orientation to dry. Rinse the containers you used, let them drip off at your bench and put them back into your drawer. Double check that the balance(s) you used are clean and if not, clean them. Take your lab notebook with you if you'll need to prepare it before next week's experiment. Wipe off your bench area and hand in your summary sheet before you leave.

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1.2: Lab 2 density

Lab 2 Density of sweet drinks

Objectives

- Explore density of solids, liquids and gases
- Explore how density is related to floating in water (buoyancy)
- Determine the density of a sweet drink (two choices, different colors)
- Evaluate sources of error in measurements and their effect on an experiment.

Safety and Notes

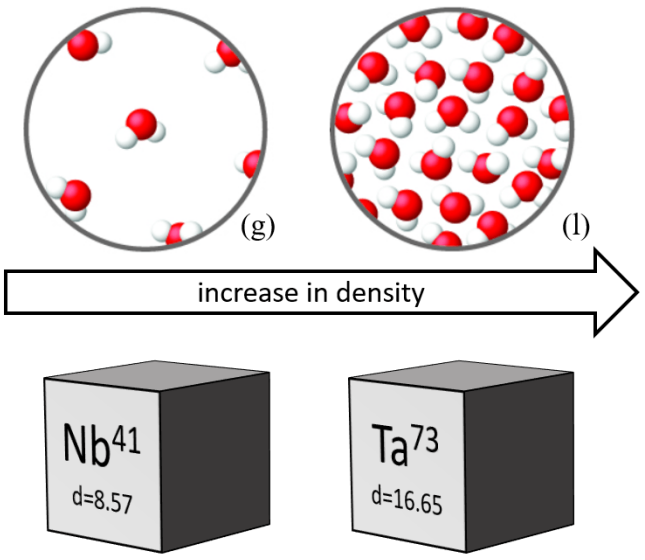
We will not generate any hazardous waste today. Once the sweet drinks enter the lab, they are no longer for human consumption, and we will discard them at the end of the week. In one task, we will heat up a metal strip to high temperature. Do not touch the strip until you cooled it down with cold water.

Introduction

Density

The density of an object is defined as the ratio of its mass to its volume:

$$\text{Density} = \frac{\text{Mass}}{\text{Volume}} \tag{1.2.1}$$



For an example of density, consider the following: Imagine a brick that is made of Styrofoam. Imagine a second brick that is made of lead. Note that even though the bricks take up the same amount of space - that is, they have the same volume - there is a major difference in their mass. We would say that the lead is denser, that is it has more mass in the same volume.

How can we explain density at the atomic level? The closer atoms are to each other, the higher the density. For example, the density of liquid water is higher than that of water vapor (steam) because the water molecules are much closer together in the

liquid (see image on the right). The heavier the atoms are, the higher the density. For example, the density of metallic tantalum is about twice that of niobium, even though they have similar chemical properties and are in the same group of the periodic table (which is why they are sometimes called twins).

Density of (liquid) water

It is important to note that liquid water has a density of approximately 1.0 g/mL. So at room temperature, 50.0 mL of water has a mass of almost exactly 50.0 g.

Two factors have an effect on the density of water:

1. Temperature will have a small effect on the density. For water, density increases as temperature decreases. For example, a 10.000 mL volume of water will increase by 0.016 mL when the temperature is raised from 18°C to 25°C. See Table 1 for the density of water at different temperatures.

2. When solids are dissolved in the water, the solution will typically have a higher density than pure water. We might see this effect in today's lab when we compare the density of sweet drinks (aqueous solutions) with the density of water.

Temp (°C)	d_{H_2O}(g/mL)	Temp(°C)	d_{H_2O}(g/mL)
18.0	0.99860	22.0	0.99777
18.5	0.99850	22.5	0.99765
19.0	0.99841	23.0	0.99754
19.5	0.99830	23.5	0.99742
20.0	0.99820	24.0	0.99730
20.5	0.99809	24.5	0.99716
21.0	0.99799	25.0	0.99704
21.5	0.99788	25.5	0.99690

Table 1. Density of water at different temperatures

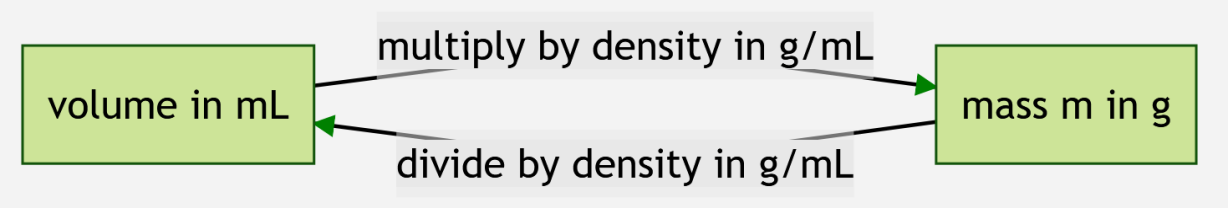
A table of the theoretical values of density for sucrose (table sugar) solutions of various (*w*/*w*)% is included in Table 2 below.

Mass %	Density (g/mL)	Mass %	Density (g/mL)
0.00	1.000	12.50	1.051
2.50	1.011	15.00	1.062
5.00	1.021	17.50	1.073
7.50	1.030	20.00	1.084
10.00	1.042	22.50	1.102

Table 2: Theoretical Density Values of Sucrose Solutions with Known Mass Percent

Calculating with density

If you know the density of a sample (in g/mL), you can determine the volume from the mass of the sample, and vice versa.



Buoyancy

The concept of buoyancy (a physics concept) is relevant to the interpretation of some of the tasks below. Buoyancy is an upward force of objects submerged in a liquid that explains why some objects sink and others float in a given liquid. The buoyancy is equal to the gravitational force on the displaced liquid, i.e. it depends on the volume of the floating object (or, if it is not fully submerged, the volume that is under the surface) and on the density of the liquid.

Objects that have a density less than water, that is, less than 1.0 g/mL, will float on the surface of the water, with more or less of the object submerged. Those that have a density greater than 1.0 g/mL will sink. Consider our two bricks again. The brick of Styrofoam will float if we toss it into water. The lead will sink "like a rock".

Relative error

In this experiment, we will discuss the relative error, expressed in percent, of our measurements. The equation for finding relative error (or percent error) is:

$$\text{Relative error } = \frac{\text{(Experimental Value-Theoretical Value)}}{\text{Theoretical Value}} \times 100\% \tag{1.2.2}$$

If we want to decide whether to use a 100 mL or a 10 mL graduated cylinder for our density measurements, the relative error will be the deciding factor. If the 100 mL cylinder has a tolerance of 0.4 mL and the 10 mL cylinder has a tolerance of 0.05 mL, which one has the smaller absolute volume error? Which one has the smaller relative error? Assuming you have a balance that has a relative error of 0.01%, which volume is better for a density determination?

Graphing Data

In one of the tasks, you will use a graphical method to determine density. It is recommended that you use a spreadsheet for this task (tutorial for Google sheets, MS Excel, IOS Numbers). Make a scatter plot of your data and fit a straight line to it. Then, jot down the formula of the line, given as y = m x + b. You might chose to set b (y-intercept) to zero, but you don't have to. If you do, the formula is simply y = m x. In either case, m is the slope you need for the analysis.

Experimental Procedure

To have less crowding at the balances, you either start with task 1 or with task 2. Do task 3 only when you are done with the two other tasks. Record your observations and calculations in your lab notebook, and use the lab summary sheet to hand in your results and reflections.

Part 1: Density of sweet drink

The protocol below is a bit weird. We are filling a graduated cylinder (which is designed "to contain") from a beaker, whose mass we monitor. We could have measured the mass of the graduated cylinder instead; however, the graduated cylinder has a higher mass (getting close to the maximum of the analytical balance) and an awkward shape for use in the balance.

1. Pour about 60 mL of sweet drink (there are two choices, with different colors) into a 100 mL beaker and measure the total mass (beaker + drink).
2. At your bench, pour 10 mL from the beaker into a 100 mL graduated cylinder. Go slow and steady, and use a transfer pipet for the last milliliter. Observe the meniscus carefully. Any leftover solution in the transfer pipet goes back into the beaker.
3. Measure the total mass of the sweet drink in the beaker again (the mass should be less because you removed 10 mL from it).
4. Repeat steps 2 and 3, adding drink to the same graduated cylinder (so it will be filled with 10 mL, 20 mL, 30 mL, 40 mL and

finally 50 mL. Make sure the balance is zeroed before you place the beaker on the weighing dish.
5. Graph the removed volume against the removed mass. The *x* axis should be the volume in mL and the *y* axis the mass in g.
6. Fit a line to the data. The slope of the line will have units g/mL and is a measure of the density of the drink.
7. In a different way of analyzing the data, subtract the final mass from the initial mass and divide by the difference in volume to get the density.
8. When you are done, pour the drink into the appropriate one of the two provided containers (we will use it again, and monitor whether the density changes over the week due to evaporation or contamination).

Part 2: Density changes with temperature, pressure, and physical state

Walk through the exhibit (any order), explore the objects and answer the questions on the summary sheet

Part 3: Alternative density determination

Choose **one** of the alternative density determination methods below. Choice 1 can be done at your bench, the others at separate stations. Not all stations might be available.

1. Graduated cylinder

Taking a clean and dry graduated cylinder, measure the mass, and then fill to 10, 20, 30, 40, and 50 mL with the drink you used for task 1, measuring the mass at each step. Use the same graphical analysis as in task 1 to determine the density. When you are done, pour the drink into the appropriate one of the two provided containers (we will use it again, and monitor whether the density changes over the week due to evaporation or contamination).

2. Volumetric flask

Taking a clean and dry 50 mL volumetric flask, measure the mass, and then carefully fill to the line with the drink you used for task 1. If you have a drink that likes to make bubbles, a funnel will be helpful. Measure about 45 mL of the drink in a beaker, and slowly pour it into the funnel whose outlet is at the bottom of the volumetric flask. Use a transfer pipet for the last bit, making sure the liquid does not wet the volumetric flask above the line, and measure the mass. Calculate the density from your data and compare it with that from task 1. When you are done, pour the drink into the appropriate one of the two provided containers (we will use it again, and monitor whether the density changes over the week due to evaporation or contamination).

3. Potato and egg

See whether a potato and a hard-boiled egg float or sink in your drink. Each object has a volume of 60-80 mL, so choose a beaker that the object fits in, place it in the beaker, and fill with the sweet drink until submerged. If they float, estimate which fraction of the object is above the surface of the liquid. From your observations, estimate the density of the liquid. When you are down, rinse and dry the potato and the egg. Pour the drink into the appropriate one of the two provided containers (we will use it again, and monitor whether the density changes over the week due to evaporation or contamination).

4. Below balance measurement

Zero the balance and attach the “sinker” (a large marble on a string) to the hook, zeroing the balance. Submerge the sinker in pure water at room temperature and record the mass difference (which will be negative). Then, dry the sinker and submerge it in your sweet drink, recording the mass difference (which will be negative). The ratio of the mass differences is equal to the ratio of densities. Look up the density of pure water and calculate the density of your sweet drink. When you are done, pour the drink into the appropriate one of the two provided containers (we will use it again, and monitor whether the density changes over the week due to evaporation or contamination).

5. Above balance measurement

Place a beaker with water on the balance and zero the balance. Submerge a “sinker” in the water and record the mass. It represents the buoyancy of the sinker (and you can determine the volume of the sinker if you know the density of the water).

Repeat with your sweet drink. If it has a higher density than water, the buoyancy will be larger. The ratio of the masses measured is equal to the ratio of densities. Look up the density of pure water and calculate the density of your sweet drink. When you are done, pour the drink into the appropriate one of the two provided containers (we will use it again, and monitor whether the density changes over the week due to evaporation or contamination).

Acknowledgement

Parts of this lab were adopted from Gerber-Morales's density lab.

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1.3: Spreadsheet tutorials

What do I hand in?

This lab is asynchronous. This means you can work on it at your own pace. Each lab section has one cancelled lab session (either the week of Indigenous People Day or the week of Thanksgiving, depending on which week day you meet. The lab is due that week, but it makes more sense to work through the topics earlier, as the skills you learn are useful to have in the lab.

To receive credit for this lab, please hand in printouts of one data table with descriptive statistics (part 1), one data table where at least one of the columns is calculated with a formula you entered yourself (part 2), and one scatterplot where you fitted data to a line. Ideally, these would be from data you collected yourself, for the first and second lab, as recorded in your lab notebook. If that is not possible, contact your instructor for an alternative.

Part 1: Data tables and descriptive statistics

Here are the slides used in the video.

Data tables

Here is an example of a data table for measuring the mass of some samples:

Object	**m_1 (g)**	**m_2 (g)**	**m_3 (g)**	**average mass (g)**
Apple	154.627			
Banana				
Carrot				

You could make the data table headers before you start the lab, and then add the data as you measure it.

Descriptive statistics

When you measure something multiple times, there will be some variation in your measurements. You can summarize the measurements by giving the average, the standard deviation, and N, the number of measurements. These values give you an expectation of what to expect of additional measurements. In our example above, we are just calculating the average, i.e. the sum of the measurements divided by N, the number of measurements. You could do this using a pocket calculator but that becomes tedious (and error-prone) when having to calculate a lot of averages. Instead, with an electronic spreadsheet, you can enter a formula into the column for the average, and the spreadsheet will do the math. As an added bonus, it is very easy to copy and paste the formula into the remainder of the column. The video tutorial will show you how to enter a formula, and how to copy and paste it.

Apart from average and standard deviation, you might also be asked to calculate median, standard error and relative error. Here is a brief summary of why these are useful.

Average: Also called "mean". If your experiments does not have any systematic errors, the average will be closer to the true value than individual measurements because random errors cancel out when averaging.

Median: You expect 50% of your measurements to be higher than the median, and 50% to be lower.

Standard deviation: Describes the spread of the measurements around the average. If the deviation is due to small random errors, about 2/3 of the measurements will be within one standard deviation from the average.

Standard error: an estimate how far from the true value your average is. The idea is that the average is a better estimate than any

single measurement because you are averaging out random errors. To get the standard error, you divide the standard deviation by the square root of the number of measurements. The more measurements, the smaller the standard error. For example, comparing the average of 10 measurements to 40 measurements (i.e. four times as many), the standard error gets cut into half. A quick way of giving average and standard error is to write something like "m = 45.33 ± 0.04 g".

Relative error: This is giving the standard deviation or standard error as a percentage of the average. You could say that the relative volume error of a pipette is 0.5%. For a volume of 100 mL, you would expect to be off by about 0.5 mL. Or you could say the average of your measurements is expected to be off by 2%, which would be the standard error divided by the average.

Part 2: Repetitive calculations

Sometimes, you have to do the same set of calculations for every measurement in your experiment. It makes sense to use a spreadsheet for these repetitive calculations because it is convenient and you can avoid mistakes. Even if the arithmetic is taken care of for you, you still are responsible for correct data entry, formulas, units and significant figures.

YouTube Video

This interactive element is not available in this format. Visit the link or scan the QR code to access it online.

View this YouTube Video online

Part 3: Plotting data and fitting a line to it

YouTube Video

This interactive element is not available in this format. Visit the link or scan the QR code to access it online.

View this YouTube Video online

This is a brief tutorial showing you how to plot your data (for a lab report or presentation). It also shows you how to fit a line to your data in the scatter plot. Data fitting within a spread sheet is more reproducible than with pencil, paper and ruler (and faster).

Here is the finished spread sheet. When you click on the link, it asks you whether you want to make a copy of it, and you should say yes if you want to edit a copy of it.

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1.4: Soap lab

Making Specialty Soap: Chemistry of Saponification

A Two-Week Guided-Inquiry Experiment

Saponification Background

Chemistry of Saponification

To make soap, a fat or oil containing fatty acid triglyceride molecules is combined with a strong base such as sodium hydroxide (NaOH) or potassium hydroxide (KOH). Under the right reaction conditions, this will form glycerol and a salt of the fatty acid. The chemical equation is

$$\text{Fatty acid triglyceride} + 3\,\text{NaOH} \longrightarrow 3\,\text{fatty acid salt} + \text{glycerol} \tag{1.4.1}$$

Each fatty acid triglyceride contains three fatty acid groups that yield fatty acids salts (soap) in the presence of NaOH. For example, the fatty acid stearic acid ($C_{17}H_{35}CO_2H$) yields sodium stearate ($C_{17}H_{35}CO_2Na$), shown below.

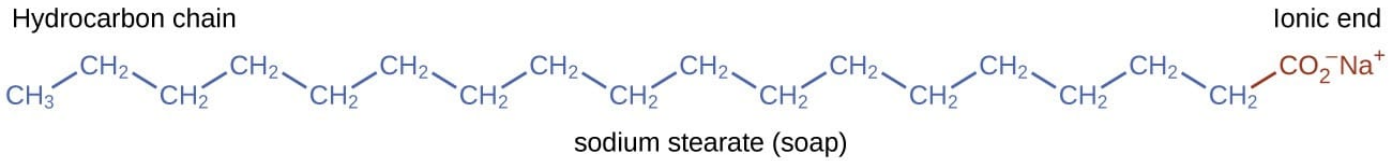


Figure 1: Sodium stearate is an example of a salt of a fatty acid which is just one example of a compound that can be found in soaps[1]

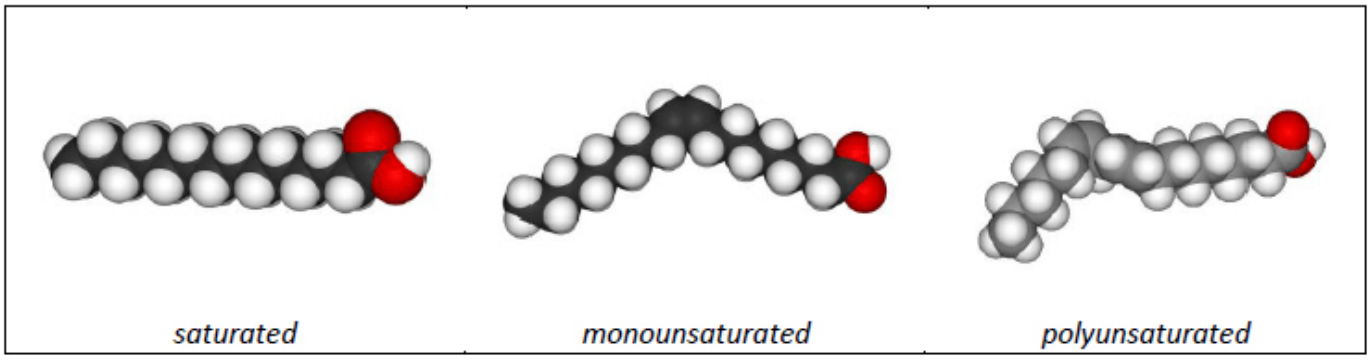
We'll be using sodium hydroxide (NaOH) as the base in the soap reaction. Check out this video which describes the interesting history and importance of potash, which contains potassium instead of sodium, used in early soap making and other applications.

Making Soap as a Specialty Product

Making specialty soaps is popular by craft enthusiasts and small businesses. Depending on the ingredients used to create the soap, the final product can have different physical properties including texture, color, and smell that appeal to a consumer. The soap can also have desirable properties such as being gentle for sensitive skin, moisturizing, exfoliating, cleansing, stain removing, etc. When manufacturing and selling soaps, cost also must be taken into consideration. For this experiment, you will choose soap ingredients to create a specialty soap that you will pitch to your classmates and try to convince them to buy your product.

The main decision you'll need to make when making your soap is what oil/fat to use. The chemical composition and the structure of the fatty acid compounds present in the fat/oil you choose is extremely important to many properties of your soap, including its final hardness. One major difference between fatty acids is if the compound is "saturated" or "unsaturated". Saturated fatty acids mean that each carbon atom is surrounded by the maximum number of hydrogen atoms. Unsaturated fatty acid molecules have at least one carbon atom that has formed a double bond with an adjacent carbon, so there are less hydrogen atoms surrounding it. See the examples of the chemical structures of saturated and unsaturated fatty acids in Figure 2.

Figure 2: Chemical structures showing examples of saturated, monounsaturated, and polyunsaturated fatty acid molecules. The black spheres are carbon atoms, the white are hydrogen, and the red are oxygen.



Experiment Objectives

For this 2-week guided inquiry experiment, you will work with a lab partner to:

- Investigate how the chemical structure of different oils/fats affect the properties of soaps.
- Use this information as well as the costs of all ingredients to determine how to make the “best” specialty soap
- Test the properties of the soap you create
- Design a method to describe how well your soap lathers
- Give a short “sales pitch” presentation to try to convince your peers that you have made the best specialty soap in the lab

Experimental Procedure for Week 1

You’ll work together in pairs for this two-week experiment. Write in your lab notebook and complete the Summary Sheet for Week 1 as you go through the procedure.

Safety Considerations

- Wear your safety goggles at all times during this experiment.
- Concentrated sodium hydroxide is caustic and will irritate and burn the eyes and skin. Handle this solution carefully and thoroughly clean up any spills. Here is an example Safety Data Sheet (SDS) for a 20% sodium hydroxide solution.

Part A: Making Your Soap[2] [3]

1. With your partner, decide which fat(s) or oil(s) to use to prepare your soap. You are welcome to use your own oil or fat if you brought any, or use one provided in lab, shown in the table below. Be sure to choose oils(s)/fat(s) containing fatty acid triglycerides – so think cooking fats and oils and not motor oil or mineral oil. You may use only one fat/oil or use a mixture of up to two different fats/oils. The total amount of fat/oil needs to be 10mL.

Fat or Oil	Approximate cost per bar of soap
Olive oil	$1.00
Canola oil	$0.30
Coconut oil	$1.40
Vegetable shortening	$0.70

The approximate costs of each oil were based on prices found at Walmart using 4 oz (a standard size for a bar of soap). If you

are using a different oil or fat, find the price per oz. online, and calculate the total cost for 4 oz.

1. Using a hot plate set at a low temperature, gently heat the fat or oil in a beaker until it reaches a temperature of 35 °C. Using a graduated cylinder, measure a total of 10 mL of warm fat or oil and then pour into a 250 mL beaker.

2. Add 3 mL of 20% sodium hydroxide solution to the beaker. Use a glass stirring rod to combine the oil/fat and sodium hydroxide. Add a few grains of stearic acid, which will help harden your soap.

3. Place a stir bar into your beaker. Then place the beaker on a stir plate, turn on and then gradually increase the speed to avoid splattering. Every so often you'll also want to take the beaker off the stir plate and use the glass stirring rod to stir the mixture by hand, paying extra attention to scrape around the edges of the beaker that might not be stirred effectively with the stir bar. Use the rest of the lab period to stir this as long as possible. Particularly for the oils that are liquid at room temperature (canola oil, olive oil, etc.), stirring a longer time will allow the reaction to complete and the mixture will thicken. Ideally, the mixture will achieve a thicker consistency called “trace” that you can see in the image below. This ensures that you have created an emulsion. If the reaction does not finish and you do not form an emulsion, you risk having unreacted sodium hydroxide left in your soap which will make it unsafe to use. At least one lab partner should stay at the lab bench to monitor your soap while it is stirring, but you both are able to multi-task and complete other tasks at the lab bench.



Figure 3: This image shows “trace,” when the soap mixture is thick you can drip some onto the surface of the mixture and it will not immediately sink back in.[4]

4. Once your reaction is complete, if desired, add one drop of fragrance to your mixture and mix thoroughly. Essential oils can be expensive, so add $1.00 to the cost of your bar of soap if you add fragrance.

5. Add one drop of coloring if desired. Add $0.20 to the cost of your bar of soap if you use coloring.

6. Use a sharpie to clearly label the bottom of an aluminum weigh boat with you and your partner’s initials, your lab section and lab time, and the current date. Pour your soap mixture into the aluminum weigh boat and let it sit until next week. During the week, your soap will finish reacting and set.

Once you are done with the experiment, be sure to thoroughly clean all of your glassware and equipment you used with warm, soapy water and a brush to remove all fat/oil and soap residues.

Experimental Procedure for Week 2

Part B. Testing Properties of Soap

Write in your lab notebook and fill out the Summary Sheet for Week 2 as you work through the procedure. Wear gloves as you handle your soap in case there is any unreacted sodium hydroxide still present.

1. Test the pH

You’ll want to test the pH of your soap to make sure there is no unreacted sodium hydroxide that would make your soap unsafe to use. An easy way to test for pH is to put a drop of *deionized* water on your soap and then dip a pH strip in the soap solution

that forms. If your soap pH is between 7-10, it is safe to use and touch. If your pH is above 10, there is still too much unreacted sodium hydroxide and it will be unsafe to touch. However, for best safety practices, continue to wear your gloves when handling your soap regardless of the pH you measure.

2. Physical Properties

Observe the color, smell, and hardness of your soap. Be descriptive in your recorded observations.

3. Ability to Lather

Compare the ability of your soap to lather compared to commercial bar soap. Come up with a qualitative or quantitative method to test which soap produces a better lather (i.e. more suds). You can compare the amount of suds from your soap to the suds from the commercial soap OR determine the volume of the suds produced from your soap compared to the volume of suds from the commercial soap. Record your method and observations. Come up with a simple statement to express your results that you can share in your presentation.

4. Ability to Clean[5]

Test the ability of your soap to clean a stain. You will be provided with index cards, lip stick, and cotton swabs. Place a small amount of lipstick on the index card. Wet the cotton swab and use your soap to create a lather. Try to “clean” the lipstick stain with your soapy swab and observe and record your results. If time allows, try to clean a lipstick stain with just water and/or commercial soap to compare your results.

Part C. Sales Pitch Presentation (Week 2)

After you have conducted all your tests investigating the properties of your soap, prepare a short presentation (3 - 5 minutes) that highlights the desirable qualities and properties of your soap. Split the presentation time between you and your lab partner.

When giving your presentation, you will stand in front of the class with your soap and, if desired, any other visuals you prepared during testing. You may also utilize the white board if there is anything you would like to write or draw to help your pitch. You are welcome to refer to the notes on your worksheet while you are giving your presentation, or you may also prepare a note card or other outline of the important points you want to cover in your presentation. The presentation will be graded according to the provided rubric.

In your presentation, be sure to include the following:

1. Start by discussing your soap recipe and why you chose it. Include relevant background research you did in Week 1 and important observations from your soap making.
2. Describe your soap's properties. Show off your soap and any other visual aids from the experimental testing.
3. Evaluate the procedures from your experimental testing of your soap's properties. What went well and what didn't? Give suggestions for ways to improve if you were given a chance to make and test another batch of soap.
4. Be persuasive in your presentation. What makes your soap stand out from all the others? Consider coming up with a brand name, logo, slogan, etc. for your soap. How did it perform compared to the commercial soap?

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1. Flowers, P., Theopold, K., Langley, R., Robinson, W.R. et. al. Section 11.5 Colloids. In *Chemistry 2e*, 2nd edition; OpenStax, 2019. https://openstax.org/books/chemistry-2e/pages/1-introduction
2. Phansteil IV, O, Dueno, E. and Wang, Q.X. Synthesis of Exotic Soaps in the Chemistry Laboratory. *J. Chem. Ed.* **1998**, 75, 5.
3. Making Soap Saponification Lab Procedure. https://chem.libretexts.org/@go/page/306794 (accessed Jun 18, 2021).
4. Best Ways to Control Trace When You Make Cold Process Soap. https://www.modernsoapmaking.com/blog/controlling-trace-in-cold-process-soapmaking (accessed Aug 20, 2021).
5. Soap and Detergent: Release the Grease. https://www.acs.org/content/dam/acso...the-grease.pdf (accessed Jun 18, 2021).

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1.5: Soap lab

Making Specialty Soap: Chemistry of Saponification

A Two-Week Guided-Inquiry Experiment

Saponification Background

Chemistry of Saponification

To make soap, a fat or oil containing fatty acid triglyceride molecules is combined with a strong base such as sodium hydroxide (NaOH) or potassium hydroxide (KOH). Under the right reaction conditions, this will form glycerol and a salt of the fatty acid. The chemical equation is

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Each fatty acid triglyceride contains three fatty acid groups that yield fatty acids salts (soap) in the presence of NaOH. For example, the fatty acid stearic acid ($C_{17}H_{35}CO_2H$) yields sodium stearate ($C_{17}H_{35}CO_2Na$), shown below.

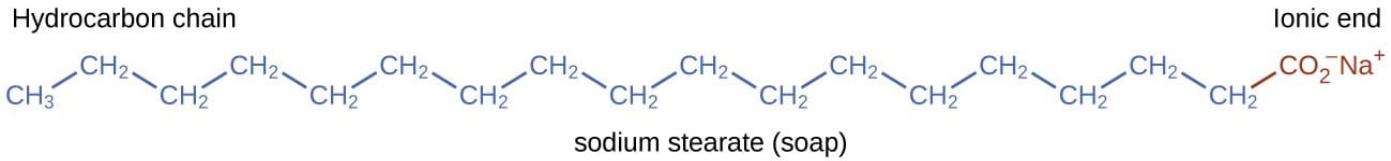


Figure 1: Sodium stearate is an example of a salt of a fatty acid which is just one example of a compound that can be found in soaps[1]

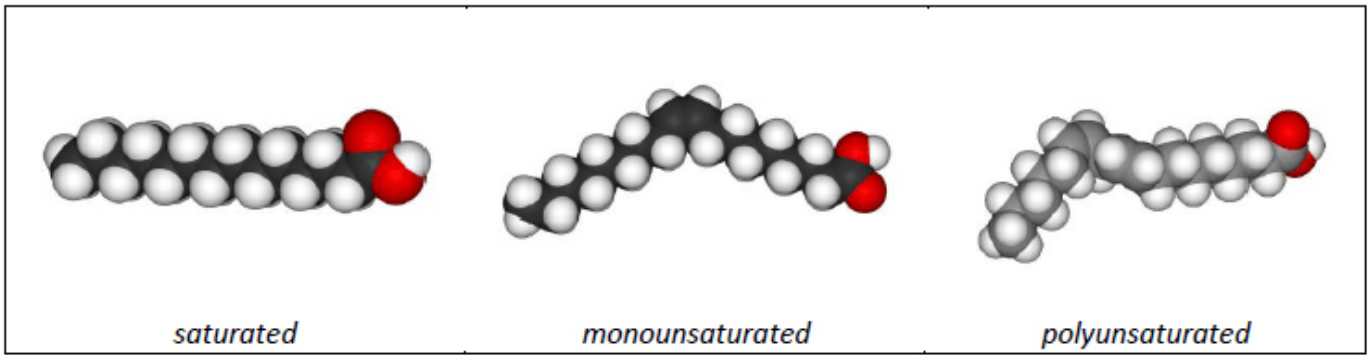
We'll be using sodium hydroxide (NaOH) as the base in the soap reaction. Check out this video which describes the interesting history and importance of potash, which contains potassium instead of sodium, used in early soap making and other applications.

Making Soap as a Specialty Product

Making specialty soaps is popular by craft enthusiasts and small businesses. Depending on the ingredients used to create the soap, the final product can have different physical properties including texture, color, and smell that appeal to a consumer. The soap can also have desirable properties such as being gentle for sensitive skin, moisturizing, exfoliating, cleansing, stain removing, etc. When manufacturing and selling soaps, cost also must be taken into consideration. For this experiment, you will choose soap ingredients to create a specialty soap that you will pitch to your classmates and try to convince them to buy your product.

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Figure 2: Chemical structures showing examples of saturated, monounsaturated, and polyunsaturated fatty acid molecules. The black spheres are carbon atoms, the white are hydrogen, and the red are oxygen.



Experiment Objectives

For this 2-week guided inquiry experiment, you will work with a lab partner to:

- Investigate how the chemical structure of different oils/fats affect the properties of soaps.
- Use this information as well as the costs of all ingredients to determine how to make the “best” specialty soap
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Experimental Procedure for Week 1

You’ll work together in pairs for this two-week experiment. Write in your lab notebook and complete the Summary Sheet for Week 1 as you go through the procedure.

Safety Considerations

- Wear your safety goggles at all times during this experiment.
- Concentrated sodium hydroxide is caustic and will irritate and burn the eyes and skin. Handle this solution carefully and thoroughly clean up any spills. Here is an example Safety Data Sheet (SDS) for a 20% sodium hydroxide solution.

Part A: Making Your Soap[2] [3]

1. With your partner, decide which fat(s) or oil(s) to use to prepare your soap. You are welcome to use your own oil or fat if you brought any, or use one provided in lab, shown in the table below. Be sure to choose oils(s)/fat(s) containing fatty acid triglycerides – so think cooking fats and oils and not motor oil or mineral oil. You may use only one fat/oil or use a mixture of up to two different fats/oils. The total amount of fat/oil needs to be 10mL.

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Coconut oil	$1.40
Vegetable shortening	$0.70

The approximate costs of each oil were based on prices found at Walmart using 4 oz (a standard size for a bar of soap). If you

are using a different oil or fat, find the price per oz. online, and calculate the total cost for 4 oz.

1. Using a hot plate set at a low temperature, gently heat the fat or oil in a beaker until it reaches a temperature of 35 °C. Using a graduated cylinder, measure a total of 10 mL of warm fat or oil and then pour into a 250 mL beaker.

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3. Place a stir bar into your beaker. Then place the beaker on a stir plate, turn on and then gradually increase the speed to avoid splattering. Every so often you'll also want to take the beaker off the stir plate and use the glass stirring rod to stir the mixture by hand, paying extra attention to scrape around the edges of the beaker that might not be stirred effectively with the stir bar. Use the rest of the lab period to stir this as long as possible. Particularly for the oils that are liquid at room temperature (canola oil, olive oil, etc.), stirring a longer time will allow the reaction to complete and the mixture will thicken. Ideally, the mixture will achieve a thicker consistency called “trace” that you can see in the image below. This ensures that you have created an emulsion. If the reaction does not finish and you do not form an emulsion, you risk having unreacted sodium hydroxide left in your soap which will make it unsafe to use. At least one lab partner should stay at the lab bench to monitor your soap while it is stirring, but you both are able to multi-task and complete other tasks at the lab bench.



Figure 3: This image shows “trace,” when the soap mixture is thick you can drip some onto the surface of the mixture and it will not immediately sink back in.[4]

4. Once your reaction is complete, if desired, add one drop of fragrance to your mixture and mix thoroughly. Essential oils can be expensive, so add $1.00 to the cost of your bar of soap if you add fragrance.

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Once you are done with the experiment, be sure to thoroughly clean all of your glassware and equipment you used with warm, soapy water and a brush to remove all fat/oil and soap residues.

Experimental Procedure for Week 2

Part B. Testing Properties of Soap

Write in your lab notebook and fill out the Summary Sheet for Week 2 as you work through the procedure. Wear gloves as you handle your soap in case there is any unreacted sodium hydroxide still present.

1. Test the pH

You’ll want to test the pH of your soap to make sure there is no unreacted sodium hydroxide that would make your soap unsafe to use. An easy way to test for pH is to put a drop of *deionized* water on your soap and then dip a pH strip in the soap solution

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2. Physical Properties

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3. Ability to Lather

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Test the ability of your soap to clean a stain. You will be provided with index cards, lip stick, and cotton swabs. Place a small amount of lipstick on the index card. Wet the cotton swab and use your soap to create a lather. Try to “clean” the lipstick stain with your soapy swab and observe and record your results. If time allows, try to clean a lipstick stain with just water and/or commercial soap to compare your results.

Part C. Sales Pitch Presentation (Week 2)

After you have conducted all your tests investigating the properties of your soap, prepare a short presentation (3 - 5 minutes) that highlights the desirable qualities and properties of your soap. Split the presentation time between you and your lab partner.

When giving your presentation, you will stand in front of the class with your soap and, if desired, any other visuals you prepared during testing. You may also utilize the white board if there is anything you would like to write or draw to help your pitch. You are welcome to refer to the notes on your worksheet while you are giving your presentation, or you may also prepare a note card or other outline of the important points you want to cover in your presentation. The presentation will be graded according to the provided rubric.

In your presentation, be sure to include the following:

1. Start by discussing your soap recipe and why you chose it. Include relevant background research you did in Week 1 and important observations from your soap making.
2. Describe your soap's properties. Show off your soap and any other visual aids from the experimental testing.
3. Evaluate the procedures from your experimental testing of your soap's properties. What went well and what didn't? Give suggestions for ways to improve if you were given a chance to make and test another batch of soap.
4. Be persuasive in your presentation. What makes your soap stand out from all the others? Consider coming up with a brand name, logo, slogan, etc. for your soap. How did it perform compared to the commercial soap?

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1.6: Limiting Reactant

Experiment Objectives

- Apply dimensional analysis and the concept of limiting reactants in an example about baking cookies
- Investigate a chemical reaction involving aluminum and copper metals
- Use both observations and stoichiometry to determine a limiting reactant
- Calculate theoretical yields and percent yield

Safety Considerations

- Wear your safety goggles at all times during the experiment.
- HCl is a strong acid and is corrosive to skin and eyes and toxic to inhale. Handle HCl solutions carefully.
- Methanol is flammable. Do not keep containers of methanol near a heat source such as a hot plate.

Experimental Procedure

Record all measurements and calculations into your lab notebook. Then, complete the questions, data, and calculations on your Summary Sheet for today's experiment.

Part 1

Today, we will work with copper and aluminum. As an element, copper is a shiny brown metal (like a penny) whose surface will slowly change color (like the statue of liberty). As an element, aluminum is a shiny silvery metal. Its surface also reacts, but without the dramatic color change. The reaction we will perform today is between $\mathrm{Al} + \mathrm{CuCl_2 \cdot 2\,H_2O}$. You will need a balanced chemical equation for the reaction to be able to analyze your results (some background reading). You should be able to figure out the products from your observations. You can watch a video on how to decant before you start the experiment.

Prepare the reactants

1. Label a 250 mL beaker as “A.” Weigh the beaker and record your measurement in the data and observations table.
2. Using an analytical balance and disposable plastic spatulas and weigh boats, weigh approximately 0.50 g $\mathrm{CuCl_2 \cdot 2\,H_2O}$ and record the exact mass. Be careful using the copper chloride salt. It can damage other metals, so clean up any spills right away.
3. Using the same analytical balance (it's good practice to use the same balance during an experiment to minimize uncertainty), weigh approximately 0.25 g Aluminum foil and record the exact mass. You can tear off small pieces of aluminum foil until you get to the total mass you need. Do not fold or crumple the foil.
4. Place the Al and $\mathrm{CuCl_2 \cdot 2\,H_2O}$ into beaker “A”. Make sure that the aluminum foil is unfolded so that it will completely react.
5. Label a second 250 mL beaker “B”. Weigh and record.
6. Using a balance and a new disposable weigh boat, weigh out 0.70 g of $\mathrm{CuCl_2 \cdot 2\,H_2O}$ and 0.05 g of aluminum foil.
7. Place the Al and $\mathrm{CuCl_2 \cdot 2\,H_2O}$ into beaker “B”. Again, make sure that the aluminum foil is unfolded so that it will completely react.
8. Even if you don't see any spills, clean off the balance by using the brush to sweep anything out of the corners or cracks and use a damp paper towel to wipe it clean.
9. Look at the contents of each beaker. Record the color of substances and any other observations (bubbling, heat formation, etc.) that are visible before the reaction begins (before water is added in step 10) in the data table.

Run the reaction

1. Using a graduated cylinder, measure 50.0 ml of deionized water and add to each beaker. When water is added to the beakers, the $\mathrm{CuCl_2 \cdot 2\,H_2O}$ will dissolve and the reaction will proceed. Note the time or start a timer.

2. Stir the substances in the beakers occasionally with the stirring rod and break up the aluminum foil as it reacts. The reaction should take about 30 minutes to complete.

WHILE YOU ARE WAITING FOR REACTION TO FINISH, COMPLETE PART 2.

3. After waiting at least 30 minutes, now record any color changes or any other observations after the reaction into your notebook.

Separate the reaction mixture to isolate the copper

1. When the reaction is complete and you no longer notice bubbles forming, carefully remove and discard any pieces of aluminum leaving the copper behind in the beaker.

2. Allow the solid Cu to settle in both beakers. Decant (pour off the liquid) the solution from the beakers into a waste container. Be careful not to lose any of the copper.

3. Wash the copper solid with 15 mL of deionized water. Let solid settle. Decant again (be careful to pour as much water off as possible without losing any of the copper solid). Repeat this decanting step once more with another 15 mL of deionized water.

4. Wash the copper solid with 10 mL of methanol. Let solid settle. Decant the methanol into the waste container.

5. Under the hood, heat the beakers on a hot plate at a low setting until dry. Avoid heating at high temperatures for longer periods of time which may cause the unwanted oxidation of the copper product. If you see any "smoke," carefully but quickly take your beaker off the hot plate to cool it down, and turn down the temperature of your hot plate.

6. When the product appears dry, carefully place the beakers on wire gauze or paper towels.

7. After cooling, weigh the beakers and their contents. Record this in your notebook.

Part 2

Now we'll consider an example showing the importance of limiting reactants in baking. On your lab bench you'll find three ingredients (the "reactants") needed to bake peanut butter cookies. Look at the provided recipe to see how much of each ingredient is needed for a batch of cookies. Next consider how much of each ingredient you have by looking at the information provided on the packaging. For the brown sugar and peanut butter, you'll need to convert the amounts into units of cups using dimensional analysis. You will need to look up conversion factors to use in your calculations. The quantity of eggs needed should be an exact number, or a count. Then determine which ingredient is the limiting reactant.

1. Individually: First write down your ideas and initial calculations to determine the limiting reactant by yourself and record into your lab notebook.
2. In a Pair: Once you have completed your calculations, find someone at the same lab bench who is also done and compare your calculations. Talk through any discrepancies, asking the instructor for guidance if needed.
3. In a group at your lab bench: Since you have the same starting ingredients at each lab bench, your final answers should all be the same at the same lab bench. Compare your answers and your work among everyone at the same bench. Do you have the same answer for what the limiting reactant is? You should! Did you all do the same calculations? Not necessarily, as there are multiple different but correct ways to perform unit conversions and limiting reactant calculations! Again, ask your instructor if there are any discrepancies you can't resolve.

Once you are done working out all your calculations in your notebook, fill in the worksheet (individually) to complete the Task 2 questions.

Acknowledgements: This experiment was adapted from Lumen Learning 2014, Chesapeake Campus. https://courses.lumenlearning.com/chemistry1labs/

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1.7: Acid/base titration

Experiment 6: My First Acid - Base Titration

Learning Outcomes

Upon completion of this lab, the student will be able to:

1. Start preparing for emergencies in the chemical lab
2. Set-up a titration stand
3. Prepare primary standard solutions of Potassium Hydrogen Phthalate (KHP) solutions
4. Standardize a solution of Sodium Hydroxide (NaOH)
5. Perform a fast titration and a slow titration

Reagents and Supplies

Approximate 1.0 M aqueous sodium hydroxide (NaOH), potassium hydrogen phthalate (KHP), and a phenolphthalein solution.

Burette, materials from your drawer

(See posted SDS, Safety Data Sheets)

Introduction

Titration is an analytical quantitative technique used to determine the concentration of a solute; a pH-titration is used to determine the concentration of an acid or a base. Titrations play an important role in determining **amount** and **purity** in many manufacturing processes. These include food processing, textile, wood product manufacturing, petroleum, pharmaceuticals and chemical manufacturing. Titrations play a critical role in the manufacturing of biodiesel, water purification and waste water treatment and even in the production of various products in the dairy industry.

Here are some examples from pharmaceutical manufacturing. The active ingredient in many cough syrups, ephedrine, is tested for purity through titration with perchloric acid. Some drugs, such as the antifungal, clotrimazole, are end products of titration reactions. Titrations are even used to determine the amount of non-pharmacologically active ingredients, such as the binding substances that make up medicine tablets.

A titration involves two solutions: the titrant and the analyte. Typically, the **titrant is slowly added from a burette** to the flask containing the analyte until you reach the endpoint. At the endpoint, you know the initial volume of analyte (how much you added to our flask) and the volume of added titrant (by reading off the burette). The titrant is a solution of known concentration and you figure out the concentration of the analyte.

Consider the reaction between aqueous solutions of sulfuric acid and potassium hydroxide. The balanced chemical equation for the reaction is shown below:

$$\mathrm{H_2SO_4\,(aq) + 2\,KOH\,(aq) \longrightarrow K_2SO_4\,(aq) + 2\,H_2O\,(l)} \tag{1}$$

Assume that the molar concentration of KOH is 0.100 M and that of $\mathrm{H_2SO_4}$ is unknown.

For this example, KOH was slowly titrated to 10.00 mL of $\mathrm{H_2SO_4}$. The volume of 13.75 mL of KOH was needed to completely react with the $\mathrm{H_2SO_4}$. The concentration of the sulfuric acid (in the flask, at the beginning of the titration) is calculated as follows:

$$c_{H_2SO_4} = \frac{1}{2} \cdot c_{KOH} \cdot \frac{V_{KOH}}{V_{H_2SO_4}}$$

$$= \frac{1}{2} \cdot 0.100 \ \frac{\text{mol}}{\text{L}} \cdot \frac{13.75 \text{ mL}}{10.00 \text{ mL}}$$

$$= 0.05000 \ \frac{\text{mol}}{\text{L}} \cdot 1.3750$$

$$= 0.0688 \ \frac{\text{mol}}{\text{L}}$$

For an interactive version with more explanation, follow this link or the QR code above.

Two takeaways from this calculation:

1. As seen from the above calculation, the stoichiometric ratio between the two reactants is the key to the determination of the concentration of the unknown solution. (**You need a balanced equation).**
2. Determining the unknown concentration requires **meticulous** determination of the **volume** of the titrant.

In order to conduct the above experiment, typically the H_2SO_4 is in an Erlenmeyer flask, and the KOH is in a burette. The KOH is added one drop at a time from the burette into the acid solution with constant stirring to ensure that the reagents combine and react.

The equivalence point of an acid/base reaction

The **equivalence point** is defined as that point in the titration when stoichiometrically equal amounts of acid and base are present. In the H_2SO_4/KOH example shown previously (eq 1), that would be when two moles of KOH have been added to one mole of H_2SO_4. Therefore, **the equivalence point depends on the reaction stoichiometry**.

At the beginning of the titration, the solution in the Erlenmeyer flask is acidic. As the base is added, it completely reacts with the acid and the solution in the Erlenmeyer flask continues to be acidic. But, at the equivalence point, the acid has completely reacted with the base. If even one tiny drop of base is added beyond that needed to arrive at the equivalence point, the solution in the Erlenmeyer flask is basic. This difference in the acid/base property of the solution in the Erlenmeyer flask is used to visually determine the end of the titration.

Using color to visualize the equivalence point

An **indicator** is a chemical substance whose color depends on the acid/base property of the medium it is present in. Phenolphthalein is an indicator, which is colorless in an acidic medium and has a pink color in a basic medium.

In this titration, a few drops of phenolphthalein should be added to the acid in the Erlenmeyer flask. The solution will remain colorless until the equivalence point.

When the equivalence point has been crossed and the solution becomes basic, the phenolphthalein will take on a pink color. This is the reason to add the base drop by drop, so that even though the equivalence point will be crossed, the titration can be stopped at the appearance of the first permanent pale pink color.

This point in the titration when the indicator changes color is referred to as the **End Point**. Note, the End Point of a titration is slightly beyond the Equivalence Point. In the case of the phenolphthalein, the intensity of the pink color increases as the solution becomes more and more basic. Therefore, **it is important to stop the titration at the appearance of a permanent pale pink color**. In order to easily observe the color changes in the solution, it is a good idea to place a sheet of plain white paper beneath

the flask.

My first titration

Today we will be learning the basics of acid-base titrations. We will be going over the following techniques:

How to

- Assemble a burette stand
- Properly condition the burette for use
- Accurately measure the volume of the burette
- Dispense titrant using a 'stop-cock'
- Determine the end point of the titration
- Use and dispose of waste
- Analyze titration data

Today, we will determine the exact concentration of a solution of sodium hydroxide. In order to accomplish this, we will titrate the sodium hydroxide (NaOH) solution with standardized solution of KHP of known concentration.

Sodium hydroxide is an extremely hygroscopic solid. When solid sodium hydroxide is weighed using an electronic balance it tends to absorb moisture from the atmosphere; this leads to inaccuracies in the mass of the sodium hydroxide and thereby inaccuracies in its molarity calculations.

In order to circumvent the above problem, a sodium hydroxide solution of **approximate concentration** is prepared first. This solution is titrated with a **primary acid standard**. The concentration of this acid is known with greater accuracy. The data from this titration is then used to calculate a more accurate value for the concentration of the sodium hydroxide solution. A commonly used primary standard for titration with sodium hydroxide solution is the weak acid potassium hydrogen phthalate or KHP ($\mathrm{C_8H_5O_4K}$).

The reaction between KHP and NaOH is given below:

$$\mathrm{HC_8H_4O_4K + NaOH \longrightarrow C_8H_4O_4KNa + H_2O} \tag{1.7.1}$$

____ mole(s) of NaOH must be added for every _____ mole(s) of $\mathrm{C_8H_5O_4K}$ in solution

The titration of NaOH with KHP involves adding NaOH from the burette to a known mass of KHP in solution. The data from the titration is then used to calculate the molarity of the NaOH.

Titrations as a technique

Today you will be performing two types of titrations:

1. **FAST**
2. **SLOW**

In a fast titration, the titrant (solution in the burette) is released quickly into the solution in the flask below, hence fast. The goal of this titration is **to determine the approximate volume of titrant needed to induce the change of color (determine the end point).** This titration is not quantitative; it will not give an accurate determination of the unknown concentration.

In contrast, a slow titration will give you an accurate determination of the unknown concentration; however, the titrant is released **very slowly into the solution in the flask below**. This is especially true as you near the end point. Remember, it is important to stop the titration at the appearance of a permanent pale pink color. This slow, methodical process will yield an accurate

measurement of volume of titrant.

Procedure

Record all your calculations, measurements, and observations into your notebook, and then fill in your Summary Sheet.

Part 1: Safety Video on Preparing for Emergencies

Please watch video #6 in the ACS lab safety series.

As you are watching, think of which emergencies could have happened in previous sessions already (with the materials and substances we have been using), and how we prepared for them. When you are done, answer the questions on the handout. Today's lab involves acids and bases. Listen carefully as your instructor explains the hazards associated with them.

Part 2: Standardization of the Sodium Hydroxide (NaOH) Solution

Preparing KHP Solution (in Flask)

1. Calculate the mass of KHP needed to prepare 25.00 mL of 0.100 M KHP and write this calculation in your notebook. Check your calculation with your instructor before moving to Step 2.
2. Weigh an amount of KHP as close as possible to the calculated mass in step 1 and record the exact mass of KHP measured.
3. Transfer the solid KHP into a 125-mL Erlenmeyer flask.
4. Add 25 mL of deionized water into the 125-mL Erlenmeyer flask containing the solid KHP.
5. Add one drop of **phenolphthalein** solution into the Erlenmeyer flask and swirl the flask to mix.

Preparing NaOH Solution (in Burette)

6. Calculate the volume of approximately 1.0 M aqueous sodium hydroxide solution needed to prepare 250-mL of an approximately 0.10 M aqueous sodium hydroxide solution. Write your calculations in your lab notebook. **Check your calculations with the lab instructor before proceeding to the next step.**
7. Measure the volume of 1.0M aqueous sodium hydroxide solution calculated in step 3 using the 25-mL graduated cylinder located in the fume hood. Pour this volume into your Florence flask and dilute the solution with enough deionized water to make a total volume of 250-mL. Remember this 0.1M is only an approximate concentration.
8. Label the flask with your name, date, lab section, and 0.1M aqueous sodium hydroxide (do not use chemical symbols) with a Sharpie.
9. Clamp a burette to the burette stand.
10. Condition the burette by filling with water. Allow the water to drain. Water can be discarded in sink. Rinse the burette with a few mL of NaOH solution and then discard this into the waste container. Then fill the burette slightly above the "zero" mark with NaOH solution.
11. Drain some of the NaOH solution so that there are no air bubbles in the tip of the burette AND so that the level of NaOH falls between 0.00mL and 1.00mL. Record the initial burette reading, which will NOT be at 0.00mL.

Fast titration (perform once)

12. Titrate NaOH into the KHP solution until a faint but permanent pale pink color is obtained. Make sure to swirl the Erlenmeyer flask thoroughly to ensure mixing of the reagents. In case any NaOH solution falls on the side of the Erlenmeyer flask, rinse the sides of the flask with deionized water. Record the volume of NaOH used to reach the endpoint.

Slow titration (perform three times)

13. Set up another fresh KHP solution in a clean Erlenmeyer flask with indicator (see steps 1-5).

14. Titrate with NaOH slowly. If the level of NaOH is close to 50 mL (i.e. bottom of the scale), write down the value, fill the burette with more diluted NaOH, and write down the new value. This allows you to calculate the total amount dispensed from the burette. Don't let the level of the NaOH go below the 50.00mL line on the burette or you won't be able to determine the volume dispensed and will need to start the titration over.

15. Remember to slow down to a drip at least 1-2 mL **before the recorded volume you observed in the fast titration**. Make sure to swirl the Erlenmeyer flask thoroughly to ensure mixing of the reagents. Stop when a sustained faint pink color is observed.

16. Repeat steps 13 – 15 to conduct three total slow titrations.

Part 3: Calculate the concentration of NaOH

17. Using the average data from the slow titrations, calculate the concentration of NaOH in units of mol/L.

18. Cover the remaining NaOH solution with parafilm and store your labeled Florence flask on the side counter.

19. Check with your instructor before cleaning up. Dispose all waste into appropriate waste disposal containers as instructed by your instructor. Clean and return all glassware. Place the burette upside down on the stand and turn the stop-cock to the open position to allow it to drain and dry.

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1.8: My Second Acid-Base Titration

Learning Outcomes

Upon completion of this lab, the student will be able to:

1. Perform another sodium hydroxide (NaOH) standardization titration to recheck its concentration
2. Use your standardized NaOH solution to perform a titration to determine the concentration of an unknown acid solution
3. Monitor the pH of a titration reaction

Introduction

Recall that a titration is an analytical quantitative technique used to determine the concentration of an acid or a base. Titrations play an important role in determining **amount** and **purity** in many manufacturing processes. These include food processing, textile, wood product manufacturing, petroleum, pharmaceuticals and chemical manufacturing. Titrations play a critical role in the manufacturing of biodiesel, water purification and waste water treatment and even in the production of various products in the dairy industry.

Experimental Design

Your solution of sodium hydroxide (NaOH) from the previous experiment whose concentration is known will be used for this experiment. You'll determine the exact concentration of a provided acid solution by titration with your standardized NaOH.

Fill in the Summary Sheet as you perform today's experiment.

Reagents and Supplies

Your standardized solution of sodium hydroxide from last experiment, a provided unknown acid solution, a phenolphthalein solution, pH paper

(See posted Safety Data Sheets)

Procedure

Part 1: Recheck NaOH concentration

1. Perform one standardization titration to check the concentration of your NaOH solution following the steps from the previous experiment, Experiment 6.

Part 2: Titrations to Determine Unknown Acid Concentration

Unknown Solution: Acid (Flask)

2. Add 25 mL of unknown acid solution into a 125 mL Erlenmeyer flask.
3. Add one drop of **phenolphthalein** solution into the 125 mL Erlenmeyer flask.

Known Solution: Sodium Hydroxide (NaOH)

3. Clamp a burette to a burette stand.

4. Condition the burette by filling with water. Allow the water to drain. Water can be discarded in sink. Then rinse the burette with a few mL of your standardized sodium hydroxide solution (NaOH) from last week. Discard this rinse into waste. Finally, fill to slightly above the zero mark with the NaOH.

5. Drain enough NaOH so that there are no air bubbles in the tip of the burette and that the level of the NaOH is between 0.00mL and 1.00mL. Record the exact initial burette reading with appropriate significant figures and record this value with units.

6. Perform a fast titration. Record the volume of NaOH used to reach the endpoint.

7. Repeat steps 3-6 to perform a total of two slow titrations. Record the volume of NaOH used to reach the endpoint.

Part 3: Final slow titration while monitoring pH

8. Perform a third (and final) slow titration as you did in the steps above, but monitor the pH of your solution as you do so by using pH paper. First measure and record the pH of the acid solution in your flask before you add NaOH (this is labeled as 0.00 mL NaOH added in the table on your summary worksheet). Then you'll record the volume of NaOH added and the pH of the solution measured at this point as you perform your titration. You'll want to plan on getting two measurements before the equivalence point, one measurement at the equivalence point, and two measurements after the equivalence point. Use these values to fill out the chart in your worksheet.

9. Check with your instructor before cleaning up. Dispose all waste into appropriate waste disposal containers as instructed by your instructor. Clean and return all glassware. Place the burette upside down on the stand and turn the stop-cock to the open position to allow it to drain and dry.

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1.9: Gas stoichiometry

Objectives

- Explore an acid / base reaction that produce CO_2
- Determine the molar mass of CO_2 using the ideal gas law
- Use stoichiometry to calculate actual and theoretical yield of product
- Evaluate and make suggestions to improve the experiment each trial
- Quantify the accuracy of your measurements

Safety and Notes

Handle the hydrochloric acid, HCl, carefully. It is a strong acid and can cause burns to skin and eyes.
Make sure you use the designated test tubes and wash bottles for this experiment, or you may get a test tube stuck in the bottle.

Introduction

Chemical reactions involving acids and bases are an important and ubiquitous type of chemical reaction. In this laboratory experiment, we'll investigate acid base reactions involving carbonates that generate carbon dioxide gas, CO_2. You may have seen this type of reaction in action in one of the favorite projects of science fairs: the baking soda volcano. To cause the "eruption", baking soda (sodium bicarbonate, which acts as the base) is mixed with vinegar (acetic acid) which causes bubbles of carbon dioxide to form and causes a foamy eruption. Check out this video for instructions to make a volcano. Acid base reactions are also important in the environment. One example acid base reaction involving carbonates can be seen by the deterioration of limestone statues and buildings (which contain calcium carbonate) exposed to acid rain over time. Washington D.C. has many examples of buildings and monuments that are damaged by acid rain, which is highlighted in this video.

YouTube Video

This interactive element is not available in this format. Visit the link or scan the QR code to access it online.

View this YouTube Video online

In this lab, you'll quantitatively determine the amount of carbon dioxide gas formed from this acid base reaction and use that to calculate an experimental molar mass of carbon dioxide. To "capture" the gas generated, you'll conduct the reaction in a closed wash bottle, and the gas generated will displace the water in the wash bottle. The experimental set-up is shown in the image below. The amount of water that is displaced will give the volume of the gas generated, V_{CO_2}.



There are three quantities we will use to describe how much carbon dioxide was generated: mass, volume and chemical amount. We can measure the mass *m* on a balance, in units of grams (g) or milligrams (mg). We can determine the volume of the gas V_{CO_2} by measuring the volume of displaced water. We don't have a direct way to measure the chemical amount *n*; here, we use the ideal gas law to figure out the chemical amount of carbon dioxide from its volume. The chemical amount uses units of moles (mol); when two samples have the same chemical amount, they contain the same number of particles. If you determine both the chemical amount and the mass in a specific sample, as you will in this experiment (i.e. how much you have in moles as well as grams), you can use those values to calculate the **molar mass** of a substance (units are grams per mole, g mol^{-1}). The molar mass of a substance is a value unique to the specific element or compound.

The acid base neutralization reaction we'll investigate is hydrochloric acid (HCl) with sodium bicarbonate ($NaHCO_3$). HCl is a monoprotic, strong acid. Monoprotic means it has one proton (H^+), and a strong acid means it completely dissociates so all the HCl molecules completely break apart into H^+ and Cl^- ions. The right pointing arrow in Reaction 1 indicates the complete dissociation of the HCl, which breaks down completely into H^+ and Cl^- ions. Reaction 2 shows the balanced acid base neutralization reaction. Notice how each reactant and product is in a 1:1 mole for this particular reaction. This means that if you have 5 moles of $NaHCO_3$, you will form 5 moles of CO_2 gas, provided there is an excess of HCl. The molar ratios given in your balanced equation are always important in stoichiometry calculations.

$$\mathrm{HCl\,(aq)} \longrightarrow \mathrm{H^+\,(aq)} + \mathrm{Cl^-\,(aq)} \qquad (\mathrm{Reaction 1})$$

$$\mathrm{HCl\,(aq)} + \mathrm{NaHCO_3\,(aq)} \longrightarrow \mathrm{H_2O\,(l)} + \mathrm{NaCl\,(aq)} + \mathrm{CO_2\,(g)} \qquad (\mathrm{Reaction 2})$$

While collecting and analyzing your experimental data, you can focus on achieving good accuracy and precision. Recall that good accuracy means that your measurements are close to the "true" or "theoretical" value. Good precision means that you have multiple measurements that are close to each other. We can perform different calculations to quantify the accuracy and precision of measurements. To determine accuracy, the percent error is a simple calculation that is commonly used, shown in Equation 1. The "actual" value is the amount you calculate based on your experimental measurements. The "theoretical" value is the "true" value, which in this case is a known value that you can look up in a reputable reference.

Equation 1 – percent error

$$\frac{\text{actual} - \text{theoretical}}{\text{actual}} * 100 \qquad (1.9.1)$$

To express the precision, you must have more than one measurement so you can compare how close they are to each other. You can calculate a standard deviation and then relative standard deviation to express your precision as a percent, shown in Equations 3 and 4. You can do these calculations by hand, or you could use Excel or Google Sheets and utilize the built-in calculations for these statistics.

Equation 2 – average, $\overline{x}$

$$\overline{x} = \frac{1}{n}\sum_{i=1}^{n} x_i$$

Equation 3 – standard deviation, s

$$s = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n} (x_i - \overline{x})^2}$$

Equation 4 – relative standard deviation, RSD

$$\text{RSD} = \frac{s}{\bar{x}} * 100\%$$

You want to work carefully to obtain good accuracy and precision, but chemical reactions in practice are a bit more complicated than how they look on paper, so there are always physical limitations beyond your control that limit how much product can form. There are also limitations in the equipment you use that contribute to uncertainty in your measurements. In addition, poor experimental techniques or mistakes will also decrease the accuracy and precision you obtain. In this lab, you'll conduct the experiment multiple times and improve your procedure and techniques each trial.

Experimental Procedure

Record observations and calculations in your lab notebook and on this summary sheet you will hand in at the end of class.

Part 1: Experimentally Determine the Molar Mass of Carbon Dioxide

1. Look up the molar mass of carbon dioxide and record this value as the "known" molar mass value (g/mol). In this procedure, you'll follow a method to measure the values needed to experimentally determine the molar mass value.
2. First prepare your wash bottle, which will act as your experimental set-up. You may refer to the picture in the introduction as an example. Fill the wash bottle with tap water up to the fill line on the bottle. To get the water level to the fill line, it might be easier to overfill the bottle and then squirt out the excess. Place the wash bottle on the stir plate. Place a 150 or 250 mL beaker below the spout of the wash bottle and adjust to catch the displaced liquid.
3. Next prepare your test tube that will contain the acid base chemical reaction that will generate $\mathrm{CO_2}$. Weigh an empty gelatin capsule and record the mass. Fill the capsule with less than 0.500 g of sodium bicarbonate and weigh and record the exact mass of the full capsule. You can find the mass of just the sodium bicarbonate by the difference of the full and empty capsule to make sure you are under 0.500 g.
4. Into a 6-inch test tube, put in a small stir bar and add 7-10 mL 4 M HCl. Weigh the full test tube and record its mass. To weigh the test tube, you may want to tare a beaker on the scale and then put the test tube in the beaker to get its mass. Then place the test tube upright into the wash bottle.
5. To start the reaction, drop the filled gelatin capsule into the test tube containing the acid. Working quickly but carefully, use a glass stir rod to poke the capsule to ensure it is not stuck on the test tube wall and that it is sitting in the acid. Close the lid of the wash bottle tightly. It will take a minute or so, but the acid will dissolve the gelatin capsule and you'll see the reaction between the acid and the bicarbonate. The carbon dioxide generated will displace the water in the wash bottle. The water will be displaced quickly at first, so be careful to capture all the water that is displaced from the reaction. Once the reaction appears complete (the water stops dripping from the wash bottle), slowly turn on the stirring. You want to ensure that the capsule or sodium bicarbonate did not get stuck to the inside of the test tube. Stir for about 5 minutes or until the reaction is complete, the capsule and sodium bicarbonate are totally dissolved, and no more water is displaced.
6. Move the beaker containing the displaced water away from the wash bottle. Open the lid of the wash bottle just enough to insert a thermometer into the top of the wash bottle where the generated carbon dioxide is and record the temperature ($T_{\mathrm{CO_2}}$).
7. Carefully remove the test tube from the wash bottle. Dry off the water from the outside of the test tube. Weigh the test tube which now contains any leftover reactants and the aqueous products and record the mass. From the initial mass and this final mass, calculate the mass of $\mathrm{CO_2}$ formed.
8. Measure the volume of the displaced water in the beaker by pouring it carefully into a large graduated cylinder. Pay attention to the markings on the graduated cylinder. If the total volume of water is more than the max volume in the graduated cylinder, you'll have to measure it in increments and sum up the measurements. The volume of the displaced water is the same as the volume of the carbon dioxide that was formed ($V_{\mathrm{CO_2}}$).
9. Record the atmospheric pressure in the laboratory. Since the apparatus you set up is open to the atmosphere, the atmospheric pressure is the same as the pressure of the carbon dioxide ($P_{\mathrm{CO_2}}$).
10. Complete all your observations and calculations for Trial 1 before moving on.
11. Repeat this entire procedure in Trial 2 to get a second calculated value for molar mass of $\mathrm{CO_2}$.
12. Clean up by cleaning and returning your stir bar. Be careful not to drop the stir bar in the sink. Dispose of all hazardous waste in the labeled container in the hood. Clean all other glassware and equipment used and return it or put it away. Wipe off and dry the counter. Wash your hands before leaving the lab.

Acknowledgement

Parts of this lab were adopted from Nayasulu, F., Paris, S., and Barlag, R. Wash Bottle Laboratory Exercises: Mass of $\mathrm{NaHCO_3}$ in an Alka-Seltzer Tablet, Molar Mass of $\mathrm{CO_2}$, and the Ideal Gas Law Constant. J Chem Ed. 2009, 86, 842-844.

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1.10: Calorimetry lab

Calorimetry

Calorimetry is a method used to measure the heat transfer between a system and its environment. For chemical reactions, we can use calorimetry to determine the enthalpy of reaction. To remind you, endothermic reactions gain energy from the surroundings (ΔH is positive) and exothermic reactions give energy to the surroundings (ΔH is negative). A calorimeter is a device used in the measurement. Ideally, it is well insulated so that the contents of the calorimeter don't exchange heat with the outside. We will design and build a calorimeter to replace our old Styrofoam cup ones. While Styrofoam is an excellent insulator, there is no secure recycling stream for it. Coffee and fast food places have stopped using them, instead offering recyclable multi-walled paper containers for their hot beverages or a discount for bringing your own thermos.

Please take good notes in your lab notebook, and summarize your results and thoughts on this summary sheet, which you will hand in before you clean up your bench and leave the lab.

Safety and hazardous waste

This lab uses sodium hydroxide solution (in task 5). You have to wear your splash goggles at all times (someone on the other side of the bench might spill while you are just doing some calculations). When someone gets sodium hydroxide in their eyes, we stop lab and the individual has to rinse their eyes for 15 minutes in the eye wash before visiting an eye doctor. We are not generating any hazardous waste today because the product of our reaction is sodium acetate. However, we have to carefully collect any leftover acetic acid and sodium hydroxide solutions.

Part 1. Warmup: dissolve some salts to experience exo- and endothermic

Source: https://www.middleschoolchemistry.co...apter5/lesson9

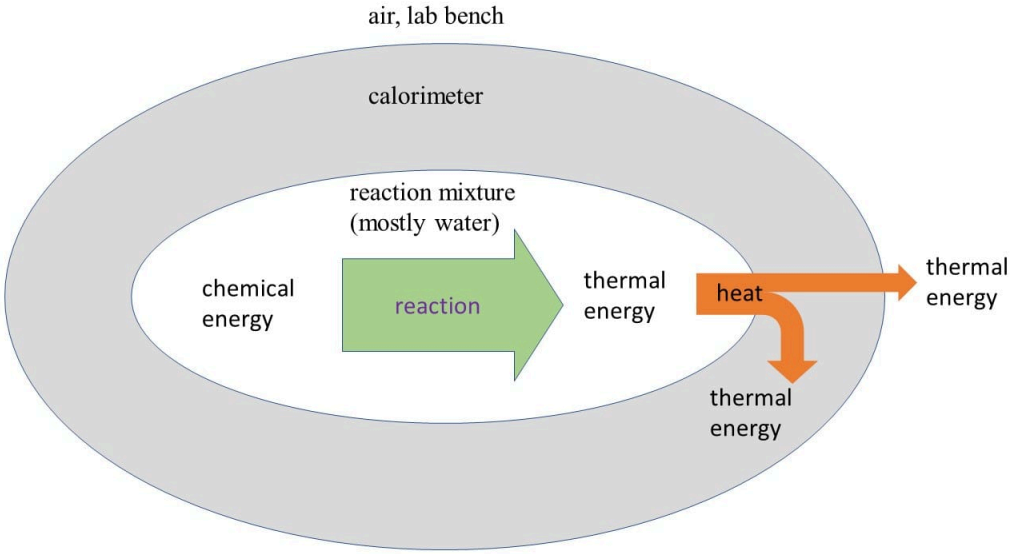
Take a plastic cup and fill it with 10 mL water kept at room temperature (not from the tap, which typically is colder). Briefly touch the bottom of the cup to get a sense of the temperature. Also, measure the temperature with your thermometer. Take two gram of $\mathrm{NaNO_3}$ **or** $\mathrm{CaCl_2}$ and dissolve it in the water. Once everything is dissolved, check the temperature of the cup again (with your hand touching the bottom of the cup, and with the thermometer). Could you use the substance in a heat pack or in a cold pack (in combination with water). Is your dissolution reaction exothermic or endothermic? Check in with others at your bench about the substance you did not choose.

1. $\mathrm{NaNO_3\,(s) \longrightarrow Na^+\,(aq) + NO_3{}^-\,(aq)}$
2. $\mathrm{CaCl_2\,(s) \longrightarrow Ca^{2+}\,(aq) + 2\,Cl^-\,(aq)}$

Part 2. Engineering task: Insulate a cup to use as calorimeter

The role of insulation

In the previous task, you experienced endothermic and exothermic reactions in a qualitative way. It was easy to feel changes in temperature on the outside of the cup because it was not insulated. For a quantitative measurement of the reaction enthalpy, we need to minimize heat transfer from the solution to the container and the rest of the lab. The diagram below shows energy transfer for an exothermic reaction (for endothermic, reverse all arrows); we want to make the orange heat transfers as small as possible so we can neglect them when measuring reaction enthalpies.



The way we set this up[1], all of the chemical energy from the reaction will initially be transformed into thermal energy of the reaction mixture. However, once the temperature of the reaction mixture is different than room temperature, there is also some heat exchange from the reaction mixture to the container, and from the container to the air and lab bench. If we wait too long, the temperature of the reaction mixture will be back to room temperature.

[1]constant pressure, no work except for pressure/volume work (GenChem 2 topics)

The task and the engineering cycle

Calorimeters are thermally insulated to minimize the heat exchange (orange arrows). Your task is to build a calorimeter from cups of different sizes, different insulation material, different lids and other material. **Each design has to have a 9 oz plastic cup securely placed inside the contraption at a height that allows magnetic stirring and covering it with the provided foam lid.** Apart from those constraints, you can be creative with the design. It would help with cleaning and drying if it is easy to take apart and reassemble the components.

Here are some hints to get you started. The heat capacity of the calorimeter is related to its total mass (a synonym for heat capacity is “thermal mass”). The most efficient insulation (apart from a vacuum layer) is air confined to pockets; foam insulation is based on this. The thicker the insulation, the more effective it is, but the heat capacity and the costs will increase. Lids that fit snugly into the 9 oz plastic cup with a hole for the thermometer are available.

We will go through two rounds of an engineering cycle, i.e. designing, building and testing two prototypes.You will design and build prototype 1 on your own. Then, you will test it and compare it to the one your partner built. From what you learned, you will design and build prototype 2 in consultation with your partner.

The tests are crucial, otherwise you don't know which prototype is the best. You will test your prototype 1 by adding hot water into the cup and monitoring the temperature with your hand for a couple of minutes. This is a qualitative test because our sense of temperature is relative and rough. You get a sense of how well the cup is insulated, but are not measuring the heat capacity. For prototype 2, we will do some quantitative tests.

Some background that might help with the design

It might help to know a bit about how heat transfer works if we want to minimize it. Taking a thermos container, a Styrofoam cup and a double-glazed window as our examples, we can discuss the three mechanisms of heat transfer:

1. **Conduction**: The basic idea is that atoms collide, transferring kinetic energy. If one side of a piece of glass is hot and the other side cold, the atoms on the hot side will have higher average kinetic energy than the atoms on the cold side, so on average thermal energy flows from the hot to the cold side. Both Styrofoam cup and double-glazed window have air gaps. Air,

with its low density and large distance between atoms, is a bad conductor. Metals like copper are very good heat conductors. In the thermos container, there is a vacuum gap which has zero conductivity.
2. **Convection**: If part of the insulating material is in the liquid or the vapor state, atoms can move over large distances. Because the density of fluids depends on their temperature (and there is gravity), we get what is called convection flow, with low density material rising and high density material falling, mixing in the process. There is no convection in the vacuum gap of the thermos, and convection is very limited in the foamy structure of Styrofoam. However, the double-glazed window has a lot of convection unless the air-gap is sufficiently narrow.
3. **Radiation**: All samples emit electro-magnetic radiation from their surfaces. At the temperatures we usually encounter, this radiation is in the infra-red range that our eyes don't see. If you go to very high temperatures, the radiation reaches into the visible range, as we "see" in an incandescent light bulb, red-hot metal or glowing embers. You can experience infra-red radiation next to a grill or a bonfire. For the thermos container, radiation across the vacuum gap is minimized by the silvery surface coating. In the Styrofoam cup, the solid walls of the foam prevent radiation across long distances. A double-glazed window lets a lot of radiation through, as you can experience on a cold, sunny day. In general, if the material is translucent to visible light, it might also be translucent to infra-red radiation.

So why don't we just use a thermos container as calorimeter? It seems to have good strategies to prevent conduction, convection and radiation. There is a safety problem (if the thermos shatters, it implodes) and it has a high heat capacity (the walls have to be thick to withstand the vacuum). In general, the heavier the insulating container, the higher heat capacity it tends to have. This is why in our design process, we are monitoring the total mass and are aiming to keep it low while minimizing conduction, convection and radiation.

Part 3. Measure the calorimeter's mass and test its heat capacity and rate of heat loss

Once you have prototype 2 (or the time is up), you will do a quantitative test. The better the insulation and the lower the heat capacity of the empty calorimeter, the better the design. The insulation will determine how fast or how slow the hot water cools down. The heat capacity will determine how much the hot water cools down the instant you add it to the calorimeter, i.e. the highest temperature you observe (which will always be lower than the initial temperature of the hot water before you pour it in).

The test will be done in pairs so you have enough hands. Ensure that a magnetic stir bar turns inside the cup (no need for liquid). Let the calorimeter sit for one minute so it reaches room temperature (when you touch it, it will get warmer). Insert the thermometer into the lid and place it on the calorimeter. Once these preparations are done:

1. Record the temperature of the thermostated water bath with your own thermometer (they are all calibrated slightly differently but are good at determining temperature differences). Dry the tip of the thermometer and place it back into the lid of your calorimeter (i.e. the circular foam piece).
2. Transfer 100 mL of water from the thermostated water bath into the calorimeter cup, using the ladle in the thermostat (it is in there so it is at the same temperature and does not cool down the hot water).
3. Put the lid with the thermometer on and read off the temperature when it is reasonably stable (your partner should close the thermostated water bath so it does not cool down too much).
4. Bring the calorimeter back to your bench, place it on the magnetic stirrer, and record temperature readings every 30 sec for 5 minutes (10 readings). While you read off the temperatures, your partner should call out the time and write down the measurements.
5. Open the calorimeter, check once more that stirring is efficient, remove the water and let the cup dry and cool down.

Plot your measurements (temperature against time), marking off the initial temperature (of the thermostated water bath) on the temperature axis, and fitting a straight line to the last six data points. Record the maximum temperature and the slope of your fitted line in the result sheet. Repeat for the second design.

Part 4. Contest

4.1 Choose your bench's champion

Check out the other designs at your bench (there should be 5-6), and compare the initial temperature drop and the slope of the temperature curve (reflecting heat loss to the outside). Together, these tell you about the heat capacity and the quality of the insulation. Choose one of the designs to compete against the other two benches, and give it a name to enter in the competition.

If looking at the different designs gives you ideas for improvements, you can decide as a group to submit a modified version of your best design (this might improve it or not, you don't know yet because you did not test it yet, so this is a risky move).

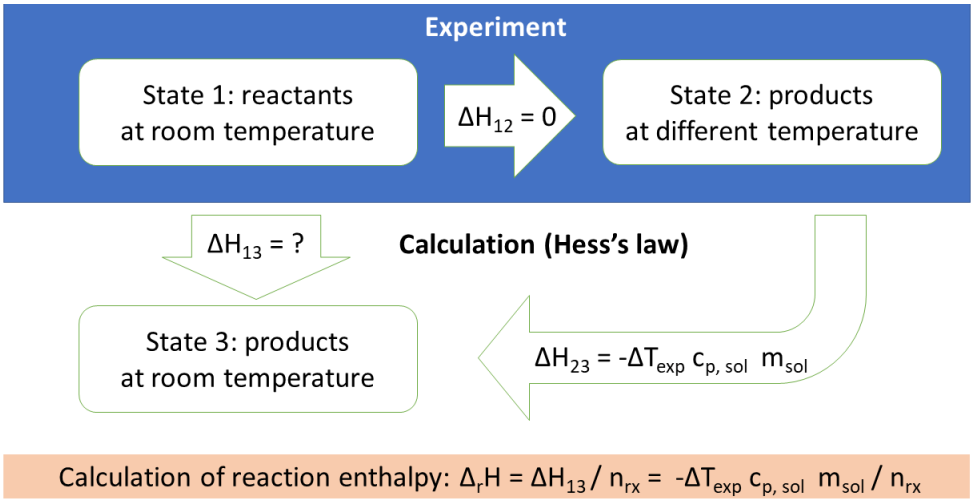
4.2 Test the three models (and find the biggest leaks)

As a group experiment, we will use an infrared thermometer to find "warm spots" on the calorimeters where most heat transfer occurs (this could be the lid, the bottom, or parts of the wall). Then, we will discuss which of the three models we would recommend using for the second semester, where we will need a calorimeter again.

Part 5. Measure the reaction enthalpy of the neutralization of acetic acid and NaOH

This will be done in pairs, using one of your own prototype 2 calorimeter. Place 50 mL 1 M of NaOH into your calorimeter cup and start stirring. Carefully (and wearing safety splash googles, of course), add 50 mL of 1 M acetic acid and close the lid. Record the temperature every 30 sec for 5 minutes.

We will estimate the reaction enthalpy of the reaction at room temperature. However, we started with reactants at room temperature (state 1) and ended with products at a different temperature (state 2). To make a statement about products at room temperature (state 3), we will use Hess's law, which says that changes in enthalpy do not depend on the path. We want to go from state 1 to state 3. We know the enthalpy change from state 1 to state 2 (it is zero, can you explain why). We also know that state 2 and state 3 only differ in temperature.



We will estimate the heat capacity of the calorimeter C_p as

$$C_p = m_{\text{solution}} \times c_{\text{p,solution}} = m_{\text{solution}} \times 3.9 \text{ J/(K g)} \tag{1.10.1}$$

and the reaction enthalpy as

$$\Delta_r H = -\Delta T \frac{m_{\text{solution}} \times c_{\text{p,solution}}}{n_{\text{reaction}}} \tag{1.10.2}$$

For n_{reaction}, use the chemical amount of either acid or base (don't add them up). ΔT is the difference between final temperature and initial temperature (i.e. should be positive for exothermic reactions).



A more detailed explanations of the calculation is here. Also, you can study this sample calculation.

Part 6. Which calorimeter would you recommend for next semester?

In the summary lab sheet, make a recommendation for a starting design for next semester. Criteria should include sustainability, cost, ease of use and quality of results.

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1.11: Atomic Spectra

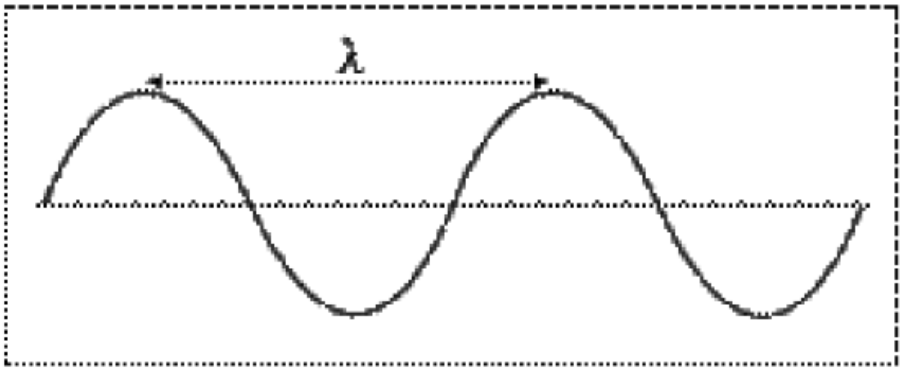
Please enter your observations and thoughts into this summary sheet, which you will hand in at the end of lab.

Objectives

- To perform flame tests of metal cations in order to observe their characteristic colors,
- To perform calculations to determine the frequency and energy of the emitted photons.
- To relate these results to the types of electronic transitions occurring in these elements.
- To observe and understand line emission spectra of atoms using a computer simulation and gas-discharge tubes.

Introduction

Electromagnetic radiation is energy in the form of waves. Waves are characterized by their *wavelength* (λ) and *frequency* (ν). Wavelength is defined as the distance between successive crests (or troughs) on a wave, and is measured in meters. Frequency is defined as the number of waves that pass a given point every second, and is measured in 1/seconds (1/s or s^{-1}), or Hertz (Hz).



All electromagnetic waves travel at the *speed of light* (c), or $3.0 \times 10^8 m/s$. The relationship between the wavelength, frequency and speed of a wave is given by the equation:

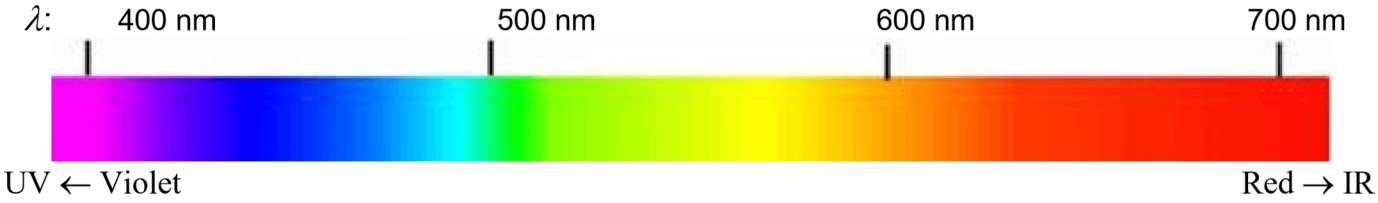
$$c = \lambda \times \nu \tag{1.11.1}$$

Electromagnetic radiation also occurs as discreet "packets" called *photons*. The *energy* (E) of a photon (in Joules, J) is given by the equation:

$$E_{photon} = h \times \nu \tag{1.11.2}$$

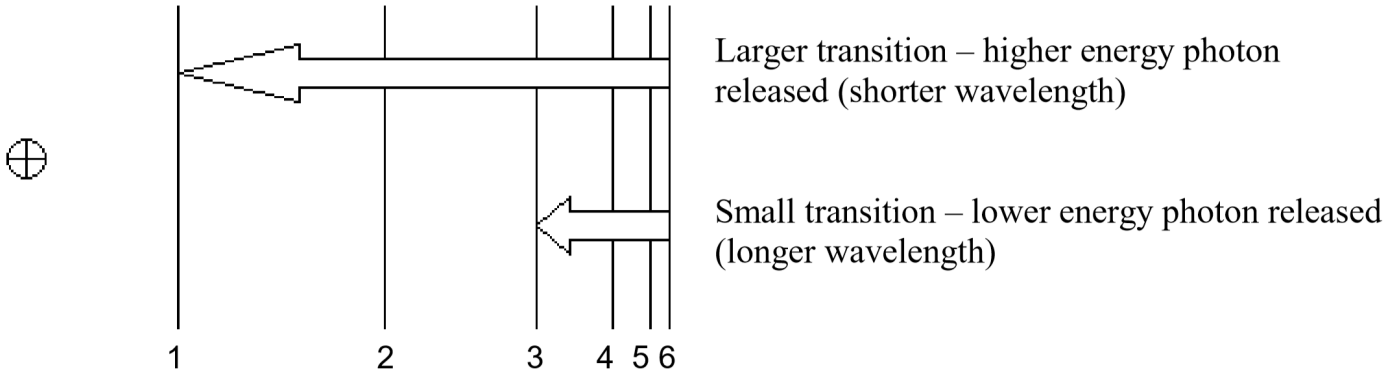
Here, h is *Planck's constant*, which has a value of $6.63 \times 10^{-34} J \cdot s$.

Visible light is the most familiar example of electromagnetic radiation. Differences in the wavelengths of visible light are manifested as different colors, shown in the color spectrum below (colors can be seen in the PDF document on-line). Other examples of electromagnetic radiation include X-rays, ultraviolet light, infrared light, microwaves and radio waves.

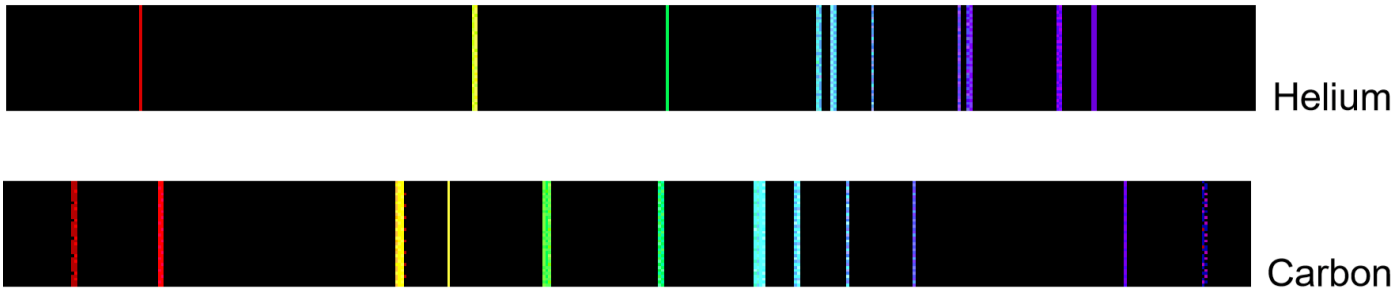


So, how does electromagnetic radiation relate to flame tests? Well, when an atom or ion absorbs energy, its electrons can make transitions from lower energy levels to higher energy levels. The energy absorbed could be in the form of heat (as in flame tests), or electrical energy, or electromagnetic radiation. However, when electrons subsequently return from higher energy levels to lower energy levels, energy is released predominantly in the form of *electromagnetic radiation*.

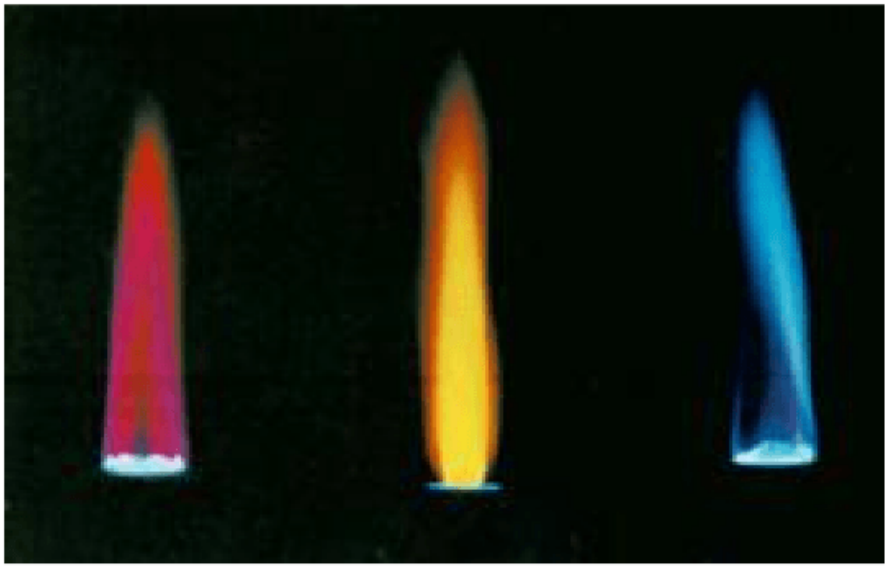
The spacing between energy levels in an atom determines the sizes of the transitions that occur, and thus the energy and wavelengths of the collection of photons emitted.



If emitted photons are in the visible region of the spectrum, they may be perceived as lines of different colors (note that photons outside the visible spectrum may also be emitted, but cannot be seen). The result is called a *line emission spectrum*, and can serve as a 'fingerprint' of the element to which the atoms belong. For example, the line spectra shown below for the elements helium and carbon are clearly quite different.



Unfortunately, techniques more sophisticated than those used in this lab are required to obtain such line spectra. To the naked eye, when an element is vaporized in a flame (or an electrical discharge)the emission spectrum will appear to be just *one color*. For example, helium gas when excited by an electrical discharge emits light that appears an orange- peach color. This one color results from a *combination of all lines* of the emission spectrum, in proportion to their intensities. As many elements will still produce distinctive colors under such conditions, simple flame tests can be used to identify these elements. In fact, flame tests were used to identify elements long before the invention of modern techniques, such as emission spectroscopy.



Procedure

Materials and Equipment

Looped platinum or nichrome wires, wash bottle with distilled water, Bunsen burner, and the following solutions: NaCl (*aq*), KCl (*aq*), $\mathrm{CuCl_2}$ (*aq*), and $\mathrm{CaCl_2}$ (*aq*). Gas discharge tubes for hydrogen, helium, and mercury. A handheld spectroscope.

Before lab, watch this video on how to use the spectroscope.

Safety

Exercise appropriate caution when using the Bunsen burner.

Part 1: Flame Tests of Metal Cations

Your instructor will dip a looped platinum wire into a solution containing a metal salt, and then hold it in the Bunsen burner flame. You will record your observations in your worksheet.

Part 2: Bohr Model of an Atom Simulation

1. Using a computer or a mobile device, access the "Models of the Hydrogen Atom" simulation from phet.colorado.edu. Set the dial to “Prediction” and select Bohr's Atomic Model. Click the box to set the Light Control to “White”. Turn on the gun to shoot photons (“packets of light” or “energy bundles”) by clicking the red button. Each photon corresponds to that color dot with fuzzy purple dots representing UV. Check the box to show the spectrometer. Also check the box to show the electron energy level diagram. You can adjust the speed if its helpful.
2. Recall that a neutral hydrogen only has one electron, which is represented by the blue dot rotating around the energy levels of the atom in this simulation. Observe what happens with the photons when the electron jumps to a higher energy level. Observe what happens when the electron relaxes back down to a lower energy level.
3. Now increase the speed of the simulation in order to fill in the signal collected by the spectrometer and observe the results.

Part 3: Line Emission Spectra

1. Gas-discharge tubes for hydrogen, helium and mercury are set up on the back lab bench. Go to any available station and use the hand-held spectroscope.
2. Align the slit on the wider end so that you can see the light through the view window on the opposite end. It should appear as a clear, vertical line, and result in several colored lines appearing in the view. You should also be able to see the nm markings

(make sure it is not upside-down) below the colored lines in the view.
3. Record the spectral lines in your worksheet, indicating colors in their specific locations.

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- 5: Flame Tests and Atomic Spectra (Experiment) by Santa Monica College is licensed CC BY-NC 4.0.

1.12: Molecular modeling - Computational chemistry

Introduction

> *The general theory of quantum mechanics is now almost complete... The underlying physical laws necessary for the mathematical theory of a large part of physics and the whole of chemistry are thus completely known, and the difficulty is only that exact application of these laws leads to equations much too complicated to be soluble. It therefore becomes desirable that approximate practical methods of applying quantum mechanics should be developed.*
>
> —P. A. M. Dirac, 1929

An Introduction to Computational Chemistry

The quantum mechanical calculations utilized in this exercise are beyond the scope of General Chemistry as the concepts require a thorough understanding of calculus and differential equations. If you are interested, these topics are covered in Physical Chemistry. That being said, a general idea of inner workings (excluding as much complex math as possible) will be detailed herein.

Quantum chemistry is centered on the Schrödinger equation:

$E\Psi = \hat{H}\Psi$ [1]

, where Ψ is the wavefunction, E is the energy of the system, and $\hat{H}$ is the Hamiltonian operator.

You might be asking yourself, "What is this wavefunction thing?" A wavefunction is a mathematical function to describe the wave-like behavior of an electron. The wavefunction itself does not describe anything physical, but if you multiply the wavefunction by its complex conjugate (Ψ^*):

$\Psi^*\Psi = |\Psi^2|$ [2]

, then you get the electron probability density (ρ), which is an observable property. However, the Schrödinger equation can currently only be solved exactly for one electron systems, such as the hydrogen atom. So what good is this model if we can only look at one electron systems? The answer is not much, so we need to make additional approximations to move beyond one electron systems.

An example of one of these approximations is the Linear Combinations of Atomic Orbitals (LCAO). In this approximation, basis functions (φ) are used in the construction of molecular orbitals (MOs). MOs are used to describe the electronic structure of a molecule whereas atomic orbitals (AOs) describe the electronic structure of an atom. The mathematical form of the LCAO approximation is given by:

$\psi_{mo} = \sum c_i \varphi_i$ [3]

,where ψ_{mo} is the wavefunction of the molecular orbital, φ_i represents the atomic orbital wavefunctions, and the c_i terms are the "percentages" for each atomic orbital. In an H_2 molecule, the form of the σ bonding MO would look like the following:

$\psi_+ = c_a \varphi_a + c_b \varphi_b$ [4]

, where φ_a is the atomic orbital on "hydrogen a", φ_b is the atomic orbital on "hydrogen b", and c_a/c_b are the contributions of atomic orbitals to the overall MO. If we use a minimal basis set then we know that the atomic orbitals for each hydrogen can be represented by a 1s orbital, and the fact that each AO of the hydrogen contributes equally in this so-called pure covalent system, then the H_2 σ bonding MO could be expressed by:

$\psi_+ = \frac{1}{\sqrt{2}}[\varphi_{a,1s} + \varphi_{b,1s}]$ [5]

Depending on the approximations made one arrives at different ways to model quantum chemical systems. Despite the large number of approximations that go into these different models, they can get within "chemical accuracy" if properly used. Some approximations include Hartree-Fock (HF) Theory, Density Functional Theory (DFT), Møller-Plesset (MP) models, Coupled-Cluster (CC), and configuration interaction (CI). Each model has its limitations and users should be aware of those limitations.

Some terminology:

Level of Theory – This is the theoretical model selected for the calculation. This includes HF, DFT, etc.

Basis Sets/Functions – A basis set is a mathematical function used in the construction of the molecular orbitals.

Practical quantum chemistry (what an oxymoron, right?) is a balance between calculation accuracy and computational expense (time). A quality quantum chemist selects the level of theory, along with the basis sets, to get a reasonable result in a reasonable amount of time. Additionally, knowing the "meaningful" digits from the results of these calculations is important.[4]

In this exercise you will be conducting quantum chemical calculations to make predictions about macroscopic properties, such as miscibility.

References:

[1] Engel, T.; Reid, P. *Physical Chemistry*, 3rd Edition. Pearson: Boston, 2013.

[2] Cooksy, A. *Physical Chemistry Quantum Chemistry and Molecular Interactions*. Pearson: Boston, 2014.

[3] Levine, I.N. *Quantum Chemistry*, 7th Ed. Pearson: Boston, 2014.

[4] Hoffmann, R.; Schleyer, P.R.; Schaefer, H.F. Predicting Molecules-More Realism, Please. *Angew. Chem. Int. Ed.*, **2008**, *47*, 7164-7167.

Procedure

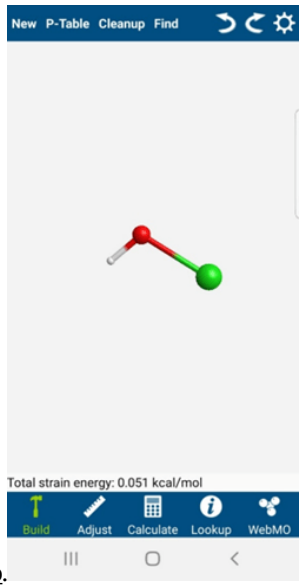
Part 1 – Which is the lowest-energy structure?

You will use the free version of WebMO, a web-based interface to perform quantum chemical calculations to investigate the energy and the equilibrium geometry of hypochlorous acid (HClO) to confirm which geometry is the lowest in energy structure. You'll consider the calculated "heat of formation," or the amount of heat that is either absorbed or released as the molecule forms from its elements. If the heat of formation is negative, then heat is released. If the heat of formation is positive, then heat is absorbed. The isomer that has a lower heat of formation (more negative) will be energetically favored and more stable than the isomer with higher heats of formation.

Computational Procedure:

You will use the program to determine the best geometry for each molecule (the best geometry will produce a minimum in the heat of formation). **After drawing the VSEPR model for the isomers of hypochlorous acid on the summary sheet**, continue with the procedure below.

You can use the "WebMO" app on your smart phone for this computational experiment.



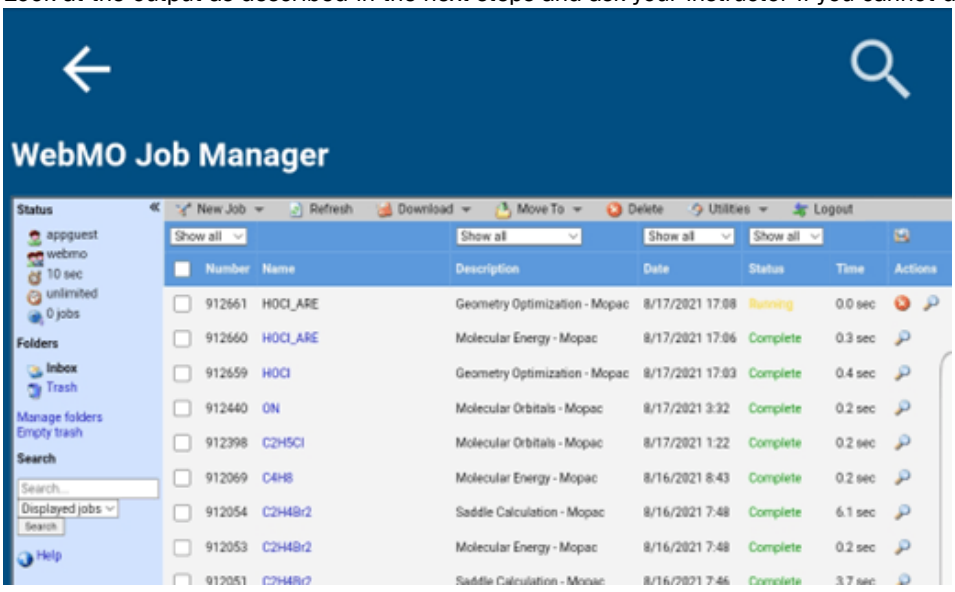
1. You will be “drawing” a representation of the HOCl molecule in the **WebMO app**.
    - To select the element for the first atom, click on “P-Table” menu item. A periodic table will appear. Pick an element, like oxygen for example.
    - The periodic table will disappear and you will be back in the editor window. Click anywhere in the white area and a red sphere will appear. This red sphere represents an oxygen atom.
    - Now you are ready to add the hydrogen atom. Bring up the periodic table again and select hydrogen. To add a hydrogen atom that is bonded to the oxygen atom, click-drag (place the cursor on the oxygen, left-click and drag away from the oxygen atom while still holding the mouse button down). The hydrogen atom will be white. Repeat for the chlorine atom, which will be green.
    - If you need to add a double bond, simply click on one atom and then drag to the nearby atom where you want to add the double bond.
    - If you need to undo a step, the left pointing curved arrow at the top Chart, line chart Description automatically generated will undo your last action.
    - After building a molecule, perform an initial geometry optimization by choosing Clean-Up → Geometry before the next step.
2. After optimizing the geometry of your molecule, click on the “WebMO” icon on the bottom tool bar.



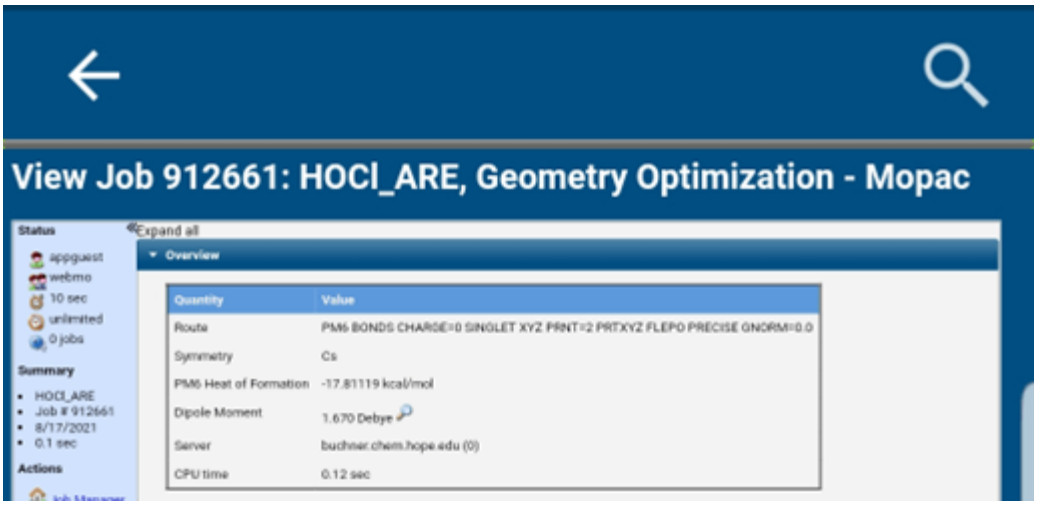
Then click “Submit” on the top tool bar. Now you will be at the “Configure Mopac Job Options” page. Supply unique a job name that includes your initials that will help you identify the calculation. An example Job Name can be seen in the figure below. There should be no commas, symbols, parentheses, and/or spaces for the job name. Make sure the other settings match those in the example below (You will most likely have to change the **Calculation** to “**Geometry Optimization**”).

Click the right pointing arrow in the bottom right of the screen to submit it.

3. Once the job is submitted, you will then be redirected back to the job manager page. The calculation should take less than one second, but it may take as much as 30 seconds for the queuing software to update the job status. Once the job status changes to a green “Complete” the calculation is finished. If the status is listed as a yellow “Running”, do not panic! The job is just waiting in line to make it to the computer cluster. If the status is listed as a red “Failed” then something went wrong. Look at the output as described in the next steps and ask your instructor if you cannot determine the issue.



4. After the job completes, open the output by clicking the small magnifying glass icon to the right of your submitted job. A graphical representation of the molecule will be shown in the “Molecular Viewer” window. Now click on “Results” on the top tool bar. Record the Heat of Formation value, in kcal/mol, in Table A-1.



Part 2 – Predicting Polarity and Building 3D chemical compounds

In this part of the exercise, you will need to:

- Use VSEPR to draw the Lewis structures of the compounds listed in the table in the worksheet.
- Use WebMO to calculate the dipole moment for these compounds.

Computational Procedure:

1. Draw the Lewis structures for the compounds in the table. Before moving on to modeling them in WebMO, ask your instructor to check your Lewis Structures.
2. Using the procedure from Task 1, perform the quantum chemical calculations for the ground state geometry for the compounds listed in the table.
3. From your calculations, find the calculated dipole moment in Debye for each compound and record in the table.

Drawing Lewis Structures (a brief review):

1. Arrange atoms: when there is a central atom, it is usually the least electronegative. Hint: chemists usually list the central atom first in chemical formulas (e.g., CCl_4 and CO_3^{2-} both have C as the central atom).
2. Determine total number of valence electrons in molecule or ion
3. Draw covalent bonds between atoms: Remember a covalent bond is two shared electrons
4. Beginning with terminal atoms, add enough electrons to each atom to give each atom an octet (or duet for hydrogen)
5. If any electrons are left over, place them on the central atom: Hint: some atoms may break the octet rule!
6. If the central atom has fewer electrons than an octet, form multiple (double or triple) bonds to the central atom to achieve an octet: Remember to keep track of the number of electrons you use!

This procedure was adapted from *Simpson, Scott; Evanoski-Cole, Ashley; Gast, Kellie; Wedvik, Madeleine; Schneider, Patrick; and Klingensmith, Isaac. (2020). Quantum chemical exercise linking computational chemistry to general chemistry topics. Chemistry Teacher International. 3. 10.1515/cti-2019-0014.*

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2: GENERAL CHEMISTRY 2 (CHEM0111)

2.1: Paper chromatography

2.2: Freezing Point Depression

2.3: Synthesis and Recrystallization of Aspirin

2.4: Acetone Iodine kinetics

2.5: Equilibrium and Le Chatelier's Principle

2.6: Determination of Kc for a Complex Ion Formation

2.7: Group I cations

2.8: pH measurement and determination of pKa value

2.9: Making a buffer with three components

2.10: Colorimetric pH measurement

2.11: Hypothesis testing

2.12: Preventing rust

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2.1: Paper chromatography

Objectives

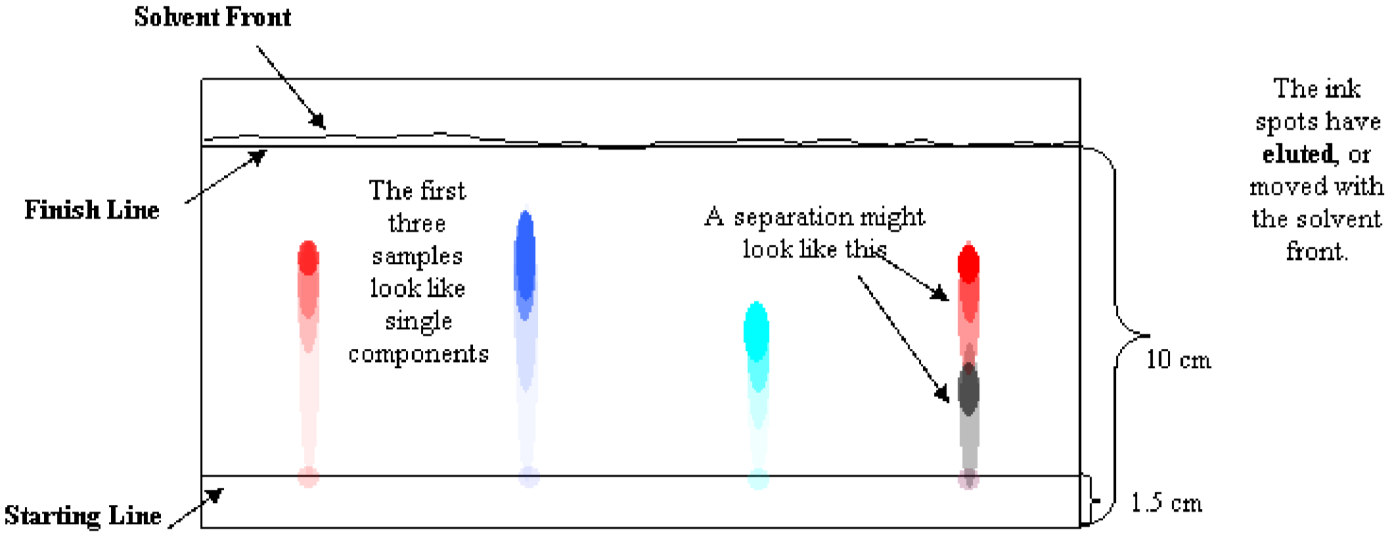
- To use paper chromatography to identify whether certain food dyes are pure substances or mixtures.
- To obtain a paper chromatogram of various food dyes
- To identify components of food dyes by calculating R_f values

Chromatography is a method of physically separating *mixtures* into its individual components. It is a common laboratory technique used to identify unknown components in mixtures. The "Chroma" in chromatography refers to colors. While early chromatography (and our experiment today) relied on our eyesight to read a chromatography result, modern instrumentation has other detection methods (absorbance in the ultraviolet or infrared, flame ionisation, mass spectrometry and many other techniques, depending on the type of samples you are separating).

There are several types of chromatography; all types employ a *mobile phase* or *eluent* (it can be liquid or gas), which is forced through a *stationary phase* (a solid or semi-solid). Mixtures are separated because some components will be more attracted to the stationary phase (and stick to it) while some components will be more attracted to the mobile phase (and travel with it).

By eye, we cannot know if each dye color is a mixture or pure substance. Using chromatography, the components in a sample will migrate along the filter paper at different rates such that they become spread out and separated from each other. The mobile phase takes advantage of differing solubility or polarity of the components in order to separate them. This component traveling process is called *elution*. Chromatography can be used to separate inks, dyes, pigments in plants, or used to determine the chemical composition of many substances.

Figure 1 shows a typical paper chromatogram. There are a few difficulties commonly encountered in the elution process. One problem is that spots tend the spread out as they elute, and can bleed into each other as they proceed up the paper. This can be confusing when interpreting the chromatogram. To avoid this problem, space the spots of sample far apart and make repeated, tiny applications of sample to prevent spreading. Another problem is an uneven solvent front. This can happen if the beaker is nudged – if the mobile phase sloshes inside, the elution trails may travel diagonally, which makes interpretation very difficult.



A component with a given solubility travels along with the mobile phase at one rate, regardless of what other components are present in the sample. If the red part of purple dye travels at the same rate as pure red dye, and both stop in the same place, the two should be the same red dye. The two red spots should have the same *Retention Factor*, R_f. The R_f is the distance, D, traveled by the spot divided by the distance traveled by the eluting solution, or *Solvent Front*, F.

$$R_f = \frac{D}{F} \tag{2.1.1}$$

Comparing the R_f values allows the confirmation of a component in multiple samples because unique components have unique R_f values (for the same solvent mixture at the same temperature).

Procedure

Materials and Equipment

Materials: chromatography paper, candy, and eluting solution.

Equipment: 600-mL beaker, pencil, ruler, binder clip and Whatman #1.

Safety

Be sure to handle only the dry part or the chromatogram when removing it from the beaker. Wash hands thoroughly if the eluting solution touches your skin. Personal protective equipment (PPE) required: closed-toe shoes

Part A: Preparation of dye solutions

1. You will use two pieces of candy, one with a primary color (red, yellow, blue), and one other one (green, orange, brown). You can choose what type of candy. Add two drops of water into a well of the spot plate. Add a piece of candy such that one side of it is suspended in water. After most of the dye is dissolved, turn the candy around to dissolve the dye on the other side. Once most of the dye is dissolved (i.e. you can see the white coating), remove the candy and discard. Do not eat the candy.
2. If you want to have a higher concentration of dye in solution, work together with someone who has the same color, and dissolve the dye from another piece of candy in the same solution. There is plenty of volume for multiple runs, so you can share the resulting dye solution.
3. Repeat for the different colors assigned to you.

Part B: Preparation of Chromatography Paper

1. Wash your hands thoroughly to remove excess oils from your skin. Obtain a ruler and a piece of chromatography paper. Handle the paper only on the edges to avoid leaving fingerprints, as these may hinder the elution process.
2. Place the chromatography paper on a sheet of clean notebook paper or paper towel to avoid picking up dirt or contaminants from the bench top. Orient the paper into a “portrait” position and write your name on the top edge of the paper in one corner. Using a pencil and ruler to measure accurately, draw a straight line across the paper, about 1.5 cm above the bottom edge. This is the **starting line**.
3. Lightly draw two small “X” centered on the starting line so that there is plenty of space between the spots and between spots and edge of the paper.
4. In the center of each X, apply a small amount of dye with a capillary tube (using the solutions you made in part A, see Figure 2 right).



Figure 2: Using the binder clip (left) and applying the sample (right)

5. Attach the top of the paper to a binder clip and insert a pencil through the metal loops (see left panel). Then, lay the pencil across a 600 mL beaker (avoiding the spout) and adjust the paper so it almost touches the bottom.

Part C: Acquisition of Chromatogram

1. Remove the paper strip, clip and pencil assembly from the beaker.
2. Find your eluting solution. Depending which bench you are working at (near the safety shower, near the fire alarm, or middle bench), you will use a different eluting solution. Pour about 25 mL of eluting solution into the beaker.

Bench	Eluting solution
near the safety shower	0.1 % aqueous sodium chloride solution
middle bench	0.1 % aqueous sodium chloride solution containing 5% ethanol
near the fire alarm light	1 % aqueous sodium chloride solution

3. Place a wick (a larger filter paper) into the beaker so it touches the side, and angle the beaker so that the wick gets wet (without pouring out the solution). Get a cover ready (aluminum foil or saran wrap).
4. Gently place the pencil on the rim of the beaker so that the paper is submerged in solution, but the dye spots are not submerged. You should see the solvent move up the paper strip. If necessary, adjust the height. Remember that the spots must be above the liquid level for the experiment to work.
5. Start a timer.
6. Cover the beaker with aluminum foil or saran wrap. Let the solvent move up least 6 cm (two-thirds of the length of the paper strip). This will take some patience.
7. When the solvent front moved far enough, lift the paper out of the solution by lifting the pencil. Let excess eluent drip into the beaker. Mark the position of the solvent front with a pencil (if you forget this step, you can later see the solvent front nicely under UV light). Let the paper air dry, attaching it to a snorkel hood to speed things up. Let it dry completely. Again, this might take some patience.

Part D: Interpretation of Chromatogram

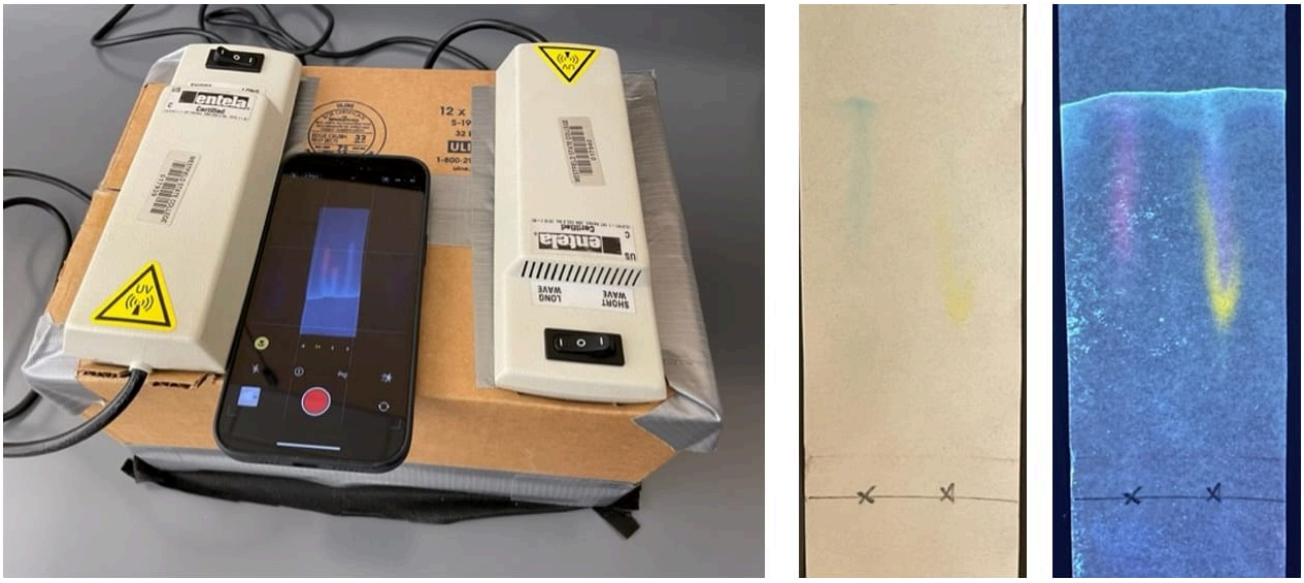


Figure 3: UV illuminator (left) and chromatogram viewed under visible vs. UV light (right)

1. Circle around each color spot. For mixtures, circle each separated color as best as you can.
2. Use the UV-cardboard illuminator (Figure 3 left panel) to see which spots fluoresce in a different color than the visible color. This will also help detecting colors like blue and yellow that might be difficult to see in visible light (Figure 3 right), and visualizing the solvent front after it dried.
3. Use a ruler and draw a plus sign in the center of each spot. Remember, if you separated a mixture, each single color gets a plus sign. Measure the distance from the starting line to each plus sign. Record this distance for each spot on your lab report. These are the D values, in cm.
4. Measure the distance between the starting line and the solvent front reached. Record this distance. This is the F value, in cm. Unless your solvent ran unevenly, this should be the same for all samples on a single strip.
5. Calculate the retention factor (R_f) for each spot and record the values in your lab report.
6. Compare the retention factor of samples with multiple spots (dye mixtures) to those with a single spot (pure dyes). What are colors used for the mixtures? Is that what you expected from your experience growing up?

Lab1 result sheet.pdf

Acknowledgements

Thanks to the Spring 2023 Monday lab section at Westfield State for many suggestions, corrections and the samples pictured here.

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2.2: Freezing Point Depression

Goals

- Measure freezing point depression.
- Determine the Van't Hoff factor for two different solutes

Safety

- The substances we are using today are household chemicals (table salt and sugar). However, the usual lab precautions apply.

Equipment and materials

- From your drawer: 50 mL beaker, 100 mL beaker
- On your bench: temperature probe
- At the balances: sucrose (s), sodium chloride (s)
- For task 4: sieve, calorimeter
- At a station: ice

Background

A colligative property is when a solute affects a solution property like its freezing or boiling point, and the effect depends only on the amount of particles dissolved, no matter which substance you use. In this lab we will investigate the effect of the different solutes on the freezing point of aqueous solutions.

You should observe that as the solute concentration increases the freezing point $T_{fp}(\text{solution})$ decreases (the freezing point depression ΔT increases), and this can be described by the following equation:

$$\Delta T = i \cdot k_f \cdot \frac{n_{\text{solute}}}{m_{\text{solvent}}} \tag{2.2.1}$$

where

$i = \text{Van't Hoff Factor}$

$k_f = \text{freezing point point constant}$

$n = \text{amount}$

$m = \text{mass}$

and

$\Delta T = T_{fp}(\text{pure solvent}) - T_{fp}(\text{solution})$

The value of k_f for the solvent water is 1860 °C g/mol, or 1.86 °C kg/mol. Check your units to decide which format is more convenient in this case.

Experimental Design Considerations

We are making the assumption that at the instance the ice melts the mixture of ice and water is at the freezing point. There can be multiple reasons why this is wrong, and we will discuss this in our pre-lab discussion.

Experimental Procedures

Record all observations in your lab notebook, and summarize your calculations and interpretations in this worksheet.

Part 1: Freezing point determination, pure water

1. Obtain a temperature probe and turn it on. Measure the mass of a clean, dry 100 mL beaker.
2. Pour approximately 25 mL of DI water into the 100 mL beaker
3. Place temperature probe into pure water in beaker
4. Add around 20 mL of fresh crushed ice to a 50 mL beaker.
5. Quickly add the ice to the 100 mL beaker, gently swirl the temperature probe. Record the lowest temperature, and the temperature just as the last bit of ice melts.
6. Weigh the mass of the beaker with the water and melted ice (so now just water). Record to the precision of the instrument. Clean and dry the 100 mL beaker and record the empty mass to the precision of the instrument.
7. Discard the water in the sink.
8. In your further calculations, use the lowest recorded temperature as the freezing point of water. The temperature probes are not calibrated, but are very good at measuring temperature differences. Don't be worried if the reading on your temperature probe is different from that of your neighbor, and don't swap temperature probes.

Part 2: Freezing point determination, sucrose solution

You can do part 2 and part 3 in any order, depending which balance is available.

1. Weigh approx 0.05 mol of sucrose (molar mass is 342.3 g/mol), record the exact mass, and transfer to the clean and dry 100 mL beaker.
2. Add approximately 25 mL water.
3. Place temperature probe into the solution in the beaker and swirl and stir to dissolve the sucrose completely.
4. Add around 20 mL of fresh crushed ice to the 50 mL beaker.
5. Quickly add the ice to the 100 mL beaker, gently swirl the temperature probe. Record the lowest temperature, and the temperature just as the last bit of ice melts.
6. Weigh the mass of the beaker with the sucrose solution and melted ice (so now a diluted sucrose solution). Record to the precision of the instrument. Clean and dry the 100 mL beaker and record the empty mass to the precision of the instrument. Then, calculate the mass of water (solvent) from the available data.
7. Calculate the van't Hoff factor for sucrose and record your work in the lab summary.

Part 3: Freezing point determination, sodium chloride solution

1. Weigh approx 0.05 mol of sodium chloride (molar mass is 58.44 g/mol), record the exact mass, and transfer to the clean and dry 100 mL beaker.
2. Add approximately 25 mL water.
3. Place temperature probe into the solution in the beaker and swirl and stir to dissolve the sodium chloride completely.
4. Add around 20 mL of fresh crushed ice to the 50 mL beaker.
5. Quickly add the ice to the 100 mL beaker, gently swirl the temperature probe. Record the lowest temperature, and the temperature just as the last bit of ice melts.
6. Weigh the mass of the beaker with the sodium chloride solution and melted ice (so now a diluted sodium chloride solution). Record to the precision of the instrument. Clean and dry the 100 mL beaker and record the empty mass to the precision of the instrument. Then, calculate the mass of water (solvent) from the available data.
7. Calculate the van't Hoff factor for sodium chloride and record your work in the lab summary.

Part 4: Try a different experimental strategy

With a partner, measure the freezing point of a solution of sodium chloride again, using a different experimental strategy based on the calorimeters we built last semester. This method is a bit faster but requires more equipment (work with a calorimeter instead of a beaker to minimize heat transfer, or use the minimum temperature in the calculation, quickly removing the remaining ice by pouring the slush through a sieve). As you are going through the steps, reflect on the pros and cons of this and the orginal method.

1. Weigh approx 0.05 mol of sodium chloride (molar mass is 58.44 g/mol), record the exact mass, and transfer to calorimeter cup.
2. Add approximately 25 mL water from a 100 mL beaker.

3. Place temperature probe into the solution in the beaker and swirl and stir to dissolve the sodium chloride completely. Place the calorimeter cup into the calorimeter.
4. Add around 20 mL of fresh crushed ice to the 50 mL beaker.
5. Quickly add the ice to the calorimeter cup, and gently stir with the temperature probe. Once the temperature stops decreasing (make sure there is still ice present), record the temperature, remove the temperature probe, and pour the mixture through a sieve into a dry 100 mL beaker. Discard the ice in the sieve.
6. Weigh the mass of the beaker with the sodium chloride solution. Record to the precision of the instrument. Clean and dry the 100 mL beaker and record the empty mass to the precision of the instrument. Then, calculate the mass of water (solvent) from the available data.
7. Calculate the van't Hoff factor for sodium chloride and record your work in the lab summary.

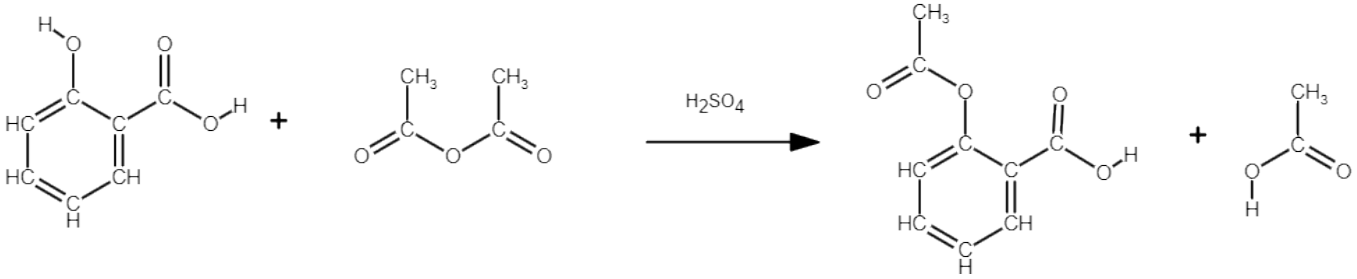
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2.3: Synthesis and Recrystallization of Aspirin

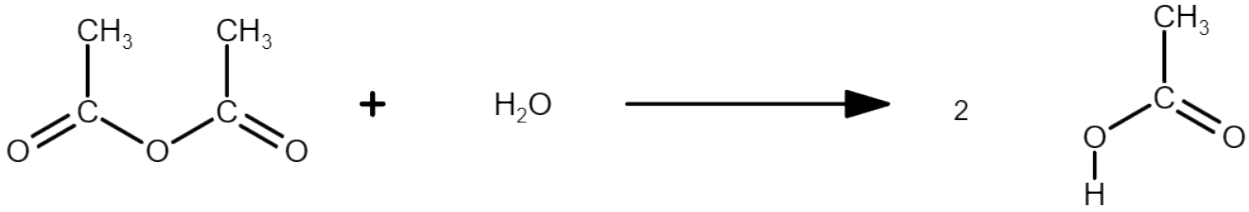
Over history, many compounds obtained from nature have been used to cure ills or to produce an effect in humans. These natural products have been obtained from plants, minerals, and animals. In addition, various transformations of these and other compounds have led to even more medically useful compounds.

Analgesics are compounds used to reduce pain, antipyretics are compounds used to reduce fever. One popular drug that does both is aspirin. The Merck Index, which is an encyclopedia of chemicals, drugs and biologicals, lists the following information under aspirin: acetylsalicylic acid; monoclinic tablets or needle-like crystals; mp 135 °C (rapid heating); is odorless, but in moist air it is gradually hydrolyzed into salicylic and acetic acids; one gram dissolves in 300 mL of water at 25 °C, in 100 mL of water at 37 °C, in 5 mL alcohol, in 17 mL chloroform.

Synthesis of aspirin (acetylsalicylic acid)



Reaction scheme 1: Salicylic acid and acetic acid anhydride react to form aspirin and acetic acid, catalyzed by sulfuric acid



Reaction scheme 2: Acetic anhydride reacts with water to form two molecules of acetic acid

Week 1: Aspirin Synthesis

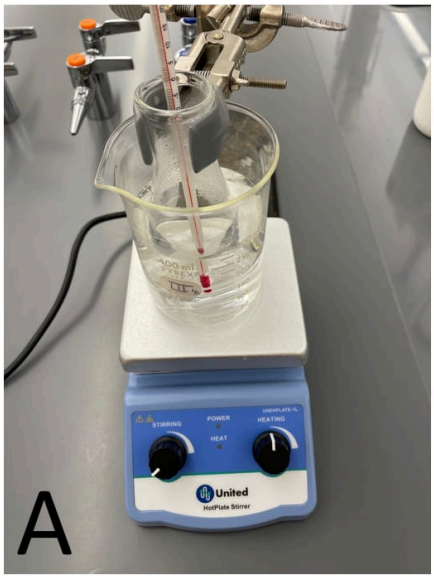
For the first week of this two-week experiment, you will synthesize and recrystalize aspirin.

Synthesis of crude aspirin

1. Set up a hot water bath (400 mL beaker filled to 100 mL with water) on a hotplate and adjust the water temperature to about 75 °C (see photo below - after the water reaches the desired temperature, set the heating dial to the minimal value as in the photo, or turn it off if the temperature is too high).
2. Place 2.0 g (about 15 mmol) of salicylic acid in a 125-mL Erlenmeyer flask.
3. Under the hood, add 5 mL (5.4 g, about 50 mmol) of acetic anhydride, followed by 5 drops of conc. H_2SO_4 (*use the provided dropper, H_2SO_4 is highly corrosive*) and swirl the flask gently most of the salicylic acid dissolves (the remainder will dissolve as you heat the reaction).
4. Heat the flask gently in the hot water bath for 15 minutes to run reaction scheme 1 shown above (if you overheat, you get side products; if you don't heat, you get less of the desired product). The flask will float on the hot water at an angle, which is fine.
5. Allow the flask to cool until you can touch the bottom. Under the hood, add 10 mL of water to react with the excess of acetic

anhydride (see reaction scheme below). Bring the flask back to your bench.

6. If acetylsalicylic acid does not begin to crystallize out, scratch the inside walls of the flask with a glass rod or add a seed crystal. Cool the mixture slightly in an ice bath until crystallization is completed. **If the mixture appears milky or there is a whitish oil on the bottom, consult your instructor.** The product will appear as a solid mass when crystallization is completed. Then, add 50 mL of water and cool the mixture in an ice bath. Do not add the water until crystal formation is complete.
7. Vacuum filter the product using wetted round filter paper in a Buchner funnel (see photo below for setup). The total amount will not fit in the funnel. Swirl before you pour to get the chunks transferred, and use the glass rod with rubber policeman to transfer any remaining solids.
8. Rinse the crystals several times with small portions (5 mL) of cold water and air dry the crystals on a Buchner funnel by suction until the crystals appear to be free of solvent (no more liquid dripping from the funnel. Place the crude solid on a pre-weighed watch glass and record the mass of the solid. Place a small amount on a watch glass to dry. We will use a portion for the ferric chloride test, and a small amount to measure the melting point next week.

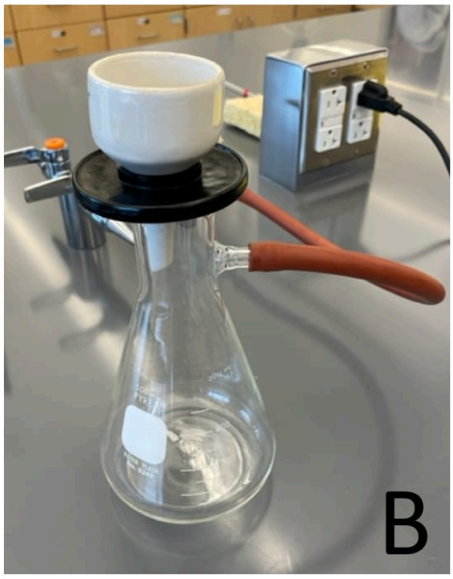


If the hot plate stirrer does not work:

1. For testing purposes, turn the stirring knob clockwise (the power light should turn on)
2. Make sure the on/off toggle on the side is in the on position
3. Make sure the power chord is pushed in all the way in the back of the stirrer
4. Make sure the electrical breaker on the outlet did not pop (orange indicator on one side of the bench)

If the filtration does not work:

1. Make sure you put a filter paper in.
2. Make sure the black gasket is in the right orientation.
3. Make sure the vacuum valve is open (parallel to hose)



Panel A: Water bath setup (instead of clamping the Erlenmeyer, you can also carefully place it without clamping). Panel B: filtration setup. For troubleshooting tips, see center.

Recrystallization of aspirin

1. Place the remaining crude product into a clean 125 mL Erlenmeyer flask. Add ethanol (about 1 mL for every gram of crude product) and warm in the water bath at about 50°C. If lots of solid remains, add more ethanol in 1-mL portions. Once most or all is dissolved, add 40 mL water. Cool down to room temperature and make sure crystals form (you can use the scratching method or add a tiny crystal of crude product if crystals don't form on their own) before you cool down further in an ice bath. **If you get a milky liquid instead of solid crystals, consult your instructor.**
2. Vacuum filter the product using a Buchner funnel.
3. Rinse the crystals several times with small portions (5 mL) of cold water and air dry the crystals on a Buchner funnel by suction until the crystals appear to be free of solvent. Test this recrystallized product for the presence of unreacted salicylic acid using the ferric chloride test (procedure below) and keep some for measuring the melting point next week.
4. Fill out the result sheet.
5. Record the mass and calculate the relative yield in percent.
6. Follow your instructor's directions to properly store your product to dry by next week.

Week 2: Aspirin Characterization

For the second week of this experiment, you will perform the following tests after your aspirin product has completely dried.

Ferric chloride test for salicylic acid

Phenols are compounds that have a hydroxyl (-OH) group attached directly to a benzene ring. Salicylic acid is a phenol while aspirin is not. Formation of an iron-phenol complex with Fe(lll) gives a definite color ranging from red to violet, depending upon the particular phenol present.

1. Add 10 drops of aqueous 1% ferric chloride solution to a test tube containing a few crystals of the compound to be tested dissolved in 5 mL water and note the color.
2. Do this test with (a) salicylic acid, (b) your crude product and (c) your recrystallized product.

Melting point determination

This is done in batches of three samples (crude product, recrystallized product, store-bought product). The idea is to start with low temperature, and watch the sample visually as you ramp up the temperature, writing down the temperature at which each sample melts:

1. Make sure the temperature of the melting point apparatus is below 70 °C.
2. Place your samples in the melting point apparatus. Make sure you can see the solids.
3. Turn on the heater. Watch the samples and the temperature (two people).
4. Once the last sample is melted, turn off the heater.
5. **Make sure the heater is turned off** to allow the next group of measurements to proceed as soon as possible.

HPLC analysis

Follow your instructor's directions to analyze your aspirin product using high performance liquid chromatography (HPLC).

Contributors and Attributions

- James Chickos, David Garin, and Valerian D'Souza. University of Missouri–St. Louis; Chemistry)

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2.4: Acetone Iodine kinetics

Introduction

In this lab, we will apply the concepts of reaction rate, order of reaction and catalyst. Our example reaction is the iodination of acetone catalyzed by hydrochloric acid.

$$\mathrm{CH_3C(=O)CH_3 + I_2 \xrightarrow{HCl} CH_3C(=O)CH_2I + I^- + H^+} \tag{2.4.1}$$

The reaction is zero order in iodine. You will figure out the order in acetone and the order in HCl, and the rate constant. Then, you will predict which initial concentrations are necessary to make the reaction run for exactly 3 minutes (180 s).

We will figure out the reaction rate by visual observation of the iodine. Iodine in water is brownish-yellow, and all other species are colorless. In the experiments, you will start a timer once iodine is added to the other substances in the reaction mixture, and measure the time until the solution appear colorless. We will run the reaction at room temperature. Try not to warm up any of the reactants or the reaction mixture with your hands or other sources of heat (e.g. sunlight near window).

Experimental design

Finding the rate

Usually, the reaction rate of a reaction changes over time as reactants are depleted. Also, the concentrations change over time. To be able to "plug in" the rate and the concentrations into the rate law and determine the order of reactions, you would measure an initial rate.

Today, we are measuring an average rate, but it turns out the rate stays constant over the course of the experiment. There are three reasons for this:

1. The HCl concentration does not change (catalyst)
2. The acetone concentration does not change significantly (in large excess over iodine)
3. The iodine concentration changes a lot, but it does not affect the rate (zero order in iodine)

To calculate the rate, we consider the time it took for the iodine to "fade", and the initial concentration of the iodine. For example, if the initial iodine concentration is 0.001 mol /L, and it takes 180 s for the reaction mixture to become clear, the rate would be:

$$rate = -\frac{\Delta[\mathrm{I_2}]}{\Delta t} = \frac{1.0\ \times 10^{-3}\mathrm{M}}{180\mathrm{s}} = 5.5\ \times 10^{-5}\mathrm{M}^{-1}\ \mathrm{s}^{-1} \tag{2.4.2}$$

Varying concentrations to find the order

You will set up multiple reactions, varying the concentrations in the reaction mix. To quickly figure out the concentrations, it makes sense to always keep the final volume the same (25 mL), and use the dilution law to figure out concentrations (or volumes you need to add to get the desired concentration). Here is the dilution law:

$$c_{\mathrm{mix}} V_{\mathrm{mix}} = c_{\mathrm{stock}} V_{\mathrm{stock}} \tag{2.4.3}$$

If you want to know the (initial) concentration of a component in the reaction mixture, plug in the volume and concentration of the stock solution. For example, if our 25 mL reaction mixture contains 5 mL iodine stock with an original concentration of 0.005 M, we get:

$$c_{\mathrm{mix}} = c_{\mathrm{stock}} \frac{V_{\mathrm{stock}}}{V_{\mathrm{mix}}} = 0.005\mathrm{M}\ \frac{5\mathrm{mL}}{25\mathrm{mL}} \tag{2.4.4}$$

If you want to know how much of the 4-M acetone stock solution you need to get 0.8 M in the reaction mixture, you solve for the

volume of stock solution:

$$V_{\text{stock}} = V_{\text{mix}} \frac{c_{\text{mix}}}{c_{\text{stock}}} = 25\text{mL} \ \frac{0.8\text{M}}{4\text{M}} \tag{2.4.5}$$

Procedure

This is the procedure for measuring a single rate. You will run this procedure twice for each different reaction mixture. You can figure out the concentrations of the mixture from the recipe (the volumes mixed) using equation 4 or this spreadsheet. Make sure the volumes in your recipe always add up to 25 mL.

1. Mix water, acetone stock and hydrochloric acid stock in a 125 mL Erlenmeyer. The reaction can not proceed at this stage (no iodine).
2. At your bench, add the correct amount of iodine stock (5 mL) to the mixture and start the timer.
3. For better visibility of the color change, pour the reaction mixture into a large test tube, and observe from the top, with a white paper under the test tubes. Prepare a large test tube with 25 mL water and one with 0.5 mL iodine solution in 24.5 mL of water for comparison (see picture below: the iodine solution labeled 1:10 looks like water in the side view, but has a faint brownish tint in the top view).
4. Stop the timer once the solution is colorless. Record the time in your lab notebook and calculate the rate for your experiment (like in equation 2).
5. Keep the test tube with water and the 1:10 mix for the remainder of the runs. Discard the reaction mix, rinse the 125 mL Erlenmeyer and the large test tube and invert to dry so you can use them again for the next run

Sideview



H_2O mix 1:10

Top view



Determining the orders of reaction

The easiest way to find an order of reaction is to halve the concentration of the species you are interested in while keeping all other concentrations the same. If the reaction time stays the same, it is zero order in the species whose concentration you changed. If the reaction time doubles (the rate halves), it is first order. If the change is more extreme, it is a higher order than one. If you learned a mathematical way of determining the reaction order in your lecture section, feel free to use that method. Because of experimental errors, we don't expect the reaction order to come out exactly as 0, 1, or 2, and you should feel free to round your result as you formulate the rate law.

To find out two orders of reaction (with respect to acetone and with respect to hydrochloric acid), you need three sets of concentrations (see data sheet). You will run each set in duplicate, so you end up running a total of six experiments.

We have aqueous stock solutions containing acetone (c = 4 M), hydrochloric acid (c = 1 M), and iodine (c = 0.005 M). For the first set of concentrations, you will use 10 mL acetone solution, 5 mL water, and 5 mL hydrochloric acid (which is corrosive and should

be handled under the hood until diluted), giving a subtotal of 20 mL. Measure out 5 mL iodine solution in a graduated cylinder, and start the reaction at your bench by adding the iodine to the other components in the Erlenmeyer. Record the time and repeat the experiment once to check for consistency.

For the second set of concentrations, we are using half the volume of acetone, and for the third set of concentrations, we are using half the volume of hydrochloric acid (compared to the first set). The volumes are given in the data sheet. Calculate the volume of water you need. You should check your recipe before you make the reaction mixture. Make sure the volumes in your recipe always add up to 25 mL.

Predicting the rate of reaction

Once you know the form of the rate law (i.e. all the orders of reaction), you can plug in all the concentrations and the measured rate to calculate the rate constant. It should be the same for all of your trials (if the temperature is constant), so you can average it to average out random errors. Once you have the rate constant, you can predict the rate (and the reaction time) for any set of concentrations. We will have a contest to see who can get their reaction to end after exactly 2 minutes (120 s), or closest to that time.

The contest will be done bench by bench. Once everyone has calculated the concentrations they want to use, and has prepared their reaction mixture (minus the iodine), you can ask for the contest to start. Your instructor starts a countdown, and you will add the iodine to the flask when the timer starts. Then, you will transfer your reaction mixture to a large test tube and place it in a test tube rack in the front. A doc camera will project the image of the tubes on the screen, so everyone in the room can see. Whenever a test tube turns colorless, we will record the time. The reaction completing closest to 120 s wins our appreciation for good calculations and good experimental technique (with a bit of luck).

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2.5: Equilibrium and Le Chatelier's Principle

Objectives

- To observe the effect of an applied stress on chemical systems at equilibrium.

A reversible reaction is a reaction in which both the conversion of reactants to products (forward reaction) and the re-conversion of products to reactants (backward reaction) occur simultaneously:

Forward reaction:

$$\mathrm{A + B \longrightarrow C + D} \tag{2.5.1}$$

$$\mathrm{Reactants \longrightarrow Products} \tag{2.5.2}$$

Backward reaction:

$$\mathrm{C + D \longrightarrow A + B} \tag{2.5.3}$$

$$\mathrm{Products \longrightarrow Reactants} \tag{2.5.4}$$

Reversible reaction:

$$\mathrm{A + B \rightleftharpoons C + D} \tag{2.5.5}$$

Consider the case of a reversible reaction in which a concentrated mixture of only A and B is supplied. Initially the forward reaction rate ($\mathrm{A + B \longrightarrow C + D}$) is fast since the reactant concentration is high. However as the reaction proceeds, the concentrations of A and B will decrease. Thus over time the forward reaction slows down. On the other hand, as the reaction proceeds, the concentrations of C and D are increasing. Thus although initially slow, the backward reaction rate ($\mathrm{C + D \longrightarrow A + B}$) will speed up over time. Eventually a point will be reached where the rate of the forward reaction will be equal to the rate of the backward reaction. When this occurs, a state of chemical equilibrium is said to exist. Chemical equilibrium is a dynamic state. At equilibrium both the forward and backward reactions are still occurring, but the concentrations of A, B, C, and D remain constant.

A reaction at equilibrium can be disturbed ("restarted", if you will, even tough the forward and reverse reactions never stop) if a "stress" is applied to it. Examples of stresses include increasing or decreasing concentrations or reactants or products, or temperature changes. If such a stress is applied, the reversible reaction will undergo a shift (show a net reaction) in order to re-establish equilibrium. This is known as Le Chatelier's Principle.

Consider a hypothetical reversible reaction already at equilibrium: $\mathrm{A + B \rightleftharpoons C + D}$. If, for example, the concentration of A is increased, the system would no longer be at equilibrium. The rate of the forward reaction ($\mathrm{A + B \longrightarrow C + D}$) would briefly increase in order to reduce the amount of A present and would cause the system to undergo a net shift to the right. Eventually the forward reaction would slow down and the forward and backward reaction rates become equal again as the system returns to a state of equilibrium.

Using similar logic, the following changes in concentration are expected to cause the following shifts:

- Increasing the concentration of A or B causes a shift to the right.
- Increasing the concentration of C or D causes a shift to the left.
- Decreasing the concentration of A or B causes a shift to the left.
- Decreasing the concentration of C or D causes a shift to the right.

In other words, if a chemical is added to a reversible reaction at equilibrium, a net reaction away from the added chemical occurs. When a chemical is removed from a reversible reaction at equilibrium, a net reaction towards the removed chemical occurs.

You can also explain this using the concepts of reaction quotient Q and equilibrium constant K. At equilibrium, Q = K. In the absence of products, Q is zero, and the reaction will go forward, gradually increasing Q until it is equal to K. At this point, we are

at equilibrium, all reactants and products are present, and there is no net reaction in either direction. Changing any concentration after the reaction reached equilibrium will change Q, but not K. As a consequence, we are no longer at equilibrium, and we will observe a net reaction until we reach equilibrium (and Q is equal to K again). The equilibrium concentrations at that point will be different for the initial equilibrium concentrations, but Q will have the same value again.

A change in temperature will also cause a reversible reaction at equilibrium to undergo a shift. The direction of the shift depends on whether the reaction is exothermic or endothermic. In exothermic reactions, heat energy is released and can thus be considered a product. In endothermic reactions, heat energy is absorbed and thus can be considered a reactant.

Exothermic:

$$\mathrm{A + B \rightleftharpoons C + D + \ heat} \tag{2.5.6}$$

Endothermic:

$$\mathrm{A + B + \ heat \rightleftharpoons C + D} \tag{2.5.7}$$

As a general rule, if the temperature is increased, a shift away from the side of the equation with “heat” occurs. If the temperature is decreased, a shift towards the side of the equation with “heat” occurs.

If we try to explain this with Q vs K, it turns out that the equilibrium constant is not constant after all but is temperature-dependent. So K changes, and Q adjusts to match it.

In this lab, the effect of applying stresses to a variety of chemical systems at equilibrium will be explored. The equilibrium systems to be studied are given below:

1. Saturated Sodium Chloride Solution

$$\mathrm{NaCl\,(s) \rightleftharpoons Na^+\,(aq) + Cl^-\,(aq)} \tag{2.5.8}$$

2. Aqueous Ammonia Solution (with phenolphthalein)

$$\underbrace{\mathrm{NH_3\,(aq)}}_{\text{Clear}} + \mathrm{H_2O\,(l) \rightleftharpoons NH_4{}^+\,(aq) + \underbrace{OH^-\,(aq)}_{\text{Pink}}} \tag{2.5.9}$$

3. Cobalt(II) Chloride Solution

$$\underbrace{\mathrm{Co(H_2O)_6{}^{2+}\,(aq)}}_{\text{Pink}} + 4\,\mathrm{Cl^-\,(aq) \rightleftharpoons \underbrace{CoCl_4{}^{2-}\,(aq)}_{\text{Blue}} + 6\,H_2O\,(l)} \tag{2.5.10}$$

4. Iron(III) Thiocyanate Solution

$$\underbrace{\mathrm{Fe^{3+}\,(aq)}}_{\text{Pale Yellow}} + \underbrace{\mathrm{SCN^-\,(aq)}}_{\text{Colorless}} \rightleftharpoons \underbrace{\mathrm{Fe(SCN)^{2+}\,(aq)}}_{\text{Deep Red}} \tag{2.5.11}$$

By observing the changes that occur (color changes, precipitate formation, etc.) the direction of a particular shift may be determined. Such shifts may then be explained by carefully examining the effect of the applied stress as dictated by Le Chatelier’s Principle. If no visible changes happen, you can assume the reaction is at equilibrium. At equilibrium, you don't see the effects of forward and reverse reactions because they occur at equal rates.

Procedure

Materials and Equipment

Equipment: 10 small test tubes, test tube rack, test tube holder, hot plate, 2 medium-sized beakers (for stock solutions), 10-mL graduated cylinder, wash bottle, stirring rod, and scoopula. Sparkling water.

Chemicals: solid $\mathrm{NH_4Cl}$ (s), saturated NaCl (*aq*), concentrated 12 M HCl (*aq*), 0.1 M $\mathrm{FeCl_3}$ (*aq*), 0.1 M KSCN (*aq*), 0.1 M $\mathrm{AgNO_3}$ (*aq*), 0.1 M $\mathrm{CoCl_2}$ (*aq*), concentrated 15 M $\mathrm{NH_3}$ (*aq*), phenolphthalein, 6 M $\mathrm{HNO_3}$ (*aq*), and 10% NaOH (*aq*).

Safety

All of the acids and bases used in this experiment (NH_3, HCl, HNO_3 and $NaOH$) can cause chemical burns. In particular, concentrated 12 M HCl is extremely dangerous! If any of these chemicals spill on you, immediately rinse the affected area under running water and notify your instructor. Also note that direct contact with silver nitrate ($AgNO_3$) will cause dark discolorations to appear on your skin. These spots will eventually fade after repeated rinses in water. Finally, in Part 3 you will be heating a solution in a test tube directly in a Bunsen burner flame. If the solution is overheated it will splatter out of the tube, so be careful not to point the tube towards anyone while heating.

Experimental Procedure

Record all observations on your report form. These should include, but not be limited to, color changes and precipitates. Note that solution volumes are approximate for all reactions below. Dispose of all chemical waste in the containers in the hood.

Part 1: Saturated Sodium Chloride Solution

1. Place 3 mL of saturated $NaCl$ (*aq*) into a small test tube. Add one tiny crystal of NaCl to make sure it is saturated.
2. ***Carefully*** add concentrated 12 M HCl (*aq*) drop-wise to the solution in the test tube until a distinct change occurs (don't add more than 3 drops). Record your observations.

Think about the following: In step 1, did the added crystal dissolve? Is this at equilibrium? In step 2, which concentrations changed, and in what direction? What was the net direction of the reaction you observed? How can you tell whether the reaction is at equilibrium again?

Part 2: Aqueous Ammonia Solution

Instructor Prep: At the beginning of lab prepare a stock solution of aqueous ammonia. Add 4 drops of concentrated 15 M NH_3 (*aq*) and 3 drops of phenolphthalein to a 150-mL (medium) beaker, top it up with 100-mL of distilled water, and mix with a stirring rod. Label the beaker and place it on the front desk. The entire class will then use this stock solution in Part 2.

1. Place 3-mL of the prepared stock solution into a small test tube.
2. Add a medium scoop of NH_4Cl powder to the solution in this test tube. Record your observations.

Think about the following: In step 1, how do you know the reaction is at equilibrium? In step 2, which concentrations changes, and in what direction? What was the net direction of the reaction you observed? How can you tell whether the reaction is at equilibrium again? In part 1, we changed the volume of the solution a bit by adding drops of another aqueous solution. What happened to the volume of the solution in part 2?

Part 3: Cobalt(II) Chloride Solution

1. Label three small test tubes 1-3. Place 3 mL of 0.1 M $CoCl_2$ (*aq*) into tubes #1 and #3, and 1 mL into tube #2.
2. The solution in test tube #1 remains untouched. It is a control for comparison with other tubes.
3. To the solution in test tube #2, carefully add concentrated 12 M HCl (*aq*) drop-wise until a drastic color change occurs. Record your observations.
4. To the solution in test tube #3, first add a medium scoop of solid NH_4Cl. Then heat this solution in the provided hot water bath for about 30 seconds, or, until a distinct change occurs. Record your observations. Then cool the solution in test tube #3 back to room temperature by holding it under running tap water (or water ice slush), and again record your observations.

Think about the following: What is the evidence that tube #1 is at equilibrium? Which concentration changed in tube #2, and in which net direction did the reaction proceed? Which concentration changed in tube #3, and in which net direction did the reaction proceed before heating? Is the reaction endothermic or exothermic based on the color change upon heating and cooling?

Part 4: Iron(III) Thiocyanate Solution

Instructor Prep: At the beginning of lab prepare a stock solution of iron(III) thiocyanate. Add 2.5 mL of 0.1 M $\mathrm{FeCl_3}$ (*aq*) and 2.5 mL of 0.1 M KSCN (*aq*) to a 400-mLbeaker, top it up with 250 mL of distilled water, and mix with a stirring rod. Label the beaker and place it on the front desk. The entire class (up to 18 students) will then use this stock solution in Part 4.

1. Place 3 mL each of the prepared stock solution into 4 small test tubes. Label these test tubes 1-4.
2. The solution in test tube #1 remains untouched. It is a control for comparison with other tubes.
3. To the solution in test tube #2, add 1 mL of 0.1 M $\mathrm{FeCl_3}$ (*aq*). Record your observations.
4. To the solution in test tube #3, add 1 mL of 0.1 M KSCN (*aq*). Record your observations.
5. To the solution in test tube #4, add 0.1 M $\mathrm{AgNO_3}$ (*aq*) drop-wise until all the color disappears. A light precipitate may also appear. Record your observations. Here the added silver nitrate is effectively removing thiocyanate ions from the equilibrium system via a precipitation reaction:

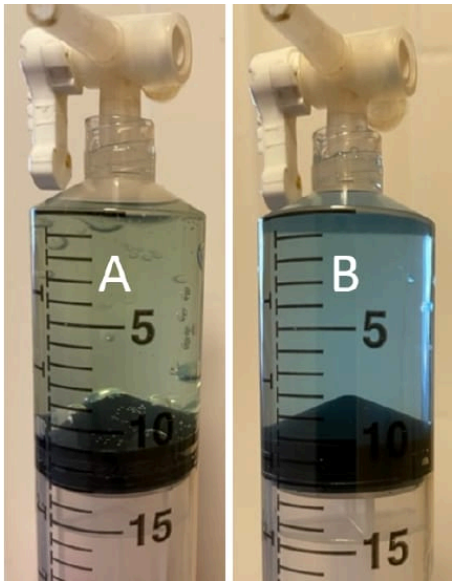
$$\mathrm{Ag^{+1}\,(aq) + SCN^{-1}\,(aq) \longrightarrow AgSCN\,(s)} \tag{2.5.12}$$

.

Think about the following: In step 3, which concentration changed, and in which net direction did the reaction proceed? What about in step 4 and step 5?

Part 5: $\mathrm{CO_2}$ dissolved in water

Predict the outcomes of the following experiments before approaching the "soda station" and testing your predictions. At the soda station, there are syringes with stoppers, a supply of soda with some pH indicator dissolved in it (not for human consumption!). The color of the indicator is greenish at the pH of the sparkling water, and blueish in pure water (compare panel A and panel B of the figure below).



The reaction you will study is

$$\mathrm{CO_2\,(aq) \rightleftharpoons CO_2\,(g)} \tag{2.5.13}$$

We can increase the concentration of carbon dioxide in the gas phase by pushing down the plunger, and decrease it by pulling out the plunger (or exchanging the gas in the syringe with air from the lab). We can decrease the concentration of carbon dioxide in the soda by mixing it vigorously in an open container (you know this: soda in an open container eventually "goes flat").

We can see that the reaction goes forward when we see bubbles rising out of the soda. We can estimate the concentration of carbon dioxide in solution by looking at the color of the pH indicator. The more carbon dioxide is dissolved, the more acidic the solution is. The simplified chemical equation for this is

$$CO_2\,(aq) + H_2O \rightleftharpoons HCO_3^-\,(aq) + H^+\,(aq) \tag{2.5.14}$$

Predictions and procedures, Part 5

For the following, predict what happens (what is the direction of the net reaction, and what will happen to the color of the solution and the volume of gas in the syringe).

1. Get a 30 mL syringe. Pull up 10 mL of soda with indicator, cap the syringe, observe and record the color, and record the volume of gas, if any.
2. Pull out the plunger to the 30 mL mark, and shake the syringe. Release the plunger and, observe and record the color, and record the volume of gas.
3. Press in the plunger as far as you can with medium force, shake the syringe. Release the plunger and, observe and record the color, and record the volume of gas.
4. Repeat this a couple of times: Pull out the plunger, shake, open the valve, push the plunger in to the 10 mL mark, close the valve. Observe and record the color.

Once you have written all of your predictions on the data sheet, find a partner and do the experiment after sharing your prediction. If you have time, watch this video after you are done: https://youtu.be/QtCRxvCxa6M

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2.6: Determination of Kc for a Complex Ion Formation

Objectives

- Find the value of the equilibrium constant for formation of $\mathrm{FeSCN^{2+}}$ by using the visible light absorption of the complex ion.
- Use the equilibrium constant to predict the equilibrium concentration of product, and the absorption of the reaction mix.

In the study of chemical reactions, we first study reactions that go to completion. Inherent in these familiar problems—such as calculation of theoretical yield, limiting reactant, and percent yield—is the assumption that the reaction can consume *all* of one or more reactants to produce products. In fact, most reactions do not behave this way. Instead, reactions reach a state where both reactants and products are present. This mixture is called the *equilibrium state*; at this point, chemical reaction occurs in both directions at equal rates. Therefore, once the equilibrium state has been reached, no further change occurs in the concentrations of reactants and products.

The equilibrium constant, K, is used to quantify the equilibrium state. The expression for the equilibrium constant for a reaction is determined by examining the balanced chemical equation. For a reaction involving aqueous reactants and products, the equilibrium constant is expressed as a ratio between reactant and product concentrations, where each term is raised to the power of its reaction coefficient (Equation 2.6.2). The value of this constant at equilibrium is always the same, regardless of the initial reaction concentrations. At a given temperature, whether the reactants are mixed in their exact stoichiometric ratios or one reactant is initially present in large excess, the ratio described by the equilibrium constant expression will be achieved once the reaction composition stops changing.

$$a\mathrm{A}(aq) + b\mathrm{B}(aq) \rightleftharpoons c\mathrm{C}(aq) + d\mathrm{D}(aq) \tag{2.6.1}$$

with

$$K_c = \frac{[\mathrm{C}]^c[\mathrm{D}]^d}{[\mathrm{A}]^a[\mathrm{B}]^b} \tag{2.6.2}$$

We will be studying the reaction that forms the reddish-orange iron (III) thiocyanate complex ion, $\mathrm{Fe(H_2O)_5SCN^{2+}}$ (Equation 2.6.3). The actual reaction involves the displacement of a water ligand by thiocyanate ligand, $\mathrm{SCN^-}$ and is often call a ligand exchange reaction.

$$\mathrm{Fe(H_2O)_6{}^{3+}\,(aq) + SCN^-\,(aq) \rightleftharpoons Fe(H_2O)_5SCN^{2+}\,(aq) + H_2O\,(l)} \tag{2.6.3}$$

For simplicity, and because water ligands do not change the net charge of the species, water can be omitted from the formulas of $\mathrm{Fe(H_2O)_6{}^{3+}}$ and $\mathrm{Fe(H_2O)_5SCN^{2+}}$; thus $\mathrm{Fe(H_2O)_6{}^{3+}}$ is usually written as $\mathrm{Fe^{3+}}$ and $\mathrm{Fe(H_2O)_5SCN^{2+}}$ is written as $\mathrm{FeSCN^{2+}}$ (Equation 2.6.4). Also, because the concentration of liquid water is essentially unchanged in an aqueous solution, we can write a simpler expression for K_c that expresses the equilibrium condition only in terms of species with variable concentrations.

$$\mathrm{Fe^{3+}\,(aq) + SCN^-\,(aq) \rightleftharpoons FeSCN^{2+}\,(aq)} \tag{2.6.4}$$

In this experiment, you will create several different aqueous mixtures of $\mathrm{Fe^{3+}}$ and $\mathrm{SCN^-}$. Since this reaction reaches equilibrium nearly instantly, these mixtures turn reddish-orange very quickly due to the formation of the product $\mathrm{FeSCN^{2+}}$ (*aq*). The intensity of the color of the mixtures is proportional to the concentration of product formed at equilibrium. As long as all mixtures are measured at the same temperature, the ratio described in Equation 2.6.4 will be the same.

Avoiding crowds at the spectrophotometer and the hood

This lab has experimental and computational tasks. If you find yourself in a line at the spec or the hood, ask yourself whether you could work on the computational tasks instead, making better use of your time.

Measurement of [$\mathrm{FeSCN^{2+}}$]

Since the complex ion product is the only strongly colored species in the system, its concentration can be determined by measuring the intensity of the orange color in equilibrium systems of these ions. To measure the color of the complex, we will use spectrophotometry at a wavelength of 447 nm and use a standard curve to estimate concentrations of the colored complex.

A standard curve is a plot of absorbances, $A_{447\mathrm{nm}}$, vs. concentrations, c, for several solutions with known concentration of $\mathrm{FeSCN^{2+}}$. We will provide the standard curve, and you will use it to estimate unknown concentrations from their absorbance.

Calculations

In order to determine the value of K_c, the equilibrium values of [redacted], $[\mathrm{SCN^-}]$, and [redacted] must be known. The equilibrium value of [redacted] will be determined by spectrophotometry as described above; its initial value was zero, since no $\mathrm{FeSCN^{2+}}$ was added to the solution.

The equilibrium values of [redacted] and $[\mathrm{SCN^-}]$ can be determined from a reaction table ('ICE' table) as shown in Table 1. The initial concentrations of the reactants—that is, [redacted] and $[\mathrm{SCN^-}]$ prior to any reaction—can be found by a dilution calculation based on the values from Table 2 found in the procedure.

Students at each bench are responsible for calculating one set of initial concentrations and writing it on the board after checking that everyone got the same answer. Be prepared to show your work to everyone in case there is a question about your results. The initial concentrations for mixture 1 are already given in the results sheet. The "shower" bench will do mixture 2, the "projector" bench will do mixture 3, and the "fire alarm" bench will do mixture 4.

Once the reaction reaches equilibrium, we assume that the reaction has shifted forward by an amount, x. The equilibrium concentrations of the reactants, $\mathrm{Fe^{3+}}$ and $\mathrm{SCN^-}$, are found by subtracting the equilibrium [redacted] from the initial values. Once all the equilibrium values are known, they can be applied to Equation 2.6.4 to determine the value of K_c.

Table 1:

Reaction 2.6.4	$\mathrm{Fe^{3+}}$	$+ \quad \mathrm{SCN^-}$	$\rightleftharpoons \mathrm{FeSCN^{2+}}$
Initial Concentration	[redacted]$_i$	$[\mathrm{SCN^-}_i]$	0
Change in Concentration	$-x$	$-x$	$+x$
Equilibrium Concentration	[redacted]$_i - x$	$[\mathrm{SCN^-}_i] - x$	$x =$ [redacted]

The reaction "ICE" table demonstrates the method used in order to find the equilibrium concentrations of each species. The values that come directly from the experimental procedure are found in the shaded regions. From these values, the remainder of the table can be completed.

Procedure

Materials and Equipment

Solutions: Iron(III) nitrate (2.00 x 10^{-3} M) in 1 M $\mathrm{HNO_3}$; Potassium thiocyanate (2.00 x 10^{-3} M).

Materials:

Test tubes (large and medium size), stirring rod, two 10- mL graduated pipets, Pasteur pipets, ruler, spectrometers and special-size test tubes at the spectrophotometer.

Safety

The iron(III) nitrate solutions contain nitric acid. Avoid contact with skin and eyes; wash hands and all glassware thoroughly after the experiment. Collect all your solutions during the lab and dispose of them in the proper waste container.

Part A: Solution Preparation

Into a clean, dry 150-mL beaker, pour 30-40 mL of 2.00 x 10^{-3} M $Fe(NO_3)_3$ (already dissolved by the stockroom in 1 M HNO_3). Then pour 25-30 mL of 2.00 x 10^{-3} M $KSCN$ into a clean, dry 50-mL beaker. At your work area, label five clean and dry medium test tubes to be used for the five test mixtures you will make. Add 5.00 mL of your 2.00 x 10^{-3} M $Fe(NO_3)_3$ solution into each of the five test tubes. Next, add the correct amount of $KSCN$ solution to each of the labeled test tubes, according to the table below. Then, add the appropriate amount of deionized water into each of the labeled test tubes. Stir each solution thoroughly with your stirring rod until a uniform orange color is obtained. To avoid contaminating the solutions, rinse and dry your stirring rod after stirring each solution.

Four solutions will be prepared from 2.00 x 10^{-3} M $KSCN$ and 2.00 x 10^{-3} M $Fe(NO_3)_3$ according to this table. Note that the total volume for each mixture is 10.00 mL, assuming volumes are additive. If the mixtures are prepared properly, the solutions will gradually be darker in color from the first to the fourth mixture. Use this table to perform dilution calculations to find the initial reactant concentrations.

Table 2: Test Mixtures

Experiment #	*V* in mL $Fe(NO_3)_3$ Solution	*V* in mL $KSCN$ Solution	*V* in mL Water
1	5.00	1.00	4.00
2	5.00	2.00	3.00
3	5.00	4.00	1.00
4	5.00	5.00	0.00

Part B: Spectrophotometric Determination of [redacted]

To measure the absorbance, transfer your solution into one of the special-size test tubes next to the spectrophotometer, and insert it into the instrument. Once the reading is stable, record it. Then, using the standard curve provided, record the corresponding concentration of the complex. Repeat for all 4 equilibrium mixtures.

Part C: Determine the equilibrium constant

For each mixture, determine all the concentrations at equilibrium (using an ICE table or other method of choice), and calculate the equilibrium constant. If our theoretical assumptions and our experimental data were perfect, you should get the same equilibrium constant for all four measurements. In practice, it probably makes sense to average the four values to get the best estimate of the "real" equilibrium constant.

Part D: Predict the equilibrium concentrations for a new set of starting concentrations

For a solution containing 1.00 x 10^{-3} M Fe^{2+} and 0.60 x 10^{-3} M SCN^-, what is the equilibrium concentration of the $FeSCN^{2+}$ complex? Use an ICE table (or other method) to predict the equilibrium concentration. Also, figure out the recipe to get those initial concentration of iron and thiocyanide ions (experiment *5* in the tables on the summary sheet). Then, make the mixture and test the concentration of complex formed spectrophotometrically to check your prediction.

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2.7: Group I cations

This is a place holder. We did not implement this lab in 2023 (snow day).

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2.8: pH measurement and determination of pKa value

Objectives:

- To measure the pH of various solutions using pH indicators and meter.
- To determine the value of K_a of acetic acid in three different ways.
- To create and study the properties of buffer solutions.

Part A: Using Indicators to Measure pH

In this part of the experiment you will use five indicators to determine the pH of four solutions to within one pH unit. An **acid-base indicator** is a chemical species that changes color at a specific pH as the pH (acidity) of the solution is varied. Acid-base indicators are themselves weak acids where the color of the aqueous acid is different than the color of the corresponding conjugate base. We can represent the dissociation of an acid-base indicator in an aqueous solution with the following equation.

$$\underbrace{\mathrm{HIn\,(aq)}}_{\text{yellow}} + \mathrm{H_2O\,(l)} \rightleftharpoons \underbrace{\mathrm{In^-\,(aq)}}_{\text{blue}} + \mathrm{H_3O^+\,(aq)} \tag{2.8.1}$$

In this hypothetical example In stands for the indicator. As you can see from Equation 2.8.1, the protonated form of the acid-base indicator, HIn (*aq*), will be one color (color I, yellow in this example) and the deprotonated form, $\mathrm{In^-}$ (*aq*), will be another color (color II, blue in this example). The equilibrium-constant expression for Equation 2.8.1 is:

$$K_a = \frac{[\mathrm{H_3O^+}][\mathrm{In^-}]}{[\mathrm{HIn}]} \tag{2.8.2}$$

This equation can also be written as:

$$\frac{K_a}{[\mathrm{H_3O^+}]} = \frac{[\mathrm{In^-}]}{[\mathrm{HIn}]} \tag{2.8.3}$$

Generally only one or two drops of indicator are added to the solution of interest and therefore the amount of $\mathrm{H_3O^+}$ due to the indicator itself can be considered negligible. The total amount of $\mathrm{H_3O^+}$ in the solution is therefore controlled by the concentrations of the other acids and/or bases present in the solution. Note that when $[\mathrm{H_3O^+}] >> K_{ai}$, $[\mathrm{HIn}] >> [\mathrm{In^-}]$ and the color of the solution will be essentially the same as color I. Similarly, when $[\mathrm{H_3O^+}] << K_a$, $[\mathrm{HIn}] << [\mathrm{In^-}]$ and the color of the solution will be essentially the same as color II.

As an example consider a very acidic solution (say pH = 0) containing the indicator HIn where $[\mathrm{H_3O^+}] >> K_a$, and therefore, $[\mathrm{HIn}] >> [\mathrm{In^-}]$. Under these conditions the solution will be yellow. Suppose we add base to the solution resulting in a decrease of $[\mathrm{H_3O^+}]$. As $[\mathrm{H_3O^+}]$ decreases the reaction indicated by Equation 2.8.1 will go net forward and $[\mathrm{HIn}]$ will decrease while $[\mathrm{In^-}]$ increases. When $[\mathrm{In^-}]$ becomes significant compared to $[\mathrm{HIn}]$ the color of the solution will begin to change. Eventually as $[\mathrm{H_3O^+}]$ decreases still further we will have, $[\mathrm{H_3O^+}] << K_{ai}$, and the color of the solution will have turned to blue. Depending on the indicator, this could happen while the solution is still acidic, only less so. In other words the solution will change color when $[\mathrm{HIn}] \approx [\mathrm{In^-}]$, and so $K_a = [\mathrm{H_3O^+}]$, or $pK_a = pH$. In general we can say that an acid-base indicator changes color at a pH determined by the value of K_a or $\mathrm{pK_a}$ for that particular indicator.

The five indicators you will use in this experiment, their color transitions, and their respective values of $\mathrm{p}K_{ai}$ are given in Table 1.

Table 1: Acid-Base Indicators

Indicator	pK_{ai}	pH and color transition (0 1 2 3 4 5 6 7)
methyl violet	0.8	yellow ⟷ blue-violet
thymol blue*	1.7	red ⟷ yellow
methyl yellow	3.3	red ⟷ yellow
congo red	4.1	violet ⟷ orange-red
bromcresol green	4.9	yellow ⟷ blue

*The actual colors in solution vary somewhat from those shown here depending on the concentration. *Thymol blue is a polyprotic acid with two pK_a values. The second pK_a is around 8.8. Adding too much NaOH, to a pH beyond its second pK_a results in a colorless solution.*

We can use the values in Table 1 to determine the approximate pH of a solution. For example, suppose we have a solution in which methyl violet is violet. This tells us that the pH of our unknown solution is greater than or equal to 2 because methyl violet turns violet at pH values of 2 or greater. Now suppose we add some congo red to a fresh sample of our solution and find that the color is violet. This tells us that the pH of our solution is less than or equal to 3 because congo red turns violet at pH values of 3 or less. From these two tests we know that the pH range our solution is between 2 and 3. Thus, we have determined the pH of our solution to within one pH unit. Proceeding in a similar manner, you will use the acid-base indicators in Table 1 to determine the pH range of four solutions to within one pH unit.

Part B: Using pH meters

In this part of the experiment you will learn to use a pH meter to measure pH. Your instructor will demonstrate how to use the pH meter appropriately at the beginning of your laboratory session, explaining why the pH probe should always be submerged in a solution, and how to avoid contamination of a sample with the one measured previously.

Part C: Using pH to determine the value of K_a for acetic acid, $\mathrm{CH_3COOH}$(aq)

In this part of the experiment you will use your pH meter to measure the pH of an acetic acid solution of known concentration, and an acetate solution (the conjugate base) of known concentration. From the measured pH and concentration you can determine the value of K_a for the acid. The general equation for the dissociation of a weak acid, HA (*aq*), in water is:

$$\mathrm{HA\,(aq) + H_2O\,(l) \rightleftharpoons A\,(aq) + H_3O^+\,(aq)} \tag{2.8.4}$$

For which

$$K_a = \frac{[\mathrm{A}][\mathrm{H_3O^+}]}{[\mathrm{HA}]} \tag{2.8.5}$$

When we construct an ICE table for this reaction we can see that at equilibrium

$$[\mathrm{A^-}] = [\mathrm{H_3O^+}] \tag{2.8.6}$$

and

$$[\mathrm{HA}] = [\mathrm{HA}]_0 - [\mathrm{H_3O^+}] \tag{2.8.7}$$

where $[\mathrm{HA}]_0$ is the initial (nominal) concentration of HA (*aq*) before equilibrium is established.

Using Equations 2.8.6 and 2.8.7 , we may express Equation 2.8.5 as

$$K_a = \frac{[\mathrm{H_3O^{+2}}]}{[\mathrm{HA}]_0 - [\mathrm{H_3O^+}]} \tag{2.8.8}$$

Because $[\mathrm{H_3O^+}]$ can be determined by measuring the pH of the weak acid and $[\mathrm{HA}]_0$ is known you can determine the value of K_a using Equation 2.8.8.

For the pH measurement of the acetate, hydroxide rather than hydronium is a major species, so it makes sense to use a different version of the acid/base reaction for the ICE table:

$$\mathrm{AH\,(aq) + OH^-\,(aq) \rightleftharpoons A^-\,(aq) + H_2O\,(l)} \tag{2.8.9}$$

So the ICE table starting with the conjugate base is similar but should include hydroxide instead of hydronium. Once you figure out the equilibrium concentrations of acetic acid and acetate, you can plug in those as well as the hydronium concentration to calculate the K_a using Equation 2.8.5.

Part D: Buffer Solutions

In this part of the experiment you will prepare a buffer solution with a certain pH from solutions of 0.1 M acetic acid and of 0.1 M acetate (sodium as counter ion).

Recall that the pH of a buffer solution is given by the Henderson-Hasselbach approximation:

$$\mathrm{pH} = \mathrm{pKa} + \log \frac{[A^-]}{[HA]} \tag{2.8.10}$$

This can be accomplished using Equation 2.8.10 to determine the ratio, $\frac{[\mathrm{A^-}]}{[\mathrm{HA}]}$, that will produce the specified pH of the buffer solution. You will confirm the pH of this solution using your pH meter.

Finally, you will compare the buffering capacity of the buffer you prepare with that of deionized water.

Procedure

All experiments will be done in pairs. This way, you can compare your calculations before you do the next step. One way to organize group work is having one student to the experiment and another writing down results. It might make sense to do calculations outside of the lab after taking off gloves and goggles.

Materials and Equipment:

You will need the following additional items for this experiment: pH meter. There will be one shared one at every bench (so 3 pairs will share a pH meter).

Safety

Students must wear safety goggles when in the lab. Do not leave the lab with gloves on.

GLOVES: Gloves are needed when handling:

- zinc sulfate
- sodium carbonate
- sodium bisulfate
- methyl yellow
- congo red
- bromocresol green

WASTE DISPOSAL: All chemicals used must go in the proper waste container for disposal.

Part A: Determination of pH using Acid-Base Indicators

1. Rinse five small test tubes using deionized water (there is no need to dry these). To each of these test tubes add about 1 mL of 0.1 M HCl (*aq*). (Estimate these volumes). To each of these test tubes add one of the five indicators listed in Table 1. Be sure to arrange or label your test tubes so that you know which indicator was added to which tube.
2. By comparing the colors you observe in each tube you should be able to determine the pH of the 0.1 M HCl solution to within one pH unit (see background discussion). Record your color observations and your determination of the pH range of the 0.1 M HCl solution on your data sheet.
3. Repeat the same procedure using each of the following solutions:

   0.1 M sodium hydrogen phosphate, $\mathrm{NaH_2PO_4}$ (*aq*)

   0.1 M acetic acid, $\mathrm{CH_3COOH}$ (*aq*)

   0.1 M zinc sulfate, $\mathrm{ZnSO_4}$ (*aq*)

4. Record your results for each on your data sheet.

Part B: Using pH Meters

Your instructor will demonstrate the proper use of the pH meters.

1. Rinse four small 100 or 150-mL beakers several times using deionized water. Do not use any soap as the residue may affect your pH measurements.
2. Into each of your four clean beakers collect about 30 mL of one of the following:

   0.1 M sodium chloride, NaCl (*aq*)

   0.1 M sodium carbonate, $\mathrm{Na_2CO_3}$ (*aq*)

   0.1 M sodium acetate, $\mathrm{NaCH_3COO}$ (*aq*)

   0.1 M sodium hydrogen sulfate, $\mathrm{NaHSO_4}$ (*aq*)

3. Use your pH meter to determine the pH of each of these four solutions. Record the results on your data sheet. Add a drop or two or bromcresol green indicator to each of these solutions. Record the color of the indicator in each solution on your data sheet.

Part C: Using pH to Determine the Value of K_a for Acetic Acid, $\mathrm{CH_3COOH}$ (*aq*)

1. Rinse two small 100 or 150-mL beakers as before. Put 30 mL of 0.1 M acetic acid solution into the first beaker and 30 mL of 0.10 M sodium acetate solution into the second.
2. Use your pH meter to determine the pH of each solution. Record the results on your data sheet. You will use these values to calculate K_a.

Part D: Buffer Solutions

First, verify the pKa measured in part C by combining 10 mL each of 0.1 M acetic acid solution and of 0.1 M acetate, and measuring the pH of this "1:1 buffer".

Each student pair will try to get a slightly different pH value for their buffer: The pair closest to the windows will try to set the pH to be 0.1 units higher (more basic) than the pKa, the pair closest to the white board will try the set the pH 0.2 units lower (more acidic) than the pKa, and the remaining pair at a given bench will try to set the pH 0.3 units higher (more basic) than the pKa.Then, change the pH to the desired pH by adding the appropriate volume of one of the stock solutions.

You have to figure out which one, and how much you have to add. Make the new buffer solution and use your pH meter to measure the pH of your buffer solution and see how much it deviates from the assigned value.

Now we will test the buffer solution you prepared against changes in pH.

1. Rinse and fill another 150-mL beaker with a volume of deionized water equal to that of your buffer solution. Using your pH meter measure the pH of the deionized water. Do not be alarmed if this pH is less than neutral. It should be between 5.2 and 7.0. *Do you know why? What species from the atmosphere dissolves in water that is left standing?*
2. Add 1 drop of 0.1 M HCl solution to both the beaker containing your buffer solution and that containing the deionized water. Measure the pH of each of these solutions following this addition and determine the change in pH of each. Record these values on your data sheet.
3. Clean up. Discard all chemicals in the proper chemical waste container. Clean and then return all borrowed equipment.

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2.9: Making a buffer with three components

Your job today is to make a buffered solution. We are not giving you the procedure, just the final concentrations in the solution. You will receive a "recipe card" such as the one below.

Solution B5 Make 50 mL of a buffer containing: · 50 mM sodium citrate, · 20 mM HBr · 100 mM KCl

Please work carefully and check your calculations. Other students (in this and other sections) will use this buffer next week for a colorimetric lab.

Research labs and industrial labs typically have a variety of solutions on hand to do their daily work. Often, these are made by one person and used by many. When hiring new people to work in the lab, one crucial question is whether they are able to make solutions that others can use with confidence.

This lab has two parts. In the first part, you are responsible for figuring out a recipe for your solution and for making it. In the second part, you team up to characterize your solutions, and to submit a solution you feel is closest to the specifications.

You have the following materials available:

- Pure sodium acetate trihydrate ($NaCH_3COO{\cdot}3\ H_2O$, powder, molar mass is ~136 g/mol)
- Pure sodium phosphate, monobasic monohydrate
- ($NaH_2PO_4{\cdot}H_2O$, powder, molar mass is ~138 g/mol)
- Solution of hydrochloric acid, HCl, concentration is 200 mM (or value on the bottle)
- Solution of sodium hydroxide, NaOH, concentration is 200 mM (or value on the bottle)
- Solution of sodium chloride, NaCl, concentration is 2.00 M
- Solution of sodium chloride, NaCl, concentration is 0.200 M

Part one: individual tasks

Record your calculations and measurements in the data sheet.

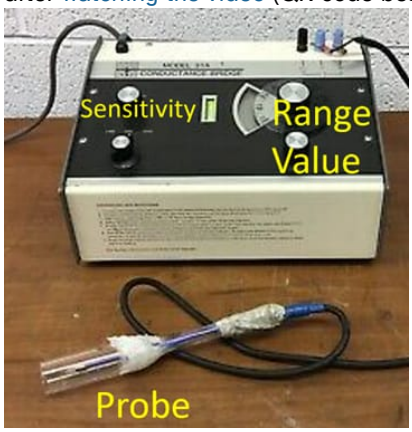
1. **Formulate a recipe** to make your buffered solution. You will weigh out a certain mass of your buffer substance, add strong acid or base (this will adjust the pH), add sodium chloride and water. Choose which NaCl stock solution you will use to minimize volume errors.
2. **Make the buffer**, using your recipe and checking off steps as you go. If there are any unexpected issues, take notes. Also, record the exact mass you used (it is fine if it is a couple of milligrams different from your recipe).
3. **Calculate the estimated pH** of your solution after looking up the relevant pKa value.

Tasks (working together)

1. Find the other individual(s) working on the same solution. Compare the recipes you used and the pH values you calculated. While the recipes might differ a bit, the calculated pH should be the same.
2. **Measure the pH** of your two solutions. You have measured pH values during lab last week. Use the same procedure, making sure the electrode is rinsed between uses. If you missed the lab, ask for a demonstration before you use the pH meter. The pH is sensitive to concentrations of acids and bases added (as well as which acid or base you added). The same recipe should always give the same or a similar pH. For comparisons, it is best to use the same pH meter (they might be calibrated

slightly differently).

3. **Measure the conductivity** of your two solutions: The conductivity is influenced by which ions are present at which concentration. Identical solutions should have the same conductivity. For the solutions we are making today, the biggest influence on conductivity is the concentration of sodium chloride. Inconsistencies in the sodium chloride concentration would show as a large difference in conductivity but only a small difference in pH (because NaCl is neither an acid nor a base). We are using an analog instrument that is a bit quirky. The following instructions will make more sense when you are at the instrument or after watching the video (QR code below).



For the conductivity measurement, make sure the probe and the provided 50 mL tube are rinsed with water before they come in contact with you solution. Transfer your solution into the 50 mL tube and insert the probe. For an accurate measurement, the probe has to be submerged past the line. Then, choose the appropriate measurement interval using the range dial while the sensitivity knob is turned all the way counterclockwise. Finally, increase the sensitivity, adjust the value dial to maximize the gap in the display, and read off the result on both dials. Don't forget to retrieve your solution once you are done. There are two sample solutions provided along with the measurements if you need to troubleshoot.

4. Enter the data in the data table in your report, and discuss how different the measurements are for the different solutions. Are the differences within the range you expected? If not, what might have happened?
5. Decide what you want to save for next week. You can either submit a mixture of your solutions (this would average out random errors) or decide to submit a single solution and dump the rest. If you discover that all solutions made had some "fatal flaw", you can also make another solution together and submit that.
6. Place your buffer in a 50 mL screw cap tube. Make a label, indicating the concentrations of all components, the data and your (first) names, and tape it on the tube. Then, put your solution in the provided Styrofoam rack in the hood.

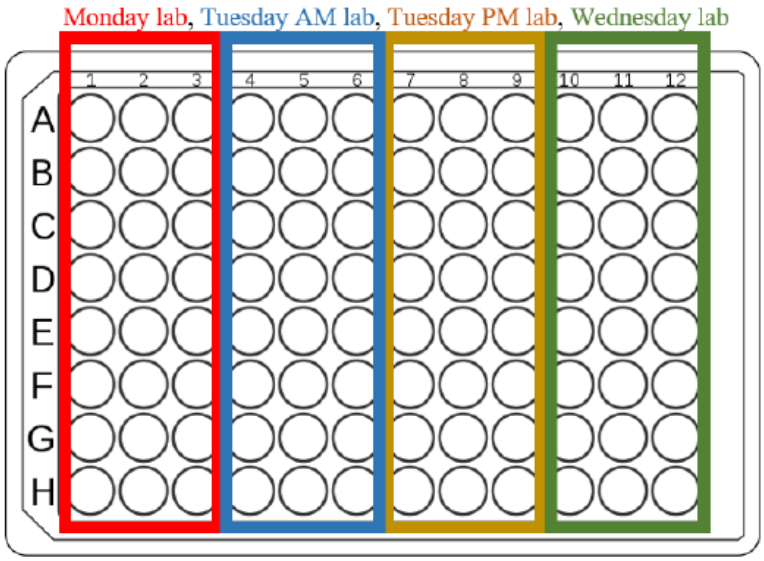
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2.10: Colorimetric pH measurement

Your task today is to estimate the pH-values of six solutions using an indicator, and then getting a better estimate by measuring the absorbance of the indicator at two different wavelengths. This dual-wavelength method is used commercially, for example in Pulse Oxymeters to measure oxygen saturation of hemoglobin in our blood, shining light through our fingertip. Similar techniques are also used to measure concentrations or pH in living cells.

The reason we are using two wavelengths is to figure out the hue of the color irrespective of its intensity. Said in another way, the two-wavelength measurement will give the same result for different concentrations of the indicator, as long as the ratio of protonated to deprotonated is the same.

For this experiment, we will be storing and measuring solutions in a 96-well plate. These are used in situations where you have a lot of samples of similar type, and are designed for automation. Each group will have their own plate, but it is used in the other sections too, so please only use the designated well:



Tasks (you are working in pairs)

There are two data sheets; one of you will use the first, and one of you will use the second. Use the first data sheet for tasks 1-8. Then, switch recorder and use the second data sheet for tasks 9 and 10. Finally, record your critical evaluation of the experiment on the back side of your data sheets.

1. Using a transfer pipette, place approximately 250 µL of each buffer you are testing in two adjacent wells in the microplate(e.g. first column and second column, rows A-F). Make sure not to contaminate (cross-contaminate) the buffers, as they will be used by other lab sections as well.
2. Place one drop of universal indicator into the six solutions in the first column. Determine the pH range of your solution (by comparing with the standards), and choose whether to work with phenol red (color change at about pH 7) or methyl red (color change at about pH 5 to 5.5). Communicate that choice to your instructor.

3. Place one drop of the chosen indicator into the buffers in the second column.
4. The exact pKa of the indicator will depend on the temperature, the solvent, the ionic strength (the concentration and charge of ions present), so we will measure it using a standard. There are four standards available (pH 5 or pH 8, low or high ionic strength). Choose the one most appropriate and consult your instructor. Place one drop of the chosen indicator into three empty wells (e.g. row H), and add 250 µL each of your pH standard solution of choice.
5. Place the microplate in the reader, measure the absorbance at two wavelengths (540 nm, 405 nm, already set) and record the absorbance. You will have to use the menu to switch the display between the two wavelengths (if you have a way of taking two pictures, that is the most reliable and fast method).

6. Using the calibration curve for the indicator (there is one for methyl red and a different one for phenol red), estimate what fraction is protonated and what fraction is deprotonated for each of the nine samples. To do this, first calculate (A540 – A405) / (A540 + A405), then read off the percentage of deprotonated indicator. The percentage of protonated indicator is 100% minus that value.
7. Estimate the pKa of your indicator from the three solutions with known pH. You will use the Henderson-Hasselbalch relationship with the pKa as an unknown (see sample calculation 1). Use the average of the three values as your best estimate of the pKa.
8. Now, again using the Henderson-Hasselbalch relationship, calculate the pH of the six buffers with unknown pH (see sample calculation 2). The pKa in the formula refers to that of the indicator, and [HA]/[A-] to the ratio of protonated to deprotonated indicator.

   Switch to the other data sheet.
9. Add 100 µL of 50 mM HCl to each of the solutions made in task 3 (second column, the ones with methyl or phenol red). Visually, which changed color the most?
10. Measure the absorption and calculate the pH. Which solution had the largest change in pH? What factors determine how much the pH changes? Do your measured pH shifts match your prediction?
11. Find the other group working on the same solutions. Compare your results to theirs and to the measurements made with a pH electrode last week.

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2.11: Hypothesis testing

Safety and hazardous waste

At the beginning of this lab, you will observe an experiment (a "magic trick") without knowing the substances it contains. This experiment will change every year, with different safety considerations. Over the semester, we have handled acids and bases, flammable substances, toxic substances, gases, high and low temperatures, etc. Observe all precautions you observed for previous labs. If there are any special considerations, your professor will announce them at the beginning of the lab. Do not discard any of the solutions until you have received instructions from your professor.

Lab organization

We will explore a "magic trick" that is based on chemical reactions. You can use all of the concepts you learned in GenChem1 and GenChem2 to try to explain what is going on. You will work in three large groups which you can organize as you wish. Your group will present twice during the lab, first announcing your initial hypothesis and observations supporting or rejecting it (about 1 hour into the lab). You will again report out at the end of the lab, presenting your updated hypothesis. Individually, your job is to write a short report stating the hypothesis. This year, we will look at the blue bottle experiment, where a blue color appears (the dye methylene blue) and then disappears again.

Part 1

Your group will receive 25 mL of the magic solution in a screw-cap 50 mL vial. The solution contains 0.5 mol/L sodium hydroxide, a strong base you should only handle while wearing goggles. To start the trick, you shake the closed vial. Before you do so, make sure everyone is wearing their goggles, and the lid is screwed on finger-tight. After you tried the magic trick, consider the following questions:

1. What type of reactions might the "bluing" and "debluing" reactions be? We learned about three types of reactions in GenChem1: Acid/base, redox, and precipitation. What kind of experiment/observation could help you decide?
2. What role could the shaking step have in starting the reaction? Could you do something other than shaking to get the same effect? What kind of experiment/observation could help you decide?

You will have various equipment available (pH meter, pH paper, capped vials with a size between 1 mL and 50 mL, a magnetic stirrer and stir bar, a vortex mixer). Design experiments you can do with the given 25 mL of magic solution. Discuss the experiments with your instructor to make sure you are aware of all the safety considerations. Once you get the go-ahead, do the experiments and record your observations.

Once you are done, prepare a short presentation where you share the experiments you did and your conclusions from them with the entire class. This is a "chalk talk", so you just talk and use the white board, if necessary.

Part 2

For this part, you will have the reagents available separately as three solutions A, B, and C. Solution A contains the strong base. This allows you to leave out reagents or change their concentration. For this part, there are three questions you should address:

1. Which reactants are necessary for debluing, and which are necessary for bluing?
2. Choose one of the following
   a. How does lowering the concentration of components in solution A added change things? Is the content of B a reactant, a catalyst, or neither?
   b. How does lowering the concentration of components in solution B added change things? Is the content of B a reactant, a catalyst, or neither?
   c. How does length of shaking/mixing change things? Does the shaking add a reactant, an catalyst, or neither?
3. What is your model of the bluing and of the debluing reaction? Which reagents do they require?

4. How fast is the debluing reaction compared to the bluing reaction?

First, repeat the magic trick, using the three solutions (mix 8 parts of A with 1 part of B and 1 part of C). Then decide on a set of experiments to address the questions. Discuss the experiments with your instructor to make sure you are aware of all the safety considerations. Once you get the go-ahead, do the experiments and record your observations.

Once you are done, prepare a short presentation where you share the experiments you did and your conclusions from them with the entire class. This is a "chalk talk", so you just talk and use the white board, if necessary.

Part 3

You can do the same magic trick by mixing solution A (the strong base) with blue sports drink. Try mixing 15 mL of the blue sports drink with 5 mL of 2 mol/L NaOH in a 50 mL vial (under the hood), cap the vial and swirl to see what happens to the color. Then, shake and observe. What ingredients in the sports drink correspond to the ingredients in solution B and C, making this variation of the trick possible?

Asking for reagents, supplies and equipment

In your exploration, you might suggest an experiment for which you need certain materials not available to you. If there is something you have used in previous experiments (thermometer, pipette pump, etc), your instructor might make it available to you. You can also ask for anything else, but don't be disappointed if your request is declined. All experimenters, even in highly funded research labs, will have real world limitations (too expensive, too dangerous, tool has not been invented yet, limited amount of sample) determining which experiments they can and which they can't pursue.

Cleanup

Discard reaction mixes in the container provided under the hood. As you rinse the tubes, remember that you are still handling 0.5 mol/L NaOH. This is a strong base, and you have to protect your eyes even while cleaning up. As a mental exercise while rinsing, try to estimate the pH of the reaction mix without a calculator (hint: the concentration is in between 0.1 mol/L and 1 mol/L).

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2.12: Preventing rust

Acknowledgement

This activity was developed based on a procedure developed by the Royal Society of Chemistry together with the Nuffield foundation.

Introduction

Some redox reactions are useful, such as the ones in our cells providing the Gibbs energy to make ATP, or those charging a battery and providing electricity from a charged battery. Many steps in making desired products, from aluminum to medications, involve redox reactions. Some redox reactions, however, are undesirably and very costly. The prime example is corrosion or rust-formation, a reaction of metals (in case of rust, specifically iron and steel) with oxygen. As you know, oxygen is about 20% of the gases in air. Oxygen also dissolves in water, which allows fish to breath. Typical conditions that promote rust are the presence of water and salt - ship wreaks in the ocean rust quickly, providing an opportunity for marine life to settle in.

Today's experiments explore how we can avoid formation of rust. Rust is a complex substance containing iron(III), but initially $\mathrm{Fe^{2+}\,(aq)}$ forms. We will detect the formation of this ion with an indicator (potassium hexacyanoferrate) that forms an intensely blue-colored substance called Prussian blue when it combines with iron(II). In this way, we don't have to wait days to see the rust itself.

$$\mathrm{Fe\,(s) \longrightarrow Fe^{2+}\,(aq) + 2\,e^-} \tag{2.12.1}$$

$$\mathrm{Fe^{2+}\,(aq) + indicator \longrightarrow Prussian\ blue} \tag{2.12.2}$$

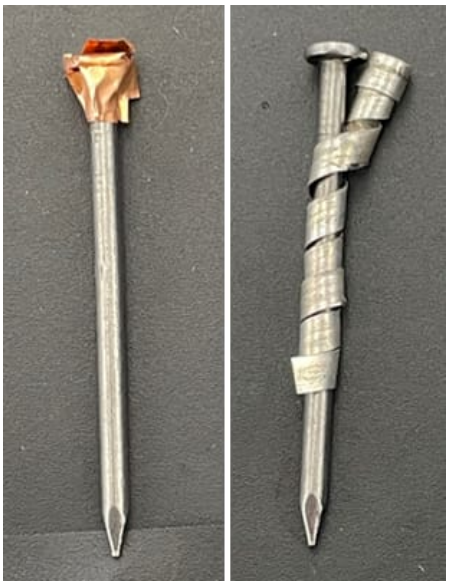
Our reaction mix contains gelatin that will set when cold. This prevents mixing and helps us see where the redox reaction occurs.

Part 1: setup

The reactions will be done in small test tubes provided. You will work in groups of two, preparing a total of 5 test tubes. Add the following to separate tubes:

1. an untreated iron nail (common nail, or bright nail, made of steel)
2. an iron nail with some magnesium strip wrapped around it
3. an iron nail with some copper strip wrapped around it

The magnesium is brittle, and both the magnesium and copper strip tend to slide off the nail if you just wrap the body of the nail. Here are two techniques that allow you to attached the metal band securely while increasing the chance of electrical contact between the iron in the nail (left, copper; right, magnesium first wrapped around body, and the coil was bent to thread the nail as shown):



For test-tube 4 and 5, every bench will try a different treatment to prevent rusting

"Shower bench"

- wrap an iron nail in saran wrap and place it into the 4th test tube
- use a stainless steel nail (these are more expensive because it contains other elements such as chromium and nickel) in the 5th test tube

"Middle bench"

- paint an iron nail, let it dry, and place it into the 4th test tube
- take a galvanized nail (coated with zinc), score it with the provided handsaw in two places, and place it into the 5th test tube

"Fire bench"

- cover an iron nail in oil and place it into the 4th test tube
- cover an iron nail in butter and place it into the 5th test tube

Once the 5 test tubes contain nails, pour hot gelatin solution (containing the indicator for iron(II)) into the test tube to a level that covers the nail completely. This will set after a couple of minutes. Once set, the redox reactions will continue, but convectional mixing will no longer occur, making it easier to see where exactly the rust forms.

Part 2

Formulate the half reactions and net redox reactions for test tube 2 and 3 on your data sheet. Here is an example for the reaction of the redox pairs $\mathrm{OH^-/O_2}$ and $\mathrm{H_2/H^+}$:

$$\mathrm{2\,H^+\,(aq) + 2\,e^- \longrightarrow H_2\,(g)} \quad E^\circ = 0V \tag{2.12.3}$$

$$\mathrm{O_2\,(g) + 2\,H_2O\,(l) + 4\,e^- \longrightarrow 4\,OH^-\,(aq)} \quad E^\circ = 0.4V \tag{2.12.4}$$

The direction of the reaction is determined by the reduction potentials. In this case, the top reaction will go in the reverse direction (oxidation), and the bottom reaction as written, giving a positive cell potential. The combined reaction (after doubling the top reaction so that electrons cancel) will be:

$$\mathrm{O_2\,(g) + 2\,H_2O\,(l) + 2\,H_2\,(g) \longrightarrow 4\,OH^-\,(aq) + 4\,H^+} \quad E^\circ = 0.4V \tag{2.12.5}$$

Relevant half reactions and reduction potentials are available in your textbook.

Part 3

Go to one of the stations and explore the electrolysis of water (we might have added some salt to improve conductivity, and we added universal indicator to visualize pH changes). There are two half reactions, one turning water into hydrogen gas and the other into oxygen gas, while changing the pH:

$$2\,H_2O + 2\,e^- \longrightarrow H_2\,(g) + 2\,OH^-\,(aq) \tag{2.12.6}$$

$$H_2O \longrightarrow \tfrac{1}{2}\,O_2\,(g) + 2\,e^- + 2\,H^+\,(aq) \tag{2.12.7}$$

Figure out which electrode is the anode and which is the cathode based on the color changes, and determine whether electrons flow from the (+) to the (-) terminal at the battery or vice versa. When you are done, mix the solution so that the pH is equal to 7 throughout again.

Part 4

Write down your observations from the reactions set up in part 1. Compare your observations for test tubes 4 and 5 with the other students at your bench, and plan a short presentation that includes projecting your results using the document camera in the lab. While the other groups are presenting, take notes.

Part 5

Check out if there is time. If there is no time, make an appointment for checkout next week.

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3: Lab safety and safe operating procedures

Chemical hygiene plan, Westfield State University

ACS safety videos, 21st century

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GLOSSARY

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