
Experiment #1: Measurements involving Mass, Volume, and Statistical Analysis of Data

The error of a measurement is defined as the difference between the experimental and the true value. This is often expressed as percent (%) error, which is calculated as:

$$\text{Percent Error} = \frac{|\text{Experimental} - \text{True}|}{\text{True}} \times 100 \% \quad (1)$$

Note: At times a true value may not be known or possible. If two experimental values are determined and the true value is unknown, then the percent difference may be calculated. The percent difference is the absolute value of the difference between the two experimental values divided by the average value and multiplied by 100.

$$\text{Percent Difference} = \frac{|\text{Value 1} - \text{Value 2}|}{(\text{Value 1} + \text{Value 2}) / 2} \times 100 \% \quad (2)$$

In chemical measurements, we try to eliminate errors, which can be divided into two broad types: systematic and random. *Systematic error* occurs regularly and predictably because of faulty methods, defective instrumentation, and/or incorrect assumptions. *Random error* is more difficult to define and is governed by chance. Examples include a weighing error due to air currents near a balance and line current fluctuations for electronic instrumentation. Systematic errors always affect the measured quantity in the same direction, while random errors can make the measured quantity either too large or too small.

Accuracy is the closeness of agreement between a measured value and the true (or accepted) value. True values can never be obtained by measurement. However, we accept values obtained by skilled workers using the best instrumentation as true values for purposes of calculation or for judging our own results.

Precision describes the reproducibility of our results. A series of measurements with values that are very close to one another is a sign of good precision. It is important to understand, though, that good precision does NOT guarantee accuracy!

The *standard deviation* of a series of measurements including at least 6 independent trials may be defined as follows: let x_m represent a measured value, n be the number of measurements, and \bar{x} be the average or mean of the various independent trials or measurements. Then d is the average deviation:

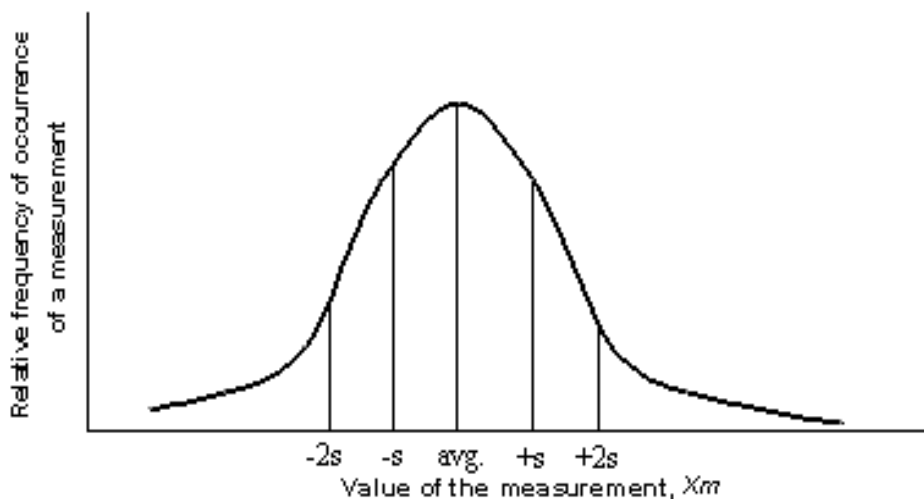
$$d = x_m - \bar{x} \quad (2)$$

and the standard deviation, s , is defined by:

$$s = \sqrt{\frac{\sum d^2}{n-1}} \quad (3)$$

where $\sum d^2$ refers to the sum of all the values of d^2 .

The value of any measurement should include some indication of the precision of the measurement. The standard deviation is used for this purpose if a large number of measurements of the same quantity is subject to random errors only. We can understand the meaning of s if we plot the number of times a given value of x_m is obtained (on the y-axis) versus the values x_m (on the x-axis). Such a *normal distribution curve* is bell-shaped with the most frequent value being the average value \bar{x} .



Most of the measurements result in values near \bar{x} . In fact, 68% of the measurements fall within the standard deviation s of \bar{x} as shown in the graph above. 95% of the measured values are found within $2s$ of \bar{x} . We call the value of $2s$ the uncertainty of the measurement. Then, if we report our value of the measurement as $\bar{x} \pm 2s$, we are saying that \bar{x} is the most probable value, and 95% of the measured values fall within this range. Consider the following example as one illustration of this statistical analysis.

Example One: A test tube is weighed on ten different balances, and the data below is obtained along with statistical analyses:

Balance Number	Mass (g) = x_m	$d = x_m - \bar{x}$	d^2
1	24.29	0.00	0.0000
2	24.26	-0.03	0.0009
3	24.17	-0.12	0.0144
4	24.31	0.02	0.0004
5	24.28	-0.01	0.0001
6	24.19	-0.10	0.0100
7	24.33	0.04	0.0016
8	24.50	0.21	0.0441
9	24.30	0.01	0.0001
10	24.23	-0.06	0.0036

From the data above, it is calculated that $\bar{x} = 242.86/10 = 24.29$ g, and the standard deviation, $s = \sqrt{\frac{0.0752}{9}} = 0.0917$. The range is found to be $\bar{x} \pm 2s = (24.29 \pm 0.18)$ g.

Therefore, the test tube weighs between 24.11 g and 24.47 g with 95% certainty. Now the values of x_m are checked against the range. We observe that the weight from balance #8 is OUTSIDE the range. This value is discarded as it is unreliable, and the values of \bar{x} , d , d^2 , and s are thus recalculated (see Problem #1 in the Pre-lab).

For most of the experiments in this course, the standard deviation is impossible to calculate because we perform too few measurements of a particular quantity. When there are such few measured values (< 6), the *Q Test* is used to decide whether to reject suspected "bad" values as outliers.

$$Q = \frac{|\text{suspect} - \text{nearest}|}{\text{largest} - \text{smallest}} \quad (4)$$

n (# of measurements)	3	4	5	6 or more
<i>Q Test</i> value (90% probability)	0.941	0.765	0.642	Do Not Use

If Q for a set of data is larger than those listed in the table above, the suspect value may be rejected, and the average of the other values is reported. Please note that the sign of Q is NOT important since we are using the absolute value. Consider the following example as one illustration of the *Q Test*.

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Example Two: A student determines the concentration of a hydrochloric acid solution by titration with standardized sodium hydroxide. S/he obtains the values: 0.555 M, 0.565 M, and 0.564 M. Should the value 0.555 M be rejected? Apply the *Q Test*:

$$Q = \frac{|\text{suspect} - \text{nearest}|}{\text{largest} - \text{smallest}} = \frac{|0.555 - 0.564|}{0.565 - 0.555} = 0.90$$

Since 0.90 is LESS THAN the tabulated value of 0.94 for three measurements, we may NOT reject the suspect value, 0.555 M. If the agreement had been greater than the tabulated value of 0.94, the chemist would repeat the measurement until s/he is satisfied (see Problem #2 in the Pre-lab).

Procedure**Part 1**

Use calipers to measure the inside diameter of a medium test tube. Use a ruler to measure the height of the test tube from the lip to where the bottom curves. Calculate the volume of this part of the test tube as a cylinder ($V_{\text{cylinder}} = \pi r^2 h$, where r is the radius, and h is the height in cm). Assume that the bottom of the test tube is HALF of a sphere with the same radius. Calculate its volume ($V_{\text{sphere}} = 4\pi r^3 / 3$). Divide the result by 2; add this number to the volume of the cylindrical portion. This is the total volume of the test tube.

Now fill the test tube with water. Determine the actual capacity of the test tube by pouring the water (from the test tube) into a 50 mL graduated cylinder and calculate the percent difference.

Part 2

Obtain an unknown metal cylinder from your instructor. Determine its density by weighing it on the analytical balance, measuring its dimensions with calipers, and calculating its volume. You will now confirm your calculated volume. Fill a 50 mL graduated cylinder with 25 mL of water, carefully slide your metal into your graduated cylinder, and note the volume of the displaced water. How does this value compare to your calculated volume? Now refer to the *CRC Handbook of Chemistry and Physics* in order to check your density by looking up the handbook value for the material of which your solid is made. Determine the percent error in your density.

Part 3

Obtain a buret and unknown sodium chloride (NaCl) solution from your instructor. Fill the buret with the salt solution. Allow some to drain out so as to fill the nozzle with the solution.

Weigh a stoppered Erlenmeyer flask on the analytical balance.

Record the initial buret reading containing your salt solution, and then add approximately 6 mL of solution to the flask. Carefully record the final buret reading, and weigh your stoppered flask containing the solution. Repeat this procedure FIVE more times; DO NOT EMPTY THE FLASK OR REFILL THE BURET BETWEEN WEIGHINGS! Each time, make the samples different sizes, allowing them to vary between 5 – 8 mL. Be certain to record the weight and volume of each sample as accurately as possible. At the end of the experiment, you should have SIX SAMPLES in the stoppered flask, but you have weighed each one separately and measured the volume of each one separately as well.

Calculate the six values of the density of the solution, making sure to carry out the calculation to 3 digits past the decimal. Look up the density of sodium chloride solutions in the *CRC Handbook of Chemistry and Physics*. Determine the percent sodium chloride by weight of your unknown by comparing your density with the densities in the *CRC*.

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Data and Calculations

Part 1

Diameter: _____ cm Radius: _____ cm Height (cylinder part): _____ cm

Volume (cylinder part): _____ cm³ Volume (half-sphere part): _____ cm³
SHOW CALCULATION: SHOW CALCULATION:

Total Volume (sum): _____ cm³ Volume (graduated cylinder): _____ mL

Average Volume: _____ mL Percent Difference: _____ %
SHOW CALCULATION: SHOW CALCULATION:

Part 2

Mass of Metal Cylinder _____

Diameter _____ Length _____ Volume_{calipers} _____

Volume_{water} _____ Volume_{metal + water} _____ Volume_{water displacement} _____

Density of the Cylinder: calipers: _____ water displacement: _____

Handbook Density _____

Identity of Metal _____

% Error: calipers: _____ water displacement: _____

SHOW CALCULATIONS:

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Part 3

Unknown Number _____

Mass of Flask with stopper _____

Initial Buret reading _____

Sample	Mass Flask+Stopper+Liquid (g)	Mass Liquid Only (g)	Final Buret Reading (mL)	Net Volume (mL)	Density (x_m) (g / mL) 4 sig. figs.	d ($x_m - \bar{x}$)	d ²
1							
2							
3							
4							
5							
6							
					sum of x_m :	sum of d ² :	

Show your calculation of the standard deviation, s, from d² below:Mean value (\bar{x}): _____

Standard Deviation (s): _____

Range: _____

% NaCl from Table: _____

Name: _____

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Pre-lab Questions

Upon reading the procedure in preparation for this experiment, you should also answer the following questions:

1. Consider Example One in the laboratory discussion. Since measurement 8 lies outside the range, it may be omitted in the calculation of the reported value. Omit measurement 8 and recalculate the mean (\bar{x}). Fill in the d and d^2 columns in the table, then calculate the standard deviation (s) and the range.

Recalculated mean (\bar{x}), without measurement 8: _____

Balance Number	Mass (g) = x_m	$d = x_m - \bar{x}$	d^2
1	24.29		
2	24.26		
3	24.17		
4	24.31		
5	24.28		
6	24.19		
7	24.33		
8 – OMITTED	24.50		
9	24.30		
10	24.23		
sum of x_m :		sum of d^2 :	

Recalculated standard deviation (s): _____ and range: _____

SHOW CALCULATIONS:

2. Now consider Example Two in the laboratory discussion. The student doing the titration repeated the experiment twice more. The following five values were obtained: 0.555 M, 0.565 M, 0.564 M, 0.567 M, and 0.563 M.

A. Use the *Q Test* to demonstrate that the first value should be rejected.

B. Recalculate the values for \bar{x} , omitting the value 0.555 M. Compare with the original value of \bar{x} .

Experiment #2: Graphical Representation of Data and the Use of Excel[®]

Scientists answer posed questions by performing experiments which provide information about a given problem. After collecting sufficient data, scientists attempt to correlate their findings and derive fundamental relationships that may exist between the acquired data. Graphical representations of data illustrate such relationships among data more readily. A graph is a diagram that represents the variation of one factor in relation to one or more other factors. These variables can be represented on a coordinate axes. The vertical axis is the y-axis (or ordinate), and the horizontal axis is the x-axis (or abscissa). When plotting a certain variable on a particular axis, experiments are normally designed so that you vary one property (represented by the *independent variable*) and then measure the corresponding effect on the other property (represented by the *dependent variable*).

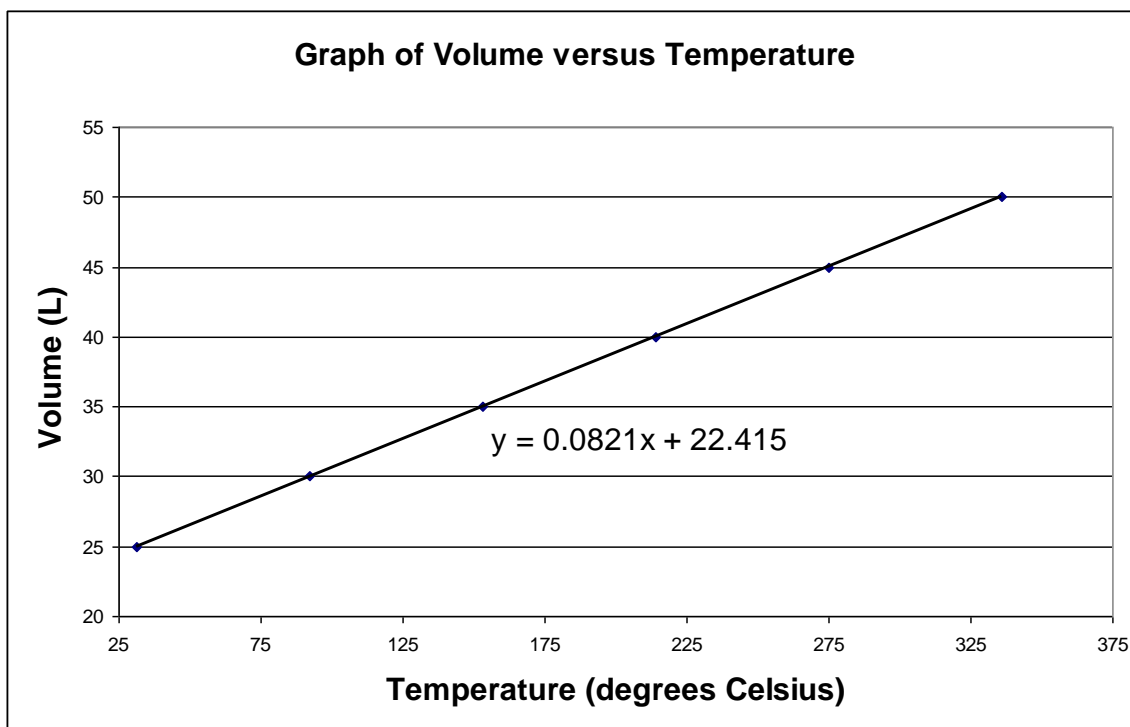
All graphs should conform to the following guidelines:

1. They should have a descriptive title.
2. The independent variable is conventionally placed on the horizontal axis; the dependent variable is plotted on the vertical axis.
3. Label both the vertical and horizontal axes with units clearly marked.
4. The scale chosen for the data should reflect the precision of the measurements. For example, if temperature is known to be ± 0.1 °C, you should be able to plot the value this closely. Moreover, the data points should be distributed so that the points extend throughout the entire page (as opposed to a small portion of the paper).
5. There should be a visible point on the graph for each experimental value.

Let us first examine a direct function involving a linear graph. Consider the following measurements made of an oxygen sample under standard pressure:

Temperature (°C)	Volume (L)
31.49	25.00
92.38	30.00
153.28	35.00
214.18	40.00
275.08	45.00
335.97	50.00

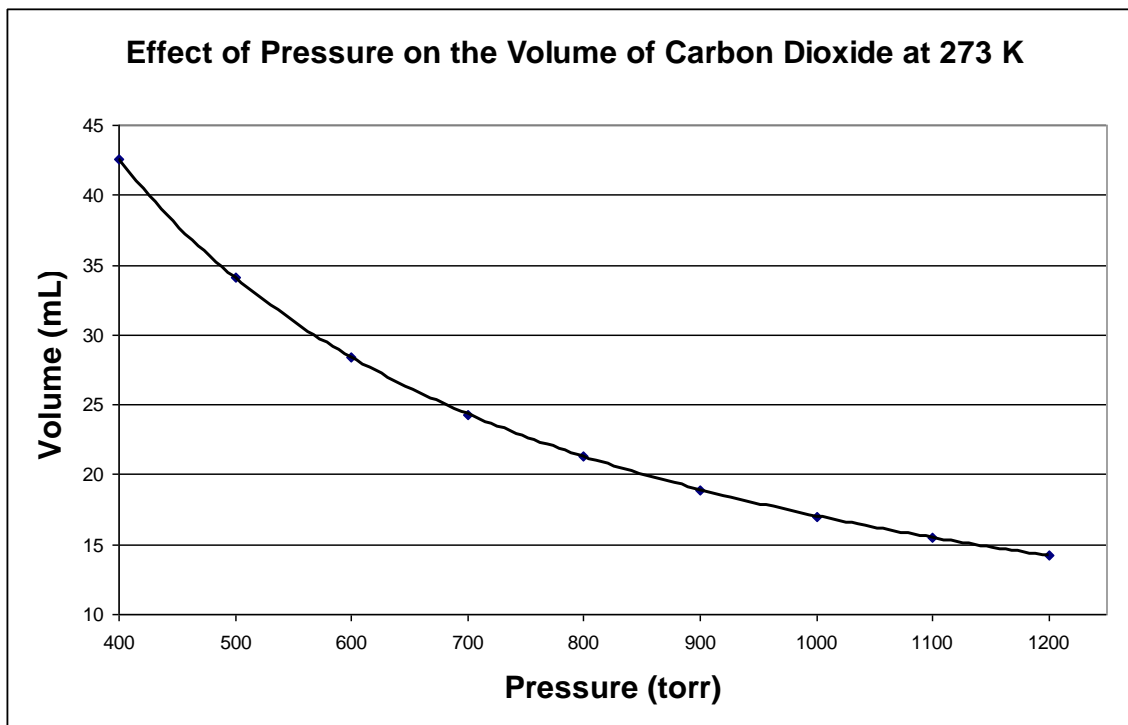
Using graph paper or any graphing program such as Microsoft Office Excel[®], one can first construct a plot of the data, where volume is determined to lie on the y-axis, and temperature is plotted on the x-axis. Once the data is plotted, a best-fitting line is constructed, and an equation of the line in slope-intercept form $y = mx + b$ is formulated, where m = slope and b = y-intercept. That is,



Now examine an indirect function involving a hyperbola. Consider the following measurements made of a carbon dioxide gas sample at 273 K:

Pressure (torr)	Volume (mL)
400	42.6
500	34.1
600	28.4
700	24.3
800	21.3
900	18.9
1000	17.0
1100	15.5
1200	14.2

Once again, using graph paper or any graphing program such as Microsoft Office Excel[®], one can construct a plot of the data, where volume is determined to lie on the y-axis, and pressure is plotted on the x-axis.



As depicted in the graph above, some chemical relationships are not linear; that is, there are no simple linear equations to represent such relationships. Instead, a plot of data for this kind of relationship gives a curved (non-linear) fit. Such a graph is useful in showing an overall chemical relationship, although the slope and the y-intercept are NOT relevant to its interpretation.

In this experiment, you will use acquired measurements and graphical analyses to determine the density of an unknown liquid, learn to use Microsoft Office Excel[®], and create computerized linear and non-linear graphs of provided experimental data. *Students without personal computers or Microsoft Office Excel[®] are invited to use the college's library computers designated for student use.*

Laboratory Procedure

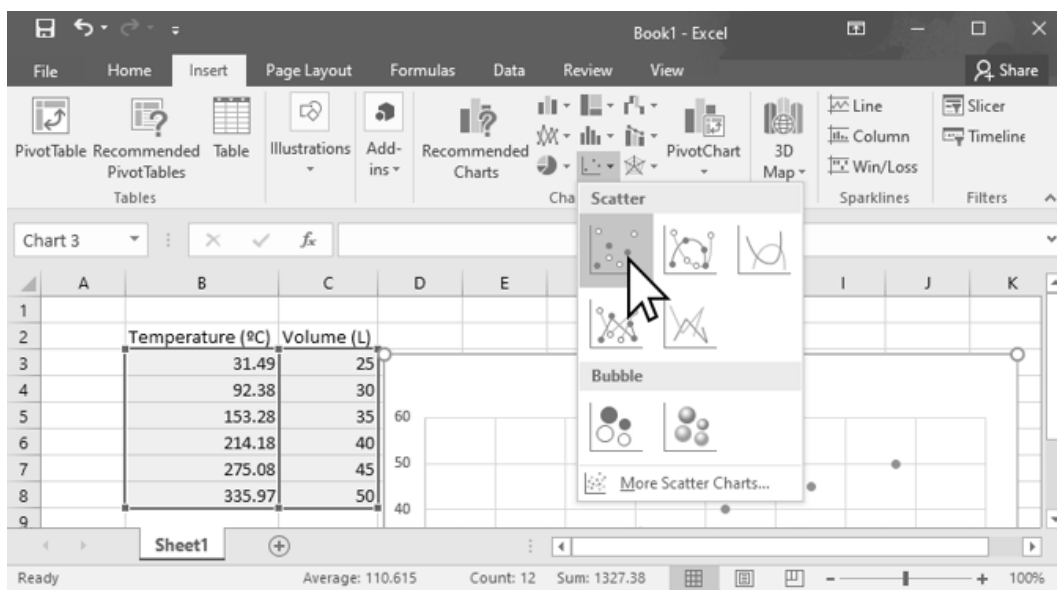
1. Fill one of the 100 mL beakers a little more than half-full of the unknown liquid assigned to you.
2. Pipette 10.00 mL of the liquid from the half-full beaker into the empty one. Pipettes are calibrated to deliver the volume of a liquid specified by the markings on the pipette. Make sure that the bottom of the concave meniscus of the liquid exactly coincides with the line marked on the upper stem of the pipette. Do NOT blow the liquid out of the pipette; let it drain naturally. If there is still a drop of liquid on the tip of the pipette, touch it gently to the side of the container to which the liquid is being transferred. Cover the beaker with the provided watch glass to avoid evaporation of the unknown liquid.

3. Weigh the beaker and cover with the 10.00 mL of liquid in it. Your measurements should be accurate to the nearest 0.001 g.
4. Transfer another 10.00 mL portion of the liquid to the beaker and reweigh the covered beaker and liquid. Repeat this process until you have SIX collective readings of volume and mass.

Excel® Procedure

Note that various versions of Excel® may function a bit differently from the directions outlined below (which work on department-owned laptop computers):

Put the title for your x-axis (include units) in one Excel® cell (box). In the cell to the right, put the title for your y-axis. Using these boxes as headings, input the numeric data (like a table) in the cells under these titles (each box should contain one number; each row represents one data point in x,y format). Click and drag your mouse to highlight just the numeric boxes. From the “Insert” tab, choose a “Scatter” plot. (See example, below.)



Your graph must include a meaningful Chart Title and Axis Titles (with units). These Chart Elements can be added to your graph by clicking on the “+” icon in the upper right corner of your graph. Your instructor may request additional Chart Elements.

To add a Trendline, right click on any data point on your graph and choose “Display Trendline” from the menu that appears. The format trendline pane will appear on the right side of your screen. Linear should be selected by default. From this pane, you should check the box next to “Display Equation on chart.” Your instructor may also ask you to check the box for “Display R-squared value on chart.”

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Data and Calculations

Liquid Unknown # _____

<u>Measurement #</u>	<u>Volume (mL)</u>	<u>Mass (g) of beaker + cover + liquid</u>
1	_____	_____
2	_____	_____
3	_____	_____
4	_____	_____
5	_____	_____
6	_____	_____

Post-lab Questions

1. Using your hand-written graph, determine the density of your unknown (recall that slope = $\Delta y / \Delta x$). When graphing, use all the data points, then draw the best fit straight line. Start at zero for the x axis, and about 10 to 20 grams less than your smallest mass for the y axis. The line may not exactly touch each point. Determine the slope using two points on the best fit line spread apart from each other. Do not use just two data points measured in the experiment as they may not be on the best fit line.
2. Using your hand-written graph, estimate:
 - A. the mass (in grams) of 27.0 mL of your liquid. _____
 - B. what volume (in mL) would 17.0 g of your liquid occupy. _____
3. Using your Excel[®] plot and constructed trendline, write the slope-intercept equation for your liquid unknown. What is the density of your unknown? Make sure to include the appropriate units. How does this density value compare to your result from question #1 above?

4. Using your slope-intercept equation, determine:

A. the mass (in grams) of 27.0 mL of your liquid.

B. what volume (in mL) would 17.0 g of your liquid occupy.

5. The data for temperature and pressure of a certain sample of gas is found to be:

Temperature (°C)	Pressure (mmHg)
-196	215
-78.5	542
-17.8	711
0.1	761
22.7	824
99.3	1037
189	1287

The researcher seals the gas inside of a container, adjusts the temperature of the container, and then measures the pressure of the gas inside. Therefore, temperature is the independent variable and pressure is the dependent variable.

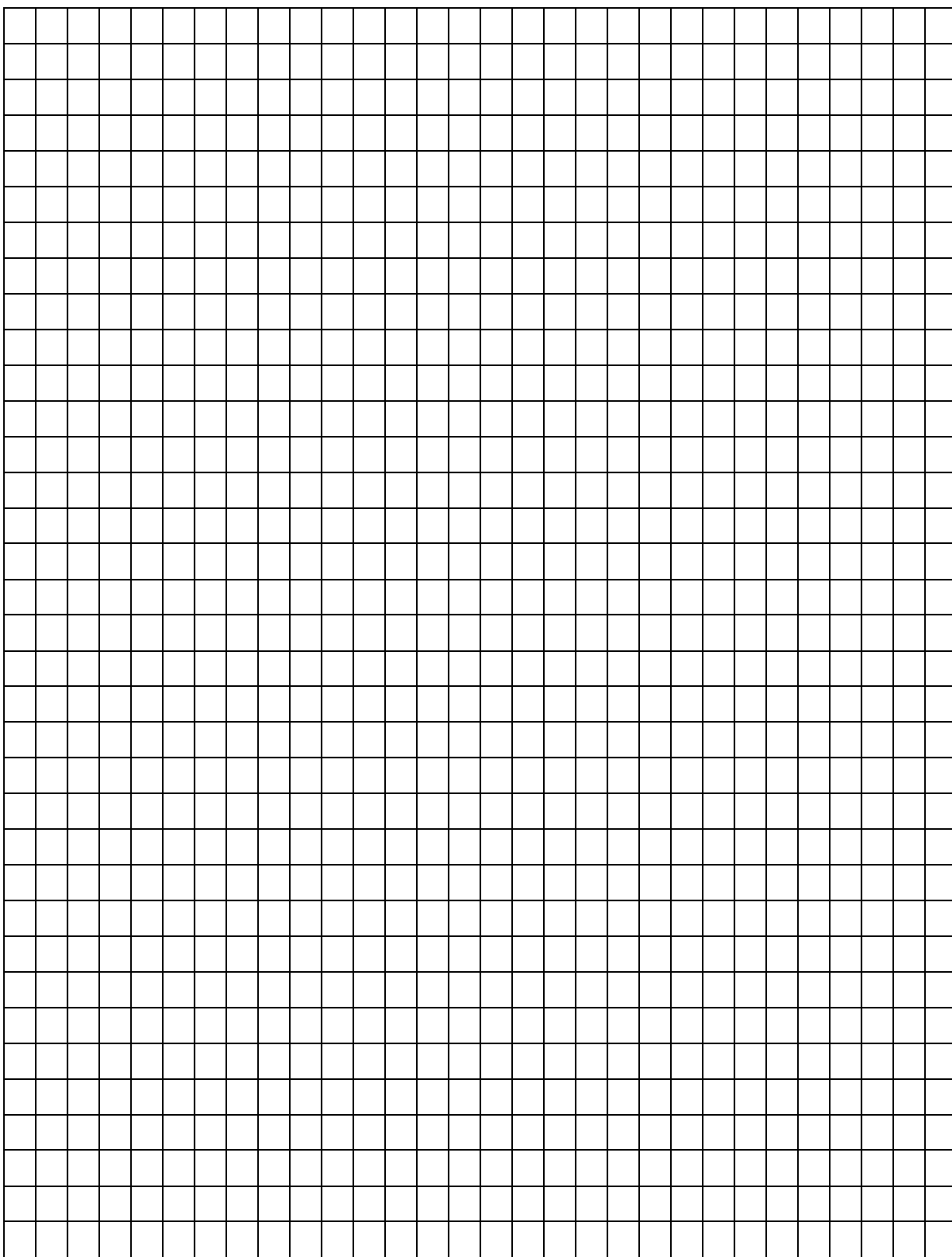
A. Construct an Excel[®] graph of this data with a linear trendline. Make sure to print out a copy of this graph with the slope-intercept equation of the trendline displayed for inclusion with this report. Rewrite the equation, below, using T and P instead of x and y to show the relationship between temperature (T) and pressure (P):

B. Use the equation of the line to calculate the expected pressure of the gas at a temperature of 42.3 °C.

C. Use the equation of the line to predict the temperature of the gas when its pressure is 437 mmHg.

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Experiment #3: Weight Analysis of a Copper Oxide

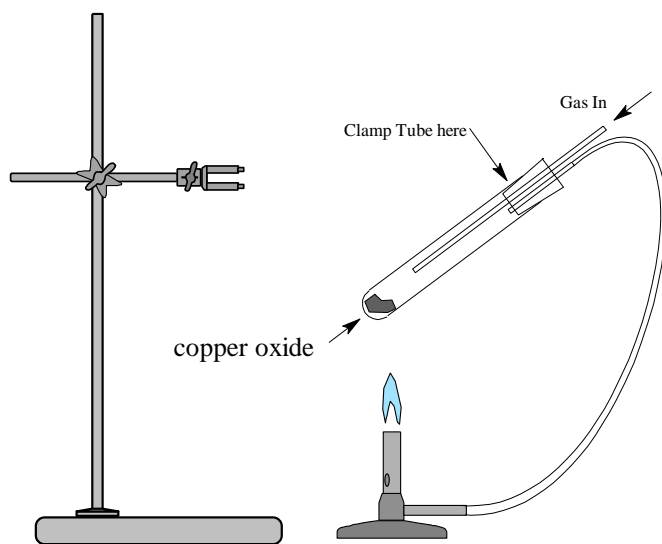
Some metals, such as gold and silver, are naturally found in their elemental form and have been used since prehistoric times. Other metals, such as mercury, can be isolated from their oxides simply by strong heating. Many more metals can be produced by strongly heating the oxides with a reducing agent such as carbon, which transfers electrons to the metal cation. In today's lab, we will use methane gas as a reducing agent, as it is faster and cleaner than charcoal. The empirical formula of an oxide of copper and the mass percentage of copper in the oxide will be determined using the weights of the metal and the oxide according to the following unbalanced chemical equation:



Be aware that there are several forms of copper oxide with formulas that depend on the charge of copper; you will determine the formula for one of these forms.

Procedure

1. Arrange the apparatus as shown in the following schematic. The gas delivery tube should reach to about 5 cm (2 inch) from the bottom of test tube.



Keep tube at 45° angle!

Stop heating if tube glows orange!

Make sure stopper is on snugly!

Use a foil wrapped stopper!

2. Weigh a clean, dry heavy walled (Pyrex) 200 mm ignition tube on the balance. The tube should have straight sides. If it has a flanged open end (i.e. flared out), don't use such a tube, it will melt in the heat!

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3. Weigh about one gram of your copper oxide on a piece of paper or in a plastic tray. (You *do* need to know the exact weight of the powder.) Pour this sample through a funnel with an extension into the weighed test tube. Use the funnel to keep the copper oxide off the sides of the test tube.
4. Weigh and record the mass of the test tube and copper oxide.
5. Connect the test tube to your apparatus. CAUTION: do NOT use plastic coated clamps as they will melt. Use the fiberglass “mitts” to cover the clamps. Check with your instructor for approval before proceeding any further.
6. Turn on the natural gas (methane) slowly. Light and adjust the flame of the Bunsen burner and heat the sample of copper oxide with your burner. Continue to heat the sample with as hot a flame as possible for about 20 minutes or until it turns completely copper colored. If the tube starts glowing orange, pull the Bunsen burner away for awhile. If you heat the tube for too long, it will develop a soft spot and eventually pop open!
7. After the heating is completed, turn off the Bunsen burner but do NOT remove the stopper from the test tube until it is cool enough to hold with your hands. Warm samples may reoxidize if exposed to air.
8. Weigh the tube and contents.
9. Reheat the system for 10 more minutes, let it cool, and reweigh it. If the weight remains the same (within 0.005 g) as the last weighing, you are done. Otherwise, reheat for an additional 10 minute period until the weight of the pellet and tube remain constant for two consecutive weighings.
10. After you have weighed the pellet, dispose of it in the proper waste container.
11. [Optional] Place the pellet on the benchtop and cover it with a tissue or paper towel. Press down on it and slide it back and forth across the benchtop. The pellet will quickly become hot from the friction. Once it is very hot, turn it over and examine the polished surface.

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Data and Calculations

Mass of empty test tube: _____

Mass of the test tube + copper oxide before heating _____

Mass of the test tube + solid after 20 minutes of heating _____

Mass of the test tube + solid after 30 minutes of heating: _____

Mass of the test tube + solid after 40 minutes of heating: *
*if necessary; add more lines if needed _____

Mass of the copper oxide *before* you heated: _____

Final mass of the copper metal powder: _____

Mass that disappeared during the heating: _____

What element or compound disappeared from the copper oxide during heating? _____

Moles of copper in the pellet: _____

SHOW CALCULATION:

Moles of oxide that escaped: _____

SHOW CALCULATION:

Empirical formula of the initial copper oxide: _____

SHOW CALCULATION:

Mass percentage of copper in copper oxide: _____

SHOW CALCULATION:

Post-lab Questions

1. Write the balanced chemical equation for the reaction of your oxide using the experimentally determined empirical formula.

2. Imagine you had just discovered copper. Which of the following formulas would be *possible* guesses (however unlikely) for the empirical formula of copper oxide? Which would be *good* guesses if you used the periodic table as a guide to understanding copper's probable charge? Briefly explain your choices below.

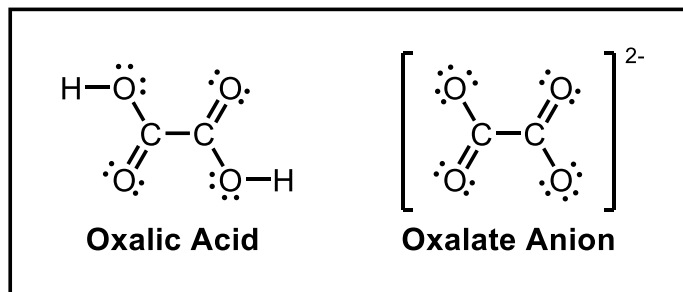


3. A sample of an iron oxide weighing 1.996 g yields 1.396 g of iron on reaction with methane gas. Determine the percent composition and the empirical formula of the iron oxide from this data. **SHOW ALL CALCULATIONS!** Random guessing will NOT earn you any credit.

Experiment #4: Synthesis and Decomposition of an Oxalate Salt

Introduction:

Oxalic acid ($\text{H}_2\text{C}_2\text{O}_4$) is an organic acid that is widespread in biology. It is the simplest diacid, and forms compounds called oxalates when it loses two hydrogens.



The best-known oxalate salt is formed from calcium. These mineral deposits, kidney stones, are a common medical condition for not just humans, but also dogs, cats, and even iguanas. Kidney stones are formed by the precipitation of calcium ions with oxalate ions ($\text{C}_2\text{O}_4^{2-}$) in the urine. Surprisingly, increased consumption of calcium in the diet is not correlated with increased occurrence of kidney stones. However, eating high content oxalic acid foods, such as spinach, nuts, rhubarb, and sesame seeds, can lead to more kidney stones.

Today, we make an oxalate from either Iron or Manganese. Then, we will decompose the oxalate with heat. (This reaction is also called *pyrolysis*.)

Procedure:Part 1 – Synthesis of the Oxalate Salt

1. Weigh between 1.9 g and 2.1 g of YOUR starting sample (hydrated metal salt with either Fe or Mn) into a 100 ml beaker. Record the metal salt's exact mass in Table 1b, keeping all the significant numbers on the balance. Add 10 mL distilled water and stir to dissolve.
2. Use a graduated cylinder to measure 25.0 mL of 0.888 M oxalic acid solution.
Solution contains 4.0 g oxalic acid ($H_2C_2O_4$) per 50.0 mL of solution.
3. Once the starting metal salt is completely dissolved, slowly pour the 25.0 ml of oxalic acid solution into the same beaker. Initially stir to mix and allow the solution to sit for 10 minutes while a precipitate forms. Try not to lose any precipitate.
4. Prepare a Büchner funnel vacuum filtration while the precipitate is forming. Before placing the filter paper in the Büchner funnel, weigh and record the filter paper mass in Table 1 (f).
5. After the 10 minute waiting period, begin vacuum suction through the filter paper in the funnel, wet the paper with your wash bottle containing DI water. Then slowly transfer the precipitate solution from the beaker onto the center of the filter paper. Attempt to keep most of the precipitate in the center of the paper and not on the funnel walls. You are keeping the solid formed. The filtrate liquid that passes through will be waste that may go down a sink.
6. Once the entire solution has been emptied into the funnel, use your wash bottle to rinse out any remaining precipitate into the vacuum Büchner funnel. Transfer all of the precipitate from the beaker.
7. Because water dries too slowly, next wash the precipitate with ethanol. Do this while the vacuum is still running. Obtain an ethanol filled bottle from under the hood. Gently squirt the ethanol over the precipitate in the funnel to saturate. During this step, water on the precipitate particles will dissolve in the ethanol and facilitate the drying of your sample. Squirt a bit more ethanol over the solid one or two more times after pausing a minute.
8. Keep the vacuum on to draw air through the filter paper. The ethanol will evaporate from the solid. Keep the vacuum on until the solid is dry, (about 10-15 minutes). You can push around the precipitate with a glass rod to test for dryness. Once the solid is completely dry, turn off the vacuum.
 - **Be patient!** The drier your solid product, the more accurate your results.
 - This solid product must be **completely dry** before beginning Part II of the procedure.

9. Weigh the colored solid and record its mass in Table 1: (*g*) *experimental yield*
10. Determine (*h*) the identity of the limiting reactant, (*i*) theoretical yield of the hydrated metal oxalate, and (*j*) the percent yield of the hydrated metal oxalate product. Complete Table 1.

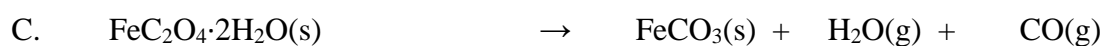
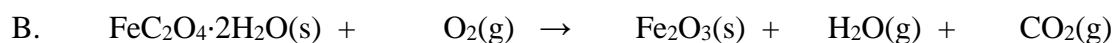
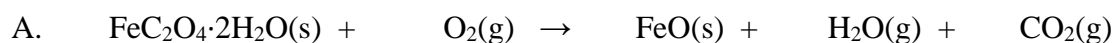
Part 2 – Pyrolysis of the Metal Oxalate

You will pyrolyze the metal oxalate you synthesized in Part I. You will use stoichiometric considerations to determine which pyrolysis reaction (of those given in prelab Question #5) your metal oxalate undergoes.

1. Record the mass of the Al dish in Table 2 (*a*).
2. Measure between 0.990 g and 1.010 g of your dry, hydrated metal oxalate into the aluminum dish. Record precise masses in Table 2 (*b*). Don't use the entire sample from the first part if you have more than 1.0 g product! If you have less than one gram, you may be able to find another group with excess of your same sample to share with you.
 - To enhance heat transfer, spread out the solid over the bottom of the dish.
 - It is important to use between 0.990 g and 1.010 g because your prelab calculations were based upon pyrolysis of 1.0 g of hydrated metal oxalate.
3. Turn the dial on a hot plate up to about 400°C or level 7. Place the aluminum dish on the hot plate. The hydrated metal oxalate will undergo dehydration (loss of water) followed by a pyrolysis reaction. You will see a visible change in the product.
4. Wait until the metal oxalate has completely changed color. Remove the aluminum dish from the hot plate using a pair of metal tongs and place it on the laboratory bench to cool to room temperature. The aluminum dish should cool quickly but the precipitate may take longer.
 - Caution: the aluminum dish will be VERY hot – Use tongs!!
 - Be patient! The cooler the precipitate, the better your results.
 - Never weigh hot objects. It's bad for the balance's electronics.
5. Record the mass of the pyrolyzed product in Table 2 (*c*).
6. Complete Table 2. The pyrolysis reaction occurs with near 100% yield, so the actual yield of the pyrolysis reaction should be very close to the theoretical yield you calculated for the correct reaction equation (as calculated in Question #5 of the prelab).
7. Dispose of all chemical waste properly.

Option Fe: Pre-lab Calculations for the Iron Based Experiment:

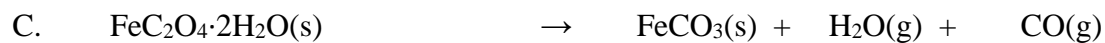
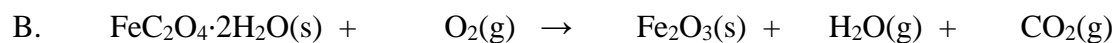
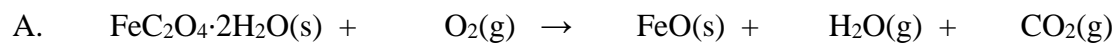
1. Calculate the molar mass of the starting hydrated salt material, $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$.
2. Your oxalate synthesis product will be $\text{FeC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$. Calculate the molar mass of $\text{FeC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$.
3. Write a balanced molecular equation that shows your $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, reacting with oxalic acid ($\text{H}_2\text{C}_2\text{O}_4$) to form $\text{FeC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$. What will be the other product(s) of the double displacement reaction? Water will need to be added somewhere to balance correctly.
4. Pyrolysis of $\text{FeC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ will follow one of the following molecular equations. Balance each molecular equation. (*It's OK to keep a fraction when balancing the O_2 molecule in A or B*)



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5. For each of the possible reactions, calculate the theoretical yield (in grams) of the solid product, assuming that you use 1.0 g $\text{FeC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ and that oxygen is the excess reactant. You will need to write in coefficients from question 4 to balance the molecular equations.



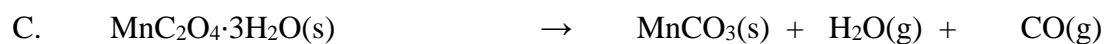
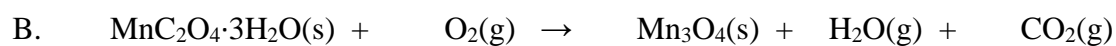
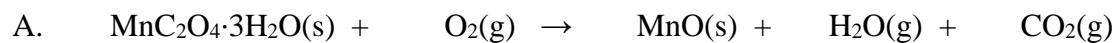
Option Mn: Pre-lab calculations for the Manganese based experiment:

1. Calculate the molar mass of the starting hydrated salt material, $\text{MnSO}_4 \cdot \text{H}_2\text{O}$.
2. Your oxalate synthesis product will be $\text{MnC}_2\text{O}_4 \cdot 3\text{H}_2\text{O}$. Calculate the molar mass of $\text{MnC}_2\text{O}_4 \cdot 3\text{H}_2\text{O}$
3. Write a balanced molecular equation that shows $\text{MnSO}_4 \cdot \text{H}_2\text{O}$, reacting with oxalic acid ($\text{H}_2\text{C}_2\text{O}_4$) to form $\text{MnC}_2\text{O}_4 \cdot 3\text{H}_2\text{O}$. What will be the other product(s) of the double displacement reaction? Water will need to be added somewhere to balance correctly.
4. Pyrolysis of $\text{MnC}_2\text{O}_4 \cdot 3\text{H}_2\text{O}$ will follow one of the following molecular equations. Balance each molecular equation. (*It's OK to keep a fraction when balancing the O_2 molecule in A or B.*)
 - A. $\text{MnC}_2\text{O}_4 \cdot 3\text{H}_2\text{O}(\text{s}) + \text{O}_2(\text{g}) \rightarrow \text{MnO}(\text{s}) + \text{H}_2\text{O}(\text{g}) + \text{CO}_2(\text{g})$
 - B. $\text{MnC}_2\text{O}_4 \cdot 3\text{H}_2\text{O}(\text{s}) + \text{O}_2(\text{g}) \rightarrow \text{Mn}_3\text{O}_4(\text{s}) + \text{H}_2\text{O}(\text{g}) + \text{CO}_2(\text{g})$
 - C. $\text{MnC}_2\text{O}_4 \cdot 3\text{H}_2\text{O}(\text{s}) \rightarrow \text{MnCO}_3(\text{s}) + \text{H}_2\text{O}(\text{g}) + \text{CO}(\text{g})$

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5. For each of the possible reactions, calculate the theoretical yield (in grams) of the solid product, assuming that you use 1.0 g $\text{MnC}_2\text{O}_4 \cdot 3\text{H}_2\text{O}$ and that oxygen is the excess reactant. You will need to write in coefficients from question 4 to balance the molecular equations.



Data and CalculationsPart 1: Synthesis of Metal Oxalate

Determine the identity of the limiting reactant, theoretical yield of the hydrated metal oxalate, and the percent yield of the hydrated metal oxalate product. Complete Table 1.

Table 1. Synthesis of Metal Oxalate

a) complete chemical formula of your starting material: (formula found on question 1)	
b) mass of hydrated metal salt used (g) <i>tare your 100 ml beaker and add 1.9-2.1g sample</i>	
c) moles of hydrated metal salt used convert grams from (b) using molar mass in question 1	
d) volume of 0.888M oxalic acid solution used <i>should be close to 25.0mL</i>	
e) moles of oxalic acid used <i>calculate from volume and molarity</i>	
f) mass of filter paper	
g) experimental yield: total mass of hydrated metal oxalate precipitate (grams) after drying by vacuum filtration. filter paper + salt mass: _____ <i>subtract the filter paper</i>	
h) identify the limiting reactant: either your starting formula in part (a) or oxalic acid <i>Refer to the balanced chemical equation in pre-lab quest. #3 and your moles of each reactant calculated in c and e</i>	
i) theoretical yield of hydrated metal oxalate product (g)	
j) percent yield of hydrated metal oxalate product	

Calculations: Show your calculations. Watch your units and report all answers with the correct number of significant figures.

Data and CalculationsPart 2: Decomposition (Pyrolysis) of Metal Oxalate

Complete Table 2. The pyrolysis reaction occurs with near 100% yield, so the actual yield of the pyrolysis reaction should be very close to the theoretical yield you calculated for the correct reaction equation (as calculated in Question #5 of the prelab).

Table 2. Pyrolysis of Metal Oxalate

a) Weigh the empty Aluminum dish before adding the filtered product.	
b) Before pyrolysis: mass of hydrated metal oxalate (grams) <i>Ideally between 0.99 to 1.01 gram</i> Al + oxalate salt mass: _____ <i>subtract the Al dish</i>	
c) After pyrolysis: final mass of pyrolysis product (grams) Al + pyrolyzed salt mass: _____ <i>subtract the Al dish</i>	
d) theoretical mass of product in reaction A From prelab (quest. #5A):	
e) theoretical mass of product in reaction B From prelab (quest. #5B):	
f) theoretical mass of product in reaction C From prelab (quest. #5C):	
g) Using the above information, which pyrolysis reactions actually occurred, A , B or C ?	
h) What is the expected chemical formula of the pyrolyzed metal salt formed?	

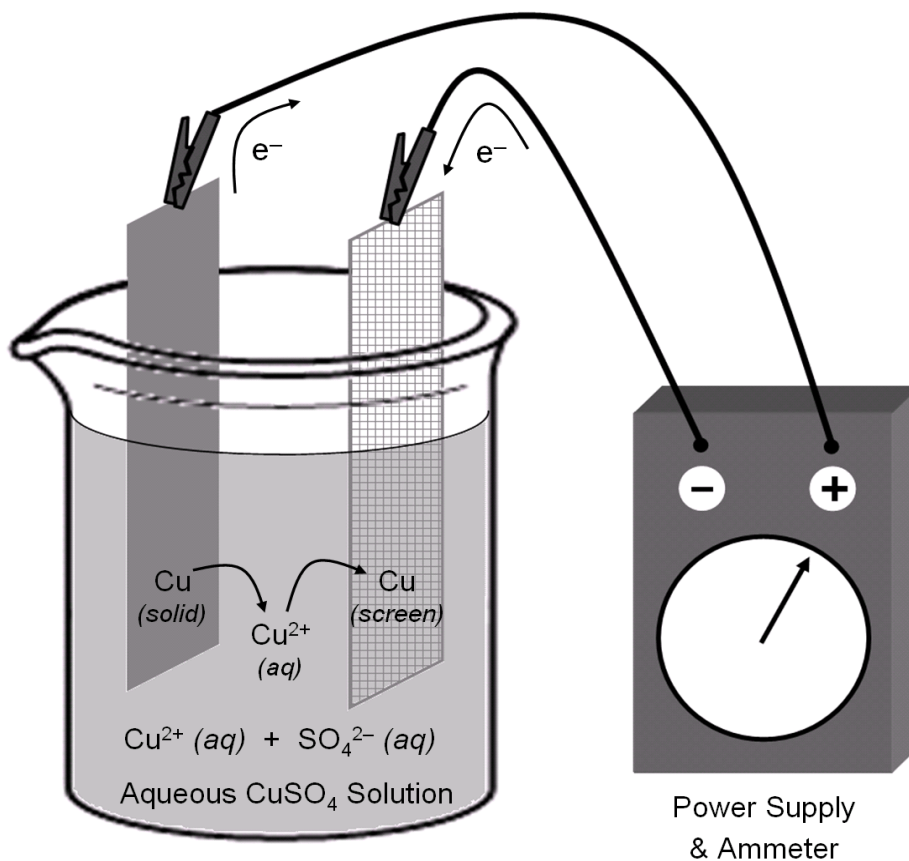
Calculations: Refer to your work from prelab Question #5 from your option. Watch your units and report all answers with the correct number of significant figures.

Experiment #5: Determination of Avogadro's Number from Electrodeposition of Copper

There are many ways of finding Avogadro's number; regardless of the method used, they all begin with an arbitrary amount of one compound or element. Currently, the mole is defined by the weight of 12.0000 grams of carbon-12 atoms. In today's lab, we will measure Avogadro's number using electroplating techniques.

You will use an apparatus (depicted below) to plate out a small amount of copper. Because you will be able to measure the current delivered using an ammeter, you can determine the number of electrons that passed through the wire. Using the equation that relates the number of electrons to the number of copper atoms, you can find the number of atoms in the freshly plated sample. Finally, you must assume that the atomic weight of copper is 63.54 grams/mole, similar to the way that the atomic weight of 12.00 grams/mole was chosen for carbon. Consider the following useful pieces of information:

1. Gram atomic weight of copper: 63.54 grams of Cu = 1 mole of Cu atoms
2. Charge on an e^- : 1.60×10^{-19} coulombs = 1 electron
3. Amps \times seconds = coulombs OR 1 amp = 1 coulomb/sec
4. The balanced half-reaction for copper is represented by $\text{Cu} \rightarrow \text{Cu}^{+2} + 2e^-$



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Procedure

1. Set up the apparatus as indicated in the picture.
2. Clean the solid Cu strip with steel wool; wash BOTH Cu strips with dilute NaOH, followed by dilute HNO₃, and finally rinse with DI H₂O. At this point, make certain to handle both Cu strips with forceps.
3. Rinse strips with acetone and when dry, weigh and record the mass of the Cu strips.
4. Place the strips in a 250 mL beaker containing about 200 mL of 1M CuSO₄. DO NOT ALLOW THE STRIPS TO TOUCH.
5. Adjust the current to approximately 0.175 Amp (175 mA) and run the system for 30 minutes. If you cannot maintain a constant amp reading, take readings at 5 minute intervals and average the current.
6. Carefully rinse both Cu strips under a slow stream of DI water, then rinse with acetone, and allow to dry. Weigh and record the mass of each dry Cu strip.

Data and Calculations

1. Current _____ amps
2. Time _____ seconds
3. Initial Mass of Copper A (solid) _____
4. Final Mass of Copper A (solid) _____
5. Change in mass of Copper A (solid) _____
6. Initial Mass of Copper B (screen) _____
7. Final Mass of Copper B (screen) _____
8. Change in mass of Copper B (screen) _____
9. Average change in mass of the Copper Strips _____

Experiment #6: Synthesis of Copper(II) Compounds

The law of conservation of mass states that matter is neither created nor destroyed during a chemical reaction. This was first observed by Antoine Lavoisier in the late eighteenth century, in which he observed that the total mass of all substances present after a chemical reaction is the same as the total mass before the reaction. Any changes which occur during a reaction merely involve the rearrangement of atoms. In theory, this means that an element may go through several reactions until it is transformed back into its original state without the loss of any mass. In practice, however, this may not always *appear* to be the case.

In this experiment, Lavoisier's theory is tested by reacting solid copper with certain reagents and synthesizing various compounds in order to arrive at the original mass of copper at the conclusion of the experiment.

Procedure

1. Weigh approximately 0.35 g of copper into a 250 mL beaker. Describe the color and form of the sample. Record the mass of the sample.
2. Part A: WORK IN THE HOOD! Add 3–5 mL of concentrated HNO_3 slowly to dissolve the copper; warm gently on a hot plate if all of the copper does not dissolve. After the copper has dissolved, remove the solution from the hot plate and add 15 mL of deionized H_2O slowly. Note the color of the solution as well as the reaction that is occurring.
3. IN THE FUME HOOD, prepare a boiling water bath for Part C: Fill a 400 mL beaker with about 200 mL of water and add 2–3 boiling stones. Heat this solution at about 50 % power on a hot plate. While waiting for the water to boil, continue to Part B.
4. Part B: Use NO HEAT during this step! Add 6 M $\text{NaOH}(\text{aq})$ very slowly while stirring until the solution turns red litmus paper blue (use a stirring rod to test a drop of solution). Do not confuse the color of the precipitate with the color of the litmus paper. Note any color changes; a precipitate will form. Describe the color and the supernatant. Note the reaction that is occurring.
5. Part C: Dilute the solution to 100 mL total with DI water. Place your 250 mL beaker in the boiling water bath that you prepared in the fume hood. If the water bath is not boiling yet, you may increase the power on the hot plate to bring the water to a boil. Allow your solution to heat up and note any color changes. When all of the solid material appears to have changed color, allow the solution to heat for an additional 3 minutes, then turn off the hot plate, remove the 250 mL beaker from the water bath and allow the solution to cool. While the solution is cooling, prepare a filter by folding a piece of filter paper in half twice to fit your funnel. Filter the cooled mixture and discard the liquid. Rinse the filter with deionized water. You will return the solid to the original beaker, so it is not necessary to completely transfer the solid. Record the color of the residue and the filtrate. Note the reaction that is occurring.

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6. Part D: Transfer the filter paper and solid back to the original beaker. Dissolve the solid copper oxide by adding approximately 10 mL of 3 M $\text{H}_2\text{SO}_4(\text{aq})$ to the filter paper containing the residue from the previous step. Once the solid has dissolved, remove the filter paper and rinse it with 10 – 20 mL of deionized water. Add the washings to the acid solution, and save the solution for the next step. Note any color change. Also note the reaction that is occurring.
7. Part E: **WORK IN THE HOOD!** Add about 0.40 g of zinc metal to the acidic copper solution. If any blue color remains after the zinc has dissolved, a bit more zinc may need to be added (record in your report!). Note the reaction that is occurring. Dissolve any excess zinc with a small amount (approximately 5 mL) of 3 M $\text{H}_2\text{SO}_4(\text{aq})$.
8. Filter the supernatant liquid from the solid and wash the solid 3 times with 20 mL portions of deionized water.
9. Transfer the solid copper onto a large watch glass and place it in the laboratory oven (PS 103 or PS 107) for 15 to 20 minutes at around 100 °C or until dry. Weigh to determine the mass of recovered copper.

Data Analysis and Calculations

Initial color and form of the copper _____

Mass of Cu(s) at the beginning of the experiment _____

Mass Recovered _____

% Recovery _____

Comment and discussion: Do your results support Lavoisier's law of conservation of mass? How does your percent recovery deviate from the expected 100%? Briefly explain.

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Post-lab Questions

1. Write the symbol or formula for the form of copper that is present in the following parts of the experiment:

A. after adding nitric acid _____

B. after adding NaOH, litmus paper turns blue _____

C. after boiling _____

D. after adding sulfuric acid _____

E. after adding zinc _____

2. Now give the color of the copper substances in each of the steps above:

A. _____ D. _____

B. _____ E. _____

C. _____

3. A student reports 115% recovery. How could he/she possibly have more copper at the end of the experiment than he/she started with? Explain.

4. If a student used a penny as the source of copper in this experiment, would it matter if a pre-1982 penny (essentially pure copper) versus a post-1982 penny (copper exterior over a zinc core) was used? Would using a post-1982 penny pose any experimental complications? Briefly explain.

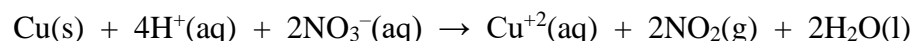
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5. Part A in today's experiment is classified as a redox reaction in which electrons are transferred via oxidation of Cu and reduction of the N in HNO₃. The balanced molecular equation is:



The net ionic equation for this reaction is:



Write balanced molecular, ionic, and net ionic equations for the other four reactions in this inorganic sequence. Classify the types of each reaction in as many possible ways (i.e. redox, synthesis, combustion, decomposition, single replacement, double displacement, precipitation, neutralization).

Part B:

Part C:

Part D:

Part E:

6. Referring to part E, write the reaction for the reduction of copper(II) ion into solid copper using zinc. Calculate the theoretical mass of zinc needed to carry out the reaction based on the initial mass of copper used. Compare this theoretical mass of zinc with the actual amount used in the laboratory. Justify any differences.

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Pre-lab Questions

Upon reading the procedure in preparation for this experiment, you should also answer the following questions:

1. Use the outlined procedure to describe:
 - a) a test for determining whether a solution is basic enough.
 - b) a test to decide whether enough zinc has been added.
2. Write out and classify the molecular, ionic, and net ionic equations that take place when H_2SO_4 is added to the excess zinc in part E.
3. Does observing a color change always indicate that a chemical change has occurred? Explain why or why not.
4. What should the student do if the solution in step E is still blue?

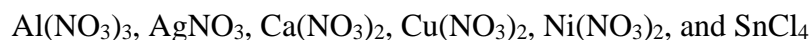
Experiment #7: Qualitative Analysis of Various Reagents

The Ten Bottle Mystery

There are two broad categories of problems in analytical chemistry: quantitative and qualitative analyses. Quantitative analysis deals with the determination of the amounts of species present in a sample. Qualitative analysis involves establishing whether given species are present in detectable amounts in a sample. In the laboratory, qualitative analysis often tests for the presence of a possible component by adding a reagent that reacts with the component in a characteristic way.

You will be furnished with ten numbered bottles, each of which will contain a single substance. Prior to the experiment, you will be assigned either Set A or Set B (listed below) of unknowns. Your task is to find out which solution is in which bottle. You will do this by mixing small volumes of the solutions in test tubes and recording your observations. *NO ADDITIONAL REAGENTS OR ACID-BASE INDICATORS SUCH AS LITMUS PAPER ARE ALLOWED.* You are permitted, however, to use the odor and color of the different species and to use your reaction matrix (to be completed BEFORE LAB and discussed below) when identifying the various bottles.

Of the ten solutions, four are common laboratory reagents. They are 6 M HCl, 3 M H₂SO₄, 6 M NH₄OH (*i.e.* NH₃ (aq)), and 6 M NaOH. The other six are 0.1 M solutions of:



To determine which solution is in each bottle, you will need to know what happens when the various solutions are mixed. In some cases, nothing happens. This often happens when two solutions with similar cations are mixed. When one of the reagents is mixed with a different cation solution, you may get a precipitate, white or colored, and that precipitate may dissolve in excess reagent by complex-ion formation (a topic for Chemistry 1B). Furthermore, some solutions become very warm and/or produce a visible vapor.

You will not be able to solve your particular ten bottle mystery without doing some preliminary work. You need to know what to expect when any two of your ten solutions are mixed. You can find this out by consulting your chemistry textbook, solubility rules, referring to various reference works on qualitative analysis such as the *CRC Handbook of Chemistry and Physics*, and searching online. A convenient way to tabulate the information you obtain is to set up a matrix with ten columns and ten rows, one for each solution. At each intersection, write the products that you'd expect to find. For example, if a precipitate forms, you might write "P" along with the color. If heat is evolved, you might write an "H". If gas or smoke is formed, you might write "G" and "S", respectively. Since mixing solution A with B is the same as mixing B with A, not all 100 spaces in the 10-by-10 matrix need to be filled.

Because you are allowed to use the odor or color of a solution to identify it, the problem is somewhat simpler than it might first appear. In each set of ten solutions, you will probably be able to identify at least two solutions by odor and color tests. Knowing the identity of

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those solutions, you can make mixtures with the other solutions in which one of the components is known. From the results obtained with those mixtures and the information in the matrix, you can identify other solutions. These can be used to identify still others, until the entire set of ten is finally identified.

Pre-lab Questions

1. Fill in the matrix below to show how various solutions react, as described in the procedure.

											HCl
											H ₂ SO ₄
											NaOH
											NH ₄ OH
											Al(NO ₃) ₃
											AgNO ₃
											Ca(NO ₃) ₂
											Cu(NO ₃) ₂
											Ni(NO ₃) ₂
											SnCl ₄
											NOTES
HCl											
	H ₂ SO ₄										
		NaOH									
			NH ₄ OH								
				Al(NO ₃) ₃							
					AgNO ₃						
						Ca(NO ₃) ₂					
							Cu(NO ₃) ₂				
								Ni(NO ₃) ₂			
									SnCl ₄		

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2. Which solutions should you expect to identify by simple observations?

3. Outline the procedure you will follow in identifying the remaining solutions. Be specific about what to look for and what conclusions you expect to draw from your observations.

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QUALITATIVE ANALYSIS Report Sheet

Final Identifications:

No. 1 _____ No. 6 _____

No. 2 _____ No. 7 _____

No. 3 _____ No. 8 _____

No. 4 _____ No. 9 _____

No. 5 _____ No. 10 _____

Use the next few pages to write balanced MOLECULAR, IONIC, and NET-IONIC equations for TEN of the reactions that occurred during this laboratory experiment. Make sure to include the physical states of all the products. These equations must be turned in along with this report sheet to receive full credit upon conclusion of the lab.

1. Molecular:

Ionic:

Net-ionic:

2. Molecular:

Ionic:

Net-ionic:

3. Molecular:

Ionic:

Net-ionic:

4. Molecular:

Ionic:

Net-ionic:

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5. Molecular:

Ionic:

Net-ionic:

6. Molecular:

Ionic:

Net-ionic:

7. Molecular:

Ionic:

Net-ionic:

8. Molecular:

Ionic:

Net-ionic:

9. Molecular:

Ionic:

Net-ionic:

10. Molecular:

Ionic:

Net-ionic:

Experiment #8: Calorimetry

When a substance undergoes a change in temperature, the quantity (q) of heat lost or gained can be calculated using the mass (m), specific heat (s), and change in temperature ($\Delta T = T_{\text{final}} - T_{\text{initial}}$):

$$q = m s \Delta T \quad (1)$$

The specific heat (s) is the amount of heat required to change the temperature of one gram of a particular substance by one degree Celsius (or 1 K). For water, the specific heat is defined as 1 cal / g °C or measured to be 4.184 J / g °C.

Heat exchange experiments are conducted in a calorimeter which is an insulated container. The calorimeter is constructed such that ideally there will be no heat exchange between the contents of the calorimeter and the surrounding environment. However, heat exchanges can occur between the components within the calorimeter. This can be expressed in the following way:

$$q_{\text{system}} = q_{\text{gain}} + q_{\text{loss}} \quad (2)$$

If the system does not exchange heat with the surroundings, then the $q_{\text{system}} = 0$, and

$$q_{\text{gain}} = -q_{\text{loss}} \quad (3)$$

When a hot substance comes in contact with a cold substance within a calorimeter, heat will spontaneously pass from the hot to the cold substance until they achieve a thermal equilibrium (the same constant temperature). The quantity of heat lost and gained is equal in magnitude, but opposite in sign. For example, if a piece of hot metal is placed in cold water within a calorimeter, the following thermodynamic equation can be used:

$$q_{\text{water}} = -q_{\text{metal}} \quad (4)$$

When a chemical reaction occurs in a water solution, the situation is similar to when a hot piece of metal is placed in water. When a reaction occurs, there is a change in the potential energy of the chemicals, resulting in a heat exchange in the calorimeter. As in the hot metal experiment, the heat flow of the reaction is equal in magnitude but opposite in sign to that of the solution as shown by the following equation:

$$q_{\text{reaction}} = -q_{\text{solution}} = -(m s \Delta T)_{\text{solution}} \quad (5)$$

By measuring the mass of the solution (the water plus the solute) and by observing the temperature change that the solution undergoes, we can calculate q_{solution} and therefore q_{reaction} . Because the experiment is performed under constant pressure conditions, the heat flow of the reaction is also equal to the enthalpy change, ΔH , for the reaction. Since the mass of reactants and products can vary for different experiments, the q_{reaction} will also vary.

Therefore, ΔH is generally reported as the heat of reaction per a fixed amount of one reactant or product, and we will calculate ΔH per gram or per mole of this substance.

$$\Delta H_{\text{rxn}} = \frac{q_{\text{rxn}}}{\text{mass}_{\text{substance}}} \quad \text{or} \quad \Delta H_{\text{rxn}} = \frac{q_{\text{rxn}}}{\text{mole}_{\text{substance}}} \quad (6)$$

If the temperature of the water goes up, heat has been given off by the reaction, so that reaction is exothermic, and ΔH of the reaction is negative. If the temperature of the water goes down, heat is absorbed by the reaction, therefore the reaction is endothermic, and ΔH for the reaction is positive.

Procedure

Construct a calorimeter, where two polystyrene coffee cups with a plastic cover on top are nested together. Weigh and record the mass of the clean, dry calorimeter. Place the calorimeter into a 400 mL glass beaker for stability during the experiment.

Measure 25.0 mL of 1.00 M H_2SO_4 and 75.0 mL of 1.50 M NaOH into separate 100 mL beakers. Record the temperature of each of the solutions to the nearest 0.01 °C using the same thermometer. Be sure to wash and dry the thermometer after putting it into each solution to avoid initiating the reaction by prematurely mixing the solutions.

Immediately and simultaneously pour both solutions into the calorimeter, replace the lid, insert the thermometer, and stir vigorously. Record the final temperature to the nearest 0.01 °C. Carefully remove the thermometer from the calorimeter, then weigh and record the mass of the calorimeter containing the reaction mixture.

Dispose of any waste down the drain, rinse the cups, lids, and thermometers with DI water, and dry them thoroughly before returning them to where you originally got them.

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Data and Calculations

Mass of clean, dry calorimeter: _____ g

Mass of calorimeter with solution after conclusion of reaction: _____ g

Final mass of reaction solution: _____ g

Initial temperature of H₂SO₄ solution: _____ °C

Initial temperature of NaOH solution: _____ °C

Average initial temperature of starting solutions: _____ °C

Final temperature of mixture: _____ °C

Change in temperature of solution (ΔT): _____ °C

1. Write the balanced chemical equation for your acid/base neutralization reaction.

2. Determine the theoretical yield of water (in grams) of the reaction that you carried out.

3. What is the limiting reactant of your reaction?

4. Calculate the mass of each reactant that is theoretically left over at the end of your reaction.

_____ g of NaOH left over _____ g of H₂SO₄ left over

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5. Assuming that the specific heat capacity of the solution that you used is equal to $3.70 \text{ J / g } ^\circ\text{C}$, calculate the heat (q_{solution}) in kJ that was absorbed by the contents of the coffee cup during the reaction.

6. Calculate ΔH of your reaction using the units specified:

_____ kJ / g H_2O formed _____ kJ / mol H_2O formed

7. Rewrite your balanced chemical equation and include the value of ΔH beside it:

_____ \rightarrow _____ $\Delta H =$ _____ kJ

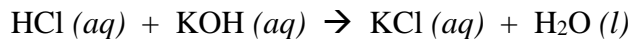
8. Use the ΔH_f° values given to calculate the theoretical value of ΔH for your reaction.

Substance	ΔH_f° (kJ / mol)
$\text{H}_2\text{SO}_4 (aq)$	-909.3
$\text{NaOH} (aq)$	-470.1
$\text{H}_2\text{O} (l)$	-285.8
$\text{Na}_2\text{SO}_4 (aq)$	-1387.1

9. Using your results from questions 7 & 8, calculate the percent error in your determination of ΔH .

Pre-lab Assignment

A student carries out the following reaction in lab by mixing 50.0 mL of a 1.00 M solution of hydrochloric acid with 50.0 mL of a 1.00 M solution of potassium hydroxide:



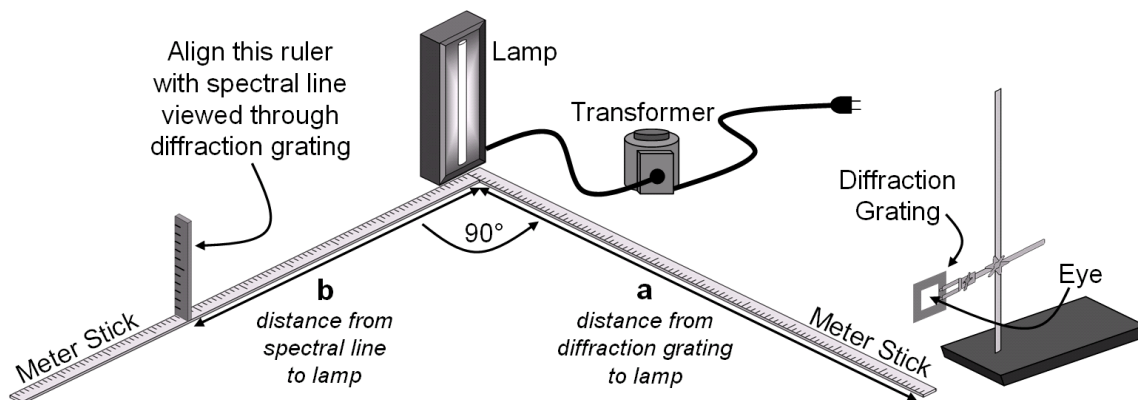
The average initial temperature of the two solutions was 25.00 °C. After mixing, the final temperature of the solution was 31.43 °C. The mass of the resulting solution was 100.2 g.

1. Determine the theoretical yield of KCl (in moles) of the student's reaction.
2. Assuming that the specific heat capacity of the solutions that the student used are equal to the specific heat capacity of pure water, calculate the heat (q_{solution}) in kJ that was absorbed by the contents of the coffee cup from the reaction.
3. Calculate the value of ΔH_{rxn} in the units kJ / mol KCl.
4. For this reaction, the theoretical value of ΔH_{rxn} is -55.8 kJ / mol KCl. What was the student's percent error when determining ΔH_{rxn} ?

Experiment #9: Emission Spectra of Hydrogen, Helium, and Mercury

According to quantum theory, electrons exist in specific energy levels. Moreover, an electron can transition from one level to another by absorbing or emitting a certain amount of energy equal to the difference between the final and initial states. When this energy takes the form of light, the frequency (ν) can be calculated using the important equation $\Delta E = h\nu$, where $h = 6.626 \times 10^{-34}$ J·s (Planck's constant).

In this experiment, you will excite electrons to higher energy levels using electricity. As electrons return to lower energy levels and emit light, you will observe various colored lines in the hydrogen spectrum, a green line in the mercury spectrum, and a yellow line in the helium spectrum. We will first measure the wavelength of the light (λ), then convert it to frequency (ν), and finally calculate ΔE .



The schematic above shows the apparatus for measuring the wavelength of light given off by hydrogen, mercury and helium. The only measurements needed to calculate λ are the distance (a) from the grating to the light source, and the distance (b) between the light source and the appearance of the spectral line. λ can then be calculated using the Bragg equation:

$$\lambda = d \sin \theta$$

where d is the distance between the lines in the diffraction grating.

Our gratings contain 600. lines per mm. You will need to convert this to cm/line. θ is the angle between looking straight at the light source and the peripheral image of the spectral line. $\sin \theta$ can be calculated in 3 steps:

1. $b/a = \tan \theta$
2. $\tan^{-1}(b/a) = \theta$
3. compute $\sin \theta$ using the sine trigonometric function on your calculator

Name: _____

Section: _____

The frequency is found using the equation $\nu = c / \lambda$, where $c = 3.00 \times 10^{10}$ cm / s (speed of light). Finally, ΔE can be calculated.

You will also be asked to predict the wavelength for various energy changes for hydrogen's electron using the Rydberg equation:

$$\frac{1}{\lambda} = 109,678 \text{ cm}^{-1} \left(\frac{1}{n_f^2} - \frac{1}{n_i^2} \right)$$

where n_i and n_f are the initial and final energy levels of the electron, respectively. Please note that this equation only works for the hydrogen atom or any other one-electron system.

Procedure

Using the apparatus previously described, set the diffraction grating one meter from the light source, distance (a). Tape the meter sticks at right angles to each other. Darken the room, and the student looking through the diffraction grating should direct a second student to move a vertical ruler along the meter stick, distance (b), until it just coincides with the spectral line. Record all your collected data in the provided data table.

Data and Calculations

Given that the diffraction grating has 600. lines / mm, determine the distance (d) between two of these lines in cm. Show your calculation and answer in the "blank space" on the next page. (Hint: It may help to think of this distance as having the units cm / line.)

Determine the energies corresponding to the colors you are instructed to observe for H, He, and Hg. Then, use the Rydberg equation to predict the energy changes for the transitions in exercises 1 – 3 and 6 – 10. Notice some of the exercises involve $n = \infty$. This means the electron has escaped from the atom, a phenomenon known as ionization; the atom losing an electron becomes a positively charged ion. Finally, determine which region of the electromagnetic spectrum (i.e. UV, Visible, or IR) corresponds with each electronic transition.

Source and Color of Line	a (cm)	b (cm)	d (cm)	Wavelength (cm)	Wavelength (nm)	Frequency (s ⁻¹)	Energy (J/photon)	Energy (kJ/mole)	Rydberg Eq calculation of λ (nm)	
1. H (red) n = 3 → n = 2										
2. H (blue) n = 4 → n = 2										
3. H (violet) n = 5 → n = 2										
4. He (yellow)									X	
5. Hg (green)									X	
6. H n = 2 → n = 1	Show calculation to determine d here:									
7. H n = ∞ → n = 1										
8. H n = 4 → n = 3										
9. H n = ∞ → n = 3										
10. H n = ∞ → n = 2										
										IR, Vis, UV
										IR, Vis, UV
										IR, Vis, UV
										IR, Vis, UV
										IR, Vis, UV

Experiment #10: Geometrical Structure of Molecules and Ions using Molecular Models

In Chemistry 1A, you are introduced to four theories involving molecular models: Lewis dot structures, Valence Shell Electron Pair Repulsion (VSEPR) Theory, Valence Bond (VB) Theory, and Molecular Orbital (MO) Theory. This experiment combines the first three theories. It gives you an opportunity to predict the Lewis dot two-dimensional structures on paper, physically build the three-dimensional models using ball and stick pieces, determine the expected angles and shapes using VSEPR Theory, and identify the hybridized orbitals and number of sigma (σ) and pi (π) bonds using VB Theory.

Lewis dot structures are two-dimensional models that use all of the valence electrons available. Covalent bonds form when nonmetals combine to share electrons, creating single, double, or triple bonds. Ions are always represented inside brackets with the overall charge indicated on the outside upper right. For ionic compounds, you separate the cation and anion, keeping both inside their own brackets. Lewis structures give you a great amount of information such as bond types, resonances, structural isomers, formal charges, and dipole arrows between atoms. Most elements follow and prefer the octet rule; exceptions include: (1) octet deficient (H, Be, B) atoms, (2) an odd number of total electrons prevent the possibility of an octet for all atoms, and (3) expanded octets possessing 10 or 12 electrons around a central atom, beginning with period three and higher elements. The BEST Lewis structures follow the octet rule and minimize formal charges.

Valence Shell Electron Pair Repulsion (VSEPR) Theory states that bonds and lone pairs are regions of high electron density in an atom that repel each other until they get as far apart as possible. This effect determines the atom's three-dimensional geometry and bond angles. Two regions will be 180° apart; three regions will be 120° apart; and four regions will be 109.5° apart. Expanded octets which are not pictured here include the trigonal bipyramidal electronic geometry that has five regions (2 axial positions 180° apart from each other and 90° from the 3 equatorial positions that are 120° apart from each other) and the octahedral electronic geometry that has six regions (all 180° and 90° apart from each other as in an x, y, z three-dimensional axis).

The Valence Bond (VB) Theory is also three-dimensional and has the same angles as predicted by VSEPR Theory. In addition, VB Theory promotes the notion that these shapes appear because the atomic orbitals have become hybridized. Hydrogen keeps its simple *s* orbital. Atoms with two regions now possess two identical *sp*-hybridized orbitals, three regions have 3 *sp²* hybrid orbitals, 4 regions become *sp³* hybridized, five regions are *sp³d* hybridized, and six regions form *sp³d²* hybrid orbitals. All bonds also have a sigma (σ) bond that directly overlaps. Multiple bonds possess sigma and pi (π) bonds that indirectly overlap perpendicular to the hybridized orbitals, where double bonds include 1 sigma and 1 pi bond, and triple bonds include 1 sigma and 2 pi bonds.

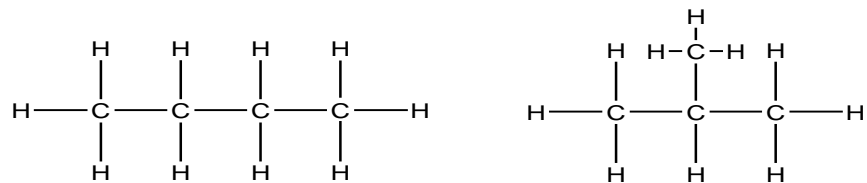
Partial VSEPR Table (look to your book or notes for expanded octets)

# of areas	# of bonds	# of lone pairs	Geometry and bond angles	Example
4	2	2	Angular or bent (~109.5°)	
4	3	1	Trigonal Pyramidal (~109.5°)	
4	4	0	Tetrahedral (109.5°)	
3	2	1	Bent (~120°)	
3	3	0	Trigonal Planar (120°)	
2	2	0	Linear (180°)	H-C≡N:
	1	any	Linear (Must have three or more atoms to form an angle.)	

Notice the convention for drawing bonds in 3-D space, where:

- the wedge () represents a bond coming out of the paper, and
- the dash () represents a bond going behind the paper.

Do not confuse resonance structures with structural isomers. Resonance structures contain atoms in the same position but electrons are delocalized throughout. Structural isomers are molecules having the same chemical formula but different connectivity of the atoms. For example, two isomers are possible for a molecule with the formula C_4H_{10} :

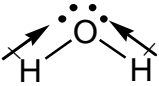
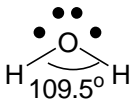


Procedure

For each of the ions or molecules listed:

- A. Draw the Lewis dot structure. For those with resonance, draw all possibilities. When requested, draw all the different structural isomers. Remember that single bonds can twist, but multiple bonds cannot.
- B. Use the model kit to build the structure.
 - The white atoms represent hydrogen and can form one bond each.
 - The black, blue, and red atoms represent atoms that desire a complete octet.
 - Use short sticks for nonbonded electron lone pairs
 - Use long sticks for single bonds.
 - Use multiple springs for double and triple bonds.
 - Use purple atoms for trigonal bipyramidal electron geometry
 - Use silver atoms for octahedral electron geometry
- C. For each central atom in the structure, determine the number of areas of electron density that lie directly on that atom. An area of electron density may be:
 - a lone (nonbonding) pair or dot that lies on the atom in question
 - a single bond
 - a double bond
 - a triple bond
- D. Fill in the empty spots on the table. Assign bond angles, formal charges, dipole arrows for polar bonds, and molecular geometries. Determine the polarity of the species, atomic and hybridized orbitals, and number of pi bonds.

Complete the following table for the indicated species:

Substance	H ₂ O	HF	O ₂	CO
<p>a) Draw the best Lewis structure(s), resonances, and structural isomers if any</p> <p>b) In your structure above, indicate polar bonds with dipole arrows toward the more electronegative atom</p> <p>c) Include formal charges if they are not zero</p>	 <p>(does NOT need to be bent at this point!)</p> <p>formal charge O = 0</p> <p>formal charge H = 0</p>			
Name the electronic geometry around central atom(s)	Tetrahedral			
Give hybridization for central atom(s)	sp^3			
Name the shape around central atom(s)	Bent (or angular)			
Show 3-D sketch of the structure and label all bond angles				
How many sigma bonds? How many pi bonds?	2 σ and 0 π bonds			
Is the substance an ionic compound, a polar molecule, a nonpolar molecule, or a polyatomic ion?	polar molecule			

Name: _____

Section: _____

Complete the following table for the indicated species:

Substance	NH_4^{+1}	Na_2S	SO_3	ClO_2^-
a) Draw the best Lewis structure(s), resonances, and structural isomers if any				
b) In your structure above, indicate polar bonds with dipole arrows toward the more electronegative atom				
c) Include formal charges if they are not zero				
Name the electronic geometry around central atom(s)				
Give hybridization for central atom(s)				
Name the shape around central atom(s)				
Show 3-D sketch of the structure and label all bond angles				
How many sigma bonds? How many pi bonds?				
Is the substance an ionic compound, a polar molecule, a nonpolar molecule, or a polyatomic ion?				

Name: _____

Section: _____

Complete the following table for the indicated species:

Substance	SO_3^{-2}	CH_2O	CO_2	SCN^-
a) Draw the best Lewis structure(s), resonances, and structural isomers if any				
b) In your structure above, indicate polar bonds with dipole arrows toward the more electronegative atom				
c) Include formal charges if they are not zero				
Name the electronic geometry around central atom(s)				
Give hybridization for central atom(s)				
Name the shape around central atom(s)				
Show 3-D sketch of the structure and label all bond angles				
How many sigma bonds? How many pi bonds?				
Is the substance an ionic compound, a polar molecule, a nonpolar molecule, or a polyatomic ion?				

Name: _____

Section: _____

Complete the following table for the indicated species:

Substance	$C_2H_2Br_2$	NF_3	CH_2Cl_2	CH_3OH
a) Draw the best Lewis structure(s), resonances, and structural isomers if any				
b) In your structure above, indicate polar bonds with dipole arrows toward the more electronegative atom				
c) Include formal charges if they are not zero				
Name the electronic geometry around central atom(s)				
Give hybridization for central atom(s)				
Name the shape around central atom(s)				
Show 3-D sketch of the structure and label all bond angles				
How many sigma bonds? How many pi bonds?				
Is the substance an ionic compound, a polar molecule, a nonpolar molecule, or a polyatomic ion?				

Name: _____

Section: _____

Complete the following table for the indicated species:

Substance	C_6H_6 (ring)	S_8	PO_4^{3-}	C_3H_8O
a) Draw the best Lewis structure(s), resonances, and structural isomers if any				
b) In your structure above, indicate polar bonds with dipole arrows toward the more electronegative atom				
c) Include formal charges if they are not zero				
Name the electronic geometry around central atom(s)				
Give hybridization for central atom(s)				
Name the shape around central atom(s)				
Show 3-D sketch of the structure and label all bond angles				
How many sigma bonds? How many pi bonds?				
Is the substance an ionic compound, a polar molecule, a nonpolar molecule, or a polyatomic ion?				

Name: _____

Section: _____

Complete the following table for the indicated species:

Substance	NO_3^-	NO_2	H_2O_2	C_2H_2
a) Draw the best Lewis structure(s), resonances, and structural isomers if any				
b) In your structure above, indicate polar bonds with dipole arrows toward the more electronegative atom				
c) Include formal charges if they are not zero				
Name the electronic geometry around central atom(s)				
Give hybridization for central atom(s)				
Name the shape around central atom(s)				
Show 3-D sketch of the structure and label all bond angles				
How many sigma bonds? How many pi bonds?				
Is the substance an ionic compound, a polar molecule, a nonpolar molecule, or a polyatomic ion?				

Complete the following table for the indicated species:

Substance	A: $C_2H_4O_2$	B: $C_2H_4O_2$	C_2H_6	BaO
<p>a) Draw the best Lewis structure(s), resonances, and structural isomers if any</p> <p>b) In your structure above, indicate polar bonds with dipole arrows toward the more electronegative atom</p> <p>c) Include formal charges if they are not zero</p>	<p>Draw one structural isomer with C–C bond that has one C connected to 3 H and the other to 2 O. This is acetic acid</p>	<p>Draw a new structural isomer keeping all formal charges = 0. More than 5 isomers are possible</p>		
Name the electronic geometry around central atom(s)				
Give hybridization for central atom(s)				
Name the shape around central atom(s)				
Show 3-D sketch of the structure and label all bond angles				
How many sigma bonds? How many pi bonds?				
Is the substance an ionic compound, a polar molecule, a nonpolar molecule, or a polyatomic ion?				

Name: _____

Section: _____

Complete the following table (the central atom for each species has an expanded octet):

Substance	I_3^-	ICl_5	SF_6	$XeOCl_2$
a) Draw the best Lewis structure(s), resonances, and structural isomers if any				
b) In your structure above, indicate polar bonds with dipole arrows toward the more electronegative atom				
c) Include formal charges if they are not zero				
Name the electronic geometry around central atom(s)				
Give hybridization for central atom(s)				
Name the shape around central atom(s)				
Show 3-D sketch of the structure and label all bond angles				
How many sigma bonds? How many pi bonds?				
Is the substance an ionic compound, a polar molecule, a nonpolar molecule, or a polyatomic ion?				

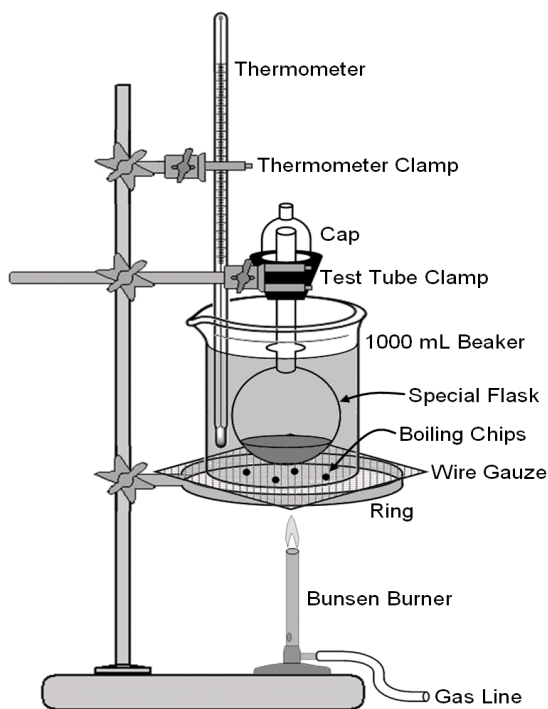
Experiment #11: Molecular Weight of an Unknown Volatile Liquid

This experiment will allow you to determine the molecular weight of a volatile liquid. The liquid will first be vaporized, then by measuring the mass, volume, temperature and pressure of this gas, you can use the Ideal Gas Law to calculate the molecular weight.

Procedure

Obtain a special flask with its stopper, cap, and plastic weighing dish. Set the flask in the plastic dish to keep it from rolling off the table. Obtain an unknown liquid assigned to you by your instructor and record its number on the data sheet.

Place the pointed rubber stopper in the clean, dry flask. Weigh on the analytical balance and record its mass. It is important to keep track of your stopper and not confuse it with a lab neighbor since they all have different masses. With a disposable pipet or medicine dropper, transfer about 5 mL of the unknown liquid into the flask. **DO NOT WEIGH THE FLASK AT THIS TIME!** You will weigh the flask after the excess liquid has vaporized). Set up the apparatus as shown in the figure below. Fill the 1000 mL beaker with about 700 mL of DI water and add 3 to 5 boiling chips. Place the flask in the water and set the cap on the neck of the flask while clamping the cap in position. The flask must not rest on the bottom of the beaker. Add water as needed so the round flask is submerged completely under water up to the narrow neck.



Name: _____

Section: _____

Heat the water to a gentle boil. Carefully watch the level of liquid inside the flask while it gradually disappears as the vapor escapes through the capillary cap. After all the liquid has disappeared, keep the flask in the boiling water for another minute. Read and record the temperature of the boiling water during this time. Shut off the burner and allow the water to stop boiling for approximately 30 seconds. Raise the flask and cap assembly from the water and quickly replace the cap with the original stopper into the neck of the flask.

Remove the flask from the hot water and immerse it into a tub of cool water or hold it under cold water from the tap. Notice that the volatile gas that had filled the flask now condenses back to liquid. After cooling the flask about 4 – 5 minutes, momentarily remove the stopper to allow air to enter the flask. Thoroughly dry the outside of the flask and again loosen the stopper a moment to equalize the pressure. Weigh and record the mass of the flask and its contents.

Refill the flask with approximately 5 mL more of your liquid and repeat this procedure. It is not necessary to clean the flask or to pour out the liquid in the flask before you start. If you suspect water has entered the flask, consult your instructor.

Record the atmospheric pressure in the room. A barometer is an instrument that is filled with mercury in an upside-down tube, above which is a vacuum and below a pool of mercury. To correctly read the pressure from the barometer in the room, first look at the bottom of the instrument and adjust the mercury level to just touch the triangular metal point. Next, while at eye level, adjust the top of the barometer so that the slider stops at the top of the meniscus. You may need to use a step stool to maintain eye level. Notice for mercury the meniscus is convex, the opposite of water which is concave. Read the barometer in millimeters of mercury on the right side (left side is inches of Hg). The atmospheric pressure is generally between 730 to 760 mm Hg (or torr) in our area. A common mistake is to miss the 700 mm Hg value since the instrument leaves out the 100's place between the intervals so the other numbers are clear to read.

Notes: The volume of the flask is written or etched on the front of the flask.

Upon completion of the experiment, return all the equipment.

Do not rinse the flask.

Remove the boiling chips from the 1000 mL beaker.

Do not dump the boiling chips into the sink.

******DO NOT PUT WATER IN THE FLASK AT ANY TIME******

Name: _____

Section: _____

Data and Calculations

Unknown Number _____

Atmospheric pressure _____ torr = _____ atm

	Trial 1		Trial 2
1. Mass of dry flask with stopper	_____	=	_____
2. Mass of flask, stopper, & <u>condensed</u> liquid	_____		_____
3. Mass of <u>condensed</u> liquid	_____		_____
4. Temperature of boiling water	_____		_____
5. Volume of flask (see flask)	_____	=	_____
6. Molecular weight of sample)	_____		_____

(These values must be within 5 % of each other. If not, a third trial must be run.)

7. Average Molecular Weight* _____
*Average only those values within the limit

SHOW CALCULATIONS:

Name: _____

Section: _____

Questions (to be completed while in the laboratory)

1. Obtain the mass percent composition information of your unknown from your instructor. Solve for the empirical formula, and then using your determined molecular weight, solve for the molecular formula of your compound.

Empirical Formula _____

Molecular Formula _____

2. Determine your percent error using your experimental molecular weight and the theoretical molecular weight determined via your molecular formula.

3. Write the ideal gas law equation for molecular weight.

Name: _____

Section: _____

Post-lab Questions

4. It was found that 0.801 gram of vapor exerted a pressure of 744 torr at 100 °C when confined to a 260 mL flask. If this vapor came from a volatile liquid, what is the molecular weight of the liquid?

5. Based on this experiment:
 - A. How do you experimentally determine the temperature of the unknown when it is a gas?

 - B. How do you experimentally determine the pressure of the unknown when it is a gas?

6. What is the purpose of cooling the flask?

7. Why will air rush into the cooled flask when the stopper is removed?

Name: _____

Section: _____

8. How would each of the following procedural errors affect the calculated molecular weight in this experiment? Give your reasoning in each case.

A. All of the liquid was not vaporized when the flask was removed from the water bath.

B. The flask was not dried before the final weighing with the condensed vapor inside.

C. The flask was left open to the atmosphere while it was being cooled, and the stopper was inserted just before the final weighing.

D. The flask was removed from the bath after all of the liquid had vaporized but before the vapor had reached the temperature of the boiling water.

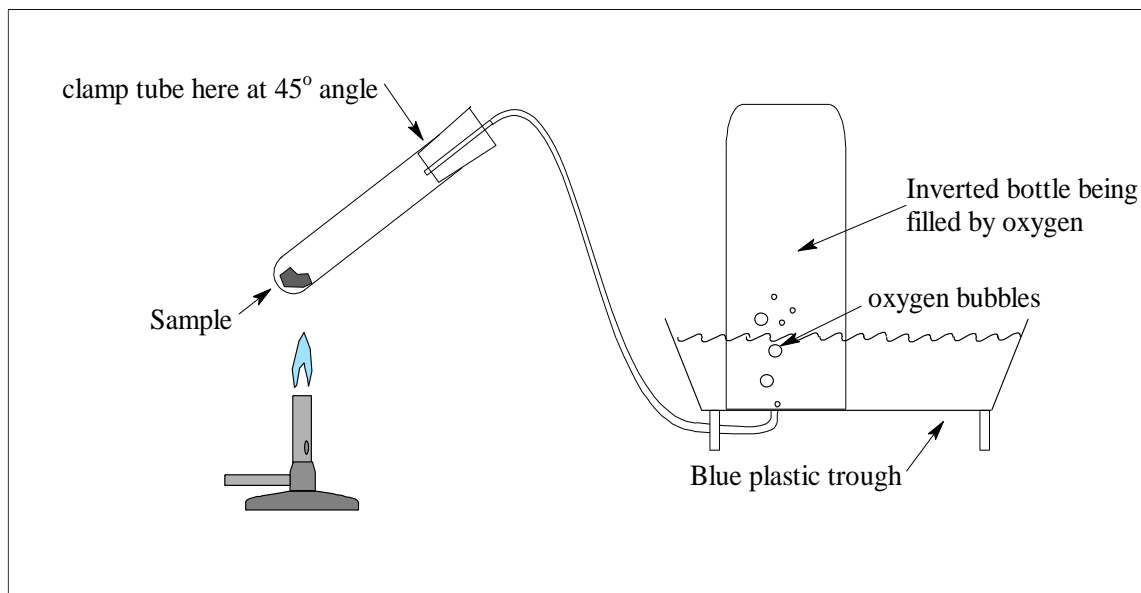
Experiment #12: Molar Volume of a Gas and Percentage of KClO_3 in an Unknown Sample

In this experiment, you will collect a gas by displacement of water and measure its volume when its pressure has been equalized with the atmospheric pressure. Then, you will calculate the molar volume of a gas. In the second part of the experiment, you will determine the percent of potassium chlorate contained in an impure sample consisting of KClO_3 , KCl , and MnO_2 catalyst.

The molar volume of a gas is simply the volume that one mole of an ideal gas occupies at one atmosphere of pressure and 0°C . Although oxygen is not an ideal gas, it will come very close to the ideal value of 22.4 L/mole.

Procedure

1. Obtain a clean, dry Pyrex test tube. Weigh and record its mass. Obtain the unknown sample assigned to you by your instructor and record its number. Weigh about 1.0 gram sample of your unknown on a weighing boat. (You need to know the exact amount, but it does not have to be exactly 1.000 g.) Transfer this sample to the test tube and record the total mass.



2. Set up the apparatus as shown above. Fill the provided wide-mouth bottle with water and slide the glass plate over its mouth. While securely holding the glass plate, invert the bottle and place its mouth under the water level in the trough. Remove the glass plate and set the mouth of the bottle over the opening in the bottom of the trough. No air should be in the bottle at this time.

3. After the set up is complete and you are certain there are no leaks, gently heat the test tube, slowly at first, then more strongly to obtain a moderate rate of evolution of oxygen gas. If white vapors appear in the test tube, stop heating until they disappear from the test tube. Continue heating until all the oxygen gas has been liberated. When the gas stops bubbling into the bottle, shut off the Bunsen burner and REMOVE THE RUBBER STOPPER FROM THE TEST TUBE IMMEDIATELY. Do this carefully as the test tube will be hot. When the test tube has cooled, weigh and record its mass.
4. Equalize the pressure of the collected gas with the atmospheric pressure by raising or lowering the bottle in the water trough to get the water level inside the bottle equal to the water level outside the bottle. When the levels are equal, securely place a glass plate over the mouth of the bottle and remove it from the water, taking care not to allow any water to leave the bottle. Invert the bottle to an upright position and remove the glass plate. Measure the volume of gas in the bottle and record this value.

(Note: If you cannot equalize the pressure in the trough, the bottle must be transferred to a larger container of water without allowing any air to enter the bottle. Place the glass plate over the mouth of the bottle while it is under water. While securely holding the glass plate over the mouth of the bottle, transfer the bottle to the larger container of water; once the mouth of the bottle is again under water, remove the glass plate and follow the procedure above to equalize the pressure.)

5. Record the temperature. Note that the oxygen gas collected should be at the same temperature as the water.
6. Finally, record the atmospheric pressure in the room. A barometer is an instrument that is filled with mercury in an upside-down tube, above which is a vacuum and below a pool of mercury. To correctly read the pressure from the barometer in the room, first look at the bottom of the instrument and adjust the mercury level to just touch the triangular metal point. Next, while at eye level, adjust the top of the barometer so that the slider stops at the top of the meniscus. You may need to use a step stool to maintain eye level. Notice for mercury the meniscus is convex, the opposite of water which is concave. Read the barometer in millimeters of mercury on the right side (left side is inches of Hg). The atmospheric pressure is generally between 730 to 760 mm Hg (or torr) in our area. A common mistake is to miss the 700 mm Hg value since the instrument leaves out the 100's place between the intervals so the other numbers are clear to read.

Name: _____

Section: _____

Data and Calculations

1. Unknown sample number _____
2. Mass of test tube: _____
3. Mass of test tube and sample *before* heating: _____
4. Mass of test tube and sample *after* heating: _____
5. Mass of sample in the tube *before* heating: _____
6. Mass of residue in test tube *after* heating: _____
7. Mass of oxygen gas released: _____
8. Volume of oxygen gas at room temperature: _____
9. Atmospheric pressure: _____
10. Vapor pressure of water: _____
11. Temperature of water: _____

Questions (to be completed while in the laboratory)

1. Calculate the pressure of the collected oxygen gas (i.e. correct for the vapor pressure of water).

2. How much volume would the gas in question #1 occupy at STP?

3. Determine the moles of oxygen gas collected from the experimental mass of the oxygen gas.

Name: _____

Section: _____

Post-lab Questions

1. A sample of an unknown metal chlorate weighing 1.725 g is heated until all of the oxygen is driven off. The residue remaining in the container weighs 0.859 g. Calculate the percentage of oxygen in this metal chlorate.

2. 340 mL of oxygen gas are collected by displacement of water at 33 °C and 742 torr, where the vapor pressure of water at this temperature is known to be 37.8 torr.
 - A. What is the pressure of the oxygen gas?

 - B. Determine the volume of the oxygen gas at STP.

Experiment #13: Classification of Chemical Substances

Most chemicals can be classified based on the type of bonding that holds the atoms together as: ionic, molecular, network-covalent, or metallic substances. Molecular compounds can be further classified as being polar or non-polar.

All of these labels are somewhat arbitrary, and some substances have properties that would place them in a borderline category somewhere intermediate between one group and another. It is useful, however, to consider some of the general characteristics of typical ionic, molecular, network, and metallic substances since many very common substances can be readily assigned to one category or another.

IONIC SUBSTANCES

Ionic substances are all solids at room temperature. They are typically crystalline but may exist as fine powders or clearly defined crystals. While most ionic substances are stable up to their melting points, some decompose upon heating. It is common for an ionic crystal to release loosely bound waters of hydration at temperatures below 200 °C. Anhydrous (or dehydrated) ionic compounds generally have high melting points, usually above 300 °C but below 1000 °C. They are not readily volatilized and boil at only very high temperatures. Typical examples include NaCl (water soluble) or CaCO₃ (which is not very water soluble).

When molten, ionic compounds conduct an electric current. In the solid state, they do not conduct electricity. The conductivity of the molten liquid is attributed to the freedom of motion of the ions, which arises when the crystal lattice is no longer present.

Ionic substances are frequently but *not always* soluble in water. The solutions produced conduct the electric current rather well. The conductivity of a solution of a slightly soluble ionic substance is often several times higher than that of the solvent water. Ionic compounds are usually not as soluble in other liquids as they are in water. For a liquid to be a good solvent for ionic compounds, it must be highly polar and contain molecules with well-defined positive and negative regions which can interact with the ions.

MOLECULAR SUBSTANCES

All gases and essentially all liquids at room temperature are molecular in nature. If the molecular weight of a substance is over about a hundred, it may be a solid at that temperature. The melting points of molecular substances are usually below 300 °C; these substances are relatively volatile, but a good many will decompose before they boil. Most molecular substances do not conduct electric current either when solid or when molten.

Organic compounds, which contain mostly carbon and hydrogen, are usually molecular in nature. If an organic compound decomposes upon heating, the residue is frequently a black, sooty material. A large number of inorganic substances are also molecular; those which are solids at room temperature include some of the binary compounds of elements in Groups IVA, VA, VIA, and VIIA.

Molecular substances are frequently soluble in at least a few organic solvents, with the solubility enhanced if the substance and the solvent are similar in molecular structure. Compounds that dissolve in solvents like xylene, toluene, or hexane are called *non-polar molecular* compounds. Compounds that dissolve in ethanol or methanol (or sometimes water) are called *polar molecular* compounds. Examples of polar organic compounds include aspirin, sugar, or vitamin C. Non-polar organic examples would include the edible triglyceride fats or the oil-soluble vitamins (*e.g.* Vitamin A)

Molecular compounds such as acids and bases are markedly polar, which tends to increase their solubility in water and other polar solvents. Such substances may ionize appreciably in water or even melt so that they become conductors of electricity. Often, the conductivity is considerably lower than that of an ionic material. Most polar molecular compounds in this category are organic, but a few, including some of the salts of the transition metals, are inorganic.

NETWORK-COVALENT SUBSTANCES

When every atom in a sample has covalent bonds whose crystal structures are more restricted by the geometrical constraints of the directional bonds, the compound is designated as a network-covalent substance. These are all solids at room temperature, possess extremely high (>1000 °C) melting points, and low volatility. They are typically very resistant to thermal decomposition, generally do not conduct electric current, and are often good insulators. Network-covalent solids are not soluble in water or any organic solvents. They are frequently chemically inert and may be used as abrasives or refractories. Examples include diamond (C), quartz (SiO₂), aluminum oxide (Al₂O₃), and tungsten carbide (W₂C).

METALLIC SUBSTANCES

The properties of metals stem mainly from the freedom of movement by their bonding electrons. Metals are good electrical conductors in the solid form with characteristic luster and malleability. Most metals are solid at room temperature and have melting points that range from below 0 °C to over 2000 °C. They are not soluble in water or organic solvents. Some metals are prepared as black powders which may not appear to be electrical conductors; if such powders are heated, the particles will coalesce to give good electrical conductivity. Examples of metals include iron, gold, or aluminum.

Procedure

In this experiment, you will investigate the properties of several substances with the purpose of determining whether they are ionic, molecular, network-covalent, or metallic. In some cases, the classification will be very straightforward. In others, you may find that the substance behaves in a way that would not clearly place it in a given category but in some intermediate group.

Determine the rough melting point range. Substances with low melting points, less than 100°C, for example, will melt readily when warmed gently in a Pyrex test tube. A test tube heated to about 300 °C will impart a yellow-orange color to the Bunsen flame. This color becomes more pronounced between 300° and 550 °C, at which temperature the Pyrex tube will begin to soften. When heating samples you should loosely cover the test tube with a metal cap. Do not breathe any vapors that are given off. Look for liquid condensing on the cooler portions of the tube and for indications that sublimation is occurring. For the higher temperature studies possible in the lab, heat the sample in a crucible with a strong Bunsen flame; a noticeable red color will appear in the crucible at about 600 °C. Do not heat samples to 600 °C unless their solubility and conductivity properties have been studied at lower temperatures with indecisive results.

Electrical conductivities of your solutions will be measured with a conductivity meter for that purpose. Distinguish between completely nonconducting, slightly conducting, and highly conducting liquids.

The substances to be studied in the first part of the experiment are on the laboratory tables along with two organic solvents, one polar and one nonpolar. Carry out enough tests on each substance to establish its classification as best you can. Report your observations on each substance, how you would classify it, and your reason for the classification.

When you have completed your tests, report to your instructor, who will check your results and issue you two unknowns for characterization. Some judgment needs to be used when determining if a compound is soluble. For example, calcium carbonate (chalk) is not soluble in water and will eventually settle to the bottom. However, if you shake some chalk in water, the water will look milky white, leading to the false conclusion that it was soluble.

1. Melting Point

- A. Place a small sample into a clean, dry test tube and loosely cover the tube with a metal cap.
- B. Heat the test tube and record observations (see previous page).
- C. If the melted sample cannot be easily removed, discard the tube. If the sample can be easily removed, discard the sample and reuse the test tube.

2. Solubility

- A. Place a very small sample into a clean, dry test tube.
- B. Add the appropriate solvent to approximately half-fill the tube.
- C. Stopper the tube and gently shake the contents. Observe and record as soluble, insoluble, or questionable.
- D. Save the aqueous solution for the conductivity test.
- E. Discard the test tube contents of the polar and nonpolar solvents into the appropriate waste container in the fume hood.

3. Conductivity - Aqueous Solution

- A. Add more solid (in small amounts) to saturate the solution. Shake contents to assure saturation.
- B. Pour the liquid into a clean, dry evaporating dish and test for electrical conductivity using the tester.
- C. Discard any remaining solid in the waste container. Clean and reuse or discard the test tubes.

4. Conductivity - Solid Sample

Use the tester directly on the solid sample in an evaporating dish. Remember to place both copper leads on the same small crystal or press down on the sample to ensure that the leads are making adequate contact.

Name: _____

Section: _____

Data and Results

Substance	Melting range (check one)	Soluble in xylene?	Soluble in ethanol?	Soluble in water?	Conducts electricity as a solid?	Conducts as an aqueous solution?	<u>Classification:</u> Metallic Ionic Molecular (P) Molecular (NP) Network-Cov.
Known A	<input type="checkbox"/> <100 °C <input type="checkbox"/> 100–300 °C <input type="checkbox"/> 300–600 °C <input type="checkbox"/> 600 °C						
Known B	<input type="checkbox"/> <100 °C <input type="checkbox"/> 100–300 °C <input type="checkbox"/> 300–600 °C <input type="checkbox"/> 600 °C						
Known C	<input type="checkbox"/> <100 °C <input type="checkbox"/> 100–300 °C <input type="checkbox"/> 300–600 °C <input type="checkbox"/> 600 °C						
Known D	<input type="checkbox"/> <100 °C <input type="checkbox"/> 100–300 °C <input type="checkbox"/> 300–600 °C <input type="checkbox"/> 600 °C						
Known E	<input type="checkbox"/> <100 °C <input type="checkbox"/> 100–300 °C <input type="checkbox"/> 300–600 °C <input type="checkbox"/> 600 °C						
Known F	<input type="checkbox"/> <100 °C <input type="checkbox"/> 100–300 °C <input type="checkbox"/> 300–600 °C <input type="checkbox"/> 600 °C						
Unknown A	<input type="checkbox"/> <100 °C <input type="checkbox"/> 100–300 °C <input type="checkbox"/> 300–600 °C <input type="checkbox"/> 600 °C						
Unknown B	<input type="checkbox"/> <100 °C <input type="checkbox"/> 100–300 °C <input type="checkbox"/> 300–600 °C <input type="checkbox"/> 600 °C						

Experiment #15: Identification and Characterization of Compounds X and Y

In this multi-week lab, each student will carry out an organic synthesis where an unknown compound X is converted into compound Y. A series of subsequent tests and characterizations will then be performed in order to deduce the structural formulas of both unknown compounds. At the end of the process, you will submit a formal laboratory report outlining the experiment, its results, and a discussion of your conclusions along with structural representations of both compounds (use of ChemSketch is optional). A rubric outlining the guidelines and expectations for the formal report is found at the end of the experimental procedure described herein.

Overview:

Part I – In this step, you will boil a white powder (unknown compound X) with hydrochloric acid to produce another white powder (unknown compound Y).

Part II – By conducting a simple flame test, the compound will be tested for the presence of chlorine.

Part III – A melting point test will be performed on compound X and two known substances.

Part IV – The solubility and pH properties of X and Y will be examined.

Part V – Using provided data from combustion analyses, the empirical formula of both compounds will be determined.

Part VI – You will titrate both compounds to determine their gram equivalent weights, or how many grams per mole of molecule are “attached” to each acidic hydrogen.

Part VII – In order to determine the molecular weight, the colligative property of freezing point depression for compounds X and Y will be examined.

Part VIII – Finally, the mass spectra (MS) and Infrared spectra (IR) of both compounds will be examined and analyzed for further structural elucidation.

PART I

1. Weigh and record the mass of a clean dry 100 mL beaker. Add about 3 grams (record the exact mass) of compound X to this beaker. Then add about 5 mL of distilled water to compound X to dissolve it completely (warm up if necessary).

Name: _____

Section: _____

2. Use the reagent pump to add 7.0 mL of 12 M (conc) HCl to the solution and cover with a small watch glass.
3. Place the beaker inside a 250 mL beaker which is about one-third full of DI water. Heat this water bath to a GENTLE boil for about 5 minutes or until a solid material forms in the smaller beaker. Keep the small watch glass on the inside beaker while boiling.
4. Cool the solution by placing the smaller beaker in a cold water bath.
5. Weigh and record the mass of a piece of filter paper. Pour your newly synthesized compound Y mixture through this filter paper so that the liquid drains into a 250 mL Erlenmeyer flask. Wash any remaining solid into the filter paper by rinsing the beaker with small amounts of cold DI water from a wash bottle. Allow the crystals to drain. Wash the precipitate 3 or 4 times with 5 mL portions of cold distilled water.
6. Transfer the crystals and filter paper to a previously weighed large watch glass. Dry the crystals by using one of the following methods: (1) place the watch glass over an appropriate sized beaker about one-third full of boiling water; or (2) place in a drying oven.
7. Once the sample is dry, weigh and record the mass of the watch glass, filter paper, and crystals. Label the crystals as compound Y and keep them in a stoppered test tube in your locker for further analysis. Do not leave the crystals in an open container in your locker, as this may damage the locking mechanism.

Mass of Compound Y: _____

PART II

1. In the fume hood, take a piece of thick copper wire and heat it in a Bunsen burner flame until its glowing. Note the flame color. Let the wire cool for 20 seconds, and dip the wire into a container with Parlon. Cover the tip in the powder, place the compound in the flame and burn it. Observe the color. Parlon contains chlorine, which burns with a distinctive color in the presence of copper.
2. Follow the same procedure using another wire to test a sample of Chemical X. This should not have any unusual color, only burning with the orange color of a typical hydrocarbon fuelled fire.
3. Follow the same procedure using another wire to test a sample of Chemical Y. Does it contain chlorine?

Parlon Flame Color: _____ Chemical X Flame Color: _____

Chemical Y Flame Color: _____

Name: _____

Section: _____

PART III

Use the mel-temp apparatus for melting point determination. To determine the melting point, place a small amount of crystals into a thin-walled capillary tube (about 0.7–1.0 cm). Place the tube into the mel-temp apparatus, turn it on, and record the temperature range at which the sample melts. You can heat quickly to start, but when you get near the melting point (10 – 20 °C), lower the rate of heating so you can get a more accurate reading. If you don't know the melting point, you can do one fast run to get an approximate reading, then do a more careful and gradual second run. Please note that you cannot rerun a sample once it has melted.

The melting point of compound Y is close to 300 °C. Do NOT attempt to take its melting point as the electronic thermometers will fail at such a high temperature.

While you are testing compound X, also test 4-aminobenzoic acid and phenacetin to calibrate your results. The Mel-temp will run three samples at the same time. Perform two separate trials for each substance. These two values should be within 3° of each other.

	<u>Trial 1</u>	<u>Trial 2</u>	<u>CRC Handbook</u>
4-Aminobenzoic Acid	_____	_____	_____
Phenacetin	_____	_____	_____
Compound X	_____	_____	_____
Compound Y	<i>(not experimentally measured)</i>		<u>~300 °C</u>

PART IV

Dissolve about 0.2 gram of compounds X and Y separately in a 100 mL beaker containing 40 mL DI water. Use these solutions for all tests in this section.

Compare the solubility of X and Y in water.

X: _____ Y: _____

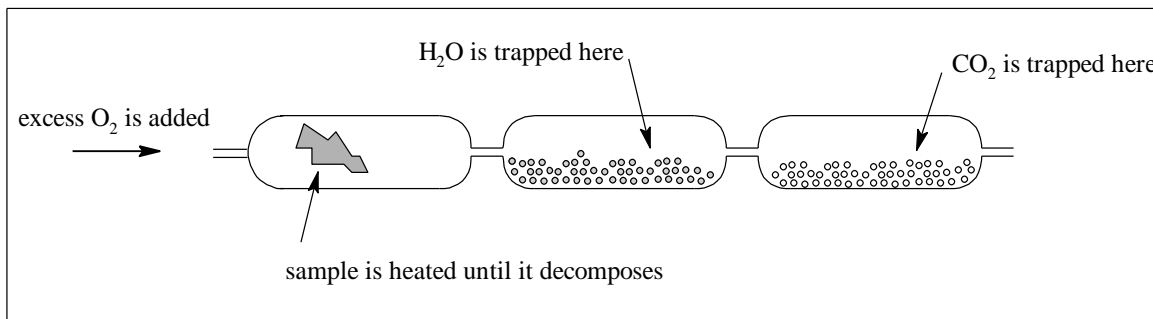
Use pH paper to determine the pH of both solutions: X: _____ and Y: _____

To each solution add a small quantity of Na₂CO₃. What happened? What class of compounds causes sodium carbonate to react this way?

X: _____ Y: _____

PART V

The empirical formula of an organic compound can be determined via combustion analysis. A typical combustion apparatus is shown below:



A sample of the chemical is burned in oxygen in a tube packed with copper oxide to ensure complete combustion. The carbon in the compound is converted to CO_2 , which is absorbed in a tube packed with ascarite. The hydrogen is converted to water vapor and is absorbed in a tube packed with drierite. The increase in weight of the absorber tubes corresponds to the CO_2 and H_2O produced during combustion. Compounds X and Y do NOT contain any nitrogen, sulfur, or halogen atoms; both compounds do contain oxygen.

A 1.0542 g sample of X was burned in a combustion apparatus. The results were:

Weight of ascarite tube before combustion	75.2382 g
Weight of ascarite tube after combustion	76.8377 g
Weight of drierite tube before combustion	81.4128 g
Weight of drierite tube after combustion	81.7418 g

Given this data, calculate the empirical formula of compound X: _____

A 1.4745 g sample of Y was burned in a combustion apparatus. The results were:

Weight of ascarite tube before combustion	80.7821 g
Weight of ascarite tube after combustion	83.0196 g
Weight of drierite tube before combustion	78.2988 g
Weight of drierite tube after combustion	78.7560 g

Given this data, calculate the empirical formula of compound Y: _____

PART VI

Before you can titrate your unknowns, you need to first determine the exact molarity of the NaOH solution you are going to use, a process known as standardization. Sodium hydroxide solutions often change concentration if they are exposed to air for long periods, and they are usually checked when first made and after a few days of use.

1. Pour about 150 mL of the approximate 0.2 M NaOH solution into a clean, dry 250 mL beaker which is labeled NaOH. Fill a clean buret with this solution.
2. Into a clean 125 mL Erlenmeyer, place about 0.5 g of KHP acid (KHP = Potassium Hydrogen Phthalate = $\text{KHC}_8\text{H}_4\text{O}_4$; MW of KHP = 204.2 g/mol). Record the exact amount of KHP in the flask.
3. Dissolve the KHP acid in your flask in about 30 mL of DI water. Add about 3 drops of phenolphthalein indicator to the flask.
4. Take the initial buret reading of the level of NaOH, and slowly add NaOH to the flask from the buret. Swirl flask continually. The solution will change from clear to light pink in exactly one drop, so watch closely. If the color disappears with swirling, then you have not reached the endpoint. A dark pink color indicates you have passed the endpoint. When you think you are close to the endpoint, begin to add the NaOH one drop at a time. Record the exact volume of NaOH solution added.
5. Repeat the titration until the molarity is consistent within 0.004 M.

Data for Part VI Standardization:

Recall that at the endpoint, moles OH^- = moles H^+ . Because each mole of KHP contains one acid group, the moles of H^+ = moles KHP.

Mass KHP acid	_____	_____	_____
Moles KHP acid	_____	_____	_____
Volume of NaOH added	_____	_____	_____
Molarity NaOH	_____	_____	_____
Average Molarity (Use values within limit)	_____	_____	_____

Name: _____

Section: _____

Now knowing that both compounds X and Y are acidic, you can titrate each using your previously standardized NaOH solution. Moreover, if you could determine the molecular weight using another technique (Part VII), you can ultimately determine how much mass of the compound is “attached” to each acidic proton. This is known as the “gram equivalent weight”.

1. To an empty 250 mL Erlenmeyer flask, add about 0.20 grams of X to the flask (record its exact mass).
2. To the flask, add about 30 mL of DI water and 3 drops of phenolphthalein indicator. Titrate the sample with standardized NaOH solution. The endpoint will be a faint pink color that persists for more than 30 seconds.
3. Repeat the titration until you get two consistent results (within 2%), and compare the mass X / mole H^+ values.

Data for Part VI Titration of Compound X

Mass X	_____	_____	_____
Volume of NaOH added	_____	_____	_____
Moles OH^-	_____	_____	_____
Moles H^+	_____	_____	_____
Grams X per mole H^+	_____	_____	_____
Average grams X per mole H^+ (Use values within limit)		_____	
Mole H^+ per mole X (Must know molecular weight from Part VII)		_____	

Name: _____

Section: _____

Repeat the titration using compound Y. Compound Y titrates slowly, and it may take longer for the pink color to persist as Y will still be dissolving during the titration. You may wish to start the titration before Y has completely dissolved. It should dissolve before you are halfway through adding the NaOH solution.

Data for Part VI Titration of Compound Y

Mass Y _____

Volume of NaOH added _____

Moles OH⁻ _____

Moles H⁺ _____

Grams Y per mole H⁺ _____

Average grams Y per mole H⁺
(Use values within limit) _____

Mole H⁺ per mole Y
(Must know molecular weight from Part VII) _____

PART VII

The freezing point of a solution is lower than that of the pure solvent. The extent of this freezing point depression depends on multiple factors including the concentration of the solution. This is the phenomenon involved when salt is spread on ice to cause it to melt. Raoult found that the depression of the freezing point of a solution is directly proportional to the molal concentration of the solution. That is,

$$\Delta T_f = i K_f m = i K_f \left(\frac{\text{mol}_{\text{solute}}}{\text{kg}_{\text{solvent}}} \right) = \left(\frac{i K_f \text{ grams}_{\text{solute}}}{\text{kg}_{\text{solvent}} \text{ MW}_{\text{solute}}} \right)$$

where ΔT_f = freezing point depression of a solvent, and $i = 1$ (*van't Hoff factor*) for compounds X and Y. K_f is the freezing point constant for the solvent. It is a characteristic only of the solvent and is independent of the solute.

$$K_f = \frac{1.86 \text{ }^\circ\text{C}}{m} = \frac{1.86 \text{ }^\circ\text{C} \cdot \text{kg}_{\text{solvent}}}{\text{mol}_{\text{solute}}}$$

Once you know the freezing point depression (ΔT_f) and the weight of solute and solvent in the solution, you can determine the molecular weight of solute.

1. Calibrate the thermometer. Place one Styrofoam cup inside another. Fill the inner cup with ice and water to fill the cup and cover with a lid. Rinse a thermometer thoroughly with DI water, and insert it into the cup through the lid. Stir the ice water and record the temperature to the nearest 0.01 °C when it becomes constant.
2. Assemble your freezing point apparatus by placing 2 – 3 scoops of rock salt in a 400 mL beaker and add about 150 mL of water. Stir this mixture with a glass stirring rod to saturate the solution and then fill the beaker with ice. Keep the glass stirring rod in this beaker and stir the ice water / salt mixture every few minutes during the experiment.
3. Weigh and record the mass of a clean, dry 200-mm test tube. Add about 1 gram of compound X (record the exact amount). Pour approximately 10 mL of DI water into the test tube, completely dissolve compound X (warm slightly if necessary), reweigh, and record the total mass. Support the test tube in a plastic beaker during these weighings.
4. Clamp the test tube to a ring stand using a utility clamp. Place a clean, dry plastic stirrer into the test tube and lower the test tube into the ice water/salt mixture. Carefully lower the thermometer into the test tube, and make certain the thermometer bulb does not touch the glass. CONSTANTLY move the plastic stirrer in a rapid manner. Continue until you observe the formation of a solid; then gently stir and read and record the freezing temperature of the compound X solution to the nearest 0.01 °C.
5. Remove the test tube and allow the solution to return to room temperature, then repeat the procedure with the same solution once more. If your molecular weight values are not within 10% of each other, do a third trial. Clean and return all equipment when done.

Name: _____

Section: _____

Data for Part VII

Mass of compound X _____

Mass of H₂O _____Trial 1Trial 2

Freezing temperature of pure water _____ = _____

Freezing temperature of solution _____

 ΔT_f _____

Molecular weight of compound X _____

Average Molecular Weight (values must be within 10%): _____

PART VIII

Chemists generally turn to instrumentation first in most research and industrial settings. In this section, you are going to use two techniques to investigate your compounds.

Mass Spectrometry (MS) is a powerful technique that gives you the molecular weight of most compounds quickly and accurately. It is easy enough to employ that it is used for multiple purposes, for example, to scan luggage for explosive residues in airports. Analysis of the small fragments and exact patterns of the mass spectra is difficult, but finding the molecular weight of the compound is usually trivial. A mass spectrum features various mass-to-charge ratios of charged particles, where unknown compounds are identified by their fragmentation patterns. For this lab, you are looking for the *molecular (or parent) ion* which corresponds to the unknown compound's molecular mass. It is important to note (as you will see) that the molecular ion can be weak with a small height on the actual spectrum; don't assume that the largest peak present in your spectrum necessarily corresponds to the molecular ion peak. Consider the following: does your average molecular weight value from Part VII correlate with any peaks in your mass spectra in Part VIII?

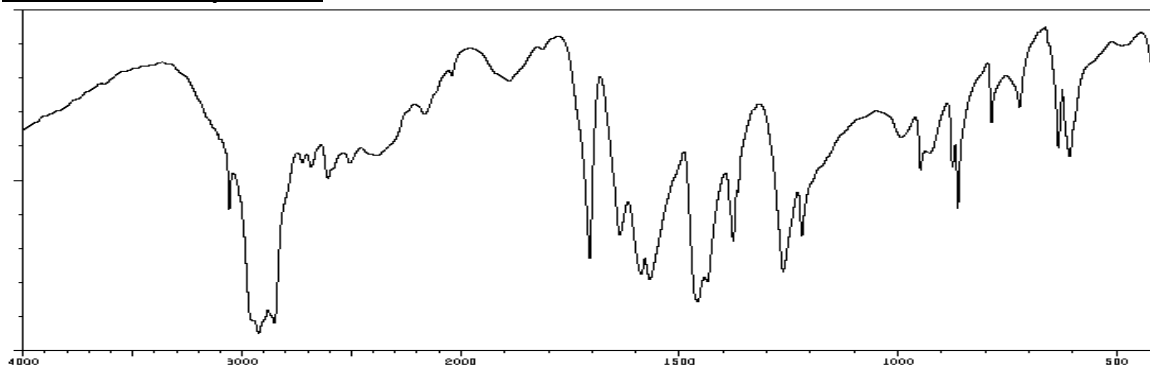
Infrared Spectroscopy (IR), which is more difficult to interpret, gives you information about which type of functional groups are present in a molecule. By comparing peaks to known values, you can determine if your molecule has those types of functional groups.

On the next page are the MS and IR for both compounds. Your instructor will give you guidance about the level of interpretation that is expected. You can find databases of spectra from the SDBS (spectral data base system from Japan's AIST) online to compare these spectra to.

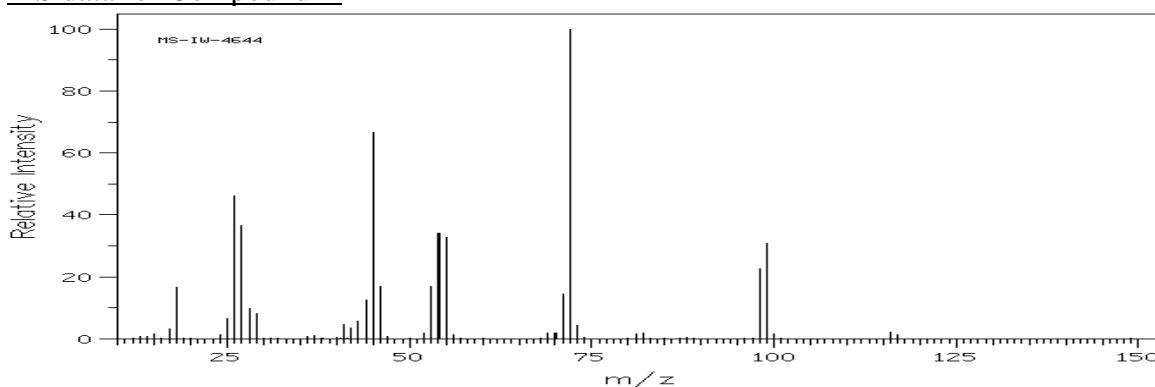
Name: _____

Section: _____

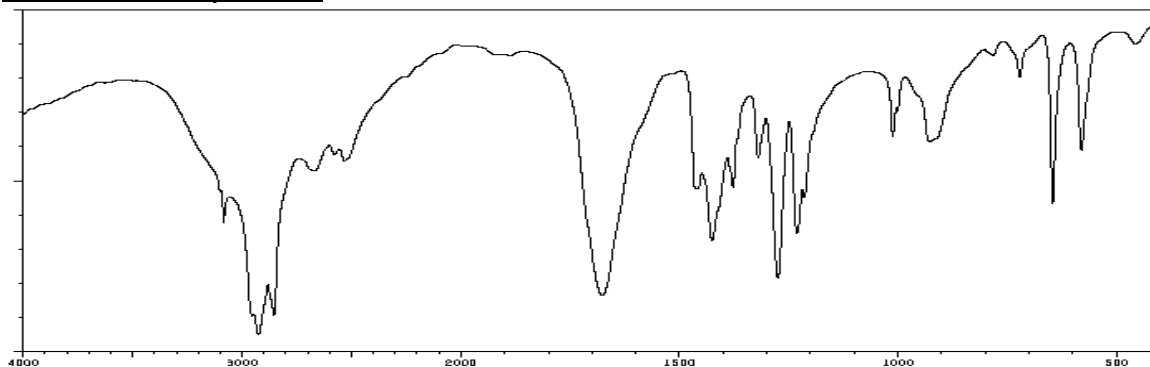
IR data for Compound X



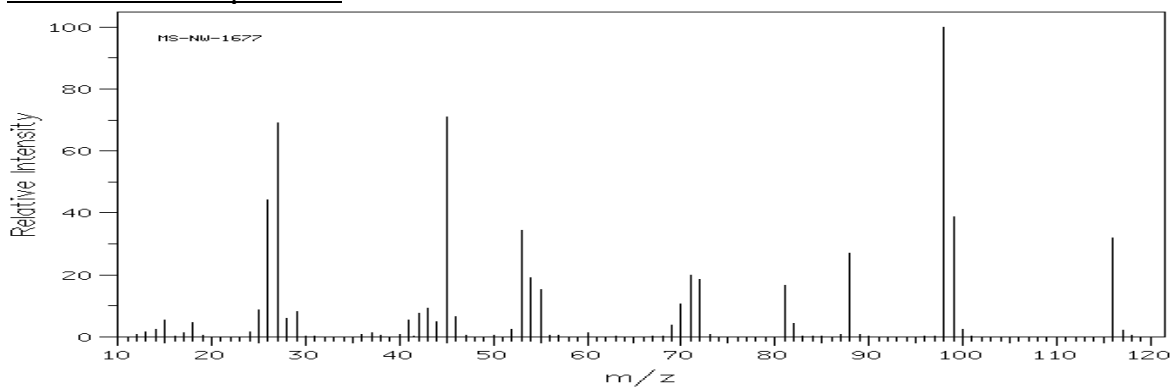
MS data for Compound X



IR data for Compound Y

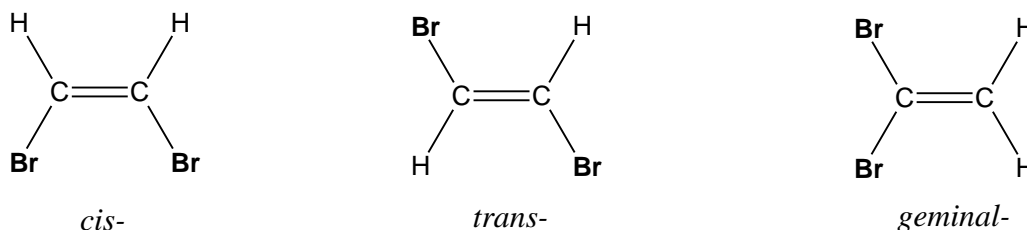


MS data for Compound Y



ANALYSIS:

Are the molecular weights of X and Y the same? Are the empirical formulas of X and Y the same? How can two compounds have the same formula but be different? A clue to this problem can come from looking at similar compounds called geometric isomers, or compounds that have the same chemical formula and basic “layout” but possess different shapes. For example, dibromoethene comes in one of three isomers: *cis*-, *trans*-, and *geminal*- as shown below:



The physical properties of all three isomers are different. Therefore, use your data from the various portions of this experiment to deduce structural representations for both compounds X and Y. Was the compound acidic? How can molecules made up of C, H and O become acidic? Recall that most organic acids have the $\text{-CO}_2\text{H}$ group present. Moreover, as another point of consideration, examine the data acquired in Part VI. The gram equivalent weights of X and Y tell you the weight of atoms (in grams/mole) that are associated with each acidic hydrogen. Using the compound's molecular weight, its empirical formula, and knowing what functional groups are present, you should be able to deduce a few plausible choices.

To determine the EXACT nature of X and Y, you need to consult a reference text (such as the *CRC Handbook* or *Merck Index*) and/or a reputable website such as the Aldrich Chemical site or Wikipedia. Compare the properties and melting points of the various compounds you find, and try to match X and Y to their real identities. Don't forget to write down specific information as to where you obtained any literature information. This should include (but is not limited to) author or editor, title of source, edition where applicable, year, and page numbers.

Your final report should contain the technical and common names along with complete structural representations (including all bonds, respective angles, hybrid orbitals invoked by all central atoms, and total number of σ and π molecular orbitals present) of compounds X and Y. This formal, typed report must adhere to the guidelines (rubric) found on the next two pages. You can neatly draw and label the molecules “by hand”; in addition, you can download ACD/ChemSketch 11.0 Freeware onto a personal computer. This free software is a chemically intelligent drawing tool that works well with Microsoft Word[®] and can be readily downloaded at: <http://www.acdlabs.com/>.

Moorpark College Chemistry Department Laboratory Report Rubric

Total: _____

CATEGORY	4 – Accomplished	3 – Good	2 – Developing	1 – Beginning	0 – Substandard	Score
Abstract	Clear, concise (~½ page), and thorough summary of results with appropriate literature references.	Refers to most of the major results; some minor details are missing or not clearly stated.	Misses one or more major aspects of the results.	Missing several major aspects of the results and merely repeats information from the	None, unrelated, or plagiarized.	_____ ×2
Introduction	A cohesive, well-written summary (including relevant reaction chemistry) of the background material pertinent to the experiment with appropriate literature references (at least one scientific reference if required by your instructor) and a statement of purpose.	Introduction is nearly complete but does not provide context for minor points. Contains relevant information but fails to provide background for one part of the experiment, or certain information is not cohesive.	Certain major introductory points are missing (e.g., background, theory, reaction chemistry), or explanations are unclear and confusing. References are not scholarly.	Very little background information is provided, and information is incorrect. No references are provided.	None, unrelated, or plagiarized.	_____ ×2
Methods & Materials	Contains a complete listing of safety information, a narrative of experimental procedures followed, and materials used. Omits information that can be assumed by peers. Includes observations when appropriate and only important experimental details.	Narrative includes most important experimental details. Missing one or more relevant pieces of safety information or experimental procedure.	Narrative is missing several experimental details and safety information or includes insignificant procedural details.	Several important experimental details and safety information are missing. Procedural steps are incorrect, illogical, or occasionally copied directly from the laboratory manual.	None, unrelated, or plagiarized (including completely copied from the laboratory manual).	_____ _____
Results & Calculations	All figures, graphs, and tables are numbered with appropriate titles and captions. Sample calculations are shown and correctly solved. All data is explicitly mentioned in the text.	All figures, graphs, and tables are correctly drawn, but some have minor problems or could still be improved. All data and sample calculations are mentioned in the text.	Most figures, graphs, and tables are included, but some important or required features are missing. Certain data and sample calculations are not explained in the text and/or solved incorrectly.	Figures, graphs, and tables are poorly constructed, have missing titles, captions or numbers. Certain data and sample calculations are not referenced in the text and solved incorrectly.	None, unrelated, or plagiarized.	_____ ×2

CATEGORY	4 – Accomplished	3 – Good	2 – Developing	1 – Beginning	0 – Substandard	Score
Discussion & Conclusion	Demonstrates a logical, coherent working knowledge and understanding of important experimental concepts, forms appropriate conclusions based on interpretations of results and/or spectrum (spectra) analysis, addresses any post-lab questions in paragraph format, includes applications of and improvements in the experiment, refers to the literature when appropriate, and demonstrates accountability by providing	Demonstrates an understanding of the majority of important experimental concepts, forms conclusions based on results and/or spectra (spectrum) analysis but either lacks proper interpretation, does not answer post-lab questions in paragraph format, suggests inappropriate improvements in the experiment, refers to the literature insufficiently, or lacks overall	While some of the results have been correctly interpreted and discussed, partial understanding of results is still evident. Student fails to make one or two connections to underlying theory.	Does not demonstrate an understanding of the important experimental concepts, forms inaccurate conclusions, does not answer post-lab questions in paragraph format, suggests inappropriate improvements in the experiment, refers to the literature insufficiently, and lacks overall	None, unrelated, insignificant error analysis and incorrect explanation, or plagiarized.	_____ ×2
References* (see example)	All sources (information and graphics) are accurately documented in ACS format. At least one reference is taken from primary scientific literature relevant to the report if required by instructor.	All sources are accurately documented, but a few are not in ACS format. Some sources are not accurately documented.	All sources are accurately documented, but many are not in ACS format. Most sources are not directly cited in the text.	All sources are accurately documented but not directly cited in the text.	Sources are not documented nor directly cited in the text.	_____ ×2
Miscellaneous (check all that apply) <input type="checkbox"/> Mechanics, grammar, & appearance <input type="checkbox"/> Appendix <input type="checkbox"/> Lab Notebook <input type="checkbox"/> VSEPR & Valence Bond drawings	Grammar and spelling are correct. All required components are included, complete, and/or illustrated correctly. Paper is not written in first person. Includes ChemSketch image(s) if required by instructor. See lab manual for more details.	Less than three grammatical and spelling errors are present. Missing one required component or features an improperly labeled molecular representation.	More than three grammatical and spelling errors are present or paper is written in first person. Features multiple errors with labeled molecular representation.	Frequent grammatical and spelling errors, and writing style lacks cohesion and fluidity. Paper is written in first person. Labeled molecule contains multiple errors.	None, unrelated, or plagiarized.	_____ ×2

*Journal citations include author or editor, *title (in italics)* followed by a period, **year (boldface)**, *volume (in italics)*, and page numbers. Example: Schrauzer, G.N.; Windgassen, R.J. *J. Am. Chem. Soc.* **1966**, *99*, 3738–3743. For more examples, see summary of ACS Style Guide (found online)