

College of Western Idaho General Chemistry First  
Semester Lab Manual: CHEM 111L



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Semester Lab Manual: CHEM  
IIIL

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# Introduction

This lab manual was designed as an inclusive lab experience covering scope and sequence for the first semester general chemistry course at the College of Western Idaho. It includes hands on activities and prompts writing and data processing associated with varied general chemistry topics following an atom's first approach and including measurement, light and energy, stoichiometry, solubility and activity, calorimetry, and gas behavior. The manual also includes a calculation and statistical analysis appendix, sample reports, and a guide to writing reports. Each lab in the sequence is organized with pre-lab questions, a safety overview, an historical and chemical background and lab instructions as well as lab reporting instructions.





# I. Exp. 1: Exploration of Matter Through Density Determination

## Pre-Lab

Use BB to quiz on the following before the lab: Define the following:

1. Density:
2. Specific gravity:
3. Buoyancy:
4. RSD (Relative Standard Deviation):
5. The density of a cardboard box is 43 pounds/cubic foot (lb./ft<sup>3</sup>). What is the density in the metric units of g/cm<sup>3</sup>?
6. What volume of liquid, expressed in mL, would a 16lb bowling

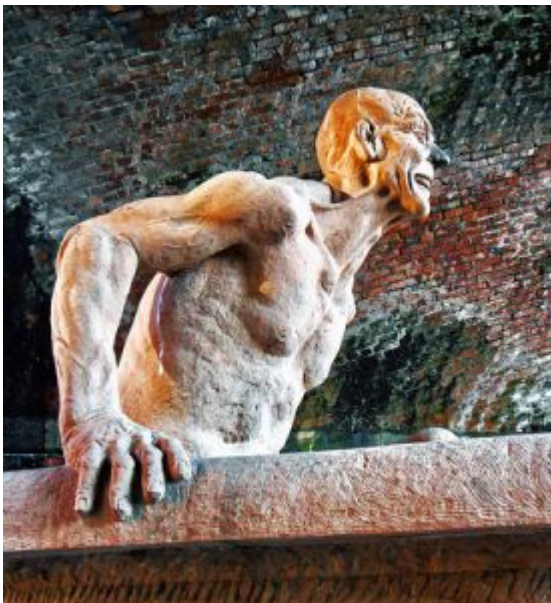
ball displace (diameter = 8.5 in)? The following information may be useful for you:

1 in = 2.54 cm, 16 oz. = 1 lb., 28.3 g = 1 oz., volume of a sphere =  $\frac{4}{3}\pi r^3$

7. A cylinder of lead weighs 1095 grams. If the density of lead is 11.4 g/cm<sup>3</sup>, what is the volume of the lead?
8. A cylinder of lead has a diameter of 0.635 cm and a length of 5.08 cm. What is the mass of the lead?
9. If a piece of aluminum foil weighs 4.08 grams, the length of the piece of foil is 10.0 cm, and the width of the piece of foil is 93.5 cm, what is the thickness of the foil in cm?

Al = 2.70 g/cm<sup>3</sup>

10. Explain the safety concerns of this lab and how to avoid being injured.



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## Introduction

Density, specific gravity, and buoyancy are related, but not identical. Steel, for example, has a density of between 7.75 g/mL and 8.05 g/mL while water has a density of very nearly 1 g/mL.

$$\rho = m/v \quad \text{Equation 1}$$

Steel sinks in water. A large steel ship, such as an aircraft carrier weighing several tons, floats on water, however. The mass of water displaced by the hull of the ship must be greater than the mass of the ship. The air-filled ship is buoyant. The Greek mathematician and physicist, Archimedes, discovered this principle, called the Archimedes principle, in present day Sicily. King Hiero II, a friend to Archimedes, commissioned a gold crown, and gave a precise mass of gold to a smith for the project. The finished crown was returned, weighing identically to the mass of the gold that was provided. King Hiero, however, suspected that silver had been alloyed with the gold, and that he had been cheated. Archimedes was tasked

with verifying the composition of the crown without destroying it. Puzzling over his task, Archimedes climbed into his filled bath, displacing his exact volume in water. The solution was apparent to him; Archimedes had solved the king's dilemma, and without pausing to put on a towel, ran through the streets crying, 'Eureka,' or I have found it! Archimedes then displaced and measured a certain volume of water with a mass of gold equal to that of the crown. When the process was repeated with the crown, it displaced more water. The smith had replaced some of the gold with silver or some other less valuable and less dense metal.

**SPECIFIC GRAVITY** – Specific gravity is related to both density and buoyancy. Specific gravity is the ratio of the density of a material at a specified temperature compared to that of water at the specified temperature. Specific gravity is a unitless term since it is a ratio of densities. Specific gravity is frequently given with temperatures added. One temperature will give the temperature at which the density of the material was measured (the top temperature) and the second temperature (the bottom temperature) will give the temperature at which the density of water was measured.

#### Objective

The objective of lab one is to use careful measurements to calculate the density of various liquids, to use ancillary measurements to calculate the thickness of aluminum foil, and to use density to discover the percent composition of an alloy.

#### Safety

- Wear goggles. Assume that any unknown liquid is caustic.
- Check for broken glassware.
- Use care to slide your brass alloy into your graduated cylinder, rather than dropping it.
- Read and understand the lab before attending and ask questions in lab. It is better to ask before than fix after.

#### Waste/Housekeeping

- Dispose of water and 'sea water' in the sink
- Place your alloy on a paper towel in the back of the room and leave to dry

## Procedure

### **Day 1:** Density Determination of a Liquid

1. Record the mass of your smallest clean, dry beaker in the data table below under Trial 1\*.
2. Pipette 5 mL of distilled water into the beaker using a volumetric pipette.

-Note the error printed on the bulb end of the pipette; it will inform your significant figures.

3. Measure the temperature and mass of the water. Record your data under the Trial 1 heading.
4. Perform a second density mass and volume measurement as Trial 2.
5. Determine the average density of water based on your two trials.

\* Remember, these tables are **only examples**, and must be reproduced in your informal lab report. One of the reasons the lab process exists is to help you learn to more effectively communicate information. One of the ways to communicate information is to record everything in a logical order with adequate labels. Start with a plan of what you will do with your recorded data and then record your data in an organized manner that makes sense to you. A question about how or why you will use a measurement will be better received than a question asking where to write a certain value. How and where you record data is at your discretion.

<b>Data Table 1: Water</b>	<b>Trial 1</b>	<b>Trial 2</b>
Mass of beaker (g)		
Tared mass of water (g)		
Volume of water (mL)		
Density of water (g mL <sup>-1</sup> )		
Average density of water (g mL <sup>-1</sup> )		
Laboratory temperature (°C)		

$$\% \text{ error} = \frac{|(\text{experimental value}) - (\text{accepted value})|}{\text{accepted value}} \times 100$$

**Equation 2**

Percent error communicates how much your experimental results differ from an accepted literature value. \*Statistical equations are provided for your first lab. In future labs, please refer to your appendix.

Using Table 1, calculate the percent error in your water density measurement. Be sure to show your work on all calculations for credit.

Table 1: Density of water at Various Temperatures	
Temperature °C	Water Density (g mL <sup>-1</sup> )
18	0.998595
19	0.998405
20	0.998203
21	0.997992
22	0.997770
23	0.997538
24	0.997296
25	0.997044
26	0.996783
27	0.996512
28	0.996232

%error, density of water

-----

Repeat the density analysis for seawater.

Data Table 2: Seawater	Trial 1	Trial 2
Mass of beaker (g)		
Tared mass of water (g)		
Volume of water (mL)		
Density of water (g mL <sup>-1</sup> )		
Average density seawater (g mL <sup>-1</sup> )		

The density and salinity of seawater varies with temperature. In this case, rather than using a percent error to analyze your results, you will calculate a percent difference between trials one and two. Percent difference communicates how precise two measurements

are, or how they agree with each other when neither can be considered the true or correct value. The percent difference is the absolute value of the difference of the experimental values over the mean times 100. Calculate the percent difference between your seawater samples. Be sure to show your work for credit.

$$\% \text{ difference} = |(E1) - (E2)| / 1/2 (E1) + (E2) \times 100$$

**Equation 3**

%	difference	seawater
---	------------	----------

Now that you have densities for D.I. water and seawater, find the specific gravity of seawater and include it in your report. Use the tabulated data for the density of water at a given temperature listed in Table 1.

Specific	gravity	seawater
----------	---------	----------

Day 2:

**Thickness of Aluminum Foil**

Obtain a piece of aluminum foil. The size, or area is up to you, but both your mass and area error will increase as size decreases. A sheet approximately the size of a sheet of notebook paper may work well. Given that the density of aluminum is 2.73 g/cm<sup>3</sup>, determine the thickness of the aluminum foil. Develop your own procedure. The following **example** data table may be helpful.

Data table 3: Aluminum Thickness			
	Length (cm)	Width (cm)	Mass (g)    Thickness (cm)
Trial 1			
Trial 2			



Thickness of Aluminum Foil:

-----

Include a percent error **or** a percent difference of your findings. Choose the most appropriate **one**. Please refer to your appendix for clarification on the use of statistical methods.

% error **or** % difference:

-----

--

### Density of an Alloy:

Now that you are well versed in using density for simple measurements, you will determine the percent composition of an alloy. Your alloy is a custom brass alloy, composed of copper and zinc. Any trace metals occur at small ratios and can be ignored. Some hints to keep in mind as you perform your analysis:

- Smaller graduated cylinders usually have more graduations, reducing volume measurement error and increasing significant figures
- If you weigh your brass sample before you wet it, you will reduce mass error
- You will need to dry your sample between trials and re-weigh.
- Increasing your displacement of water or  $\Delta V$  will decrease volume error

Come up with a sound strategy for finding the density of your custom brass sample. Points will be awarded for obtaining the correct percent composition. The following **example** data table may be helpful. Your brass sample may be a cylinder or pellets, and differing glassware may be appropriate.

---

**Data Table 4: Percent Composition of an Alloy**

	Trial 1	Trial 2	Trial 3	Trial 4 (if needed)
Total mass brass (g)				
Initial volume water (mL)				
Final volume water (mL)				
Volume brass (mL)				
Density brass (g/mL)				
Average density brass				

---

The **density** measurements for trials one and two and three should agree within **+/-0.5 g mL<sup>-1</sup>**. If the trials differ by more than this value, repeat your calculations a fourth time. You may choose to throw out the offending density measurement at this point.

Relative standard deviation, RSD, can inform you of the precision of your measurements. In this case you will not be using standard deviation from excel, although you may check your work using the program. You will calculate it the long way, showing all parts of the calculation.

**Formula does not parse**

**Equation 4**

**Formula does not parse**

**Equation 5**

Standard deviation =  $s$

$x_i$  = each individual measurement

$\bar{x}$  = mean (average) of the measurements

$n$  = the number of measurements

%RSD of your three or four measurements

---

Now that you have measured and calculated a reliable density in your brass sample, you are prepared to calculate the percent composition between copper and zinc. The mathematical operation can be expressed as follows:

$$x (\text{copper density}) + (1-x) (\text{zinc density}) = \text{average density of brass}$$

#### Equation 4

where

$x$  = the fraction of the alloy composed of copper

$1-x$  = the fraction composed of all other metals (zinc)

The densities of some coinage metals are found in table 3.

**Table 2: Density and Cost of Coinage Metals**

Metal	Density (g mL <sup>-1</sup> )	Cost (\$ g <sup>-1</sup> )
Zinc	7.13	0.00201
Copper	8.96	0.00591
Nickel	8.92	0.0129
Silver	10.5	0.570
Gold	19.3	55.68

<https://www.moneymetals.com/precious-metals-charts/gold-price> accessed 06/18/20

Composition of alloy = \_\_\_\_\_% copper  
 \_\_\_\_\_% zinc

Post Lab:

1. What would happen to your density calculation if you measured the volume of seawater with your burette, but class ended, and you kept the sample uncovered in your drawer to measure the mass the following week in lab?
2. Assuming your brass samples are identical in composition, or percent copper and zinc, how would your density

measurements compare if you used differently sized brass samples during different trials? Be specific in your descriptions of density and its constituents, mass and volume.

3. You have a chunk of material about the size of a marble that appears to be antique ivory. Describe in detail how you would determine the mass and the volume so that you could calculate the density?
4. Considering the data in Table 3, would equal masses of gold and copper result in the same volume change if added to water? Please explain.
5. What are the respective purposes for using %RSD and percent error? Name an instance from any discipline or application where **each** is applicable.

#### Your Report:

1. Complete the pre-lab and take the pre-lab quiz in your Blackboard shell before attending lab.
2. Use a lab notebook to record observations and make calculations.
3. Create an informal lab document to turn in at the beginning of the next lab. Your lab report should include clearly labeled and tabulated or graphed raw data, any formulas or equations, unknown number, or letter (if applicable), conclusive data such as density, % error, etc. with clear example calculations or sample calculations\*. Discuss results relevant to your findings. Think about the main point of the lab, or the results that you

worked for and be sure to include it or them. Refer to *How to Write an Informal Lab Report* as you draft your report.

4. Include numbered responses to any post lab questions. You do not need to re-write the questions but will use complete sentences or a short paragraph as appropriate. Be specific. If you describe an error, for example, you must describe the direction the data would skew and why.
5. Submit your document **before** your next lab appointment under the assignment tab on your laboratory Blackboard shell.

#### References:

Davila, R. In *Lab 3: Density* College of Southern Idaho; College of Southern Idaho; 2020.

Dieckmann, G., Sibert, J. In *An Atoms First Approach to the General Chemistry Laboratory*; McGraw Hill: New York, NY, 2014; pp 3–12.

Ross, R. Eureka! The Archimedes Principle.  
<https://www.livescience.com/58839-archimedes-principle.html>  
(accessed Jun 29, 2020).

## 2. Exp. 2: Light and Nanotechnology; How do we 'See' Something too Small to See?

Pre-Lab; may be included on BB pre-quiz:

Read the lab experiment and answer the following questions prior to coming to lab. Note that the last question will require a spreadsheet file.

1. Define spectrophotometry, absorbance, cuvette, and wavelength.
2. Find the molarity of a 1.00 L sample that contains 47.6 g NaCl.
1. Find the wavelength (in nanometers) of light with a frequency of  $4.47 \times 10^{14} \text{ s}^{-1}$ .
4. What is the amount of energy in one photon of light emitted by the red laser (assume 670 nm)?
1. Graph the following data using excel®. Enter concentration

units into the A column, as the independent variable. Excel will assign these values to the x axis of a scatter plot. Enter absorbance into the B column. Excel will assign these values to the y axis of a scatter plot. In excel, choose *insert, scatter plot* (no line) Add the equation of the line (right click on a data point). Use the linear equation to solve for the concentration (x) of a sample whose absorbance (y) is 0.231. See your lab appendix for excel help.

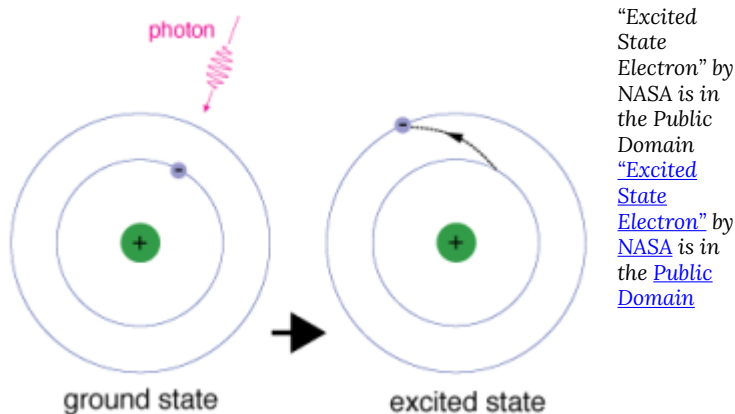
Absorbance (nm)	Concentration (M)
0.050	0.100
0.104	0.200
0.198	0.400
0.301	

6. Explain the safety concerns in this lab and how to avoid getting injured.
  
7. In using the Vernier spectrophotometer,
  1. Why must you always use the same cuvette for your sample?
  2. Why was it important to wipe your fingerprints off the cuvette or shake bubbles out of the cuvette before measuring with it?

## Pre-Introduction

A mole of particles is defined as  $6.022 \times 10^{23}$ . Molarity (M) is a concentration unit defined as the number of moles solute per liter solution and describes the concentration of a solution. For example, a 1.0 M aqueous solution of NaCl contains one mole of NaCl, or  $6.022 \times 10^{23}$  formula units of NaCl for each liter of the solution. Molarity will be one measure of concentration used later in your lecture course. If a 1.00 L solution contained 65 g NaCl, the mass NaCl would first be converted to moles  $65\text{g}/58.44\text{g/mol} = 1.11\text{mol}$  NaCl. The moles NaCl would then be divided by the liter's solution (1.00) to yield a 1.11 M solution.

## Introduction



Nanotechnology is both a very old and new discipline of study. The antimicrobial properties of colloidal (nano) silver were known to both the ancient Greeks and Babylonians. Nano-sized conglomerations of metals have long been used in creating stained glass. Red, for example, is tinted by gold aggregating at the nanoscale and yellow created with silver particles. More recently



carbon nanotubes, valued for both strength and lightness, have been introduced to bicycle frames and tennis rackets, among other applications. NASA has used silver nanotechnology in water purification to be used in space. Modern sunscreens use nano-sized molecules to absorb certain wavelengths of 'light.' Biomedical applications of nanoscience are some of the most rapidly progressing fields of nanoscience.

Nano science refers to the application of materials with a dimension of 100 nm or less, with nano referring to a billionth of a meter, much too small to evaluate with standard measurement techniques. In this lab, you will indirectly measure the size of nano sized silver aggregations as well as the width of human hair using interaction with light. Although the width of hair is larger than nanoscale, it is too small to use standard measurement techniques. Some background and theory will facilitate your lab work.

Spectrophotometry is essentially the measurement ("metry") of light particles ("photo") coming from the electromagnetic spectrum ("spectro"). We are dealing with moving electrons around in the outer parts of an atom or a molecule. When these electrons move from one radius or distance from the nucleus to another there is a certain quantity of energy involved (Figure 1). This particular quantity of energy is what we are measuring.

You are going to be looking at how much energy is involved in moving an electron from one place to another in a real atom. There are several relationships we need to look at next:

$$E = h \nu$$

**Equation 1**

$E$  = energy                       $h$  = Planck's constant                       $\nu$   
 = frequency  
 Where  $h = 6.626 \times 10^{-34} \text{ J}\cdot\text{s}$

(The frequency is how many waves of light pass a given point in one second).

$$c = \lambda \nu \quad \text{or} \quad \nu =$$

### Equation 2

Where  $c$  is the speed of light and  $\lambda$  is the length of one wave of the light, or wavelength. If we combine these two, we get . . .

$$E =$$

### Equation 3

Where  $c = 2.998 \times 10^8 \text{ m s}^{-1}$

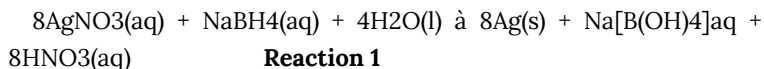
What is of most importance is that energy is directly related to the wavelength of light. If we monitor the wavelength of light, we can know something about the energy involved, and even learn about the path that an electron takes. Specific amounts of energy have specific wavelengths associated with them.

The narrow region of 'light' in the electromagnetic spectrum that we can see, ~400 to 800nm, is called the visible spectrum. Some birds, crustaceans, and insects, however, can see beyond our visible spectrum to a second violet.

A continuous visible spectrum is detected as white light, and an absence of energy in the wavelengths of the visible spectrum is detected as black, or absence of light. Light, as a wave, can be influenced, or reflected, refracted, or absorbed. If a particle or molecule absorbs white light at 620 nm, or orange, the eye will see the remaining reflected wavelengths as white minus orange, or orange's complimentary color, blue. From this absorption, the size of particles can be inferred.

### Silver Nanoparticles:

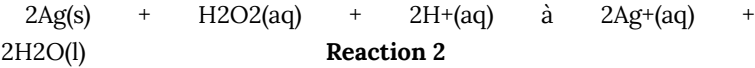
The color visible in solutions of silver nanoparticles depends on the size and shape of the particles present. In this lab you will perform a precise and sequential series of reactions which will reduce  $\text{Ag}^+$  ions to  $\text{Ag(s)}$ . Sodium borohydride ( $\text{NaBH}_4(\text{aq})$ ) is used as the reducing agent according to the following reaction.



### Reaction 1

The silver atoms then begin to aggregate, which will continue until the particles are coated with a charges species that inhibits

further aggregation. Hydrogen peroxide oxidizes, or removes electrons from the atom, quenching the aggregation as follows.



The borohydride and peroxide cooperate to form the appropriate size nanoparticle to produce visible color and a signature absorption. Bromide is added to control the size of particles. As more bromide is added, the size of the nano prisms decreases (Table 1).

The absorption of white light can then be seen visually as color as the size of nanoparticle dictates interaction with specific wavelength of white light. The absorption at a certain wavelength can also be quantified via a spectrometer. As discussed earlier, if a sample absorbs in the yellow (580 nm) region, an observer will see a violet solution, or complimentary color. By measuring the wavelength of peak absorbance, nanoparticle size can be inferred, as the size of the aggregation dictates the wavelength of light influenced. Absorption will not be needed in this lab, but is dependent on both concentrations, and the path length of the light. In this lab, you will build a calibration between sodium bromide concentration and peak wavelength ( $\lambda$ ) absorption, which will then be used to approximate the concentration of sodium bromide.

Table 1: Effect of Bromide on Silver Nano prism Size			
Volume KBr added ( $\mu\text{L}$ )	Molarity KBr in soln.	Expected Color	Average Width Silver Nano prism (nm)
25	$2.63 \times 10^{-9}$	Blue	~64
35	$3.68 \times 10^{-9}$	Orange-red	~35
45	$4.74 \times 10^{-9}$	Yellow	~20

**Separating Light:** Perhaps you are familiar with a prism, which separates light via refraction. A second method of separating light is the single slit diffraction, which will be approximated using light bending around a human hair. In a single slit diffraction, light passes through a narrow slit of width  $w$  and strikes a screen at a distance  $L$  from the slit.

When light of the same wavelength, such as one color of laser light, passes through a small single slit, light bends around the corners of the slit and spreads. When bent light arrives at the screen in phase, a constructive or bright line emerges and when it arrives out of phase a destructive or dark space becomes visible on the screen (figure 4). The diffraction pattern,  $z$ , or distance between the center and dark lines, can be used in calculating  $w$  or in our experiment, width of hair, or any other small opaque body based on Babinet's principle. Diffraction patterns from apertures of known size and shape, for instance, are compared with the pattern of an unknown solid object. The size of red blood cells can be found with such a technique. In order to use the principle, the relationship between  $\lambda$ ,  $L$ ,  $z$  and  $w$  must be established.

$$= zn/L$$

**Equation 4**

$$= n\lambda$$

**Equation 5**

$n = 1, 2, 3, zn$ , and is the number of destructive diffractions, or dark spots from the center. If you measure  $z$  from the center to the first dark spot,  $n = 1$ , to the second spot,  $n = 2$ , etc.  $L$  is the distance between the laser source and the screen. Be careful to use the same units for both  $zn$  and  $L$  as the distance units must cancel in equation #4. Using the distances  $z$  and  $L$ , can be solved for and can be used in equation #5. The diffraction angles, are too small to be measured mechanically with a protractor.  $\lambda$  must be inferred based on the color of laser. Some of the laser pointers are labeled with a wavelength range. You may pick the mid-point of that range, look at a neighbor's laser label, or infer wavelength from the color of laser

(figure 2). You can now solve for  $w$ , or width of hair. Please note: the distance unit of hair width corresponds to the unit that you used for wavelength ( $\lambda$ ) in equation #5.

### Objectives

The objective of this lab is to use light interaction to perform distance measurements that are too small to use standard measurement techniques. The width of hair and the peak absorbance of silver aggregate will be measured using diffraction and absorption. The particle size of the silver aggregate will be estimated. A calibration curve will be used in order to find the bromide concentration of an unknown silver nanoparticle solution.

### Safety/Housekeeping

- Do not point the laser toward anyone's eyes
- Sodium Citrate and Peroxides are strong oxidizers, use caution and wear goggles
- Discard all silver nanoparticle waste, including from cuvettes to the waste container in hood #1
- Discard your syringes to the marked waste container in hood #1 rather than the normal trash
- Rinse special glassware including beakers vials and cuvettes with DI water and return to the tray that you got it from

### Procedure: Silver Nano-Prism:

Materials:

Solutions; Prepared Weekly:

1. A) 0.375 M sodium citrate
2. B) 0.00375 M silver nitrate
3. C) 0.50 M hydrogen peroxide
4. D) 0.010 M potassium bromide (instructor only)
5. E) 0.050 M sodium borohydride (prepared daily)
6. F) Unknown sample for analysis

Glassware and apparatus:

1. A) Plastic syringes – 4 per group
2. B) Acid washed beakers supplied by stockroom, **do not use the beakers from your drawers, they are not clean enough.** – 4 per group
3. C) Acid washed 20 mL screw cap vials – 3 per group
4. D) Cuvette: 1 per group
5. E) Disposable pipettes – 5 per group

### Procedure

The solutions you will be using are provided in stock bottles and labeled with the chemical name and a letter designation A, B, C, and E. Label each of four beakers A, B, C, and E. **Each group should pour approximately 20 ml of each solution into the corresponding labeled beaker. DO NOT WORK OUT OF THE STOCK SOLUTION BOTTLES, FILL EACH RESPECTIVE BEAKER AND TAKE BACK TO YOUR WORK BENCH. There should be plenty of solution for each of 8 groups.**

\_\_\_\_\_

\_\_\_\_\_

E

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

C

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

B

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_  
\_\_\_\_\_  
**A**  
\_\_\_\_\_  
\_\_\_\_\_

\_\_\_\_\_  
\_\_\_\_\_  
**D**  
\_\_\_\_\_  
\_\_\_\_\_

**Solution D – Will be in a mall glass vial and it will be added by your instructor or stockroom personnel. Your instructor may want you to add solution D. If so, follow his or her instructions**

**Label three screw cap vials 1, 2, and 3.**

**25  $\mu$ l KBr**

**35  $\mu$ l KBr**

**45 $\mu$ l KBr**

You will be adding the respective solutions from the beakers to the screw cap vials with the syringes. **DO NOT MIX UP ANY OF THE SYRINGES. This experiment will not work correctly if you have cross contamination of any of the chemicals. Do not use the same syringe for different solutions.**

**The order of addition of the chemical reagents is critical. Do not deviate from the steps outlined below. Using the syringes provided, add each reagent drop wise and do not touch the syringes to the sides of the vials. Do not mix or shake the vials until all the reagents are added. Then replace the caps gently swirl the vial.**

**Step 1 – Using a clean syringe, add 2 ml of solution (A) Sodium Citrate 0.0375 M to each vial 1, 2, and 3.**

**Step 2**– Using a clean syringe; add drop wise, 5 ml of solution **(B)** Silver Nitrate 0.000375 M to each vial, 1, 2, & 3.

**Step 3** – Using a clean syringe, add drop wise, 5 ml of solution **(C)** Hydrogen Peroxide 0.05 M to each vial 1, 2, and 3.

**Step 4. Your instructor will supervise the addition of solution D using the micro pipette. A new pipette tip will be used for each addition. Discard the pipette tips in the container provided.**

Vial 1 – Using a clean pipette tip carefully add 25  $\mu$ l of solution D, 0.001M Potassium Bromide, (KBr) to vial 1 and replace cap on vial.

Vial 2 – Using a second clean pipette tip carefully add 35  $\mu$ l of solution D, 0.001M Potassium Bromide, (KBr) to vial 2 and replace cap on vial.

Vial 3 – Using a third clean pipette tip carefully add 45  $\mu$ l of solution D, 0.001M Potassium Bromide, (KBr) to vial 3 and replace cap on vial.

**Step 5** – With a clean syringe, slowly add drop wise, 2.5 ml of solution **(E)** Sodium Borohydride 0.005 M to vial 1. This step will initiate the reduction reaction and other reactions forming the silver nanoparticles.

**Step 6** – Replace the cap on the vial and gently swirl the vial to mix. A yellow color will begin to appear, and it will change over time. It should take about 3 – 5 minutes for the color to develop.

**Step 7** – Repeat steps 5 and 6 for vials 2 and 3.

After development, the color of each of the respective solutions, with darker or lighter variations, should develop as follows:



25  $\mu\text{L}$  KBr    35  $\mu\text{L}$  KBr    45 $\mu\text{L}$  KBr

**Step 8.** You will be determining the wavelength of maximum absorbance,  $\lambda_{\text{max}}$ , of each sample by use of the Vernier Spectrometer and the Logger Pro Software installed on the laboratory computers. Your instructor will provide you with instructions on using the Vernier Spectrometer. The light path should travel through the clear or un-ribbed path of the cuvette. The cuvette must hold enough solution for the light to travel through. Be careful to avoid bubbles or fingerprints on the cuvette as this will change the light absorption. You will begin with deionized water in your cuvette for calibration. In subsequent measurements, rinse your cuvette with the solution to be measured. This rinse is closer to your desired solution than D.I. water, which may dilute your desired measurement.

**Step 9.** You will find the wavelength of maximum absorbance,  $\lambda_{\text{max}}$ , of an unknown made by following the laboratory procedures, and doped with an unknown volume of KBr. The volume of KBr added is of the same order of magnitude as the volumes that you have used. Record the peak absorption for reference with a calibration curve which you will create.

The following **example** data table may be useful. The reaction is sensitive to environmental factors, and the volume KBr may need to be adjusted by your instructor.

---

Data Table 3: Volume KBr ( $\mu\text{L}$ ) Against Wavelength of max Absorption, $\lambda_{\text{max}}$ , (nm)			
Volume KBr ( $\mu\text{L}$ )	25 $\mu\text{L}$	35 $\mu\text{L}$	45 $\mu\text{L}$ Unknown
$\lambda_{\text{max}}$ (nm)			

---

**Step 10.** You will create a calibration curve in order to find the volume KBr in your unknown data using your known data. Using a spreadsheet formula such as excel, plot the volume KBr in  $\mu\text{L}$  (x-axis) against the peak absorption wavelength,  $\lambda$ , in nm (y-axis). In excel, the A column defaults to the x-axis and the B column to the y. Your calibration curve will have three points. With your data selected, insert an XY (Scatter) plot (no line).

Right click on a point on the graph and select [Add Trendline]. This will give you the option of selecting [Display Equation] of the line. See the student appendix for more information on graphing with excel. You can also display the  $R^2$  value, which informs the linearity of your data. A perfect line has an  $R^2$  value of 1. You now have a linear equation in slope intercept form. Recall that the y value is the wavelength of peak absorption ( $\lambda$ ) of your unknown compound. Use the linear equation and your known y value to find the x value, or volume KBr added. Points will be awarded for approximating the correct unknown value, or volume of KBr in  $\mu\text{L}$ . You should include your regression in your informal report, including a graph title and axis titles. The linear equation should be visible as well. You can use the *snip* app to capture your regression and should not include the entire spreadsheet.

### **Procedure: Width of Hair**

Materials: Laser pointer (red), hair (red or other), paper, ruler, clear tape, measuring tape.

1. Obtain a single strand of hair. You may be able to measure the differences in fine and full-bodied hair. You will use two samples, so it may be interesting to choose two different types of hair and predict the results.
2. Tape the hair across the laser pointer. Only the hair should cross the path of the laser.

3. Point the laser pointer at the wall or paper. You will notice that as you move further from the wall, the diffraction pattern becomes larger, minimizing your measurement error. It also, however, becomes fainter. Find a distance that takes both of these factors into account.
4. Measure the distance,  $L$ , from the front of the laser pointer to the screen. Be sure to note the distance unit. You may tape the laser pointer to a counter or book if helpful.
5. Measure the distances,  $z_1$  and  $z_2$  from the center of the light to the center of the first and second dark (destructive) bands. If you have a clear diffraction pattern, the third diffraction band can work as well, minimizing measurement error, and yielding easily measured distance.

The following **example** data table may be helpful in collecting your measurements.

Data Table 1: Diffraction from Hair Measurement	
Hair Sample 1	Hair Sample 2
$L$ with unit	$L$ with unit
$z_1$ with unit	$z_1$ with unit
$z_2$ with unit	$z_2$ with unit
Hair Type: <i>fine, medium, coarse</i>	Hair Type: <i>fine, medium, coarse</i>

Analysis: Width of Hair

1. Calculate the value for each hair sample using equation # 4. Be sure to include sample calculations in your report.
2. Calculate the width of each hair  $w$  for each sample using equation #5. Be sure to include sample calculations in your report. You are likely to have calculated your hair width,  $w$ , in

nanometers. Convert and report your hair width to micrometers,  $\mu\text{m}$ . Hair width has surprising variation, but you should be able to report hair width in micrometers without exponential notation.

3. You now have two width measurements for each hair sample. Report the % Difference of the width ( $w$ ) originating from  $z_1$  and  $z_2$  for each hair sample. Analysis comparing different samples beyond width will not tell us much about our measurement and calculation precision. You will recall % difference from lab 1.

$\% \text{ difference} = x \ 100$

**Equation 6**

The following **example** data table may be helpful in collecting your calculation data. As always, include sample calculations in your report.

Data Table 2: Hair Width Calculation	
Hair Sample 1	Hair Sample 2
Hair type (course/fine):	Hair type (course/fine):
:	:
$w$ based on $z_1$ with unit:	$w$ based on $z_1$ with unit:
$w$ based on $z_2$ with unit:	$w$ based on $z_2$ with unit:
$w$ based on $z_1 \ \mu\text{m}$ :	$w$ based on $z_1 \ \mu\text{m}$ :
$w$ based on $z_2 \ \mu\text{m}$ :	$w$ based on $z_2 \ \mu\text{m}$ :
% Difference 1:	% Difference 2:

Post Lab Questions

1. If you used a green laser pointer in place of red, what would happen to?

2. If the color of a silver nanoparticle solution remains constant, what can you infer about the aggregation of silver nanoparticles?
3. Find the absorption peak absorbing at the shortest wavelength from part two of the lab, or from your spectrophotometric data. What is the amount of energy associated with the absorption of one photon of light in order to form this peak?

### Conclusion

Discuss your findings from this experiment by addressing the following questions in paragraph form.

1. Describe how you used your data to figure out the concentration of your unknown sample. Why and how did you use all of the prior data to find a single number?

Try to write this conclusion as if you were explaining this experiment to your Chem 101 self.

2. a. Find a real-world application to finding the diameter or width of something small using the interaction with light. Do not use an example from this lab.
3. Find a real-world application using a calibration curve in order to quantify an unknown. Do not use an example from this lab.

### Your Report

1. Complete the pre-lab and take the pre-lab quiz in Blackboard before attending lab.
2. Use a lab notebook to record observations and make

calculations.

3. Create an informal lab document to turn in at the beginning of the next lab. Your lab report should include clearly labeled and tabulated or graphed raw data, any formulas or equations, unknown number, or letter (if applicable), conclusive data such as width, volume of an analyte, % error, etc. with clear example calculations or sample calculations\*. Use graphs or tables where applicable. Discuss results relevant to your findings. Think about the main point of the lab, or the results that you worked for and be sure to include it or them. Refer to *How to Write an Informal Lab Report* as you write your report.
4. Include numbered responses to any post lab questions. You do not need to re-write the questions but will use complete sentences or a short paragraph as appropriate. Be specific. If you describe an error, for example, you must describe the direction the data would skew and why.
5. Submit your document **before** your next lab appointment under the assignment tab on your laboratory Blackboard shell. Please note: Lab two will take two weeks. Submit the lab prior to the start of lab three.

#### References:

Davila, R. In *Lab 10: Spectrophotometry* College of Southern Idaho; College of Southern Idaho; 2020.

Adapted From: Dieckmann, G., Sibert, J. In *An Atoms First Approach to the General Chemistry Laboratory*; McGraw Hill: New York, NY, 2015; pp 21–32.

Personal Communication: Vladimir Kitaev, First-Year Laboratory Experiment on Synthesis of Silver Nano prisms with variable size

and Investigation of Their Optical Properties Andrew J. Frank,  
Nicole Cathcart, Kenneth E. Maly, Vladimir Kitaev\* Department of  
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Personal Communication: Dong Hee Son

DEPARTMENT OF CHEMISTRY TEXAS A&M UNIVERSITY Ver. 1.1

Materials in nanoscale: Size matters. Dong Hee Son

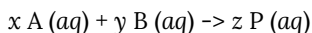
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# 3. Exp. 3: Reaction Stoichiometry and Formation of a Metal Ion Complex

## Pre-Lab

Use BB to quiz on the following before the lab

1. The method of continuous variation was used to determine the stoichiometry for:



In this experiment, the molarity concentrations of reactants A and B are both  $1.50 \times 10^{-3} \text{ M}$ . The amount of product was measured for a variety of mixtures of A and B, where the total mixture volume was always kept constant at 10.0 mL. The following plot was generated:



1. a) Using the equations of the trendlines, perform a simultaneous equations calculation to determine the volume of A used to obtain the maximum amount of product (= the value of  $x$  at the point of intersection of the lines).
  
1. b) Determine the volume of B used to obtain the maximum amount of product (hint – what was the total volume of the mixture of A and B?).
  
2. c) Using these two volumes, determine the simplest whole number volume ratio of reactants (volume A : volume B) used to obtain the maximum amount of product. Note that this ratio yields the reaction stoichiometry, i.e., volume A : volume B =  $x$  :  $y$ . One method you may use is to divide both volumes by the smaller volume. One volume (and mole ratio in an equimolar solution) will then be one. The second may be a whole number or may need to be multiplied by a factor to get a whole number. If the second ratio, for example, is 2.25, your volume (and mole) ratio is 1:2.25. Multiplying both by 4 yields a volume and mole ratio of 4:9.
3. In the experiment to be performed in this lab, what is the unbalanced reaction that will be studied?
4. What technique will be used in this experiment to determine the amount of product produced?
5. What special feature of the product makes this a good technique to use?
6. Why may happen if fingerprints or bubbles are present in your filled cuvette? Why should you handle the 'ribbed' side of the cuvette?

## Objectives

The objectives of this laboratory are as follows:

- To determine the reaction stoichiometry for the formation of a metal ion complex between iron (II) cations and 1,10-phenanthroline using continuous variation.
- To monitor the relative concentration of a colored metal ion complex in solution with absorption spectrometry.

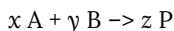
## Introduction

In 1928, French chemist Paul Job captured the imagination of the scientific community with a graphical representation of the UV absorption of  $\text{Ti}(\text{NO}_3)/\text{NH}_3$  against the mole fraction,  $X_a$ , of  $\text{Ti}(\text{NO}_3)$ . This plot is now called a plot of continuous variation, or a Job's Plot. Much like Job, you will use a continuous variation plot in order to discover the stoichiometric ration of a metal ligand complex. First, some background on stoichiometry.

The net result of a reaction can be summarized by a chemical equation. In order to write a chemical equation, a chemist must identify the reactants and products, as well as the ratios in which these species react and are produced, i.e., the stoichiometry of the reaction.

When two or more reactants are mixed together, it is possible to determine whether a reaction occurs by observing whether any property of the mixture changes. Further, by investigating how the change in an observed property varies when different ratios of reactants are mixed, the stoichiometry of the reaction can be determined.

Consider the study of a reaction where solutions of reactants A and B are mixed and product P is formed:



The reactant mixtures are carefully chosen so that *sum* of the moles of A and B are constant, and the amount of product that forms for each mixture is measured. This is known as the method of continuous variation. If either A or B is in excess, the excess will remain in solution rather than be used to form product. The maximum amount of P is formed when A and B are mixed in the correct stoichiometric amounts, when there is just enough of each to react with nothing left over.

Suppose for example that  $x = 3$  and  $y = 2$ , and that total number of moles of A and B is kept fixed at 0.10 moles. The amount of product formed will be at a maximum when the ratio A: B is 3:2, that is when  $A = 0.06$  moles and  $B = 0.04$  moles:

Amount of P formed

Moles of A	0.00	0.01	0.02	0.03	0.04	0.05	0.06	0.07	0.08	0.09	0.10
Moles of B	0.10	0.09	0.08	0.07	0.06	0.05	0.04	0.03	0.02	0.01	0.00

Note that to the left of the maximum on the plot ( $A < 0.06$  moles); there is not enough of A to react with all the B present. Thus, less than the maximum amount of product will be generated. To the right of the maximum ( $B < 0.04$  moles), there is not enough of B to react with all the A present. So once again, less than the maximum amount of product will be generated. As long as the total amount of  $A + B$  is constant, the maximum amount of product forms when the A: B ratio is the stoichiometric ratio for that reaction.

The reaction to be studied in this lab involves the formation of a metal ion complex. Metal ions, especially transition metal ions, possess the ability to form complexes with both organic and inorganic molecules called ligands. These complexes are produced when lone pair electrons from the ligand are donated into empty orbitals of the metal ion (resulting in a coordinate covalent bond). Here, iron (II) cations will be mixed with the ligand 1,10-phenanthroline to produce an iron (II)-phenanthroline complex:

\_\_\_\_  
 \_\_\_\_  
 \_\_\_\_  
 \_\_\_\_



where phen = 1,10-phenanthroline =

Using the method of continuous variation as outlined earlier, several reactant solutions are prepared in which the mole quantities of the metal ion and the ligand are varied but the sum of the mole quantities is kept constant. The amount of complex produced will be measured, the maximum indicating when the correct stoichiometric ratio of  $\text{Fe}^{2+}$ : phen is used.

In this experiment, both the  $\text{Fe}^{2+}$  and phenanthroline solutions will have the same molarity concentration. Mixtures prepared with the same total number of moles will therefore have the same total volume (recall that moles of solute can be calculated by multiplying

the solution volume by its molarity). As an additional consequence, in each prepared mixture the volume ratio of reactants used will be identical to the mole ratio of reactants used:

$$n_{\text{Fe}} = M_{\text{Fe}} \times V_{\text{Fe}} \text{ and } n_{\text{phen}} = M_{\text{phen}} \times V_{\text{phen}}$$

$$\text{if } M_{\text{Fe}} = M_{\text{phen}}$$

$$\text{then } n_{\text{Fe}}:n_{\text{phen}} \propto V_{\text{Fe}}:V_{\text{phen}}$$

Thus, it will be more convenient to analyze the amount of complex product formed as a function of reactant volumes, rather than as a function of reactant moles.

The challenge in this experiment is choosing an appropriate technique to measure the amount of complex formed. Since the complex is a red-orange color (while both reactants are colorless), the technique of Absorption Spectroscopy will be used. When visible light is directed at a colored compound in solution, the compound will absorb some wavelengths of the light while transmitting other wavelengths. The higher the concentration of the colored compound, the more light it will absorb.

Consider, for example, a blue compound in solution. When visible light is directed at this solution, the compound will predominantly absorb wavelengths in the orange region of the color spectrum, while predominantly transmitting wavelengths in the blue region of the spectrum (note that orange and blue are complementary colors). The transmitted light gives rise to the solution color that we see. Since the iron (II)-phenanthroline complex is a red-orange color, it is expected to absorb blue-green wavelengths, between 460 and 550 nm.

A UV-Visible spectrophotometer is the device used to measure how much light of a specific wavelength is absorbed by a colored species in solution. A schematic diagram of a typical spectrophotometer is shown below.

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

\_\_\_\_\_  
\_\_\_\_\_  
Computer  
A  
Wavelength  
\_\_\_\_\_  
\_\_\_\_\_

Beer's Law quantitatively describes the relationship between the light absorbed (A) and the concentration of the colored species in solution (c) as:

$$\text{Equation 2: } A = e l c$$

where  $e$  is the molar extinction coefficient (a constant that depends characteristics of the compound analyzed and the wavelength used for analysis) and  $l$  is the path length, or the distance traveled by light through the sample (a constant of 1.0 cm in this experiment). The important thing to note is that concentration and Absorbance are directly proportional.

Absorbance can thus be used as a direct measure of the amount of complex produced in each reactant mixture analyzed.

## Procedure

### Safety

Both reactant solutions are prepared in an acidic buffer. In addition, 1,10-phenanthroline is a mild irritant. If any of these solutions comes into contact with your skin or eyes, rinse with copious amounts of water for 15 minutes, and inform your instructor.

### Materials and Equipment

200 – 1000  $\mu\text{L}$  pipette, two pipette tips, one cuvette for spectrophotometer, spectrophotometer, ten small test tubes, two 50-mL beakers, wash bottle filled with distilled water, Parafilm® (or a small stopper), Kim Wipes®,  $2.50 \times 10^{-4}$  M  $\text{Fe}^{+2}$  solution and  $2.5 \times 10^{-4}$  M 1,10-phenanthroline solution.

### Experiment Instructions

There will be ~8 spectrophotometers available for student use. Your instructor will provide directions on how to use them correctly at the beginning of the lab session. Use the same cuvette for all measurements.

### *Preparing Mixtures of Reactant Solutions*

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**Table 1: Iron (II): Phen volumes**

Tube	iron (II)	phen
1	3.50 mL	1.50 mL
2	3.00 mL	2.00 mL
3	2.50 mL	2.50 mL
4	2.00 mL	3.00 mL
5	1.50 mL	3.50 mL
6	1.25 mL	3.75 mL
7	1.00 mL	4.00 mL
8	0.75 mL	4.25 mL
9	0.50 mL	4.50 mL
10	0.25 mL	4.75 mL

---

1. Obtain approximately 25-mL of  $2.50 \times 10^{-4}$  M  $\text{Fe}^{+2}$  and 40-mL of  $2.5 \times 10^{-4}$  M phenanthroline in each of your small beakers. Use the appropriate micro pipettes to prepare the following solution mixtures in your small test tubes. You will use only two micropipette tips. Recall that  $\mu\text{L}$  represent  $1 \times 10^{-6}$  L and mL represent  $1 \times 10^{-3}$  L. Perform conversions in your notebook if necessary. You will fill and dispense the pipette multiple times per test tube for the larger volumes.

The rose to orange color of the iron (II)-phenanthroline complex should fully develop within ten minutes of mixing.

#### *Determining $\lambda_{\text{max}}$ of the Complex*

2. Rinse the cuvette with distilled water and dry it carefully with a Kim Wipe tissue. Fill your cuvette to the mark with **phenanthroline only**. This solution will be used as your calibration blank. Your colorimeter will prompt a zero calibration, which can also be accessed in the *Experiment* drop



menu under the *Calibrate* tab. Choose *Spect. 1*, then *Finish Calibration*. You have now zeroed the spectrophotometer so that it will measure the solution absorbance and 'zero' the cuvette and solvent absorbance.

### *Measuring the Absorbance of the Solutions with Different Mole/Volume Ratios*

3. Measure the absorbance and  $\lambda_{\text{max}}$  of each of the ten prepared solution mixtures. Record this data on your report form.
  - Rinse the clean, empty cuvette with a small volume of the solution to be measured and rinse to waste\*. Fill the cuvette at least  $\frac{3}{4}$  full of the solution mixture in tube #1. Place it in the sample holder and measure the solution absorbance and peak wavelength. This can be done by placing the cursor at the highest absorbance peak and reading the x (wavelength, nm) and y (absorbance) readings at the bottom left of the graph. Use the *Auto scale* and *zoom* functions to calibrate your graph.
  - Return the solution mixture into its original test tube. Rinse the same cuvette with a small volume of the next solution to be analyzed. Discard the rinse solution to your waste.
  - Repeat this process with all other solution mixtures in tubes #2-10.

\*You are accustomed to rinsing glassware with D.I. water. In this

case, however, a small volume of water will dilute your sample. Each successive solution is closer than D.I water in both target species concentration and absorbance. You will have greater success by rinsing your cuvette with the solution to be analyzed, especially as you move from low to high concentration or absorbance.

4. When completely finished, dispose of your chemical waste as directed by your instructor.

### Data Analysis

Using Microsoft Excel®, graph your absorbance data as a function of the volume of ligand (1, 10 Phenanthroline) used. Refer to student Appendix VII for help with an excel® scatterplot and series data. You will graph this data as two data sets with overlay. Treat ascending and descending values as two separate data sets, plotting them both on the same set of axes. You can do this by right clicking on your graph (or the filter icon) to select data. You can then input the  $x$  and  $y$  values that are ascending as a series and the  $x$  and  $y$  values that are descending by adding a second series. \*The **example** graph from contrived data will slope and intercept the  $y$ -axis differently than your data. Add best-fit trendlines to both data sets and obtain the equations of these trendlines. Then solve for the point of intersection of these lines using your two equations in a simultaneous equations calculation.

From the point of intersection, you can determine the volume mixture of reactants used to obtain the maximum absorbance (hence, the maximum amount of complex formed). Finally, using this volume mixture, you can determine the simplest whole number volume ratio of  $\text{Fe}^{+2}$ : Ph (identical to the mole ratio), which is the stoichiometric ratio for this reaction. One method you may use is to divide both volumes by the smaller volume. One volume (and mole ratio in an equimolar solution) will then be one. The second may be

a whole number or may need to be multiplied by a factor to get a whole number. If the second ratio, for example, is 2.25, your volume (and mole) ratio is 1:2.25. Multiplying both by 4 yields a volume and mole ratio of 4:9. The following **example** data tables may help as you create your informal report.

#### Experimental Data

- *Absorbance of Reactant Solution Mixtures*

Molarity of Iron (II):

Molarity of

Phenanthroline: \_ \_ \_

Mixture	Volumes of Reactants Mixed (mL)	Absorbance (unitless)	$\lambda_{max}$ (nm)
Iron (II)	Phenanthroline		
1	3.50	1.50	
2	3.00	2.00	
3	2.50	2.50	
4	2.00	3.00	
5	1.50	3.50	
6	1.25	3.75	
7	1.00	4.00	
8	0.75	4.25	
9	0.50	4.50	
10	0.25	4.75	

#### Data Analysis/Post Lab

- 1) Using Microsoft Excel®, create a graph of Volume of Phenanthroline vs absorbance. Plot this data as two data sets with overlay Treat ascending and descending values as two separate data sets, plotting them both on the same set of axes

(if needed, review the instructions in your student appendix). Add best-fit trendlines to both data sets and obtain the equations of these Attach this graph to your report using the *Snip app*. The graph should take up no more than  $\frac{1}{2}$  page and should be labeled appropriately.

Ascending	datatrendlineequation: _____		
Descending	data	trendline	equation: _____

- 2) Using the equations of the trendlines, perform a simultaneous equations calculation to determine the volume of phenanthroline used to achieve the maximum product absorbance (= the value of x at the point of intersection of the lines). Show your work.
- 3) Determine the volume of iron (II) used to achieve the maximum product absorbance (hint, what was the total volume of the iron (II)–phenanthroline mixture?).
- 4) Using these two reactant volumes (at the maximum absorbance) determine the simplest whole number volume ratio of Fe<sup>2+</sup>: phen, which is the stoichiometric ratio for this Use the following method: do not round until the last step:
  - Convert mass or volume to moles (this is assumed as you are using equal molar ratios.
  - Divide by the smaller number of moles.
  - Multiply until you get a whole number.

- 5) Write the balanced equation for the reaction studied in this **Refer to Equation 1 for guidance.** You will replace the  $x$  and  $y$  variables with your mole ratio data. You may refer to 1, 10 phenanthroline as phen.
- 6) Since the two reactant solutions in this experiment have the same molarity, the volume ratio at maximum absorbance will be the same as the mole ratio at maximum absorbance. Perform appropriate calculations to prove that this is indeed the
- 7) How does your data acquisition process support or contradict Beer's law? Be specific and use the variables contained in Beer's law.
- 8) Find an outside application for continuous variation and/or Beer's law.

### **Your Report:**

1. Complete the pre-lab and take the pre-lab quiz in your Blackboard shell before attending lab.
2. Use a lab notebook to record observations and make calculations.
3. Create an informal lab document to turn in at the beginning of the next lab. Your lab report should include clearly labeled and tabulated or graphed raw data, any formulas or equations, unknown number, or letter (if applicable), conclusive data such as chemical formula, etc. with clear example calculations or

sample calculations\*. Use tables or graphical data where applicable. Discuss results relevant to your findings. Think about the main point of the lab, or the results that you worked for and be sure to include it or them. Refer to *How to Write an Informal Lab Report* as you draft your report.

4. Include numbered responses to any post lab questions. You do not need to re-write the questions but will use complete sentences or a short paragraph as appropriate. Be specific. If you describe an error, for example, you must describe the direction the data would skew and why.
5. Submit your document **before** your next lab appointment under the assignment tab on your laboratory Blackboard shell.

#### References:

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Renny JS, Tomasevich LL, Tallmadge EH, Collum DB. Method of continuous variations: applications of job plots to the study of molecular associations in organometallic chemistry. *Angew Chem Int Ed Engl*. 2013;52(46):11998-12013. doi:10.1002/anie.201304157

# 4. Exp. 4: Formation of an Oxide

Determination of the Empirical Formula of Two Compounds:

Pre-Lab, may be included on BB pre-quiz:

1. Define empirical formula
2. Define the Law of Definite Composition
3. Write the empirical formula for each of the following compounds:
  1. 0.200 mol Al and 0.600 mol Cl
  2. 0.200 mol Fe and 0.400 mol S
  3. 0.200 mol Cu and 0.400 mol S
  4. 0.200 mol Cu and 0.400 mol O
  5. 0.200 mol Ba and 0.400 mol S
  6. 0.200 mol Ba and 0.400 mol O
  7. 0.200 mol S and 0.400 mol O
  8. 0.200 mol S and 0.400 mol Cl
  9. 0.200 mol S and 0.400 mol Cu
  10. 10.98 g Ba, 2.57 g S, and 5.12 g O
4. When 2.50 g of copper (Cu) react with oxygen, the copper oxide product has a mass of 2.81 g. Copper oxide has two possible formulas; CuO or Cu<sub>2</sub>O. Which formula is supported by the given data? Be sure to show a calculation.

5. Calculate the theoretical percent composition, by mass, for each element in the compound  $\text{MgO}$ .
6. A compound contains only C, H and O. If the combustion of a 0.255 g sample of the compound produces 0.561 g of  $\text{CO}_2$  and 0.306 g of  $\text{H}_2\text{O}$ , what is the empirical formula of the compound?
7. Explain the safety concerns in this lab and how to avoid getting injured.

### Introduction

Joseph Proust did much of his pioneering work using inorganic binary compounds including sulfates, sulfides, and metallic oxides. Today you will be working with both sulfates and metallic oxides, and much like Proust, you will use mass relationships to come up with the chemical compound formulas associated with the masses of the elements present. Proust published the law of Constant Composition in 1794 in a paper detailing his work on iron oxides; "A chemical compound always contains the same elements combined together in the same proportion by mass." In order to frame this law, let's first review chemical and physical change.

When a substance undergoes a physical change, bonds between



atoms are not re-arranged. When iron is melted, it undergoes a physical change from solid to liquid, but the identity of the metal remains the same. In a chemical change, however, the substance gains a different composition and different properties. As iron oxidizes, for example, it changes from a dull gray to iron (III) oxide to a ‘rust’ colored substance with different properties and a different composition.

You will be observing a chemical reaction today that falls under the category of a combustion, or combination with oxygen. We are most familiar with the combustion process that occurs with a hydrocarbon such as wood, oil, wax, or gasoline. Such combustions form the most stable oxygen compounds possible,  $\text{CO}_2(\text{g})$  and  $\text{H}_2\text{O}$ . The combustion of a metal, such as magnesium, will also form the most stable oxide of the metal with the formula  $\text{Mg}_x\text{O}_y$ . With careful mass measurement and stoichiometric analysis, you will be able to determine the formula of the metal oxide formed with magnesium, or the ratio of oxygen to metal in the product, much as Proust did centuries ago. It is important to note that the ratio is an atom to atom or mole to mole ratio, so the mass values you obtain will all be converted into moles before analysis.

Table 1: Comparison of Physical and Chemical Changes

Physical	Chemical
Change in State	Formation of a gas (bubbles)
Change in Size	Formation of a solid (precipitate)
Tearing	Disappearance of a solid (dissolving)
Breaking	Change in color*
Grinding	Heat given off or absorbed

\*Change in color can also be physical. A hot stove is one example.

Most chemical reactions can be classified into five types, shown in table 2.

Table 2: Types of Chemical Reactions

Type of Reaction	Description	Example Equation
Combination/ Synthesis	Elements or compounds form a more complex product.	$P + O_2 \rightarrow P_2O_5$
Decomposition	A reacting substance is split into simpler products.	$KClO_3 \rightarrow KCl + O_2$
Single replacement	One element replaces another in a compound	$NaCl + F_2 \rightarrow NaF + Cl_2$
Double replacement	Elements in two compounds switch places	$AgI + Fe_2(CO_3)_2 \rightarrow FeI_3 + Ag_2CO_3$
Combustion	Reactant and oxygen form an oxide product.	$Fe + O_2 \rightarrow Fe_2O_3$

### Objective

The objective of this lab is to discover the empirical formula of two compounds, the first by combustion, the second by dehydration of a hydrate. The law of definite proportions will be used.

### Safety/Housekeeping

- When using flame, tie back long hair, remove loose clothing, especially synthetics, wear your lab coat and goggles but not gloves while burning
- Don't look directly at the burning magnesium.
- Remember that hot objects take a while to cool
- Discard your solid compounds in the inorganic solid waste and aqueous compounds in the labeled waste container in hood 1.

### Procedures

#### Finding the Simplest Formula of an oxide

Materials: Crucible, crucible lid, crucible tongs, clay triangle, iron ring, ring stand, Bunsen burner, magnesium ribbon, steel wool, ceramic square.

\*Use a procedure that makes sense to you. The crucible lid, for

example, can fall and break. If it makes sense to weigh your compound without the lid, do so and modify the data table.

1. Obtain a clean and dry crucible and lid. Secure stains in the crucible will not affect the mass calculations. Place the crucible and cover in the clay triangle and heat for one (1) minute. Cool until close to room temperature. Using crucible tongs, weigh the crucible and cover (together) and record. Do not round any digits from masses that you collect.
2. Obtain a piece of magnesium ribbon that has a mass of 0.15 to 0.3 g. Remove any tarnish found on the ribbon by polishing with steel wool or sandpaper and weigh once more. Wind the ribbon into a loose coil and place the coil into the crucible. Weigh the crucible and cover with the magnesium.
3. Begin to heat the crucible with the lid. Oxygen must, however, be replaced by lifting the lid periodically. Watch closely for smoke or fumes, which is the first indication of the magnesium and oxygen reacting. As the magnesium reacts, leave the lid off for a bit to make sure the magnesium finishes reacting. **Avoid looking directly at the burning magnesium as it emits enough light to damage your eyes.**

Observe the reaction of the magnesium and oxygen about every minute. When the magnesium no longer produces smoke or a flame, heat the crucible strongly for another five (5) minutes. Turn off the burner and let the crucible and its contents cool to room temperature. It may cool more quickly on the ceramic square than on the warm triangle.

\*Do not place your crucible on a cold surface such as your ring stand. The thermal shock will crack your crucible. Do not add water to your warm crucible. It will crack.

4. Reweigh the crucible, cover, and oxide contents. Record their mass and describe the appearance of the oxide product.
5. Perform the combustion twice. The ratio of Mg to O should be

very close. If it is not, perform a third combustion.

You may find the following **example** data table useful as you work through your calculations; you will need to reproduce it or one like it for your informal report:

---

**Data Table 1: Magnesium Oxide Formula**

	<b>Trial 1</b>	<b>Trial 2</b>
Mass of Crucible and Lid (g)		
Mass Magnesium (g)		
Mass Magnesium Oxide Product (g)		
Mass Oxygen (g)		
Moles Magnesium (mol)		
Moles Oxygen (mol)*		
Average Moles Mg (mol)		
Average Moles Oxygen (mol)		

---

\*Note: Use O rather than O<sub>2</sub> to perform your calculations as your calculated oxygen is combined with Mg.

**Calculations:**

Atoms in a chemical formula combine in whole number ratios. If this is true, this must remain true for moles of atoms in a formula. Divide the numbers of moles magnesium and oxygen by the smaller of the two values. This dictates that the quotient of the smaller value is one (1). The other quotient must be an integer, 1, 2, 3, etc., or easily lend itself to a whole number ratio. For example, 1:1.5, when doubled, becomes a whole number ratio of 2:3. 1:1.33, when tripled, becomes a whole number ratio of 3:4 etc. Using the mole values for magnesium and oxygen, use this method to find the simplest (empirical) formula for the oxide of magnesium. With careful measuring and recording, your data will lend itself to a whole number ratio. You must report your data as a whole number and may not use decimals or fractions. Points will be awarded for obtaining the correct formula. Many compounds have small whole number ratios. If you find a large ratio,

8 to 9 for example, you may consider checking your measurements and simplifying to a smaller ratio.

Data:

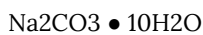
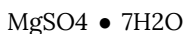
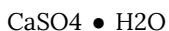
Report the ratio of Mg to O as a chemical formula with whole numbers:

-----  
-----

## Part 2: Formation of a Hydrate

### Discussion:

A hydrate is a salt that contains a specific number of water molecules called the water of hydration. The number of water molecules is fixed for each kind of hydrate but differs from one salt to another. In the formula of a hydrate, the number of water molecules is written after the salt formula and is separated by a large, raised dot.



Heating the hydrate provides energy to remove the water molecules. The salt without water is called an anhydrate. **The following is an example but is not the dehydration that you will perform.**



Through careful measurement, you will determine the water of hydration associated with the salt **CuSO<sub>4</sub>** by separating the anhydrate from the water.

### Procedure Part two:

Materials: Crucible, clay triangle, crucible tongs, hydrate of CuSO<sub>4</sub>, iron ring and stand, Bunsen burner, ceramic square.

1. Scrape out your crucible into the inorganic solid waste and heat for 2-3 minutes. Let it cool and weigh carefully.
2. Fill the crucible about 1/3 full of a hydrate of CuSO<sub>4</sub>. Record the mass of the hydrate and crucible.

- Record the appearance of the hydrate.
- Set the crucible and hydrate on the clay triangle and iron ring. Heat gently for five (5) minutes. You may need to gently stir the compound using a metal spatula. Increase the intensity of the flame, and heat strongly for ten (10) minutes. The bottom of the crucible may glow a dull red. Remove the crucible to the ceramic square to cool. Record the mass.
- Heat the crucible and contents a second time for five (5) minutes. Cool and reweigh. If the mass from the second heating is within 0.05 g of the mass obtained after the first heating, you have completely dehydrated the salt. If not, heat again until you have agreement between the final masses. Use the final mass for your calculation. Do not average the masses.
- Record the appearance of the hydrate after heating.
- Place the compound on a watch glass and slowly add water dropwise to the compound using a disposable pipet. Record your observations.

The following **example** data table may be helpful in recording your data.

---

**Data Table 2: Formula of a Hydrate of  $\text{CuSO}_4$**

	First Heating	Second Heating
Mass of dry crucible (g)		xxxxxxxxxxxxxx
Mass of crucible and hydrate (g)		xxxxxxxxxxxxxx
Mass of crucible and anhydrate (g)		
Mass of hydrate (g)		
Mass of anhydrate (g)		
Mass of water (g)		
Moles anhydrate		
Moles water		

---

Data:

Calculate the formula of the hydrate: This calculation is similar to an empirical formula calculation. You must, however, find a ratio of **one (1)** for the anhydrate and a whole number for the water. In other words, consider dividing by the smallest number of moles.

Post Lab Questions:

1. Through experimentation you found a 9.0 gram of water sample from Nampa contained 1.0 g hydrogen and 8.0 g of oxygen. Another water sample, 13.5 g total mass, taken from Crouch contained 1.5 g hydrogen and 12.0 g oxygen. Show how this illustrates the Law of Definite Composition.
2. Two different chemicals, A and X, were reacted. The first time they were combined, the ratio was AX<sub>1.51</sub>. The second time they were reacted, the ratio was AX<sub>3.32</sub>. Give at least two possible reasons for the difference. (The Law of Definite Composition is valid in both cases). Consider what ASSUMPTIONS you are making when doing an experiment and whether these assumptions are valid.
3. An old car rusts in place. If none of the rust is removed by weathering, will the car gain or lose mass? Defend your answer.

4. What practical applications would knowing the percent composition of an element in a compound have in chemistry or another discipline?
5. If the law of definite composition is obeyed every time, how is it possible to obtain two different formulas for copper oxide?

Your Report:

1. Complete the pre-lab and take the pre-lab quiz in your Blackboard shell before attending lab.
2. Use a lab notebook to record observations and make calculations.
3. Create an informal lab document to turn in at the beginning of the next lab. Your lab report should include clearly labeled and tabulated or graphed raw data, any formulas or equations, unknown number or letter (if applicable), conclusive data such as chemical or hydrate formula etc. with clear example calculations or sample calculations\*. Discuss results relevant to your findings. Think about the main point of the lab, or the results that you worked for and be sure to include it or them. Refer to *How to Write an Informal Lab Report* as you write your report.
4. Include numbered responses to any post lab questions. You do not need to re-write the questions but will use complete sentences or a short paragraph as appropriate. Be specific. If you describe an error, for example, you must describe the direction the data would skew and why.
5. Submit your document **before** your next lab appointment under the assignment tab on your laboratory Blackboard shell.



References:

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# 5. Exp. 5: Molecular Geometry and Hybridization

Pre-Lab:

Name:

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Print this lab (double sided). Read the lab. Complete the following table by hand. Quiz on the tabled structures via BB before lab.

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<b>Chemical Species</b>	$\text{KrF}_2$	$\text{PH}_4^+$	$\text{TeCl}_6$	$\text{ClBr}_3$	$\text{H}_2\text{S}$
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Lewis Structure

Perspective Drawing

Number of atoms bonded to central atom

Number of lone pairs on central atom

Electronic geometry

Molecular Geometry

Polarity \*Note: This is the polarity of the molecule, not the bond polarity

---

## Objectives

The objectives of this exercise are:

- To analyze bond angles of a variety of molecules and ions using molecular model kits.
  - To draw Lewis structures (both projection and perspective drawings) for each of these molecules and ions.
- 
- To determine the hybridization of the central atoms, the number and types of bonds, the geometries, and the polarities of the molecules and ions.

### **Safety/Housekeeping:**

Wear splash goggles when anyone in the lab uses solvents or chemicals

- Wear splash goggles when anyone in the lab uses solvents or chemicals
- Dispose of solvents in the labeled beaker in hood 1
- Dispose of naphthalene in the labeled beaker in hood 1. This may require an acetone rinse. **Do not rinse into the sink. It is volatile**, and water insoluble.
- Return your disassembled model kit to the back shelf.

### **Background**

#### *Lewis Structures*

It would be hard to overstate Gilbert Lewis' contributions to modern chemistry. His prolific innovations span work in acid base theory, thermodynamics, covalent bond theory and the behavior of electrons in the covalent bond. Lewis Structures bear his name. Lewis also coined the word 'photon' as 'light quanta' in a 1926 letter to *Nature*. Lewis mentored countless PhD students including twenty Nobel laureates, many of which collaborated with Lewis. The

'greatest scientist to never win the Nobel prize'; Lewis was nominated for the Nobel award 41 times, but evidence exists that his nominations were blocked for political reasons stemming from early criticisms of his graduate mentor while working in Germany (Kean, 2011). Sadly, Lewis was found dead in his Berkley laboratory after working with hydrogen cyanide. The circumstances behind his death are unclear.

Today you will be creating structures named after Lewis, as well as VSEPR theory to predict bond angle and hybridization. A Lewis Structure is a representation of covalent molecules (or polyatomic ions) where all the valence electrons are shown distributed about the bonded atoms as either shared electron pairs (bond pairs) or unshared electron pairs (lone pairs). A shared pair of electrons is represented as a short line (a single bond). Sometimes atoms can share two pairs of electrons, represented by two short lines (a double bond). Atoms can even share three pairs of electrons, represented by three short lines (a triple bond). Pairs of dots are used to represent lone pair electrons. Examples are shown for the molecules SF<sub>2</sub> and CH<sub>2</sub>O below.

Please review (in your text or notes) the rules for drawing Lewis structures before performing this exercise. This includes rules for structures which obey the octet rule as well as those which involve expanded or reduced octets and how to calculate and optimize formal charge.

### *Resonance Structures*

Resonance refers to bonding in molecules or ions that cannot be correctly represented by a single Lewis structure. These structures are often equivalent, meaning that they contain the same number of bonds at different locations. The molecule SO<sub>2</sub> (shown above) has two such resonance forms. Resonance structures can also be non-equivalent, in which case they will have different numbers and/or

locations of bonds. Note that any valid resonance structure of a molecule can be used to determine its shape and polarity.

### *VSEPR Theory*

The VSEPR (Valence Shell Electron Pair Repulsion) model is used to predict the geometry of molecules based on the number of effective electron pairs around a central atom. The main postulate for the VSEPR theory is that the geometrical structure around a given atom is principally determined by minimizing the repulsion between effective electron pairs. Both the molecular geometry and the polarity of individual bonds then determine whether the molecule is polar or not.

Before determining the shape of a molecule, the Lewis structure must be properly drawn. The shape of a molecule is then determined by the number of areas of electron density (or, number of effective electron pairs) around a central atom. Areas of electrons density include:

- Lone pairs of electrons: these electrons tend to take more space than the bonded pairs in space leading into somewhat distorted structures.
- Bonds (single, double, and triple bonds count as one (1) area of electron density or one effective electron pair).

Before performing this exercise, please review (in your text or notes) the various geometries and bond angles that can be produced by different numbers of effective electron pairs around the central atom.

### *Molecular Polarity*

A polar bond is one in which the electron cloud is closer to the nucleus of one atom (the more electronegative one) than the other (the less electronegative one). Knowledge of both the bond polarities and the shape are required in the determination of the

molecule's overall polarity (dipole moment). A polar molecule is one that shows an imbalance in its electron distribution. When placed in an electric field, these molecules tend to align themselves with the electric field.

Some molecules have polar bonds but no dipole moment. This happens when the bonds in a molecule are arranged in a way in which polarities cancel each other. One way this occurs is when molecules have all identical bonds and there is no lone pair on the central atom (for example,  $\text{CO}_2$ ). Molecules that do not fit both of these criteria may be polar or not depending on how atoms are bonded, and the electron pairs arranged around the central atom (for example,  $\text{XeCl}_2\text{F}_2$  shown below).

Take care to distinguish ionic compounds from polar covalent compounds. None of the Lewis structures that you created today depicts an ionic compound. Ionic compounds require a lot of energy to free ions from their crystalline structure and have both high melting and boiling points.  $\text{NaCl}$ , for example, melts at just over  $800^\circ\text{C}$ . Melting of polar covalent structure such as sucrose, on the other hand, is more nuanced. Sugar is found in observable crystals as well. Initial heating will separate the glucose and fructose molecules, an apparent melting called an inversion. Further heating will remove water, leaving nothing but carbon. If this is controlled, caramelization occurs. If the process continues, only carbon remains, and you are left with burned sugar. In contrast to melting an ionic compound, inverting, or even burning sugar occurs at temperatures easily reached in a home kitchen. The melting point of sucrose, for example, is listed at  $186^\circ\text{C}$ .

### *Hybridized Central Atom Bonding*

Bond hybridization is a complex theory that can be summarized for the central atom by counting the number of electron domains about the central atom. An electron domain is defined as an area of electron density including a lone pair of electrons or a single, double, or triple bond. The following table will help you assign central atom bond hybridization to your molecules.

---

**Table 1: Electron Domains and Central Atom Hybridization**

Number of Electron Domains	Central Atom Bond Hybridization
2	sp
3	sp <sup>2</sup>
4	sp <sup>3</sup>
5	sp <sup>3</sup> d
6	sp <sup>3</sup> d <sup>2</sup>

---

### Procedure

Complete the following for each of the molecules and ions on your Report form:

1. Draw Lewis structures, including all resonance structures if applicable (1).
2. Build models and then draw perspective structures (2) that accurately represent bond angles and molecular shapes.

The molecular model kits contain different colored balls and different size stick connectors. Three-dimensional models will be constructed from these balls and sticks.

The stick connectors represent bonds. Use the short rigid sticks for single bonds. The long flexible sticks must be used to create double (2 sticks) and triple (3 sticks) bonds.

The different colored balls represent different atoms. There is a key on the inside of the lid of the model kit which indicates

which colors correspond to which atoms. There are, however, some exceptions to this. For example, the kit indicates that the green balls with just one hole are to be used for the halogens. But if a halogen (such as Cl) appears in a molecule as a central atom with an expanded octet, you would need to use a ball that has 5 or 6 holes to build the model (brown or silver ball). Another example is oxygen. The kit indicates that the red balls with two holes should be used for oxygen. However, if an oxygen atom in a compound requires more than two bonds, the red balls cannot be used. In this case you would substitute a blue ball for oxygen.

The 3-D models will serve as a visual guide to help you with your perspective structures. Use the following guidelines to draw them correctly:

- For bonds lying in the plane of the paper, use a regular solid line (—).
- For bonds that project down into the paper away from you, use a hatched wedge-shaped
- For bonds that project up out of the paper towards you, use a solid wedge-shaped line

(     )

An example showing both the Lewis structure and perspective representation of CH<sub>4</sub> is provided below.

3. Determine the number of atoms bonded to the central atom (or, number of  $\sigma$ -bonds) (3).
4. Determine the number of lone electron pairs on the central atom (4).
5. Predict the electronic geometry using all areas of electron density (or, effective electron pairs) and the ideal bond angles



- associated with this geometry (5).
- Predict the actual geometry of the molecule or ion (6).
  - Determine the hybridization of the central atom (7).
- 
- Determine the polarity of the molecule (8). Use an arrow to show the direction of electron density for polar molecules on the perspective drawing.

**Please be sure to keep the model kits discrete and complete for the next group.**

### **Part 2: Polarity of Solvents**

- Create the following solvent mixtures in a test tube with one pipette full of each solvent. You may invert a test tube if covered by a gloved thumb, parafilm, or a small stopper.

---

**Data Table 1: Mixing Liquid Solvents**

<b>Solvents Tested</b>	<b>Observed Results</b>
Water/ethanol	
Water/cyclohexane	
Ethanol/cyclohexane	

---

- Test the following solids in each solvent by adding a small amount of solid to one pipette full of each solvent. Note: adding too much solid will overwhelm the solvent, giving a

false negative for solubility. Unknowns #1 and #2 are selected from the given list of solutes.

3. If you are still unsure of the identities of unknowns #1 and #2, devise another test based on the information you read in the lab introduction. Be sure to use proper safety techniques such as the fume hood and clear your idea with your lab instructor. Be prepared to clean any mess that occurs in your glassware. You may consider testing what does not happen, rather than what does.

---

**Data Table 2: Mixing Solids and Liquids**

**Solutes**

**Solvents**

**Water**

**Ethanol   Cyclohexane**

NaCl

Sucrose

Naphthalene

Unknown #1

Unknown #2

---

**Additional Tests:**

**Results:**

**Lab Data:**

You may hand draw and fill in the appropriate structures and data for submission. See report requirements for details.

**Table 2: Electron Domain Geometries**

**HCN**

1. Lewis Structure	2. Perspective drawing	
3. Number of atoms bonded to central atom	4. Number of non-bonding electron pairs on the central atom	5. Electronic geometry:
6. Molecular geometry with ideal bond angles	7. Hybridization of central atom	8. Polarity:

**CH3OH (carbon as center)**

1. Lewis Structure	2. Perspective drawing	
3. Number of atoms bonded to central atom (Carbon)	4. Number of non-bonding electron pairs on the central atom	5. Electronic geometry:
6. Molecular geometry with ideal bond angles	7. Hybridization of central atom	8. Polarity:

### CH<sub>3</sub>OH (oxygen as center)

3. Number of atoms bonded to central atom (Oxygen)	4. Number of non-bonding electron pairs on the central atom	5. Electronic geometry:
6. Molecular geometry with ideal bond angles	7. Hybridization of central atom	

### SeF<sub>6</sub>

1. Lewis Structure	2. Perspective drawing	
3. Number of atoms bonded to central atom	4. Number of non-bonding electron pairs on the central atom	5. Electronic geometry:
6. Molecular geometry with ideal bond angles	7. Hybridization of central atom	8. Polarity:

### NO<sub>2</sub><sup>-</sup>

1. Lewis Structure	2. Perspective drawing	
3. Number of atoms bonded to central atom	4. Number of non-bonding electron pairs on the central atom	5. Electronic geometry:
6. Molecular geometry with ideal bond angles	7. Hybridization of central atom	8. Polarity:

### AsF<sub>5</sub>

1. Lewis Structure	2. Perspective drawing	
3. Number of atoms bonded to central atom	4. Number of non-bonding electron pairs on the central atom	5. Electronic geometry:
6. Molecular geometry with ideal bond angles	7. Hybridization of central atom	8. Polarity:

### TeF<sub>5</sub><sup>-</sup>

---

1. Lewis Structure	2. Perspective drawing	
3. Number of atoms bonded to central atom	4. Number of non-bonding electron pairs on the central atom	5. Electronic geometry:
6. Molecular geometry with ideal bond angles	7. Hybridization of central atom	8. Polarity:

---

### **XeF<sub>2</sub>**

---

1. Lewis Structure	2. Perspective drawing	
3. Number of atoms bonded to central atom	4. Number of non-bonding electron pairs on the central atom	5. Electronic geometry:
6. Molecular geometry with ideal bond angles	7. Hybridization of central atom	8. Polarity:

---

### **H<sub>2</sub>CO**

1. Lewis Structure	2. Perspective drawing	
3. Number of atoms bonded to central atom	4. Number of non-bonding electron pairs on the central atom	5. Electronic geometry:
6. Molecular geometry with ideal bond angles	7. Hybridization of central atom	8. Polarity:

#### SF<sub>4</sub>

1. Lewis Structure	2. Perspective drawing	
3. Number of atoms bonded to central atom	4. Number of non-bonding electron pairs on the central atom	5. Electronic geometry:
6. Molecular geometry with ideal bond angles	7. Hybridization of central atom	8. Polarity:

#### PO<sub>4</sub><sup>3-</sup>

---

1. Lewis Structure	2. Perspective drawing	
3. Number of atoms bonded to central atom	4. Number of non-bonding electron pairs on the central atom	5. Electronic geometry:
6. Molecular geometry with ideal bond angles	7. Hybridization of central atom	8. Polarity:

---

### **XeF<sub>4</sub>**

---

1. Lewis Structure	2. Perspective drawing	
3. Number of atoms bonded to central atom	4. Number of non-bonding electron pairs on the central atom	5. Electronic geometry:
6. Molecular geometry with ideal bond angles	7. Hybridization of central atom	8. Polarity:

---

### **BrF<sub>3</sub>**



1. Lewis Structure	2. Perspective drawing	
3. Number of atoms bonded to central atom	4. Number of non-bonding electron pairs on the central atom	5. Electronic geometry:
6. Molecular geometry with ideal bond angles	7. Hybridization of central atom	8. Polarity:

### NH<sub>3</sub>

1. Lewis Structure	2. Perspective drawing	
3. Number of atoms bonded to central atom	4. Number of non-bonding electron pairs on the central atom	5. Electronic geometry:
6. Molecular geometry with ideal bond angles	7. Hybridization of central atom	8. Polarity:

### CH<sub>3</sub>NH<sub>2</sub> (carbon as center)

1. Lewis Structure	2. Perspective drawing	
3. Number of atoms bonded to central atom	4. Number of non-bonding electron pairs on the central atom	5. Electronic geometry:
6. Molecular geometry with ideal bond angles	7. Hybridization of central atom	8. Polarity:

### CH<sub>3</sub>NH<sub>2</sub> (nitrogen as center)

3. Number of atoms bonded to central atom	4. Number of non-bonding electron pairs on the central atom	5. Electronic geometry:
6. Molecular geometry with ideal bond angles	7. Hybridization of central atom	

### SBr<sub>2</sub>

1. Lewis Structure	2. Perspective drawing	
3. Number of atoms bonded to central atom	4. Number of non-bonding electron pairs on the central atom	5. Electronic geometry:
6. Molecular geometry with ideal bond angles	7. Hybridization of central atom	8. Polarity:

**Molecule (Choose any molecule that interests you):**

---

1. Lewis Structure

2. Perspective drawing

3. Number of atoms  
bonded to

4. Number of  
non-bonding

5. Electronic  
geometry:

central atom

electron pairs on the  
central atom

6. Molecular geometry  
with ideal

7. Hybridization of  
central atom

8. Polarity:

bond angles

---

**Find each molecule that displays resonance; include the structures here:**

\_\_\_\_\_

\_\_\_\_\_

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**Summary of Geometries**

Areas of Electron Density	Electron Geometry	Molecular Geometry	Example Species (from your examples)	Polarity of Example Species
Number of Atoms bonded to Central Atom ( $\sigma$ bonds)	Number of Lone Pairs (on central atom)			
2	0			
3	0			
2	1			
4	0			
3	1			
2	2			
5	0			
4	1			
3	2			
2	3			
6	0			

5 1

4 2

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**Post Lab:**

1. List the formula for each molecule that has lone pairs on the central atom and is non-polar. Explain why these molecules are non-polar.
2. Ionic compounds have extremely high melting and boiling points. NaCl, for example, melts at just over 800 ° Celsius. It takes a-lot of energy to free the ions from a rigid crystalline structure. In light of this fact, why does it appear so effortless for a polar solvent such as water to dissolve or separate an ionic compound?
3. Write a short paragraph contrasting the importance of earning a prize, formal recognition, getting published, etc. with advancing the body of scientific or other knowledge and increasing in personal learning.
4. Connect the shape or geometry of a molecule or ion to its function in paragraph form. Pharmaceuticals, metal ion ligands, hormone receptors, and piezoelectric detectors are some options to begin your search. \*In order to help you practice journal searches and citation for the upcoming formal

report, your citation must be journal sourced and cited (ACS (American Chemical Society) preferred, APA accepted). The CWI library is a great starting point; <https://cwi.edu/current-students/library>

Please see the *Journalistic Format Reference Document* in you BB shell for parameters.

### **Lab Report:**

You will complete your lab report differently than in prior labs.

1. Complete the pre-lab and take the pre-lab quiz in Blackboard before attending lab.
2. Print or reproduce and complete the data tables for work during and after lab. You must build the model associated with each compound. You may hand draw Lewis structures and perspective diagrams, and may label the remaining data requirements by hand, including polarity, # lone pairs, etc., as well as the summary data. If the molecule appears to have no central atom, assign carbon as the central atom. One section of data is marked *Molecule*. Choose a molecule or geometry that interest you. It must be different from your lab partner's molecules.
3. Include a brief printed document with answers to any post lab questions. Include data and observations for the wet portion of the lab including solubility and other observations organized into a table. You will turn in all your **printed** materials; your printed lab with drawings and post lab document stapled together at the beginning of your next lab appointment.

### **Sources:**

Adapted from: Chem 11 Experiments, Santa Monica College  
<http://www.smc.edu/AcademicPrograms/PhysicalSciences/>

Chemistry/Lab-Manual/Pages/Chem-11-Experiments.aspx  
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(accessed Jun 27, 2020).

# 6. Exp. 6: Gravimetric Analysis of Limestone from Mackay, ID

**Pre-Lab:** Use BB to quiz on the following before the lab, but after reading the lab.

1. Using reactions 1 and 2, or 3 from your lab, if 0.250 g  $\text{CO}_2(\text{g})$  escape from your reaction flask after addition of  $\text{HCl}(\text{aq})$ , how many grams of  $\text{CaCO}_3(\text{s})$  are present in your limestone sample?
2. If your limestone sample from the previous problem weighs 0.80 g, what is the percent composition of  $\text{CaCO}_3(\text{s})$  in your sample?
3. Solid calcium sulfate is collected in a Büchner funnel and dried. The mass of the solid product is 1.03 g. How many grams of  $\text{CaCO}_3(\text{s})$  are present in your sample?
4. If your limestone sample from the previous problem weighs 0.80 g, what is the percent composition of  $\text{CaCO}_3(\text{s})$  in your sample?



5. What is the percent difference between the two methods of measuring a percent composition.
6. How will you avoid injury in this lab?

**Introduction\*** This lab will be reported as a formal lab report. You should collect careful measurements and observations to help you complete the report. You may want to take pictures of your lab set up and observations to include in your report (optional).

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Figure 1: Mackay, ID population 517

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Gravimetric analysis, a quantitative analysis technique, can be applied to various processes including determination of the composition of minerals or rock to quality control measures quantifying finished products and raw materials in industry. Used to measure the unknown mass of an analyte, it consists of precipitating the analyte as a known compound. The compound can then be isolated and weighed, yielding the percent composition of that analyte, or ratio of mass of the analyte to the entire sample. In a related process, removing an analyte can yield the percent composition of the compound by subtraction. With either method, a balanced equation relates either the precipitate or volatile compound that is removed to the entire sample. In this experiment, you will perform both a gravimetric analysis of a precipitate and subtraction of a volatile compound to find the percent composition of calcium carbonate,  $\text{CaCO}_3$ , in a sample of limestone from Mackay, Idaho, a town sometimes called the ‘top’ of Idaho, due to its proximity to Mt. Borah and the Lost River Range.

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Figure 2: Wenlock Limestone, showing fossils of marine creatures like crinoids and brachiopods.

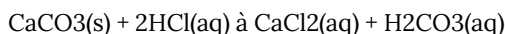
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What makes the geology of this region more interesting is that the limestone that you will analyze formed in what was anciently a warm, shallow sea, allowing invertebrates to absorb the minerals from the ocean water in the form of shells, corals, algae, and other creatures. Limestone represents an important sedimentary rock with a biological origin.  $\text{CaCO}_3$  is of particular interest, as solubility decreases with increasing temperature. Its solubility properties allow it to dissolve to create caves and sink holes as well as precipitate to form stalactites and stalagmites.

In this laboratory, you will analyze two samples, first via subtraction and then via gravimetric analysis for a total of four data points. You will then compare the results from two different methods in order to determine the percent composition of your analyte.

% composition = **Equation 1**

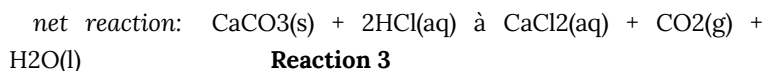
The following reactions will be useful as you perform your analysis. Your analyte of interest,  $\text{CaCO}_3(\text{s})$  reacts readily with  $\text{HCl}$ , producing carbonic acid (Reaction 1) which then decomposes to  $\text{CO}_2(\text{g})$ , (Reaction 2). The reaction of  $\text{CaCO}_3(\text{s})$  and a strong acid such as  $\text{HCl}$  is often used by field geologists as a test for limestone or other carbonates.



**Reaction 1**



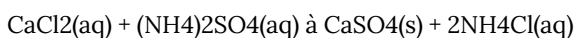
**Reaction 2**



**Reaction 3**

By carefully measuring the mass of your sample and acid before and after the effervescence of  $\text{CO}_2(\text{g})$  occurring with the dissolution with  $\text{HCl}$ , you will be able to determine the amount of  $\text{CaCO}_3(\text{s})$  in your original sample. This determination is possible based on the reaction stoichiometry from reactions one and two. This measurement depends on an assumption. What is that assumption? Be sure to include it in your formal report.

In addition, you will quantify the amount of  $\text{CaCO}_3(\text{s})$  through precipitation of the  $\text{Ca}^{2+}$  ion (Reaction 1) as  $\text{CaSO}_4(\text{s})$ .



#### **Reaction 4**

By isolating the Ca as  $\text{CaSO}_4(\text{s})$ , you will be able to quantify the Calcium present in the original sample as  $\text{CaCO}_3(\text{s})$ . This quantification also depends on an assumption. What is this assumption? Be sure to include it in your formal report.

By finding the percent composition through two different methods, the evolution of  $\text{CO}_2(\text{g})$  and the precipitation of  $\text{CaSO}_4(\text{s})$ , a comparison is possible. Would a percent error or percent difference be more appropriate? See lab one for examples and formulae.

Expt.

#### **Materials and Procedures**

Limestone

Vacuum Filtration with Trap

Buchner Funnels with removable filtering surface

Quantitative Filter Paper Q5

3.00 M Hydrochloric Acid ( $\text{HCl}$ )

2–125 mL Erlenmeyer flask

150 mL Erlenmeyer flask – 2

Methyl red indicator

Ammonium sulfate ( $\text{NH}_4\text{SO}_4$ )  
2.00 M aqueous ammonia ( $\text{NH}_3$ ) also labeled Ammonium Hydroxide ( $\text{NH}_4\text{OH}$ )  
Transfer Pipette  
Glass stir rods  
Watch Glass – in your Drawer-2  
Drying Oven Set at  $110^\circ\text{C}$

### **Procedure Carbonate in Limestone**

#### ***\*Some notes on careful weighing and measuring.***

The validity of your data in this lab depends on careful measurement and recording technique. The changes in mass are minute; even small errors will affect your calculated results. Writing your formal report without reliable results will be difficult.

- Plan your data collection before you start. Understand why you are recording a measurement.
- Use the same balance for a sequence of measurements. Be sure to tare the balance after another group uses it.
- Choose whether to record and subtract the mass of glassware used or to tare the balance with the glassware and record the mass of reagents.
- Plan ahead with the final drying step. You must choose to attempt to scrape your dried product from your Büchner funnel, or you must record the mass of your dry Büchner funnel and filter paper before you filter. Each funnel and paper have a different mass.

### **Part 1**

1. Obtain enough limestone sample so that you have two separate rock samples weighing ~0.70 grams to 0.80 grams and record the exact mass in your lab book. **You will want to choose small pieces.** They are already broken up for you. The bigger your pieces the longer it will take to dissolve your

limestone.

2. Add approximately 15 mL of 3.00 M HCl to each of a clean and dry (**important!**) 125 mL Erlenmeyer flask(s). Label your flasks with a Sharpie or label tape before weighing them. Record the total mass of the container with the HCl in your lab book.
3. Add the limestone to the flask with the HCl. Swirl gently to mix the solution and allow the reaction to proceed until bubbling has stopped. If you are unsure if the reaction is complete, wait a bit and re-weigh to see if the mass is still changing. You may still see some solids left in the flask and it may be slightly dark. This is the portion of the rock that is not calcium carbonate and is not involved in the reaction.

### **Figure 3. Reacted Limestone and HCl**

4. Record the mass of the reacted limestone and HCl.
5. Keep this solution as you will use it in **Part 2**.
6. If your sample has any undissolved solids, carefully filter the

solution using gravity filtration as depicted.

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#### GRAVITY FILTRATION

- 6a.      **#1 Whatman Filter Paper 90mm– As shown in Diagram 1**
  - 6b.      **Set up your filter paper and place it in the funnel. Place the funnel in the 125 mL Erlenmeyer flask.**
  - 6c.      **Wet the filter paper with distilled water. Your Instructor can help you.**
  - 6d.      **Pour the solution into the funnel in portions so that you do not overfill the filter paper. Use your wash bottle with distilled water to remove all of the solids from the flask on to the filter paper. Add a minimal amounts of water as possible to wash your flask. Ensure your flask is entirely rinsed of all solution**
  - 6e.      **Let the funnel sit until there are no drops of solution in the stem of the funnel. About 10 minutes**
- 

Diagram 1: Gravity Filtration

The following **sample** data table may be helpful as you record your data and perform your stoichiometric calculation: Be sure to include sample calculations in your formal report. Analyze reactions 1-3 in order to discern your reaction stoichiometry.

#### Figure 4. Gravity Filtration set up

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**Data Table 1: Subtraction of CO<sub>2</sub>(g)**

	Sample 1	Sample 2
Mass Limestone		
Mass of Flask + HCl		
Mass of reacted limestone and HCl		
Mass of CO <sub>2</sub> (g) released		
Moles of CO <sub>2</sub> (g) released		
Moles of CaCO <sub>3</sub> (s) in sample		
Mass CaCO <sub>3</sub> (s) in sample		
% Composition CaCO <sub>3</sub> (s)		
Average % Composition CaCO <sub>3</sub> (s)		

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## Part 2

### **\*Helpful notes about precipitation:**

*Although your precipitation occurs based on the mole ratio of Ca<sup>2+</sup> and SO<sub>4</sub><sup>2-</sup>, adding excess water will inhibit precipitation. Use small volumes of water for any rinse steps that occur before precipitation.*

1. Add 10 drops of methyl red indicator to the filtrate from part 1. The sample, stored in a 125 or 150 Erlenmeyer flask, should turn a pink color.
2. While stirring with a stir rod or swirl flask (you can also use a stirring plate), add at least 1.5 grams of Ammonium Sulfate (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> to the flask. Continue stirring until the ammonium sulfate completely dissolves.
3. Using a 3 ml transfer pipette, slowly add 20 ml of 2.00 M Ammonium Hydroxide NH<sub>3</sub>OH, Continuing adding the Ammonium Hydroxide drop wise using a 3 ml transfer pipette

until the color *just* shifts from pink to pale yellow. It is important not to overshoot this color change the change will occur with one additional drop of Ammonium Hydroxide. The color change should be maintained with stirring or shaking. You can check the pH with pH paper. With the addition of the base the pH should change from 4.4 to 6.4, with the pale-yellow color.

**See the sequence below.**

**Figure 5: Precipitation of  $\text{CaSO}_4(\text{s})$**

- 1. Dissolved  $(\text{NH}_4)_2\text{SO}_4$  B. Addition of  $\text{NH}_4\text{OH}$  precipitate forming C. Color change with one drop of  $\text{NH}_4\text{OH}$**
2. Obtain a clean and dry Büchner funnel and piece of #5 filter paper.
5. Consider the goal of filtering and drying Calcium Sulfate precipitate. What is the final measurement you will use in your calculations? This will inform your next few steps.
6. Record the mass of the dry filter paper to be used in your Büchner funnel. You won't be able to dry your sample in your funnel, so you may need to weigh an evaporating dish or watch glass. Plan a few steps ahead. Would you get the most accurate result by weighing your dry precipitate and filter paper in a tared weigh boat? If so, you do not need to weigh an evaporating dish or watch glass. Will you get the best results by scraping your dry precipitate from your filter paper? If so, you don't need to weigh your filter paper. Adjust your data table to reflect your process. \*If you fail to weigh your filter



paper in this step, weighing a second paper and assuming the masses are the same will introduce substantial error.

*A Büchner filtration system has been set up for you. Your analyte has now precipitated as a solid, Be sure to empty the filtrate from your Buchner set up into the waste container when finished.*

7. Place the weighed filter paper in the Büchner funnel. **Wet the paper thoroughly with distilled water.** Turn on the vacuum to allow the paper to be sucked onto the funnel to ensure a good seal.
8. Collect the crystals by pouring the solution onto the filter paper in the funnel. Do not disturb the crystals on the filter paper or you will break the filtration vacuum. Wash any product left in the flask into the funnel with **cold** distilled water; rinse your stirring rod as well. Remember, any product you lose here will affect your mass percent.
9. Wash the **precipitate** with distilled water and allow the vacuum to pull air through to dry your sample for a few minutes.
10. \*(Optional: If your sample will dry in the oven between lab periods, you may skip this step) Allow the filtration to continue until you see no more water droplets fall from the funnel. Turn the vacuum off water off. Rinse the product with a little acetone from an acetone rinse bottle to drive off excess water.

Wait a minute and turn the vacuum on again to pull the acetone through the filter.

11. Place your filter papers and precipitates in the oven using a watch glass. Be sure to label your sample (prior to weighing).
12. To ensure that your sample is dry, record the mass and return to the oven. After another 10 minutes in the oven, re-weigh. If your sample mass is still changing, it is not yet dry. Once you are satisfied that your sample has dried, let it cool and record the mass in your lab book. During successive weighing, the mass of precipitate should differ by no more than 5%.

**Check that you have your own sample and not some else's sample.**

The following **sample** data table may be helpful as you record your results. Be sure to include sample calculation in your final report. Be sure to refer to reactions 1-4 for reaction stoichiometry. You may use two Buchner tops available or may have to filter and dry during successive lab periods.

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**Data Table 2: Precipitation of  $\text{CaSO}_4(\text{s})$  \*Adjust to your method**

	Sample 1	Sample 2
Mass dry filter paper		
Mass dry filter paper and watch glass or evaporating dish		
Mass dry precipitate, filter paper and evaporating dish or watch glass 1st Weighing		
Mass dry precipitate, filter paper and evaporating dish or watch glass 2nd Weighing		
Mass $\text{CaSO}_4(\text{s})$		
Moles $\text{CaSO}_4(\text{s})$		
Moles $\text{CaCO}_3(\text{s})$		
Mass $\text{CaCO}_3(\text{s})$		
% Composition $\text{CaCO}_3(\text{s})$		
Average % Composition $\text{CaCO}_3(\text{s})$		

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### Your Report

You will use the data and observations from this lab to complete your first formal lab report. Please see the *Formal Lab Documents* tab included in your BB shell, including the requirements found document, *How to Write a Formal Lab Report* found in your *Formal Lab Documents* file. To help you reference the document, use this incomplete list of requirements.

- A journal sourced application of the lab technique to be included in your *Introduction*
- Any assumptions made in your *Introduction*
- Any formulas or reactions used in your *Introduction*
- Any deviation from procedure as well as actual masses and concentrations in your *Experimental* section
- Tabulated raw data in your *Results and Discussion*
- Sample calculations in your *Results and Discussion*

- Discussion of any differences in % composition between samples first with gas evolution then with precipitation in the *Results and Discussion* section.
- Discussion of any differences in % composition within technique 1 and technique 2 in the *Results and Discussion* section
- Statistical treatment and discussion of any differences in average % composition between technique 1 and technique 2 in the *Results and Discussion* section including % error **or** % difference as appropriate (see your student appendix).
- Statistical treatment and discussion of the deviation between the four data points you obtained in the lab. Use a 95% confidence interval as described in your lab appendix. Discuss the reported error in your *Results and Discussion* section. (see Appendix IV)
- Findings should be listed in the *Abstract*, *Results and Discussion*, and *Conclusion* and discussed in the *Results and Discussion* section.
- Address whether or not the assumptions listed in the introduction were validated or were not validated in the *Conclusion* section.
- Reference citations (not URL) after the *Conclusion*
- **An originality report will be automatically generated upon your submission of the exam and will be visible to you. Please note that in science writing, direct quotations aren't used. You must, therefore, convey the ideas and theories as well as past work cited by literature without plagiarizing another's work. The originality report will alert me to similar reports across sections and semesters. It is reasonable to report the same measurements and numerical data as your lab partner. Duplicated work of less than 15% is generally accepted in journal writing. Unoriginal content of greater than 25% is generally considered plagiarized in journal writing. Reproduced writing, except as described previously, whether from a literature source or another student represents**

**plagiarism, and the first instance will result in a zero grade for the assignment without the opportunity to re-write the assignment.**

Your formal report (6) and informal report for lab seven (7) *Testing the Solubility Rules* will be turned in via your lab Blackboard shell during the week prior to final exams. Please plan accordingly.

### **References**

Adapted from Gregg Dieckmann and John Sibert's "Percent Composition from Gravimetric Analysis: Calcium Carbonate in Texas Limestone." *An Atoms First Approach to the General Chemistry Laboratory*. New York: McGraw-Hill. 201

British Geological Survey 1998 – 2017 (c)NERC mailto:www-bgs@bgs.ac.uk. How is limestone formed? <https://www.bgs.ac.uk/discoveringGeology/geologyOfBritain/limestoneLandscapes/whatIsLimestone/howFormed.html> (accessed Jun 30, 2020).

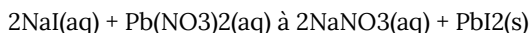
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[Quantifying the Composition of Limestone – Chem21Labs](https://www.chem21labs.com/labfiles/UKY_111_Exp4.pdf)m University of Kentucky, Lexington  
[www.chem21labs.com/labfiles/UKY\\_111\\_Exp4.pdf](https://www.chem21labs.com/labfiles/UKY_111_Exp4.pdf)

# 7. Exp. 7: Testing the Solubility Rules Through Quantitative Analysis

**Pre-Lab:** Use BB to quiz on the following before coming to lab.

1. Determine the amount of solid NaBr required to make 10. mL of a 0.20 M solution.
2. Find the spectator ions in the following precipitation reaction:



3. Find an anion that is soluble with silver cations.
4. Why must you use gloves and wash your hands when working with heavy metals such as Pb and Cd?

## **Introduction:**

In the *Density Determination* and *Percent Composition from Gravimetric Analysis* labs, you performed quantitative analysis by finding the amount of an analyte contained in a sample. This lab uses qualitative analysis to identify a substance through its solubility but does not quantify the substance. Qualitative analysis is used in fields such as forensics, arson and explosive investigation, and drug identification, among others. Physical and chemical properties, such as color, smell, fluorescence, reactivity, melting point, solubility, etc.

can be used in identifying a chemical species. Cations and anions will be identified qualitatively in this lab by aqueous solubility.

Svante Arrhenius, 1903 Nobel prize winner and great grandfather of Greta Thunburg, is credited as the first person to predict that carbon emissions in the form of  $\text{CO}_2(\text{g})$  were large enough to change earth's climate. He won the Nobel prize in 1903, however, for his work describing the dissolution of ionic compounds and the stability of the resulting ions through ion-dipole interactions. Svante's description of ions separating in water is elegantly simple and widely accepted now. At the time, the theory was poorly received by his professors. He in fact narrowly passed his graduate thesis defense on the topic.

Not all ionic compounds are soluble in water, giving rise to solubility tables like the one you will use in this lab. Some ionic solubilities are clear cut.  $\text{AgNO}_3$ , for example is very soluble while  $\text{AgCl}$  is not. Some solubilities are more nuanced.  $\text{Ag}_2\text{SO}_4$ , for instance, is listed as an exception to the rule that sulfates are generally water soluble. In fact, five grams of silver sulfate will dissolve in one liter of water. Solubility is most accurately described quantitatively. Tabulating solubility qualitatively provides clear and easy to use rules that allow for the qualitative identification of unknown cations and anions. The designations of soluble or insoluble, however, are arbitrary, giving rise to discrepancies even among test books. The second semester of this course uses  $K$  values or ratios of dissociated ions to the solid compound to quantitatively describe solubility but is beyond the scope of this lab.

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**Table 1: Aqueous Solubility of Ionic Compounds**

**Soluble Compounds**

**Soluble Compounds**

Li<sup>+</sup>, Na<sup>+</sup>, K<sup>+</sup>, Rb<sup>+</sup>, Cs<sup>+</sup>, NH<sub>4</sub><sup>+</sup>

NO<sub>3</sub><sup>-</sup>, C<sub>2</sub>H<sub>3</sub>O<sub>2</sub><sup>-</sup>, ClO<sub>3</sub><sup>-</sup>

Cl<sup>-</sup>, Br<sup>-</sup>, I<sup>-</sup>

SO<sub>4</sub><sup>2-</sup>

**Insoluble Exceptions**

Ag<sup>+</sup>, Hg<sub>2</sub><sup>2+</sup>, Pb<sup>2+</sup>

Ag<sup>+</sup>, Hg<sub>2</sub><sup>2+</sup>, Pb<sup>2+</sup>, Ca<sup>2+</sup>, Sr<sup>2+</sup>, Ba<sup>2+</sup>

**Insoluble Compounds**

**Insoluble Compounds**

CO<sub>3</sub><sup>2-</sup>, PO<sub>4</sub><sup>3-</sup>, CrO<sub>4</sub><sup>2-</sup>, S<sup>2-</sup>

OH<sup>-</sup>

**Soluble Exceptions**

Li<sup>+</sup>, Na<sup>+</sup>, K<sup>+</sup>, Rb<sup>+</sup>, Cs<sup>+</sup>, NH<sub>4</sub><sup>+</sup>

Li<sup>+</sup>, Na<sup>+</sup>, K<sup>+</sup>, Rb<sup>+</sup>, Cs<sup>+</sup>, Ba<sup>2+</sup>, NH<sub>4</sub><sup>+</sup>

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\*Notable Complication: Ag<sub>2</sub>SO<sub>4</sub> is moderately soluble. See introduction.

**Precipitation Reactions**

By using the solubility rules the products of a double replacement reaction can be predicted. The *molecular* reaction lists all species as (aq) aqueous or (s) solid. No ions are shown in the molecular reaction. The *total ionic* equation lists all aqueous species as ions, and the *net ionic* equation excludes spectator ions. Let's look at an example of each using sodium iodide and lead (II) nitrate. Please note that precipitation double replacement reactions will start with two aqueous compounds.

**Molecular:**  $2\text{NaI(aq)} + \text{Pb(NO}_3)_2\text{(aq)} \rightarrow 2\text{NaNO}_3\text{(aq)} + \text{PbI}_2\text{(s)}$

**Total Ionic:**  $2\text{Na}^+ + 2\text{I}^- + \text{Pb}^{2+} + 2\text{NO}_3^- \rightarrow 2\text{Na}^+ + 2\text{NO}_3^- + \text{PbI}_2\text{(s)}$

**Net Ionic:**  $2\text{I}^- + \text{Pb}^{2+} \rightarrow \text{PbI}_2\text{(s)}$



**Objective:**

The objective of this lab is to identify an unknown set of cations and an unknown combination of one to two anions using precipitation reactions.

**Safety:**

Barium and Lead ions are toxic. Use gloves and hand washing to minimize exposure to these chemicals. Wash your hands before leaving the lab, eating, or drinking. 0.1 M NaOH is a strong base. Use caution and wear splash goggles.

**Waste/Housekeeping:**

All solutions will be disposed of in the same waste in hood #1.

Avoid contaminating solutions. Do not return any solution to its container. Use the beral pipettes located in the solution holsters and return them without touching the counter.

**Procedure:**

Preparation of a 0.2 M NaBr solution

1. Calculate and weigh enough NaBr(s) to make 10 mL of a 0.2 M NaBr solution. Use your smallest Erlenmeyer flask and a graduated cylinder to approximate the molarity to one significant figure.
2. The other three anions, 0.1 M Na<sub>2</sub>SO<sub>4</sub>, 0.1 M Na<sub>3</sub>PO<sub>4</sub> and 0.1 M NaOH, can be distributed dropwise with a pipette.

\*Note that the anion is of interest. Sodium is the counter ion and will always dissociate.

**Cation Analysis**

\*The letters used to label solutions are repeated in this lab. Be sure to use the solutions marked **Cation** for this step.

3. The cations K<sup>+</sup>, Cu<sup>2+</sup>, Ag<sup>+</sup>, Ba<sup>2+</sup>, Fe<sup>3+</sup>, Mg<sup>2+</sup>, and Pb<sup>2+</sup> are randomly designated A – G. Each unknown cation is paired with nitrate ion, NO<sub>3</sub><sup>-</sup>, as a counter ion. Arrange your test tubes in such a way as to test each cation, A – G with each of

the four known anions from steps 1 and 2. Add or approximate 10 -12 drops of each cation and anion to test all possible combinations. For example, you might arrange a column of four 'A' cations, four 'B' cations and so on, then add Br<sup>-</sup> anion across the entire row. Create your own system, but label or keep notes to track your observations. Be sure to use one beeral pipette for each solution and add the solutions without touching the sides of the test tube. It is better to waste some solution than to risk contamination.

4. Record your observations such as no change, precipitation, color, etc.
5. Identify your unknown cations, A – G. Use all your observations, including the color of the unreacted reagent.
6. During or after lab, write molecular, total ionic, and net ionic equations for each **precipitation reaction** that you observe from your cation analysis. Combinations with no observable change will not yield a net ionic reaction and may be ignored.

The following **sample** data table may help you track your observations. Be sure to modify it based on your procedure.

---

**Data Table**  
**1: Cation**  
**Analysis**

	<b>Cation A</b>	<b>Cation B</b>	<b>Cation C</b>	<b>Cation D</b>	<b>Cation E</b>	<b>Cation F</b>	<b>Cation G</b>
<b>Br-</b>							
<b>OH-</b>							
<b>SO42-</b>							
<b>PO43-</b>							

---

Identify cations A – G.

Anion Analysis:

\*The letters used to label solutions are repeated in this lab. Be sure to use the solutions marked **Anion** for this step.

Now that you have found the identities of cations A – G, you can use these identities along with the solubility rules to find the identity of **one** unknown anion or anion combination. You will be assigned **one** anion unknown randomly lettered A – D from the following possibilities: Br<sup>-</sup>, Br<sup>-</sup>/SO<sub>4</sub><sup>2-</sup>, Br<sup>-</sup>/PO<sub>4</sub><sup>2-</sup>, Br<sup>-</sup>/OH<sup>-</sup>.

Combine 10-12 drops of your unknown anion with your known cations A – G to find the identity of your unknown anion(s).

The following **sample** data table may help. You can now label your cations based on their identities.

---

**Data Table**  
**2: Anion**  
**Analysis**

	Cation A	Cation B	Cation C	Cation D	Cation E	Cation F	Cation G
Unknown Anion(s)							
-----							

---

Anion letter: -----

Identify your anion(s): -----

### Post Lab:

- Consider a solution that contains a mixture of Ba<sup>2+</sup> and Fe<sup>3+</sup>.
  - What anion would you add to precipitate only Fe<sup>3+</sup>?
  - What anion would you add to only precipitate Ba<sup>2+</sup>?
- In what way is the title of a table, 'Solubility Rules' a misnomer? How would you better define solubility.
- Find 'rules' from two sources and list at least one contradiction among the rules. You may use this lab or your text as one of the sources.
- Find and describe an outside application of precipitating one

cation or anion while leaving another or others in solution, i.e., selective precipitation.

### **Your Report:**

1. Complete the pre-lab and take the pre-lab quiz in Blackboard prior to attending lab.
2. Use a lab notebook to take notes and make observations.
3. Create an informal lab document to turn in at the beginning of the next lab. Your lab report should have your name, letter, and identity of unknown anion, clearly labeled collected data in tables and or graphs where applicable, and molecular, total ionic, and net ionic equations of all precipitations. Think about the main point of the lab, or the results that you worked for and be sure to include it or them.
4. Numbered responses to any post lab questions. You don't need to re-write the questions.
5. Submit your document **before** your next lab appointment under the assignment tab on your laboratory Blackboard shell.

### **References:**

Adapted from Dieckmann, G., Sibert, J. In *An Atoms First Approach to the General Chemistry Laboratory*; McGraw Hill: New York, NY, 2014; pp 81–96.

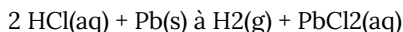
The Nobel Prize in Chemistry 1903. <https://www.nobelprize.org/prizes/chemistry/1903/arrhenius/biographical/> (accessed Jul 1, 2020).

# 8. Exp. 8: The Activity Series of Metals

**Pre-Lab:** Quiz on the following

Define:

1. Oxidation-reduction reaction
2. Oxidation half reaction
3. Reduction half reaction
4. Do the following half reactions represent oxidation or reduction?
  1.  $\text{Na(s)} \rightarrow \text{Na}^+ + \text{e}^-$
  2.  $\text{Cu}^{2+} + 2\text{e}^- \rightarrow \text{Cu(s)}$
5. Which metal is more reactive if the following oxidation-reduction reaction occurs? Consider hydrogen a metal in this context.



6. How will you avoid injury in this lab?

## Introduction:

---

Figure 1: Uluru, Australia. [adventure.com](http://adventure.com)

---

Over three quarters of the known elements can be classified as metals. In earth's oxygen rich environment, however, most metals exist as cations in rocks, minerals, or dirt. The red rock features found in the Southwest United States and Australia and the red clay found in the Southeast United States and Eastern Africa contain iron oxide deposits, or iron cations. In their metallic or uncombined form, metals conduct both heat and electricity, and are familiar

building, decorative, and conductive materials as they are both ductile and malleable. This reduced form of a metal is typified by a shiny appearance and is most likely what you think of as metallic. Coinage metals, such as gold and silver are readily reduced, and are found uncombined in nature. Alkali metals, such as sodium and potassium are readily oxidized, and so are less familiar in an uncombined form. These reactive examples are also lustrous or shiny when uncombined, however. Metal oxides also differ from porous iron oxides or rust, which allows for continual oxidation to thin dense oxide coatings on aluminum and chromium which protect from further oxidation to the dull grey oxides that forms on calcium or other alkali earth metals.

Metals differ significantly in their ability to lose electrons or oxidize. Reactivity among metals is defined as the ability to lose electrons and to form a cation. You may be familiar with the fact that gold is not very reactive, meaning it does not readily give up electrons, or oxidize, making it a good choice for jewelry, some of which has preserved for millennia. Calcium, on the other hand, is more readily oxidized, and does not last long in an uncombined state. Calcium, therefore, is more reactive, or easily oxidized than gold, and is more likely to be found as a cation combined with another element or elements.



*Less Reactive*

#### **Reaction 1**

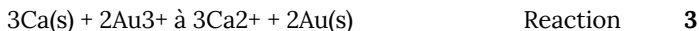


*More Reactive*

#### **Reaction 2**

This relative reactivity gives rise to the activity series of metals. More reactive, or more easily oxidized metals are found higher on the series. Today you will combine cation solutions of metals as well as reduced (metallic) metal solids to observe their relative reactivity. You will create an activity series based on these observations.

Reactions one and two, listed above, are oxidation half reactions, and show electrons leaving a solid metal sample. The electrons, however, must go somewhere. A more complete oxidation-reduction or redox reaction accounts for the transfer of electrons.

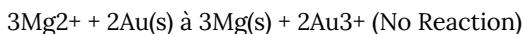


In this reaction, the more reactive calcium ends up as a cation in solution, and the less reactive gold ends up as a solid. Calcium, therefore, ranks higher in activity. Six electrons are transferred from solid calcium metal to gold cations. Calcium, therefore, is oxidized, or increases in charge, while gold is reduced, or decreases in charge. Reaction 3 can be divided into oxidation and reduction half reactions.



Note that the half reactions are multiplied by a coefficient in order to cancel the electrons. The overall redox reaction balances by mass and charge, as do the half reactions.

You probably know that gold is less reactive than calcium. To test the reactivity of these metals you would place a solid piece of calcium metal in a solution of gold cations, which is represented in Reaction 3. If the calcium is ionized into solution and solid gold precipitates, the reactions and assumptions listed are correct. A second testing method is to test the reverse of Reaction 3. If solid gold and magnesium cations in solution are combined, and no reaction occurs, magnesium ranks higher in activity than gold.



#### Reaction 5

##### **Objective:**

Deductively create a brief activity series of metals. Depict the reactions with half reactions and net ionic equations.

##### **Safety:**

- 6M HCl is caustic. Wear splash goggles, coats, and gloves
- Some of the reactions occur quickly, and generate heat

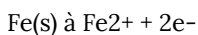
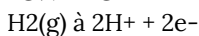
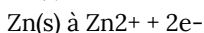
##### **Waste/Housekeeping:**

- Plan how much reagent you will need, and take a little more
- Dispense solutions into beakers and flasks, not into graduated cylinders at the common lab bench
- Don't return solutions to the stock containers. It is better to waste than risk contamination.

### Procedure:

In this lab you will obtain solutions containing the following cations:  $\text{Cu}^{2+}$  (as a sulfate),  $\text{Ag}^{+}$  (as a nitrate),  $\text{Zn}^{2+}$  (as a sulfate), and  $\text{H}^{+}$  (as a chloride). In addition, you will use solid samples of Cu, Zn, Mg, Ag, and Fe. By combining these solutions as described in the introduction, you will rank  $\text{Mg(s)}$ ,  $\text{Fe(s)}$ ,  $\text{Ag(s)}$ ,  $\text{Zn(s)}$ ,  $\text{Cu(s)}$ , and  $\text{H}_2\text{(g)}$  based on their relative activities.

You will also write oxidation and reduction half reactions and a balanced redox reaction. See example reaction 4. The relevant half reactions, written as oxidations are as follows:



1. Dispense approximately 20 mL of the following metal cation solutions into small, labeled Erlenmeyer flasks or beakers: 0.1 M  $\text{CuSO}_4$ , 0.1 M  $\text{ZnSO}_4$ , 0.1 M  $\text{AgNO}_3$ , 6.0 M  $\text{HCl}$ . Designate a beaker for each solution.
2. Design and arrange your test tube rack and test tubes so that each solid metal sample can be submerged in each solution. You don't need to mix the same solid and cation, such as  $\text{Cu}^{2+}$  and  $\text{Cu(s)}$ , for example. You may label test tubes with tape or can draw a key on paper. Design a way to track your reactions. The following example may help.



---

**Data Table 1: Metals Activity**

	<b>Cu<sup>2+</sup></b>	<b>Zn<sup>2+</sup></b>	<b>Ag<sup>+</sup></b>	<b>H<sup>+</sup></b>
<b>Cu(s)</b>	XXXXXXXXXXXX			
<b>Zn(s)</b>		XXXXXXXXXXXX		
<b>Ag(s)</b>			XXXXXXXXXXXX	
<b>Fe(s)</b>				
<b>Mg(s)</b>				

---

---

**Data Table 2: Metals Activity Series**

Most Reactive

Least Reactive

---

3. Add one small piece of solid metal (wire, ribbon, etc.) to the test tubes in your grid. Your observations will be qualitative; you will need just enough solid sample to observe a reaction. You may need to scrape or polish the copper or iron solid if an oxidized layer exists. \*You cannot obtain a chunk of hydrogen. It is noticeably absent from the metal solids yet must be accounted for in your activity series. How do you evaluate the reactivity of H<sub>2</sub>(g)?
4. Fill your test tubes with a few mL of solution. **You only need enough solution to cover the solid sample and observe what happens.**
5. Observe the results. Some combinations react quickly; others may take several minutes. Pay attention to deposition of a solid, evolution of a gas, and temperature changes.

\*Some helpful hints as you evaluate the results of your redox reactions:

- An unreacted combination yields valuable information.
- Deposition of a solid signifies the reduction of a metal.
- If iron is not present in the reaction combination, you are not observing rust or iron (II) oxide. If you see something that looks 'rusty,' what are you observing?
- $\text{H}^+$  reduces to  $\text{H}_2(\text{g})$ , what will you observe if hydrogen is less reactive than a metal?

You will find the most information and can corroborate your findings as you evaluate your reactions from an oxidation standpoint as well as a reduction standpoint. Did solid copper, for example, oxidize in  $\text{Zn}^{2+}$  solution? If so, did  $\text{Zn}(\text{s})$  remain reduced or unreactive in the  $\text{Cu}^{2+}$  solution? Since you don't have solid hydrogen, you will assign  $\text{H}_2(\text{g})$  on the activity series based on which metals reduce  $\text{H}^+$  in solution.

6. Assign your metal samples, **not cations**, from most reactive to least reactive.
7. Write an oxidation half reaction, reduction half reaction and a balanced redox reaction for each observed reaction. You don't need to include unreacted combinations.

### Post Lab:

1. Based on your lab observations:
  1. Which of the metals used in this lab is most likely to be found in an uncombined or metallic state in nature? Does this metallic state represent an oxidized or reduced state? Find and cite an example of the discovery of this metal in an elemental state.
  2. Which of the metals would be most likely to be found combined with other elements as a cation? Does this

represent an oxidized or reduced state? Find and cite an example of the formula and use of this metal in a combined state?

2. Steel, primarily iron metal, is used in the construction of sea going ships and boats. Consider your reactivity data for iron and zinc.
  1. Zn metal is often placed in contact, either directly or through wires, with the steel hull of a ship. Provide a reasoned argument for why this is a widespread practice. Use the concepts that you learned in lab. Be specific in describing the movement of electrons.
  2. Will the zinc need to be replaced periodically? If so, what happens to the missing zinc?
3. Provide a real-world application of the metals activity series.

### **Your Report:**

1. Complete the pre-lab and take the pre-lab quiz in Blackboard before attending lab.
2. Use a lab notebook to take notes and make observations.
3. Create an informal lab document to turn in at the beginning of the next lab. Your lab report should have your name, data arranged in graphs or tables where applicable, conclusive data such as an activity series. Relevant oxidation, reduction, and balanced redox reactions (see Reaction 4). Think about the main point of the lab, or the results that you worked for and be sure to include it or them. Writing the half and redox reactions as outlined (7) is part of your reporting.
4. Numbered responses to any post lab questions. You don't need to re-write the questions.
5. Submit your document **before** your next lab appointment under the assignment tab on your laboratory Blackboard shell.

### **Sources:**

Biello, D. The Origin of Oxygen in Earth's Atmosphere.

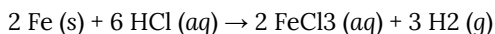
<https://www.scientificamerican.com/article/origin-of-oxygen-in-atmosphere/> (accessed Jul 2, 2020).

(Adapted From) Dieckmann, G., Sibert, J. In *An Atoms First Approach to the General Chemistry Laboratory*; McGraw Hill: New York, NY, 2014; pp 3–12.

# 9. Exp. 9: Calorimetry and Hess' Law

## Pre-lab:

1. When 1.104 grams of iron metal are mixed with 26.023 grams of hydrochloric acid in a coffee cup calorimeter, the temperature rises from 25.2 °C to a maximum of 33.5 °C. The reaction that occurs is given below.

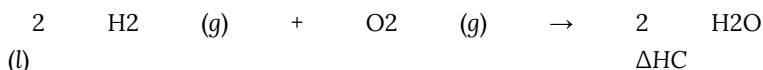
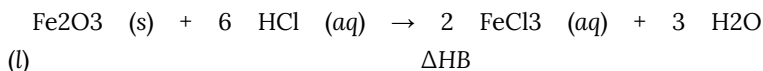
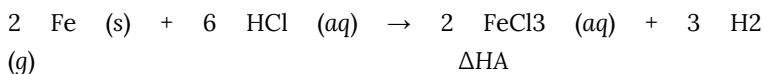


1. a) Determine the amount of heat (in J) absorbed by the reaction mixture. Assume that the specific heat of the reaction mixture is the same as the specific heat of water. The sum of the metal and solution masses will give you the mass of the reaction solution.
1. b) How much heat (in J) was released by the reaction that occurred?
1. c) Is this reaction exothermic or endothermic? Is  $\Delta H_{\text{reaction}}$  positive or negative?
2. d) Under constant pressure conditions (as used in this experiment), the heat released by the reaction equals the reaction enthalpy,  $q_{\text{released}} = \Delta H_{\text{reaction}}$ . Determine  $\Delta H_{\text{reaction}}$  in Joules per gram of metal used (J/g).

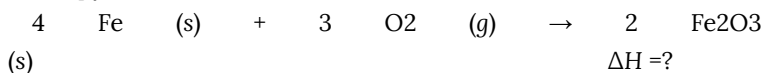
1. e) Determine  $\Delta H_{\text{reaction}}$  in kilojoules per mole of metal used (kJ/mol).

1. f) Determine  $\Delta H_{\text{reaction}}$  in kilojoules per mole of reaction for the balanced reaction equation provided (kJ/mol reaction).

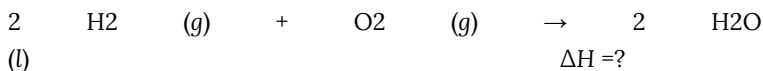
2. Consider the following three reactions:



Show how these equations must be summed together according to Hess's Law to determine  $\Delta H$  for the combustion of iron (target equation shown below). Also show clearly how the  $\Delta H$  values of each of the three reactions must be manipulated to determine the enthalpy of combustion of iron.



3. Using tabulated  $\Delta H_f^\circ$  values in the text appendix two determine the enthalpy change (in kJ) that occurs during the formation of water from its elements:



Note that this value (and the equation) will be used in your data analysis for this lab.

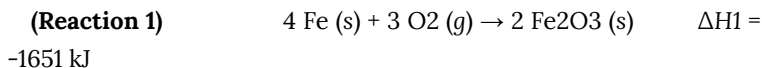
### Objectives:

The objectives of this laboratory are as follows:

- To experimentally measure the  $\Delta H$  values of two reactions using the technique of constant pressure calorimetry.
- To apply these  $\Delta H$  values in a Hess's Law calculation to determine the enthalpy of combustion of a metal

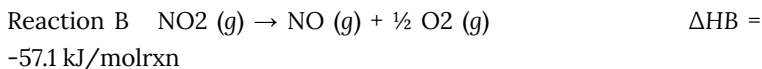
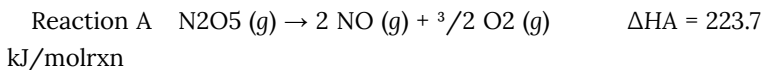
### Introduction:

As you observed in lab two, *The Formation of an Oxide*, the combustion of a metal in oxygen produces the corresponding metal oxide as the only product. Such reactions are exothermic and release heat. For example, the combustion of iron releases 1651 kJ of heat energy for every four moles of iron burned:



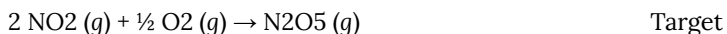
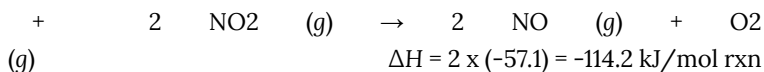
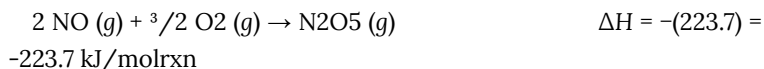
Since it is difficult to measure the enthalpy of combustion of a metal directly, in this lab it will be determined indirectly by applying Hess's Law of Heat Summation. Hess's Law states that the enthalpy change of an overall process is equal to the sum of the enthalpy changes of its individual steps.

*Hess's Law Example:* Determine  $\Delta H$  for the target reaction  $2 \text{ NO}_2 \text{ (g)} + \frac{1}{2} \text{ O}_2 \text{ (g)} \rightarrow \text{N}_2\text{O}_5 \text{ (g)}$  given the following information,



*Solution:* Reactions A and B must be carefully manipulated before they can be summed to produce the target reaction. Reaction A

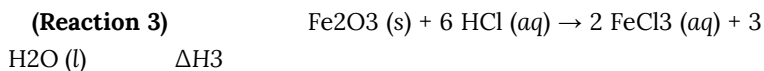
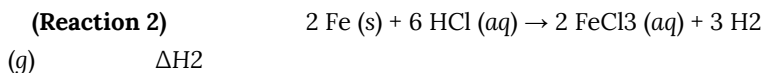
must be reversed, causing a sign change to  $\Delta H_A$ . Reaction B must be multiplied by a factor of 2, causing  $\Delta H_B$  to be multiplied by 2. Only then will they yield the target equation when added together:



Thus,  $\Delta H_{\text{Target}} = -223.7 + (-114.2) = -337.9 \text{ kJ/molrxn}$

In order to use Hess's Law to find the heat of combustion of a metal, it is first necessary to

find reaction enthalpies ( $\Delta H$  values) for equations that can be summed together appropriately. To accomplish this, two reactions will be studied in this lab. In one reaction, a given metal will react with hydrochloric acid producing hydrogen and the metal chloride. In the other reaction, the corresponding metal oxide will react with hydrochloric acid producing water and the metal chloride. For example, the reactions involving iron and iron (III) oxide are as follows:



Since both reactions are exothermic, the heat released ( $q$ ) will be absorbed into the surrounding reaction mixture. As long as the reactions are performed in an insulated container (such as a coffee cup calorimeter), there will be negligible heat exchange with the container walls or outside air. By monitoring the temperature of the reaction mixture when specific quantities of reactants are used, the amount of heat (in J) released by these reactions can be determined by applying the equation:



heat released by reaction ( $-q_{\text{reaction}}$ ) = heat absorbed by reaction mixture

**(Equation 1)**  $(+q_{\text{mixture}}) = (m \times C_s \times \Delta T)_{\text{mixture}}$

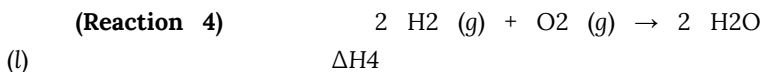
Here  $m$  is the total mass of the reaction mixture (in g),  $\Delta T$  is the maximum temperature change that occurs during the reaction (in  $^{\circ}\text{C}$ ), and  $C_s$  is the specific heat of the mixture (in  $\text{J/g}\cdot^{\circ}\text{C}$ ). Note that since the reactions occur in aqueous solution, it is reasonable to substitute the specific heat of water ( $C_s = 4.184 \text{ J/g}\cdot^{\circ}\text{C}$ ) for the specific heat of the mixture.

Recall that at constant pressure (the conditions of this experiment), the heat released by the reaction equals the reaction enthalpy:

$$q_P = \Delta H$$

Since the heat released by each reaction is proportional to the amount of metal/metal oxide used,  $\Delta H_2$  and  $\Delta H_3$  can be easily calculated per gram or mole of metal/metal oxide used.

It should be noted that reactions (2) and (3) by themselves still cannot be summed to produce Reaction (1). Another reaction is required:



$\Delta H$  for this reaction (the formation of water from its elements) must be obtained from tabulated thermodynamic data,  $\Delta H_f^{\circ}$  appendix two in the textbook. Finally, the reactions (2), (3) and (4) and their enthalpies may be summed together according to Hess's Law to determine the enthalpy of combustion of the given metal (1).

**Safety:**

- Hydrogen gas will be generated during this experiment. Hydrogen is flammable; keep all heat and flames away from your reaction vessel.
- Hydrochloric acid (HCl) is extremely caustic. Wear splash goggles, gowns, and gloves. If HCl comes into contact with your skin or eyes wash immediately under running water for at least ten minutes. The sodium bicarbonate solution by the sinks may be used to neutralize and clean up any acid spills.

**Waste/Housekeeping:**

Dispose of all waste in the labeled container located in Hood #1.

**Materials and Equipment:**

Mg (s), MgO (s), Zn (s), ZnO (s), (Al (s), Al<sub>2</sub>O<sub>3</sub> (s) not used 2020), 1M HCl (aq), 6M HCl (aq), coffee cup calorimeter with lid\*, logger pro temperature probe, glass stirring rod, 100-mL graduated cylinder, 50-mL beaker, utility clamp, stand, electronic balance, and wash bottle.

**Temperature Reading**

Instead of a thermometer, you may use a temperature probe and LoggerPro® software to directly monitor temperature changes over time. Detailed instructions for setting up this system will be provided at the beginning of your lab period. Please note that your experimental procedure will still be the same regardless of the method used to monitor temperature.

**The Heat of Combustion of a Metal/Metal Oxide:**

1. You will be assigned a specific metal/metal oxide pair to investigate by your instructor.  
Record their identities on your report form. Note that you will perform the following procedure for a total of four times, twice with the metal, then twice with the metal oxide.

2. The table below indicates the quantities of reactants to be used for each metal/metal oxide combination. Note that the reactions involving Zn and Al require the concentrated 6M acid.

**Table 1: Recommended Mass of Metal and Metal Oxide**

Mg / MgO	Zn / ZnO	Al / Al <sub>2</sub> O <sub>3</sub> (not used 2020)
0.15 g Mg, 25 mL 1M HCl	0.40 g Zn, 25 mL 6M HCl	0.15 g Al, 25 mL 6M HCl
0.25 g MgO, 25 mL 1M HCl	0.60 g ZnO, 25 mL 6M HCl	0.75 g Al <sub>2</sub> O <sub>3</sub> , 25 mL 6M HCl

3. Use an electronic balance to weigh your empty, dry calorimeter (the two nested Styrofoam® cups). Remove it from the balance, then pour approximately 25-mL of HCl (aq) into it and weigh it again. Record these masses on your report (the difference is the mass of HCl (aq) used).
4. Now weigh the recommended mass of your assigned metal using a tared or recorded beaker or weigh boat. Do not place the metal or oxide directly on the balance.
5. Assemble your equipment as shown in the figure below. A temperature probe will replace your thermometer. You may use a stir rod, or a swirling method. The temperature probe and the stirring rod must be inserted through the holes in the calorimeter lid. The tip of the temperature probe should be immersed in the acid, but not touch the bottom of the calorimeter. Clamp the thermometer in place using a utility clamp.

## 6. **Logger Pro Data Collection:**

1. Connect your temperature probe to the *Lab Quest* interface and open the *Logger Pro* program. The program will display a temperature reading.
2. Under the *Experiment* tab, choose *Data Collection* and increase the time of data collection. 350 seconds should suffice. \*If time runs out during your experiment, re-push *Collect* and *Append to Latest* in order to keep your data.
3. With the probe tip in the acid solution, push *Collect* to get a baseline temperature reading of the solution at thermal equilibrium.
4. Once you have a baseline, add your metal or oxide, and quickly replace the lid. Keep swirling or stirring until the reaction is complete. You will know the reaction is finished when the temperature has reached a maximum and begins to decrease.
5. When you are sure that the reaction is finished, push *Stop*.
6. Under the *Analyse* drop down, select the *Statistics* tab. This will give you the minimum (baseline) and maximum temperatures needed for your calculations\*.

\*Make sure that the min and max in your *Statistics* data makes sense to you. If it does not, you can manually record temperature data by placing the cursor on the minimum temperature and recording the y-axis value at the bottom left of the graph.

7. When finished, dispose of your chemical waste in the labeled container in hood one, rinse the calorimeter, thermometer and stirring rod thoroughly with distilled water, dry, and repeat the experiment again. Once you have completed both trials

with the metal, perform your two trials using the metal oxide using the identical procedure.

Note\*: Nesting your calorimeter in a medium beaker and clamping your temperature probe or thermometer will add stability. The following **example** data tables may help you collect your data.

### **Metal + HCl Reaction**

#### **Experimental Data**

Assigned Metal:

---

**Data Table 1: Collection Data (Metal)**

	Trial 1	Trial 2
Mass of dry, empty calorimeter (g)		
Mass of calorimeter plus HCl (g)		
Mass of HCl used (g)		
Mass of metal used (g)		
Total mass of solution (HCl and metal) (g)		
Initial (equilibrium) temperature of HCl (°C)		
Final (maximum) temperature of mixture (°C)		

---

#### **Data Analysis:**

1) Write the balanced equation for the reaction between your assigned metal and HCl. All balancing coefficients should be whole numbers.

2) Complete the table below with the results of your calculations.

---

**Data Table 2: Calorimetry Calculation Data (Metal)**

	Trial 1	Trial 2
Total mass of mixture, $m$		
Temperature change of mixture, $\Delta T$		
Specific Heat. of mixture, $s$ (use $C_s$ water) of water)		
Heat of mixture, in J (*what sign should this have?)		
Heat of reaction, in J (*what sign should this have?)		
$\Delta H_{rxn}$ in J/g of metal used		
$\Delta H_{rxn}$ in kJ/mol of metal used		
$\Delta H_{rxn}$ in kJ/mol(rxn) for the rxn as balanced in Data analysis#1(above)		
Average $\Delta H_{rxn}$ in kJ/mol(rxn)		

---

3) Show your work for the following calculations using your Trial 1 data only:

- Heat absorbed by mixture, in J
- Heat released by reaction, in J
- $\Delta H_{rxn}$  in J/g of metal used
- $\Delta H_{rxn}$  in kJ/mol of metal used
- $\Delta H_{rxn}$  in kJ for reaction as balanced in (1)

4) Is this reaction exothermic or endothermic? What is your experimental evidence supporting this? Is  $\Delta H_{rxn}$  positive or negative?

Assigned Metal Oxide:

---

**Data Table 1: Collection Data (Metal Oxide)**

	Trial 1	Trial 2
Mass of dry, empty calorimeter (g)		
Mass of calorimeter plus HCl (g)		
Mass of HCl used (g)		
Mass of metal oxide used (g)		
Mass of solution (HCl and metal oxide) (g)		
Initial (equilibrium) temperature of HCl (°C)		
Final (maximum) temperature of mixture (°C)		

---

### Data Analysis

1) Write the balanced equation for the reaction between your assigned metal oxide and HCl. All balancing coefficients should be whole numbers.

2) Complete the table below with the results of your calculations.

---

**Data Table 2: Calorimetry Calculation Data (Metal Oxide)**

	Trial 1	Trial 2
Total mass of mixture, $m$		
Temperature change of mixture, $\Delta T$		
Specific Heat. of mixture, $s$ (use Cs water)		
Heat of mixture, in J (*what sign should this have?)		
Heat of reaction, in J (*what sign should this have?)		
$\Delta H_{rxn}$ in J/g of metal oxide used		
$\Delta H_{rxn}$ in kJ/mol of metal oxide used		
$\Delta H_{rxn}$ in kJ/mol(rxn) for the rxn as balanced in #1(above)		
Average $\Delta H_{rxn}$ in kJ/mol(rxn)		

---

Note: You are not required to show your work for the calculations you performed to complete the above table.

**Post Lab; Enthalpy of Combustion of a Metal:**

1) Write the balanced equation for the combustion (Metal + O<sub>2</sub>(g) → Metal oxide) of your assigned metal. All balancing coefficients should be whole numbers.

2) Using Hess's Law, determine the enthalpy of combustion of your assigned metal as

balanced above. To do this, you will need the balanced thermochemical equations for the two reactions performed in this lab, i.e., the metal and HCl(aq) and the metal oxide and HCl(aq) plus the balanced thermochemical equation for the formation of water from its elements. **Clearly show how these three equations (and their reaction enthalpies) must be combined to give the target combustion reaction.** See the introductory examples for help. Pay close attention to the sign of  $\Delta H_{rxn}$ . \*You will only need to look up



one literature value in this step; this is the enthalpy of formation of water used in the third equation. Be sure to note the number of moles water produced. Why don't you need the enthalpy of formation ( $\Delta^\circ H_f$ ) for  $H_2(g)$  or  $O_2(g)$ ? Provide a clear calculation.

3) Calculate the theoretical value for the enthalpy of reaction, ( $\Delta^\circ H_{rxn}$ ) for combustion of your assigned metal using the tabulated  $\Delta H_f^\circ$  values from the text (Burdge Overby: Atoms First) appendix two. \*You will only need to calculate the enthalpy of reaction value for your overall (summed) reaction using Hess' law. This is your assigned metal combining with oxygen to form a metal oxide, see equation . You will only need to look up one literature value in this step; this is the enthalpy of formation ( $\Delta^\circ H_f$ ) of the metal oxide produced in the overall or target reaction. Why don't you need the enthalpy of formation ( $\Delta^\circ H_f$ ) for the solid metal or oxygen gas? Provide a clear calculation.

4) Determine the percent error in your experimentally determined value for the enthalpy of combustion. See your student lab appendix for formulae and explanation. Discuss the significance of the error and theorize what may have caused the error.

5) Provide a reaction and practical application for a chemical process that is:

1. a) Endothermic
2. b) Exothermic

### **Your Report:**

1. Complete the pre-lab and take the pre-lab quiz in Blackboard before attending lab.
2. Use a lab notebook to take notes and make observations.
3. Create an informal lab document to turn in at the beginning of

the next lab. Your lab report should have your name, data arranged in graphs or tables where applicable, conclusive data such as enthalpies of reaction. Relevant sample calculations, including balanced reactions. **You must clearly show heat calculations, enthalpy calculations and Hess' law manipulations for full credit. These should appear in the results and discussion section.** Think about the main point of the lab, or the results that you worked for and be sure to include it or them.

4. Include any *Data Analysis* and associated questions.
5. Numbered responses to any post lab question. You do not need to re-write the questions.
6. Submit your document **before** your next lab appointment under the assignment tab on your laboratory Blackboard shell.

#### **References:**

Adapted from: Chem 11 Experiments, Santa Monica College  
<http://www.smc.edu/AcademicPrograms/PhysicalSciences/Chemistry/Lab-Manual/Pages/Chem-11-Experiments.aspx>  
(accessed Jul 04, 2020).

# 10. Exp. 10: Toward the Creation of an Airbag

**Pre-Lab:**

**Name:**

-----

1. This lab requires chemistry calculation work and an engineering process. Collaborate with members of your group to come up with some engineering and computational ideas that will give you the edge in the class competition. Read the lab to define your equipment and parameters. Quiz on the following via BB before lab. It may be instructive to run through mock calculations before lab.
2. Consider that a typical driver-side airbag contains 50.0 g of sodium azide, what volume of nitrogen gas would be produced upon ignition assuming sodium azide is the limiting reagent? You may assume a temperature of 25°C and an atmospheric pressure of 760 mmHg for your prelab calculation.
3. You are in competition with other groups, so are encouraged to not share information.

## **Introduction:**

Your lab work this semester has spanned topics as diverse as calorimetry and stoichiometry. This lab will require you to synthesize many of the practical and computational skills that you have acquired during your lab tenure. You will generate your own procedure and data collection and will work in competition with the other lab groups in your section. You will submit this lab as a handwritten lab report today and will write your second formal report on your lab findings for submission as a signature project at a future date. In addition to your handwritten report, your airbag,

calculations, and engineering will be judged in class. Some of the questions that will be asked in an interview are:

- How did you determine the volume of the bag?
- What volume of acetic acid did you choose?
- What mass of baking soda did you choose?
- What was the limiting reagent?
- Did you perform a “test” experiment first?
- Did your reaction go to completion? How do you know?
- Is your reaction endothermic or exothermic? How does this affect the usefulness of this application of your reaction?

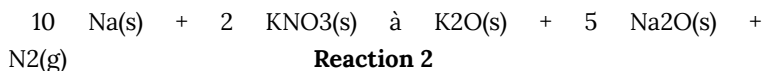
The chemistry process and calculations that you use in creation of an airbag supersede the performance of the airbag itself.

### **How does an Airbag Work?**

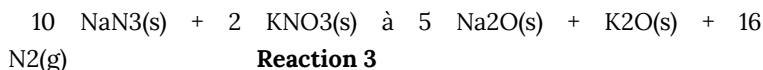
An automobile airbag must both inflate quickly and produce inert, non-flammable products to be both safe and useful. Modern air bags accomplish both through a sodium azide ( $\text{NaN}_3(\text{s})$ ) reaction, which inflates an airbag in less than 0.3 seconds. An open bag design ‘softens’ the large airbag, which is designed to both reduce acceleration and distribute force over a greater area than a steering wheel or car interior, reducing injuries. The chemistry involved is both elegant and precise. Stoichiometric masses of reactants must be used to ensure a complete reaction toward the inert nitrogen gas and silicon dioxide (glass) products according to the following reactions. The first of which is the electrical ignition of the sodium azide explosive when a sensor communicates a collision.



The highly reactive sodium metal produced in reaction one is further reacted with solid potassium nitrate in a second reaction to produce a less reactive oxide and still more nitrogen gas.

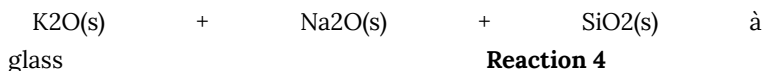


Reactions one and two can be summed to the following reaction.



### Reaction 3

The solid products are then combined with solid silicon dioxide in a fourth reaction to produce a safe and inflammable product, glass.



### Reaction 4

Some elements of the sodium azide detonation can be reproduced in lab. Using an explosive in a lab setting that allows for trials is unsafe. You can, however, safely produce an inert gas in a more controlled (slow) manner. Like the sodium azide detonation, you should consider that all the products of your reaction will be present after a collision.

#### Objective:

- Using provided chemistry concepts learned through the semester, design, calculate, and build a simulation of an automobile airbag.
- Design your own procedure and data collection and calculation.

#### Safety:

- Wear splash goggles at all times. You are creating pressure, and the bag may open.
- 6 M acetic acid is toxic by ingestion and inhalation and corrosive to skin and eyes; avoid contact with bodily tissues.  
\*The acetic acid used is more concentrated than household vinegar.

#### Waste/Housekeeping:

- Dispose of spent airbag liquid waste by dumping the contents

of your bag into the waste container located in hood one then disposing your bag into the regular trash.

- Clean your bench top and any acid spills with baking soda solution.

### **Materials:**

- Two (2) quart Ziplock® bags
- Baking soda (sodium bicarbonate,  $\text{NaHCO}_3(\text{s})$ )
- 6 M acetic acid ( $\text{HC}_2\text{H}_3\text{O}_2$ )
- Any glassware from drawer (within reason and as approved for safety by your instructor)
- Classroom consumables
- Ruler
- Analytical Balance
- Barometer: Pressure sensor
- Alcohol thermometer

### **Procedure/Data Collection/Analysis:**

**Post Lab:**

1. List any chemical reactions used in your airbag design.

2. Calculations: Include any necessary calculations in a logical and readable format.

**Your Report:**

- When ready, demonstrate the collision and reaction of your airbag components to your instructor and answer the interview questions.
- Turn this lab in at the end of class today for a lab grade in conjunction with the class competition and interview.
- Keep your lab notebook with all notes and calculations for use in your second formal lab report, due in one week. Your formal report is your signature project and will be uploaded to your



BB shell under assignments.

### **Formal Report**

You will use the data and observations from this lab to complete your second formal lab report. Please see the *Formal Lab Documents* tab included in your BB shell, including the requirements found document, *How to Write a Formal Lab Report* found in your *Formal Lab Documents* file. To help you reference the file, this incomplete list may help.

- A journal sourced application of the lab technique to be included in your *Introduction*
- Any assumptions made in your *Introduction*
- Any formulas or reactions used in your *Introduction*
- Develop a clear problem statement in your *Introduction*, identifying the information necessary to solve the problem
- Evaluate at least two possible methods that you can use to solve the problem and defend the method you have chosen. For example, you may use two different gas laws to calculate the volumes and masses to fill your airbag and evaluate which is appropriate for the experimental conditions
- An *Experimental* section (bullets, numbered, or paragraph) providing a reproducible procedure, including the actual masses and concentrations that you used
- Tabulated raw data in your *Results and Discussion*
- Sample calculations in your *Results and Discussion*
- Discussion of any differences in theoretical and actual volumes of gas produced
- Findings should be listed in the *Abstract*, *Results and Discussion*, and *Conclusion* and discussed in the *Results and Discussion* section
- Address whether the assumptions listed in the introduction were validated or invalidated in the *Conclusion* section
- Reflect on your results in your *Conclusion*, for example, was

your method the appropriate for the conditions that you tested

- Reference citations (not URL) after the *Conclusion*
- **An originality report will be automatically generated upon your submission of the exam and will be visible to you. Please note that in science writing, direct quotations aren't used. You must, therefore, convey the ideas and theories as well as past work cited by literature without plagiarizing another's work. The originality report will alert me to similar reports across sections and semesters. It is reasonable to report the same measurements and numerical data as your lab partner. Duplicated work of less than 15% is generally accepted in journal writing. Unoriginal content of greater than 25% is generally considered plagiarized in journal writing. Reproduced writing, except as described previously, whether from a literature source or another student represents plagiarism, and the first instance will result in a zero grade for the assignment without the opportunity to re-write the assignment.**

### **References:**

Adapted From Dieckmann, G., Sibert, J. In *An Atoms First Approach to the General Chemistry Laboratory*; McGraw Hill: New York, NY, 2014; pp 159–164.

# Appendix

## Appendix I

How good is a measurement?

In science, when we measure something, we report our findings so that other scientists can confirm what we've measured. There are three factors that go into reporting a measured value:

1. We must report how sensitive our equipment is.
1. We must report how confident we are that the measurement was performed correctly. This is a measure of precision.
1. We must report how accurate the measurement is by comparing to a known value.

We use significant figures to report our sensitivity; we use a statistical measure – called the 95% confidence limit – to report how precise, or confident, we are in our measurement; we use percent error to report on how accurate our measurement is.

These three factors are summarized nicely when we report our measurement using the following format:

$5.2 (\pm 0.6) \times 10^{-5} \text{ s}^{-1}$  with a percent error of 1.9% as compared to the literature value of  $5.3 \times 10^{-5} \text{ s}^{-1}$ .

This gives us our value ( $5.2 \times 10^{-5} \text{ s}^{-1}$ ), that we are 95% confident our measurement will lie within  $(\pm 0.6) \times 10^{-5} \text{ s}^{-1}$  of the measured value, and that the measured value differs by 2.3% as compared to independent measurements of this value.

It is important to include both the 95% confidence limit and the percent error when reporting your value. The 95% confidence limit tells you how confident you are that you've done a good job in making the measurement. The smaller the confidence interval the more precise your measurement is. For example, a 95% confidence limit of  $\pm 0.1$  isn't as precise as a 95% confidence limit of  $\pm 0.001$ . Although in both cases we are 95% confident the measured value will lie within the limits, since the second limit is 100 times smaller than the first we can be confident that the second measurement was more precise.

However, what we don't know from this measurement is if there was a systematic error that we made when collecting the data. A systematic error is an error that shifts our value by a set amount. For example, if you were to weigh yourself every day, but someone left a five-pound weight on the scale that you didn't notice, then you would have a precise measurement of your weight, but it would always be five pounds heavier than your true weight. This is a systematic error. To account for this, we compare the value we measure with a literature, or known value, using percent error. In this example we would measure our weight using several different scales and then compare the results using percent error. We would quickly be able to determine which scale was incorrect by looking at the percent errors between each scale.

In the following pages you will be provided instructions on how to properly use significant figures (Appendix II), calculate the 95% confidence interval (Appendix IV), and how to calculate percent error (Appendix III).

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## Appendix II

### Note on significant figures

Significant figures are critical when reporting scientific data because they give the reader an idea of how sensitive your measurement was. Before looking at a few examples, let's summarize the rules for significant figures.

1. ALL non-zero numbers (1,2,3,4,5,6,7,8,9) are ALWAYS significant.
1. ALL zeroes between non-zero numbers are ALWAYS significant.
1. ALL zeroes, which are SIMULTANEOUSLY to the right of the decimal point AND at the end of the number, are ALWAYS significant.
1. ALL zeroes to the left of a written decimal point and in a number greater than or equal to 10 are ALWAYS significant.

A helpful way to check rules 3 and 4 is to write the number in

scientific notation. If you can/must get rid of the zeroes, then they are NOT significant.

Examples: How many significant figures are present in the following numbers?

---

Number	# of Significant Figures	Rule(s)
48,923	5	1
3.967	4	1
900.06	5	1,2,4
0.0004 (= 4 E-4)	1	1
8.1000	5	1,3
501.040	6	1,2,3,4
3,000,000 (= 3 E+6)	1	1
10.0 (= 1.00 E+1)	3	1,3,4
5100.	4	1,4
5100	2	1

---

\*\*\*Note\*\*\*

Conversion factors, such as 1 in = 2.54 cm, are exact. Since conversions are exact they have an infinite number of significant

figures. Your significant figures will always be limited by your measurement, never by any conversion you apply.

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## Appendix III

### Accuracy – Percent Difference and Percent Error

Scientists want to compare their results with those of others, or with a theoretically derived prediction or literature value. The closer independently measured values are to each other, the more confident we can be that the measured values are correct.

Percent Difference: Applied when comparing two experimental results, E1 and E2, neither of which is an accepted literature value. The percent difference is the absolute value of the difference between E1 and E2 divided by the average of the values times 100. This comparison is used when no literature value is available and you want to compare a result measured in two different ways.

% Difference =

$$\frac{|E_1 - E_2|}{\frac{E_1 + E_2}{2}} \times 100$$

Percent Error: Applied when comparing an experimental quantity, E, with a theoretical or true value, T, which is considered the “correct” value, for example, an accepted literature value. The percent error

is the absolute value of the difference between T and E, divided by the “correct” value times 100.

% Error =

$$\frac{|T - E|}{|T|} \times 100$$

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## Appendix IV

If you use a Mac, you can download StatPlus for free to perform the data analysis that Excel does.

### Precision – Calculating 95% confidence interval error

A reported data value by itself is meaningless unless it is accompanied by a confidence interval, or the precision to which you are confident you know the value. For example, a grain of sand may be reported to have a diameter of 52 microns but how sure are you of this value? If you used a ruler with 1 mm gradations (1 mm = 1000 microns) then anything less than 1 mm you’re guessing at. If this value were reported with error, it would be 52 ( $\pm 1000$ )  $\mu\text{m}$ . In this case the 52-micron measurement is garbage because it is smaller than our ability to measure it precisely. However, if the measurement were made using red light that has a 1000 nanometer wavelength (~1000 nanometers = 0.01 microns) then the value reported with error would be 52.48 ( $\pm 0.01$ )  $\mu\text{m}$ ; a very precise measure of the diameter of the sand grain!



In science, when an experimental value is reported it is customary to report how confident in the value the experimenter is. The level of confidence that is generally accepted is 95%. Therefore, error is reported to a 95% confidence level. This means that the experimenter is 95% confident that the true value falls within the error range determined experimentally. For example, if you determined experimentally the molar mass of sodium chloride at the 95% confidence level was 59.2 ( $\pm 0.8$ ) g/mol, you would be 95% confident that the true mass of sodium chloride was between 58.4 and 60.0 g/mol, assuming that there was only random error in your measurements. When compared to the true mass (58.44 g/mol) you would note that the true molar mass falls within your error range and are 95% confident that your experimental method is valid.

To calculate error at the 95% confidence level the following formula is used:

$$95\% \text{ CL} = 1.96 \sigma \sqrt{N} \quad \text{---} \quad 95\% \text{ CL} = 1.96 \diamond N-1$$

In this formula,  $\sigma$  is the standard deviation and  $N$  is the number of measurements. The standard deviation can quickly be calculated using spreadsheet software such as Excel. For example, if you had the following data:

---

A	
1	Mass, g
2	45.11
3	43.97
4	44.46
5	45.45
Stdev 0.66133	

---

In Excel the standard deviation,  $\sigma$ , would be calculated by typing the following formula into cell A6, =stdev(A2:A5). After hitting enter, the result 0.66133 is displayed in cell A6. The 95% confidence interval would then be  $1.96 \times (0.66133) / \sqrt{3} = 0.74837$ , where  $N = 4$  in this example. Page Break

## Appendix V

### When to use linear regression versus standard deviation

During the Ideal Gas Law experiment, you will need to perform a linear regression on the data to determine the error in your experimental gas constant. A linear regression is required because the experimental conditions change from run to run. Fortunately, when graphed the data lie along a line. In this case, a linear regression analysis is used to determine the error in the slope of the line that has been fit to the data.

In the case where the experimental conditions do NOT change from each run, like the freezing point depression experiment where you repeat the same measurement several times, the standard calculation for 95% confidence interval described in Appendix IV is used.

To perform a linear regression analysis in Excel, you must use the Data Analysis command within the Analysis group on the Data tab. You may have to load in the Data Analysis ToolPak if you do not see the Analysis group on the Data tab. See your software help for information on how to load in the Data Analysis ToolPak.

Once you have access to the ToolPak, select Linear Regression from the list and then choose the appropriate dataset for the dependent (y) and independent (x) variables. Be sure that the confidence interval is set to 95%. Once you click “OK” Excel should output the calculated results to a new workbook. You should see something similar to the following:

	Coefficients	Standard Error	LCL	UCL	t Stat	p-level	H0 (5%) rejected?
Intercept	-0.08292	0.11036	-0.45429	0.28845	-0.75133	0.48629	No
Slope	0.08268	0.00173	0.07685	0.08851	47.74243	0.	Yes
T (5%)	3.36493						
LCL – Lower value of a reliable interval (LCL)							
UCL – Upper value of a reliable interval (UCL)							

The slope of the line, which is under the coefficients column, is 0.08268. The LCL and UCL are the lower and upper range of your error, respectively.

To report your error at the 95% confidence level, subtract the LCL from the UCL and divide by 2. For the example,  $(0.08851-0.07685)/2 = 0.00583$ . When reported to the correct number of significant figures, this gives 0.083 ( $\pm 0.006$ ) L-atm-mol-1-K-1 (See Appendix VI).

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Appendix VI

How to report experimental values with 95% confidence level error

When you report your data in a lab write-up, you should report experimentally determined values using the following format:

[experimental value] [(± 95% confidence level)] × [magnitude] [units]  
@ [temperature]

The experimental value is the value you measure in the experiment. This could be the slope of a linear regression fit or the averaged value of a repeated set of measurements.

For example, if the averaged rate constant,  $k$ , was determined to be  $5.2338 \times 10^{-5} \text{ s}^{-1}$  with a 95% confidence of  $6.3085 \times 10^{-6} \text{ s}^{-1}$  at  $23.4 (\pm 0.2) ^\circ\text{C}$ , you should report the result as,

$5.2 (\pm 0.6) \times 10^{-5} \text{ s}^{-1} \text{ at } 23.4 (\pm 0.2) ^\circ\text{C}$

Here, [experimental value] = 5.2

[(± 95% confidence level)] = ( $\pm 0.6$ )

× [magnitude] =  $\times 10^{-5}$

[units] =  $\text{s}^{-1}$

@ [temperature] =  $23.4 (\pm 0.2) ^\circ\text{C}$

There are several key things to note about this format.

- 1. The error is only reported at the 95% confidence level and only to one significant figure. The error can be reported to two significant figures ONLY if the second figure is a 5, for example 0.15.
- 1. The experimental value is reported to the same number of decimal places as the error. For example, 5.2 (±0.6). Both have one decimal place.
- 1. The error value is put into parenthesis directly after the experimental value and both the experimental value and the error are reported to the same magnitude (i.e. the same power of 10).
- 1. If the experimental value has units associated with it, they would be added after the magnitude multiplier.

Examples:

Experimental Value	95% Confidence Level error	Correct reporting notation
$4.2246 \times 10^{-5}$	$3.9001 \times 10^{-7}$ (= 0.039001× 10 <sup>-5</sup> )	$4.22 (\pm 0.04) \times 10^{-5}$
5206	139	5200 (±100) or 5.2 (±0.1) × 10 <sup>3</sup>
45.228	$15.26 \times 10^{-2}$ (= 0.1526)	45.2 (±0.2) or 45.23 (±0.15)

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## Appendix VII

Note on how to make a graph using Excel

Most of the data that you will plot using Excel is what's called pairwise data. This is data that is collected in pairs, i.e. xy data (like time and temperature). To plot this type of data we will arrange all the x data in a column and corresponding y data in the column just to the right of the x data, making sure to keep the ordering of the data pairs correct. Then from the main menu, select "Insert" followed by "Chart..." from the drop down list. From the options listed, select "Scatter" and from the subset of charts select "Marked Scatter". A blank graph will appear. Right click inside the graph and choose "Select data..." from the list. Click on the "Add" button under Series and then select the x values and y values that you wish to graph. Click "OK".

In general, we will always select a scatter plot because our x data will not be in uniform steps of 1, 2, 3, etc. A scatter plot allows for the x data to have non-uniform steps. Also, in general, we don't want Excel to draw a line through our data, we will do that using a trendline. This allows us to better judge how well our data fits a specific model or theory. It is also good form to use hollow markers to represent your data so that you can see if the trendline goes through the middle of your data points.

To add a trendline, right click on a data point in your graph and choose “Add trendline...” from the list. Choose the type of fit you wish to use (usually linear), and then select “Options” from the left-hand pane. Be sure to select the checkbox next to “Display equation on chart”. In addition, you can extend the line forward or backwards by using the Forecast feature.

Note that a trendline and a linear regression fit are different. A trendline is a visual method that draws a “best fit line” on top of the data. A trendline does not provide the error in the fit. To calculate and report the 95% confidence error, you must perform a linear regress fit (Appendix V).

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## Appendix VIII

Note on how to fit data and subsets of data using Excel

If you only wish to fit a portion of your data, rather than the entire data set, you need to create a series that contains the subset of data that you wish to fit. For example, if you graphed the following data and fit a line through all the points, your fit wouldn't be very good.



---

10 100

15 90

20 80

25 70

30 65

35 62

40 60

45 58

50 55

---

However, we can create a series for each subset of the data we wish to fit. In this example we will create two series: one will contain the first four paired data points, and the second will contain the last five paired data points.

To create a series, right click on a data point in the graph and select “Select data...” from the pop up menu. Under Series in the dialog box that pops up, click “Add”. Now select the x and y values from the data that you wish this series to contain. Repeat this process for additional series if needed. Each series can now have a trendline fitted to it. The graph now looks like this.

Note that the forecast feature was used to extend both trendlines so that they overlap.

If you choose, you can set the markers to be the same so that you can't tell you have multiple series, however, this is purely aesthetics.

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Appendix X

Basic Vernier Probe set-up using LoggerPro with troubleshooting

(This needs to be generalized) Open LoggerPro on the computer and connect the temperature probe to the Vernier USB interface. The software should automatically detect the temperature probe and a temperature reading should show up in the lower left hand corner of LoggerPro. Go into Settings and set the collection parameters for 1 point per second for 900 seconds.

If the probe is not detected, try unplugging the temperature probe and the Vernier USB hub. First reconnect the Vernier hub. It should play some short audible beeps, and then plug in the temperature probe. If the probe is still not detected alert your instructor.

Calibration of pH meter

In order for the pH electrode to correctly display pH it must be calibrated using a set of solutions with known pH. To calibrate the electrode perform the following steps.

1. Open LoggerPro on the computer.

1. Once the program is open, plug the pH electrode into the LabPro controller unit. Be sure that you plug into the controller unit that is connected to the computer you are using.
1. LoggerPro should auto-detect the electrode and pH should be displayed in the lower left hand corner. If the electrode is not auto-detected, perform the following steps otherwise skip to step 4:
  1. Under the Experiment menu, select Set Up Sensors and then select Show All Interfaces.
  1. A diagram of the LabPro controller unit will appear. Click on the dropdown box corresponding to the channel that the sensor is plugged into and select Add Sensor.
  1. Choose the pH sensor.
  1. To start calibration, under the Experiment menu select Calibrate and then select LabPro:pH from the flip out window. A menu will appear.
  1. To prepare the electrode for calibration perform the following washing procedures EACH TIME THE ELECTRODE IS PLACED INTO A NEW SOLUTION.
    1. Remove the electrode from the solution it is currently in and rinse the glass bulb at the tip using deionized water from a rinse bottle.
    1. Gently blot the tip of the electrode with a KimWipe to remove most of the water. You do not need to remove all the water.
    1. Place the electrode into the new solution. Be sure that the glass bulb at the tip of the electrode is completely submerged

in the solution.

1. You will be using pH 4.0 and 7.0 standard calibration solutions for this experiment. Place the cleaned electrode into the calibration buffer you have chosen.
1. Click Calibrate Now from the displayed menu.
1. Gently stir the electrode in the first standard solution while watching until the voltage displayed in the calibration window stabilizes. This should take about 30 seconds. Be sure not to warm the solution with your hand as this will throw off the calibration.
1. Once the voltage has stabilized enter the pH value for the standard solution you are using into the box labeled Reading 1 and press Keep.
1. Remove the electrode from the standard solution, rinse and dry the tip of the electrode, and repeat steps 6 – 9 for the second standard solution.

After you have completed the calibration procedure you must check the accuracy of the calibration you just performed. Choose any of the standard solutions and insert the electrode into this solution. The reading on LoggerPro should match the labeled value of the buffer within 0.1 pH units. If the readings do not match you must repeat the calibration procedure. You can use these calibration buffers to check your electrode calibration throughout your experiment to ensure accurate pH measurements. The pH electrode must be stored upright in its storage solution. Place the electrode and storage container in a beaker to keep it from falling over when not in use.

Page Break

## Appendix XI

### How to condition a volumetric pipette

In order to obtain the most precise results for an experiment that uses volumetric glassware, such as a volumetric burette or a volumetric pipette, the glassware must be conditioned before use. Conditioning removes any contaminants in the glassware, it removes any residual water that may dilute your stock solution thereby changing the known concentration, and conditioning ensures accurate delivery of the correct volume.

To condition a volumetric pipette, perform the following steps:

1. Fill an appropriate sized beaker with just enough stock solution to cover the bottom of the beaker to a height of 1 mm.
1. Swirl the stock solution around in the beaker and then dispose of this solution into the appropriate waste container.
1. Fill the beaker with about 1.5 – 2 times the volume of the volumetric pipette you will be using. Note: Do not use too large a beaker or you will aspirate air into the pipette. For example, a 25.00 mL pipette should use a 100 mL beaker.
1. Fill the pipette about half full with the stock solution you will be dispensing.
1. Roll the pipette around in your hands in order to coat the

inside of the glassware.

1. Drain the pipette completely, placing the solution into an appropriate waste container, and use a pipette bulb to blow out the rinse solution from the tip of the pipette.

The pipette is now fully conditioned.

Use the following steps to fill and accurately deliver an amount of solution from the pipette.

1. Refill the dispensing beaker with 1.5 – 2 times the volume of the volumetric pipette you are using.
1. Squeeze the pipette bulb to void it of air and then place it onto the top of the empty pipette. DO NOT jam the bulb onto the top of the pipette. You will need to remove the bulb later and if jammed on it could be difficult to remove.
1. Slowly draw up your stock solution into the pipette using the pipette bulb. You must KEEP THE TIP OF THE PIPETTE SUBMERGED in the stock solution. DO NOT allow the tip of the pipette to pull in air or it was aspirate into the pipette bulb and contaminate the bulb and your stock solution.
  - It is helpful to keep the tip of the pipette touching the bottom of the beaker and slightly tip the pipette to allow liquid to flow into the pipette. This helps to reduce the chance of aspirating air into the pipette.
1. Fill the pipette with solution until the solution is above the etched line in the neck of the pipette. GO SLOW when the solution starts to enter into the neck of the pipette as it will rapidly ascend the narrow tubing. DO NOT allow any solution

into the pipette bulb.

1. Remove the thumb of your dominant hand from your glove. For example, if you are right handed remove your right thumb only from your glove. Use your thumb to slide the pipette bulb off the pipette and then to quickly cover the top of the pipette so no solution leaks out.
1. With the tip of the pipette pressed down against the bottom of the beaker, SLOWLY twist your thumb to allow the solution to drain down until the bottom of the meniscus is just touching the etched line in the neck of the pipette. Remember to keep your eye at the level of the etched line to remove parallax and increase your precision.
1. Transfer your pipette to where you wish to dispense the solution and allow it to drain. DO NOT blow out the last drop of solution from the pipette. The pipette is designed to accurately deliver its specified volume with solution remaining in the tip of the pipette.

## Appendix XII

### How to condition a volumetric burette

To condition and prepare a volumetric burette for use, perform the following steps:

1. Fill a 250 mL beaker with just enough stock solution to cover the bottom of the beaker to a height of 1 mm.
1. Swirl the stock solution around in the beaker and then dispose of this solution into the appropriate waste container.

1. Fill the beaker with about 150 mL of your stock solution. Close the stopcock on the burette and use a funnel to fill the burette about one third full.
1. Slowly tip the burette on its side and drain the solution out of the top end of the burette into an appropriate waste container. DO NOT drain the solution through the tip of the burette.
1. Place the burette into a burette stand and clamp. Using a funnel, fill the burette to near the top. Check for leaks around the tip of the burette. If the burette is leaking report it to your instructor, get a new burette, and go back to step 3.
1. Open the stopcock and allow some solution to drain into an appropriate waste container. WHILE THE SOLUTION IS DRAINING, you should tap the stopcock of the burette with your finger aggressively to dislodge any bubbles from the tip of the burette. You should see bubbles flow down through the burette tip. Failure to remove bubbles from the tip will cause error in your measurement, as the burette will not dispense the correct volume of solution.
1. Once all bubbles have been removed from the tip of the burette, close the stopcock. Refill the burette until your solution is ABOVE THE ZERO LINE.
1. Remove the funnel from the top of the burette.
1. Open the stopcock and drain the burette until the solution is below the zero line. BE SURE TO LOOK AWAY FROM THE BURETTE when you close the stopcock so that you do not set your burette to any specific volume. Setting your burette to a specific volume will bias your results.
1. Read the bottom of the meniscus to the hundredths place. For



example, your initial burette volume may be 0.17 mL. You must estimate the last digit. You can place a piece of white paper behind the burette to help visualize the meniscus and the gradations on the burette. It is important to have your eye at the same level and the burette gradations to minimize parallax in your measurement.

# Lab Reporting: How to Write an Informal Lab Report

## How to Write an Informal Lab Report:

Labs turned in with formatting other than an informal report such as filling in the blanks and data tables from the printed lab, etc. will start grading at half credit.

In this course, two labs will require a formal lab write up and eight labs will require an informal lab report write up. The informal report will focus on the results and analysis from your lab. Although it is abbreviated, it contains tabulated data as well as writing including complete sentences and short paragraphs where required. First person writing is acceptable in an informal lab report. In general, your informal report should be organized with the following clearly labeled sections organized in this order.

### Tabulated Raw Data:

Your data validates your findings and can help problem solve errors in your results. Include your raw data such as mass, volume, temperature, concentration, etc. Sample data tables are often included in your lab, but you must create organized and labeled data tables that match your procedure. A linear regression displays corresponding data points as a line and must be displayed with axis labels and an equation of the line. Include the number or letter of an unknown with your raw data.

### Formulas and Equations:

Include any chemical equation used in your lab work. If an equation is included as background or an example, but not used for calculation, you may omit it. You must include mathematical

equations used such as density, wavelength and diffraction, percent composition, calorimetry, ideal gas, etc. Stoichiometric functions and Hess' law formula additions are not equations, and your sample calculations suffice in this regard.

#### Sample Calculations:

You must include sample calculations. If you repeat a calculation more than once, for multiple trials, etc. you may include one sample calculation. Stoichiometric analysis should be included. You should include a sample calculation for statistical analysis such as percent difference, relative standard deviation, and percent error as well.

#### Findings:

Include your findings with correct significant figures and units. Why did you perform the lab and what results did you produce? Examples of your results include density, percent composition, a chemical formula or ratio, width, concentration or identity of an unknown, energy per mole, or the mass and volume needed to create a product. Include the letter or number of an unknown once again in this section. Include statistical analysis. Examples of statistical analysis include percent error, percent difference or relative standard deviation. You must have at least two quantitative data points to perform statistical analysis. Not every lab will, therefore, include statistics. Discussion of your findings is appropriate in this section. Were there errors that account for a deviation from expected results? What caused the errors, and in which direction did the data skew?

#### Post Lab:

Include numbered responses to lab questions. Do not re-write the question. Use complete sentences or short paragraphs as appropriate. You do not need to include the pre-lab questions in your post lab report. They will be addressed in the pre-lab quiz.

In addition to these guidelines, each lab contains instructions specific to the lab's measurements and analysis.

# Example Informal Lab Report: Copper Cycle

Lab 6: The Chemistry of Copper

Mike Fisher: Chem 111L-001

Tabulated Data:

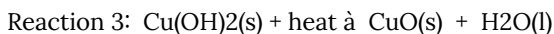
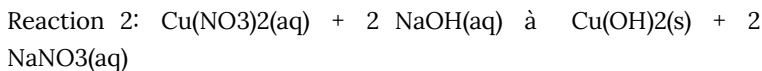
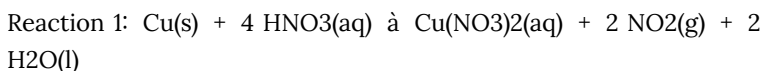
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Data Table 1: Mass Measurement of Copper Sample

Initial Solid Copper Wire	0.513 g
Dry Büchner Top w/ #5 Whatman Filter	5.217 g
Final Sample Drying 1 in Büchner Top	5.709 g
Final Sample Drying 2 in Büchner Top	5.604 g
Final Sample Drying 3 in Büchner Top	5.605 g

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Formulas and Equations:



Reaction 4:  $\text{CuO(s)} + \text{H}_2\text{SO}_4\text{(aq)} \rightarrow \text{CuSO}_4\text{(aq)} + \text{H}_2\text{O(l)}$

Reaction 5:  $\text{CuSO}_4\text{(aq)} + \text{Zn(s)} \rightarrow \text{ZnSO}_4\text{(aq)} + \text{Cu(s)}$

\*No mathematical equations used

Calculations:

Mass copper final:

$$5.605 \text{ g} - 5.217 \text{ g} = 0.388 \text{ g Cu(s)}$$

Percent recovery:

$$0.388 \text{ g} / 0.513 \text{ g} * 100\% = 75.6 \% \text{ recovery}$$

Findings with Discussion:

75.6% of the copper sample was recovered. I am confident that the sample is dry because successive oven dryings and weighing yielded consistent mass. No zinc granules are visible in the final product, suggesting a pure copper sample. Some copper oxide was lost in the copper oxide filtration step. In addition, our dried sample 'jumped' due to some apparent static discharge causing some sample loss. Our percent recovery could be improved by washing the copper oxide step with the sulfuric acid and care to discharge static before handling the final dried sample.

Numbered Questions Within the Lab:\*(not every lab includes such questions)

1. The reaction between solid copper and nitric acid is exothermic. The resulting solution is warm to the touch.
1. A blue/green color resulted from the nitric acid addition. Solid copper oxidized to  $\text{Cu}^{2+}$  in the copper (II) nitrate solution. All

of the blue/green solutions must have this ion.

1. The blue litmus paper turned red in the presence of a solution made acidic by the addition of nitric acid.
1. A cloudy blue precipitate formed.
1. The red litmus paper turned blue in the presence of basic sodium hydroxide.
1. The blue litmus paper turned red in the presence of sulfuric acid.
1. The solution is once again a blue to blue green color. The copper (II) ion must again be present to produce the color.
1. 75.6% of the copper was recovered.

#### Post Lab:

1. Copper wire is cut into smaller pieces in order to increase surface area and speed up the oxidation reaction.
1. Dissolving refers to ion solubility, or separation of ions with a polar solvent. It could also refer to non-polar solute and solvent interactions. In each case, solubility is a physical process. In our lab, Cu(s) oxidized to Cu<sup>2+</sup> ion in the presence of HNO<sub>3</sub>. This represents a new species and a chemical change.

1.  $\text{NO}_3^-$  and  $\text{Na}^+$  are the only ions present in the solution assuming that  $\text{CuO(s)}$  was filtered out and that the solution was neutralized prior to precipitation.

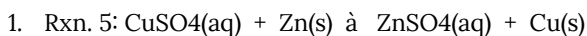
1. b. Double Replacement

c. Decomposition

d. Double Replacement

e. Single Replacement

1.  $\text{Cu(s)}$  is oxidized to  $\text{Cu}^{2+}$  ion in reaction one. In the same reaction nitrogen is reduced from a +5 oxidation state in  $\text{HNO}_3(\text{aq})$  to a +4 oxidation state in  $\text{NO}_2(\text{g})$ .



In reaction five, solid zinc is oxidized from a neutral state to a +2 oxidation state in  $\text{ZnSO}_4(\text{aq})$ . In the same reaction  $\text{Cu}^{2+}$  is reduced from a +2 oxidation state in  $\text{CuSO}_4(\text{aq})$  to a neutral oxidation state in solid copper.

1. Our second filtration left a black copper oxide stain on the Whatman #5 filter. In addition, some of our dried final solid copper 'jumped' in contact with some static charge and was lost.



1. Any water remaining in the sample after drying or zinc remaining after the hydrochloric acid addition would cause the final copper mass to measure high.

\*Note: This example lab report conforms to the given lab. Please refer to How to Write an Informal Lab Report as well as your specific lab requirements.