

General Chemistry Lab Manual
College of Alameda

This text is a revision of the Laney College Lab Manual, which was written mostly by Bill Scott and Charles Keilin, and previously revised by Cheli Fossum and Jennifer Loeser. Some experiments (Experiments 1, 5, 6, 12 and 13) are new, as are all Pre-Lab sheets. This revision and the new experiments were prepared by Emily Eames in 2015, with comments from Peter Olds and Eileen Clifford.

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Chem 1A Lab Notebook Guidelines

Clarity and completeness count! Leave plenty of space between lines so it's easy to read.

Each notebook entry for an experiment should include the following sections, clearly labelled:

Date of experiment

Title of experiment

Lab partner

Objective: in one to four sentences, summarize the goal of the experiment. What are you trying to measure? Be very specific. For instance, if you are measuring a property of a substance, make sure you say which property of what substance. Also summarize what technique or measurements you will use to achieve this goal.

Procedure: You don't need to copy the procedure from the lab manual, but write down anything that happens that isn't in the lab manual. Record observations (eg, lots of smoke, color changes, etc), mistakes you made (spilled a little, didn't stir continuously, etc), and anything else that might affect your results.

Data: any measurements you made, and other quantitative or qualitative results. All data should be clearly labelled so anyone can tell what it is. Numerical data must include units and correct significant figures based on how you collected the data. Data should be in clear, organized tables if there are more than a few results. For any experiment in which you do multiple trials, you should collect all the data in a clearly-labelled table. When you set up the table, check what calculations you'll need to do and leave space to include the results in the table so you can look at everything at once.

Calculations: you must show at least one example of each type of calculation, written out step-by-step, fully and clearly in logical order. Start with standard formulas, rearrange them to isolate the desired quantity (using variables, not numbers), show the numbers plugged into the formula with correctly canceling units, and the final answer with unit and correct significant figures. For useful formulas and definitions, see the section on Data Analysis.

Results: Your final, calculated results can be included in the data table or simply labelled on separate lines, depending on the circumstances. If you did multiple trials, it's most convenient to include calculation results in the original data table so you can see everything at once. If you made multiple measurements of a single value, report the value as "average \pm uncertainty"; for the uncertainty use 2 times the average deviation. The last reported decimal place for the average should be the same as the uncertainty. (More detail in the Data Analysis section.)

Chem 1A Lab Grading and Policies

(Unless otherwise specified by your instructor)

Each lab is worth 40 points: 10 points for attendance and lab conduct, and 30 points for written work (usually 10 points for the Pre-Lab and 20 points for the Post-Lab). One lab will be dropped, so you may miss one lab without penalty. The second missed lab will count as zero (no written work may be submitted if you do not attend the lab), and **you will automatically fail the whole class if you miss three labs.**

Written work: Please complete the written work before coming to class/lab. Sometimes it will require the use of a computer. **You must complete written work by yourself in your own words.** If multiple assignments have identical wording, neither student will receive points for those questions.

Attendance and Conduct: You will receive 10 points for each lab where you show up on time, fully prepared, observe safety rules and stay until the instructor is satisfied that you have completed the lab. You will lose points for tardiness, poor preparation, not wearing safety glasses, bringing food or water inside the lab, leaving without cleaning up, etc.

Lab Preparation: In order to complete the lab in a safe and timely fashion, you need to arrive prepared. **The instructor may refuse you participation in the lab if you arrive more than 15 minutes late, or if you arrive unprepared (without a Pre-Lab sheet, for example).**

Pre-Lab Sheets: The Pre-Lab sheets are intended to ensure that you prepare adequately before coming to lab. They will alert you safety issues and common mistakes that you should be aware of. They will also give you a chance to practice the relevant calculations. **To get credit for Pre-Lab sheets, they must be submitted within 15 minutes after the beginning of the lab.** You may forget your Pre-Lab sheet once during the semester without penalty, but if you do not bring a Pre-Lab sheet a second time your instructor may not permit you to participate in the lab.

Post-Lab Sheets: The Post-Lab sheets for each experiment are due the next lab session, one week after the experiment was completed. You may submit one late post-lab during the semester without penalty. Please complete them at home, in your own words.

Lab Partners: Some experiments are performed alone, but most are intended to be performed with a partner. **You may switch lab partners at any time, for any reason.** If you find that your partner does not participate, or does not allow you to participate fully, you are encouraged to speak to the instructor and change partners.

Lab Preparedness Checklist:

If you have questions about the procedure, pre-lab, post-lab, etc, please contact your instructor before the lab (such as by email, before class, office hours). You don't have to do all this completely on your own: you are encouraged to get help from the instructor!

- You have your lab notebook, pen, calculator, Lab Manual, Pre-Lab sheet, goggles.
- You are wearing long pants or skirt and closed-toed shoes.
- If you have long hair, it is tied back.
- In your notebook, you have written the title and detailed, specific objectives of the experiment.
- In your notebook, you have set up a table for your data, if appropriate. The table includes space for all the raw data you will collect, and the calculated results.
- You have read the complete experiment in the Lab Manual.
- You have **completed** the Pre-Lab sheet. If you left any questions blank, you are not prepared!
- You can explain clearly, in your own words, the purpose of the experiment, how and why you will make all the measurements, and how you will use them in the calculations. You are ready to answer basic questions about the techniques that will be used.

Lab (Safety) Rules:

- You must wear goggles or safety glasses **at all times** in the lab (even if you are just doing calculations!) unless your instructor gives you permission to take them off.
- You must wear long pants or skirt and closed-toed shoes in the lab.
- If you have long hair, it must be tied back.
- You may not have any food, drink, gum, etc in the lab. (If it is in your backpack, don't take it out!)
- Be careful, move slowly, and don't fool around in the lab.
- If you spill chemicals or break glassware, clean it up (ask for help if you are nervous or unsure how—definitely ask for help for any large spills or strong acid/base spills)
- Be careful with the analytical balances, try not to spill anything on them.
- Once you have removed a chemical from a shared source bottle, do not ever put it back in. Discard excess or put it in specially-labelled containers.
- Lighting Bunsen burners: with a match, light the match before turning on the gas, and bring the match up along the side of the tube so you don't burn yourself. With a striker, check to make sure you can produce sparks with the striker, then turn on the gas and use the striker.
- When you are done for the day, rinse your glassware with water, wipe your bench area with wet paper towels and put away everything you used.
- If a reagent is stored in the hood, you should probably leave the bottle there!
- Dispose of waste as instructed in the lab manual or by your instructor.
- Be careful with strong acids and bases (anything your lab manual warns is corrosive). Leave the bottles in the hood, and keep them on the trays to contain spills.

Significant Figures and Raw Data

Significant Figures (**sig figs**) are a way to record and communicate how carefully you made measurements in the lab. When recording data, it's important to use the right number of digits. Imagine you read the temperature on a thermometer. You're in a hurry, and you glance at it and see that it is closer to 23 °C than to the other lines, so you write down 23 °C. Now imagine that you are being careful and conscientious. You are going to read the thermometer to 1 decimal place. You study the thermometer and see that it is exactly on the line at 23 °C, or 23.0 °C. You record 23 °C, because the decimal place is just a zero. Is this correct? Not quite: when you look back later, you won't know whether you were careful or not careful. If you read the thermometer to 1 decimal place, you should record that decimal place, even if it's just a zero. That zero is significant, because it distinguishes between carefully reading the thermometer and *not* carefully reading the thermometer.

Sig figs are the meaningful part of a number. The rule is that you should include exactly 1 uncertain digit when you report a number. In our previous example, when you look at the thermometer carelessly and it seems close to 23 °C, the 2 is certain because it is almost certainly twenty-something degrees. The 3 is uncertain because it might have been in between 22 and 23, or 23 and 24. The actual temperature might really be closer to 22 or 24, so the last digit is uncertain. When you read the thermometer carefully and it looks like 23.0 °C, it's still kind of hard to estimate that decimal place, so it might be 23.1 °C, or 22.9 °C. When we write 23.0 °C, we're still including 1 uncertain digit: the 0 is uncertain, but now the 3 is certain.

Here are the rules for counting sig figs in a number:

- Any non-zero digit is significant (4.5 g: 2 sig figs)
- zeros between non-zero digits are significant (102 g: 3 sig figs)
- zeros before all the non-zero digits are **not** significant (0.02 L: 1 sig fig)
- zeros required to show where the decimal place is are not significant, unless the decimal point is written (300 mL: 1 sig fig; 300. mL: 3 sig figs; 300.0 mL: 4 sig figs)

Example: give the number of sig figs

1.32 g 0.00286 L 3200 mL 20.4 mL 20. mL 20.0 mL 410 mL
(answers: 3, 3, 2, 3, 2, 3, 2)

When you record raw data in the lab:

- If the number comes from a **digital instrument**, like a balance, always record all the digits from the balance! Digital instruments are designed to read the correct number of significant figures. You'll notice that the last digit may change while you wait: this is the uncertain digit.
- If the number comes from a **graduated instrument** (something with marked lines, like a regular thermometer or graduated cylinder), you should always estimate to 1/10th of the space between the smallest lines. For example, on a thermometer marked with a line for each degree, you should guess 1 decimal place as best you can. This digit is the uncertain digit, and it's totally fine that it is uncertain: this won't introduce any error because you know it's uncertain.

Significant Figures in Calculations

Now, let's imagine using the thermometer reading in two possible calculations. (Don't worry about the details, just try to follow the formulas and sig figs)

Example A: Maybe you are doing a calculation using the ideal gas law.

1. You convert the temperature to kelvin, which you do by adding 273.15 to the temperature in °C. So if the temperature was 23.0 °C, it's 296.15 K. How many sig figs should this number have? Our original measurement has 3 sig figs, and the conversion factor has 5 as written, but in this case it is exact, so it really has infinite sig figs. Since we add them together, the uncertain digit continues to be the tenths place (because that's the largest uncertainty). We should express our temperature as 296.2 K, or keep one extra sig fig for further calculation. This time, we gained sig figs when we did addition, but the number of decimal places stayed the same.
2. Now we use the following formula with some additional measurements, say $V = 0.02444$ L, $P = 756.2$ torr, and the constant $R = 62.36$ L torr K⁻¹ mol⁻¹:

$$\begin{aligned}n &= PV/RT = (756.2 \text{ torr})(0.02444 \text{ L})/(296.15 \text{ K})(62.36 \text{ L torr K}^{-1} \text{ mol}^{-1}) \\ &= 0.0010007 \text{ mol}\end{aligned}$$

How many sig figs should this have? Our temperature and pressure had uncertain tenths places: is the tenths place still the uncertain place? We can check by redoing the calculation with slightly different values. How much bigger can the answer be if the temperature is smaller by a few tenths, and the pressure is bigger by a few tenths?

$$\begin{aligned}n &= PV/RT = (756.4 \text{ torr})(0.02444 \text{ L})/(295.95 \text{ K})(62.36 \text{ L torr K}^{-1} \text{ mol}^{-1}) \\ &= 0.0010017 \text{ mol}\end{aligned}$$

The difference is in the sixth decimal place, not the first. This suggests that we should round our answer to the sixth decimal place (the uncertain digit). It would be 0.001001 mol. This number has four sig figs, which is the same number of sig figs we started with in the measurements. The number of sig figs didn't change, although the number of decimal places did change.

Example B: Maybe you measured a change in temperature for calorimetry.

1. Now, suppose you measured two temperatures. Originally the water in your cup was 23.0 °C, then you did something that heated it to 31.6 °C. We need to find the change in temperature: $\Delta T = T_{\text{final}} - T_{\text{initial}} = 31.6 - 23.0 = 8.6$ °C. How many sig figs does this have? Our measurements had 3 sig figs, but the change in temperature only has 2 sig figs, because the tenths place is uncertain. This time we lost sig figs, although the number of decimal places didn't change.
2. Now we will use the change in temperature and with another measurement, $m = 101.841$ g, and a constant $C = 4.18$ J/g°C, in the formula

$$q = mC\Delta T = (101.841 \text{ g})(4.18 \text{ J/g}^\circ\text{C})(8.6 \text{ }^\circ\text{C}) = 3661 \text{ J}$$

How many sig figs should the answer have? Let's repeat from the beginning assuming that our measurements were off by a few tenths of a degree: initially 22.8 °C and afterward 31.8

°C. Now $\Delta T = 9.0$ °C. Notice that the difference between this and the original ΔT is still in the tenths place (8.6 rounds up to 9). Our ΔT correctly had 2 sig figs. For the second part,
 $q = mC\Delta T = (101.841 \text{ g})(4.18 \text{ J/g}^\circ\text{C})(9.0 \text{ }^\circ\text{C}) = 3831 \text{ J}$

The difference between the two final values is in the hundreds place. If we round to show one uncertain digit, we'll have 3700 J for the first, and 3800 J for the second. As before, when we multiply, we change the number of decimal places, but the number of sig figs matches the smallest number we started with (2, from ΔT).

Summary rules for calculating with sig figs

(to ensure that the answer has exactly one uncertain digit)

- When doing addition or subtraction, round the answer to the same number of decimal places as the measurement with the least decimal places.
- When doing multiplication or division, round the answer to the same number of sig figs as the measurement with the smallest number of sig figs.
- Never round in the middle of the calculation to fewer than the final number of sig figs. It's best to keep an extra sig fig until the very end.

Sig figs provide the most obvious way to estimate the uncertainty of a number. If it is expressed with the correct number of sig figs, then the uncertainty should be in the last sig fig.

Examples: 0.0014 (± 0.0002), 1400 (± 200), 1.4 (± 0.2)

Rounding

If you need to decrease the number of sig figs in a number (for instance, a result from your calculator) use the following rule:

- if the first digit you're cutting off is 5-9, increase the last digit you're keeping (round up);
- if the first digit you're cutting off is 0-4, don't change the last digit you're keeping (round down).

Data Analysis

Once you've finished performing calculations with your data, you may need to do a few more calculations to assess your data. First, we'll cover what to do with repeated measurements of the same thing, then how to compare your results to expected values.

Averages

If you make repeated measurements, you will probably want to find a single value that reflects all your measurements. The simplest way to do this is with an average. Suppose you have 3 values of some quantity X (X_1 , X_2 , X_3).

$$X_{average} = (X_1 + X_2 + X_3)/3$$

In general, to find an average, add all the individual results together, then divide by the number of results you added. It's usually best to average your final calculated results, not your raw data (in example A above, it would be better to average the number of moles n , not the temperatures and pressures).

Outliers

When you go to average your results, you might notice that some of your trials are very different from the others. For example, if you measure the mass of 10 mL of water 5 times, and get 1.003 g, 9.998 g, 9.986 g, 6.581 g, and 1.005 g, one of those values is really different from the others. It looks like maybe you spilled a lot of the water in trial 4, making it an outlier. Four of the results are within 0.019 g, while the other is off by 3.5 g. In this case, it makes sense to leave out trial 4, which would significantly change your average. However, although trial 3 is also a little farther off (the other results are within 0.007, and it's 0.012 off from the next nearest), probably it's best to leave this one in. There isn't really enough data to be sure trial 3 is an outlier.

Average deviation

If you do repeated measurements of a single quantity, you can calculate a better uncertainty by comparing the measurements to each other, rather than just using sig figs.

1. Calculate the **average** of the trials
2. Find the **deviations**: calculate the difference between each trial and the average. Use the absolute value (drop any minus signs). Use 2 sig figs for the deviations.
3. Take the **average of the deviations for all the trials** (same way you'd calculate any average). Use 2 sig figs.

Your uncertainty for the measurement is roughly twice the average deviation. Usually you use 1 sig fig for the uncertainty, unless the first digit in your uncertainty is a 1, in which case save 2 sig figs. For example, 0.05, 6, or 12 have appropriate numbers of sig figs for uncertainties.

Summary: reporting results of multiple trials

In your post-labs, you may be asked for the "reported value". This means the answer you measured, to the best of your knowledge. You should report values as the average of your trials, with the uncertainty equal to two times the average deviation. You should round your average so that it includes one uncertain digit. Thus, if the uncertainty is 0.006, you should round to the third decimal place, and your reported value would be something like 0.123 ± 0.006 mol/L. If the

uncertainty is 20, you should round to the tens place, and the reported value would be something like 2340 ± 20 J.

Example: Reporting results of multiple trials

if you measure the density of water 3 times and get 1.002, 0.998 and 0.999 g/ml,

Average density = $(1.002 + 0.998 + 0.999)/3 = 0.9997$ g/ml

Deviation = average – individual measurement (but use the absolute value)

so the deviations are $0.9997 - 1.002 = 0.0023$, $0.9997 - 0.998 = 0.0017$, $0.9997 - 0.999 = 0.0007$

Average Deviation = $(0.0023 + 0.0017 + 0.0007)/3 = 0.0016 \sim 0.002$

(Notice that I save extra significant figures during the calculation, and round the average deviation to 1 significant figure at the end)

Average: 0.9997 g/ml

Average Deviation: 0.0016

Average Deviation x2 (use 1 sig fig): 0.003

uncertainty is in the third decimal place, so round 0.9997 to 1.000.

Reported value of density: 1.000 ± 0.003 g/ml

Comparing your results

Several comparison measures are commonly used in the lab.

% Yield: When you do an experiment to prepare and isolate a sample of a chemical, you usually compare the amount of the chemical you got to what you expected to get using % yield. Ideally, the % yield is close to 100% (meaning you didn't lose much of the stuff).

$$\% \text{ yield} = \frac{\text{actual amount}}{\text{theoretical amount}} \times 100 \%$$

% Error: When you measure a number and want to compare it to the accepted value, you use % error. In this case, a very small % error is ideal. For the measured value, if you did multiple trials, use the average value.

$$\% \text{ error} = \frac{\text{measured value} - \text{accepted value}}{\text{accepted value}} \times 100 \%$$

% Difference: You might use this to measure the range of values when you repeat a measurement. Ideally, it would be small.

$$\% \text{ difference} = \frac{\text{biggest result} - \text{smallest result}}{\text{average result}} \times 100 \%$$

Using Spreadsheets for data analysis and graphing

It may have occurred to you that calculating averages and average deviations will be a pain if you have more than a few trials. In this case, it's a good idea to use a spreadsheet program to do it. The results will be more reliable, it's faster, and it's useful to know how to do this. The following instructions are for using Google Sheets, which is free and easily does the things you'll want. You can also use Microsoft Excel, Apple Numbers, or other programs which are similar but perhaps slightly less useful (Numbers is terrible for adding fit lines, and Excel is clumsy for histograms).

Data Analysis: average and average deviation:

First, you'll need to enter data. You can do this by hand, or you might be able to import a .csv file provided by your instructor in some cases. Usually you want to put your trials in rows, and the different quantities you're calculating in columns. For example, enter the sample results from the density example above something like this (A1: A3, B1:B3).

	A	B	C	D
1	trial 1		1.002	"=ABS(\$B\$4 - B2)"
2	trial 2		0.998	
3	trial 3		0.999	
4		"=AVERAGE(B1 : B3)"	"=AVERAGE(C1 : C3)"	

Then go to the density column, below the last result. Type "=average(", select the three results and type ")". Hit enter or return. You'll see the average appear.

In the next column over, type "=ABS(". Select the average, type "-", then select the individual trial. Type ")" and hit enter or return. You'll see the first deviation appear.

Then double-click that cell (C1) and insert the \$ symbols into the "address" for the average. This tells it not to change this part. Then select C1:C3, and hit "Meta D" on PC or "⌘+D" on Mac. This "fills down" the formula for the deviation, so the second and third deviations appear. Now you can calculate the average deviation just as you did the average.

Graphing: using histograms to find outliers

If you want to decide which values are outliers, a good way to do this is by looking at a histogram. Select the column or cells with your data (it should all be in the same column). Hit the "insert chart" button, or use "Insert ... Chart" in the menu. Choose histogram from the type options (only available if you have more than a few data points), select the customize tab, and adjust the bucket size appropriately (use the preview to decide what's good; probably small enough to separate the data well). Now you should be able to see clearly if any results are really outliers.

Graphing: XY scatter plot and linear fit

To make a traditional graph (XY or scatter type) you'll need two columns of paired data points. For example, you might have a column with mass data and a second column with the volume that corresponds to each mass. In this case, the mass data will be on the x -axis and the volume data will be on the y -axis (volume vs mass). If you want it the other way, switch the order of the columns. Select both columns and click the chart button. In the Charts tab, choose Scatter. Then in the Customize tab, if you want, you can scroll all the way down and choose trendline. If you think your points should all be on a straight line, choose linear, and then choose "use equation" for label. Now the graph will show the best fit line for your data, and the equation of the line. You can see how well you think it fits.

You can also set the title, axis labels, etc. If you're going to print your graph and hand it in, you should definitely include labels on the axes, with units. Probably it will choose sensible ranges for the axes, but you can change these if you want by double-clicking on the axes. Ideally, your data points should fill the whole graph on both axes so you can see them clearly.

Graphing by hand

Graphing by hand is not recommended. Hopefully you remember how to do it in a pinch, but in the modern world you should know how to graph with a spreadsheet. This is much more precise and accurate, and it's much faster and easier once you get the hang of it. You'll also get much better best fit lines from a computer.

Errors and Their Effects

Types of Errors

There are three important types of errors that can affect your experiments.

Blunders: these are the avoidable mistakes you make, like spilling your sample, misreading an instrument, not following instructions, etc. Since blunders are avoidable, they are never interesting. In real life, if you make a blunder, you'll always repeat the experiment until you do it without blunders. In your post-lab sheets, you should usually not mention blunders.

Random error: all measurements have some amount of random error, meaning if you repeat a measurement many times, it might be slightly different each time because of tiny changes that we can't keep track of. For example, if you take the mass of an object five times on a digital balance, you'll probably find that the mass is slightly different in the last decimal place. This is fine and normal: the last decimal place is the uncertain digit, and it will change a little depending on air currents or other small effects. If you repeat the measurement many times, the average of all the measurements should be the true value, assuming that the instruments are calibrated correctly and only random errors are present. Random errors will probably increase if you are careless, but no matter how careful you are there will be some variation in repeated results.

Bias: this means errors that always affect the result in the same direction even when the procedure is followed carefully. For example, suppose you are measuring density of a liquid. You measure the volume in a tall graduated container that doesn't fit on the balance, then pour it into another shorter container to find the mass. This could introduce bias, because in each trial you will leave a little bit of the liquid in first container when you pour it. Your mass measurements will always correspond to a slightly smaller amount of the liquid than your volume measurements, meaning that your calculated densities (mass/volume) will always be a little low and your average density will be a little wrong, no matter how many trials you do. In this case, you aren't making a mistake, because you're following the procedure in a sensible way. (Probably the tall container reduces random error in measuring the volume.) However, it would be better to change the procedure to avoid this error, if possible. A bias in your experiment won't necessarily cause your repeated measurements to have a larger uncertainty, but it does make your result inaccurate, so if you don't pay close attention to possible bias you might be badly misled.

Notice that although bias sometimes refers to a person's preference for a particular result, human bias is not the only source of bias in experiments. In your work, human bias is not likely to be all that important, assuming that you carefully read your instruments.

Accuracy and Precision

In an **accurate** experiment, the true value is within the uncertainty range of the measured result. For example, if the reported result is measured to be 1.89 ± 0.3 , then the uncertainty range is 1.86 - 1.92. If the true value is between 1.86 and 1.92, the result is accurate. If enough trials are made, random error will not make an experiment inaccurate, because the average will still match the true value.

In a **precise** experiment, repeated measurements are very close together. In other words, the % difference is small. The uncertainty in your reported results is a measure of the precision, since you find it using the difference between the individual results. If you don't do multiple

trials, you can estimate the precision from the number of sig figs. In theory, each individual measurement should not vary beyond the last sig fig, so the combination of all the raw data should also not vary beyond the last sig fig. However, this might not be true in real life, especially if you are not very careful. Random error will affect the precision of an experiment, but bias may not.

If a particular procedure has little random error but a lot of bias, you could get a very misleading result. The reported value would have a small uncertainty, so it would “look good” and be precise, but it might be quite inaccurate. If you don’t know the true value, you would not be able to figure out that your measurement is wrong except by understanding what you did and whether it made sense. If you do know the true value, you can measure the accuracy of the experiment by calculating the % error.

Experiment 2 - Using Physical Properties to Identify an Unknown Liquid

Introduction

Chemists use **properties** to identify the **substances** they encounter. Properties are the distinguishing characteristics of the materials, such as their color, phase (solid, liquid or gas), smell, etc. Substances are separated into several categories:

- **elements**, which contain atoms of only a single element (examples: aluminum metal, charcoal)
- **pure substances** or **compounds**, which contain only a single type of molecule, or a specific, consistent ratio of elements (examples: pure salt, sugar, and water)
- **mixtures**, which contain several different compounds (examples: air, milk, seawater)

Properties are also divided (somewhat arbitrarily) into **physical properties** and **chemical properties**. Generally, chemical properties are properties that have to do with reactions between substances, while physical properties do not involve reactions. For example, chemical properties include the ability to burn in air, or to dissolve in acid. Physical properties include density, color, hardness, boiling point, and melting point. Solubility (the ability to dissolve in another substance, for instance solid sugar dissolving in water to make syrup) is sometimes considered a chemical property and sometimes a physical property.

In this lab, you will assess the following properties of an unknown pure liquid:

- **boiling point** (the temperature at which the liquid boils, such as 100 °C for water)
- **melting point** (the temperature at which the liquid freezes, such as 0 °C for water)
- **density** (the ratio of mass/volume, usually measured in g/ml)
- **solubility** (or more correctly **miscibility**) in water (whether the liquid mixes completely with water, or forms two separate layers, like oil and water)

Your measurements will give you a possible range for most of the properties, rather than a precise number, but this is sufficient to determine which of the liquids in the table below you have. Specifically, we will measure the boiling point to within ± 5 °C, whether it freezes above or below -78 °C and 0 °C, and if it is not soluble in water, whether it is more or less dense than water (density above or below 1 g/ml). After you measure these properties, you should be able to eliminate all but one of the liquids in the table, leaving only one possibility. It may be more helpful to eliminate impossible liquids rather than trying to match the correct one.

Safety Precautions:

- Wear your safety goggles at all times.
- Recommended: wear latex or nitrile gloves

Waste Disposal:

- Pour the used samples into the **organic waste** container in one of the fume hoods.

Physical Properties of some liquids

name of compound	formula of compound	boiling point (°C)	melting point (°C)	density (g/mL)	solubility in water
acetone	C ₃ H ₆ O	56	- 94	0.79	soluble
acetonitrile	CH ₃ CN	82	- 45	0.75	soluble
carbon tetrachloride	CCl ₄	77	- 23	1.59	insoluble
cyclohexane	C ₆ H ₁₂	81	+ 6	0.78	insoluble
cyclopentane	C ₅ H ₁₀	49	- 94	0.75	insoluble
ethanol	C ₂ H ₅ O	78	- 115	0.79	soluble
methylene chloride	CH ₂ Cl ₂	40	- 95	1.32	insoluble
t-butyl chloride	C ₄ H ₉ Cl	51	- 27	0.85	insoluble
“Freon-113”	C ₂ Cl ₃ F ₃	48	- 35	1.6	insoluble
water	H ₂ O	100	0	1.00	N/A

Procedure

First obtain a sample of an unknown liquid. Record the unknown code on your report sheet. Three different properties of the sample will be measured: its solubility in water, its boiling point, and its melting point. You may do the three parts of the lab in any order.

Part 1 - Solubility in Water

Take an empty test tube and add enough water to it to fill approximately the bottom inch to two inches of the test tube. Then add a small portion of your unknown liquid to the test tube. You now have a mixture of two liquids. Stir or gently swirl the mixture to mix the two liquids. Does the unknown liquid dissolve in the water, or does it form a separate layer? If the unknown is **soluble** in water, it will completely mix with it to form a **homogeneous mixture**. The mixture will appear as one clear liquid, and will look the same throughout. If the unknown is **insoluble** in water, the mixture will be a **heterogeneous mixture** in which two separate phases are visible. The unknown will either float above the water or will sink below it to the bottom of the test tube, and you will be able to see a line in between the two layers.

If the unknown liquid is insoluble in water, this test will also provide some information about another property of the liquid, its **density**. The density of a substance is the weight of a standard volume, usually one cubic centimeter (1 cc or 1 cm³) of the substance. Density is a measure of the relative "heaviness" of a substance. If two liquids that are not soluble in each other are mixed together, the one with the greater density will sink to the bottom, and the one with the smaller density will float on top. Oil floats on water because the density of oil is less than that of water.

If your unknown was insoluble in water, add a few drops of copper sulfate (CuSO_4) solution. This blue solution will dissolve only in the water layer, and will color that layer blue, so you can tell which layer is water. The density of water is exactly 1.00 g/cm^3 . Any liquid that sinks when added to water has a density greater than 1.00 g/cm^3 , and any liquid that floats on water has a density of less than 1.00 g/cm^3 . Observing if the unknown sinks or floats will not tell you its exact density, but will only tell you if its density is greater than or less than 1.00 g/cm^3 . If the unknown was soluble in water, its density cannot be estimated by this method. Whenever two liquids are soluble in each other, they mix evenly, regardless of their densities.

Part 2 - Boiling Point

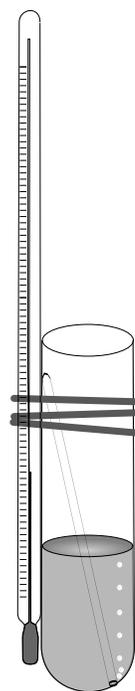
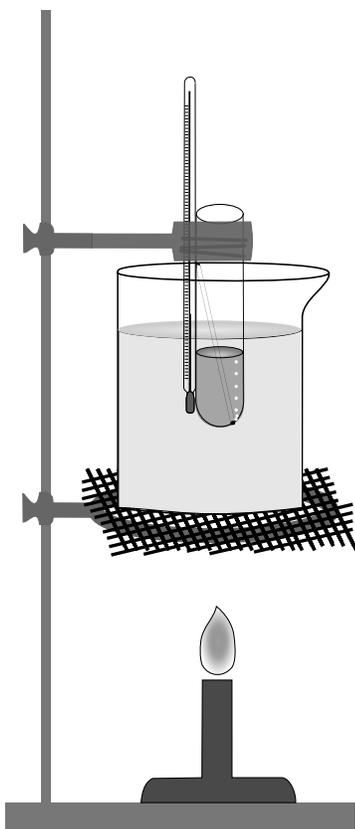
The boiling point of the liquid will be measured by a microscale technique, which uses a very small amount of the liquid unknown. Refer to the following drawing when setting up the equipment for this part of the experiment.

Set up a ring stand with a water bath: attach a ring to the ring stand, put a piece of wire gauze on top of it, and then place a beaker of water on the wire gauze. Place about 0.5 mL of the unknown liquid in a small test tube, and place an empty capillary tube *open end down* into the liquid. Attach a thermometer to the small test tube using several rubber bands (stacked on top of

each other). Make sure that the bulb of the thermometer is lined up with the level of the liquid inside the test tube. Clamp the assembly to the ring stand, submerging it into the water (make sure that the unknown liquid inside the small test tube is below the water level so that the unknown heats evenly, but do not let any water enter the test tube!).

Start heating the water bath using a Bunsen burner, and watch the unknown continuously. Keep heating the sample until a *rapid and continuous* stream of bubbles comes out of the small capillary tube. At this point, turn off the Bunsen burner. The bubbles will slow down, stop, and then some liquid will be drawn up into the capillary tube. Measure the temperature when the liquid enters the capillary: this is the boiling point of the liquid (within 5°C).

If your liquid has a low boiling point, it may evaporate completely from the test tube before you finish your measurement. If this occurs, repeat the measurement



using a little bit more liquid in the test tube.

Variations in barometric pressure, impurities in the liquid, or difficulty choosing the correct moment to record the temperature may cause your measured boiling point to be a little different

from the accepted boiling point listed in the table. Use your measured boiling point with its error range ($\pm 5\text{ }^{\circ}\text{C}$) to eliminate liquids outside this range.

Part 3 - Freezing point

Most of the liquids on the chart do not freeze until they have been cooled to very low temperatures. Acetone, for example, only freezes if it has been cooled to $-94\text{ }^{\circ}\text{C}$, or -137°F . In this experiment, we will not measure the exact freezing temperature of the unknown liquids, but will only compare their freezing temperatures to the temperature of **dry ice** and ice-water. Dry ice is solid carbon dioxide (CO_2). It vaporizes to form gaseous CO_2 at $-78\text{ }^{\circ}\text{C}$. While it is vaporizing, the solid dry ice maintains a temperature of $-78\text{ }^{\circ}\text{C}$. A mixture of dry ice and acetone will be provided. The dry ice cools the acetone to $-78\text{ }^{\circ}\text{C}$, but the acetone remains liquid because its freezing point is lower. An ice-water bath will also be provided; in this case, as the ice melts the bath maintains a constant temperature of $0\text{ }^{\circ}\text{C}$.

To test your unknown, take a dry test tube and add some unknown liquid to it. Add enough to fill the bottom one inch or so of the test tube. Hold the test tube with a test tube clamp in the dry ice-acetone bath. Leave the liquid totally immersed for at least two minutes. Remove the tube and check if the liquid has frozen. If the liquid appears to have partly frozen, immerse it in the dry ice-acetone bath again to see if it will completely freeze.

If the liquid freezes in the dry ice-acetone bath, its freezing point is higher than -78°C . If it does not freeze, its freezing point is lower than -78°C . If it does freeze in the dry ice-acetone bath, let it thaw, then immerse it in the ice-water bath and see if it freezes. This will tell you if the freezing point is above or below $0\text{ }^{\circ}\text{C}$. The actual freezing point of the liquid cannot be determined by this experiment.

Data Analysis

Use your results to eliminate all the impossible liquids in the table, leaving only one. The remaining liquid is your unknown. In the results section in your notebook, clearly indicate everything you know about the physical properties of your unknown and identify it by name.

Experiment 2 Pre-Lab Sheet

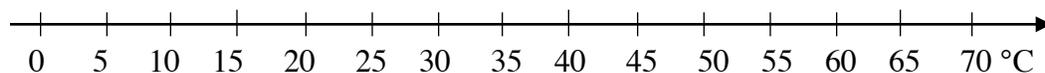
Name:

Section:

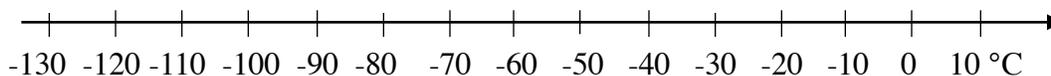
Illustrate the possible range for each property on the number line, based on the experimental results given. Then identify the liquid from the list.

Sample 1: The boiling point is recorded as 43 °C, it does not freeze in the dry ice-acetone bath, and the sample is insoluble in water, with the blue layer above the clear layer.

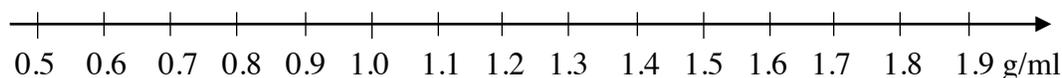
Boiling point range (1pt):



Freezing point range (1pt):



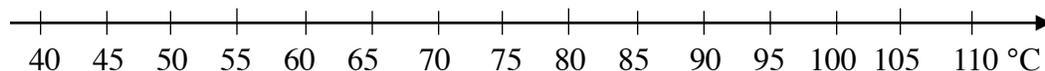
Density range (1pt):



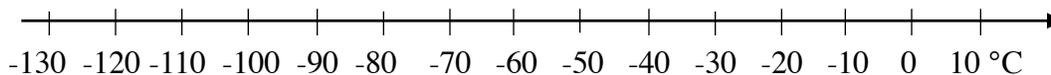
Identity of sample 1 (2pts):

Sample 2: The boiling point is recorded as 80 °C, it freezes in the dry ice-acetone bath but not in the ice-water bath, and is soluble in water.

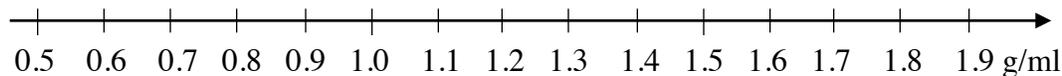
Boiling point range (1pt):



Freezing point range (1pt):



Density range (1pt):



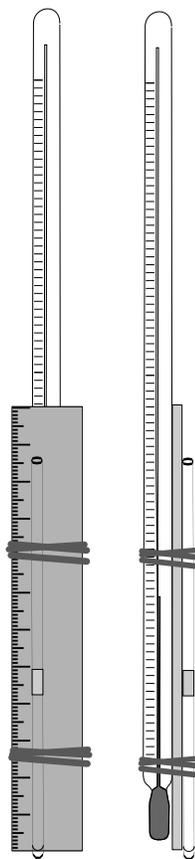
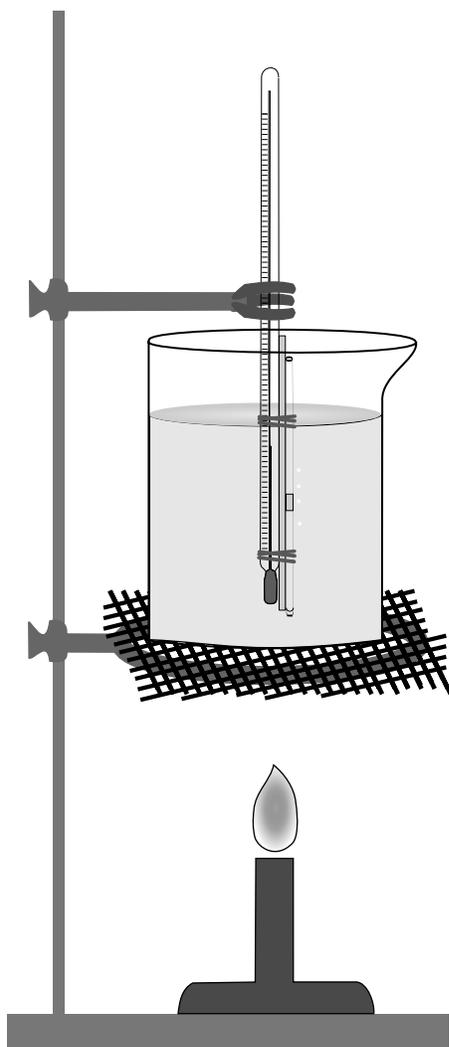
Identity of sample 1 (2pts):

Experiment 3 - Relationship between Temperature and Volume of Gases

Introduction

Gases are distinguished from liquids and solids by their **compressibility**, the ability to change volume depending on circumstances. In this experiment, you will study the relationship between the temperature and the volume of a gas, which will lead to a new temperature scale, the absolute or Kelvin scale.

In your experiment, the gas sample will be a small amount of air that is trapped beneath a tiny plug of oil in a very narrow glass tube called a “capillary tube.” When the air sample is heated, it will expand and the oil plug will move upward in the tube. The opposite will occur when the air sample is cooled. You will measure the volume of the air indirectly, by measuring and recording how far the plug moves, using a ruler to which the capillary tube is fastened along with a thermometer. (See below.) The tube should be fastened in such a way that the bottom of the tube is at the bottom of the ruler.



The sample capillary tube (attached to thermometer and ruler) is then immersed in a water bath. The temperature of the bath is read off the thermometer in Celsius degrees. The volume of the sample is read off the ruler in millimeters. We are assuming that the capillary tube has a constant diameter, so that the volume of the tube is proportional to its length ($V = \pi r^2 h$, so if r is constant, V is proportional to h). You will thus measure the length of the air column in the tube at each temperature. We don't need to know the *actual* volume in the capillary tube; knowing a quantity proportional to volume is sufficient.

You will finish the lab by analyzing a graph of the data.

Safety Precautions:

- Wear your safety goggles at all times.

Waste Disposal:

- Place the used capillary tubes in the glass waste containers. The water from the water baths can be poured down the drain.

Procedure

First you will prepare the gas sample in a capillary tube, then collect data, then graph the data.

Part 1 - Capillary Tube Preparation

1. Assemble the following materials: a Bunsen burner, a pair of crucible tongs (NOT a test tube clamp), an empty capillary tube, and a small dish of oil. Light the Bunsen burner.
2. Pick up the capillary tube with the crucible tongs so that you have a firm grip on it and so that the open end of the tube is pointing down.
3. Wave the tube back and forth through the flame while you count to 20. (The purpose of this step is to heat up the air inside the tube without actually melting the tube itself. Make sure to keep it moving, or it will melt. Keep it mostly in the flame.)
4. When you have counted to about 20, quickly dip the end of the tube into the oil (have the oil within easy reach so it won't cool before you get it in the oil). When 4-8 mm of oil has been drawn into the tube, remove it from the oil.
5. After it cools, you should have a sample of air trapped under a small bubble of oil about halfway up the tube. You now have a trapped sample of air in a "flexible" container: the volume adjusts itself to keep the pressure constant.
6. Make sure that your tube is properly set up: there should be a single bubble of oil, 4-8 mm long, and it should be at least one third of the way down the tube.

Part 2 - Measurement of Volume at Varying Temperatures

1. Attach your capillary sample tube to a metal ruler and thermometer using rubber bands. Make sure that the bottom of your air sample is lined up with the zero mark of the ruler so you can easily read the height of the air column. The bulb of the thermometer should be level with the air sample.
2. Set up a ring stand with a water bath: attach a ring to the ring stand, put a piece of wire gauze on top of it, and then place a 400 ml beaker of water on the wire gauze.
3. Put your sample tube with ruler and thermometer in the beaker, and clamp it in place using a "3-finger" clamp coated with rubber. The air sample should be under the water, but the top of the capillary tube should be above the water. Make sure you can easily read the length of the air sample and the temperature.

4. Heat the water bath until it boils using a Bunsen burner. If necessary to keep the air sample under water, add more water.
5. When the water boils, turn off the Bunsen burner. As the water bath cools, take readings of the temperature and the height of the bottom of the oil bubble every 5 °C, starting close to 100 °C and stopping at about 30 °C. Record the data in a table in your notebook. Record your measurements with 3 significant figures! (It's ok that the last sig fig is uncertain.)
6. You can speed up the cooling by stirring the water bath with a test tube of ice.
7. If time allows, repeat the experiment with a new sample tube.

Data Analysis

1. Plot the length of the gas sample (which is proportional to its volume) against the temperature in °C. (This means length on the y -axis and T on the x -axis.) You can do it by hand in your lab notebook or in a spreadsheet program like Excel. Show data points clearly as dots or markers. Scale the graph so that the **data** (not just the axes) take up at least half a page.
2. Find the equation of the “best-fit” straight line through the data points. You can do this by hand or with the program. Show the best-fit line on the graph.
3. Find the x -intercept of the line. This is the temperature where (in theory) the volume of the gas sample would be zero. Write your value of the x -intercept on the board, along with your length at 100 °C, before you leave. If you don't find the x -intercept before you leave, email it and your length at 100 °C to your instructor within 48 hours. The instructor will collect everyone's results and send the list to the class for analysis in the post-lab.

Experiment 3 Pre-Lab Sheet

Name:

1. (2 pts) What is inside the capillary tube, under the oil bubble?
2. (1 pt) What happens to the position of the oil bubble as the tube is heated?
3. (1 pt) Draw the precise position of the ruler relative to the capillary tube shown.
4. (2 pts) Why is the ruler position important? (“To make it accurate” is *not* a useful answer—be specific.)
5. (2 pts) What is a reasonable unit to record the position of the oil bubble? How many decimal places should you include, for this unit?
6. (1 pt) What will the label on the x -axis of your graph be? (Include units)
7. (1 pt) What will the label on the y -axis of your graph be? (Include units)



Experiment 4 - Determination of the Empirical Formula of Magnesium Oxide

Introduction

A **molecular formula** tells the number of atoms in the molecule (such as H₂O for water or H₂O₂ for hydrogen peroxide). The **empirical formula** of a compound merely gives the ratio of atoms in the compound based on experimental evidence (such as H₂O for water or HO for hydrogen peroxide).

When magnesium and oxygen are heated together, they react:



From the masses of magnesium and oxygen that combine, we can calculate the **empirical formula** of magnesium oxide. We will weigh the magnesium before it combines with the oxygen, and we will also weigh the product of the reaction, magnesium oxide. The final weighing is necessary because we need to subtract the original weight of magnesium from this weight of product. We "weigh" the oxygen in this indirect way because it is easier than weighing the oxygen gas before it combines with the magnesium.

If magnesium is heated in open air, its reaction with oxygen is rapid and spectacular. The metal catches fire, burning with an intense white flame and with production of white smoke. (The metal has been used in flares to provide light for night-time military operations.) We can slow down the reaction by limiting the supply of oxygen that reaches the magnesium; we do that by putting a cover on the crucible that contains the magnesium sample.

Our procedure in this experiment is complicated by another fact. Magnesium is such an active metal that it reacts with the relatively inactive element nitrogen:



This occurs in competition with the reaction of magnesium with oxygen, so it is called a "side reaction." You should realize that this side reaction uses up some of the magnesium that is supposed to be combining with oxygen. Fortunately it is possible to "undo" the reaction in this way:



and



To get reactions (3) and (4) to occur, we add water to the crucible contents at the end of the first heating period. We then heat again to speed up reactions (3) and (4) and to evaporate any excess water. Ammonia is a gas, so it will also diffuse out of the crucible.

Safety Precautions:

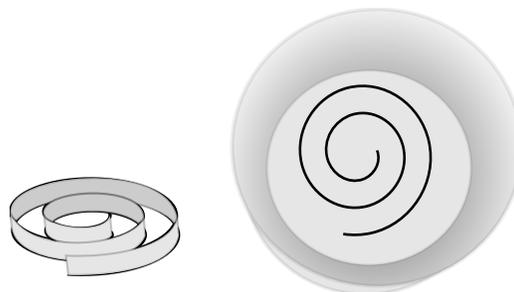
- Wear your safety goggles.
- When the magnesium is burning, do not look directly at the flame. It is bright enough to damage your eyes.
- The crucibles can shatter when heated. This is not a day to be careless about goggles!
- Be careful not to burn yourself on the hot crucible, ring, etc.

Waste Disposal:

- The solid product of this reaction can be discarded in the regular garbage can after the experiment. (Scrape it out with a spatula.)

Procedure

1. Choose a clean crucible with no cracks or flaws. Heat a clean, dry porcelain crucible with cover on a clay triangle (supported on a ring stand), using a direct flame, for about 5 minutes. This will dry the crucible.
2. Turn off the burner, and let the crucible and cover cool. Leave the crucible and cover resting on the clay triangle. (If you wish to remove the hot crucible from the clay triangle, set it on one of the wooden particle boards.)
3. Weigh the empty, dry crucible and cover on the digital balance. Record this mass. Record the mass of the crucible and cover separately also, in case the cover breaks.
4. Obtain a strip of magnesium ribbon that is about 10 to 15 cm long. If it is not shiny, polish it briefly with steel wool to remove any oxide coating.
5. Wrap the ribbon around a pencil to make a loose, flat spiral. Set the spiral in the crucible; it should sit flat against the bottom of the crucible. (See diagram below.)
6. Carefully weigh the crucible, cover, and magnesium ribbon together. Record this total mass, from which the mass of the ribbon will be calculated.
7. Put the cover aside for a moment, and start heating the crucible with the magnesium in it. Have the cover close by, with some crucible tongs ready to handle it with.
8. The instant that the magnesium starts to burn, put the cover on the crucible (using the crucible tongs) to put out the fire.
9. Continue heating the covered crucible for a minute or so, then take off the cover again. Wait for the magnesium to catch fire again, then quickly re-cover the crucible. When handling the cover, notice that some material sticks to the inside of it. This is also your product. Hold the cover upside-down and try not to knock the product off it to get the best data.
10. Repeat steps 9 and 10 until the magnesium no longer catches fire when the cover is removed.
11. Heat the sample strongly for 5 minutes, with the cover on the crucible. Make sure the bottom of the crucible becomes red hot.
12. Turn the burner off and let the crucible cool until it is only barely warm to the touch. Record the mass of the crucible, cover and product.
13. To the cooled crucible, add about 10 drops of deionized water. Make sure to wet the entire surface of the sample, not just one spot.



14. Warm the crucible with the damp sample using a gentle flame for a minute or so, then heat it moderately strongly for about 10 minutes. (The crucible need not become red hot for this phase of the experiment, and the cover is not needed for this heating.)
15. Let the crucible and contents cool until the crucible is barely warm to the touch.
16. Weigh the cooled crucible, cover, and contents of the crucible, recording this total mass.
17. Perform the calculations, and write your mole ratio (from step 5) of Mg to O on the board along with your names. The instructor will distribute a photo of the board with all the data so you can use it for your post-lab analysis.

Calculations

1. From the total mass recorded in step 16 and the mass of the empty crucible and cover, you can calculate the mass of the crucible contents. These contents should be fairly pure magnesium oxide.
2. Calculate the mass of oxygen that combined with your mass of magnesium in this experiment using subtraction.
3. Calculate the number of moles of magnesium you started with.
4. Calculate the number of moles of oxygen that reacted with the magnesium.
5. Calculate the mole ratio of magnesium to oxygen. To do this, divide the moles of magnesium by the moles of oxygen. Express the result to the correct number of significant figures.
6. Using the above ratio, write the empirical formula of magnesium oxide by rounding off the ratio to the nearest whole number.

Name:

Section: Tu Th Fam Fpm

Experiment 4 Pre-Lab Sheet

1. (1 pt) Why shouldn't you look straight at the burning magnesium?
2. (1 pts) Why do you add water?
3. (1 pt) Why do you heat the crucible again after adding water?
4. (6 pts) The following data were collected during a very similar experiment using tin. Perform the following sample calculations (show your work, with units canceling):
 - mass of crucible and cover: 19.8413 g
 - mass of crucible, cover and Sn: 21.2325 g
 - mass of crucible, cover and product: 21.6006 g

mass of Sn:

mass of O reacted:

moles of Sn:

moles of O reacted:

mole ratio (mol Sn/mol O):

empirical formula:
5. (1 pt) Will your mole ratio increase or decrease if a lot of white powder falls off the lid of the crucible?

Experiment 5 - Exploring Dissociation

Introduction

In this lab we'll explore **dissociation**, which refers to substances splitting into separate ions in solution. There are different ways to see if dissociation is happening, but one of the easiest is **conductivity**. Electricity can only flow when charged particles are free to move, like electrons in metal wires or ions in solution. If the solution contains only neutral molecules, then electricity can't flow. The more charged particles that can move, the higher the conductivity. Here, you'll test the conductivity of solutions of different compounds and at different concentrations, to get a feel for what compounds form ions, and how this depends on concentration. Some compounds dissociate completely and are called **strong electrolytes**. Other compounds dissociate only a little bit, and are called **weak electrolytes**. Others don't dissociate at all, and are called **non-electrolytes**. We'll use conductivity probes connected to a digital display to measure the conductivity of the solutions, and you will graph the conductivity vs the concentration.

Many students confuse **dissolution** with dissociation. Dissolution means dissolving, or forming a solution. The substance that dissolves is called the **solute**, and the substance it dissolves in (here, water) is called the **solvent**. Not all substances dissociate when they dissolve: that's what we'll be measuring.

In Part 2, we will prepare a solutions by **dilution**, which means making solutions less concentrated by mixing them with more solvent. We will measure the concentration of the solutions using molarity, which is defined by

$$M = (\text{moles of solute})/(\text{liters of solution}) \quad \text{Eq 1}$$

For instance, a 1 M ("one molar") solution of salt has one mole of NaCl in each liter of solution. Thus, in one milliliter, there is one millimole. If you take 50 mL of this solution and mix it with 50 mL of water, what is the new molarity? The way to think about this is to recognize that however many moles were in the 50 mL of 1 M solution are now in 100 mL of solution. The new concentration is given by

$$M_{\text{final}} = M_{\text{initial}}V_{\text{initial}}/V_{\text{final}} \quad \text{Eq 2}$$

We find the total numbers of moles in the final solution by multiplying initial volume and concentration (0.05 L * 1 mol/L), then divide this number of moles by the end volume (0.1 L), so the result is 0.5 M. You can use Eq 2 (maybe in the form $M_1V_1 = M_2V_2$) to find the new concentration each time you do a dilution. In case you encounter more complicated situations, the thing to remember is that you need to find the total number of moles, and the total volume, and divide them.

When you are making solutions of particular concentration, it's important to control the amount of water. For example, you probably need to dry your glassware before using it if it is wet, so it doesn't get diluted extra.

For this lab, you will have to "calibrate" the pipettors we use for dilution. Since we use each pipettor for only 1 volume, we don't have to do a real calibration, we'll just adjust the settings until we get the right amount.

Safety Precautions:

- Wear your safety goggles at all times.
- Be careful with the strong acids and bases (HCl, NaOH)
- If a bottle is in the fume hood, leave it there!
- Close bottles as soon as you are done using them

Waste Disposal:

- Pour your solutions into the waste bottle in the hood when you're done. Use the water squirt bottles to rinse the glassware into the waste bottle.

Procedure

Use **deionized (DI) water for everything!** Record all observations directly into your lab notebook.

Part 1 - Pipettor Use and Calibration

1. Get 2 pipettors with tips, probably a 5000 μL and 1000 μL (you'll need one to measure 1 mL, and one to measure 5 mL). (You may have to share the pipettors with another group since we don't have very many. Stick the tips on very firmly.
2. Get a big beaker of DI water, and another smaller beaker.
3. When using the pipettors, **be careful** not to twist the dial past the point where it twists easily, since this can break the pipettor, and they are very expensive.
4. To use the pipettor, set the dial (gently!) to the desired amount (the digit in red is the number of mL, and the digits in black are the decimal places. Wrap your fingers around the dark blue part and push down on the top part with your thumb. Before you dip it in liquid, push on the top and notice that it goes a set distance to the "stop point", then it becomes much harder to push although it will go down farther. To draw in liquid, dip the tip in the liquid, and push until the "stop point" (not beyond it!) then release the top slowly to draw in liquid. Make sure the tip stays under the liquid level the whole time, and do it slowly. To dispense the liquid, don't touch the tip to the liquid. Push down again on the top and this time push past the "stop point" to get the last bit of liquid out. Make sure the tip is not touching liquid when you release the top.
5. Put the smaller beaker on a digital balance. Tare it. Set one pipettor to dispense 5 mL, and dispense 5 mL of DI water into the beaker. The scale should read 5.00 g, because the density of water is 1.00 g/mL. If it does not, adjust the pipettor until it dispenses the correct amount of water (just twist the dial, don't worry about the new reading). When it seems to be set correctly, dispense 5 mL into the tared beaker 5 times, recording the mass after each addition in a table in your notebook. Make sure that it reliably dispenses 5.00 mL. Record the serial number of the pipettor and the precise reading of the dial that you are actually using, to 3 decimal places.
6. Repeat with the second pipettor until it correctly dispenses 1.00 mL. As before, record the 5 correct measurements, the setting you are using and the serial number.

Part 2 - Dilution

1. Take an index card with a solute (1 card / group).
2. Get a big test tube rack and 6 of the short wide test tubes.
3. Collect about 40 mL of the 0.100 M stock solution of your solute in a clean, dry small beaker. Use this for everything that follows.
4. Use the pipettor to put 10 mL of the 0.100 M stock solution in one test tube. Label it with the solute and the concentration.
5. You don't need to change tips on the pipettors between dispensing stock solution and water because the solutions don't usually stick to the tips (and because you are not dipping the tip in a shared stock bottle!). If there are any drops on the tip, just dry them with a paper towel before pipetting the next thing.
6. Into a second test tube, dispense 8 mL of the stock solution and 2 mL of DI water. Mix the solution thoroughly. Calculate the concentration of the solution in the test tube, and label it with solute and concentration.
7. Into a third test tube, dispense 10 mL of the stock solution and 5 mL of DI water. Mix, calculate the concentration and label it as before.
8. In the fourth test tube, prepare 10 mL of 0.05 M solution. Mix and label as before. In your notebook, record your calculations, and how much stock and water you added.
9. In the fifth test tube, prepare 12 mL of 0.025 M solution. Mix, label and record as before.
10. In the sixth test tube, dilute some of the 0.025 M solution (in the fifth test tube) to make 10 mL of 0.0025 M solution. Mix, label and record as before.

Part 2 - Conductivity Measurements

1. Setup your probe. Plug the LabView box into the outlet, then plug the conductivity probe into the LabView box. On the small box connected to the probe, make sure the switch is set to 0-20000.
2. Put some DI water in a clean test tube or beaker. Dip the conductivity probe in the water and swirl it. Keep the top half of the probe dry. Wait about 15-30 seconds for the reading to stabilize, then record the conductivity of the pure water. Remove the probe. (The last digit of the reading will change rapidly: just write down a number that seems in the middle of the range.)
3. Add a scoop of sugar to the water and dissolve it. Measure the conductivity of this solution. Is sugar an electrolyte?
4. After each conductivity measurement, rinse the probe with DI water from a squirt bottle. Then dry the probe with paper towel. This prevents diluting or contaminating the solutions.
5. Measure and record the conductivity of each of your solutions from Part 1 (dip the probe, swirl, estimate the average reading after 30 seconds or so). Record your results on the board.
6. Next you will mix your solutions with another group's solutions. **Ask your instructor** which solute you should mix your solutions with (don't choose at random). Then find a group with the right solute, and mix the solutions of the same concentration. For example, pour your 0.15 M solution into their 0.15 M solution, and stir. Repeat with all solutions.
7. Measure the conductivity of the combined solutions (dip the probe carefully, since the tubes are now fuller!). Record this data on the board also.

8. Take a look at the mixtures made by other groups (compare to the stock solutions). Record any observations: was there a visible change after mixing?
9. Based on the conductivity data and your observations, decide whether you think a reaction occurred for each mixture. If so, write the equation.
10. The instructor will provide a file with the data for the whole class. Here or at home, you will import the file into an appropriate spreadsheet program. If you aren't sure how to do this, ask the instructor or a classmate to show you.
11. Graph the conductivity (y-axis) vs. the concentration (x-axis) of all the solutions on the same axes. You should use a spreadsheet program to do this (see instructions in the Reference section). Make the graph really big and choose the range of the axes carefully so you can easily see the data. If you aren't sure how to do this, ask the instructor or a classmate to show you during lab.
12. Looking at your graph, what do you notice? Which solutes are linear (a straight line graph)? Do any of them curve? Does the conductivity match the number of ions you expect the solution has? Which are strong and weak electrolytes?

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Experiment 5 Pre-Lab Sheet

If you have a laptop, tablet or other device that can run a spreadsheet program, today is a great day to bring it to class!

1. (1 pt) What type of water should you use for *everything* in this lab?
2. (1 pt) Which part of the conductivity probe should you keep dry?
3. (1 pt) What should you be careful of so you don't break the pipettor?
4. (1 pt) When can you go past the stop point on the pipettor and when shouldn't you?
5. (2 pts) You will collect data for each solution in the form of (x, y) pairs to be graphed. What quantity will you put on the x -axis and what will you put on the y -axis? What other information do you need for each pair?
6. (2 pts) Sample calculation: if you make a solution by mixing 16 mL of 0.2 M NaCl with 4 mL of water, how many moles of solute are present? What is the concentration of the solution?
7. (2 pts) If you wish to prepare 40 mL of 0.01 M NaOH and you have 0.2 M NaOH available, what should you do?

Experiment 6 - Exploring Double Displacement Reactions

Introduction

This week, we are going to try a different style of lab. Instead of following a strict procedure, I want you to have an opportunity to try things out and explore your own ideas. Please be safe and responsible, but feel free to play around!

Many reactions depend on **dissociation**, the splitting of substances into ions when they dissolve. Recall that substances that split into ions are called **electrolytes**. Most molecular substances, including water, are non-electrolytes, meaning they don't split into ions enough to conduct electricity. Most ionic compounds are strong or moderately strong electrolytes, meaning they split completely into ions, or at least mostly into ions. Strong acids and bases are strong electrolytes, and weak acids and bases are weak electrolytes.

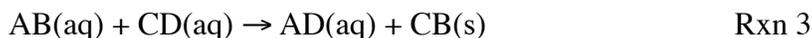
When two electrolyte compounds are mixed together, the ions can in theory rearrange in a **double-displacement reaction**:



However, if all the combinations are soluble strong electrolytes, then nothing actually happens:



The reason is that you had four kinds of ions separate in solution at the beginning, and they are still completely separate at the end; no new chemical bonds have formed. Interesting reactions happen when some combinations are weak or non-electrolytes, or when some combinations are insoluble. If an insoluble combination forms, it will **precipitate**, and you'll see either cloudiness or solid in the mix, such as:



If a soluble weak or non-electrolyte forms, you may notice a temperature or color change (or you may not be able to tell that anything happened at all, even though something did). If a gas forms, you may see bubbling. In this experiment, you'll observe reactions and look for combinations that produce gas, solid, and color and temperature changes. When something interesting happens, figure out what the reaction was! For example, you can try other combinations to isolate which ions produced the observed effect.

Record your observations by writing two types of equations when you believe a reaction happened. First, write a **molecular equation**, like Rxn 3, that shows what two compounds you mixed and what compounds were formed. Then, write a **net ionic equation**. Net ionic equations show only the part that really changed. For example, if AB, CD, and AD are soluble strong electrolytes, but CB is insoluble, Rxn 4 (the **complete ionic equation**) is actually a better description than Rxn 3:



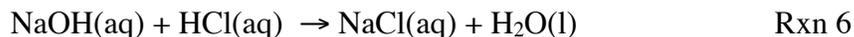
But, complete ionic equations are laborious to write out, and notice that nothing has happened to the A^+ and D^- ions, which are called **spectator ions**. We can cancel them out (cross off both sides), and that leaves the **net ionic equation**, which focuses on the only new interaction:



How to write equations: keep the following very important points in mind:

- Identify the correct charges on each ion! Use the periodic table, or for the polyatomic ions like ammonium and sulfate, if you haven't memorized them (you should!) look them up or figure out the charge based on the formulas given.
- When writing ionic formulas, the ion with the positive charge is always written first
- Only cation/anion (+/- ion) combinations that are *neutral* overall will form. So it's essential to identify the correct charge on all the ions, then write a formula with balanced charges.
- Check that you have simplified the formulas: Na_2Cl_2 should be NaCl .
- Balance the equation.
- Make sure the *charges* balance on both sides of the equation! This is just as important as balancing the atoms. For example, in Rxn 5, the total charge on the left and on the right is zero.
- For net ionic equations, only show aqueous strong electrolytes as dissociated. Weak electrolytes, non-electrolytes, and anything that is solid or gas should not be written as ions, and therefore won't be cancelled out.

Acid-base reactions are a special type of double displacement reaction in which hydrogen ions (one or more) change partners. The simplest example of acid-base neutralization involves hydrogen and hydroxide ions combining to form water, a non-electrolyte. In this case, the salt ions are spectators.



Acid-base reactions can be less obvious to write than some other types of double displacement reactions because you might not know how many hydrogen ions move if some of the compounds have more than one. In general, this will depend on the amounts of each compound used, and you might not be able to tell for sure. For example, carbonate ion is basic, as many anions are. It can react with one hydrogen ion to make bicarbonate (Rxn 7), and with a second to make carbonic acid (Rxn 8). The carbonic acid can undergo a reverse anhydride reaction and produce CO_2 and water.



If you mix sodium carbonate and acid, you might get either bicarbonate or carbon dioxide as product. In this case, you would be able to identify the latter because it would produce bubbling, but in some similar situations you might not be sure what the ratios are. If so, don't worry, just write down one or both of the possibilities.

Usually if a strong acid or base is involved, it will react completely. If both acid and base are weak, it will be much harder to predict what happens. Because they are both weak electrolytes, the reaction can run in both directions. For example,



This reaction will only happen a little bit, maybe to 1% of the total ammonia molecules, but you might still see some evidence that it occurs. If you mix an ammonium salt and a hydroxide salt, it can easily go back in the other direction; now you have a strong base present so the reaction will probably finish.

Safety Precautions:

- Wear your safety goggles at all times.
- Be careful with the strong acids and bases (HCl, Ba(OH)₂, NaOH, etc)
- If a bottle is in the fume hood, leave it there!
- Make very small test mixes: don't use more than 10 drops of any solution
- Wear latex or nitrile gloves
- Close bottles as soon as you are done using them

Waste Disposal:

- Pour your solutions into the waste bottle in the hood when you're done. Use the water squirt bottles to rinse the glassware into the waste bottle.

Procedure

Use the spot plates and/or small test tubes. Try combining the solutions provided by adding about 5 drops each of two solutions to a well or test tube. A few solids are also provided; just add a tiny amount of solid. For each mix you make, record which solutions you used and what you observed. If you think a reaction happened, try to determine what the reaction was by comparing to other combinations! Once you have decided what the products were, if possible, write molecular and net ionic equations for the reaction.

In some cases, you may notice interesting effects that depend on how much of each substance you use. If you want to explore this, start with about 5 drops of one substance and gradually add up to 15 drops of another. Keep the quantities small!

In your notebook, record the balanced molecular and net ionic equations (with s/l/g/aq indicated for each reactant and product) for:

- at least 4 combinations that formed a precipitate
- at least 1 combination that formed a gas
- at least 3 combinations that changed color but stayed clear
- at least 1 combination that changed temperature (for this, use a test tube!)

Suggestions to get you started:

1. Combine a bit of silver(I) nitrate solution with a bit of sodium chloride solution. What happens? If you aren't sure what the product is, try some other combinations to eliminate the possibilities.
2. Combine a bit of barium hydroxide solution and a bit of sulfuric acid solution. What are the products? Can you make this product by other combinations?
3. Combine a concentrated acid solution with a concentrated base solution. Does the temperature change? Try a different combination of acid and base. Does the concentration matter? (Acids include hydrochloric acid, nitric acid, and sulfuric acid; bases include hydroxide compounds, ammonia, and carbonate compounds.)
4. Add a solution of a base to a solution with transition metal cations (iron, copper, or silver). What is the product? Do you get the same product no matter which base you use? Can you tell?

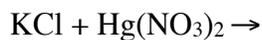
5. Add ammonia dropwise to a bit of copper(II) chloride. What do you observe? How can you explain this? Does anything happen if you add dropwise hydrochloric acid to the mixture, or to plain copper(II) chloride solution?
6. Add a drop of potassium thiocyanate to a bit of iron(III) nitrate solution. What could explain this? What is the phase of the product? What happens when you add a bit of potassium thiocyanate to other solutions, like copper(II) chloride or calcium chloride?

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Experiment 6 Pre-Lab Sheet

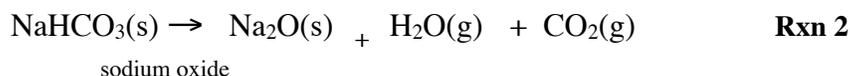
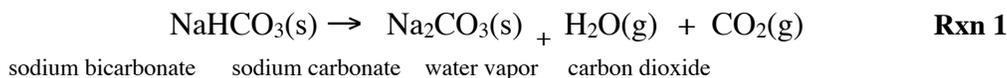
1. (3 pts) Identify the following compounds as acids (A), bases (B), or neutral salts (N):
 H_2SO_4 , NaOH , AgCl , NH_3 , HNO_3 , FeCl_3 , Na_2CO_3 , HCl , NaCl
2. (3 pts) Identify each of the following as strong (S), weak (W) or non-electrolytes (N):
 H_2O , NH_3 , HCl , NaCl , H_2SO_4 , NaOH , KNO_3 , H_2CO_3 , $\text{HC}_2\text{H}_3\text{O}_2$
3. (1 pt) How much of any solution should you use for test mixes, at maximum?
4. (1 pt) Write the net ionic equation for this reaction, assuming CD and AD are strong electrolytes and all ions have single charges:
$$\text{AB(s)} + \text{CD(aq)} \rightarrow \text{AD(aq)} + \text{CB(s)}.$$
5. (2 pts) What new compounds can form from the following mixes? Write balanced molecular equations for double displacement reactions.



Experiment 7 - Thermal Decomposition of Sodium Bicarbonate

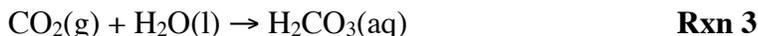
Introduction

Decomposition reactions involve a single substance splitting into several substances. Many ionic compounds containing polyatomic ions can decompose when heated. In the case of sodium bicarbonate (NaHCO_3), when heated above $110\text{ }^\circ\text{C}$ (but not heated to "red heat") both H_2O and CO_2 are produced, and a white solid residue is left when the decomposition is complete. Among possible reactions, the two following unbalanced reactions seem the ones that are most likely to explain these facts:



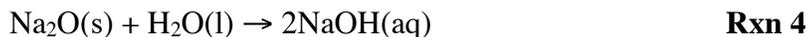
You are to choose whether **Rxn 1** or **Rxn 2** is most supported by your results. Keep in mind that in many chemical processes, multiple reactions occur in competition, so that the reaction that happens fastest will produce the most product before the reactants run out. First, you will heat a weighed sample of sodium bicarbonate in a crucible and determine the mass of solid product. The mass of the solid product ("residue") will suggest whether the product is more likely Na_2CO_3 or Na_2O .

For additional confirmation, you will try a few chemical tests to identify the presence of carbonate ions. To understand these chemical tests, it will be useful to keep in mind the **anhydride** pattern of reactivity. Anhydride means "without water"; anhydrides are oxide compounds that can undergo combination reactions with water. These reactions are often reversible under the right circumstances. There are **acid anhydrides** (non-metal oxides that produce an acid when combined with water) and **basic anhydrides** (metal oxides that produce a base when combined with water). Both appear in this lab. Carbon dioxide can undergo a reversible anhydride combination with water, making carbonic acid, as follows



The reverse of this reaction produces fizzing when acid is added to carbonate salts. The acid restores the carbonic acid (a weak acid, so it is mostly not dissociated), which can then release the carbon dioxide. Thus, if you observe fizzing on adding acid to your product, carbonate or bicarbonate ions are likely present.

In contrast, if the product is sodium oxide, it can still react with acid. In this case, when acid is added, two reactions occur very quickly (probably in competition with each other). One is a basic anhydride reaction, in which sodium oxide reacts with water to make sodium hydroxide:



The second is a standard acid-base reaction between hydroxide ions and the acid. Both of these reactions release heat, so the mixture will probably get hot.

Safety Precautions:

- Wear your safety goggles.
- Use care when handling HCl(aq). If any HCl splashes on your skin or clothes, rinse it off immediately with plenty of water.

Waste Disposal:

- The solid waste may be discarded in one of the regular garbage cans.
- Liquid waste containing barium should be placed in the waste bottle in the hood. Other liquid waste can be washed down the sink.

Procedure**Decomposition Reaction**

1. Obtain a clean, dry crucible that has no cracks. (Ideally, use a copper crucible.)
2. Place the crucible in a clay triangle, on a ring attached to a ring stand. Make certain that the crucible hangs securely in the triangle; ask for help if it seems shaky. Put the cover on the crucible.
3. Heat the crucible and cover for 10 minutes with a high flame, until the bottom of the crucible glows red hot. Allow the crucible to cool in the clay triangle. (This step is to clean out the crucible.)
4. When they have reached room temperature, weigh the crucible and cover. It's a good idea to weigh them separately in case the cover breaks.
5. Add sodium bicarbonate to the crucible until it is 3/4 full. Weigh the crucible, sample, and cover. Obtain the mass of the sample by subtraction.
6. Using a medium-sized flame, heat the sample for 15 minutes. Do NOT heat the sample until it is red hot. Cool it in place as before, and then weigh it. (While you are waiting for the sample to heat and cool, you may start on the calculations.)
7. Heat again, this time for 10 minutes, then cool and weigh as before. If the crucible, cover, and sample have not changed mass during this heating step, we can be reasonably confident that the sodium bicarbonate has been completely decomposed (i.e. no more H₂O or CO₂ is being produced.)
8. If the crucible plus sample weighs less after this second heating than it did after the first heating, repeat the heating/cooling/weighing sequence until the mass is constant (usually three times is sufficient).

Chemical Tests

Carbonate salts such as Na₂CO₃(s) react with acids to form CO₂ gas. If the solid fizzes when some HCl solution is added to it, this suggests that the solid is a carbonate or bicarbonate.

However, Na₂O will also react vigorously with acid, because it is a base, so it may not be easy to distinguish in this way.

9. After all of your heatings and weighings are done, place a bit of your product in a test tube and add a few drops of 6 M hydrochloric acid. Do you observe fizzing or change in temperature? Record your observations.
10. For comparison, put a bit of MgO (a compound similar to Na₂O that we happen to have available) in a new test tube, and add a few drops of 6 M HCl. Does it still fizz? Does it change temperature? Record your observations.
11. As a second test for carbonate ion, dissolve a bit of your product completely in water in another test tube. Then add a few drops of barium hydroxide solution. What do you observe? If you believe a reaction happened, write the molecular and net ionic equation. What does this prove, if anything?

Calculations

1. Balance equations (A) and (B).
2. Starting with the mass of sodium bicarbonate that you used, calculate the mass of Na₂CO₃ that should be produced in theory, assuming that reaction A is the one that actually occurs. Show your work.
3. Starting with the mass of sodium bicarbonate that you started with, calculate the mass of Na₂O that should be produced in theory, assuming that reaction B is the one that actually occurs. Show your work.
4. Compare your observed mass of product with those predicted in step 2 of the calculations for Na₂O_(s) and for Na₂CO_{3(s)}. On the basis of these answers, which product was actually formed?
5. Calculate the percent yield of the reaction. Does the result make sense? Explain.
6. Write molecular and net ionic equations for each of the reactions you observed in steps 9-11.

Name:

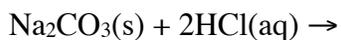
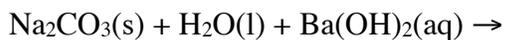
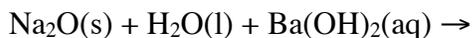
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Experiment 7 Pre-Lab Sheet

1. (2 pts) What is the purpose of this lab?

2. (1 pts) How can you tell when the decomposition reaction is complete?

3. (3 pts) Complete the reactions:



4. (1 pt) Why would it be a problem if you don't dissolve the product completely before adding barium hydroxide?

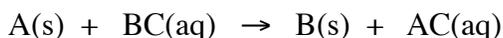
5. (3 pts) Sample calculation: If you perform a similar experiment decomposing $\text{Cu}(\text{OH})_2$ according to the following equation, starting with 6.831 g of $\text{Cu}(\text{OH})_2$, how much CuO should you get, in grams?



Experiment 8 - Single Replacement Reactions

Introduction

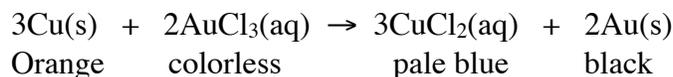
A **single replacement reaction** is a type of **oxidation-reduction reaction**. In a single replacement reaction (also called a single displacement reaction), an element reacts with an ionic compound to give a different free element and a different ionic compound. The general form of a single replacement reaction looks like this:



Where A is an element, BC is an ionic compound consisting of positively charged B ions and negatively charged C ions, B is an element, and AC consists of positively charged A ions and negatively charged C ions. It is called a “single replacement” reaction because it appears as though the element A is replacing B in its compound. (In actuality, BC is usually a soluble compound, so it consists of separated B^{x+} ions and C^{x-} ions. Since they are already separated, it isn't really accurate to say that any replacement is occurring.) If this reaction ($A + BC \rightarrow B + AC$) occurs as written, it is said that A is more active than B. If it does not occur, B must be more active than A. (We are assuming, of course, that A and B do not have the same exact activity.)

Recall that oxidation-reduction reactions involve a transfer of electrons. The substance that gets oxidized loses electrons and its oxidation number increases. On the other hand, the substance that gets reduced gains electrons and its oxidation number decreases. In a reaction of this type, the element A goes from a zero oxidation number (pure elements are always neutral and uncharged) to a positive oxidation number. Its oxidation number increases, so it is being oxidized. The substance B, however, goes from a positive to a zero oxidation number, and is therefore being reduced. Therefore, **the more active element is the one that is oxidized more easily**.

To use a specific example, if a strip of copper metal is immersed in a solution of gold (III) chloride, a reaction occurs:



Two electrons are taken from each copper atom, and transferred to a gold (III) ion, converting it to a neutral gold atom. The process simultaneously converts Cu to Cu^{2+} , the copper (II) ion. Copper is thus more active than gold.

The reverse is not the case. That is, Cu^{2+} cannot oxidize Au (it cannot take electrons from Au). This can be proven by adding solid gold to a solution of CuCl_2 : nothing happens. If a certain reaction occurs spontaneously (in our example, the transfer of 2 electrons to Au^{3+} from Cu metal) the reverse process (transfer of 2 electrons from solid Au to Cu^{2+}) will NOT be spontaneous.

The purpose of this experiment is to determine relative activities of different elements by combining elements with aqueous solutions of ionic compounds. By looking at the surface of the metal, we can decide whether or not the oxidation-reduction reaction has occurred. If the reaction is happening, a coating will form on the surface of the metal. In cases where reaction does occur, it will mean that the metal of the strip is undergoing oxidation and that the other metal's ions are gaining electrons (being reduced), forming a coating of that element on the surface of the

original metal. Redox reactions are not always immediate, so check after 10 minutes to see if anything has happened.

Acidic solutions can also undergo single replacement reactions with some metals. For example, when a piece of manganese metal is placed in a solution of HCl, there is visual evidence of a reaction. The metal begins to dissolve and a gas is formed, which bubbles out of the solution. The reaction that is happening is the following:



The gas is hydrogen gas, H₂. The manganese dissolves to become manganese(II) ions. In this case, the manganese is displacing the hydrogen from its compound, so the manganese is more active than hydrogen. The hydrogen goes from a +1 oxidation state to a zero oxidation state in the elemental form. Metals that react with any acid are referred to as **active metals**. (Some metals react only with specific acids, such as nitric acid or aqua regia (“king’s water”), a mix of HCl and HNO₃ which is the only acid that dissolves gold.)

Whether or not the reactions occur, each combination will tell you which of the two elements is more active. You will test five different metals and acid in this experiment. When all of the data is obtained, you will rank the substances in order of activity.

Safety Precautions:

- Wear safety goggles.
- Lead (Pb) metal and solutions containing lead ions are poisonous. Make sure to wash your hands after handling any lead or lead compounds.
- Silver nitrate (AgNO₃) solutions will stain skin and clothes. If you suspect you may have spilled AgNO₃ on yourself, rinse it off immediately. The stains are dark brown and they don’t show up right away. You’ll know the next day whether or not you spilled AgNO₃ on yourself.

Waste Disposal:

- While you are doing the experiment, pour your liquid waste into a beaker. Separate the solid metal pieces from the waste solution and sort them by type.
- Silver pieces must be saved, rinsed off, and given back to the instructor.
- Other metal pieces should be placed in the appropriate container in the waste hood.
- When you are finished with the experiment, pour the contents of the waste beaker (liquid waste only) into the **inorganic waste** container in the fume hood.

Procedure

With some of the combinations used in these experiments the reactions may be slow or difficult to detect. If you see no immediate evidence of reaction, set the tube aside and allow it to stand for about 10 minutes, then reexamine it.

Evidence of reaction will be either evolution of a gas (such as many bubbles on the metal surface) or appearance of a metallic deposit on the surface of the metal strip. Metals deposited from a solution are often black or gray (in the case of copper, very dark reddish brown) and do not resemble commercially prepared metals.

See the following page for a sample data table. Use a whole page for the table so you can record detailed observations for each reaction.

1. Obtain five pieces each of zinc, copper, lead, and magnesium. Obtain one piece of silver from the instructor. Clean the metal pieces with fine sandpaper or steel wool until their surfaces are clean and shiny. This is important, because coatings on their surfaces can inhibit (slow down) chemical reactions.
2. Rinse a well plate with deionized water and shake out the excess water.
3. In separate wells, place one piece of each metal except copper. Fill the well almost to the top with copper (II) nitrate solution. Observe the metal pieces for evidence of reaction. Record your results.
4. In separate wells, place one piece of each metal except lead. Fill the well almost to the top with lead (II) nitrate solution. Observe the metal pieces for evidence of reaction. Record your results.
5. In separate wells, place one piece of each metal except magnesium. Fill the well almost to the top with magnesium sulfate solution. Observe the metal pieces for evidence of reaction. Record your results.
6. In separate wells, place one piece of each metal except zinc. Fill the well almost to the top with zinc (II) sulfate solution. Observe the metal pieces for evidence of reaction. Record your results.
7. In separate wells, place one piece of each metal except silver. Fill the well almost to the top with silver (I) nitrate solution. Observe the metal pieces for evidence of reaction. Record your results.
8. Rinse and shake **five** test tubes and set them up in a test tube rack. Place about 1 mL of hydrochloric acid solution into each tube. Add a different metal piece to each tube. Record your results.
9. Dispose of all waste as previously directed. Rinse off the silver metal and return it to the instructor.
10. Write net ionic equations for each reaction that happened in your notebook. Also write one molecular and complete ionic equation for each type of solution that reacted.
11. On the basis of your observations, rank all of the metals and hydrogen in order from the most to the least active.

Suggested Data Table (but use a whole page of your notebook so you have space for detailed observations!)

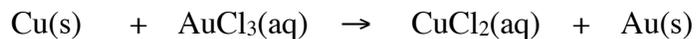
	Cu(s)	Pb(s)	Mg(s)	Zn(s)	Ag(s)
copper(II) nitrate (aq)					
lead(II) nitrate (aq)					
magnesium sulfate (aq)					
zinc(II) sulfate (aq)					
silver(I) nitrate (aq)					
HCl (aq)					

Name:

Section: Tu Th Fam Fpm

Experiment 8 Pre-Lab Sheet

1. (2 pts) What is the objective of this lab?
2. (1 pts) Does the volume of solution used matter? How much should you use for each test?
3. (1 pts) How long should you wait to see if a reaction happens?
4. (2 pts) Why is polishing the metals pieces useful?
5. (4 pts) Write the balanced net ionic equation for each reaction (assume aqueous ionic compounds are strong electrolytes, although this may not be completely true since the cations are transition metals):



Experiment 9 - Heats of Reactions

Introduction

The most important applications of chemistry (from factories to your body) involve making chemicals that are useful, or burning chemicals for energy. To do either one efficiently, we have to understand energy changes associated with chemical reactions: how much energy it costs to make a compound, or how much energy we can gain from a reaction. This part of chemistry is called **calorimetry**, which means “heat measurement”. The most important concept to remember is the **Conservation of Energy**, which means that energy is not created or destroyed, it just changes form and moves between substances. For example, when gasoline is burned in a car engine, it gets hot because energy is released by the combustion reaction. The reactants had more internal energy than the products, and the energy released by the reaction heats up the products, and their surroundings, and is used to drive the car. (Don’t get confused: the engine gets hot because of energy released by the fuel, not because energy is entering from the surroundings. In fact, heat leaves the engine and warms the surroundings, because the engine is hotter.)

If we want to know how much heat (and thus how much energy to do work, like driving the car) we can get from burning a gallon of gasoline, we have to collect the energy released by the reaction in some measurable way. Usually this is done using a **calorimeter**. The calorimeter is an insulated container in which we run the reaction. The calorimeter, or the material inside it, has a known **heat capacity**. Heat capacity (unit J/°C) is the amount of energy required to raise the temperature of the material by 1 °C. Since the container is insulated, we assume that the energy absorbed or released by the reaction is all used to change the temperature of the calorimeter and the material inside it. If we measure the temperature change, and know the heat capacity, then we can calculate the energy absorbed or released by the reaction.

In this experiment, we will measure heats of reaction using styrofoam cups as calorimeters. Our reactions will run in aqueous solution. Because water has a high heat capacity, we can assume that the heat capacity of the whole system (cups, products and water) is equal to the heat capacity of the solution. We will find the heat capacity of the solution using the **specific heat**, or heat capacity/gram, which has units of J/g°C. The equation to relate specific heat to heat is (nicknamed mCAT), is

$$q = mC_s\Delta T \quad \text{Eq 1}$$

where q is the heat required to cause temperature change ΔT (where $\Delta T = T_{final} - T_{initial}$) in m grams of a substance with specific heat C_s .

To summarize, the conservation of energy tells us that

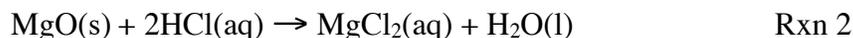
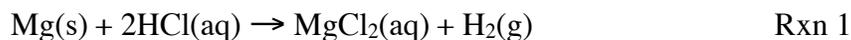
$$q_{rxn} + q_{solution} = 0 \quad \text{Eq 2}$$

where q_{rxn} is the enthalpy change of the reaction, or ΔH_{rxn} , and $q_{solution}$ is the heat required for the observed change in temperature of the solution. By plugging in Eq 1 (“mCAT”) for $q_{solution}$, we can solve Eq 2 to find ΔH_{rxn} . Eq 2 tells us that if ΔH_{rxn} is positive (endothermic) then $q_{solution}$ must be negative, implying that the temperature of the solution decreases. Likewise, if ΔH_{rxn} is negative (exothermic) then $q_{solution}$ must be positive, implying that the temperature of the solution increases. The enthalpy of reaction is defined as the difference in enthalpy of the products and reactants:

$$\Delta H_{rxn} = \Delta H_{products} - \Delta H_{reactants} \quad \text{Eq 3}$$

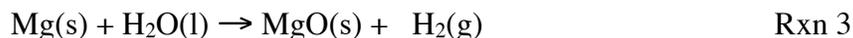
Finally, enthalpies of reaction are usually expressed as kJ/mol of reaction, so you will need to divide by the moles of reaction that occurred.

The reactions we will study are:

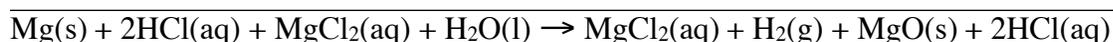
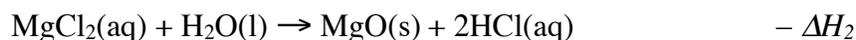
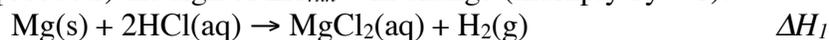


In each case, the solid reactant (Mg or MgO) will be the limiting reactant. Since both have a coefficient of 1 in the equation, moles of Mg or MgO is equal to moles of reaction.

Finally, once we have measured the ΔH_{rxn} for Rxn 1 and Rxn 2, we can use the conservation of energy again in the form of Hess' Law to find the ΔH_{rxn} for Rxn 3:



Hess' Law says that if you add 2 reactions together, the total enthalpy of reaction will be the sum of the individual ΔH_{rxn} values. Remember that if you reverse the direction of a reaction (swap the reactants and products) the sign of ΔH_{rxn} will change (multiply by -1).



Add up the reactions, then cancel "spectators" to get reaction 3:



Safety Precautions:

- Wear your safety goggles.
- Solutions of hydrochloric acid (HCl) are corrosive. If HCl splashes on you, rinse it off immediately with plenty of running water.

Waste Disposal:

- Neutralize your liquid waste with sodium bicarbonate in a large beaker, then pour it down the sink with plenty of running water.
- Please be careful when weighing the MgO. If you spill any, clean it up!
- Rinse and save the styrofoam cups unless they are broken.

Part 1

First, we will measure the enthalpy of Rxn 1. A measured amount of hydrochloric acid solution will be placed in a calorimeter. Magnesium ribbon will be added, and the heat produced by the reaction will be measured by observing the temperature change (ΔT) of the HCl solution. Record all your raw data (masses and temperatures) directly into a table in your notebook.

Procedure – Part 1

1. Weigh a dry, empty calorimeter (two Styrofoam cups in a stack).
2. Measure out about 100 mL of 1 M HCl in a graduated cylinder. Record the precise volume used.

3. Add the 1 M HCl solution to the calorimeter and weigh it again. Subtraction gives the mass of the HCl solution. The specific heat of 1 M HCl solution is 4.04 J/g°C.
4. Allow the temperature of the acid and the calorimeter to equilibrate for a few minutes. While you are waiting, proceed with step 5.
5. Obtain a strip a magnesium ribbon that weighs between 0.2 and 0.3 grams. If the magnesium doesn't look shiny, clean it with steel wool before weighing. Weigh the magnesium strip to the nearest ± 0.001 or ± 0.0001 grams on a digital balance and record the mass.
6. Wind the magnesium ribbon around a pencil to give it a compact, spiral shape.
7. Set up a ring stand with an iron ring, and set the calorimeter under the ring. (This ring will be used to help support the thermometer.) Read and record the temperature of the HCl in the calorimeter. The temperature should be read to the nearest ± 0.1 °C. Leave the thermometer in the calorimeter for the rest of the experiment, supported inside the ring on the ring stand so that it doesn't fall over and break.
8. Drop the magnesium into the acid solution in the calorimeter. If necessary, use the thermometer to hold the ribbon under the surface of the acid.
9. Stir or swirl the calorimeter frequently. As the magnesium reacts, observe the temperature. Record the maximum temperature attained (again, to the nearest 0.1°C). This should be reached as the reaction is ending. Wait for a while after the magnesium has completely reacted to be sure you have observed the maximum temperature.
10. Pour the contents of the calorimeter into a large beaker. Neutralize the solution with sodium bicarbonate, if available, and discard the solution in the sink.
11. **Repeat the entire procedure to collect a second data set.**

Calculations – Part 1

1. Use your experimental data to calculate the energy absorbed by the solution, using Equation 1. Assume the specific heat of the solution is 4.04 J/g°C.
2. Calculate the number of moles of reaction that occurred (using the moles of the limiting reactant).
3. Find ΔH_{rxn} using Equation 2, in units of kJ/mol.
4. Repeat calculations 1-3 for your second trial.
5. Calculate the average value of ΔH_{rxn} and the percent difference between the two values.
6. Write your name and results on the board.

Part 2

Second, we will measure the enthalpy of reaction for Rxn 2. The procedure for studying this reaction is essentially the same as that used in Part 1. Magnesium oxide (MgO) is a white powder. The MgO will be poured into an excess of HCl in a calorimeter and the temperature change will be measured. In this part of the experiment, 2 M HCl (specific heat = 3.89 J/g°C) will be used instead of 1 M HCl. In this part, you will calculate how much of the reactants to use using an approximate value for the enthalpy of reaction, then find the experimental enthalpy of reaction using your actual data.

Pre-Calculations - Part 2

Assuming that you use about 100 mL of solution, and the enthalpy of reaction for Rxn 2 is approximately -130 kJ/mol, how many moles of reaction will cause the solution to increase in temperature by 25 °C?

1. How many kJ are needed to heat the 100 mL of solution by 25 °C? Assume the solution has a density of about 1 g/mL.
2. How many moles of MgO are needed to produce this much heat, if the heat of reaction is approximately -130 kJ/mol?
3. How many grams of MgO does this correspond to?
4. Check that MgO is the limiting reactant under these conditions (there should be plenty of excess HCl).
5. Check your calculations with the instructor before proceeding.

Procedure – Part 2

1. **Before you start, you have to do the pre-calculations!** See above. You will use approximately the same amount of MgO you calculated. It doesn't need to be exactly the same amount.
2. Repeat the same procedure you used in Part 1, but use 2 M HCl and the mass of MgO that you calculated (to within 0.05 g). Weigh the MgO on a weigh boat and slowly pour it directly from the paper to the calorimeter, then stir vigorously. Record the masses and temperatures in a table in your notebook, as you did in Part 1. Again, be sure to observe the maximum temperature reached by the system (± 0.1 °C).
3. When the reaction is complete, check to see that a clear solution remains in the calorimeter. If the solution isn't clear, then some unreacted MgO remained, and you will have to repeat the experiment with less MgO so that it is the limiting reactant.
4. **Repeat the procedure**, as before. The amount of MgO can be changed, if desired, for the second run, but make sure it is still the limiting reactant.

Calculations – Part 2

Calculate ΔH_{rxn} for Part 2 as you did in Part 1. Again, calculate ΔH_{rxn} separately for each trial, and report the average and the percent difference of the two trials. Although you've been told that it is close to -130 kJ/mol, you can't assume this value in your calculations: the purpose of the experiment is to measure this value!

Part 3

If you want to know ΔH_{rxn} for Rxn 3, what do you do?

1. Try putting a small scrap (1 cm or less) of Mg ribbon in plain water. Does anything happen? Could you measure a heat change for this reaction as you did in Part 1 and Part 2? Why or why not? If you could, would the heat you measured correspond exactly to Rxn 3 as written?
2. Predict whether Rxn 3 is endothermic or exothermic. Explain (you can use your observations, general knowledge, etc).
3. Use Hess's Law to find ΔH_{rxn} for Rxn 3 using your average ΔH_{rxn} values for Rxn 1 and Rxn 2. (Figure out how to add Rxn 1 and Rxn 2 to give you Rxn 3. You can cancel formulas that appear on both sides of the reaction; they are "spectator substances" just like when you write net ionic equations.)

Experiment 10 - Evidence for Quantum Mechanics

Introduction

According to quantum mechanics, electrons exist in atoms in distinct, clearly separated **energy levels** or **states**, like stairs, instead of a smooth ramp of energies. The energy of the states is like the height of the step. When atoms absorb energy (by being heated or irradiated with high energy light) the electrons in that atom are excited to higher energy levels (higher steps). These electrons are unstable in their **excited state**, and they release their excess energy by falling back down to lower energy levels (lower steps), giving off light in the process. Electrons can jump or fall many steps at a time, but must start and end *on* a step, not *in between* steps. The wavelengths of light given off correspond to the energy changes that the electrons undergo (the height difference of initial and final steps). The change in energy of the electron is given by

$$\Delta E_{\text{electron}} = E_f - E_i \quad \text{Eq 1}$$

where E_i is the energy of the electron's initial state and E_f is the energy of the electron's final state. Lower energy states are more stable (electrons tend to "fall" to lower energy states, just like objects tend to fall to lower positions); the lowest energy state is called the **ground state**. The zero of energy is usually chosen to be the energy of a "free electron" that isn't bound to any atom. Electrons in atoms or molecules are more stable than free electrons, and thus have negative energies. Thus, the energy of an energy level is the energy required to remove the electron from that energy level. When electrons change states, they often either emit or absorb a **photon** (light particle) whose energy is the change in energy of the electron (absorbing a photon excites electrons to higher energy states, while relaxing to lower energy states releases a photon). The energy of the photon is given by

$$\Delta E_{\text{electron}} = E_{\text{photon}} \quad \text{Eq 2}$$

Recall that there is a simple relationship between the energy of a photon and its frequency and wavelength:

$$E_{\text{photon}} = h\nu = hc/\lambda \quad \text{Eq 3}$$

Where h is Planck's constant (6.626×10^{-34} Js), ν is the frequency of the photon, c is the speed of light (3.0×10^8 m/s) and λ is the wavelength of the photon.

Each element has a different emission spectrum, because each element's electrons have different energy levels and different spacings of those energy levels. These energy levels depend

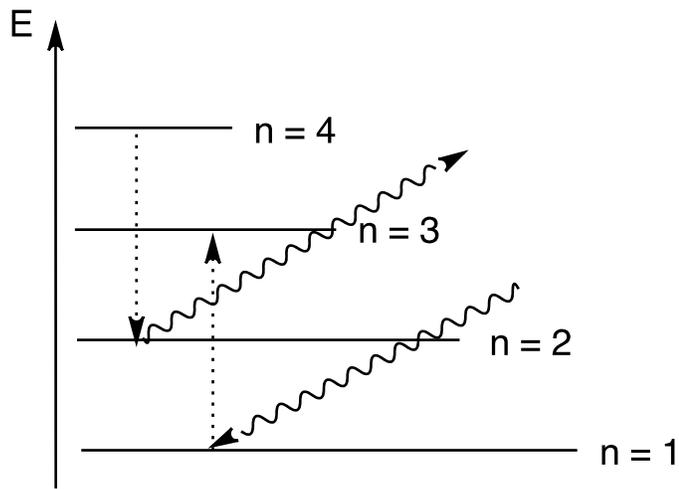


Figure 1: Energy level diagram. Dotted arrows show transitions between energy levels. Wavy arrows show photons being emitted (as in the transition from 4 to 2) or absorbed (as in the transition from 1 to 3).

largely on electrostatic interactions between electrons and nuclei. The nuclear charge, number of electrons, and distance between the electrons and nucleus help determine energy levels. In this experiment the spectra of a number of elements will be generated in two ways: by heating in a burner flame, and by electric discharge in a sealed glass tube. The objectives of this lab are generally to explore the experimental evidence for quantum mechanics, and more specifically:

1. To observe the color of light emitted when a sample of a chemical is heated, and to use this color as a means of identifying a few positive ions;
2. To observe various light sources with a spectroscope, which works much like a prism works to separate white light into a rainbow of colors. In particular, we'll look at the emission spectra of gaseous elements in discharge tubes, in which a pure elemental gas is heated very hot by a high voltage, until it emits light.

Safety Precautions:

- Wear your safety goggles.
- Use caution when handling the HCl.

Waste disposal:

- Used solutions of metal ions should go in the INORGANIC WASTE bottles (which have a blue label) in one of the fume hoods after the experiment.

Procedure

You can do the parts in either order. The hydrogen emission tube can only run for about 30 minutes or it will stop giving good data. Take turns observing it while it is running, and work on other parts of the lab while you wait for a turn.

Part 1 - Spectroscopic Examination

Most natural light is a mixture of different wavelengths, each a different color. The spectroscope is an instrument that separates any beam of light into its constituent wavelengths, and spreads these separated beams out, so they can be seen. (The action of a spectroscope is similar to the action of a prism.)

If the light that is shone into a spectroscope consists of a very large number of wavelengths, each only very slightly different from the ones next to it, the separated light makes what is called a **continuous spectrum**. It looks like a rainbow of colors, each one merging into the next. An actual rainbow is a continuous spectrum that you see when the atmosphere acts like a giant spectroscope.

Discrete spectra or line spectra are those that consist of just a very few wavelengths of light, all of which are sufficiently different so that when separated, they fall at widely-spaced intervals, with large dark gaps in between them. This type of spectrum looks like separate lines of colored light that have spaces between them. Such spectra occur when elements in the form of very thin gases are heated. It was the study of these line emission spectra that led to the modern theory of electronic energy levels in the atom. The fact that only certain wavelengths of light were produced was evidence that the energy levels of electrons in atoms were *quantized* (only certain energies were possible).

1. Look through a spectroscope at daylight or a regular tungsten light bulb. Point the small window directly at the light, then look through the narrow end, and off to the side where the numbers are. Is this a continuous or line spectrum?

2. Look through the spectroscope at the overhead fluorescent light bulb. (This works best if you stand directly under the light and point the spectroscope opening directly at the tube.) Which type of spectrum is this? How is it different from the regular light bulb?
3. If you want, take a spectroscope outside and look at the streetlights with it. What colors do you see?
4. Use the spectroscope to look through the cobalt glass at the regular lightbulb. What changes compared to the spectrum without the glass? What colors are transmitted, and which are absorbed by the glass?
5. Without moving any of the spectroscopes, look at the displayed elements in gas discharge tubes, and record the spectrum produced by each. Sketch the spectrum of each discharge tube, using colored pencils if you wish.
6. Carefully record the wavelengths (in nanometers) of the lines in the hydrogen emission spectrum from the hydrogen discharge tube. Also record the energies in eV (the other scale on the spectrometer.)
7. Carefully record the wavelengths and energies of the lines from the helium discharge tube.

Calculations

1. Calculate the frequency (in Hz) and energy (in J/photon and kJ/mol) of the photons emitted by the hydrogen emission tube using the wavelengths you recorded.
2. Using the Bohr formula for the energies of stable orbits (energy levels),

$$E_n = -Z^2(13.6 \text{ eV})/n^2$$

where n is the quantum number which identifies the energy level, Z is the nuclear charge (atomic number), and E_n is the energy of the n^{th} level, find the energies of the first six levels ($n = 1 - 6$).

3. Draw an energy level diagram (similar to Figure 1) for the hydrogen atom showing the relative spacing of the energy levels, and the transitions you observed. Be careful to draw a real energy axis, to scale, and show the actual differences in the energy levels.
4. Your energies for the hydrogen emission lines should be equal to the difference between some of these energy levels. Which transitions do your lines match? Give the initial and final n for each line and show how it fits Eq 2.

Part 2 - Flame Tests

In this part of the experiment, different solutions or powders containing metal ions are heated in a Bunsen burner flame. The flame excites the electrons to higher energy levels. As the excited electrons fall back down to lower energy levels, they give off light of specific wavelengths. A number of different wavelengths (each with a different color) are emitted in each case, but without a spectroscope to separate the wavelengths, the eye merely sees them as a single color. Sometimes this color is distinctive enough that it can be used to identify the atom or molecule that produces it.

1. Obtain a piece of nickel/chrome wire about 15 cm long. Collect a few milliliters of 6 M HCl in a test tube for use as a cleaning solution. Small short test tubes are best. You may also use several test tubes with HCl for a two-step rinse.
2. Bend one end of the wire into the smallest possible loop, about 1 mm in diameter. This loop will hold a drop of solution when you are doing the flame tests.

3. Clean the wire by first dipping it into 6 M HCl, then holding it in the hottest part of the flame. Repeat until you no longer see any significant color to the flame that comes off of the wire. *Repeat this cleaning process between each sample tested.*
4. To test a solution, let a drop of the solution fall onto the loop of the wire. (Do not let the dropper itself touch the wire; this could cause contamination of the solution in the dropper bottle.)
5. Hold the drop of solution in the burner flame, recording the color you see. **Note:** Na⁺ ion gives an especially strong and persistent color. This ion is present as an impurity in most solutions, since glass bottles contain Na⁺ which contaminates the solution. The strong color of Na⁺ in the flame can obscure other colors such as the pale lavender of K⁺. To avoid this difficulty, some people find it helpful to look at the flame through a piece of blue glass, which should remove the sodium flame color. Colors that are not orange should come through. It is also a good idea to test the sodium solutions last.
6. Test the known solutions containing K⁺, Ba²⁺, Ca²⁺, Cu²⁺, Sr²⁺, and Na⁺. In each case, record the color of the flame produced; check the colors with and without cobalt glass. Then do flame tests on various miscellaneous items, as follows:
 1. purified water, from a squeeze bottle
 2. tap water
 3. a drop of your saliva
7. Test an unknown liquid to find out which positive ion is present. (It will be one of the six ions you already tested in solution.) Report the flame color observed and your conclusion as to the identity of the ion. **IMPORTANT:** each person should test his or her own unknown. You and your lab partner must use different unknowns.
8. For fun, more spectacular results you may test some solids as well. To test solids, get a bit of the solid or powder stuck to your loop by dipping in distilled water, then in powder, then hold the loop in the flame. Use very little of the solid or it might be very difficult to clean the loop. As before, clean the loop with HCl in between each measurement, and you may use the cobalt glass to eliminate the sodium flame color if you wish. Solid powders provided might include: Mn²⁺, Fe²⁺, and Fe³⁺ salts, CuCl₂, CuSO₄, boric acid, and cream of tartar.

Name:

Section: Tu Th Fam Fpm

Experiment 10 Pre-Lab Sheet

1. (2 pts) What is the difference between a continuous and a discrete spectrum?
2. (2 pts) Where do the lines in a line spectrum come from?
3. (2 pts) The energy levels in the hydrogen atom have energies $E_n = -C/n^2$. Why are the energies negative?
4. (2 pts) What is the frequency of a photon with a wavelength of 315 nm? Show the calculation with all the units.
5. (2 pts) What is the energy in kJ/mol of photons with a wavelength of 315 nm? Show the calculation with all the units.

Experiment 11 - Acid-Base Titration

Introduction

A **titration** is an experimental technique for determining the **molarity** of a solution by reaction with something else. Recall that molarity of a solution is defined as moles of solute per liter of solution, so a 1 M (“one molar”) solution has 1 mole of solute in 1 liter of solution.

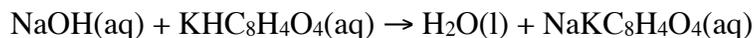
To perform a titration, a carefully measured amount of one reactant is added to an Erlenmeyer flask. An **indicator** is added that will signal the **endpoint** of the titration by a visible color change. Then the other reactant is added slowly to the flask using a buret. When the indicator changes color, the reaction is complete. At this point, you assume that you have added exactly the right number of moles of the second reactant to completely react with the first (the mole ratio used exactly matches the ratio in the balanced equation). Since you know how much of the first you started with, and how much of the second you added (using the buret), you can calculate the concentration of the unknown solution using stoichiometry.

Titration are often performed to measure concentration of acidic or basic solutions. Acid-base titrations are convenient because there are many appropriate indicators. In this lab, we will use **phenolphthalein** as our indicator. Phenolphthalein is clear (colorless) in acidic solution, but pink in basic solution. In each titration, we will put the acidic solution in Erlenmeyer flask, and titrate in the basic solution from the buret. The solution in the flask will start clear, and turn pink when the reaction is complete.

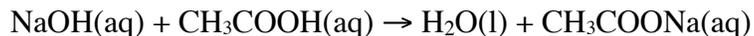
The ultimate goal of this lab is the measure the concentration of acetic acid in vinegar. However, we will do this in several steps over 2 weeks. In Part 1, we will make a **standard solution** of an acid called potassium hydrogen phthalate, abbreviated KHP. (It is *not* potassium hydrogen phosphide—this doesn’t charge balance correctly because phosphide ions are 3-.) The chemical formula of potassium hydrogen phthalate is $\text{KHC}_8\text{H}_4\text{O}_4$. This is a solid weak acid that can be measured conveniently and exactly and is available very pure, allowing you to make a solution of precisely-known concentration (to 4 significant figures).

In Part 2, you will use your KHP standard solution to determine the precise concentration of a NaOH solution. The reason we can’t simply prepare the sodium hydroxide solution with a concentration known to 4 significant figures is that NaOH is hygroscopic, meaning it absorbs water from the air. Thus, it won’t be perfectly pure, so we can’t weigh it out and know exactly how much we have. For this reason, we find the NaOH concentration by titrating with the standard solution of KHP. This process is called “standardization” of the NaOH solution. Once you know the concentration of the NaOH solution, you can use it to titrate vinegar, and find the precise concentration of the vinegar.

The reaction between NaOH and KHP is



and the reaction between NaOH and vinegar is



As you may have noticed, this experiment emphasizes accuracy and precision. Unlike many experiments we do, with titrations it is possible to be extremely accurate and precise, because there are no major limitations in the equipment or the method. Your goal is to achieve the maximum accuracy and minimum uncertainty you can. Since we want our final answer to have 4

significant figures, we care about the difference between, for example, 0.1001 and 0.1008 M. Because this difference is quite small, mistakes that may seem small can have an important effect! For this reason, you have to think carefully about what you are doing, such as when it is acceptable to use wet glassware or add DI water, and when any additional water would destroy your accuracy.

Chemists often use square brackets around a formula to indicate the molarity of that substance. For example, [NaOH] means the molarity of NaOH. So, for example, the KHP solution you prepare should have [KHP] = 0.1 M.

Safety Precautions:

- Wear your safety goggles.
- If any acid or base solution splashes on you, rinse it off immediately.

Waste Disposal:

- Waste from this experiment may be safely discarded down the drain using plenty of running water.

Procedure

You can work in pairs for Part 1, but you must work by yourself for Part 2 and Part 3.

Part 1: Preparation of Standard KHP Solution

In Part 1, you will prepare exactly 250 mL of a solution that is approximately 0.1 M in KHP (the standard acid). The solution doesn't have to be exactly 0.1 M, but you do need to know precisely what the concentration actually is (to at least 4 significant figures). To know the concentration precisely, you will need to know precisely how much KHP you used, and precisely how much solution you made. This means that all the KHP you weigh out must go into the solution, and you must know the total volume of the solution exactly, which you do by using a volumetric flask and filling it exactly to the line.

1. Wash a 250 mL volumetric flask with DI water (it should have a single line on the narrow neck—check if you aren't sure what a volumetric flask is). While you wash it, practice filling it exactly to the line on the neck. Use a squirt bottle with DI water or a dropper to get the meniscus exactly on the line. It's worth practicing because when you make your solution for real, if you add water over the line, you'll have to start over.
2. Collect the required mass (within 0.5 g of what you calculated on the pre-lab) of KHP in a clean, dry weigh boat and record the mass to at least 3 decimal places. (Be sure to tare the weigh boat on the balance before you put the KHP in, so you don't include the mass of the weigh boat.)
3. Very carefully, pour the KHP into your volumetric flask. You can use a clean funnel (rinsed with DI water). Use a squirt bottle with DI water to rinse all the KHP off the weigh boat and funnel and into the flask. Also wash the KHP down the neck of the flask into the bottom of the flask. Make sure you don't spill any KHP or leave any in the weigh boat. If you spill any, you need to wash the flask and start over.
4. Add DI water until the round part of the flask is half full, then swirl vigorously to dissolve the KHP completely. This might take a few minutes, and it's much easier if there is still

plenty of space in the flask. Make sure you don't spill any of the solution! If you do, you need to start over.

5. Add more DI water until the round part of the flask is mostly full, rinsing the sides of the flask and the neck as you add it. Mix again.
6. Add DI water carefully until it gets close to the line on the neck. Then add DI water *very carefully* (use a dropper) until the meniscus of the solution is exactly on the line. **Do not go over** the line; if you do you need to start over.
7. Use a stopper with parafilm to cover the flask. Invert and swirl the flask continuously for 5 minutes to thoroughly mix the solution. Make sure you don't spill any of the solution before it's fully mixed! If you do, you need to start over.
8. Keep the solution stoppered when you aren't using it.
9. Using the mass of KHP you actually used, calculate the concentration of your solution to 4 significant figures. The final volume (if you got it exactly on the line) is 0.2500 L. (This means you'll need to use at least 5 sig figs in the atomic weights when you find the molecular weight of KHP. Make sure you keep at least 5 sig figs in all the middle steps of the calculation.)

Part 2: Standardization of NaOH

For this part, your job is to measure the concentration of the NaOH to 4 significant figures. To do this, you will titrate the NaOH with your standard KHP solution at least 3 times, until your three best results have an average deviation less than 0.003 M. If they don't, you will need to do additional titrations. Save all the data—don't cross out trials that deviate more, even if you choose not to include them in your averages.

1. Collect about 250 mL of the NaOH solution (roughly 0.1 M) which is provided in a clean 500 mL flask. It's ok if the flask is wet with DI water from cleaning. Mix it thoroughly and stopper it. Label it with your name, the approximate concentration ("0.1 M NaOH") and the number on the bottle (record this in your notebook also). Don't refill this bottle later.
2. Get a small (25 mL) buret. Rinse it with DI water several times, letting the water run out the bottom and rinsing the sides. Then rinse it **3 times** with your KHP solution. Each rinse, use about 5 mL. Roll the buret so the sides are completely rinsed, then let the solution run out the bottom of the buret. This is called "**conditioning the buret**". This ensures that your solution isn't diluted or contaminated by anything when you put it in the buret, so the concentration doesn't change.
3. Once the buret is conditioned, fill it (no higher than the markings) with your KHP solution. You can share the KHP buret with your lab partner (who also shares your KHP solution). Drain 1-2 mL of the solution out the bottom of the buret to make sure there are no air bubbles. (Use a buret clamp to hold it up.)
4. Get another 50 mL (preferably) or 25 mL buret. Condition it again, this time using first water, then your NaOH solution. Fill it with NaOH and drain 1-2 mL out the bottom. You will probably want to have your own NaOH buret separate from your partner.
5. Separately from your partner, get a clean Erlenmeyer flask (100 or 250 mL is fine). You can rinse it with DI water and it's ok if it is wet.
6. In a table in your notebook, record the initial reading of the buret to 2 decimal places (such as 1.24 mL). Then drain 10-15 mL of the KHP into the Erlenmeyer flask. Touch the tip of the

buret to the side of the flask to get the last drop in. Record the final buret reading in the table in your notebook (always to 2 decimal places).

7. Add 2-3 drops of phenolphthalein indicator to your flask. Swirl to mix.
8. Record the initial reading on your NaOH buret. Drain the NaOH into your flask, swirling constantly. You can rinse the sides of the flask sometimes with DI water. Add the NaOH quickly at first, then slower when you expect you are getting close to the end point.
9. When the solution in the flask stays very slightly pink after swirling, you have reached the end point. Record the final volume on the NaOH buret.
10. Rinse the flask and repeat the titration (steps 6-9) at least twice. For each titration, you need to record the initial and final reading on each buret. This is the data you will use for your calculations. Try to get the color of the solution at the endpoint as light as you can (add the NaOH dropwise, swirling in between each drop, when you get close to the endpoint).
11. Do your calculations and check with your instructor. If your trials are not close enough together, you will have to do more trials.
12. Once your instructor approves your results, close the NaOH solution securely and label it with your name and the precise average concentration you calculated. Store it as directed for next week; you will use this same solution for Part 3.
13. Dispose of the KHP solution in the waste bottle in the hood.

Calculations for Part 2

1. For each trial (titration), calculate the concentration of the NaOH to 4 significant figures. (Find the moles of KHP in the flask at the beginning, and assume you added the same number of moles of NaOH.)
2. Calculate the average of your trials and the average deviation. If the average deviation is greater than 0.003 M, do more titrations and be very careful of your technique.

Part 3: Mass Percent of Acetic Acid in Vinegars

In Part 4, you determine the precise acetic acid content in a specific brand and type of vinegar by titration with your standardized NaOH. As in Part 2, you will do at least 3 titrations, until the results do not differ by more than 1.5 %.

1. Collect a small amount of vinegar (each person will need about 15 mL for titration and each buret will need about 15 mL for conditioning). Condition a small (20 mL) buret with the vinegar you are using. (See the instructions for Part 2 to remind you how to condition the buret.) You can share the vinegar buret with a partner. Make sure to use the same brand of vinegar for the whole thing.
2. Condition a large buret (if available) with your NaOH solution from last week.
3. Weigh a clean, dry Erlenmeyer flask (100 mL is fine) on an analytical balance to a precision of at least ± 0.001 g and record the mass in your notebook.
4. Record the initial reading of the vinegar buret in your data table. Add about 2.5 mL of vinegar to the flask. Record the final reading of the buret. Weigh the flask again and record the mass of the flask with the vinegar. Subtract to determine the mass of vinegar used.
5. Add a few drops of phenolphthalein indicator into the flask with the vinegar. If the vinegar has a dark color, add more purified water to lighten it.

6. Use your standardized NaOH solution to titrate the vinegar. Be sure to record the initial buret reading, as well as the buret reading at the endpoint, to two decimal places. (Review the titration instructions in Part 2 if necessary.)
7. After the first titration, dump the contents of the flask down the sink, rinse the flask well with deionized water, shake it dry, and carefully dry off the outside of the flask with a paper towel.
8. Repeat the procedure from step 3-7 at least twice. (Mass the flask, add vinegar, re-weigh the flask, and titrate. The flask does not have to be absolutely dry, but it should be dry on the outside. You will need to weigh it before each trial, because it will contain a slightly different amount of water each time.)
9. Calculate the concentration of the vinegar and check the results with your instructor. If necessary, repeat the titrations until your results are close enough together.
10. Once your instructor approves your results, clean up. The waste can go down the sink.

Calculations for Part 3

1. Calculate the concentration of the vinegar separately for each trial, as you did for Part 2, using the precise concentration you found for the NaOH.
2. Find the % difference between your least similar trials (of 3 trials total). If it is not less than 1.5%, do more trials until you have 3 within 1.5% of each other.
3. Find the average concentration (in molarity) and the average deviation of the vinegar.
4. Find the density of the vinegar from each trial. Calculate the average density and average deviation.
5. Using the average concentration and the average density, find the mass % of acetic acid in the vinegar.
6. Record the density, molarity and mass % with their error ranges (as described in the lab notebook guidelines).

Name:

Section: Tu Th Fam Fpm

Experiment 11 Pre-Lab Sheet: First Day

1. (2 pts) Find the mass of KHP (formula $\text{KHC}_8\text{H}_4\text{O}_4$) needed to prepare 250 mL of 0.10 M solution. Show your work in detail, with all the units.
2. (1 pt) Why must you start over if you add too much water to the volumetric flask in Part 1?
3. (1 pt) How many moles of KHP are there in 10 mL of 0.10 M solution? Show your work in detail, with all the units.
4. (1 pt) Write the equation for the reaction that occurs during the titration in Part 2.
5. (3 pts) Explain *in detail* how you calculate the molarity of a solution using titration data. Show that you understand the purpose of titration as a lab technique, and the principle on which it works.
6. (2 pts) How and why must you condition your burets?

Experiment 12 - Lewis Structures and Molecular Geometry

Introduction

This exercise is intended to help you understand Lewis structures and the shapes of molecules. If you want to understand the shape of a molecule, the first thing you have to do is draw a Lewis dot structure. Before you move on, check the Lewis structure carefully to make sure it passes:

- does it have the correct number of valence electrons? (Remember to include the overall charge if it's an ion)
- does each atom have an appropriate number of electrons? (H-2; Be-4; B-6; C,N,O,F-8; heavier elements 8+)
- does each atom have an appropriate formal charge? (more electronegative atoms should be more negative, all formal charges should be fairly small, opposite formal charges should be on neighboring atoms, like formal charges should be separated)
- do the formal charges of each atom add up to the total charge on the whole molecule or ion?

If your Lewis structure doesn't pass, correct the number of electrons or move them around until it does pass.

Once you have a good Lewis structure, you can find the shape of the whole molecule by considering the shape of each atom that has multiple atoms bonded to it. You predict how these atoms are arranged (what angles) using the **Electron Domain Model** (very similar to VSEPR). The basic idea is that all the electron pairs repel each other, so they will spread out as much as they can. Count how many **electron domains** (groups of electrons) there are around the atom: each lone pair and each bonded atom is an electron domain. The electron domains will spread out as much as possible, taking the following shapes described in Table 1.

Table 1: names and angles of electron domain geometries

number of electron domains	name of shape	angle between domains
2	linear	180°
3	trigonal planar	120°
4	tetrahedral	109.5°
5	trigonal bipyramidal	120°, 90°
6	octahedral	90°

However, some of the electron domains may be invisible because they are lone pairs, not atoms. So usually we describe the geometry of the molecule, not the geometry of the electron domains. We use the following names for molecular geometries when there are lone pairs. When there are no lone pairs, the molecular geometry is the same as the electron domain geometry.

Table 2: names of molecular geometries

number of electron domains	number of lone pairs	name of shape
3	1	bent
4	1	trigonal pyramidal
4	2	bent
5	1	seesaw
5	2	T-shape
5	3	linear
6	1	square pyramidal
6	2	square planar

Once you know the shape of the molecule, you can decide whether it is **polar**. If all the individual **bond dipoles** cancel out (because they are pointed in opposite directions), then there is no net **dipole moment**. If they don't cancel out, then there is a dipole and the molecule is polar.

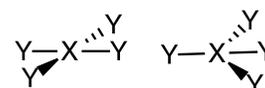
The actual shapes of the molecules may be **distorted**, which means the angles or bond lengths might not be perfectly the same as each other, or the same as in the ideal shape. For example, lone pairs take up more space than single bonds, and multiple bonds take up less space than lone pairs, but more space than single bonds. So the observed **bond angles** (between atoms, not lone pairs) in a structure with lone pairs are probably smaller than the predicted bond angles, because the bonds get squished together by the lone pairs. Also, this is why lone pairs take equatorial positions on trigonal bipyramidal structures: they can push the single bonds out of the way better from here.

How to draw 3D structures in 2D without being an artist:

The traditional way chemists represent 3D structures on paper uses several conventions. First, bonds drawn as regular lines are interpreted as in the plane of the page. For example, in the left BH_3 drawing, the structure must be planar because each bond is a normal line. An alternate way to draw the molecule uses **wedges** and **dashes** to show bonds that come out of the page toward you, and bonds that go back behind the page. In the BH_3 drawing on the right, the same flat molecule is now viewed edge-on, instead of from the top.



When you draw, orient the molecule in your mind to put one or more bonds in the plane of the page, and draw these using regular lines. Then show other bonds coming out or going back. Try to draw the bonds at roughly the correct angles, so if two bonds are directly across from each other, their lines, wedges or dashes should be parallel. Which of the XY_4 drawings shows square planar correctly?



Clean-up:

- After you have showed your models to the instructor, take them apart and return the atoms and bonds to the correct boxes and beakers. If you have trouble taking them apart, use the flat tool to help separate the pieces.

Procedure**Part 1 - Getting comfortable with models and shapes**

Find one atom with exactly 3 holes (black), one with exactly 4 holes, one with exactly 5 holes, and 1 with exactly six holes. Get a bunch of white hydrogen atoms, and a bunch of short, straight single bonds. Also get a few oval "lone pair" pieces.

The purpose of this part is to get a feel for what the shape names mean. The same name always refers to the same shape and the same number of atoms attached to the central atom, no matter what specific elements and molecules are involved.

3 electron domains:

Connect 3 hydrogens to the black atom with 3 holes. The hydrogens should be flat, in the same plane. This is the shape of an atom 3 electron domains. What is it called (see Table 1)? Draw it. Notice that the hydrogen atoms are as far from each other as they can be. Why does this name make sense?

Replace 1 hydrogen with a lone pair. What is the shape now (see Table 2)? Why does this name make sense? Since we can't actually see lone pairs, remove the lone pair piece. This is the shape we would see if there are 2 bonds and 1 lone pair. Draw and name it.

*****Make sure your drawings are clear enough to tell the difference between different geometries with the same number of bonded atoms, such as trigonal planar and trigonal pyramidal. You don't need to show lone pairs in the drawings.*****

4 electron domains:

Connect 4 hydrogens to the atom with 4 holes. This is a tetrahedral shape. Anything that is tetrahedral should have exactly this shape. Notice how the hydrogens are as far from each other as possible. Draw the shape.

Remove 1 hydrogen. What is the shape now? Why does this name make sense? This is the shape we would see if there are 3 bonds and 1 lone pair. Draw and name it.

Remove 1 more hydrogen. What shape is the molecule if there are two bonds and 2 lone pairs? Draw and name it.

5 electron domains:

Attach 5 hydrogens to the atom with 5 holes. What shape is this? Why does this name make sense? Draw it.

Find the 2 hydrogens that are right across from each other. These are in the **axial** positions (like the axle of a wheel). What is the angle between them and their nearest neighbors?

Find the other 3 hydrogens that are in the same plane. These are in the **equatorial** positions (like the equator). How many nearest neighbors does each equatorial hydrogen have, at what angle?

When there are lone pairs in an atom with 5 electron domains, they usually take the equatorial positions. Remove a hydrogen from an equatorial position. What shape is this? Draw it and name the shape. Why does the name make sense?

Remove another equatorial hydrogen. Draw and name this shape. Why does this name make sense?

Remove another equatorial hydrogen. Draw and name this shape.

6 electron domains:

Attach 6 hydrogen atoms to the atom with 6 holes. This is the octahedral shape. Draw it.

Remove 1 hydrogen. What is this shape? Draw it and name it.

Remove 1 more hydrogen (choose the hydrogen that makes sense based on the name in Table 2). Draw and name the shape.

Shapes by number of bonds:

2 bonds: compare a linear model and a bent model. Notice that both have 2 bonds, but the shapes are different. The bent shape has room for 1 or more lone pairs.

3 bonds: compare T-shape, trigonal planar and trigonal pyramidal models. Make sure you know which is which. The trigonal pyramidal shape has room for 1 lone pair, and the T-shape molecule has room for 2 or more lone pairs.

4 bonds: compare square planar, seesaw and tetrahedral models. Make sure you know which is which. The square planar shape has room for 2 lone pairs, the seesaw has room for 1 lone pair, and the tetrahedral model has no room for lone pairs.

5 bonds: compare square pyramidal and trigonal bipyramidal models. Make sure you know which is which. Square pyramidal has room for 1 lone pair, and trigonal bipyramidal has no room for lone pairs.

Part 2 - Lewis structures, shapes, models, etc

Complete Part 2 on the lab sheet. For each central atom, give the number of electron domains, lone pairs on it, and atoms bonded to it. Recall that the number of electron domains of an atom is equal to the number of atoms attached plus the number of lone pairs it has. Predict shape, bond angles, and distortion at that center.

Build models of each molecule or ion, using the correct color atoms if possible. Show the instructor your finished sheet and all the models.

Pre-Lab Exercise

Before coming to lab, complete the Lewis structures for Part 2 on the lab sheet. There's no post-lab this week, so the pre-lab is worth the whole 30 points for pre-lab and post-lab!

Experiment 12 (Pre-During-Post-Lab) Sheet

Name:

Part 1: Make sure your drawings **clearly** show (using wedges and dashes) the differences between different shapes with the same number of bonds!

	draw the shape	name the shape		draw the shape	name the shape
3 electron domains			4 electron domains		
0 lone pairs			0 lone pairs		
1 lone pair			1 lone pair		
			2 lone pairs		
5 electron domains			6 electron domains		
0 lone pairs			0 lone pairs		
1 lone pair			1 lone pair		
2 lone pairs			2 lone pairs		
3 lone pairs					

Part 2:

Formula	Lewis structure	on central atom: electron domains	electron domain geometry name	-bond angles (e.g. 90°)
number of valence e^-		bonded atoms	molecular geometry name	polar? (y/n)
		lone pairs		distortion? (y/n)
CO ₂				
H ₂ O				
BH ₃				
CH ₄				
NH ₃				
POF ₃				
SF ₄				
PCl ₅				

Formula	Lewis structure	on central atom: electron domains	electron domain geometry name	-bond angles (e.g. 90°)
number of valence e^-		bonded atoms	molecular geometry name	polar? (y/n)
		lone pairs		distortion? (y/n)
SF ₆				
ICl ₂ ⁻				
ClF ₃				
XeF ₄				
IF ₅				
SO ₄ ²⁻				
N ₂ H ₄				
HCN				

Experiment 13 - Lewis Structures and Molecular Geometry

Introduction

This exercise builds on the previous one to help you get comfortable with shapes and structures of molecules. This time, we will focus on molecules that obey the octet rule and look at more complicated molecules. In molecules that have more than one central atom, rather than learning names for an infinite variety of larger shapes, we will name the shape at each central atom using electron domain model. Then, if we know the possible rotations around the bonds, we can understand the shape (and flexibility) of the whole molecule.

According to the Valence Bond Theory, single bonds are made by an electron pair occupying the overlapping space in two orbitals that point straight toward each other. This type of bond is called a σ -bond (Greek letter sigma). To get orbitals that point in the directions required by the electron domain model (and the actual shapes of the real molecules), we can combine the s and p orbitals on atoms such as C, N and O into new combinations called **hybrids**. The number of orbitals combined is the same as the number of hybrids formed, and also matches the number of electron domains. For example, when you mix one s orbital with one p orbital, you get two sp hybrids which are pointed 180° from each other, perfect for a linear geometry. Since there are three p orbitals to start with and only one was used to make hybrids, there are two p orbitals leftover, which can be used to make multiple bonds.

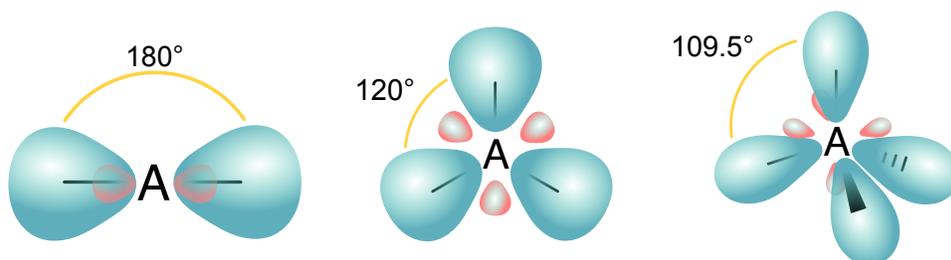
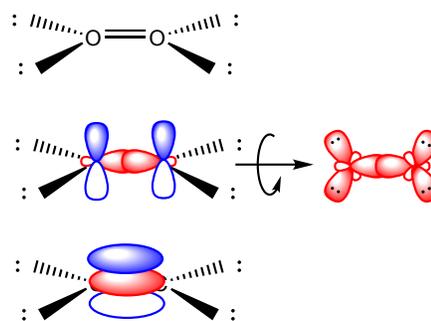


Table 1: Important hybrid orbitals

number of electron domains	name of shape	angle between domains	hybrid orbitals
2	linear	180°	sp
3	trigonal planar	120°	sp^2
4	tetrahedral	109.5°	sp^3

The second and third bond between two atoms can't be a σ -bond, because there's only room for one bond that goes directly between the atoms. Instead, these bonds are called π -bonds (Greek letter pi) and they are formed by two perpendicular p orbitals. Because σ -bonds are symmetric around the bond, the molecule can rotate around σ -bonds to change shape. However, π -bonds break when rotated, because then there is no longer good overlap between the p orbitals they are made of. The diagram show the σ -bond and lone pairs in O_2 in red and the π -bond in blue. When you build models with multiple bonds, there are two options. Normally, you will use the regular atoms with the correct number of electron domains and use two or three of the

flexible multiple bond connectors to make the bonds. This method will give the right shape, and show that you can't rotate around a multiple bond. If you want to see the π -bonds more clearly, you can use the 5-domain dark blue atoms and put on teardrop-shaped orbitals to show the p orbitals used to make π -bonds.



Resonance structures are used when one Lewis structure doesn't adequately describe the molecule. Often this occurs because π -bonds can be spread out over more than two atoms. For example, in carbonate ion, the Lewis structures suggest carbon makes single bonds to two oxygen atoms and a double bond to the remaining oxygen atom. In reality, the π -bond is spread out over all three oxygen atoms. By drawing three Lewis structures, with the double bond in a different place each time, we can (clumsily and laboriously) show the true situation. Note that when drawing resonance structures, the lone pairs and bonds move, but the atoms do not.

In many molecules, it's possible to make several different arrangements of atoms that have the same formula. These arrangements are called **isomers**. Some isomers have obviously different arrangements, like straight chains and branched chains are carbon. Others are more subtle. If a double bond is present, it may be possible to make two different isomers by changing the arrangement of atoms or groups around the non-rotating double bond. Arrangements that look different but can be made the same by rotating around single bonds are not different isomers. Another type of isomers are mirror images that aren't the same, called stereoisomers. These are very important in biology.

Clean-up:

- After you have showed your models to the instructor, take them apart and return the atoms and bonds to the correct boxes and beakers. If you have trouble taking them apart, use the flat tool to help separate the pieces. Make sure the number of holes on the atom matches the label on the box.

Procedure

Work with a partner. As you finish each part, show the instructor your models and drawings to get checked off. Use the correct colors for each atom type: black for carbon, light blue for nitrogen, red for oxygen, and white for hydrogen.

Part 1 - Multiple Bonds and Resonance

Build models of C_2H_6 , C_2H_4 , and C_2H_2 . Can you rotate around the bonds? Clearly and carefully draw the 3D structures based on your models.

Draw a Lewis structure and predict the shape of H_2CCCH_2 . Will it be all in the same plane? Then build a model and clearly, carefully draw the 3D structure. Was your prediction correct? Carefully draw the π -bonds in the molecule.

Draw a Lewis structure and build a model of benzene, C_6H_6 , using the flexible bonds. What does it get right and what does it get wrong about the shape of the real molecule? Look at the instructor's models of carbonate and benzene. How are they different from the Lewis structures?

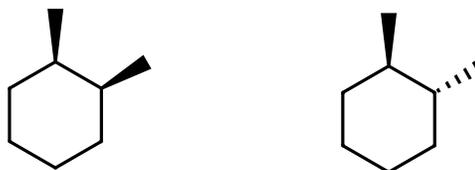
Refer to your pre-lab for resonance structures for $\text{CH}_3\text{CONHCH}_3$. Build models of both resonance structures. What do these structures predict about the shape of the molecule? What is different about what they predict? How flexible will it be?

Part 2 - Isomers

Draw and build three or four isomers of C_5H_{10} . Use “organic-style” line drawings, not Lewis structures. Make sure your isomers are really different molecules!

Build a model of CHFCIBr . Then build the mirror image of the model you built. Are the two models identical?

Build models of the two molecules at right. What is different between them?

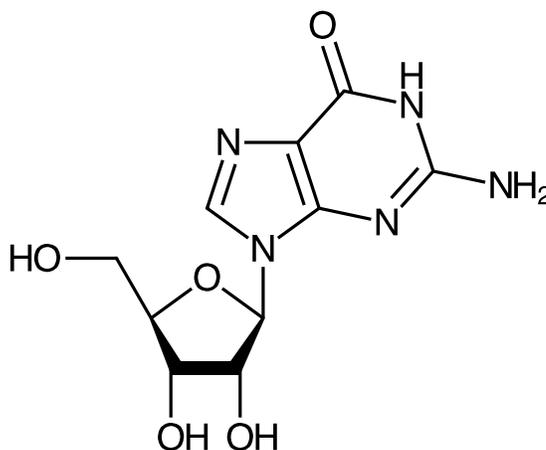
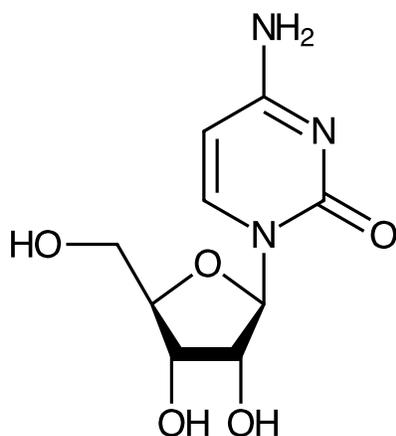


Part 3 - Larger Molecules

Draw a Lewis structure for CH_3COOH and predict the shape at each central atom. What hybrids does each atom use? Build a model and confirm your predictions.

Convert the “organic-style” diagrams below to Lewis structures by making each end or bend of the lines a carbon with enough hydrogen to make octet, and by adding appropriate lone pairs to other atoms. Then build models: be careful, make sure you use the right atom types with the right numbers of holes, so the shape is what it should be! Wiggle them around and see how flexible they are. Does the organic-style drawing show the real shapes of the molecules better or worse than regular Lewis structures?

The two large structures are units of DNA. Match up the hydrogen bonds to see how DNA works.



Experiment 13 Pre-Lab Sheet

Name:

This experiment has a 20 pt pre-lab and a 10 pt post-lab.

- (1 pt) Fill in the following:
 - In Lewis structures, C usually makes ___ bonds
 - In Lewis structures, H usually makes ___ bonds
 - In Lewis structures, N usually makes ___ bonds
 - In Lewis structures, O usually makes ___ bonds
 - In Lewis structures, Cl usually makes ___ bonds
- (3 pts) What is the difference between isomers and resonance structures?
- (6 pts) For each molecule, draw a Lewis structure, say what type of hybrid orbitals are used, and make two separate drawings, one showing the σ -bonds and the other showing the π -bonds (if applicable).



4. (4 pts) Draw two resonance structures for $\text{CH}_3\text{CONHCH}_3$. Arrange the atoms in groups as the formula suggests: $(\text{CH}_3)(\text{CO})(\text{NH})(\text{CH}_3)$. One resonance structure should have all-zero formal charges.

5. (4 pts) Draw a Lewis structure for CH_3COOH and predict the shape at each central atom. What hybrids does each atom use?

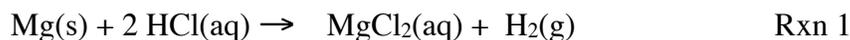
6. (2 pts) Draw a few isomers of C_5H_{10} . Use organic-style line drawings.

Experiment 14 - Calculation of the Ideal Gas Constant

According to both theory and experiment, the pressure (P) of any sample of an ideal gas is inversely proportional to its volume (V) and directly proportional to its absolute temperature (T) and to the number of moles (n) of the gas present in the sample. It is more convenient to put this relationship in the form of an equation, adding a constant to ensure that units work out correctly. This constant, R , is called the **ideal gas constant**. The resulting equation is the familiar **ideal gas law**:

$$PV = nRT \quad \text{Eq 1}$$

The object of the present experiment is to measure R using a sample of hydrogen gas, H_2 . To do this, we must simultaneously measure P , V , T , and n . The value of R can then be calculated and compared with the accepted value of R . In this experiment, P , V , and T will be measured directly, while n will be calculated from the amount of magnesium used to produce the hydrogen gas according to the equation:



To calculate the partial pressure of hydrogen in the tube, there are two corrections to take into account. First, the gas is collected over water, so the vapor pressure of water must be subtracted from the total pressure. The second correction is due to the column of solution remaining in the tube at the end of the reaction. Because the level of solution inside the tube is higher than the water level in the beaker at the end of the reaction, the pressure of gas inside the tube is lower than the atmospheric pressure. You will need to convert the height of the acid column to the corresponding height of mercury. Since **mm Hg** is the same as **torr**, a common unit of pressure, it may then be subtracted from the total pressure.

To convert the height of the column of solution to the corresponding height of mercury, the differing densities of mercury ($d = 13.6 \text{ g/mL}$) and the acid solution ($d = 1.05 \text{ g/mL}$) must be taken into account. The pressure due to a column of liquid is related to its height and density by

$$P = gdh \quad \text{Eq 2}$$

where P is pressure due to a column of liquid, g is the constant acceleration of gravity, d is the density of the liquid, and h is the height of the column of liquid. In a more useful form, you can relate the height of any liquid to the height of mercury that corresponds to the same pressure

$$d_{\text{liq}}h_{\text{liq}} = d_{\text{Hg}}h_{\text{Hg}} \quad \text{Eq 3}$$

The height of a mercury column in millimeters is equal to the pressure in torr, so you can use Eq 3 to find a pressure correction in torr for the height of the water. The atmospheric pressure on the water surface is balanced by the partial pressure of hydrogen gas P_{H_2} , the partial pressure of the water vapor P_{WV} , and the pressure of the water column ($P_{WC} = h_{Hg}$).

$$P_{\text{atmosphere}} = P_{H_2} + P_{WV} + P_{WC} \quad \text{Eq 4}$$

Safety Precautions:

- Wear your safety goggles.

- Use caution when handling 6 M HCl. It is corrosive and can burn your skin. If any HCl comes into contact with your skin, rinse it off immediately and thoroughly with lots of water.

Waste Disposal:

- At the end of the experiment, the HCl solution will be much more dilute. The water/HCl/MgCl₂ mixture may be rinsed down the sink with plenty of water.

Procedure

1. Obtain a piece of magnesium ribbon approximately 2 cm long. Clean the ribbon with fine steel wool and weigh it to the nearest 0.0001 g (use the balances in the balance room). Make sure that it weighs less than 0.04 grams, and that your recorded mass has three significant figures. (If it weighs more, cut off a small piece and re-weigh.)
2. Roll the ribbon loosely and then wrap it in a little ball of fine copper wire (see the display in the laboratory), leaving a "handle" of copper wire. The wrapping is designed to prevent small pieces of magnesium from breaking off and escaping during the experiment. Make sure that the ball is not too large to fit into the gas measuring tube.
3. Set up a ring stand with a buret clamp in position to hold a 50-mL gas-measuring tube (eudiometer). Fill a 400-mL beaker about 2/3 full of tap water and place it near the ring stand.
4. Tilt the gas-measuring tube slightly and pour in about 10 mL of 6 M HCl. (Estimate volume using the marks on the tube, and don't worry about getting exactly 10 mL.) Then, with the tube still in the same tilted position, gently add some water from a wash bottle, being careful not to mix the water too much with the acid. Then, gently fill the tube to the top with water (pour from a beaker or wash bottle). While pouring, rinse down any acid that may have wet the sides of the tube. The object is to have acid at the bottom of the tube and water at the top. Try to avoid stirring up the acid layer at the bottom of the tube. Air bubbles that may cling to the insides of the tube can be dislodged by gentle tapping of the tube.
5. Add water to the tube until it overflows slightly. Holding the copper coil by the handle, insert the cage about 3 cm down into the tube. Hook the wire over the edge of the tube where it will be pinched by the rubber stopper and held in place. When you insert the stopper, don't put your finger over the hole in the stopper. Let the water overflow as you insert the stopper so that there are absolutely no air bubbles trapped in the tube.
6. Add some more water to the hole in the stopper so that it is completely filled with water. Cover the hole in the stopper with your finger and invert the tube in the beaker of water, so that the stoppered end is under water. Once the hole is under water, you can remove your finger; the water cannot now run out. Clamp the tube in place.
7. Check if there is any bubble at the top of the tube (ideally, there won't be). Record if there is a bubble at the top of the tube, and how big it is.
8. The acid, being more dense than the water, will diffuse down through the water and soon reach the metal sample. When the reaction begins, you will see bubbles of hydrogen gas form. Check to see if there is a temperature change associated with this reaction and record your observations.
9. After the bubbles stop forming, you know that the reaction is completed, but you should wait for a few minutes for the tube to come to room temperature.

10. Tap the tube (gently) to dislodge the bubbles stuck to the sides. Try to get them all to join the main gas sample as the top.
11. Read and record the volume of the hydrogen gas in the eudiometer to the nearest 0.1 mL.
12. *Without changing the position of the eudiometer*, hold a ruler at the top surface of the water level inside the beaker, and measure the distance from the water level in the beaker to the water level inside the eudiometer. Record this height (in centimeters). This will be the "height of the liquid column" referred to in the calculations.
13. Measure and record the temperature of the gas.
14. Record the atmospheric pressure (the instructor will read the barometer and write today's atmospheric pressure on the board).
15. Repeat the entire procedure with a second sample of magnesium (you can probably use a second tube and other apparatus, and clean both up at once).
16. Perform your calculations before cleaning up your gas sample. This ensures that you can repeat any measurements if necessary.
17. Disassemble the apparatus (remove the stopper carefully from the tube, and collect all the liquid in the beaker). Slowly add some sodium carbonate (Na_2CO_3) or sodium bicarbonate (NaHCO_3) to the HCl solution in the beaker until there is no further fizzing (this step neutralizes the remaining acid). Dump the neutralized solution down the drain. If there is no sodium carbonate or bicarbonate available, dump the solution down the sink followed by lots of water. Rinse the eudiometer with water.
18. Write your name and your two results for R on the board in the classroom.

Calculations

1. From the mass of magnesium used and the balanced equation, calculate the number of moles of hydrogen gas expected for each trial.
2. Use Eq 3 to find the pressure correction for the height of the water column, in mm Hg (torr). Make sure to convert the height of the liquid column to units of millimeters before using the equation. Do this for each trial.
3. For each trial, find the partial pressure of hydrogen using Eq 4. All the units should be mm Hg (torr).
4. Using the Ideal Gas Law (Eq 1), calculate the experimental value for R for each trial.
5. Find your average value of R . Calculate the percent error using the accepted value of R ($62.36 \text{ L torr K}^{-1} \text{ mol}^{-1}$) and your average experimental value of R .
6. Write your results for R on the board with your name. The instructor will post the class results so you can use them for your post-lab.

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Experiment 14 Pre-Lab Sheet

1. (1 pt) What is the purpose of this lab?
2. (1 pt) Why don't we need to measure the acid volume precisely?
3. (2 pts) How many moles of hydrogen gas are produced when 0.2145 g of Mg are reacted completely with HCl?
4. (1 pt) What is the purpose of wrapping the Mg ribbon in copper wire?
5. (1 pt) Why should you measure the mass of the Mg ribbon to 0.0001 g, not 0.001 g?
6. (4 pts) A sample of nitrogen gas is collected over water, like in this lab. The atmospheric pressure is 745 torr, the vapor pressure of water is 17.5 torr, and the height of the column of water is 22.4 cm. Find the partial pressure of the nitrogen gas.

Experiment 15 - Heat of Fusion and Heat of Solution

Phase changes and dissolution are physical processes that absorb or release heat. In this experiment, you will determine the heat of fusion of ice (the energy required to melt ice) and the heat of solution of two different ionic compounds.

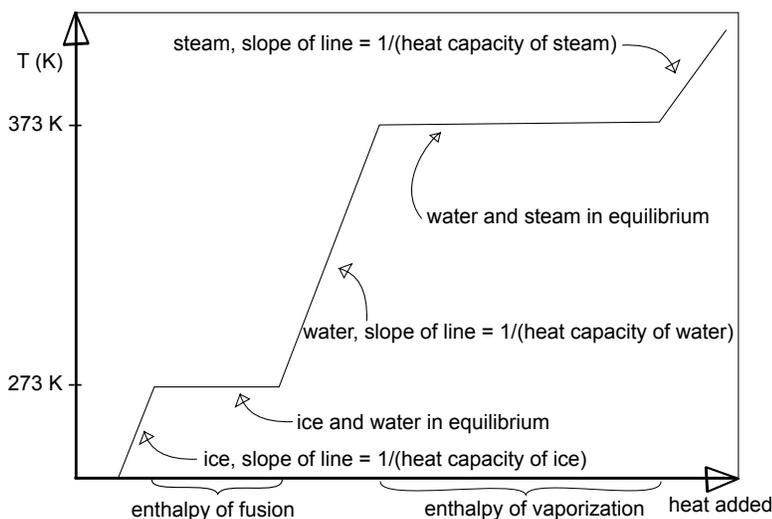
Part 1: The Heat of Fusion of Ice

The melting (fusion) of any solid substance is an endothermic process. Heat must be absorbed by a substance in order to be converted from the solid phase to the liquid phase. In this experiment, the amount of heat required to melt one gram of solid H₂O (ice) will be determined. This quantity is called the **heat of fusion** of ice.

Normally, when a substance is heated, the temperature rises. This is because the heat energy is used to increase the **kinetic energy** of the molecules of the substance. Increasing kinetic energy means that the molecules of the substance are moving faster, and increased average molecular velocity is observed and measured as increased temperature.

When a substance is at its melting point or boiling point, however, its temperature will not change as it is heated. Instead, it will undergo a **phase change** - it will either melt or boil - and phase changes are processes that occur at constant temperature. The heat energy is used in these cases to increase the **potential energy** of the molecules. In melting, energy is required to break apart the crystalline lattice of the solid state. In boiling, the molecules must be separated from each other in order to vaporize. Heat energy is used to overcome intermolecular forces holding the molecules together, increasing the potential energy (a process analogous to increasing the gravitational potential energy of a weight by lifting it).

The amount of heat energy that is required to melt a sample of ice can be measured by



allowing the ice to melt in a known amount of liquid water. When the ice is placed in water, an exchange of thermal energy takes place. The ice absorbs energy from the water and melts (potential energy increases), while the water gets colder (the kinetic energy of the molecules decreases) as it loses thermal energy to the ice. Once the ice has melted, it then warms up (the newly melted H₂O molecules now gain kinetic energy) until its temperature becomes the same as that of the originally liquid water.

If the process of ice melting in water occurs in an insulated container, no heat energy will enter the system from the outside. All of the energy gained by the ice in melting and warming will come from the originally liquid water. In other words, the heat absorbed by the ice, q_{abs} , must be equal, but opposite in sign, to the heat released by the liquid water, q_{rel} . This can be expressed by the equation:

$$q_{abs} + q_{rel} = 0 \quad \text{Eq 1}$$

The amount of heat energy lost by the originally liquid water can be calculated by using the **specific heat**, temperature change and mass of the water. The specific heat, or heat capacity/gram, has units of J/g°C. The equation to relate specific heat to heat is (nicknamed mCAT), is

$$q = mC_s\Delta T \quad \text{Eq 2}$$

where q is the heat required to cause temperature change ΔT (where $\Delta T = T_{final} - T_{initial}$) in m grams of a substance with specific heat C_s . The definition of the calorie states that 1.00 calorie is required to heat one gram of water by 1 °C. Since we will be working in joules, the specific heat of water is 4.18 J/g°C, which implies that 1 cal = 4.18 J.

Our goal will be to determine the amount of energy per gram required to melt ice. In order to do this, we must realize that q_{abs} has two terms: the energy absorbed by the ice as it is melting at 0 °C (we'll call this q_{melt}), and the energy absorbed by the resulting water as it warms up from 0 °C to the final temperature (we'll call this q_{warm}).

$$q_{abs} = q_{melt} + q_{warm} \quad \text{Eq 3}$$

While q_{melt} is related to the heat of fusion, q_{warm} can be calculated from the Eq 2, where m will be equal to the mass of ice, since this is the water that was originally ice. The initial temperature of this water sample is 0 °C (exactly), and the final temperature is the measured final temperature for the entire water sample at the end of the experiment.

Therefore, you will be able to calculate the value for q_{rel} and q_{warm} . To find the heat of fusion of ice, you need to know q_{melt} .

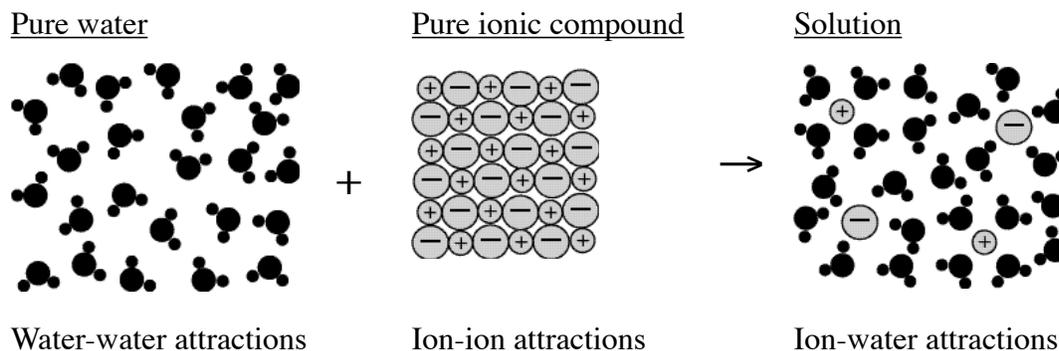
$$-q_{rel} = q_{abs} = q_{melt} + q_{warm} \quad \text{Eq 4}$$

Use the above relationships to solve for q_{melt} . This is the amount of energy used to melt the amount of ice you used. To find the heat of fusion, divide the q_{melt} by the number of grams of ice used, and you will have the heat of fusion in units of J/g.

Part 2: Heat of Solution

When a solute dissolves in a solvent, energy can be absorbed or released. In this experiment, you will be dissolving two different ionic solids in water and determining the heat of solution of each substance. (The heat of solution is the energy involved in dissolving a specific amount of the substance in a particular solvent).

When water and an ionic compound are mixed to form a solution, the heat of solution ($\Delta H_{solution}$) depends not only on the attractions between water and the ions in the solution, but also on the water-water attractions in pure water, and on the ion-ion attractions when the pure crystal.



For some substances, the heat of solution is endothermic, and for others the solution process is exothermic. The overall energy change depends on two main factors. Energy must be added to break the solid apart into separate ions and to separate the solvent molecules to make room for the ions, and energy is released when ions are hydrated by water molecules. When these two amounts of energy are added together, the result can be either positive or negative (endothermic or exothermic) overall.

If the attractions in the solution are *stronger* than the attractions in the pure substances, the dissolving process will be **exothermic**. By dissolving, the water and the ions will reach a more stable state (lower potential energy). The difference in potential energy will appear as heat (kinetic energy). The solution will be hotter than the pure substances were before mixing. If the attractions in the solution are *weaker* than the attractions in the separate solute and solvent, then the dissolving process will be **endothermic**, and will absorb heat from the solution, making the solution colder than it started. However, even if the dissolution is endothermic, the substance may be soluble because there is a natural tendency toward mixing.

The names and symbols here can get confusing. Since we don't yet know what's releasing and absorbing energy, let's call the heat released or absorbed by the process of dissolution q_{dis} , and the heat released to or absorbed from the solution as a result q_{sol} . Then, just like Eq 1,

$$q_{dis} + q_{sol} = 0 \qquad \text{Eq 5}$$

You can find q_{sol} using Eq 2, and q_{dis} is the heat of solution multiplied by the number of moles.

Safety Precautions:

- Wear your safety goggles.

Waste Disposal:

- The waste from this experiment may be safely disposed of in the sink with plenty of running water.
- Rinse and save the cups unless they are broken.

Procedure – Part 1

The procedure here is similar to that used in Experiment 8. We will add some ice to a known mass of water at a known temperature, and record the final minimum temperature after all the ice has melted. The exact weight of the ice will need to be known, but it is inconvenient to weigh the ice *before* adding it to the water, since it will begin to melt as it is being weighed. A large source of error in this experiment would be to add liquid water (on the surface of the ice) and count this liquid water as ice, since melted ice has already absorbed its heat of fusion.

To minimize the melting of the ice before it reaches the water in the cup, the ice will simply be added without weighing it. The weight of the ice added can be found later, at the end of the experiment, by weighing the cup to see how much weight has been added.

1. Set up an appropriate table in your notebook to hold the data.
2. Weigh the empty Styrofoam cups (use two stacked cups) that will be used to hold the ice and water.
3. Add about 100 - 150 mL of water to the Styrofoam cup. The water should be at or slightly above room temperature before it is added. Weigh the cup again to determine the weight of the water.

4. Set up a ring stand with an iron ring. Place the cups in a beaker (for extra stability) and place the beaker below the ring. Between temperature measurements, the thermometer can rest against the inside of the iron ring so that it won't fall over and break.
5. Measure the temperature of the water in the cup ($\pm 0.1\text{ }^{\circ}\text{C}$). Be sure that the temperature has reached a constant value and is not changing. Record this temperature.
6. Take about 10 - 15 grams of ice (estimated by eye as about 10 - 15 mL of ice, which is about 3 connected pieces or 5 separate pieces from our machine), dry it quickly with a paper towel, and add it to the water in the cup. Be sure that the ice is as dry as possible before adding it.
7. Allow the ice to melt, **stirring constantly** with the thermometer. Record the lowest temperature reached as the final temperature ($\pm 0.1\text{ }^{\circ}\text{C}$). This should be reached just as the ice has completely melted.
8. Weigh the cup and its contents to obtain the mass of melted ice by subtraction.
9. Repeat the entire procedure.
10. Calculate the heat of fusion of ice in J/g for each trial using the equations in the introduction. Average the two ΔH_{fus} values.
11. Calculate the percent error using the theoretical value of ΔH_{fus} for ice (333 J/g) and your average value of ΔH_{fus} as the experimental value.
12. Write down your results for ΔH_{fus} on the board, along with your name. The instructor will distribute the results for the class so you can use them in your post-lab.

Procedure – Part 2

1. Dry and reweigh your cups if you have doubt of their mass.
2. Transfer about 100 mL of deionized water into the cup, and weigh again.
3. Weigh out about 5 grams of solid anhydrous ammonium chloride (NH_4Cl) on a piece of weighing paper or in a weighing boat. (Put the weighing paper or boat on the top-loading centigram balance, press the tare button to zero the balance, and then transfer some of the solid to the paper or boat.) Record the precise mass of solid used.
4. Carefully measure and record the temperature of the water in the cup to $\pm 0.1^{\circ}\text{C}$.
5. Add the ammonium chloride to the cup, and vigorously stir the mixture with the thermometer until all of the solid has dissolved. Watch the temperature reading, and record the reading that differs the most from the initial temperature.
6. Dump the solution down the sink, and rinse and dry the calorimeter.
7. Repeat steps 1-6, using about 10 grams of anhydrous sodium carbonate (Na_2CO_3) instead of 5 g of NH_4Cl . The sodium carbonate will not dissolve as easily as the ammonium chloride, so be sure to stir the solution vigorously. The sodium carbonate must dissolve in less than 40 seconds, or your results will be inaccurate.

Calculations – Part 2

Note: Do these calculations separately for each trial.

1. Determine the mass of water and mass of solution in the cup.
2. From the mass of solution, the temperature change, and the heat capacity of the solution (assume the heat capacity of the solution is very close to the heat capacity of water), determine the amount of energy absorbed or released by the water/solution in the cup using Eq 2.

3. Determine the amount of energy absorbed or released by the dissolving solid using Eq 1 (which applies in general, not just in the specific instance described in the introduction).
4. Determine the number of moles of solid used.
5. Calculate the “heat of solution” of this compound (ΔH_{soln}) in units of kJ/mol of solute. Make sure to include the appropriate sign.

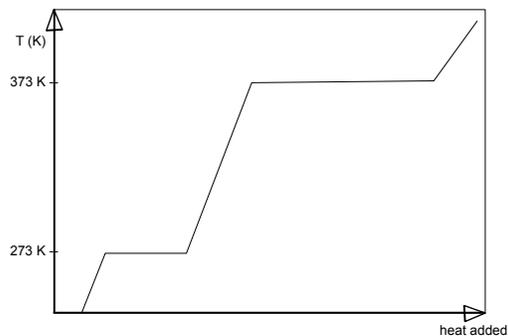
Name:

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Experiment 14 Pre-Lab Sheet

1. (1 pt) To find the heat of fusion of ice in J/g, you (divide/multiply) _____ by the _____.
2. (1 pt) To find the heat of solution in kJ/mol, you (divide / multiply) _____ by the _____.
3. (1 pts) Why should you dry the ice before adding it to the calorimeter?
4. (1 pts) Why is it important to stir constantly during your trials?

5. (3 pts) On the simple heating diagram, label the following: A: where the “ice” starts if it is wet, B: where the ice starts if it is below 0°C, C: where the ice starts if it is dry at 0°C, D: where the water starts (approximately), E: where everything finishes, approximately.



6. (3 pts) Sample calculation: You add 5 g of ice to 100 g of water. The initial temperature of the water is 23 °C. The minimum temperature of the water is 19 °C. Show that according to this data, the enthalpy of fusion of water is 255 J/g.

