# Valencia College Osceola/Lake Nona Campus 

# Experimental Laboratory Manual <br> for General Chemistry-I 

First Edition

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2012
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## Timothy Barnett

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# Experiment \#1: <br> Density of Rubber and Four Pure Metals 

## Introduction to Density:

1) Density is an important physical feature of matter and is equal to its mass per unit volume. $(d=m / \mathrm{V})$
2) The International System of Units (SI) maintained by the International Committee of Weights and Measures states that the proper unit for density is kilograms per cubic meter $\left(\mathrm{kg} / \mathrm{m}^{3}\right)$.
a. This unit of measure is not practical for both solids and liquids so the conversion is commonly made to $\mathrm{g} / \mathrm{cm}^{3}$.
b. $\mathrm{kg} / \mathrm{m}^{3}=\mathrm{kg} / \mathrm{m}^{3} \times(1000 \mathrm{~g} / 1 \mathrm{~kg}) \times(1 \mathrm{~m})^{3} /(100 \mathrm{~cm})^{3}=1 \times 10^{-3} \mathrm{~g} / \mathrm{cm}^{3}$.
c. In addition, $1 \mathrm{~cm}^{3}=1 \mathrm{~mL}$, so density can be reported as $\mathrm{g} / \mathrm{mL}$.
i. For sensible purposes, solid densities are reported as $\mathrm{g} / \mathrm{cm}^{3}$ and liquid densities are reported as $\mathrm{g} / \mathrm{mL}$.
d. Gases, however, have reported densities in $g / L$.
3) Density is a property of matter can help identify unknown substances.
a. If the mass and volume are measured, the density can be calculated.
4) Since density is characteristic of a substance, it can help assess purity.
a. The purity of a substance can be assessed by comparing the determined value to the actual value of density.
5) The density is a conversion factor between mass and volume.
6) Density is an intensive property of matter.
a. Intensive properties are independent of the amount of a substance.
b. Extensive properties depend on the amount of a substance such as mass, volume, and moles.
7) To determine the density of all solid materials in this experiment, masses will be recorded via a balance and volume will be recorded by water displacement in a graduated cylinder.
a. This data will be plotted and the density of the material will be determined by determining the slope of the best-fit straight line.

## Plotted Data to Determine Density:

1) For each substance analyzed, masses and volumes displaced in water will be recorded.
2) A graph of cumulative mass versus the cumulative volume displaced will be produced using a computerized graphing program.

| A | B |
| :---: | :---: |
| 0.00 | 0.000 |
| 1.45 | 5.228 |
| 2.80 | 10.162 |
| 3.40 | 12.991 |
| 3.95 | 14.892 |
| 4.80 | 21.205 |

a. The variable of cumulative mass will go on the $y$-axis and the cumulative volume displaced will be placed on the x -axis.
i. If using Microsoft Excel, place volume data into cell A and mass data into cell B as indicated.
b. Create a scatter plot using the chart generator.
c. This plot will be given an appropriate title and both axes will be labeled with the variables and units.
d. Using a linear regression, the best-fit straight line will be produced.
i. This can be added to the graph by right-clicking any point and selecting the "Add Trendline" option in Excel.
e. After the best-fit straight line is produced, an equation for the line will be reported on the graph using the following generic formula: $y=m x+b$.
f . The density of the material is the slope of the line indicated by the coefficient of $x$.
g. If the equation is not available, to calculate the slope, two points that fall on the line will be selected and a calculation of the density will be the change in $y$ over change in $x$ via the formula: $\Delta y / \Delta x=\left(y_{2}-y_{1}\right) /\left(x_{2}-x_{1}\right)$.
$h$. The value for the $y$-intercept, or the point where the line intersects the $y$ axis, should be zero. This is because when an object has zero volume it will also have no mass.
i. If your $y$-intercept is not zero, then experimental error is apparent.
2) An example graph is shown below using data from table A.


## Procedure for Part 1: Density of Rubber Stoppers

1) Add 50.0 mL of distilled (or deionized) water to a 100.0 mL graduated cylinder.
a. Measure the exact volume of water added by reading from the lowest point of the meniscus and record to the nearest tenth or first decimal place.
2) Place graduated cylinder onto a top-loading balance or analytical balance.
a. Once a stable mass is displayed, tare the balance (or set the mass to zero).
3) Obtain a combination of 5 rubber stoppers with sizes of $00,0,1$, and/or 2.
4) Add one stopper at a time to the graduated cylinder and record the mass (or cumulative mass) of the stopper(s), the total volume to the nearest tenth, and the volume of water displaced.
a. Be careful not to splash out any of the water.
b. Record the mass indicated on the balance using all decimal places provided.
5) Continue to add rubber stoppers (without removing the previous ones) and record the cumulative masses, total volumes to the nearest tenth, and volumes of water displaced.
6) Produce a graph of the cumulative mass versus the cumulative volume displaced using a computerized graphing program.
a. Compare the experimental/observed density to the true/theoretical density and calculate a percent error.
b. Percent error $=[($ true value - experimental value $) /$ true value $] * 100 \%$
i. Percent error cannot be a negative value. Take the absolute value of the number if necessary.
c. For the true/theoretical density, cite references for sources using proper bibliographic technique.

## Procedure for Part 2: Density of Four Pure Metals

1) The four pure metals provided will be aluminum, copper, nickel, and zinc.
a. If one of the metals above is unavailable, lead or iron will be used as an alternative.
2) Place 12.00 mL of distilled (or deionized) water into a 25.00 mL graduated cylinder (that is marked with 0.2 mL increments.)
a. Measure the exact volume of water added by reading from the lowest point of the meniscus and record to the nearest hundredth or second decimal place.
b. If a 25.00 mL graduated cylinder is unavailable, add 25.0 mL to a 50.0 mL graduated cylinder.
i. For this instrument, measure the exact volume of water added by reading from the lowest point of the meniscus and record to the nearest tenth or first decimal place.
3) Place graduated cylinder onto an analytical balance.
a. Once a stable mass is displayed, tare the balance (or set to zero).
4) Carefully, place enough mass of the first metal into the graduated cylinder to produce a volume displacement of approximately $1-2$ milliliters.
a. Record the mass (or cumulative mass) of the metal, the total volume to the nearest hundredth (or tenth), and the cumulative volume of water displaced.
b. Be careful not to splash out any of the water.
5) Without removing the previous mass, add enough metal to produce another volume displacement of approximately $1-2$ milliliters.
a. Record the mass (or cumulative mass) of the metal, the total volume to the nearest hundredth (or tenth), and the cumulative volume of water displaced.
6) Continue adding mass of the metal using the same procedure for 3 additional data points.
a. Record the mass (or cumulative mass) of the metal, the total volume to the nearest hundredth (or tenth), and the cumulative volume of water displaced.
7) Repeat the above experiment with the other three metals.
8) Produce a graph of the cumulative mass versus the cumulative volume displaced using a computerized graphing program.
a. Compare the experimental/observed density to the true/theoretical density and calculate a percent error.
b. Percent error $=[($ true value - experimental value $) /$ true value $] * 100 \%$
i. Percent error cannot be a negative value. Take the absolute value of the number if necessary.
c. For the true/theoretical density, cite references for sources using proper bibliographic technique.

## Waste Disposal and Lab Clean-Up:

1) Without allowing any of the metal fragments to fall into the drain, dispose of the water down the sink.
2) Place wet metal fragments into the appropriately labeled waste containers for drying.
3) Stoppers should be dried off and returned to their storage bins.

## The Lab Write-Up:

1) Using the plots of mass versus volume displaced, determine the density of rubber and each metal.
2) In the conclusion, discuss all experimental/observed densities and compare to true values.
3) Following this comparison with the true values, discuss reasons for error and how these errors affected results.
4) Determine the percent error for each metal and discuss in conclusion in the error discussion.
a. Percent error $=[$ (true value - experimental value) $/$ true value $] * 100 \%$
b. Percent error cannot be a negative value. Take the absolute value of the number if necessary.
5) Which metal is the densest and which is the least dense?
6) Define intensive properties and extensive properties. Provide a few examples for each, including those used in this experiment.
7) Would your density results have been the same if ethanol was used as the liquid in the graduated cylinder? Explain.

## Example Data Table:

Table A: Mass and Volume Measurements for Titanium Metal

| Cumulative Mass of <br> Metal (g) <br> (y-axis on graph) | Total Volume of Water (mL) | Cumulative Volume <br> Displaced (mL) <br> (x-axis on graph) |
| :---: | :---: | :---: |
| 0.000 | 15.00 | 0.00 |
| 5.228 | 16.45 | 1.45 |
| 10.162 | 17.80 | 2.80 |
| 12.991 | 18.40 | 3.40 |
| 14.892 | 18.95 | 3.95 |
| 21.205 | 19.80 | 4.80 |

Draw the best fit straight line and the density will be the slope of the graph: $\Delta y / \Delta x$.

## Sample Data Tables:

Mass and Volume Measurements for Rubber Stoppers

| Stopper <br> Size(s) <br> $(\mathbf{0 0}, \mathbf{0}, \mathbf{1}$, or 2) | Cumulative Mass of <br> Stoppers (g) <br> $\left(\begin{array}{c}\text { (axis on graph })\end{array}\right.$ | Total Volume of <br> Water (mL)* <br> ${ }^{*}$ to tenth place | Cumulative Volume <br> Displaced (mL) <br> (x-axis on graph) |
| :---: | :---: | :---: | :---: |
|  | 0.00 or 0.000 |  | 0.0 |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |

Experimental/Observed Density for Rubber: $\qquad$ $\mathrm{g} / \mathrm{mL}$
True/Theoretical Density for Rubber: $\mathrm{g} / \mathrm{mL}$
Percent Error for Rubber Density: $\qquad$ \%

| Mass and Volume Measurements for Aluminum |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: |
| Data Point | Cumulative Mass of <br> Metal (g) <br> $(y$-axis on graph) | Total Volume of <br> Water (mL)* <br> *to hundredth place | Cumulative Volume <br> Displaced (mL) <br> $(x$-axis on graph) |  |
| 0 | 0.000 |  | 0.00 |  |
| 1 |  |  |  |  |
| 2 |  |  |  |  |
| 3 |  |  |  |  |
| 4 |  |  |  |  |
| 5 |  |  |  |  |

Experimental/Observed Density for Aluminum: $\qquad$ $\mathrm{g} / \mathrm{mL}$
True/Theoretical Density for Aluminum: $\qquad$ $\mathrm{g} / \mathrm{mL}$
Percent Error for Aluminum Density: $\qquad$ \%

Mass and Volume Measurements for Copper

| Data Point | Cumulative Mass of <br> Metal (g) <br> $(y$-axis on graph) | Total Volume of <br> Water (mL)* <br> *to hundredth place | Cumulative Volume <br> Displaced (mL) <br> $(x$-axis on graph) |
| :---: | :---: | :---: | :---: |
| 0 | 0.000 |  | 0.00 |
| 1 |  |  |  |
| 2 |  |  |  |
| 3 |  |  |  |
| 4 |  |  |  |
| 5 |  |  |  |

Experimental/Observed Density for Copper: $\qquad$ $\mathrm{g} / \mathrm{mL}$
True/Theoretical Density for Copper:
Percent Error for Copper Density:
$\qquad$ $\mathrm{g} / \mathrm{mL}$
$\square$ \%

| Mass and Volume Measurements for Nickel |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: |
| Data Point | Cumulative Mass of <br> Metal (g) <br> (y-axis on graph) | Total Volume of <br> Water (mL)* <br> *to hundredth place | Cumulative Volume <br> Displaced (mL) <br> (x-axis on graph) |  |
| 0 | 0.000 |  | 0.00 |  |$|$|  |
| :---: |
| 1 |

Experimental/Observed Density for Nickel: $\qquad$ $\mathrm{g} / \mathrm{mL}$
True/Theoretical Density for Nickel:
Percent Error for Nickel Density: $\qquad$ $\mathrm{g} / \mathrm{mL}$ \%

Mass and Volume Measurements for Zinc

| Data Point | Cumulative Mass of <br> Metal (g) <br> $(y$-axis on graph $)$ | Total Volume <br> of Water (mL)* <br> *to hundredth place | Cumulative Volume <br> Displaced (mL) <br> $(\boldsymbol{x}$-axis on graph) |
| :---: | :---: | :---: | :---: |
| 0 | 0.000 |  | 0.00 |
| 1 |  |  |  |
| 2 |  |  |  |
| 3 |  |  |  |
| 4 |  |  |  |
| 5 |  |  |  |

Experimental/Observed Density for Zinc: $\qquad$

## Experiment \#2: <br> Water of Hydration; <br> The Removal of Water from a Barium Chloride Hydrate

## Introduction to Hydrates:

1) Salts are compounds that are metal elements bound to non-metal elements through ionic bonding.
a. Examples: $\mathrm{NaCl}, \mathrm{CaSO}_{4}$

Table A

| $\#$ of <br> $\mathrm{H}_{2} \mathrm{O}$ | prefix |
| :---: | :---: |
| 1 | mono- |
| 2 | di- |
| 3 | tri- |
| 4 | tetra- |
| 5 | penta- |
| 6 | hexa- |
| 7 | hepta- |
| 8 | octa- |
| 9 | nona- |
| 10 | deca- |
| 11 | undeca- |
| 12 | dodeca- |

2) Inorganic hydrates (or hydrated salts) are compounds which contain a specific amount of water chemically bound.
a. The general formula for a hydrate is $\mathrm{MX}_{\neq} \cdot \mathbf{n} \mathrm{H}_{2} \mathrm{O}$.
i. $\mathrm{M}=$ metal ion, $\mathrm{X}=$ anion, $\mathrm{n}=$ moles of water bound
b. For example, iron (II) sulfate monohydrate (or ferrous sulfate monohydrate) has a formula of $\mathrm{FeSO}_{4} \cdot \mathrm{H}_{2} \mathrm{O}$.
i. This means that 1 molecule (or mole) of water is incorporated into the salt.
ii. The polar water molecule is held by the attractive force to the positively charged iron (II), $\mathrm{Fe}^{2+}$, ion.
3) Since many of these metal salts form hydrates as they crystallize, these materials are said to contain water of crystallization or water of hydration.
4) There are many other examples of inorganic hydrates with fixed amounts of bound water. See Table A for all the hydrate prefixes.
a. $\mathrm{SnCl}_{2} \cdot 2 \mathrm{H}_{2} \mathrm{O}$, tin (II) chloride dihydrate
b. $\mathrm{LiClO}_{4} \cdot 3 \mathrm{H}_{2} \mathrm{O}$, lithium perchlorate trihydrate
c. $\mathrm{NiSO}_{4} \cdot \mathbf{6} \mathrm{H}_{2} \mathrm{O}$, nickel (II) sulfate hexahydrate

## Introduction to Molar Mass (Formula Mass) and Percent Water:

1) Molar mass, $\mathrm{M}_{\mathrm{m}}$, or formula mass is the sum of the atomic weights of all atoms in a substance reported in grams per moles.
a. For hydrates, the water of hydration must be included.
2) The molar mass for $\mathrm{FeSO}_{4} \cdot \mathrm{H}_{2} \mathrm{O}$ is shown by the following calculation.

| Amount of atoms/molecules multiplied by atomic/molecular weight | Total to add to the molar mass (or formula mass) |
| :---: | :---: |
| $1 \times \mathrm{Fe}(55.85 \mathrm{~g} / \mathrm{mol})=$ | $55.85 \mathrm{~g} / \mathrm{mol}$ |
| $1 \times \mathrm{S}(32.07 \mathrm{~g} / \mathrm{mol})=$ | $+32.07 \mathrm{~g} / \mathrm{mol}$ |
| $4 \times \mathrm{O}(16.00 \mathrm{~g} / \mathrm{mol})=$ | $+64.00 \mathrm{~g} / \mathrm{mol}$ |
| $1 \mathrm{x} \mathrm{H}_{2} \mathrm{O}(18.02 \mathrm{~g} / \mathrm{mol})=$ | $+18.02 \mathrm{~g} / \mathrm{mol}$ |
| Molar mass ( $\mathrm{M}_{\mathrm{m}}$ ) for $\mathrm{FeSO}_{4} \cdot \mathrm{H}_{2} \mathrm{O}$ : | $=169.94 \mathrm{~g} / \mathrm{mol}$ |

3) To determine the percent water in a hydrate, divide the mass of water in one mole of hydrate by the molar mass of the hydrate and multiply this fraction by $100 \%$.
a. For $\mathrm{FeSO}_{4} \cdot \mathrm{H}_{2} \mathrm{O}$, the percent water in a hydrate is calculated as follows: $(18.02 \mathrm{~g} / \mathrm{mol}) /(169.94 \mathrm{~g} / \mathrm{mol}) \times 100 \%=10.60 \%$
4) Following heating with a Bunsen burner, the attractive forces can be overcome and the water can be liberated as a gas.
a. This leaves behind an anhydrous salt.
5) A balanced equation can be created by placing the solid hydrate on the reactant side and the anhydrous salt with gaseous water on the product side.
a. Example: $\mathrm{FeSO}_{4} \cdot \mathrm{H}_{2} \mathrm{O}(s) \xrightarrow{\Delta} \mathrm{FeSO}_{4}(s)+\mathrm{H}_{2} \mathrm{O}(\mathrm{g})$
b. Before heating, the molar mass (formula mass) for iron (II) sulfate monohydrate ( $\mathrm{FeSO}_{4} \cdot \mathrm{H}_{2} \mathrm{O}$ ), as shown above, is $169.94 \mathrm{~g} / \mathrm{mol}$.
i. Of this, $18.02 \mathrm{~g} / \mathrm{mol}$ is comprised of water.
c. After heating, the molar mass (formula mass) of anhydrous iron (II) sulfate $\left(\mathrm{FeSO}_{4}\right)$ is $151.91 \mathrm{~g} / \mathrm{mol}$.
i. The mass should have decreased by $10.60 \%$.

## Procedure for Hydrated Barium Chloride $\left(\mathrm{BaCl}_{2} \cdot \mathbf{n H}_{2} \mathrm{O}\right)$ :

1) Obtain one crucible. Clean and dry if necessary.
a. Record an initial mass of the crucible.
2) Set up the apparatus shown in the model attached.
a. Attach an iron ring to a ring stand or to the lab metal scaffolding.
b. Place a clay triangle onto the iron ring and place your crucible into the triangle.
3) Place a Bunsen burner away from apparatus and light with a striker. Adjust flame so that a blue flame is observed with a hot inner blue cone.
4) Move lit Bunsen burner into position underneath the apparatus.
a. Adjust flame so that the hot part of the flame heats the bottom of the crucible.
5) Heat the empty crucible (without a lid) for 5 minutes to drive away adsorbed water.
a. Be careful to handle only with crucible tongs when hot!
6) Allow to cool to room temperature.
7) Record the mass of the cooled crucible.
8) Place crucible back onto the setup and reheat the crucible for 2 minutes.
a. Allow to cool to room temperature and determine mass.
b. If there has not been a significant change in mass, record the mass and continue on with the experiment.
i. A significant change in mass can include a mass difference of $\geq$ 0.01 g .
c. If there has been significant change in mass, reheat (for 2 minutes) until a constant mass is obtained. Record the constant mass.
9) Weigh out approximately 1.0 g of the hydrated barium chloride on an anatical balance.
a. It would be advisable to place the crucible of constant mass on the balance and tare it (set it equal to zero) and then weigh the sample into the crucible.
b. Record the mass indicated on the balance using all decimal places provided.
10) Heat the crucible with the sample for $10-15$ minutes.
a. Note the initial and final appearance of your crystals.
11) Allow to cool to room temperature.
12) Record the mass of the cooled crucible with the sample inside.
a. Subtract the mass of the empty crucible from this recorded mass to obtain the mass of the anhydrous salt.
b. Create a data table similar to the example attached.
13) Repeat the above process for a second trial using a separate quantity (between $1-2 \mathrm{~g}$ ) to ensure results are reproducible.

## Waste Disposal and Lab Clean - Up:

1) Place all anhydrous barium chloride powder into the appropriately labeled container.
2) Make sure that all gas valves are turned off!

## The Lab Write - Up:

1) In the conclusion, explain all relevant data points collected.
2) Discuss the experimental formula determined for barium chloride hydrate and compare to the known formula.
3) Write a balanced equation for the process that took place.
4) After calculation of the percent water in the barium chloride hydrate for your experiment, compare this to the true value. Discuss percent error, reasons for error, and how errors affected data/results.
5) Why was it necessary to heat the empty crucible before performing the experiment? How would your results have been different if this step were not performed?
6) Name all of the hydrates in the table below using the IUPAC nomenclature system.

Common Hydrate Table:

| Hydrate | IUPAC Name |
| :---: | :---: |
| $\mathrm{CuSO}_{4} \cdot 5 \mathrm{H}_{2} \mathrm{O}$ |  |
| $\mathrm{MgSO}_{4} \cdot 7 \mathrm{H}_{2} \mathrm{O}$ |  |
| $\mathrm{CrCl}_{3} \cdot 6 \mathrm{H}_{2} \mathrm{O}$ |  |
| $\mathrm{Pb}\left(\mathrm{C}_{2} \mathrm{H}_{3} \mathrm{O}_{2}\right)_{2} \cdot 3 \mathrm{H}_{2} \mathrm{O}$ |  |

Sample Data Table:

| Data Recorded/Calculated | Trial \#1 | Trial \#2 |
| :---: | :---: | :---: |
| a) Initial mass of crucible in grams: |  |  |
| b) Mass of crucible after first heating in grams: |  |  |
| c) Mass of crucible after second heating in grams: |  |  |
| d) Stable mass of crucible in grams*: <br> *Use mass after final heating |  |  |
| e) Initial mass of barium chloride hydrate in grams: |  |  |
| f) Mass of sample plus crucible after heating in grams: |  |  |
|  |  |  |
| g) Mass of barium chloride after heating in grams ( $\mathrm{f}-\mathrm{d}$ ): |  |  |
| h) Moles of barium chloride after heating: ( $\mathrm{g} / \mathrm{M}_{\mathrm{m}}$ ): |  |  |
|  |  |  |
| i) Mass of water lost after heating in grams (e-g): |  |  |
| j) Moles of water lost after heating (i/ $\mathrm{M}_{\mathrm{m}}$ ): |  |  |
|  |  |  |
| k) Experimental mole to mole ratio of $\mathrm{BaCl}_{2}$ to $\mathrm{H}_{2} \mathrm{O}^{*}$ : | : | : |
| 1) Experimental formula for hydrated barium chloride: |  |  |
|  |  |  |
| k) True mole to mole ratio of $\mathrm{BaCl}_{2}$ to $\mathrm{H}_{2} \mathrm{O}^{*}$ : | : | : |
| 1) True formula for hydrated barium chloride: |  |  |
|  |  |  |
| o) Experimental/Observed percent water in hydrate ((i/e) $\times 100 \%$ ): |  |  |
| p) True/Theoretical percent water in hydrate: |  |  |
| q) Percent error for percent water in hydrate: |  |  |

*To determine the mole to mole ratio in whole numbers, divide the number of moles for both $\mathrm{BaCl}_{2}$ and $\mathrm{H}_{2} \mathrm{O}$ by the smallest number of moles from the two. Round to the nearest whole number.

## Model of the Apparatus:



## Experiment \#3A: The Stoichiometry of a Double Displacement Reaction; Synthesis of Calcium Carbonate

## Introduction to Chemical Reactions:

1) Most chemical reactions are placed in three categories:
a. Precipitation reactions
i. Two ionic solutions are mixed and produce an ionic solid.
ii. The most common type of precipitation reaction is a double displacement reaction or metathesis reaction.
1. This reaction can also be referred to as an exchange reaction as shown below:
2. $2 \mathrm{KBr}(a q)+\mathrm{Pb}\left(\mathrm{NO}_{3}\right)_{2}(a q) \rightarrow \mathrm{PbBr}_{2}(s)+2 \mathrm{KNO}_{3}(a q)$
iii. To predict precipitation reactions, the solubilities of reactants and products must be assessed.
3. If all reactants and products are soluble in water, no product results.
4. If one or more products is insoluble, the reaction will proceed and a solid substance will be observed.
b. Acid-base neutralization reactions
i. An acid and base are combined to form an ionic compound (i.e. salt) and possibly water (if the base is a hydroxide).
ii. A Brønsted-Lowry acid is defined as a proton donor.
5. Example: HBr or $\mathrm{H}_{2} \mathrm{SO}_{4}$
iii. A Brønsted-Lowry base is defined as a proton acceptor.
6. Example: LiOH or $\mathrm{NH}_{3}$
iv. An example reaction: $\mathrm{LiOH}(a q)+\mathrm{HBr}(a q) \rightarrow \mathrm{LiBr}(a q)+\mathrm{H}_{2} \mathrm{O}(l)$
v. Some ionic salts, when reacted with acid, form gases.
7. The most common gases produced are $\mathrm{CO}_{2}, \mathrm{SO}_{2}$, and $\mathrm{H}_{2} \mathrm{~S}$.
8. Example: $\mathrm{K}_{2} \mathrm{CO}_{3}(a q)+2 \mathrm{HBr}(a q) \rightarrow 2 \mathrm{KBr}(a q)+\mathrm{H}_{2} \mathrm{O}(l)+$ $\mathrm{CO}_{2}(\mathrm{~g})$
a. The $\mathrm{H}_{2} \mathrm{O}(l)+\mathrm{CO}_{2}(g)$ result from the spontaneous decomposition of $\mathrm{H}_{2} \mathrm{CO}_{3}(a q)$.
c. Oxidation-reduction reactions
i. These reactions involve electron transfer where one species is oxidized and another species is reduced.
9. Oxidation: loss of electrons or an increase in the oxidation number.
10. Reduction: gain of electrons or a decrease in the oxidation number.
ii. The most common types of oxidation-reduction reactions are:
11. Combination (or synthesis) reactions
12. Decomposition reactions
13. Single displacement reactions
14. Combustion reactions
2) There are a number of driving forces for reactions to occur:
a. Formation of molecular compounds
i. Example: $\mathrm{H}_{2} \mathrm{O}$
b. Formation of a gas
i. Examples: $\mathrm{H}_{2} \mathrm{~S}, \mathrm{CO}_{2}, \mathrm{SO}_{2}$
c. Formation of an insoluble precipitate
i. See Table A attached for a comprehensive list of soluble and insoluble salts in water.

## Introduction to Double Displacement Reactions:

1) A double displacement reaction (or metathesis reaction) is one in which two ionic compounds in an aqueous solution exchange anions to produce two new species.
2) The driving force for this type of reaction is typically the production of an insoluble precipitate.
a. If all products are soluble, no reaction will take place.
3) Most double displacement reactions can be predicted by analyzing water solubility of the products.
4) An example precipitation reaction is depicted below.
a. $\mathrm{Al}_{2}\left(\mathrm{SO}_{4}\right)_{3}(a q)+6 \mathrm{NaOH}(a q) \rightarrow 2 \mathrm{Al}(\mathrm{OH})_{3}(s)+3 \mathrm{Na}_{2} \mathrm{SO}_{4}(a q)$
5) Most acid-base neutralization reactions can also be classified as double displacement reactions.

## Introduction to Stoichiometry:

1) Stoichiometry is the calculation of the quantities of reactants and products involved in a chemical reaction.
a. It is based on the balanced chemical equation and on the relationship between mass and moles.
2) The balanced chemical equation can be interpreted in numbers of molecules, but generally chemists interpret equations as "mole-to-mole" relationships.
3) For example, the Haber process for producing ammonia involves the reaction of hydrogen and nitrogen.
a. The balanced equation: $\mathrm{N}_{2}(g)+3 \mathrm{H}_{2}(g) \rightarrow 2 \mathrm{NH}_{3}(g)$
b. This balanced chemical equation shows that 1 mole of $\mathrm{N}_{2}$ reacts with 3 moles of $\mathrm{H}_{2}$ to produce 2 moles of $\mathrm{NH}_{3}$.
c. Because moles can be converted into mass (using the molar mass or molecular weight), a mass interpretation of the chemical equation can be performed.
4) The limiting reactant (or limiting reagent) is the reactant that is entirely consumed when the reaction goes to completion.
5) The moles of product are always determined by the starting moles of the limiting reagent.
6) The theoretical yield is the maximum amount of product that can be obtained from a reaction from given amount of reactants.
a. The theoretical yield of product is determined by the starting moles of the limiting reagent.
7) The percent yield is the actual yield (or experimental yield) expressed as a percentage of the theoretical yield.
a. Percent Yield $=($ Actual Yield/Theoretical Yield $) X 100 \%$

Table A: Solubility Table for Common Anions \& Cations

| Anion/Cation Name(s) | Anion/Cation Formula(s) | Soluble | Insoluble |
| :---: | :---: | :---: | :---: |
| acetate | $\begin{gathered} \mathrm{CH}_{3} \mathrm{CO}_{2}^{-} \text {or } \\ \mathrm{C}_{2} \mathrm{H}_{3} \mathrm{O}_{2}^{-} \end{gathered}$ | most cations | none |
| arsenate, phosphate | $\begin{aligned} & \mathrm{AsO}_{4}{ }^{3-} \\ & \mathrm{PO}_{4}^{3-} \\ & \hline \end{aligned}$ | $\mathrm{NH}_{4}{ }^{+}$, Group IA (except $\mathrm{Li}^{+}$) | most cations |
| bromide, chloride | $\begin{aligned} & \mathrm{Br}^{-} \\ & \mathrm{Cl}^{-} \\ & \hline \end{aligned}$ | most cations | $\begin{gathered} \mathrm{Ag}^{+}, \mathrm{Hg}_{2}{ }^{2+}, \mathrm{Pb}^{2+} \\ \left(\text { and } \mathrm{Cu}^{+}, \mathrm{Tl}^{+}\right) \\ \hline \end{gathered}$ |
| carbonate | $\mathrm{CO}_{3}{ }^{2-}$ | $\mathrm{NH}_{4}{ }^{+}$, Group IA (except $\mathrm{Li}^{+}$) | most cations |
| chlorate, perchlorate | $\begin{aligned} & \mathrm{ClO}_{3-} \\ & \mathrm{ClO}_{4-}^{-} \\ & \hline \end{aligned}$ | most cations | none |
| chromate | $\mathrm{CrO}_{4}{ }^{2-}$ | $\begin{gathered} \mathrm{NH}_{4}^{+}, \text {Group IA }, \mathrm{Ca}^{2+}, \\ \mathrm{Mg}^{2+}, \mathrm{Cu}^{2+} \end{gathered}$ | most cations |
| cyanide | $\mathrm{CN}^{-}$ | $\mathrm{NH}_{4}{ }^{+}$, Group IA (except $\mathrm{Li}^{+}$) | most cations |
| fluoride | F | $\mathrm{NH}_{4}^{+}$, Group IA, $\mathrm{Ag}^{+}$ | most cations |
| hydroxide, oxide | $\underset{\mathrm{O}^{2-}}{\mathrm{OH}^{-} \text {or } \mathrm{HO}^{-}}$ | $\begin{gathered} \mathrm{NH}_{4}^{+}, \text {Group IA, }^{2+} \mathrm{Sr}^{2+}, \\ \mathrm{Ba}^{a^{+}}, \mathrm{Ca}^{2^{+}} \text {(sparingly) } \end{gathered}$ | most cations |
| iodide | $\mathrm{I}^{-}$ | most cations | $\begin{gathered} \mathrm{Ag}^{+}, \mathrm{Hg}_{2}{ }^{2+}, \mathrm{Pb}^{2+} \\ \left(\text { and } \mathrm{Cu}^{+}, \mathrm{Tl}^{+}, \mathrm{Bi}^{3+}, \mathrm{Sn}^{4+}\right) \end{gathered}$ |
| nitrate nitrite | $\begin{aligned} & \mathrm{NO}_{3}^{-} \\ & \mathrm{NO}_{2}^{-} \\ & \hline \end{aligned}$ | most cations | none |
| oxalate | $\mathrm{C}_{2} \mathrm{O}_{4}{ }^{2-}$ | $\mathrm{NH}_{4}{ }^{+}$, Group IA (except Li ${ }^{+}$) | most cations |
| permanganate | $\mathrm{MnO}_{4}{ }^{-}$ | most cations | none |
| silicate | $\mathrm{SiO}_{3}{ }^{2-}$ | Group IA | most cations |
| sulfate | $\mathrm{SO}_{4}{ }^{2-}$ | most cations | $\begin{gathered} \mathrm{Ca}^{2+}, \mathrm{Sr}^{2+}, \mathrm{Ba}^{2+}, \mathrm{Pb}^{2+}, \\ \mathrm{Ag}^{+} \text {(sparingly) } \end{gathered}$ |
| sulfide | $\mathrm{S}^{2-}$ | $\mathrm{NH}_{4}^{+}$, Group IA \& IIA | most cations |
| sulfite | $\mathrm{SO}_{3}{ }^{2-}$ | $\mathrm{NH}_{4}{ }^{+}$, Group IA (except $\mathrm{Li}^{+}$) | most cations |
| thiosulfate | $\mathrm{S}_{2} \mathrm{O}_{3}{ }^{2-}$ | most cations | $\mathrm{Ag}^{+}, \mathrm{Pb}^{2+}$ |
| ////////////////////////\| | /////////////////////////// | ///\|/|/|/|/|/|/|/|/|/|/|/|/|/|/| | ////////////////////////////////// |
| Group IA | $\mathrm{Li}^{+}, \mathrm{Na}^{+}, \mathrm{K}^{+}$ | most anions | none (except for some $\mathrm{Li}^{+}$salts) |
| ammonium | $\mathrm{NH}_{4}{ }^{+}$ | most anions | none |

## The Experimental Procedure for the Synthesis of Calcium Carbonate:

1) Using a wax/grease pencil, label two 125 mL Erlenmeyer flasks as Flask \#1 and Flask \#2.
2) Place 100.0 mL of deionized water into each Erlenmeyer flask using a 100.0 mL graduated cylinder.
3) On an analytical balance and in separate weigh boats, obtain approximately 5.00 g of anhydrous calcium chloride $\left(\mathrm{CaCl}_{2}\right)$ and approximately 5.00 g of anhydrous sodium carbonate $\left(\mathrm{Na}_{2} \mathrm{CO}_{3}\right)$.
a. Note: Calcium chloride is extremely hygroscopic which means that it readily absorbs moisture from the air.
b. Return cap of the calcium chloride immediately after using and proceed expediently after measuring the mass of the calcium chloride.
c. Record the mass of each ingredient and place data in data table.
4) Add anhydrous calcium chloride to flask \#1.
a. Swirl to completely dissolve.
b. Note the evolution of heat as the calcium chloride dissolves.
5) Add anhydrous sodium carbonate to flask \#2.
a. Swirl to completely dissolve.
b. Note in observations if any bubbling occurs following the addition of the sodium carbonate salt to the water.
6) While the reactants are dissolving, obtain a single piece of Whatman filter paper and a watch glass.
a. Record the mass of the filter paper and watch glass together on an ANALYTICAL balance and record in your data table.
7) Once dissolved, immediately add both solutions together into a $250-500 \mathrm{~mL}$ Erlenmeyer flask and record observations in data table.
a. Note: Calcium carbonate is unusually more soluble in water when the temperature is decreased. Thus, it would be advisable to work expediently while the calcium chloride solution is warm.
8) Rinse both 125 mL Erlenmeyer flasks with small amounts of deionized water, and add to the large Erlenmeyer flask.
9) You are now ready to filter; please proceed to the filtration process designated by your instructor.

## Gravity Filtration Procedure:

1) Flute the filter paper by repeatedly folding in half and place into a glass funnel.
2) Place onto a 500 mL Erlenmeyer flask.
3) Wet the filter with a small amount of deionized water.
4) Once the filter paper is secure, transfer the contents of the Erlenmeyer flask into the filter paper.
a. The water should pass through the filter leaving behind the precipitate.
b. Do not allow the liquid volume to exceed the height of the filter paper.
c. To prevent loss of product, it may be advisable to transfer the solution carefully down the length of a glass stir rod and onto the filter paper.
5) After the liquid has passed through the filter, observe the filtrate solution in the filter flask.
a. If a large amount of precipitate has passed through the filter, remove the solution from the flask and re-filter through the funnel.
b. If a significant amount of precipitate has not passed through the filter, continue with the experiment.
6) Rinse out your Erlenmeyer flask with deionized water and place into the filter to ensure all precipitate has been collected.
7) Once most of the water has passed through the filter and a wet cake is evident, proceed to the next step.
8) Transfer your filter paper and precipitate onto the pre-weighed watch glass and allow to dry as instructed.
a. The sample can be air dried, dried in a dessicator, or dried in a warm oven.
9) Once the precipitate dries, record the mass of the watch glass, filter paper, and precipitate.
10) Create a balanced equation and a net ionic equation for the reaction between these two aqueous solutions.
11) Determine the limiting reactant and the theoretical yield of calcium carbonate.
12) Calculate the percent yield and discuss reasons for error.

## Vacuum Filtration Procedure:

1) Set up a vacuum filtration apparatus as shown in the attached diagram.
a. Clamp the filter flask securely to a ring stand before affixing vacuum tubing and turning on vacuum.
2) Pre-weigh a piece of filter paper and then place into the Büchner funnel of the vacuum filtration system.
3) Turn on the vacuum first and then wet the filter paper with small amounts of deionized water.
a. Ensure that the filter paper has created a good seal inside the Büchner funnel and no bubbling of the filter paper is observed.
4) Slowly, transfer the contents of the Erlenmeyer flask into the filter paper.
a. The water should pass through the filter leaving behind the precipitate.
b. Do not allow the liquid volume to exceed the height of the filter paper.
c. To prevent loss of product, it may be advisable to transfer the solution carefully down the length of a glass stir rod and onto the filter paper.
5) After the liquid has passed through the filter, observe the filtrate solution in the filter flask.
a. If a large amount of precipitate has passed through the filter, remove the solution from the flask and re-filter through the funnel.
b. If a significant amount of precipitate has not passed through the filter, continue with the experiment.
6) Rinse out your Erlenmeyer flask with deionized water and place into the filter to ensure all precipitate has been collected.
7) Once all of the liquid has passed through the filter paper, proceed to the next step.
8) Transfer your filter paper and precipitate onto the pre-weighed watch glass and allow to dry as instructed.
a. The sample can be air dried, dried in a dessicator, or dried in a warm oven.
9) Once the precipitate has dried, record the mass of the watch glass, filter paper, and precipitate.
10) Create a balanced equation and a net ionic equation for the reaction between these two aqueous solutions.
11) Determine the limiting reactant and the theoretical yield of calcium carbonate.
12) Calculate the percent yield and discuss reasons for error.

## Waste Disposal and Lab Clean - Up:

1) All aqueous filtrates can be placed into the sink.
2) Place all calcium carbonate waste into the trash can.

## The Lab Write - Up:

1) Determine all values in your data table including your balanced equations, limiting reactants, etc.
2) Determine your theoretical yield and percent yield of calcium carbonate.
a. $\%$ Yield $=($ Actual Yield/Theoretical Yield $) \mathrm{X} 100 \%$.
b. How come you did not achieve $100 \%$ yield?
i. Discuss potential places for error that accounted for high/low percent yield.
3) In your conclusion, explain all data collected and discuss reasons for error.
4) What would happen to the yield results if the sample was not completely dried?
5) What would happen if the water used to dissolve the sodium carbonate was slightly acidic? How may the limiting reactant/yield be affected?
a. Hint: This side reaction should appear under the side reaction section of your lab notebook.

## Sample Data Table:

| Data Point to be Recorded/Calculated | Data Point |
| :---: | :---: |
| a) Mass of anhydrous $\mathrm{CaCl}_{2}$ in grams: |  |
| b) Mass of anhydrous $\mathrm{Na}_{2} \mathrm{CO}_{3}$ in grams: |  |
| c) Balanced equation with states of matter: | $\longrightarrow$ |
| d) Net ionic equation with states of matter: | $\longrightarrow$ |
| e) Evidence of reaction/Reaction observations: |  |
| f) Molar mass of $\mathrm{CaCl}_{2}(\mathrm{~g} / \mathrm{mol})$ : |  |
| $\mathrm{g})$ Moles of $\mathrm{CaCl}_{2}(\mathrm{~mol})$ : |  |
| h) Molar mass of $\mathrm{Na}_{2} \mathrm{CO}_{3}(\mathrm{~g} / \mathrm{mol})$ : |  |
| i) Moles of $\mathrm{Na}_{2} \mathrm{CO}_{3}(\mathrm{~mol})$ : |  |
| j) Molar mass of $\mathrm{CaCO}_{3}(\mathrm{~g} / \mathrm{mol})$ : |  |
| k) Limiting reactant: |  |
| $l)$ Theoretical yield of $\mathrm{CaCO}_{3}$ in grams: |  |
| m) Mass of filter paper + watch glass in grams: |  |
| n) Mass of filter paper + watch glass + dried product in grams: |  |
| o) Actual yield of $\mathrm{CaCO}_{3}$ in grams ( $\mathrm{n}-\mathrm{m}$ ): |  |
| p) Percent yield of $\mathrm{CaCO}_{3}(\mathbf{o} / \boldsymbol{l} \times 100 \%)$ in $\%$ : |  |
| q) Errors observed throughout the experiment: |  |

## Model of Vacuum Filtration Using a Büchner Funnel



Clamp filter flask securely to a ring stand before affixing vacuum tubing and turning on vacuum.

# Experiment \#3B: <br> Empirical Formula Determination of Magnesium Compounds and the Stoichiometry of Reactions 

## Introduction to Experiment:

1) Magnesium metal reacts with oxygen and chloride (from hydrochloric acid) to form magnesium salts.
a. $\mathrm{Mg}(\mathrm{s})+\mathrm{O}_{2}(\mathrm{~g}) \rightarrow$ magnesium oxide salt
b. $\mathrm{Mg}(s)+\mathrm{HCl}(a q) \rightarrow$ magnesium chloride salt + byproduct.
2) From determining the mass of reactant magnesium metal and the mass of magnesium salt product, the mass of oxygen and chloride can be determined.
3) Once the mass of reactants is determined, these amounts can be converted to moles.
a. The ratio between the number of moles of magnesium used and the number of moles of oxygen or chlorine bound can be calculated. This will provide the empirical formula.

## Introduction to Empirical Formula:

1) The empirical formula is the formula of a substance written with the smallest integer subscripts (whole numbers).
2) It is the simplest of formulas that is directly related to the percent composition.
3) For most ionic compounds, the empirical formula is the one reported for the compound.
a. Examples:
i. $\mathrm{LiCl}(1: 1$ ratio of elements)
ii. $\mathrm{CaF}_{2}$ (1:2 ratio of elements)
iii. $\mathrm{CrO}_{3}$ (1:3 ratio of elements)
iv. $\mathrm{Na}_{2} \mathrm{~S}(2: 1$ ratio of elements)

## Introduction to Stoichiometry:

1) Stoichiometry is the calculation of the quantities of reactants and products involved in a chemical reaction.
a. It is based on the balanced chemical equation and on the relationship between mass and moles.
2) The balanced chemical equation can be interpreted in numbers of molecules, but generally chemists interpret equations as "mole-to-mole" relationships.
3) For example, the Haber process for producing ammonia involves the reaction of hydrogen and nitrogen.
a. The balanced equation: $\mathrm{N}_{2}(g)+3 \mathrm{H}_{2}(g) \rightarrow 2 \mathrm{NH}_{3}(g)$
b. This balanced chemical equation shows that 1 mole of $\mathrm{N}_{2}$ reacts with 3 moles of $\mathrm{H}_{2}$ to produce 2 moles of $\mathrm{NH}_{3}$.
c. Because moles can be converted into mass (using the molar mass or molecular weight), a mass interpretation of the chemical equation can be performed.
4) The limiting reactant (or limiting reagent) is the reactant that is entirely consumed when the reaction goes to completion.
5) The moles of product are always determined by the starting moles of the limiting reagent.
6) The theoretical yield is the maximum amount of product that can be obtained from a reaction from given amount of reactants.
b. The theoretical yield of product is determined by the starting moles of the limiting reagent.
7) The percent yield is the actual yield (or experimental yield) expressed as a percentage of the theoretical yield.
a. Percent Yield $=($ Actual Yield/Theoretical Yield $) \mathrm{X} 100 \%$

## Part 1

## Procedure for Reacting Magnesium Metal with Oxygen:

1) Obtain one crucible and a lid. Clean and dry if necessary.
a. Record an initial mass of the crucible WITH lid.
2) Set up the apparatus shown in the model attached.
a. Attach an iron ring to a ring stand or to the lab metal scaffolding.
b. Place a clay triangle onto the iron ring and place your crucible into the triangle.
3) Place a Bunsen burner away from apparatus and light with a striker. Adjust flame so that a blue flame is observed with a hot inner blue cone.
4) Move lit Bunsen burner into position underneath the apparatus.
a. Adjust flame so that the hot part of the flame heats the bottom of the crucible.
5) Heat the empty crucible with the lid slightly ajar for 5 minutes to drive away adsorbed water.
a. Be careful to handle only with crucible tongs when hot!
6) Allow to cool to room temperature.
7) Record the mass of the cooled crucible with lid.
8) Place crucible back onto the setup and reheat the crucible for 2 minutes.
a. Allow to cool to room temperature and determine mass.
b. If there has not been a significant change in mass, record the mass and continue on with the experiment.
i. A significant change in mass can include a mass difference of $\geq$ 0.01 g .
c. If there has been significant change in mass, reheat (for 2 minutes) until a constant mass is obtained. Record the constant mass.
9) Obtain one 12 cm ribbon of pure magnesium metal which has a mass of $\sim 0.1 \mathrm{~g}$. If tarnished, use sandpaper to polish to a shine.
10) Create a loose roll, place into the crucible, and obtain the mass of this strip of magnesium metal on an analytical balance.
a. It would be advisable to place the crucible + lid of constant mass on the balance and tare it (set it equal to zero) and then weigh the sample into the crucible.
b. Record the mass of the magnesium metal strip.
11) Place crucible with your magnesium back onto the setup with the lid slightly ajar.
12) Place a Bunsen burner away from apparatus and light with a striker. Adjust flame so that a blue flame is observed with a hot inner blue cone.
13) Move lit Bunsen burner into position underneath the apparatus.
a. Adjust flame so that the hot part of the flame heats the bottom of the crucible.
14) If heated strongly enough, the magnesium should ignite and white smoke should appear.
a. Do not look directly into the burning magnesium flame!
b. Cover the crucible temporarily using the crucible tongs to hand the lid.
c. Once the smoke subsides, allow the lid to sit slightly ajar for the remainder of the experiment.
15) Next, allow the Bunsen burner to heat the crucible with the lid slightly ajar for at least 10 minutes.
a. The bottom of the crucible should glow red if enough heat is applied.
b. After 10 minutes, remove the lid with the crucible tongs to check if all magnesium metal has been burned.
16) Once all magnesium metal has burned, remove the heat and place the crucible and lid on a ceramic square to cool.
17) Once cool, deliver 5 drops of deionized water into the crucible, place back onto the heating apparatus, and heat the crucible with the lid slightly ajar for at least 5 minutes.
18) After the allotted time, remove the heat and place the crucible and lid on a ceramic square to cool.
19) Once crucible is cool, record the mass of the crucible with lid containing the oxide.

## Waste Disposal and Lab Clean - Up:

1) Place all oxide product into the trash can.
2) Make sure that all gas valves are turned off!

## Model of the Apparatus for Part 1:



## Sample Data Table for Magnesium Metal with Oxygen:

a) Initial Mass of Crucible + Lid in grams: $\qquad$
b) Mass of Crucible + Lid After First Heating in grams:
c) Mass of Crucible + Lid After Second Heating in grams: $\qquad$
d) Stable Mass of Crucible + Lid in grams: $\qquad$
e) Mass of Magnesium Metal in grams:
f) Moles of Magnesium Metal (e/ $\mathrm{M}_{\mathrm{m}}$ of Mg ): $\qquad$
g) Mass of Crucible + Lid + Oxide product in grams: $\qquad$
h) Mass of Oxide Product in grams: $(\mathrm{g}-\mathrm{d})$ : $\qquad$
i) Mass of Oxygen/Oxide in grams $(\mathrm{h}-\mathrm{e})$ : $\qquad$
j) Moles of Oxygen/Oxide ( $\mathrm{i} / \mathrm{M}_{\mathrm{m}}$ of O ): $\qquad$
k) Experimental Mole to Mole Ratio of Mg to O*: $\qquad$ : $\qquad$

1) Experimental Empirical Formula for Oxide Product: $\qquad$
m) True Mole to Mole Ratio of Mg to O : $\qquad$ :
n) True Empirical Formula for Oxide Product: $\qquad$
o) Balanced Equation for the Reaction: $\qquad$

## Yield Information:

Actual Yield for Magnesium Oxide in grams:
Theoretical Yield for Magnesium Oxide in grams:
$\qquad$

Percent Yield for Magnesium Oxide: $\qquad$

[^0]
## Part 2

## Procedure for Reacting Magnesium Metal with HCl:

1) Obtain one evaporating dish. Clean and dry if necessary.
a. Record an initial mass of the evaporating dish.
2) Set up the apparatus shown in the model attached.
a. Attach an iron ring to a ring stand or to the lab metal scaffolding.
b. Place a wire mesh onto the iron ring and place evaporating dish onto the mesh.
3) Place a Bunsen burner away from apparatus and light with a striker. Adjust flame so that a blue flame is observed with a hot inner blue cone.
4) Move lit Bunsen burner into position underneath the apparatus.
5) Adjust flame so that the hot part of the flame heats the bottom of the wire mesh.
6) Heat the empty evaporating dish for 5 minutes to drive away adsorbed water.
a. Be careful to handle only with crucible tongs when hot!
7) Allow to cool to room temperature.
8) Record the mass of the cooled evaporating dish.
9) Place evaporating dish back onto the setup and reheat the crucible for 2 minutes.
a. Allow to cool to room temperature and determine mass.
b. If there has not been a significant change in mass, record the mass and continue on with the experiment.
i. A significant change in mass can include a mass difference of $\geq$ 0.01 g .
c. If there has been significant change in mass, reheat (for 2 minutes) until a constant mass is obtained. Record the constant mass.
10) Obtain one 12 cm ribbon of pure magnesium metal which has a mass of $\sim 0.1 \mathrm{~g}$. If tarnished, use sandpaper to polish to a shine.
11) Create a loose roll, place into the evaporating dish, and obtain the mass of this strip of magnesium metal on an analytical balance.
a. It would be advisable to place the dish of constant mass on the balance and tare it (set it equal to zero) and then weigh the sample into the dish.
b. Record the mass of the magnesium metal strip.
12) Measure out at least 2.0 mL of 6 M hydrochloric acid $(\mathrm{HCl})$.
a. Be extremely careful with this acid; it is corrosive!
13) Before adding the acid to the evaporating dish, line the benchtop with paper towels and place evaporating dish with magnesium inside onto the paper towels.
14) Slowly pour the acid overtop of the magnesium metal in the evaporating dish.
a. Record observations of the process that occurs.
b. Do not inhale vapors.
15) Once all of the magnesium has reacted, place onto the apparatus shown below UNDER THE FUME HOOD.
16) Cover the dish with a watch glass.
17) Place a Bunsen burner away from apparatus and light with a striker. Adjust flame so that a blue flame is observed with a hot inner blue cone.
18) Move lit Bunsen burner into position underneath the apparatus.
19) Adjust flame so that the hot part of the flame heats the bottom of the wire mesh.
20) Heat the solution until dryness.
a. Notice that unreacted hydrochloric acid and water will steam off.
b. Use crucible tongs to carefully remove the watch glass, and place into a paper towel.
21) Once a dry powder is observed, place evaporating dish onto a cooling square, allow for cooling, and then obtaining the mass of the powder product.

## Waste Disposal and Lab Clean - Up:

1) Place chloride product into the trash can.
2) Make sure that all gas valves are turned off!

## Model of the Apparatus for Part 2:



## Sample Data Table for Magnesium Metal with HCl:

a) Initial Mass of Evaporating Dish in grams:
b) Mass of Evaporating Dish After First Heating in grams: $\qquad$
c) Mass of Evaporating Dish After Second Heating in grams: $\qquad$
d) Stable Mass of Evaporating Dish in grams: $\qquad$
e) Mass of Magnesium Metal in grams:
f) Moles of Magnesium Metal (e/ $\mathrm{M}_{\mathrm{m}}$ of Mg$)$ : $\qquad$
g) Mass of Evaporating Dish + Chloride Product in grams: $\qquad$
h) Mass of Chloride Product in grams: $(\mathrm{g}-\mathrm{d})$ : $\qquad$
i) Mass of Chlorine/Chloride in grams $(\mathrm{h}-\mathrm{e})$ : $\qquad$
j) Moles of Chlorine/Chloride ( $\mathrm{i} / \mathrm{M}_{\mathrm{m}}$ of Cl ): $\qquad$
k) Experimental Mole to Mole Ratio of Mg to $\mathrm{Cl}^{*}$ : $\qquad$ :

1) Experimental Empirical Formula for Chloride Product: $\qquad$
m) True Mole to Mole Ratio of Mg to Cl : $\qquad$ $:$
n) True Empirical Formula for Chloride Product: $\qquad$
o) Balanced Equation for the Reaction: $\qquad$

## Yield Information:

Actual Yield for Magnesium Chloride in grams:
Theoretical Yield for Magnesium Chloride in grams:
Percent Yield for Magnesium Chloride: $\qquad$
*To determine the mole to mole ratio in whole numbers, divide the number of moles for both Mg and Cl by the smallest number of moles from the two. Round values to the nearest whole number.

## The Lab Write - Up:

1) Determine all values in your data table including your empirical formulas and balanced equations.
2) Determine your theoretical yield of magnesium salts and percent yields.
a. $\%$ Yield $=($ Actual Yield/Theoretical Yield) X 100\%.
b. How come you did not achieve $100 \%$ yield?
3) Explain all data collected and discuss reasons for error.
4) In the formation of the magnesium oxide salt, why was it necessary to leave the crucible lid slightly ajar?
5) In the formation of the magnesium oxide salt, magnesium nitride is a possible byproduct. Provide the balanced reaction for this byproduct formation.
6) In the formation of the magnesium chloride salt, what byproduct was produced and how could this component have been collected?

## Experiment \#4: <br> Unknown Solution Determination by Precipitation and Acid-Base Reactions

## Introduction to Chemical Reactions:

1) Most chemical reactions are placed in three categories:
a. Precipitation reactions
i. Two ionic solutions are mixed and produce an ionic solid.
ii. The most common type of precipitation reaction is a double displacement reaction or metathesis reaction.
1. This reaction can also be referred to as an exchange reaction.
2. Example: $2 \mathrm{KBr}(a q)+\mathrm{Pb}\left(\mathrm{NO}_{3}\right)_{2}(a q) \rightarrow \mathrm{PbBr}_{2}(s)+$ $2 \mathrm{KNO}_{3}(a q)$
iii. To predict precipitation reactions, the solubilities of reactants and products must be assessed.
3. If all reactants and products are soluble in water, no product results.
4. If one or more products is insoluble, the reaction will proceed and a solid substance will be observed.

## b. Acid-base neutralization reactions

i. An acid and base are combined to form an ionic compound (i.e. salt) and possibly water (if the base is a hydroxide).
ii. A Brønsted-Lowry acid is defined as a proton donor.

1. Example: HBr or $\mathrm{H}_{2} \mathrm{SO}_{4}$
iii. A Brønsted-Lowry base is defined as a proton acceptor.
2. Example: LiOH or $\mathrm{NH}_{3}$
iv. An example reaction: $\mathrm{LiOH}(a q)+\mathrm{HBr}(a q) \rightarrow \mathrm{LiBr}(a q)+\mathrm{H}_{2} \mathrm{O}(l)$
v. Some ionic salts, when reacted with acid, form gases.
3. The most common gases produced are $\mathrm{CO}_{2}, \mathrm{SO}_{2}$, and $\mathrm{H}_{2} \mathrm{~S}$.
4. Example: $\mathrm{K}_{2} \mathrm{CO}_{3}(a q)+2 \mathrm{HBr}(a q) \rightarrow 2 \mathrm{KBr}(a q)+\mathrm{H}_{2} \mathrm{O}(l)+$ $\mathrm{CO}_{2}(\mathrm{~g})$
a. The $\mathrm{H}_{2} \mathrm{O}(l)+\mathrm{CO}_{2}(g)$ result from the spontaneous decomposition of $\mathrm{H}_{2} \mathrm{CO}_{3}(a q)$.
c. Oxidation-reduction reactions
i. These reactions involve electron transfer where one species is oxidized and another species is reduced.
5. Oxidation: loss of electrons or an increase in the oxidation number.
6. Reduction: gain of electrons or a decrease in the oxidation number.
ii. The most common types of oxidation-reduction reactions are:
7. Combination (or synthesis) reactions
8. Decomposition reactions
9. Single displacement reactions
10. Combustion reactions
2) There are a number of driving forces for reactions to occur:
a. Formation of molecular compounds
i. Example: $\mathrm{H}_{2} \mathrm{O}$
b. Formation of a gas
i. Examples: $\mathrm{H}_{2} \mathrm{~S}, \mathrm{CO}_{2}, \mathrm{SO}_{2}$
c. Formation of an insoluble precipitate
i. See Table A attached for a comprehensive list of soluble and insoluble salts in water.

## Introduction to Double Displacement Reactions:

1) A double displacement reaction (or metathesis reaction) is one in which two ionic compounds in an aqueous solution exchange anions to produce two new species.
2) The driving force for this type of reaction is typically the production of an insoluble precipitate.
a. If all products are soluble, no reaction will take place.
3) Most double displacement reactions can be predicted by analyzing water solubility of the products.
4) An example precipitation reaction is depicted below.
a. $\mathrm{Al}_{2}\left(\mathrm{SO}_{4}\right)_{3}(a q)+6 \mathrm{NaOH}(a q) \rightarrow 2 \mathrm{Al}(\mathrm{OH})_{3}(s)+3 \mathrm{Na}_{2} \mathrm{SO}_{4}(a q)$
5) Most acid-base neutralization reactions can also be classified as double displacement reactions.

## The Experimental Goal:

1) The goal of this experiment is to identify seven aqueous solutions using qualitative analysis.
a. Qualitative analysis is a systematic method of recording precipitation reactions, color changes, and other visible changes to determine chemical composition.
2) Acids and bases will be identified using pH paper.
3) After a pH analysis, all solutions will be mixed together to determine if a reaction has taken place.
4) Once all data have been recorded and potential balanced equations have been written, the unknown solutions can then be determined.

Table A: Solubility Table for Common Anions \& Cations

| Anion/Cation Name(s) | Anion/Cation Formula(s) | Soluble | Insoluble |
| :---: | :---: | :---: | :---: |
| acetate | $\begin{gathered} \mathrm{CH}_{3} \mathrm{CO}_{2}^{-} \text {or } \\ \mathrm{C}_{2} \mathrm{H}_{3} \mathrm{O}_{2}^{-} \end{gathered}$ | most cations | none |
| arsenate, phosphate | $\begin{aligned} & \mathrm{AsO}_{4}{ }^{3-} \\ & \mathrm{PO}_{4}^{3-} \\ & \hline \end{aligned}$ | $\mathrm{NH}_{4}{ }^{+}$, Group IA (except $\mathrm{Li}^{+}$) | most cations |
| bromide, chloride | $\begin{aligned} & \mathrm{Br}^{-} \\ & \mathrm{Cl}^{-} \\ & \hline \end{aligned}$ | most cations | $\begin{gathered} \mathrm{Ag}^{+}, \mathrm{Hg}_{2}{ }^{2+}, \mathrm{Pb}^{2+} \\ \left(\text { and } \mathrm{Cu}^{+}, \mathrm{Tl}^{+}\right) \\ \hline \end{gathered}$ |
| carbonate | $\mathrm{CO}_{3}{ }^{2-}$ | $\mathrm{NH}_{4}{ }^{+}$, Group IA (except $\mathrm{Li}^{+}$) | most cations |
| chlorate, perchlorate | $\begin{aligned} & \mathrm{ClO}_{3-} \\ & \mathrm{ClO}_{4-}^{-} \\ & \hline \end{aligned}$ | most cations | none |
| chromate | $\mathrm{CrO}_{4}{ }^{2-}$ | $\begin{gathered} \mathrm{NH}_{4}^{+}, \text {Group IA }, \mathrm{Ca}^{2+}, \\ \mathrm{Mg}^{2+}, \mathrm{Cu}^{2+} \end{gathered}$ | most cations |
| cyanide | $\mathrm{CN}^{-}$ | $\mathrm{NH}_{4}{ }^{+}$, Group IA (except $\mathrm{Li}^{+}$) | most cations |
| fluoride | F | $\mathrm{NH}_{4}^{+}$, Group IA, $\mathrm{Ag}^{+}$ | most cations |
| hydroxide, oxide | $\underset{\mathrm{O}^{2-}}{\mathrm{OH}^{-} \text {or } \mathrm{HO}^{-}}$ | $\begin{gathered} \mathrm{NH}_{4}^{+}, \text {Group IA, }^{2+} \mathrm{Sr}^{2+}, \\ \mathrm{Ba}^{a^{+}}, \mathrm{Ca}^{2^{+}} \text {(sparingly) } \end{gathered}$ | most cations |
| iodide | $\mathrm{I}^{-}$ | most cations | $\begin{gathered} \mathrm{Ag}^{+}, \mathrm{Hg}_{2}{ }^{2+}, \mathrm{Pb}^{2+} \\ \left(\text { and } \mathrm{Cu}^{+}, \mathrm{Tl}^{+}, \mathrm{Bi}^{3+}, \mathrm{Sn}^{4+}\right) \end{gathered}$ |
| nitrate nitrite | $\begin{aligned} & \mathrm{NO}_{3}^{-} \\ & \mathrm{NO}_{2}^{-} \\ & \hline \end{aligned}$ | most cations | none |
| oxalate | $\mathrm{C}_{2} \mathrm{O}_{4}{ }^{2-}$ | $\mathrm{NH}_{4}{ }^{+}$, Group IA (except Li ${ }^{+}$) | most cations |
| permanganate | $\mathrm{MnO}_{4}{ }^{-}$ | most cations | none |
| silicate | $\mathrm{SiO}_{3}{ }^{2-}$ | Group IA | most cations |
| sulfate | $\mathrm{SO}_{4}{ }^{2-}$ | most cations | $\begin{gathered} \mathrm{Ca}^{2+}, \mathrm{Sr}^{2+}, \mathrm{Ba}^{2+}, \mathrm{Pb}^{2+}, \\ \mathrm{Ag}^{+} \text {(sparingly) } \end{gathered}$ |
| sulfide | $\mathrm{S}^{2-}$ | $\mathrm{NH}_{4}^{+}$, Group IA \& IIA | most cations |
| sulfite | $\mathrm{SO}_{3}{ }^{2-}$ | $\mathrm{NH}_{4}{ }^{+}$, Group IA (except $\mathrm{Li}^{+}$) | most cations |
| thiosulfate | $\mathrm{S}_{2} \mathrm{O}_{3}{ }^{2-}$ | most cations | $\mathrm{Ag}^{+}, \mathrm{Pb}^{2+}$ |
| ////////////////////////\| | /////////////////////////// | ///\|/|/|/|/|/|/|/|/|/|/|/|/|/|/| | ////////////////////////////////// |
| Group IA | $\mathrm{Li}^{+}, \mathrm{Na}^{+}, \mathrm{K}^{+}$ | most anions | none (except for some $\mathrm{Li}^{+}$salts) |
| ammonium | $\mathrm{NH}_{4}{ }^{+}$ | most anions | none |

## The Seven Reactant Solution Unknowns:

A) $\mathrm{AgNO}_{3}(0.10 \mathrm{M})$
B) $\mathrm{BaCl}_{2}(0.10 \mathrm{M})$
C) $\mathrm{Ca}(\mathrm{OH})_{2}$ (saturated or 0.024 M )
D) $\mathrm{HCl}(3.0 \mathrm{M})$
E) $\mathrm{MnSO}_{4}(0.10 \mathrm{M})$
F) $\mathrm{Na}_{2} \mathrm{CO}_{3}(0.10 \mathrm{M})$
G) $\mathrm{Na}_{3} \mathrm{PO}_{4}(0.10 \mathrm{M})$

## The Experimental Procedure:

1) Obtain seven test tubes. Clean and dry if necessary.
a. Label them with the numbers $1-7$.
2) Obtain approximately 15.0 mL of each unknown solution and place them into the appropriately labeled test tube.
a. In addition, obtain seven disposable plastic pipettes.
i. Make sure each pipette is used only for one solution.
ii. It may be advisable to only use the graduated cylinder to measure out the first unknown liquid and "eyeball" the correct amount of liquid to fill the other test tubes. This will prevent contamination.
3) Observe the color and clarity of the solution.
a. Record this physical information into a data table like the one attached.
i. If the solution has a color, report the color.
ii. If the solution has no color, it is referred to as colorless.
iii. If you can see through the solution, it is referred to as clear.
iv. If you cannot see through the solution, it is referred to as opaque.
4) Acquire wide range pH paper ( pHydrion ) and a stirring rod.
5) Immerse the stir rod into the first three unknown solutions and test for the presence of an acid or base.
a. Report the color of the pH paper following the addition of a drop of the unknown solution.
i. Using the card indicator, report an approximate pH and indicate whether the solution is acidic, basic, or neutral.
b. Document results in data table.
c. After testing each solution, clean and dry the stir rod before analyzing the next solution!
6) Obtain 21 clean and dry test tubes.
a. Label the test tubes with all of the 21 possible unknown combinations.
7) Mix each solution combination in the appropriately labeled test tube.
a. Record the initial temperature of the first solution before adding the second solution.
b. Mix between $1-2 \mathrm{~mL}^{*}$ of the aqueous solutions with each other.
i. *This is only an approximation. Add enough of first solution to cover the bulb of thermometer. Next, add roughly the same volume of second unknown.
c. Record the final temperature following mixing.
d. Report the presence or absence of gas formation.
e. Report the presence or absence of a precipitate.
i. If present, indicate the color.

## Waste Disposal and Lab Clean - Up:

1) Place all combinations in the appropriately labeled waste containers.
2) Do not discard any materials down the sink; especially the precipitates.

## The Lab Write - Up:

1) Provide the balanced equation for all reactions including the proper states of matter for all reactants and products.
a. If no reaction occurs, use NR as the abbreviation.
2) Determine the identities of all seven unknown solutions.
a. Also, include the appropriate name for each unknown.
3) In your conclusion, discuss the identity of the seven unknown solutions and how this was determined. This will require a discussion of the data and observations recorded.
4) Discuss all reasons for error and how these errors can affect results.

Sample Data Table for Unknown Color \& pH Test:

| Unknown \# | Solution Color <br> and Clarity |  <br> Approximate $\mathbf{p H}$ | Acid, Base, or <br> Neither |
| :---: | :---: | :---: | :---: |
| 1 |  |  |  |
| 2 |  |  |  |
| 3 |  |  |  |
| 4 |  |  |  |
| 5 |  |  |  |
| 7 |  |  |  |
| 7 |  |  |  |

Sample Data Table for the IUPAC Names for the Reactants:

| Chemical Formula | IUPAC Name |
| :---: | :---: |
| $\mathrm{AgNO}_{3}(0.10 \mathrm{M})$ |  |
| $\mathrm{BaCl}_{2}(0.10 \mathrm{M})$ |  |
| $\mathrm{Ca}(\mathrm{OH})_{2}($ saturated or 0.024 M$)$ |  |
| $\mathrm{HCl}(3.0 \mathrm{M})$ |  |
| $\mathrm{MnSO}_{4}(0.10 \mathrm{M})$ |  |
| $\mathrm{Na}_{2} \mathrm{CO}_{3}(0.10 \mathrm{M})$ |  |
| $\mathrm{Na}_{3} \mathrm{PO}_{4}(0.10 \mathrm{M})$ |  |

## Sample Data Table for Unknown Mixture Reactions:

| Unknown Combination | Heat Evolution: <br> Initial \& Final Temp ( ${ }^{0} \mathrm{C}$ ) | $\begin{gathered} \text { Gas Formation } \\ (+ \text { or }-) \end{gathered}$ | Precipitation \& Color <br> (+ or - w/ color) |
| :---: | :---: | :---: | :---: |
| $1+2$ | Initial: $\qquad$ <br> Final: |  |  |
| $1+3$ | Initial: <br> Final: |  |  |
| $1+4$ | Initial: $\qquad$ <br> Final: |  |  |
| $1+5$ | Initial: <br> Final: |  |  |
| $1+6$ | Initial: <br> Final: |  |  |
| $1+7$ | Initial: <br> Final: |  |  |
| $2+3$ | Initial: $\qquad$ <br> Final: |  |  |
| $2+4$ | Initial: $\qquad$ <br> Final: |  |  |
| $2+5$ | Initial: <br> Final: |  |  |
| $2+6$ | Initial: $\qquad$ <br> Final: |  |  |
| $2+7$ | Initial: <br> Final: |  |  |
| $3+4$ | Initial: $\qquad$ <br> Final: $\qquad$ |  |  |
| $3+5$ | Initial: <br> Final: |  |  |
| $3+6$ | Initial: $\qquad$ <br> Final: |  |  |
| $3+7$ | Initial: <br> Final: |  |  |
| $4+5$ | Initial: $\qquad$ <br> Final: |  |  |
| $4+6$ | Initial: <br> Final: $\qquad$ |  |  |
| $4+7$ | Initial: $\qquad$ <br> Final: |  |  |
| $5+6$ | Initial: $\qquad$ <br> Final: |  |  |
| $5+7$ | Initial: <br> Final: |  |  |
| $6+7$ | Initial: <br> Final: |  |  |

## Sample Data Table for the Balanced Reactions:

| The Reactant(s): | Yields | The Product(s): |
| :---: | :---: | :---: |
| $\mathrm{AgNO}_{3}(a q)+\ldots \mathrm{BaCl}_{2}(a q)$ | $\longrightarrow$ |  |
| $\mathrm{AgNO}_{3}(\mathrm{aq})+\ldots \mathrm{Ca}(\mathrm{OH})_{2}(\mathrm{aq})$ | $\longrightarrow$ |  |
| $\mathrm{AgNO}_{3}(a q)+\ldots \mathrm{HCl}(a q)$ | $\longrightarrow$ |  |
| $\mathrm{AgNO}_{3}(a q)+\ldots \mathrm{MnSO}_{4}(a q)$ | $\longrightarrow$ |  |
| $\mathrm{AgNO}_{3}(\mathrm{aq})+\ldots \mathrm{Na}_{2} \mathrm{CO}_{3}(\mathrm{aq})$ | $\longrightarrow$ |  |
| $\mathrm{AgNO}_{3}(a q)+\ldots \mathrm{Na}_{3} \mathrm{PO}_{4}(\mathrm{aq})$ | $\longrightarrow$ |  |
| $\mathrm{BaCl}_{2}(a q)+\ldots \mathrm{Ca}(\mathrm{OH})_{2}(a q)$ | $\longrightarrow$ |  |
| $\mathrm{BaCl}_{2}(a q)+\ldots \mathrm{HCl}(a q)$ | $\longrightarrow$ |  |
| $\mathrm{BaCl}_{2}(a q)+\ldots \mathrm{MnSO}_{4}(a q)$ | $\longrightarrow$ |  |
| $\mathrm{BaCl}_{2}(\mathrm{aq})+\ldots \mathrm{Na}_{2} \mathrm{CO}_{3}(\mathrm{aq})$ | $\longrightarrow$ |  |
| $\mathrm{BaCl}_{2}(a q)+\ldots \mathrm{Na}_{3} \mathrm{PO}_{4}(a q)$ | $\longrightarrow$ |  |
| $\mathrm{Ca}(\mathrm{OH})_{2}(\mathrm{aq})+\ldots \mathrm{HCl}(\mathrm{aq})$ | $\longrightarrow$ |  |
| $\mathrm{Ca}(\mathrm{OH})_{2}(a q)+\ldots \mathrm{MnSO}_{4}(a q)$ | $\longrightarrow$ |  |
| $\mathrm{Ca}(\mathrm{OH})_{2}(\mathrm{aq})+\ldots \mathrm{Na}_{2} \mathrm{CO}_{3}(\mathrm{aq})$ | $\longrightarrow$ |  |
| $\mathrm{Ca}(\mathrm{OH})_{2}(\mathrm{aq})+\ldots \mathrm{Na}_{3} \mathrm{PO}_{4}(\mathrm{aq})$ | $\longrightarrow$ |  |
| $\mathrm{HCl}(a q)+\ldots \mathrm{MnSO}_{4}(a q)$ | $\longrightarrow$ |  |
| $\mathrm{HCl}(a q)+\ldots \mathrm{Na}_{2} \mathrm{CO}_{3}(\mathrm{aq})$ | $\longrightarrow$ |  |
| $\mathrm{HCl}(\mathrm{aq})+\ldots \mathrm{Na}_{3} \mathrm{PO}_{4}(\mathrm{aq})$ | - |  |
| $\mathrm{MnSO}_{4}(\mathrm{aq})+\ldots \mathrm{Na}_{2} \mathrm{CO}_{3}(\mathrm{aq})$ | $\longrightarrow$ |  |
| $\mathrm{MnSO}_{4}(\mathrm{aq})+\ldots \mathrm{Na}_{3} \mathrm{PO}_{4}(\mathrm{aq})$ | $\longrightarrow$ |  |
| $\mathrm{Na}_{2} \mathrm{CO}_{3}(\mathrm{aq})+\ldots \mathrm{Na}_{3} \mathrm{PO}_{4}(\mathrm{aq})$ | $\longrightarrow$ |  |

Sample Data Table for the Identities of the Unknown Solutions:

| Chemical Formula | Solution Number: |
| :---: | :---: |
| $\mathrm{AgNO}_{3}(0.10 \mathrm{M})$ |  |
| $\mathrm{BaCl}_{2}(0.10 \mathrm{M})$ |  |
| $\mathrm{Ca}(\mathrm{OH})_{2}($ saturated or 0.024 M$)$ |  |
| $\mathrm{HCl}(3.0 \mathrm{M})$ |  |
| $\mathrm{MnSO}_{4}(0.10 \mathrm{M})$ |  |
| $\mathrm{Na}_{2} \mathrm{CO}_{3}(0.10 \mathrm{M})$ |  |
| $\mathrm{Na}_{3} \mathrm{PO}_{4}(0.10 \mathrm{M})$ |  |

## Experiment \#5: Identification of an Unknown Salt by Determination of the Percentage Copper

## Introduction to Percent Composition:

1) Percent composition is defined as the mass percentages of each element in a compound.
2) The mass percentage of element A in a compound is equal to the parts of A per hundred parts of the total, by mass.
a. The equation is as follows: Mass $\% \mathrm{~A}=$ (Mass of A in the compound / Mass of the entire compound) X $100 \%$.
3) An example calculation for nitrobenzene, $\mathrm{C}_{6} \mathrm{H}_{5} \mathrm{NO}_{2}$
a. Step 1: Determine the molar mass of nitrobenzene.
i. Molar mass $=(6 \times 12.011 \mathrm{~g} / \mathrm{mol})+(5 \mathrm{x} 1.0079 \mathrm{~g} / \mathrm{mol})+(1 \mathrm{x}$ $14.0067 \mathrm{~g} / \mathrm{mol})+(2 \times 15.9994 \mathrm{~g} / \mathrm{mol})=123.11 \mathrm{~g} / \mathrm{mol}$
b. Step 2: Calculate the percent composition for each element.
i. C: $(72.066 \mathrm{~g} / \mathrm{mol} / 123.11 \mathrm{~g} / \mathrm{mol}) \times 100 \%=58.54 \%$
ii. $\mathrm{H}:(5.0395 \mathrm{~g} / \mathrm{mol} / 123.11 \mathrm{~g} / \mathrm{mol}) \times 100 \%=4.09 \%$
iii. $\mathrm{N}:(14.0067 \mathrm{~g} / \mathrm{mol} / 123.11 \mathrm{~g} / \mathrm{mol}) \times 100 \%=11.38 \%$
iv. $\mathrm{O}:(31.9988 \mathrm{~g} / \mathrm{mol} / 123.11 \mathrm{~g} / \mathrm{mol}) \times 100 \%=25.99 \%$

## Introduction to the Reactivity Series:

1) A single displacement (single replacement) reaction occurs when an element reacts with a compound to displace an element from that compound.
a. Example \#1: $\mathrm{Zn}(s)+2 \mathrm{AgNO}_{3}(a q) \rightarrow \mathrm{Zn}\left(\mathrm{NO}_{3}\right)_{2}(a q)+2 \mathrm{Ag}(s)$
b. Example \#2: $2 \mathrm{Al}(s)+6 \mathrm{HCl}(a q) \rightarrow 2 \mathrm{AlCl}_{3}(a q)+3 \mathrm{H}_{2}(g)$
2) Whether a reaction takes place depends on the relative reactivity of the elemental metal to the displaced element.
a. If the elemental metal is more reactive than the displaced element, then a reaction will take place.
b. If the elemental metal is less reactive than the displaced element, then a reaction will NOT take place.
3) The reactivity series provides a list of the common metals in order of decreasing reactivity (from the most reactive to the least.)
a. The reactivity series is as follows: $\mathrm{Li}, \mathrm{K}, \mathrm{Ba}, \mathrm{Ca}, \mathrm{Na}, \mathrm{Mg}, \mathrm{Al}, \mathrm{Zn}, \mathrm{Cr}, \mathrm{Fe}$, $\mathrm{Cd}, \mathrm{Co}, \mathrm{Ni}, \mathrm{Sn}, \mathrm{Pb}, \mathrm{H}_{2}, \mathrm{Cu}, \mathrm{Hg}, \mathrm{Ag}, \mathrm{Au}$
4) The reactivity series also reveals which elements react with water and/or acids to produce hydrogen gas.
a. Metals lithium to sodium ( $\mathrm{Li}-\mathrm{Na}$ ) react violently with liquid water (and acid) to produce hydrogen gas.
b. Metals magnesium to cadmium ( $\mathrm{Mg}-\mathrm{Cd}$ ) react slowly with liquid water but considerably with acids to produce hydrogen gas.
c. Metals cobalt to lead $(\mathrm{Co}-\mathrm{Pb})$ do NOT react with liquid water but considerably with acids to produce hydrogen gas.
d. Metals copper to gold $(\mathrm{Cu}-\mathrm{Au})$ react neither with liquid water nor acids.

## The Copper Salts*:

1) $\mathrm{Cu}_{3}\left(\mathrm{PO}_{4}\right)_{2} \cdot 3 \mathrm{H}_{2} \mathrm{O}$
2) $\mathrm{CuCl}_{2} \cdot 2 \mathrm{H}_{2} \mathrm{O}$
3) $\mathrm{CuSO}_{4} \cdot 5 \mathrm{H}_{2} \mathrm{O}$
4) $\mathrm{Cu}\left(\mathrm{ClO}_{4}\right)_{2} \cdot 6 \mathrm{H}_{2} \mathrm{O}$
5) $\mathrm{CuSeO}_{4} \cdot 5 \mathrm{H}_{2} \mathrm{O}$
6) $\mathrm{Cu}\left(\mathrm{CH}_{3} \mathrm{CO}_{2}\right)_{2} \cdot \mathrm{H}_{2} \mathrm{O}$

## The Experimental Procedure:

1) On an analytical balance, weigh out approximately 3.0 grams of the unknown copper salt in a weigh boat or on a piece of weigh paper.
a. Record the unknown number/letter and the color of the salt in your lab notebook.
b. Record the mass in your lab notebook.
2) Transfer the unknown copper salt into a 250 mL beaker.
a. Ensure all powder is in the beaker, avoid spilling.
b. If material clings to weigh boat, transfer into the beaker using the deionized water in the next step.
3) Using a 100 mL graduated cylinder, obtain 100.0 mL of deionized water and add to the beaker containing the unknown copper salt.
4) Place beaker onto a wire gauze on an iron ring clamped to a ring stand.*
a. A replica of the apparatus is shown below.
b. *The procedure can be modified by using a hot plate instead of a Bunsen burner.
5) Gently heat the solution to dissolve salt using a low Bunsen burner flame.
a. Stir with a glass stir rod until dissolved.
b. Do NOT allow for a significant boil.
6) Weigh out 3.0 grams of mossy zinc and cautiously add to the solution.
a. Do NOT allow any of the liquid material to splash out.
7) Continue heating the solution using a stirring rod to mix thoroughly.
a. Do NOT allow for a significant boil.
b. Do NOT remove the stirring rod from the beaker; some of the copper solution will cling to the glass.
8) Continue stirring the heated solution until the solution color completely fades.
a. Add an additional 1.0 g of mossy zinc if all zinc reacts and the color remains.
9) Once the solution is completely colorless, discontinue heating and allow to cool for a few minutes.
10) Obtain 15.0 mL of 6.0 M sulfuric acid.
11) After cooling for a few minutes, slowly add the sulfuric acid to the solution 1 mL at a time.
a. This addition step should take at least 5 minutes.
b. This step is to remove unreacted zinc.
12) A few minutes after this addition, remove all chunks of unreacted zinc with forceps.
a. Be careful to rinse off all bits of zinc with deionized water before removing from the solution.
13) You are now ready to filter; please proceed to the filtration process designated by your instructor.

## Gravity Filtration Procedure:

1) Obtain a piece of filter paper and a watch glass and record the mass on an analytical balance.
2) Create a cone with the filter paper by folding it in half twice to form a quarter of a circle and then separating one side from the other three.
3) Place filter paper into a glass filter and then place filter onto a 500 mL Erlenmeyer flask.
4) Wet the filter with a small amount of deionized water.
a. Ensure that you remove the bubbles between the filter paper and funnel.
5) Once the filter paper is secure, transfer the contents of the beaker into the filter paper.
a. The water should pass through the filter leaving behind the copper.
b. Do not allow the liquid volume to exceed the height of the filter paper.
c. To prevent loss of product, it may be advisable to transfer the solution carefully down the length of a glass stir rod and onto the filter paper.
d. Ensure all of the copper is collected by rinsing beaker well and transferring onto filter paper.
6) Measure out approximately 50.0 mL of deionized water and place into a 100 250 mL beaker.
7) Measure out 5.0 mL of 6.0 M sulfuric acid with a graduated cylinder and then add to the beaker of deionized water to create an acid rinse.
a. This step is necessary to ensure all zinc has been eliminated.
b. Make sure you add the acid TO the deionized water and not the reverse.
8) Add this acid rinse to your collected copper and allow liquid to filter through.
9) Following the addition of the acid rinse, wash thoroughly with copious amounts of deionized water.
a. If some acid remains, the filter paper will burn in the oven.
b. Ensure the filter paper is free from acid.
10) After the filter paper has drained from the last rinse, transfer filter paper with contents onto the pre-weighed watch glass.
11) Place sample into a warm oven to dry.
a. Allow at least 20-30 minutes to ensure dryness.
12) Once dry, carefully remove from the oven and allow to cool to room temperature.
13) Once cool, obtain the mass of the sample and record on data table.

## Vacuum Filtration Procedure:

1) Set up a vacuum filtration apparatus as shown in the attached diagram.
a. Clamp the filter flask securely to a ring stand before affixing vacuum tubing and turning on vacuum.
2) Pre-weigh a piece of filter paper and then place into the Büchner funnel of the vacuum filtration system.
3) Turn on the vacuum first and then wet the filter paper with small amounts of deionized water.
a. Ensure that the filter paper has created a good seal inside the Büchner funnel and no bubbling of the filter paper is observed.
4) Slowly, transfer the contents of the beaker onto the filter paper.
a. The water should pass through the filter paper leaving behind the copper.
b. Do not allow the liquid volume to exceed the height of the filter paper.
c. To prevent loss of product, it may be advisable to transfer the solution carefully down the length of a glass stir rod and onto the filter paper.
d. Ensure all of the copper is collected by rinsing beaker well and transferring onto filter paper.
5) Measure out approximately 50.0 mL of deionized water and place into a 100 250 mL beaker.
6) Measure out 5.0 mL of 6.0 M sulfuric acid with a graduated cylinder and then add to the beaker of deionized water to create an acid rinse.
a. This step is necessary to ensure all zinc has been eliminated.
b. Make sure you add the acid TO the deionized water and not the reverse.
7) Add this acid rinse to your collected copper and allow liquid to filter through.
8) Following the addition of the acid rinse, wash thoroughly with copious amounts of deionized water.
a. If some acid remains, the filter paper will burn in the oven.
b. Ensure the filter paper is free from acid.
9) After the filter paper has drained from the last rinse, transfer filter paper with contents onto the pre-weighed watch glass.
10) Place sample into a warm oven to dry.
a. Allow at least 20-30 minutes to ensure dryness.
11) Once dry, carefully remove from the oven and allow to cool to room temperature.
12) Once cool, obtain the mass of the sample and record on data table.

## Waste Disposal and Clean-Up:

1) Place all acidic filtrates into labeled waste beakers under the hood for neutralization.
2) All copper and unreacted zinc should be placed in the appropriate waste containers.

## The Lab Write - Up:

1) Provide the IUPAC name, molar mass, and percent copper for all five unknown copper salts.
2) Determine all values in your data table.
3) Provide a balanced equation for all possible reactions.
4) Calculate the percent copper in your unknown copper salt and correctly identify which copper salt you were given. In your conclusion, compare the experimental percent copper determined with all possible copper salts.
5) In addition, explain all relevant data collected in conclusion and discuss reasons for error.
6) Reflecting back onto previous labs, what would be another way to identify the unknown copper salt? Briefly provide a step-by-step procedure for this alternate method.

## Sample Data Table for Percent Copper in Reactants:

| No. | Formula of Copper <br> Salt | IUPAC Name | Molar Mass <br> (g/mol) | Percent <br> Copper |
| :---: | :---: | :---: | :---: | :---: |
| 1 | $\mathrm{Cu}_{3}\left(\mathrm{PO}_{4}\right)_{2} \cdot 3 \mathrm{H}_{2} \mathrm{O}$ |  |  |  |
| 2 | $\mathrm{CuCl}_{2} \cdot 2 \mathrm{H}_{2} \mathrm{O}$ |  |  |  |
| 3 | $\mathrm{CuSO}_{4} \cdot 5 \mathrm{H}_{2} \mathrm{O}$ |  |  |  |
| 4 | $\mathrm{Cu}\left(\mathrm{ClO}_{4}\right)_{2} \cdot 6 \mathrm{H}_{2} \mathrm{O}$ |  |  |  |
| 5 | $\mathrm{CuSeO}_{4} \cdot 5 \mathrm{H}_{2} \mathrm{O}$ |  |  |  |
| 6 | $\mathrm{Cu}\left(\mathrm{CH}_{3} \mathrm{CO}_{2}\right)_{2} \cdot \mathrm{H}_{2} \mathrm{O}$ |  |  |  |

## Sample Data Table:

a) Unknown number/letter of copper salt: $\qquad$
b) Mass of unknown copper salt in grams: $\qquad$
c) Mass of mossy zinc in grams: $\qquad$
d) Mass of watch glass + filter paper in grams:
e) Mass of watch glass + filter paper + copper in grams: $\qquad$
f) Mass of copper in grams (e -d ): $\qquad$
g) Experimental percent copper in salt (f/b X 100\%): $\qquad$
h) Identity of copper salt based on percent copper: $\qquad$
i) Balanced equation for the reaction: $\qquad$
j) Balanced equation for zinc elimination: $\qquad$

## Model of the Apparatus:



Note: A second iron ring can be placed around to top of the beaker for added security if necessary.

## Model of Vacuum Filtration Using a Büchner Funnel



Clamp filter flask securely to a ring stand before affixing vacuum tubing and turning on vacuum.

## Experiment \#6A: <br> Gravimetric Analysis of Heavy Metal Solutions; Lead Analysis of a Contaminated Water Sample

## Introduction to Heavy Metals:

1) Heavy metals are commonly classified as metallic elements that contain a $d$ orbital and have high molar masses.
2) Many of these heavy metals are regulated by the Environmental Protection Agency (EPA) due to their toxicity to plant and animal life.
a. The EPA sets a maximum contaminant level (MCL) that indicates the highest concentration allowed in drinking water. These levels are enforced by this agency.
3) Furthermore, many of these elements, following long-term exposure, can accumulate in the body leading to ill health effects.
4) The common heavy metals that are under strict EPA control are the following elements:
a. Arsenic (As)
i. Arsenic is a metalloid element that has been linked to cancer of the bladder, lungs, skin, kidneys, and liver ${ }^{1}$.
ii. In addition, acute arsenic poisoning can cause severe stomach pain, nausea, vomiting, diarrhea, peripheral neuropathy, partial paralysis, delirium, blindness, and death ${ }^{1,2}$.
iii. The maximum contaminant level (MCL) in drinking water is 0.010 parts per million $(0.010 \mathrm{mg} / \mathrm{L})^{1,2}$.
b. Cadmium (Cd)
i. Cadmium is a group 12 (IIB) transition element that has utility in photography, machinery, and in batteries.
ii. Acute cadmium poisoning can cause nausea, vomiting, diarrhea, convulsions, muscle cramping, liver and kidney failure, and death ${ }^{1,2}$.
iii. Long term exposure to cadmium results in significant bioaccumulation which can cause cancer, osteoporosis, osteomalacia, hyperuricemia, and liver and kidney failure ${ }^{1,2,3}$.
iv. The MCL in drinking water is 0.5 parts per billion (0.005 $\mathrm{mg} / \mathrm{L})^{1,2,3}$.
c. Chromium (Cr)
i. Chromium is a group 6 (VIB) transition element that exists in multiple valence states such as divalent (II), trivalent (III), and hexavalent (VI).
ii. Trivalent chromium is an essential trace mineral in the human body with a recommended daily intake of $50-200 \mu \mathrm{~g} /$ day in adults ${ }^{1}$.
iii. Hexavalent chromium or chromium (VI) is the most toxic valence state and acute poisoning can cause nausea, vomiting, diarrhea, coughing, shortness of breath, skin reactions, and hemorrhage ${ }^{1,2,3}$.
iv. Longterm exposure to hexavalent chromium can cause cancer and genotoxicity ${ }^{1,2,3}$.
v. The MCL in drinking water for total chromium (trivalent plus hexavalent chromium) is 100 parts per billion $(0.1 \mathrm{mg} / \mathrm{L})^{1,2,3}$.
d. $\operatorname{Iron}(\mathrm{Fe})$
i. Iron is a group 8 (VIIIB) transition element that is essential to the human body.
ii. Iron is incorporated into the heme complex system for many animals, which allows iron to be carried by hemoglobin or myoglobin for oxygen transport.
iii. The two common valences for iron are iron(II) or ferrous iron and iron(III) or ferric iron.
iv. Iron poisoning is the leading cause of death for accidental overdose in children under six ${ }^{1,3}$.
v. Acute iron toxicity has a three phase effect ${ }^{1,3}$.
1. During phase one, immediately following the overdose, the stomach lining becomes ulcerated causing nausea and vomiting.
2. During the second phase, called the latent phase, the pain subsides as the iron begins to damage the internal organs.
3. During the last phase, metabolic acidosis develops and severe damage to the brain and liver occur.
vi. The MCL in drinking water is $0.3 \mathrm{mg} / \mathrm{L}^{1}$.
e. Lead (Pb)
i. Lead is a group 14 (IVA) element that was used in the manufacture of paint prior to 1978 and used as an antiknock gasoline additive in the 1980 's ${ }^{1,3}$.
ii. Acute exposure to lead can cause irritability, insomnia, headache, severe gastrointestinal discomfort, seizure, and coma ${ }^{1,2,3}$.
iii. Long - term exposure is associated with anemia, kidney dysfunction, reproductive harm, and extreme learning disabilities ${ }^{1,2,3}$.
iv. The MCL in drinking water is 15 parts per billion $(0.015 \mathrm{mg} / \mathrm{L})^{1,3}$.
f. Mercury ( Hg )
i. Mercury, like cadmium, is a group 12 (IIB) transition element.
ii. Organic mercury compounds, such as methyl mercury, are the most toxic mercury species and are one of the most regulated materials in the Earth's water systems.
iii. Upon exposure to mercury compounds, the central nervous system, endocrine system, and hepatic systems are at high risk of damage ${ }^{1,3}$.
iv. The World Health Organization states that the primary sources of environmental exposure is through consumption of contaminated fish ${ }^{1,3}$.
v. The MCL in drinking water is 2 parts per billion $(0.002 \mathrm{mg} / \mathrm{L})^{1,3}$.

Table A: Solubility Table for Common Anions \& Cations

| Anion/Cation Name(s) | Anion/Cation Formula(s) | Soluble | Insoluble |
| :---: | :---: | :---: | :---: |
| acetate | $\begin{gathered} \mathrm{CH}_{3} \mathrm{CO}_{2}^{-} \text {or } \\ \mathrm{C}_{2} \mathrm{H}_{3} \mathrm{O}_{2}^{-} \\ \hline \end{gathered}$ | most cations | none |
| arsenate, phosphate | $\begin{aligned} & \mathrm{AsO}_{4}^{3-} \\ & \mathrm{PO}_{4}{ }^{3-} \end{aligned}$ | $\mathrm{NH}_{4}{ }^{+}$, Group IA (except Li ${ }^{+}$) | most cations |
| bromide, chloride | $\begin{aligned} & \mathrm{Br}^{-} \\ & \mathrm{Cl}^{-} \end{aligned}$ | most cations | $\begin{gathered} \mathrm{Ag}^{+}, \mathrm{Hg}_{2}{ }_{2+}, \mathrm{Pb}^{2+} \\ \left(\text { (and } \mathrm{Cu}^{+}, \mathrm{Tl}^{+}\right) \end{gathered}$ |
| carbonate | $\mathrm{CO}_{3}{ }^{2-}$ | $\mathrm{NH}_{4}{ }^{+}$, Group IA (except $\mathrm{Li}^{+}$) | most cations |
| chlorate, perchlorate | $\begin{aligned} & \mathrm{ClO}_{3-}- \\ & \mathrm{ClO}_{4} \end{aligned}$ | most cations | none |
| chromate | $\mathrm{CrO}_{4}{ }^{2-}$ | $\begin{gathered} \mathrm{NH}_{4}^{+}, \text {Group IA }, \mathrm{Ca}^{2+} \\ \mathrm{Mg}^{2+}, \mathrm{Cu}^{2+} \end{gathered}$ | most cations |
| cyanide | $\mathrm{CN}^{-}$ | $\mathrm{NH}_{4}{ }^{+}$, Group IA (except $\mathrm{Li}^{+}$) | most cations |
| fluoride | F | $\mathrm{NH}_{4}^{+}$, Group IA, $\mathrm{Ag}^{+}$ | most cations |
| hydroxide, oxide | $\begin{gathered} \mathrm{OH}^{-} \text {or } \mathrm{HO}^{-} \\ \mathrm{O}^{2-} \\ \hline \end{gathered}$ | $\begin{gathered} \mathrm{NH}_{4}^{+}, \text {Group IA, } \mathrm{Sr}^{2+}, \\ \mathrm{Ba}^{2+}, \mathrm{Ca}^{2+} \text { (sparingly) } \end{gathered}$ | most cations |
| iodide | I | most cations | $\begin{gathered} \mathrm{Ag}^{+}, \mathrm{Hg}_{2}{ }^{2+}, \mathrm{Pb}^{2+} \\ \left(\text { and } \mathrm{Cu}^{+}, \mathrm{Tl}^{+}, \mathrm{Bi}^{3+}, \mathrm{Sn}^{4+}\right) \end{gathered}$ |
| nitrate nitrite | $\begin{aligned} & \mathrm{NO}_{3}^{-} \\ & \mathrm{NO}_{2}^{-} \end{aligned}$ | most cations | none |
| oxalate | $\mathrm{C}_{2} \mathrm{O}_{4}{ }^{2-}$ | $\mathrm{NH}_{4}{ }^{+}$, Group IA (except $\mathrm{Li}^{+}$) | most cations |
| permanganate | $\mathrm{MnO}_{4}^{-}$ | most cations | none |
| silicate | $\mathrm{SiO}_{3}{ }^{2-}$ | Group IA | most cations |
| sulfate | $\mathrm{SO}_{4}{ }^{2-}$ | most cations | $\begin{gathered} \mathrm{Ca}^{2+}, \mathrm{Sr}^{2+}, \mathrm{Ba}^{2+}, \mathrm{Pb}^{2+}, \\ \mathrm{Ag}^{+} \text {(sparingly) } \end{gathered}$ |
| sulfide | $\mathrm{S}^{2-}$ | $\mathrm{NH}_{4}^{+}$, Group IA \& IIA | most cations |
| sulfite | $\mathrm{SO}_{3}{ }^{2-}$ | $\mathrm{NH}_{4}{ }^{+}$, Group IA (except $\mathrm{Li}^{+}$) | most cations |
| thiosulfate | $\mathrm{S}_{2} \mathrm{O}_{3}{ }^{2-}$ | most cations | $\mathrm{Ag}^{+}, \mathrm{Pb}^{2+}$ |
| //////////////////////// |  | ///\|/|/|/|/|/|/|/|/|/|/|/|/|/|/|/ |  |
| Group IA | $\mathrm{Li}^{+}, \mathrm{Na}^{+}, \mathrm{K}^{+}$ | most anions | none (except for some <br> $\mathrm{Li}^{+}$salts) |
| ammonium | $\mathrm{NH}_{4}^{+}$ | most anions | none |

## Introduction to Gravimetric Analysis:

1) Gravimetric Analysis is a type of quantitative analysis in which the amount of a species in a material is determined by converting to a product that can be isolated and weighed.
a. Precipitation reactions are performed to produce quantitative amounts of a known insoluble heavy metal complex.
2) The advantages of using gravimetric analysis over other methods are as follows:
a. It is provides an exceedingly precise measurement.
b. There is little room for instrumental error.
c. A series of standards are not required.
d. Inexpensive equipment (i.e. an analytical balance) is used.
3) The disadvantages for using gravimetric analysis over other methods are as follows:
a. This technique is limited to analysis of a single element/material at once.
b. Other elements/materials, such as colloids, can interfere with the analysis.
4) Using this technique, the molarity ( $\mathrm{mol} / \mathrm{L}$ ) and mass concentration ( $\mathrm{g} / \mathrm{L}$ or $\mathrm{mg} / \mathrm{L}$ ) of the heavy atom can be determined in the original solution.

## The Heavy Metal Contaminant:

1) $\mathrm{Pb}\left(\mathrm{NO}_{3}\right)_{2}$

## The Known Solution:

1) $0.50 \mathrm{M}-1.0 \mathrm{M} \mathrm{Na}_{2} \mathrm{SO}_{4}$

## The Experimental Goal:

1) Each student will be assigned a water sample contaminated with $\mathrm{Pb}\left(\mathrm{NO}_{3}\right)_{2}$ of an unknown concentration.
2) The goal is to determine the concentration of lead in the water sample and determine if passes the EPA standard for human consumption.

## The Experimental Procedure for Formation of Heavy Metal Precipitate:

1) Please wear goggles, gloves, and proper attire throughout this experiment and do not discard any lead samples down the drain.
2) You will be assigned a water sample contaminated with $\mathrm{Pb}\left(\mathrm{NO}_{3}\right)_{2}$ of an unknown concentration.
a. Record the unknown number/letter and the color \& clarity of the solution in your lab notebook.
3) Using a 10.0 mL pipette, transfer exactly 10.0 mL of your unknown solution into a $50-125 \mathrm{~mL}$ Erlenmeyer flask.
4) Using a 10.0 mL graduated cylinder, measure out 10.0 mL of $0.50 \mathrm{M}-1.0 \mathrm{M}$ $\mathrm{Na}_{2} \mathrm{SO}_{4}$ solution.
a. Record color \& clarity of the sodium sulfate solution.
5) Transfer the sodium sulfate solution into the Erlenmeyer flask with your unknown heavy metal solution.
a. Observe the precipitate and record the color in your lab notebook.
6) You are now ready to filter; please proceed to the filtration process designated by your instructor.

## Gravity Filtration Procedure:

1) Obtain a Whatman filter paper and a watch glass.
a. Record the mass of the filter paper and watch glass together on an ANALYTICAL balance.
b. For best results, use 9.0 cm Whatman filter paper \#44 or \#42 (slow, ashless).
2) Flute the filter paper by repeatedly folding in half and place into a glass funnel.
3) Place onto a $250-500 \mathrm{~mL}$ Erlenmeyer flask.
4) Wet the filter with a small amount of deionized water.
5) Once the filter paper is secure, transfer the contents of the Erlenmeyer flask into the filter paper.
a. The water should pass through the filter leaving behind the precipitate.
b. Do not allow the liquid volume to exceed the height of the filter paper.
c. To prevent loss of product, it may be advisable to transfer the solution carefully down the length of a glass stir rod and onto the filter paper.
6) After the liquid has passed through the filter, observe the filtrate solution in the filter flask.
a. If precipitate has passed through the filter, remove the solution from the flask and re-filter through the funnel.
b. If no precipitate has passed through the filter, continue with the experiment.
7) Rinse out your Erlenmeyer flask with deionized water and place into the filter to ensure all precipitate has been collected.
8) Once most of the water has passed through the filter and you are left with a wet cake, you can proceed to the next step.
9) Transfer your filter paper and precipitate onto the pre-weighed watch glass and allow to dry as instructed.
a. The sample can be air dried, dried in a dessicator, or dried in a warm oven.
10) Once the precipitate has dried, record the mass of the watch glass, filter paper, and precipitate.

## Vacuum Filtration Procedure:

1) Set up a vacuum filtration apparatus as shown in the attached diagram.
a. Clamp the filter flask securely to a ring stand before affixing vacuum tubing and turning on vacuum.
2) Obtain a Whatman filter paper and a watch glass.
a. Record the mass of the filter paper and watch glass together on an ANALYTICAL balance.
b. For best results, use Whatman filter paper \#44 or \#42 (slow, ashless).
3) Turn on the vacuum first and then wet the filter paper with small amounts of deionized water.
a. Ensure that the filter paper has created a good seal inside the Büchner funnel and no bubbling of the filter paper is observed.
4) Slowly, transfer the contents of the Erlenmeyer flask into the filter paper.
a. The water should pass through the filter leaving behind the precipitate.
b. Do not allow the liquid volume to exceed the height of the filter paper.
c. To prevent loss of product, it may be advisable to transfer the solution carefully down the length of a glass stir rod and onto the filter paper.
5) After the liquid has passed through the filter, observe the filtrate solution in the filter flask.
a. If precipitate has passed through the filter, remove the solution from the flask and re-filter through the funnel.
b. If no precipitate has passed through the filter, continue with the experiment.
6) Rinse out your Erlenmeyer flask with deionized water and place into the filter to ensure all precipitate has been collected.
7) Once most of the water has passed through the filter and you are left with a wet cake, you can proceed to the next step.
8) Transfer your filter paper and precipitate onto the pre-weighed watch glass and allow to dry as instructed.
a. The sample can be air dried, dried in a dessicator, or dried in a warm oven.
9) Once the precipitate has dried, record the mass of the watch glass, filter paper, and precipitate.

## Waste Disposal and Lab Clean - Up:

1) If no lead precipitate is present, place all filtrate solutions down the sink.
2) If lead precipitate is still present, place filtrate solutions into designated waste container.
3) All solid precipitates should be placed in the appropriate waste containers.

## The Lab Write - Up:

1) Determine all values in data table \#1 and discuss this data in the conclusion.
a. Calculate the molarity and the mass concentrations (in both $\mathrm{g} / \mathrm{L}$ and $\mathrm{mg} / \mathrm{L}$ ) of your heavy metal in your original solution.
2) Would this water sample have been safe for drinking?
a. Explain by comparing your value to EPA limits.
3) Report the balanced equation for your reaction in your conclusion including observations.
4) Discuss reasons for error and how these errors affect results.
a. What other ions present in the water sample could affect results?
b. What are some disadvantages to using gravimetric analysis over other techniques?
5) Provide IUPAC names for all four heavy metal nitrates in data table \#2.
6) Provide balanced equations (including states of matter) for all gravimetric analysis reactions in data table $\# 3$. For $\mathrm{Cd}\left(\mathrm{NO}_{3}\right)_{2}$, select an appropriate reagent that would produce a precipitate and provide products.

## Data Table \#1: Gravimetric Analysis:

| Data Point to be Recorded/Calculated | Data Point |
| :---: | :---: |
| a) Unknown solution letter/number given: |  |
| b) Volume of unknown heavy metal solution: | 10.0 mL |
| c) Volume of $0.50 \mathrm{M}-1.0 \mathrm{M} \mathrm{Na}_{2} \mathrm{SO}_{4}$ : | 10.0 mL |
| d) Evidence of reaction/Reaction observations: |  |
| e) Mass of watch glass + filter paper in grams: |  |
| f) Mass of watch glass + filter paper + precipitate in grams: |  |
| g) Mass of sulfate precipitate in grams ( $\mathrm{f}-\mathrm{e}$ ): |  |
| h) Molecular formula of heavy metal sulfate: |  |
| i) Molar mass of heavy metal sulfate in $\mathrm{g} / \mathrm{mol}$ : |  |
| j) Percent composition of heavy metal in heavy metal sulfate in $\%$ : |  |
| k) Mass of heavy metal in precipitate in grams ([j/100\%] x g): |  |
| l) Moles of heavy metal $\left(\mathrm{k} / \mathrm{M}_{\mathrm{m}}\right.$ of a single heavy metal): |  |
| m) Molarity of heavy metal in original solution in $\mathrm{mol} / \mathrm{L}(\ell / 0.0100 \mathrm{~L})$ : |  |
| n) Mass concentration of heavy metal in original solution in $\mathrm{g} / \mathrm{L}(\mathrm{k} / 0.0100 \mathrm{~L})$ |  |
| o) Mass concentration of heavy metal in original solution in $\mathbf{m g} / \mathbf{L}(\mathrm{n} \times[1000 \mathrm{mg} / \mathrm{g}])$ |  |
| p) EPA mass concentration limit in drinking water in $\mathrm{mg} / \mathrm{L}$ : |  |
| o) Safe for human consumption (yes or no): |  |

Data Table \#2: IUPAC Nomenclature of Heavy Metal Nitrates:

| Heavy Metal Nitrate | IUPAC Name |
| :---: | :---: |
| $\mathrm{Pb}\left(\mathrm{NO}_{3}\right)_{2}$ |  |
| $\mathrm{Hg}_{2}\left(\mathrm{NO}_{3}\right)_{2}$ |  |
| $\mathrm{Fe}\left(\mathrm{NO}_{3}\right)_{3}$ |  |
| $\mathrm{Cd}\left(\mathrm{NO}_{3}\right)_{2}$ |  |

## Data Table \#3:

Balanced Equations for Common Gravimetric Analyses:

| The Reactant(s): | Yields | The Product(s): |
| :---: | :---: | :---: |
| $\mathrm{Pb}\left(\mathrm{NO}_{3}\right)_{2}(a q)+\ldots \mathrm{Na}_{2} \mathrm{SO}_{4}(a q)$ | $\longrightarrow$ |  |
| $\mathrm{Hg}_{2}\left(\mathrm{NO}_{3}\right)_{2}(\mathrm{aq})+\ldots \mathrm{NaCl}(\mathrm{aq})$ | - |  |
| 1) $\mathrm{Fe}\left(\mathrm{NO}_{3}\right)_{3}(a q)+\ldots \mathrm{NaOH}(a q)$ | $\longrightarrow$ | $(s)+\quad(a q)$ |
| $2)$ | $\xrightarrow{\Delta}$ | $\mathrm{Fe}_{2} \mathrm{O}_{3}(s)+\ldots \mathrm{H}_{2} \mathrm{O}(g)$ |
| $\mathrm{Cd}\left(\mathrm{NO}_{3}\right)_{2}(a q)+\ldots$ | - |  |

References:

1. www.epa.gov
2. http://water.epa.gov/drink/contaminants/upload/mcl-2.pdf
3. http://www.atsdr.cdc.gov/

# Experiment \#6B: <br> A Volumetric Titration; Quality Control Analysis of the Acetic Acid Content in Vinegar 

## Introduction to Volumetric Analysis and Titrations:

1) Volumetric analysis is an investigative method used to quantitatively determine the concentration of an unknown analyte in a specified volume.
2) A titration is the laboratory technique employed to perform a volumetric analysis.
a. In a titration, a standard solution of a known concentration (the titrant) is reacted with the analyte of an unknown concentration (or titrand).
i. The titrant is commonly a strong acid or base.
ii. The analyte (titrand) is commonly a weak acid or weak base.
b. As the titrant is slowly added to the analyte (which generally contains an added indicator), a visible color change is observed when the endpoint is reached.
c. This visual observance of the endpoint of a titration is commonly used to determine the equivalence point, the point where equivalent amounts of titrant and unknown are present and the reaction is complete.
3) A volumetric analysis is most often performed with the aid of a buret.
4) A buret (or burette) is a laboratory instrument used to precisely measure the amount of a titrant delivered to an analyte solution.
a. Burets are cylindrical glass tubes, used vertically, with a stopcock on the end which controls titrant delivery.
b. These instruments are very accurate analytical tools to measure delivered volumes.
c. A depiction of a buret reading is show on the left.

## Introduction to Concentration:

1) Concentration is the amount of solute present in a given amount of solvent or solution.
2) Concentration can be expressed using different units.
a. Molarity $(M)$ is defined as the moles of solute dissolved in one liter (cubic decimeter) of solution.
i. Molarity $(M)=($ moles of solute $) /$ (liter of solution)
b. Mass percentage (or percentage by mass) is defined as the mass of solute dissolved in the mass of solution times 100 percent.
i. Mass percentage $=[($ mass of solute $) /($ mass of solution $)] \times 100 \%$

## Introduction to Quality Control and Statistical Data:

1) Quality control ( QC ) is a practice for ensuring the preservation of proper standards in manufactured materials by employing regular but random inspection of product quality.
2) QC is utilized in almost every industry including manufacturers of food, drugs, and industrial and commercial products.
3) Statistical analysis can be employed to analyze and interpret data collected.
a. In this experiment, the measure of central tendency (or average) will be determined by the mean concentration.
i. The mean is the sum of all data points divided by the sample size (or number of data points.)
b. This mean concentration will be compared to the manufacturer's labeled concentration to determine a percent difference (much like a percent error).
i. Percent difference $=$ [(|Experimental value - Manufacturer's value|] x 100\%
ii. Unless more stringent guidelines are applied, a percent difference of less than $5 \%$ is considered an acceptable difference.

## Introduction to the Titration Reaction:

1) The acetic acid content in a vinegar sample will be titrated with a standardized sodium hydroxide solution.
2) The balanced equation is shown below.

$$
\mathrm{HC}_{2} \mathrm{H}_{3} \mathrm{O}_{2}(a q)+\mathrm{NaOH}(a q) \rightarrow \mathrm{H}_{2} \mathrm{O}(l)+\mathrm{NaC}_{2} \mathrm{H}_{3} \mathrm{O}_{2}(a q)
$$

## The Experimental Goal:

1) The goal of this experiment is to determine the concentration of acetic acid in vinegar and compare this experimental value to the manufacturer's label for purposes of quality control.

## Procedure for the Volumetric Titration of the Acetic Acid in Vinegar:

3) Obtain three 250 mL Erlenmeyer flasks, a 100 mL beaker, a 25.0 mL graduated cylinder, two 250 mL beakers, a wax/grease pencil, a buret, a buret clamp, a buret or ring stand, a short stem funnel, 10 mL volumetric pipet, and a pipetting device/pump.
4) Using the wax/grease pencil, label the three 250 mL Erlenmeyer flasks as "titration \#1," "titration \#2," and "titration \#3."
5) Obtain the mass of each empty titration flask on an analytical balance and record on your data table.
6) For the vinegar to be analyzed, record the following information in your data table:
a. Brand of vinegar (company)
b. Expiration date
c. Percent acidity (mass percent acetic acid) on manufacturer's label
7) Using the 100 mL beaker, obtain approximately 50 mL of vinegar.
8) Using the 10.00 mL volumetric pipet with pipetting device/pump, add 10.00 mL of vinegar into each of the three labeled 250 mL Erlenmeyer flasks.
9) Obtain the mass of each titration flask with the vinegar within on an anytical balance and record on your data table.
10) Add 5-6 drops of phenolphthalein indicator to each titration flask.
a. Note the initial color of all solutions before and after addition in the observation section of your laboratory notebook.
11) Using a 25.0 mL graduated cylinder, measure out 25.0 mL of deionized/distilled water and place into each titration flask.
a. It would be advisable to rinse the inside walls of each flask during this addition to ensure all vinegar is off of the side walls.
b. Boiled deionized water is preferred to remove dissolved carbon dioxide which can affect results.
c. Note: this water addition should not affect the acetic acid concentration.
12) Clamp the buret to a buret/ring stand using a buret clamp.
13) Place a 250 mL beaker below the buret to catch any spilt materials.
14) Obtain approximately 100.0 mL of a standard 0.1000 M NaOH solution into a dry 100 mL beaker.
a. Label beaker as "NaOH."
b. Note: a standardized 0.2000 M solution can be substituted for this titrant.
c. Record this standardized concentration in your data table.
15) Ensure the stopcock is in the closed position. Using a short stem funnel, add approximately 10 mL of NaOH to the buret.
16) Condition the buret by holding it horizontally and rotating to allow the NaOH to wet the inside of the glass completely. Discard the rinse into the sink.
17) Fill the buret with NaOH and adjust the meniscus to zero.
a. Record the initial buret reading as 0.00 mL .
18) Place titration flask \#1 under the tip of the burette and slowly begin to deliver the solution into the reaction mixture.
a. Swirl flask continually.
b. You may add it quickly at first but slow the addition of the titrant when the pink color lingers.
c. It may be advisable to place a piece of white paper underneath the flask for better viewing contrast.
19) Cease adding the sodium hydroxide titrant when the endpoint is reached.
a. The endpoint of the titration is when the addition of a single added drop of sodium hydroxide solution produces a faint but permanent pink color for at least 30 seconds.
b. The pink will eventually fade, however, due to the absorption of carbon dioxide (producing carbonic acid) from the air which will slowly react with the base.
20) Once the endpoint has been reached, record the final level of sodium hydroxide solution on the buret.
21) Refill the buret with sodium hydroxide solution to the 0.00 mL mark.
22) Repeat the titration procedure for titration flask \#2 and \#3.
23) Discard all of the contents of the titration flasks down the sink.
24) For each trial, calculate the moles of NaOH delivered by multiplying the standardized molarity by the amount of NaOH delivered in liters.
25) Next, calculate the moles of acetic acid neutralized by applying using proper stoichiometry.
26) Calculate the molarity of acetic acid in vinegar and the mean (average) value from the three trials.
27) Calculate the mass of acetic acid in vinegar titrated, mass percent of acetic acid in vinegar, and the mean value from the three trials.
28) Calculate the percent difference (between the manufacturer's label and the experimental value) and determine if this sample passes the quality control test of less than $5 \%$ difference.

## Waste Disposal and Lab Clean - Up:

1) Discard all of the contents of the titration flasks down the sink.
2) Place all excess vinegar and sodium hydroxide solutions in the same waste beaker. Neutralize, if needed and discard in the sink.
3) Clean all materials well with soap and water, remove wax/grease labels, and dry and return items to their original locations.

## The Lab - Write Up:

1) Determine all of the values in the data table and discuss how these values were ascertained in your conclusion.
2) Determine the experimental percent acetic acid in vinegar and compare to the manufacturer's value. Calculate the percent difference and determine if it passes the quality control test of less than $5 \%$ difference.
3) Discuss all reasons for error and how these errors affected the data.
4) How would using non-boiled distilled/deionized water (containing carbon dioxide) affect your results? How would this affect the experimental mass percent of acetic acid?

## Sample Data Table:

| Data Recorded/Calculated | Trial \#1 | Trial \#2 | Trial \#3 |
| :--- | :--- | :--- | :--- |
| a) Brand of vinegar analyzed: |  |  |  |
| b) Expiration date of vinegar analyzed: |  |  |  |
| c) Percent acidity (mass percent acetic acid) <br> on manufacturer's label: |  |  |  |
| d) Mass of empty titration flask in grams: |  |  |  |
| e) Volume of vinegar to be titrated in <br> milliliters: | 10.00 mL | 10.00 mL | 10.00 mL |
| f) Mass of titration flask with vinegar in <br> grams: |  |  |  |
| g) Mass of vinegar titrated in grams (f - d): |  |  |  |
|  |  |  |  |
| h) Standardized molar concentration of <br> NaOH used in moles per liter: |  |  |  |
| i) Initial buret reading before addition of <br> NaOH solution in milliliters: |  |  |  |
| j) Final buret reading after addition of <br> NaOH solution in milliliters: |  |  |  |
| k) Volume of NaOH delivered in milliliters <br> (j - i): |  |  |  |
| l) Moles of NaOH delivered in mol: |  |  |  |
| m) Moles of acetic acid neutralized in mol: |  |  |  |
| n) Molarity of acetic acid in vinegar in <br> moles per liter: |  |  |  |
| o) Mean (average) molarity of acetic acid in <br> vinegar in moles per liter: |  |  |  |
|  |  |  |  |
| p) Mass of acetic acid neutralized in grams <br> (m x Mm): |  |  |  |
| q) Mass percent of acetic acid in vinegar in <br> \% (p/g): |  |  |  |
| r) Mean (average) mass percent of acetic <br> acid in vinegar in \%: |  |  |  |
| s) Percent difference: |  |  |  |
| t) Does this sample pass the quality control <br> test of less than 5\% difference? (Yes/No) |  |  |  |

# Experiment \#7: <br> Determination of the Molar Mass of a Volatile Liquid by the Ideal Gas Equation 

## Introduction to Gases:

1) Gases are highly compressible fluids with no definite shape.
a. Their molecules are widely separated and they have high kinetic energies when compared to solids and liquids.
2) Gases are described by the four physical properties of pressure, temperature, volume, and the number of particles (or moles.)
3) Pressure of a gas is defined as the force exerted by a gas per surface area.
a. The International System of Units (SI) states that the standard metric unit for pressure is $\mathrm{kg} / \mathrm{m} \cdot \mathrm{s}^{2}$ called a pascal ( Pa ).
i. Important pressure conversions: $1 \mathrm{~atm}=760 . \mathrm{mm} \mathrm{Hg}=101,325 \mathrm{~Pa}$ $=1,013.25 \mathrm{hPa}=101.325 \mathrm{kPa}$.
b. Atmospheric pressure is commonly measured by a barometer.
i. An antiquated mercury barometer consists of a 1 meter long glass tube filled with mercury inverted into a dish of mercury.
1. At sea level, the height of mercury is 760 mm Hg (or Torr) and this value (or 1 atm ) is considered standard pressure ( $\mathrm{P}_{\mathrm{STP}}$ ).
4) Temperature is a measure of the average kinetic energy of gaseous particles.
a. The standard temperature ( $\mathrm{T}_{\text {STP }}$ ) of a gas is considered $0.00^{\circ} \mathrm{C}$ or 273.15 K .
5) Volume is an extensive property of matter that defines the space occupied in three-dimensional space.
a. At standard temperature and pressure (STP), the volume for 1 mole of an ideal gas is 22.4 L .
6) A mole is an extensive property of a substance that defines a quantity of elementary particles (atoms, molecules, ions, etc.) equal to 12 grams $(0.012 \mathrm{~kg})$ of carbon-12.
a. The number of particles in 1 mole of any material is described as Avogadro's number $\left(\mathrm{N}_{\mathrm{A}}\right)$ which is equal to $6.022 \times 10^{23}$ particles per mole.

## Introduction to the Empirical Gas Laws \& the Ideal Gas Equation:

1) Boyle's Law relates the volume and pressure of a gas to one another. This law states that, considering a constant number of gaseous moles at a constant temperature, the volume varies inversely with the applied pressure.
a. $\mathrm{V} \propto 1 / \mathrm{P}$ or $\mathrm{PV}=$ constant
2) Charles's Law relates the volume and absolute temperature of a gas to one another. This law states that, considering a constant number of gaseous moles at a constant pressure, the volume of a gas is directly proportional to its absolute temperature.
a. $\mathrm{V} \propto \mathrm{T}_{\mathrm{abs}}$ or $\mathrm{V} / \mathrm{T}=$ constant
3) Gay-Lussac's Law relates the pressure and absolute temperature of a gas to one another. This law states that, considering a constant number of gaseous moles at a constant volume, the exerted pressure by a gas is directly proportional to its absolute temperature.
a. $\mathrm{P} \alpha \mathrm{T}_{\mathrm{abs}}$ or $\mathrm{P} / \mathrm{T}=\mathrm{constant}$
4) Avogadro's Law states that equal volumes of any two gases at the same temperature and pressure contain the same number of gas particles.
a. The volume of one mole of a gas is referred to as molar volume, $\mathrm{V}_{\mathrm{m}}$.
b. As stated above, at STP, the volume for 1 mole of an ideal gas is 22.4 L .
5) Combining Boyle's Law, Charles's Law, and Avogadro's Law, the ideal gas equation was formulated.
6) The ideal gas law allows chemists to determine and derive important features of compounds by formula manipulation.
a. The formula: $P V=n R T$
i. $\mathrm{P}=$ pressure, $\mathrm{V}=$ volume, $\mathrm{n}=$ moles, $\mathrm{R}=$ ideal gas constant, $\mathrm{T}=$ temperature

$$
\text { 1. } \mathrm{R}=0.08206 \mathrm{~L} \bullet \mathrm{~atm} /(\mathrm{K} \bullet \mathrm{~mol})
$$

7) Volatile liquids are compounds that can be easily be vaporized.
a. If this vaporization takes place in a known volume and at a given temperature and pressure, the number of moles can be determined.
i. The rearranged formula: $n=P V / R T$
ii. The pressure in the container is equal to the barometric pressure as long as the container is open to the atmosphere.
b. The molar mass of the volatile liquid can then be determined by measuring the mass of the vapor.
i. Molar mass $\left(\mathrm{M}_{\mathrm{m}}\right)=($ mass of vapor $) /($ moles of vapor $)=\mathrm{g} / \mathrm{mol}$.

## Kinetic - Molecular Theory of an Ideal Gas:

1) To consider the ideal gas equation, a number of assumptions are proposed which help simplify gas particle behavior.
2) The kinetic theory of an ideal gas is based on five postulates:
a. Postulate \#1: The volume of gas particles is negligible.
b. Postulate \#2: Gas particles are in constant motion and move randomly and in straight lines in all directions and at various speeds.
c. Postulate \#3: The inherent attractive or repulsive force between gas molecules is negligible, except upon collision.
d. Postulate \#4: When gas particles collide, the collisions are elastic.
e. Postulate \#5: The average kinetic energy of a collection of particles is proportional to the absolute temperature.

## The Experimental Goal:

1) Each student will be assigned one known compound.
2) Following at least two trials of the experiment, the molar mass will be calculated and compared to the true value.

## The Known Volatile Liquids:

A) Acetone (2-propanone)
B) Cyclohexane
C) Ethanol (ethyl alcohol)
D) Ethyl acetate (ethyl ethanoate)
E) Isopropyl acetate (1-methylethyl ethanoate)
F) Isopropyl alcohol (2-propanol)
G) Methanol (methyl alcohol)
H) Pentane

## The Experimental Procedure:

1) Although the small amounts of gaseous materials produced in this lab are not harmful, it is recommended that this lab be performed under a fume hood, if available.
2) Obtain a clean and completely dry 125 mL Erlenmeyer flask.
3) Obtain a cap for the flask.
a. The cap is a size 4 stopper with a hole in the center. A small open glass tube (such as an eye dropper) has been placed through the stopper.
4) Record the mass of the clean and completely dried flask with the cap affixed an analytical balance.
5) Using a 10.0 mL graduated cylinder, measure out approximately 7.5 mL of your assigned volatile liquid.
a. Record the name of the volatile liquid assigned.
6) Add the measured volume of the volatile liquid to the inside of the flask.
7) Set up a water bath in a $600-1000 \mathrm{~mL}$ beaker using a hot plate as a heat source.
a. Place a few boiling stones into the water to promote even heating.
8) Immerse your flask into the water to cover the entire flask without allowing any water to enter the flask.
a. Secure the flask by using a utility clamp.
b. It is very important that water does not get into the flask; make sure the cap is securely fashioned.
9) Quickly bring the water to a boil and then lower the heat to allow for a gentle boil.
a. Record the temperature of the water once stabilized. (This temperature will also be the heat of your vapor.)
b. Record the pressure in the lab using a barometer.
10) Allow your sample to heat for 10 minutes after a gentle boil has been achieved. Ensure that all liquid material has been vaporized.
11) After the allotted time, remove flask from water bath and allow to cool to room temperature.
a. The vapor may condense back into a liquid; do not be alarmed.
12) Remove ALL TRACES of water from the outside of the flask with a paper towel.
a. Water can condense on the edges of the cap; ensure removal of this water.
13) Obtain the mass of the cool, dried flask on an analytical balance.
14) Remove the cap and discard the remaining volatile liquid into an appropriately labeled waste container.
15) Repeat experiment for a second trial.
a. For this trial, add approximately 5.0 mL of the volatile liquid to the inside of the flask.
b. If results differ significantly, perform a third trial.
16) After the last trial, remove the cap discard the remaining volatile liquid into an appropriately labeled waste container.
a. Do not get rid of flask at this point; you need to determine the volume of the container.
17) Rinse the Erlenmeyer well with water and discard down the drain. Reaffix the cap.
18) Using a wax/grease pencil, mark a line on the flask where the stopper ends and the empty space begins.
19) Remove the cap and fill the flask up to this wax/grease mark with deionized water.
20) CAREFULLY, transfer the volume into graduated cylinder(s) and record the total volume of the water that the flask could hold.
21) Repeat this volume determination for maximum precision.
22) Using the example data table attached, determine the molar mass of your volatile liquid and compare to true value.

## Waste Disposal and Lab Clean - Up:

1) All water baths can be poured down the sink and the boiling stones discarded into the trash.
2) Extra volumes of volatile liquids should be placed in the appropriately labeled waste containers.

## The Lab - Write Up:

1) Determine the experimental molar mass of your assigned volatile liquid and compare to the true value.
a. In conclusion, explain all relevant data collected to determine the experimental molar mass.
2) Calculate the percent error and discuss in the conclusion.
a. Percent error $=[$ (true value - experimental value)/true value $] * 100 \%$
b. Percent error cannot be a negative value. Take the absolute value of the number if necessary.
3) Discuss all reasons for error and how these errors affected the data.
4) What would have happened to the molar mass of your volatile liquid if water droplets were left onto the flask? Would your molar mass have been higher or lower? Thoroughly explain.
5) What assumptions have been made throughout this experiment that could have accounted for error?
a. Hint: Consider the Kinetic-Molecular Theory of Gases.

| Sample Data Table: |  |  |  |
| :--- | :--- | :--- | :--- |
| Data Recorded/Calculated | Trial \#1 | Trial \#2 | Trial \#3 |
| a) Volatile liquid assigned: |  |  |  |
| b) Mass of flask with cap in grams: |  |  |  |
| c) Volume of unknown liquid added (mL): |  |  |  |
| d) Stable temperature of water ( $\left.{ }^{\circ} \mathrm{C}\right):$ |  |  |  |
| e) Stable temperature of water (K): |  |  |  |
| f) Barometric pressure (hPa): |  |  |  |
| g) Barometric pressure (mm Hg): |  |  |  |
| h) Barometric pressure (atm): |  |  |  |
| i) Mass of flask with cap with sample after <br> heating in grams: |  |  |  |
| j) Mass of volatile liquid in grams (i - b): |  |  |  |
|  |  |  |  |
| k) Volume of water taken up by flask in <br> liters: |  |  |  |
|  |  |  |  |
| l) Moles of volatile liquid using $n=P V / R T$ <br> (mol): |  |  |  |
|  |  |  |  |
| m) Experimental molar mass of volatile <br> liquid in grams per mole (j/l): |  |  |  |
|  |  |  |  |
| n) Average experimental molar mass of <br> volatile liquid in grams per mole: |  |  |  |
|  |  |  |  |
| o) True molar mass of volatile liquid in <br> grams per mole: |  |  |  |
|  |  |  |  |
| p) Percent error (\%): |  |  |  |

Model Apparatus Set - Up:


## Experiment \#8A: <br> Thermochemistry Lab \#1; <br> Determination of the Molar Heat of Fusion for Water and Heat of Solution of a Solid

## Introduction to Thermochemistry \& Heat:

1) Thermochemistry is one area of chemistry dealing with the quantitative analysis of heat absorbed or evolved by physical and chemical processes.
2) Heat ( $q$ ) can be defined as the flow of energy into and out of a system due to a temperature difference between the thermodynamic system and its surroundings.
3) Heat energy will flow from the region of higher temperature to the region of lower temperature until thermal equilibrium has been reached.
a. At thermal equilibrium, the temperature of the two regions will be equal at any one time.
4) Heat and temperature are both measures of kinetic energy but there is an important difference.
a. Heat measures total energy.
b. Temperature measures the average energy.
c. An example to distinguish these two terms is as follows.
i. Considering a teaspoonful of boiling water versus a cup of boiling water (both at $100^{\circ} \mathrm{C}$ ), the cup of boiling water would be more hazardous to spill because it has more heat (total energy).
5) The formula for heat is as follows: $q=s \bullet m \bullet \Delta t$
a. $\quad q=$ heat, $s=$ specific heat, $m=$ mass, $\Delta t=$ change in temperature $\left(\mathrm{t}_{\mathrm{f}}-\mathrm{t}_{\mathrm{i}}\right)$.
i. Specific heat for water $=4.184 \mathrm{~J} /\left(\mathrm{g} \bullet{ }^{\circ} \mathrm{C}\right)$
6) In chemical reactions, heat is often transferred from the "system" to its "surroundings," or vice versa.
a. The substance or mixture of substances under study in which a change occurs is called the thermodynamic system (or just the system.)
b. The surroundings are everything around that thermodynamic system that can interact.
7) The sign of $q$ is very important!
a. If $q$ is positive, heat is absorbed by the system.
i. This process is described as endothermic.
b. If $q$ is negative, heat is released from the system.
i. This process is described as exothermic.

## Introduction to Calorimetry:

1) A calorimeter is a device used to measure the heat absorbed or evolved during a physical or chemical change.
2) There are two types of calorimeters:
a. A bomb calorimeter is an isolated metal container with a known mass and known heat capacity.
b. A coffee cup calorimeter is a plastic-foam cups that serves as a vessel for heat exchange.
i. These cups have a very low heat capacity and the small amount of heat gained/emitted is ignored.
ii. The foam is a good thermal insulator that allows very little heat to escape from the calorimeter.
3) The essential formula that governs calorimetry is as follows.
a. $q_{\text {system }}=-q_{\text {surroundings }}$

## Introduction to Heat of Fusion:

1) The heat needed to melt 1 mole of a pure substance is called the heat of fusion and denoted as $\Delta \mathrm{H}_{\text {fus }}$.
2) To melt a pure substance at its melting point requires an extra boost of energy to overcome lattice energies.
3) The heat of fusion $\left(\Delta \mathrm{H}_{\mathrm{fus}}\right)$ can be calculated.
a. At constant pressure, the heat exchanged is equal to the heat of fusion:
i. $q_{p}=\Delta \mathrm{H}_{\text {fus }}$.

## Introduction to Heat of Solution:

1) A solution is a homogeneous mixture of two or more substances, consisting of ions or molecules.
2) The solute is the dissolved substance for gases or solids in a liquid solution.
a. In other cases, it is the component that is in a smaller amount.
3) The solvent is the liquid substance in which the solids or gases are dissolved.
a. In other cases, it is the component that is in a larger amount.
4) When a solid dissolves in water, an energy change will always result.
a. Heat can be evolved or absorbed when an ionic compound dissolves in water.
5) The dissolving process occurs in two distinct steps:
a. The first step is the breaking down of the solid crystal lattice.
i. This process is endothermic (absorbing energy).
b. The second step is the hydration of the individual particles by the solvent.
i. This process is exothermic (releasing energy).
6) If the first step is dominant in the course of dissolving, then the process will be endothermic.
7) If the second step is dominant in the course of dissolving, then the process will be exothermic.
8) The heat of solution $\left(\Delta \mathrm{H}_{\text {sol }}\right)$ can be calculated.
a. At constant pressure, the heat exchanged is equal to the heat of solution:

$$
\text { i. } \quad q_{p}=\Delta \mathrm{H}_{\mathrm{sol}}
$$

## Procedure for the Molar Heat of Fusion of Water:

1) Place about $400-500 \mathrm{~mL}$ of deionized water into a 600 mL beaker and place onto a hotplate.
a. Heat up the water to between $60^{\circ} \mathrm{C}$ and $70^{\circ} \mathrm{C}$.
b. Note: If the water is hotter than needed, remove from the hotplate with beaker tongs or hot mitts and then add a quantity of room temperature deionized water.
2) While the water is heating up, obtain the mass of two clean and dried empty Styrofoam cups with a cap.
a. These two cups should be stacked on top of one another and the cap should have a hole in it to facilitate the use of your thermometer.
b. This set of Styrofoam cups will function as your calorimeter.
c. Record mass in data table.
3) Once the water temperature is appropriate, add enough warm water to the preweighed set of cups to fill between $65-75 \%$ full.
a. Make sure that you do not add too much warm water if an analytical balance is used because this large mass may overwhelm the balance.
4) Obtain the mass of the set of cups containing the warm water with the cap on top.
a. Ensure that no water is clinging to the outside walls of the Styrofoam cups. Dry with a paper towel if necessary.
5) Measure the temperature of the warm water to the nearest tenth to hundredth of a degree depending on the thermometer calibrations $\left(0.1^{\circ} \mathrm{C}-0.01^{\circ} \mathrm{C}\right)$.
6) Immediately following this measurement, add enough ice to cover the surface of the water.
a. Ensure that none of the warm water is allowed to splash out.
7) Replace the cap and gently stir the mixture with the thermometer or gently swirl the container.
a. Do not allow any of the water to splash out during this process.
8) Once the ice has melted and the temperature has stabilized, record the final temperature of the cool water to the nearest tenth to hundredth of a degree depending on the thermometer calibrations $\left(0.1^{\circ} \mathrm{C}-0.01^{\circ} \mathrm{C}\right)$.
9) Following this measurement, remove the thermometer and record the mass of the cool water in the set of cups with the cap.
10) Repeat this experiment (steps $1-9$ ) for a second trial.

## Procedure for Heat of Solution of a Solid:

1) Obtain the mass of two clean and dried empty Styrofoam cups with a cap.
a. These two cups should be stacked on top of one another and the cap should have a hole in it to facilitate the use of your thermometer.
b. Record mass in data table.
2) Add between $150-200 \mathrm{~mL}$ of room temperature deionized water to the Styrofoam cups.
3) Obtain the mass of the set of cups containing the water with the cap on top.
a. Ensure that no water is clinging to the outside walls of the Styrofoam cups. Dry with a paper towel if necessary.
4) Measure out approximately 10 - 15 grams of solid ammonium nitrate on a weigh boat using an analytical balance.
a. Record the mass in data table.
5) Measure the temperature of the water to the nearest tenth to hundredth of a degree depending on the thermometer calibrations $\left(0.1^{\circ} \mathrm{C}-0.01^{\circ} \mathrm{C}\right)$.
6) Immediately following this measurement, add the ammonium nitrate to the set of cups.
a. Ensure that you do not allow any of the water to splash out.
7) Replace the cap and gently stir the mixture with the thermometer or gently swirl the container.
a. Do not allow any of the water to splash out during this process.
8) Once the solid has completely dissolved and the temperature has stabilized, record the final temperature of the cool water to the nearest tenth to hundredth of a degree depending on the thermometer calibrations $\left(0.1^{\circ} \mathrm{C}-0.01^{\circ} \mathrm{C}\right)$.
9) Following this measurement, discard mixture down the drain with plenty of water.
10) Rinse out and completely dry the set of cups.
11) Repeat this experiment with $10-15$ grams of anhydrous calcium chloride.
a. Note: Calcium chloride is extremely hygroscopic which means that it readily absorbs moisture from the air.
b. Return cap of the calcium chloride immediately after using and proceed expediently after measuring the mass of the calcium chloride.

## Waste Disposal and Lab Clean - Up:

1) All water and salt solutions can be disposed of down the sink with plenty of water.
2) Do not discard the Styrofoam cups with caps; they are reused.

## The Lab - Write Up:

1) Determine the molar heat of fusion of water and the heats of solution for ammonium nitrate and calcium chloride. Discuss relevant data in conclusion that lead to this information.
2) Compare experimental results with the true values \& determine the percent error in each experiment using the following formula:
a. [(true value - experimental value)/true value] $* 100 \%$
b. Error cannot be negative; take the absolute value if necessary.
3) Discuss all reasons for error and how these errors affected the data.
4) In the heat of dissolution experiment, were the processes endothermic or exothermic? Explain by the use of the heat equation.
5) What assumptions have been made throughout this experiment that could have accounted for error?

Sample Data Table for the Molar Heat of Fusion of Water:

| Data Recorded/Calculated | Trial \#1 | Trial \#2 |
| :---: | :---: | :---: |
| a) Mass of Styrofoam cups with cap in grams: |  |  |
| b) Mass of Styrofoam cups with cap plus warm water in grams: |  |  |
| c) Mass of warm water in grams (b-a): |  |  |
| d) Stable temperature of warm water in ${ }^{\circ} \mathrm{C}$ : |  |  |
| e) Stable temperature of warm water in Kelvin: |  |  |
| f) Stable temperature of cool water in ${ }^{\circ} \mathrm{C}$ : |  |  |
| g) Stable temperature of cool water in Kelvin: |  |  |
| h) Temperature change of warm water in ${ }^{\circ} \mathrm{C}(\mathrm{f}-\mathrm{d})$ : |  |  |
| i) Mass of Styrofoam cups with cap plus cool water in grams: |  |  |
| j) Mass of cool water in grams (i-a): |  |  |
| k) Mass of ice in grams ( $\mathrm{j}-\mathrm{c}$ ): |  |  |
| 1) Amount of heat lost (in Joules) by the warm water ${ }^{\dagger}$ : |  |  |
| m) Amount of heat gained (in Joules) by the ice $\ddagger$ : |  |  |
| n) Amount of heat (in Joules) that melted ice ( $\|1\|-\|\mathrm{m}\|$ ): |  |  |
| o) Experimental heat of fusion (in Joules/gram) of water ( $\mathrm{n} / \mathrm{k}$ ): |  |  |
| p) Experimental molar heat of fusion (in Joules/mole) of water $\left(0 \cdot \mathrm{M}_{\mathrm{m}}\right)$ : |  |  |
| q) Experimental molar heat of fusion (in kilojoules/mole) of water ( $\mathrm{p} / 1000$ ): |  |  |
| r) Average experimental molar heat of fusion (in kilojoules/mole) of water $\left[\left(\mathrm{q}_{1}+\mathrm{q}_{2}\right) / 2\right]$ : |  |  |
| s) True molar heat of fusion (in kilojoules/mole) of water*: |  |  |
| t) Percent error for molar heat of fusion of water (\%): |  |  |
|  |  |  |
| * Reference for true molar heat of fusion for water: |  |  |

$$
{ }^{\dagger} \mathrm{q}=(\mathrm{c})\left(4.184 \mathrm{~J} / \mathrm{g} \bullet{ }^{\circ} \mathrm{C}\right)(\mathrm{h})
$$

$$
\ddagger \mathrm{q}=(\mathrm{k})\left(4.184 \mathrm{~J} / \mathrm{g} \bullet{ }^{\circ} \mathrm{C}\right)\left(\mathrm{f}-0.00^{\circ} \mathrm{C}\right)
$$

## Sample Data Table for Heat of Solution of $\mathbf{N H}_{4} \mathbf{N O}_{3}$ and $\mathrm{CaCl}_{2}$ :

| Data Recorded/Calculated | $\mathrm{NH}_{4} \mathrm{NO}_{3}$ | $\mathrm{CaCl}_{2}$ |
| :---: | :---: | :---: |
| a) Mass of Styrofoam cups with cap in grams: |  |  |
| b) Mass of Styrofoam cups with cap plus water in grams: |  |  |
| c) Mass of water in grams (b-a): |  |  |
|  |  |  |
| d) Mass of salt $\left(\mathrm{NH}_{4} \mathrm{NO}_{3}\right.$ or $\left.\mathrm{CaCl}_{2}\right)$ in grams: |  |  |
|  |  |  |
| e) Stable temperature of water in ${ }^{\circ} \mathrm{C}$ : |  |  |
| f) Stable temperature of in Kelvin: |  |  |
|  |  |  |
| g) Stable temperature of water after solid addition in ${ }^{\circ} \mathrm{C}$ : |  |  |
| h) Stable temperature of water after solid addition in Kelvin: |  |  |
|  |  |  |
| i) Temperature difference in ${ }^{\circ} \mathrm{C}(\mathrm{g}-\mathrm{e})$ : |  |  |
| j) Temperature difference in Kelvin (h-f): |  |  |
|  |  |  |
| k) Amount of heat lost or gained (in Joules) by the water ${ }^{\dagger}$ : |  |  |
| 1) Amount of heat lost or gained (in Joules) by the salt $\left(\mathrm{NH}_{4} \mathrm{NO}_{3}\right.$ or $\left.\mathrm{CaCl}_{2}\right)$ used ${ }^{\dagger \dagger}$ : |  |  |
|  |  |  |
| m) Number of moles of salt $\left(\mathrm{NH}_{4} \mathrm{NO}_{3}\right.$ or $\left.\mathrm{CaCl}_{2}\right)$ used $\left(\mathrm{d} / \mathrm{M}_{\mathrm{m}}\right)$ : |  |  |
| n) Experimental heat of solution (in J/mol) of salt $\left(\mathrm{NH}_{4} \mathrm{NO}_{3}\right.$ or $\mathrm{CaCl}_{2}$ ) used ( $1 / \mathrm{m}$ ): |  |  |
| o) Experimental heat of solution (in $\mathrm{kJ} / \mathrm{mol}$ ) of salt $\left(\mathrm{NH}_{4} \mathrm{NO}_{3}\right.$ or $\left.\mathrm{CaCl}_{2}\right)$ used $(\mathrm{n} / 1000)$ : |  |  |
| p) True heat of solution (in $\mathrm{kJ} / \mathrm{mol}$ ) of of salt $\left(\mathrm{NH}_{4} \mathrm{NO}_{3}\right.$ or $\mathrm{CaCl}_{2}$ ) used ${ }^{*}$ : |  |  |
| q) Percent error for heat of solution of of salt $\left(\mathrm{NH}_{4} \mathrm{NO}_{3}\right.$ or $\mathrm{CaCl}_{2}$ ) used (\%): |  |  |
| * Reference for true molar heat of fusion for $\mathrm{NH}_{4} \mathrm{NO}_{3}$ : |  |  |
| * Reference for true molar heat of fusion for $\mathrm{CaCl}_{2}$ : |  |  |

[^1]
# Experiment \#8B: <br> Thermochemistry Lab \#2; <br> Determination of the Specific Heat of an Unknown Metal and Heat of Reaction by Hess's Law 

## Introduction to Thermochemistry \& Heat:

1) $x$ 'Thermochemistry is one area of chemistry dealing with the quantitative analysis of heat absorbed or evolved by physical and chemical processes.
2) Heat $(q)$ can be defined as the flow of energy into and out of a system due to a temperature difference between the thermodynamic system and its surroundings.
3) Heat energy will flow from the region of higher temperature to the region of lower temperature until thermal equilibrium has been reached.
a. At thermal equilibrium, the temperature of the two regions will be equal at any one time.
4) Heat and temperature are both measures of kinetic energy but there is an important difference.
a. Heat measures total energy.
b. Temperature measures the average energy.
c. An example to distinguish these two terms is as follows.
i. Considering a teaspoonful of boiling water versus a cup of boiling water (both at $100^{\circ} \mathrm{C}$ ), the cup of boiling water would be more hazardous to spill because it has more heat (total energy).
5) The formula for heat is as follows: $q=s \bullet m \bullet \Delta t$
a. $\quad q=$ heat, $s=$ specific heat, $m=$ mass, $\Delta t=$ change in temperature $\left(\mathrm{t}_{\mathrm{f}}-\mathrm{t}_{\mathrm{i}}\right)$.
i. Specific heat for water $=4.184 \mathrm{~J} /\left(\mathrm{g} \bullet{ }^{\circ} \mathrm{C}\right)$
6) In chemical reactions, heat is often transferred from the "system" to its "surroundings," or vice versa.
a. The substance or mixture of substances under study in which a change occurs is called the thermodynamic system (or just the system.)
b. The surroundings are everything around that thermodynamic system that can interact.
7) The sign of $q$ is very important!
a. If $q$ is positive, heat is absorbed by the system.
i. This process is described as endothermic.
b. If $q$ is negative, heat is released from the system.
i. This process is described as exothermic.

## Introduction to Calorimetry:

1) A calorimeter is a device used to measure the heat absorbed or evolved during a physical or chemical change.
2) There are two types of calorimeters:
a. A bomb calorimeter is an isolated metal container with a known mass and known heat capacity.
b. A coffee cup calorimeter is a plastic-foam cups that serves as a vessel for heat exchange.
i. These cups have a very low heat capacity and the small amount of heat gained/emitted is ignored.
ii. The foam is a good thermal insulator that allows very little heat to escape from the calorimeter.
3) The essential formula that governs calorimetry is as follows.
a. $q_{\text {system }}=-q_{\text {surroundings }}$

## Introduction to Heat of Reaction:

1) The heat of reaction is the value of $q$ required to return a system to the given temperature at the completion of a reaction.
2) The enthalpy of reaction $\left(\Delta H_{r x n}\right)$ is an extensive property of a system that can be used to obtain the heat absorbed/evolved in a reaction at a given temperature and pressure.
a. If at constant pressure, the $q_{p}$ can be determined from the following formula:
i. $\Delta H_{r x n}=q_{p}$.

## Introduction to Hess's Law:

1) Hess's law of heat summation states that for a chemical equation that can be written as the sum of two or more steps, the enthalpy change for the overall equation is the sum of the enthalpy changes for the individual steps.
2) An example calculation for the decomposition of 2 moles of $\mathrm{CaCO}_{3}$.
a. Unknown equation: $2 \mathrm{CaCO}_{3}(s) \rightarrow 2 \mathrm{CaO}(s)+2 \mathrm{CO}_{2}(g) \quad \Delta H=$ ?
b. Known equations:
```
i. \(\mathrm{Ca}(s)+\mathrm{C}(s)+3 / 2 \mathrm{O}_{2}(g) \rightarrow \mathrm{CaCO}_{3}(s)\)
\(\Delta \mathrm{H}^{\mathrm{o}}{ }_{1}=-1208 \mathrm{~kJ}\)
ii. \(\mathrm{C}(\mathrm{s})+\mathrm{O}_{2}(g) \rightarrow \mathrm{CO}_{2}(g)\)
\(\Delta \mathrm{H}^{\mathrm{o}}{ }_{2}=-393.5 \mathrm{~kJ}\)
iii. \(\mathrm{Ca}(s)+1 / 2 \mathrm{O}_{2}(g) \rightarrow \mathrm{CaO}(s)\)
\(\Delta \mathrm{H}^{\mathrm{o}}{ }_{3}=-635.0 \mathrm{~kJ}\)
```

c. Step 1: Flip Equation \#1 (and the sign of $\Delta \mathrm{H}^{\mathrm{o}}{ }_{1}$ )
i. $\mathrm{CaCO}_{3}(s) \rightarrow \mathrm{Ca}(s)+\mathrm{C}(s)+3 / 2 \mathrm{O}_{2}(g)$
$\Delta \mathrm{H}^{\mathrm{o}}{ }_{1}=-(-1208 \mathrm{~kJ})$
d. Step 2: Cancel Like Terms and Add $\Delta \mathrm{H}^{\circ}$
i. $\mathrm{CaCO}_{3}(s) \rightarrow \mathrm{Ca}(s)+\mathrm{C}(s)+3 / 2 \mathrm{O}_{2}(g) \quad \Delta \mathrm{H}^{\mathrm{o}}{ }_{1}=+1208 \mathrm{~kJ}$
ii. $\mathrm{C}(s)+\mathrm{O}_{2}(g) \rightarrow \mathrm{CO}_{2}(g) \quad \Delta \mathrm{H}^{\mathrm{o}}{ }_{2}=-393.5 \mathrm{~kJ}$
iii. $\mathrm{Ca}(s)+1 / 2 \mathrm{O}_{2}(g) \rightarrow \mathrm{CaO}(s) \quad \Delta \mathrm{H}^{0}{ }_{3}=-635.0 \mathrm{~kJ}$
iv. $\mathrm{CaCO}_{3}(s) \rightarrow \mathrm{CaO}(s)+\mathrm{CO}_{2}(g) \quad \Delta \mathrm{H}_{\mathrm{rxn}}{ }^{\circ}=+179.5 \mathrm{~kJ}$
e. Step 3: Multiply by Entire Equation by $2 \& \Delta \mathrm{H}^{\circ}{ }_{\mathrm{rxn}}$
i. $2\left(\mathrm{CaCO}_{3}(s) \rightarrow \mathrm{CaO}(s)+\mathrm{CO}_{2}(g)\right)$
f. Final Answer:
i. $2 \mathrm{CaCO}_{3}(s) \rightarrow 2 \mathrm{CaO}(s)+2 \mathrm{CO}_{2}(g)$
$\Delta \mathrm{H}_{\mathrm{rxn}}^{\mathrm{o}}=2(+179.5 \mathrm{~kJ})$
$\Delta \mathbf{H}^{\mathbf{0}}{ }_{\mathrm{rxn}}=+359 \mathrm{~kJ}$

## Introduction to Specific Heat:

1) Specific heat is the quantity of heat required to raise the temperature of one gram of a substance, one degree Celsius (or Kelvin) at constant pressure.
a. Specific heats are characteristic for that substance at $25^{\circ} \mathrm{C}$ and constant pressure.
b. Example specific heats of pure materials are listed below:

| Element/Compound | Specific Heat (J/(g•$\left.\left.{ }^{\mathbf{0}} \mathbf{C}\right)\right)$ |
| :---: | :---: |
| Aluminum | 0.901 |
| Iron | 0.450 |
| Lead | 0.128 |
| Copper | 0.387 |
| Water $(l)$ | 4.184 |

## Procedure for the Specific Heat of an Unknown Metal:

1) Place approximately 400 mL of tap water into a 600 mL beaker and place onto a hotplate.
a. Heat the water to the boiling point.
2) While waiting for the water to boil, obtain an unknown metal cylinder and determine the mass on an ANALYTICAL balance.
a. Record this mass in the data table attached.
b. Record the unknown number/letter in data table.
c. The unknown metals are aluminum, iron, lead, and copper.
3) Carefully, place the metal cylinder into a dry test tube and clamp the tube into the boiling such that the metal cylinder is below surface.
a. The metal should NOT be in direct contact with the water.
4) Obtain the mass of two clean and dried empty Styrofoam cups with a cap on an ANALYTICAL balance.
a. These two cups should be stacked on top of one another and the cap should have a hole in it to facilitate the use of your thermometer.
b. This set of Styrofoam cups will function as your calorimeter.
c. Record mass in data table.
5) Using a graduated cylinder, add 50.0 mL of deionized water to the calorimeter and obtain the mass on an ANALYTICAL balance.
a. Ensure that no water is clinging to the sides of the Styrofoam cups.
b. Record the mass in the data table.
6) Measure the stable temperature of the boiling water to the nearest tenth to hundredth of a degree depending on the thermometer calibrations $\left(0.1^{\circ} \mathrm{C}-\right.$ $0.01^{\circ} \mathrm{C}$ ).
a. Record measurement in data table.
b. This temperature is not just that of your water, but of your metal cylinder as well.
7) Measure the stable temperature of the deionized water in the calorimeter to the nearest tenth to hundredth of a degree depending on the thermometer calibrations $\left(0.1^{\circ} \mathrm{C}-0.01^{\circ} \mathrm{C}\right)$.
a. Record measurement in data table.
8) Remove the clamped test tube and quickly transfer ONLY the heated metal cylinder into the calorimeter.
a. Ensure that you do not transfer any boiling water into the calorimeter.
b. Ensure that you do not allow any of the deionized water from inside the calorimeter to splash out.
9) Replace the cap and gently stir the mixture with the thermometer or swirl the calorimeter.
10) Record the final temperature in the calorimeter after stabilization to the nearest tenth to hundredth of a degree depending on the thermometer calibrations $\left(0.1^{\circ} \mathrm{C}\right.$ $\left.-0.01^{\circ} \mathrm{C}\right)$.
11) Following this measurement, remove the metal and thoroughly dry.
12) Repeat this experiment for a second trial.
a. Use $60.0-75.0 \mathrm{~mL}$ of deionized water in the calorimeter for this trial.

## Procedure for Heat of Reaction by Hess's Law:

1) Obtain the mass of two clean and dried empty Styrofoam cups with a cap on an ANALYTICAL balance.
a. These two cups should be stacked on top of one another and the cap should have a hole in it to facilitate the use of your thermometer.
b. This set of Styrofoam cups will function as your calorimeter.
c. Record mass in data table.
2) Measure out 75.0 mL of 6.0 M HCl and carefully transfer into pre-weighed calorimeter.
b. Be extremely careful when handling the hydrochloric acid solution; it is very caustic.
c. Wear goggles, gloves, and appropriate clothing at all times.
3) Obtain the mass of the set of cups with the cap on top containing the hydrochloric acid solution.
a. Be extremely careful while transporting this solution and do not spill onto the balance.
4) Measure the temperature of the acid solution to the nearest tenth to hundredth of a degree depending on the thermometer calibrations $\left(0.1^{\circ} \mathrm{C}-0.01^{\circ} \mathrm{C}\right)$.
5) Obtain approximately 0.65 g of powdered, elemental zinc ( Zn ) using an ANALYTICAL balance.
a. If available, the use of weigh paper is preferred over the use of weigh boats.
b. Record mass in data table.
6) Add the metal to the calorimeter, replace the cap, and gently stir the mixture with the thermometer.
7) Record the final temperature in the calorimeter after stabilization to the nearest tenth to hundredth of a degree depending on the thermometer calibrations $\left(0.1^{\circ} \mathrm{C}\right.$ $-0.01^{\circ} \mathrm{C}$ ).
8) Following this measurement, place the solution in the appropriate waste container and completely clean and dry the set of cups.
9) Repeat this experiment with 75.0 mL of 6.0 M HCl and 1.20 g of zinc oxide.

## Waste Disposal and Lab Clean-Up:

1) All water baths can be disposed of down the sink.
2) All hydrochloric acid solutions should be placed in the appropriate waste container for neutralization.
3) Do not discard the Styrofoam cups with caps; they are reused.

## The Lab - Write Up:

1) Determine the specific heat of the unknown metal and the identity of the metal after comparison with known metal values.
a. Discuss relevant data in your conclusion and compare experimental specific heat to all true values.
b. Determine the percent error in each experiment using the following formula and discuss in conclusion:
i. [(true value - experimental value)/true value] * $100 \%$
ii. Percent error cannot be a negative value. Take the absolute value of the number if necessary.
2) Use Hess's Law to determine the heat of reaction $(\Delta H)$ for $\mathrm{Zn}(s)+1 / 2 \mathrm{O}_{2}(g) \rightarrow$ $\mathrm{ZnO}(s)$.
a. Look up the true value for heat of formation for $\mathrm{ZnO}(s)$, properly reference, and compare. Discuss in conclusion.
b. Determine the percent error in each experiment using the following formula and discuss in conclusion:
i. [(true value - experimental value)/true value] $* 100 \%$
ii. Percent error cannot be a negative value. Take the absolute value of the number if necessary.
3) Discuss all reasons for error and how these errors affected the data.
4) What assumptions have been made throughout this experiment that could have accounted for error?

Sample Data Table for the Specific Heat of an Unknown Metal:

| Data Recorded/Calculated | Trial \#1 | Trial \#2 |
| :---: | :---: | :---: |
| a) Unknown metal cylinder number/letter: |  |  |
| b) Mass of unknown metal cylinder in grams: |  |  |
|  |  |  |
| c) Mass of Styrofoam cups with cap in grams: |  |  |
| d) Mass of Styrofoam cups with cap plus deionized water in grams: |  |  |
| e) Mass of deionized water in grams (d-c): |  |  |
|  |  |  |
| f) Stable temperature of boiling water/metal cylinder ( ${ }^{\circ} \mathrm{C}$ ): |  |  |
| g) Stable temperature of boiling water/metal cylinder (K): |  |  |
|  |  |  |
| h) Stable temperature of water before metal addition ( ${ }^{\circ} \mathrm{C}$ ): |  |  |
| i) Stable temperature of water before metal addition (K): |  |  |
|  |  |  |
| j) Stable temperature of water after metal addition ( ${ }^{\circ} \mathrm{C}$ ): |  |  |
| k) Stable temperature of water after metal addition (K): |  |  |
|  |  |  |
| 1) Amount of heat gained (in Joules) by the deionized water: |  |  |
| m) Amount of heat lost (in Joules) by the metal cylinder: |  |  |
| n) Specific heat (s) of unknown metal (J/(g• $\left.\left.{ }^{\circ} \mathrm{C}\right)\right)^{\dagger}$ : |  |  |
| o) Average specific heat of unknown metal in $\mathrm{J} /\left(\mathrm{g} \cdot{ }^{\circ} \mathrm{C}\right)$ $\left[\left(\boldsymbol{s}_{1}+\boldsymbol{s}_{2}\right) / 2\right]$ : |  |  |
| p) Identity of unknown metal: |  |  |
| q) Percent error for specific heat (\%): |  |  |

$$
\begin{gathered}
\dagger s=q /(m \bullet \Delta t) \\
\text { (Derived from } q=s \bullet m \bullet \Delta t)
\end{gathered}
$$

## Sample Data Table Heat of Reaction by Hess's Law:

## Part 1: Elemental Zinc + HCl

a) Balanced equation:
b) Mass of Styrofoam cups with cap in grams:
c) Volume of hydrochloric acid solution in milliliters:
d) Mass of Styrofoam cups with cap plus HCl solution in grams: $\qquad$
e) Mass of HCl solution in grams $(\mathrm{d}-\mathrm{b})$ : $\qquad$
f) Density of HCl solution in $\mathrm{g} / \mathrm{mL}(\mathrm{e} / \mathrm{c})$ : $\qquad$
g) Heat capacity for HCl solution: $3.86 \mathrm{~J} /\left(\mathrm{g} \bullet{ }^{\circ} \mathrm{C}\right)$
h) Temperature of HCl solution before metal addition $\left({ }^{\circ} \mathrm{C}\right)$ : $\qquad$
i) Temperature of HCl solution before metal addition (K): $\qquad$
j) Mass of zinc $(\mathrm{Zn})$ metal in grams: $\qquad$
k) Moles of zinc ( Zn ) metal $\left(\mathrm{j} / \mathrm{M}_{\mathrm{m}}\right.$ ): $\qquad$

1) Temperature of HCl solution after metal addition $\left({ }^{\circ} \mathrm{C}\right)$ : $\qquad$
$\mathrm{m})$ Temperature of HCl solution after metal addition (K): $\qquad$
n) Temperature change of HCl solution in ${ }^{\circ} \mathrm{C}(1-\mathrm{h})$ :
o) Heat $(q)$ of the HCl solution in Joules $(\mathrm{g} \bullet(\mathrm{e}+\mathrm{j}) \bullet \mathrm{n})$ : $\qquad$
p) Heat $(q)$ of the reaction in Joules $(-o)$ :
q) Molar heat of reaction for Zn reaction in $\mathrm{J} /$ mole $(\mathrm{p} / \mathrm{k})$ : $\qquad$
r) Molar heat of reaction for Zn reaction in $\mathrm{kJ} / \mathrm{mole}(\mathrm{q} / 1000)$ :

## Part 2: Zinc Oxide + HCl

a) Balanced equation:
b) Mass of Styrofoam cups with cap in grams:
c) Volume of hydrochloric acid solution in milliliters:
d) Mass of Styrofoam cups with cap plus HCl solution in grams: $\qquad$
e) Mass of HCl solution in grams $(\mathrm{d}-\mathrm{b})$ : $\qquad$
f) Density of HCl solution in $\mathrm{g} / \mathrm{mL}(\mathrm{e} / \mathrm{c})$ : $\qquad$
g) Specific heat for HCl solution: $3.86 \mathrm{~J} /\left(\mathrm{g} \bullet{ }^{\circ} \mathrm{C}\right)$
h) Temperature of HCl solution before oxide addition $\left({ }^{\circ} \mathrm{C}\right)$ : $\qquad$
i) Temperature of HCl solution before oxide addition (K): $\qquad$
j) Mass of zinc oxide in grams:
k) Moles of zinc oxide $\left(\mathrm{j} / \mathrm{M}_{\mathrm{m}}\right)$ :
$\qquad$

1) Temperature of HCl solution after oxide addition $\left({ }^{\circ} \mathrm{C}\right)$ : $\qquad$
$\mathrm{m})$ Temperature of HCl solution after oxide addition (K): $\qquad$
n) Temperature change of HCl solution in ${ }^{\circ} \mathrm{C}(1-\mathrm{h})$ : $\qquad$
o) Heat $(q)$ of the HCl solution in Joules $(\mathrm{g} \bullet(\mathrm{e}+\mathrm{j}) \bullet \mathrm{n})$ : $\qquad$
p) Heat $(q)$ of the reaction in Joules ( -o ): $\qquad$
$\qquad$
q) Molar heat of reaction for zinc oxide reaction in $\mathrm{J} / \mathrm{mol}(\mathrm{p} / \mathrm{k})$ :
r) Molar heat of reaction for zinc oxide reaction in $\mathrm{kJ} / \mathrm{mol}(\mathrm{q} / 1000)$ : $\qquad$

## Part 3: Hess's Law for the Heat of Formation of ZnO:

$$
\text { Unknown Reaction: } \mathbf{Z n}(s)+1 / 2 \mathbf{O}_{\mathbf{2}}(\mathrm{g}) \rightarrow \mathbf{Z n O}(\boldsymbol{s}) \quad \Delta H=?
$$

a) Balanced equation for elemental zinc: $\qquad$
b) Molar heat of reaction for Zn reaction: $\qquad$
c) Balanced equation for zinc oxide:
d) Molar heat of reaction for Zn oxide reaction:
e) Balanced equation for synthesis of water: ${\underline{\mathrm{H}_{2}}(g)+1 / 2 \mathrm{O}_{2}(g) \rightarrow \mathrm{H}_{2} \mathrm{O}(l)}_{(l)}$
f) Molar heat of reaction for synthesis of water: $-285.8 \mathrm{~kJ} / \mathrm{mol}$
g) Experimental value for heat of formation of $\mathrm{ZnO}(s)$ : $\qquad$
h) True value for heat of formation of $\mathrm{ZnO}(s)^{*}$ :
i) Percent error for heat of formation of $\mathrm{ZnO}(s)$ : $\qquad$
Reference for true heat of formation of $\mathrm{ZnO}(s)$ :
*You must look up this value online or in a text $\&$ properly reference.

## Experiment \#9: The Electromagnetic Spectrum \& Atomic Line Spectra

## Introduction to the Electromagnetic Spectrum:

1) The entire range of frequencies or wavelengths of electromagnetic radiation is called the electromagnetic spectrum.
a. Electromagnetic radiation has properties of oscillating waves with an electric and magnetic vector that are perpendicular to one another.
2) The waves of the electromagnetic spectrum are characterized by their frequencies and wavelengths.
a. The wavelength, designated by $\lambda$, is defined as the distance between any two adjacent identical points of a wave.
i. The SI unit for wavelength is the meter.
ii. Since the visible spectrum is often analyzed, values for these measurements are provided in nanometers ( nm ).
1. $1 \mathrm{~nm}=1 \times 10^{-9} \mathrm{~m}$
b. The frequency, designated by $v$, is defined as the number of wavelengths of that wave that pass through a fixed point in one unit of time (commonly the second).
i. The SI unit of frequency is $\mathrm{s}^{-1}$ (also called the hertz or Hz ).
c. The frequency and wavelength of a wave are inversely proportional to one another: $c=\nu \lambda$.
i. $\quad c=$ the speed of light $=2.998 \times 10^{8} \mathrm{~m} / \mathrm{s}$
3) Light also has properties of particles.
a. These particles of energy are called quanta or photons.
b. The energy ( E ) of these particles is characterized by the following equation: $\mathrm{E}=\mathrm{h} v=\mathrm{h} c / \lambda$
i. $h=$ Plank's constant $=6.626 \times 10^{-34} \mathrm{~J} \bullet s$.
ii. Notice that frequency ( $v$ ) is directly proportional to the energy while wavelength $(\lambda)$ is inversely proportional to the energy.
1. Gamma rays contain the highest amount of energy with wavelengths $(\lambda)$ of $\sim 1 \times 10^{-12}$ m and frequencies $(v)$ of $\sim 1 \times$ $10^{20} \mathrm{~s}^{-1}$.
2. Radio waves contain the lowest amount of energy with wavelengths $(\lambda)$ of $\sim 1 \times 10^{-1} \mathrm{~m}$ and frequencies $(v)$ of $>1.0$ x $10^{10} \mathrm{~s}^{-1}$.
4) A heated solid (such as tungsten) emits polychromatic light that can be separated into a continuous spectrum when played through a prism.
a. The continuous spectrum contains all wavelengths of the visible spectrum.
b. The visible spectrum ranges from 390 nm to 800 nm .
i. The 400 nm region is the violet end of the spectrum.
ii. The 700 nm region is the red end of the spectrum.
iii. The seven colors of visible light are: red, orange, yellow, green, blue, indigo, and violet.
c. The following picture depicts the entire visible spectrum.
i. Reference for picture: "General Chemistry", Ebbing \& Gammon, $9^{\text {th }}$ Edition, 2009/2011.

5) A heated gas (such as hydrogen) does not emit polychromatic light, however.
a. A line spectrum is produced which only contains certain colors or specific wavelengths of visible light.
6) A grating spectrometer is a useful tool designed to analyze the certain wavelengths of light emitted from a gas discharge tube.
a. Gas discharge tubes are air evacuated tubes with small amounts of elemental gases within which have electrodes on both ends.
i. Once the high voltage power supply is activated, a beam of high energy electrons is passed through the gas.
ii. As electrons collide with the gas particles, electrons may be excited to higher energy levels.
iii. Once these electrons fall back to their initial energy levels (their ground states), photons of specific energies are emitted.
b. Light is passed through the grating slit and travels through a prism where the light is split into the characteristic wavelengths of the visible spectrum.

## The Elements:

1) Argon
2) Bromine
3) Chlorine
4) Helium
5) Hydrogen
6) Iodine
7) Krypton
8) Mercury
9) Neon
10) Nitrogen
11) Oxygen
12) Sodium
13) Xenon

## Safety Precautions:



## Diagram of a Grating Spectrometer

1) Please do not touch the gas discharge tubes until they have cooled.
2) Please do not remove or insert a discharge tube if the power supply is on. The power supplies are a source of high voltage electricity!

## Experimental Procedure:

1) Place a hydrogen gas discharge tube into the high voltage power supply and turn on.
a. Note: Your instructor may have this already prepared ahead of time. Do not disturb setup.
2) Note the color of the glowing gas in a data table like the one provided.
3) Place a grating spectrometer in front of the discharge tube and allow the light in through the slit.
a. The slit width can be adjusted to allow more or less light to pass into the prism.
b. It may be advisable to turn off all overhead lights (and other sources of ambient light) to allow for better viewing and decrease interference.
4) Determine the four main colors associated with the hydrogen emission line spectrum.
a. Press the push button switch on the illuminator accessory, if necessary, to view the wavelength scale. Record wavelengths.
i. The scale extends from 400 to 750 nm and measurements can be made directly to within 5 nm .
b. Depending on the sensitivity of the equipment, there may only be three atomic lines visible.
5) Calculate the frequencies and energies of these four colors and record in the data table.
6) Repeat the above mentioned procedure with helium, mercury, a fourth element (to be selected by instructor and/or class) and an unknown element.
a. Ensure that you record the unknown letter/number.
b. Determine the identity of the unknown element by the emission spectrum.
i. Compare atomic line and wavelength data from the other elements provided.
ii. A helpful website that provides the atomic line spectra (emission) for all elements is as follows: http://jersey.uoregon.edu/vlab/elements/Elements.html

## The Lab Write - Up:

1) Determine the colors, wavelengths, frequencies, and energies of the emission lines for hydrogen, helium, mercury, a fourth element, and an unknown. Discuss important data collected in your conclusion.
2) Determine the identity of the unknown by its characteristic emission lines. Explain how this was determined in your conclusion.
3) Why would it have been expected for iodine to have emission lines that included all colors of the visible spectrum? Explain.
4) Why would it be feasible to use tungsten as a filament for the light bulb? Discuss multiple reasons.
5) Discuss reasons for error and how these errors can affect results.

## Waste Disposal and Lab Clean - Up:

1) Place materials back into their storage locations as indicated by your instructor.

Sample Data Table for Atomic Line Spectrum for Hydrogen: Color of hydrogen discharge tube:

| Color | Wavelength (nm) | Frequency (s ${ }^{\mathbf{- 1}}$ ) | Energy (J) |
| :---: | :--- | :--- | :--- |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |

Sample Data Table for Atomic Line Spectrum for Helium:
Color of discharge tube:

| Color | Wavelength (nm) | Frequency (s ${ }^{\mathbf{- 1}}$ ) | Energy (J) |
| :---: | :--- | :--- | :--- |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |

Sample Data Table for Atomic Line Spectrum for Mercury: Color of discharge tube:

| Color | Wavelength (nm) | Frequency (s ${ }^{\mathbf{1}}$ ) | Energy (J) |
| :---: | :--- | :--- | :--- |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
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|  |  |  |  |

Sample Data Table for Atomic Line Spectrum for $\qquad$ : Color of discharge tube:

| Color | Wavelength (nm) | Frequency (s ${ }^{\mathbf{1}}$ ) | Energy (J) |
| :---: | :--- | :--- | :--- |
|  |  |  |  |
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Sample Data Table for Atomic Line Spectrum for Unknown $\qquad$
Color of discharge tube:

| Color | Wavelength (nm) | Frequency (s ${ }^{\mathbf{1}}$ ) | Energy (J) |
| :---: | :--- | :--- | :--- |
|  |  |  |  |
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Identity of unknown element: $\qquad$

## Experiment \#10: Electron Configuration and Periodic Trends Pre-Lab

Note to Instructor: These materials are to be provided to students in advance of the laboratory exercise and must be completed prior to performing the experiment. On the day of the laboratory exercise, the document entitled "Electron Configurations and Periodic Trends Lab" is to be provided.

Note to Student: Using the guide and tables below, complete the pre-laboratory information in your laboratory notebook as indicated. You will be provided with the experimental procedure during the lab period, if you have completed this assignment in advance.

Student Name: Your Name
Experimental Title: Electron Configuration and Periodic Trends Lab
Date started: Date of Lab
Unknown \#: N/A
Reference to Source of Procedure: Barnett, "Electron Configurations and Periodic Trends," pg 1-4, 2012.

Balanced Equations for Reactions: *Leave enough space for the following areas*

## Part 9:

Explosive reactions:

1) $\qquad$
2) 
3) 
4) $\qquad$
5) $\qquad$

## Part 10:

Reactions of alkali metals:
1)
2) $\qquad$
Reactions of alkaline Earth metals:
1)
2)

Reactions of Group 13/IIIA elements:
1)
2)
)

## Reactions of Group 14/IVA elements:

1) 
2) 

## Reactions of the pnictogens:

1) 
2) 

Reactions of the chalcogens:
1)
2)

## Reactions of the halogens:

1) 
2) $\qquad$
Reactions of the noble gases:
3) 
4) 

## Side Reactions and Complete Reaction Mechanism: N/A

Purpose: The purpose of this lab is to use the periodic chart to derive the following information about the elements: 1) electron configurations, 2) orbital diagrams, 3) magnetic properties, 4) atomic radii, 5) ionization energies, 6) electron affinities, and 7) types of oxide formation. This analysis will be performed on the following elements: Al, $\mathrm{Ba}, \mathrm{Be}, \mathrm{C}, \mathrm{Ca}, \mathrm{Cr}, \mathrm{Cs}, \mathrm{Cu}, \mathrm{F}, \mathrm{Ge}, \mathrm{He}, \mathrm{Hg}, \mathrm{I}, \mathrm{K}, \mathrm{Kr}, \mathrm{Li}, \mathrm{Na}, \mathrm{P}, \mathrm{Rb}, \mathrm{Si}$, and Sb .

Table \#1: The Periodic Table: Cut out and paste/staple onto a data page of your lab notebook.

Table \#2: Table of Important Information for the Elements: Complete attached table and provide references. Place onto a data page of your lab notebook.

Table \#3: Procedures and Observations and Waste Disposal Information: Cut out and paste/staple onto a data page of your lab notebook. The procedures and waste disposal information will be provided during the lab.

Table \#4: Table of Physical and Chemical Properties for Reactants and Products: Complete attached table and provide references. Place onto a data page of your lab notebook.

Table \#5: Data Table of Oxides in Water: Cut out and paste/staple onto a data page of your lab notebook. The procedures will be provided during the lab.

> After the data tables have been affixed to your lab notebook, leave additional blank data pages for further data collection.

## Table \#1: The Periodic Table



Table \#2: Table of Important Information for the Elements:

| Elemental Symbol (Name) | Atomic Number | Group or Family Name | Metal, NonMetal, or Semimetal (Metalloid)? | Atomic Radius (pm) | First Ionization Energy (kJ/mol) | Electron Affinity (kJ/mol) |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Al (aluminum) |  |  |  |  |  |  |
| $\mathrm{Ba}(\square)$ |  |  |  |  |  |  |
| $\mathrm{Be}(\square)$ |  |  |  |  |  |  |
| C ( |  |  |  |  |  |  |
| Ca ( |  |  |  |  |  |  |
| $\mathrm{Cr}(\square)$ |  |  |  |  |  |  |
| Cs ( |  |  |  |  |  |  |
| $\mathrm{Cu}(\square)$ |  |  |  |  |  |  |
| F ( |  |  |  |  |  |  |
| Ge ( |  |  |  |  |  |  |
| He ( |  |  |  |  |  |  |
| Hg ( |  |  |  |  |  |  |
| I ( |  |  |  |  |  |  |
| K ( |  |  |  |  |  |  |
| $\mathrm{Kr}(\square)$ |  |  |  |  |  |  |
| $\mathrm{Li}(\underline{\square}$ ) |  |  |  |  |  |  |
| Na ( |  |  |  |  |  |  |
| P ( |  |  |  |  |  |  |
| Rb ( |  |  |  |  |  |  |
| $\mathrm{Si}(\square)$ |  |  |  |  |  |  |
| $\mathrm{Sb}(\square)$ |  |  |  |  |  |  |

References: $\qquad$
$\qquad$
$\qquad$

Table \#3: Procedure and Observations:

| Procedure (In your own words) | Observations |
| :--- | :--- |
|  |  |
|  |  |
|  |  |
|  |  |
|  |  |

Waste Disposal and Clean Up:

| Waste Disposal/Clean Up | Action |
| :--- | :--- |
|  |  |
|  |  |

Table \#4: Table of Physical and Chemical Properties for Reactants and

## Products:

| Chemical Name | Atomic, Ionic, Molecular Formula and/or Structure | Atomic, Ionic, Molecular Mass or Weight: (in g/mol) | Melting Point (MP): (in ${ }^{\circ} \mathrm{C}$ ) | Boiling Point $\begin{aligned} & (\mathbf{B P}): \\ & \left(\text { in }^{\circ} \mathrm{C}\right) \end{aligned}$ | Density: (g/mL) | Other <br> Info: (such as $\mathbf{p K}_{\mathrm{a}}$, solubility, etc.) |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| aluminum oxide |  |  |  |  |  |  |  |
| calcium oxide |  |  |  |  |  |  |  |
| carbon dioxide |  |  |  |  |  |  |  |
| diiodine pentoxide |  |  |  |  |  |  |  |
| diphosphorus pentoxide |  |  |  |  |  |  |  |
| germanium dioxide |  |  |  |  |  |  |  |
| silicon dioxide |  |  |  |  |  |  |  |
| sodium oxide |  |  |  |  |  |  |  |
| water |  |  |  |  |  |  |  |

References: $\qquad$

Table \#5: Data Table of Oxides in Water:

| Name of Oxide | Oxide <br> Formula | pH of Water <br> Before Oxide <br> Addition | pH of Water <br> After Oxide <br> Addition | Acidic, <br> Basic, or <br> Amphoteric |
| :---: | :---: | :---: | :---: | :---: |
| aluminum oxide |  |  |  |  |
| calcium oxide |  |  |  |  |
| carbon dioxide |  |  |  |  |
| diiodine pentoxide |  |  |  |  |
| diphosphorus |  |  |  |  |
| pentoxide |  |  |  |  |
| germanium dioxide |  |  |  |  |
| silicon dioxide |  |  |  |  |
| sodium oxide |  |  |  |  |

## Experiment \#11A: Thermochemistry versus Bond Energy; The Enthalpy of Catalytic Decomposition of Hydrogen Peroxide

## Introduction to Thermochemistry \& Heat:

1) Thermochemistry is one area of chemistry dealing with the quantitative analysis of heat absorbed or evolved by physical and chemical processes.
2) Heat ( $q$ ) can be defined as the flow of energy into and out of a system due to a temperature difference between the thermodynamic system and its surroundings.
3) Heat energy will flow from the region of higher temperature to the region of lower temperature until thermal equilibrium has been reached.
a. At thermal equilibrium, the temperature of the two regions will be equal at any one time.
4) Heat and temperature are both measures of kinetic energy but there is an important difference.
a. Heat measures total energy.
b. Temperature measures the average energy.
c. An example to distinguish these two terms is as follows.
i. Considering a teaspoonful of boiling water versus a cup of boiling water (both at $100^{\circ} \mathrm{C}$ ), the cup of boiling water would be more hazardous to spill because it has more heat (total energy).
5) The formula for heat is as follows: $q=s \bullet m \bullet \Delta t$
a. $\quad q=$ heat, $s=$ specific heat, $m=$ mass, $\Delta t=$ change in temperature $\left(\mathrm{t}_{\mathrm{f}}-\mathrm{t}_{\mathrm{i}}\right)$.
i. Specific heat for water $=4.184 \mathrm{~J} /\left(\mathrm{g} \bullet{ }^{\circ} \mathrm{C}\right)$
6) In chemical reactions, heat is often transferred from the "system" to its "surroundings," or vice versa.
a. The substance or mixture of substances under study in which a change occurs is called the thermodynamic system (or just the system.)
b. The surroundings are everything around that thermodynamic system that can interact.
7) The sign of $q$ is very important!
a. If $q$ is positive, heat is absorbed by the system.
i. This process is described as endothermic.
b. If $q$ is negative, heat is released from the system.
i. This process is described as exothermic.

## Introduction to Calorimetry:

1) A calorimeter is a device used to measure the heat absorbed or evolved during a physical or chemical change.
2) There are two types of calorimeters:
a. A bomb calorimeter is an isolated metal container with a known mass and known heat capacity.
b. A coffee cup calorimeter is a plastic-foam cups that serves as a vessel for heat exchange.
i. These cups have a very low heat capacity and the small amount of heat gained/emitted is ignored.
ii. The foam is a good thermal insulator that allows very little heat to escape from the calorimeter.
3) The essential formula that governs calorimetry is as follows.
a. $q_{\text {system }}=-q_{\text {surroundings }}$

## Introduction to Heat of Reaction:

1) The heat of reaction is the value of $q$ required to return a system to the given temperature at the completion of a reaction.
2) The enthalpy of reaction $\left(\Delta H_{r x n}\right)$ is an extensive property of a system that can be used to obtain the heat absorbed/evolved in a reaction at a given temperature and pressure.
a. If at constant pressure, the $q_{p}$ can be determined from the following formula:
i. $\Delta H_{r x n}=q_{p}$.

## Introduction to Hess's Law:

1) Hess's law of heat summation states that for a chemical equation that can be written as the sum of two or more steps, the enthalpy change for the overall equation is the sum of the enthalpy changes for the individual steps.
2) An example calculation for the decomposition of 2 moles of $\mathrm{CaCO}_{3}$.
a. Unknown equation: $2 \mathrm{CaCO}_{3}(s) \rightarrow 2 \mathrm{CaO}(s)+2 \mathrm{CO}_{2}(g) \quad \Delta H=$ ?
b. Known equations:
i. $\mathrm{Ca}(s)+\mathrm{C}(s)+3 / 2 \mathrm{O}_{2}(g) \rightarrow \mathrm{CaCO}_{3}(s)$
ii. $\mathrm{C}(\mathrm{s})+\mathrm{O}_{2}(g) \rightarrow \mathrm{CO}_{2}(g)$
iii. $\mathrm{Ca}(s)+1 / 2 \mathrm{O}_{2}(g) \rightarrow \mathrm{CaO}(s)$
c. Step 1: Flip Equation \#1 (and the sign of $\Delta \mathrm{H}^{\mathbf{0}}{ }_{1}$ )
i. $\mathrm{CaCO}_{3}(s) \rightarrow \mathrm{Ca}(s)+\mathrm{C}(s)+3 / 2 \mathrm{O}_{2}(g) \quad \Delta \mathrm{H}^{\mathrm{o}}{ }_{1}=-(-1208 \mathrm{~kJ})$
d. Step 2: Cancel Like Terms and Add $\Delta \mathrm{H}^{\circ}$
i. $\mathrm{CaCO}_{3}(s) \rightarrow \mathrm{Ca}(s)+\mathrm{C}(s)+3 / 2 \mathrm{O}_{2}(g) \quad \Delta \mathrm{H}^{\mathrm{o}}=+1208 \mathrm{~kJ}$
ii. $\mathrm{C}(s)+\mathrm{O}_{2}(g) \rightarrow \mathrm{CO}_{2}(g) \quad \Delta \mathrm{H}^{\mathrm{o}}{ }_{2}=-393.5 \mathrm{~kJ}$
iii. $\mathrm{Ca}(s)+1 / 2 \mathrm{O}_{2}(g) \rightarrow \mathrm{CaO}(s) \quad \Delta \mathrm{H}^{0}{ }_{3}=-635.0 \mathrm{~kJ}$
iv. $\mathrm{CaCO}_{3}(s) \rightarrow \mathrm{CaO}(s)+\mathrm{CO}_{2}(g) \quad \Delta \mathrm{H}_{\mathrm{rxn}}^{\mathrm{o}}=+179.5 \mathrm{~kJ}$
e. Step 3: Multiply by Entire Equation by $2 \& \Delta \mathrm{H}_{\mathrm{rxn}}^{0}$

$$
\text { i. } 2\left(\mathrm{CaCO}_{3}(s) \rightarrow \mathrm{CaO}(s)+\mathrm{CO}_{2}(g)\right) \quad \Delta \mathrm{H}_{\mathrm{rxn}}^{\mathrm{o}}=2(+179.5 \mathrm{~kJ})
$$

f. Final Answer:

$$
\text { i. } 2 \mathrm{CaCO}_{3}(s) \rightarrow 2 \mathrm{CaO}(s)+2 \mathrm{CO}_{2}(g) \quad \Delta \mathbf{H}_{\mathbf{r x n}}^{0}=+\mathbf{3 5 9} \mathbf{~ k J}
$$

## Introduction to Bond Energy:

1) Bond energy (BE) is defined as the average heat (enthalpy) needed to break a molecular bond in the gaseous phase.
a. All bond energies are endothermic because bond cleavage can only occur following the absorption of energy.
b. The following table provides the bond energies for common covalent bonds in $\mathrm{kJ} / \mathrm{mol}$ :

Table A: Bond Energies for Common Covalent Bonds in kJ/mol*

| Bond | Bond Energy |  | Bond |
| :---: | :---: | :---: | :---: |
| Bond Energy |  |  |  |
| $\mathrm{C}-\mathrm{H}$ | 411 | $\mathrm{O}-\mathrm{H}$ | 459 |
| $\mathrm{C}-\mathrm{O}$ | 358 | $\mathrm{O}-\mathrm{O}$ | 142 |
| $\mathrm{C}=\mathrm{O}$ | 745 | $\mathrm{O}=\mathrm{O}$ | 494 |
| $\mathrm{C}-\mathrm{C}$ | 346 | $\mathrm{C}-\mathrm{N}$ | 305 |
| $\mathrm{C}=\mathrm{C}$ | 602 | $\mathrm{C} \equiv \mathrm{N}$ | 887 |
| $\mathrm{C} \equiv \mathrm{C}$ | 835 | $\mathrm{~N}-\mathrm{H}$ | 386 |

*Assuming gaseous state
c. The enthalpy, $\Delta \mathrm{H}$, of a reaction is approximately equal to the sum of the bond energies (BE) of the reactants minus the sum of the bond energies (BE) of the products.
i. $\quad \Delta \mathrm{H}=\Sigma \mathrm{nBE}_{\text {(bonds broken) }}-\Sigma \mathrm{nBE}_{\text {(bonds formed) }}$
ii. Note: This method is only an approximation and using thermochemical data is more accurate at determining the enthalpy for a reaction.

## Catalytic Decomposition of Hydrogen Peroxide:

3) Hydrogen peroxide, $\mathrm{H}_{2} \mathrm{O}_{2}$, decomposes by disproportionation, into liquid water and oxygen gas.
a. This decomposition is spontaneous at room temperature but quite slow.
b. It can be catalyzed by the addition of the iodide ion ( $\mathrm{I}^{-}$), manganese dioxide $\left(\mathrm{MnO}_{2}\right)$, or an iron(III) catalyst $\left(\mathrm{Fe}^{3+}\right)$.
c. The balanced equation is depicted below:

$$
\mathrm{H}_{2} \mathrm{O}_{2}(a q) \xrightarrow{\mathrm{Fe}\left(\mathrm{NO}_{3}\right)_{3}} \mathrm{H}_{2} \mathrm{O}(l)+1 / 2 \mathrm{O}_{2}(g)
$$

## Procedure for the Heat of Reaction via Calorimetry:

1) Obtain the mass of two clean and dried empty Styrofoam cups with a cap on an ANALYTICAL balance.
a. These two cups should be stacked on top of one another and the cap should have a hole in it to facilitate the use of your thermometer.
b. This set of Styrofoam cups will function as your calorimeter.
c. Record mass in data table.
2) Using a clean and dry graduated cylinder, add 50.0 mL of $3 \%$ hydrogen peroxide $\left(\mathrm{H}_{2} \mathrm{O}_{2}\right)$ to the calorimeter and obtain the mass on an ANALYTICAL balance.
a. Ensure that no water or hydrogen peroxide solution is clinging to the sides of the Styrofoam cups.
b. Record the mass in the data table.
3) Using a clean and dry 10.0 mL graduated cylinder, measure out 10.0 mL of 0.20 $-0.50 \mathrm{M} \mathrm{Fe}\left(\mathrm{NO}_{3}\right)_{3}$ solution.
4) Measure the stable temperature of the hydrogen peroxide solution the nearest tenth to hundredth of a degree depending on the thermometer calibrations $\left(0.1^{\circ} \mathrm{C}\right.$ $-0.01^{\circ} \mathrm{C}$ ).
a. Record measurement in data table.
5) Transfer the $\mathrm{Fe}\left(\mathrm{NO}_{3}\right)_{3}$ solution into the calorimeter.
a. Ensure that you do not allow any of the hydrogen peroxide solution from inside the calorimeter to splash out.
6) Replace the cap and gently stir the mixture with the thermometer or swirl the calorimeter.
7) Record the final (and maximum) temperature in the calorimeter after stabilization to the nearest tenth to hundredth of a degree depending on the thermometer calibrations $\left(0.1^{\circ} \mathrm{C}-0.01^{\circ} \mathrm{C}\right)$.
a. Continually swirl/stir for no less than 5 minutes.
b. Discontinue swirling/stirring the temperature has stabilized and the reaction has been completed.
c. It may be advisable to collect temperature and time data every 30 seconds to ensure accuracy.
8) Following this measurement, discard solution down the sink and clean cups thoroughly.
9) Repeat this experiment for a second trial.

## Procedure for Calculation of the Heat of Reaction by Bond Energy:

1) Draw the Lewis dot structure for the reactant and two products for the decomposition of hydrogen peroxide.
a. Place these structures into your data table.
2) Using the bond energies in Table A , calculate the enthalpy for the reaction using the formula below.
d. $\Delta \mathrm{H}=\Sigma \mathrm{nBE}_{\text {(bonds broken) }}-\Sigma \mathrm{nBE}_{\text {(bonds formed) }}$.
3) Compare to calorimetric data.

## Procedure for Calculation of the Heat of Reaction by Hess's Law:

1) Using the two equations provided in the data table with their enthalpies, apply Hess's Law to solve for the true heat of reaction.
2) Compare the true value to the enthalpy via calorimetry and via BE.
e. Calculate a percent error for each.

## Waste Disposal and Lab Clean-Up:

1) All hydrogen peroxide solutions can be placed down the sink.
2) Do not discard the Styrofoam cups with caps; they are reused. Wash and dry.

## The Lab - Write Up:

1) Determine the enthalpy of reaction for catalytic the decomposition of hydrogen peroxide via calorimetry.
a. Discuss relevant data in your conclusion.
2) Determine the enthalpy of reaction for catalytic the decomposition of hydrogen peroxide via bond energy provided in Table A.
a. Discuss relevant data in your conclusion.
3) Determine the true enthalpy of reaction for catalytic the decomposition of hydrogen peroxide via Hess's Law.
a. Discuss relevant data in your conclusion.
4) Compare experimental enthalpy of reaction via calorimetry and bond energy to the true value and to one another.
a. Determine the percent error in each experiment using the following formula and discuss in conclusion:
i. [(true value - experimental value)/true value] * $100 \%$
ii. Percent error cannot be a negative value. Take the absolute value of the number if necessary.
b. Which method of determine the enthalpy for the reaction was more accurate? Explain.
5) Discuss all reasons for error and how these errors affected the data.
6) What assumptions have been made throughout this experiment that could have accounted for error?

Sample Data Table for the Enthalpy of Reaction via Calorimetry:

| Data Recorded/Calculated | Trial \#1 | Trial \#2 |
| :---: | :---: | :---: |
| a) Mass of Styrofoam cups with cap in grams: |  |  |
| b) Volume of $3.0 \%$ hydrogen peroxide solution measured in milliliters: |  |  |
| c) Mass of Styrofoam cups with cap plus $3.0 \%$ hydrogen peroxide solution in grams: |  |  |
| d) Mass of hydrogen peroxide solution in grams (c-a): |  |  |
| e) Density of $3.0 \%$ hydrogen peroxide solution in $\mathrm{g} / \mathrm{mL}$ (d/b): |  |  |
| f) Moles of hydrogen peroxide in 3.0\% solution in mol: |  |  |
| g) Volume of aqueous iron (III) nitrate solution measured in milliliters: |  |  |
| h) Stable temperature of hydrogen peroxide solution before catalyst addition $\left({ }^{\circ} \mathrm{C}\right)$ : |  |  |
| i) Stable temperature of hydrogen peroxide solution after catalyst addition $\left({ }^{\circ} \mathrm{C}\right)$ : |  |  |
| j) Specific heat of a 3.0\% hydrogen peroxide solution in $\mathrm{J} /\left(\mathrm{g} \bullet^{\circ} \mathrm{C}\right)$ : | 4.184 | 4.184 |
| k) Amount of heat gained (in Joules) by 3.0\% hydrogen peroxide solution ${ }^{\dagger}$ : |  |  |
| l) Amount of heat lost (in Joules) by the hydrogen peroxide reacting (or heat of reaction) ${ }^{+1}$ : |  |  |
| m) Amount of heat lost (in kilojoules) by the hydrogen peroxide reacting (or heat of reaction) $(\ell / 1000)$ : |  |  |
| n) Enthalpy of reaction (in kilojoules per mole) for the decomposition of hydrogen peroxide $(\mathrm{m} / \mathrm{f})$ : |  |  |
| o) Average enthalpy of reaction (in kilojoules per mole) for the decomposition of hydrogen peroxide $\left(\left(\mathrm{n}_{1}+\mathrm{n}_{2}\right) / 2\right)$ : |  |  |

Sample Data Table the Heat of Reaction by Bond Energy:


| Data Recorded/Calculated |  |
| :--- | :--- |
| a) Sum of bonds broken* (in $\mathrm{kJ} / \mathrm{mol}):$ | Data |
| b) Sum of bonds formed* (in $\mathrm{kJ} / \mathrm{mol}):$ |  |
| c) Enthalpy of reaction (in $\mathrm{kJ} / \mathrm{mol})$ for the <br> decomposition of hydrogen peroxide via BE $(\mathrm{a}-\mathrm{b}):$ |  |

*Use information from Table A from the "Introduction to Bond Energy" section
Sample Data Table for the Enthalpy of Reaction via Hess's Law:

| Data Recorded/Calculated | Data |
| :--- | :--- |
| a) Known reaction \#1: $\mathrm{H}_{2}(g)+\mathrm{O}_{2}(g) \rightarrow \mathrm{H}_{2} \mathrm{O}_{2}(a q)$ | $\Delta \mathrm{H}_{\mathrm{f}}=-191.2 \mathrm{~kJ} / \mathrm{mol}$ |
| b) Known reaction \#2: $\mathrm{H}_{2}(g)+1 / 2 \mathrm{O}_{2}(g) \rightarrow \mathrm{H}_{2} \mathrm{O}(l)$ | $\Delta \mathrm{H}_{\mathrm{f}}=-285.8 \mathrm{~kJ} / \mathrm{mol}$ |
|  |  |
| c) Unknown reaction: $\mathrm{H}_{2} \mathrm{O}_{2}(a q) \rightarrow \mathrm{H}_{2} \mathrm{O}(l)+1 / 2 \mathrm{O}_{2}(g)$ | $\Delta \mathrm{H}_{\mathrm{rxn}}=$ |
|  |  |

Sample Data Table for the Enthalpy of Reaction Comparison:

| Data Recorded/Calculated | Data |  |
| :--- | :--- | :---: |
| a) True enthalpy of reaction (in kJ/mol) for the <br> decomposition of hydrogen peroxide via Hess's Law: | Via <br> Calorimetry |  |
| Via Bond <br> Energy |  |  |
| b) Experimental enthalpy of reaction (in kJ/mol) for the <br> decomposition of hydrogen peroxide: |  |  |
| c) Percent error (\%): |  |  |
| d) Which method is more accurate?* |  |  |

[^2]
## Experiment \#11B: Periodicity of Oxides and Lewis Dot - Diagrams

## Introduction to Oxides:

1) Oxygen is the most prevalent element on Earth.
a. Free diatomic oxygen $\left(\mathrm{O}_{2}\right)$ makes up about $20 \%$ of the air by volume.
b. Water is approximately $89 \%$ oxygen by mass.
c. Oxides $\left[\mathrm{M}_{\mathrm{x}} \mathrm{O}_{\mathrm{y}}\right]$ are the most abundant salts in the Earth's crust.
2) Oxygen at room temperature is fairly unreactive; its reactivity is enhanced by increasing the temperature.
3) Pure diatomic oxygen gas can be generated by the decomposition of chlorates.
a. Our reaction: $2 \mathrm{KClO}_{3}(s) \xrightarrow{\mathrm{MnO}_{2}} 2 \mathrm{KCl}(s)+3 \mathrm{O}_{2}(g)$
b. Manganese dioxide is a catalyst implemented to increase the rate of the reaction.
4) Oxides can be classified by their acidity and basicity.
a. Basic oxides produce alkaline solutions $(\mathrm{pH}>7)$ and react with acids.
i. Most basic oxides are produced from reactions of metals with oxygen.
ii. Basic oxide synthesis: $2 \mathrm{Ba}(s)+\mathrm{O}_{2}(g) \rightarrow 2 \mathrm{BaO}(s)$
iii. Basic oxide in water: $\mathrm{BaO}(s)+\mathrm{H}_{2} \mathrm{O}(l) \rightarrow \mathrm{Ba}(\mathrm{OH})_{2}(a q)$
b. Acidic oxides produce acidic solutions $(\mathrm{pH}<7)$ and react with bases.
i. Most acidic oxides are produced from reactions of non-metals with oxygen.
ii. Acidic oxide synthesis: $\mathrm{Se}(s)+\mathrm{O}_{2}(g) \rightarrow \mathrm{SeO}_{2}(s)$
iii. Acidic oxide in water: $\mathrm{SeO}_{2}(s)+\mathrm{H}_{2} \mathrm{O}(l) \rightarrow \mathrm{H}_{2} \mathrm{SeO}_{3}(a q)$
c. Amphoteric oxides can have properties of both acidic and basic oxides.
i. Many amphoteric oxides are produced from reactions of semimetals with oxygen.
ii. Depending on the environment, amphoteric oxides can produce acid or basic compounds.
iii. Example:
1. Amphoteric oxide synthesis: $4 \mathrm{Al}(s)+3 \mathrm{O}_{2}(g) \rightarrow 2 \mathrm{Al}_{2} \mathrm{O}_{3}(s)$
2. Reaction of amphoteric oxide under basic conditions: $\mathrm{Al}_{2} \mathrm{O}_{3}(s)+3 \mathrm{H}_{2} \mathrm{O}(l)+2 \mathrm{NaOH}(a q) \rightarrow 2 \mathrm{NaAl}(\mathrm{OH})_{4}(a q)$
3. Reaction of amphoteric oxide under acidic conditions:

$$
\mathrm{Al}_{2} \mathrm{O}_{3}(s)+3 \mathrm{H}_{2} \mathrm{O}(l)+6 \mathrm{HCl}(a q) \rightarrow 2 \mathrm{AlCl}_{3}(a q)+6 \mathrm{H}_{2} \mathrm{O}(l)
$$

The Reactive Elements: Aluminum (Al), Carbon Charcoal (C), Magnesium (Mg), Oxygen ( $\mathrm{O}_{2}$ ), Red Phosphorus ( P ), Sulfur ( $\mathrm{S}_{8}$ ).

## The Oxides:

1) Aluminum oxide*
2) Calcium oxide
3) Carbon dioxide*
4) Diphosphorus pentoxide*
5) Diiodine pentoxide
6) Magnesium oxide*
7) Sodium oxide
8) Silicon dioxide
*The indicated oxides will be synthesized from the reactive elements and pure oxygen and then reacted with water.

The other oxides will be provided and only reacted with water.
9) Sulfur dioxide*

## Part 1: Procedure for Generating Pure Diatomic Oxygen:

14) Obtain 6 glass bottles, 6 glass squares, a rubber stopper with a hollow tube of glass through it, and a large test tube.
a. Ensure that this glassware is clean and dried.
15) Acquire a metal or plastic tray with a rubber hose attached to the overflow valve.
16) Fill the tray to the overflow line with tap water and ensure the hose is placed into the sink.
17) Weigh out approximately 10 grams of potassium chlorate and 2 grams of manganese dioxide.
a. Mix together thoroughly in test tube ensuring that powder along the rim is wiped off with a Kimwipe.
18) Clamp the test tube at a $45^{\circ}$ angle on a ring stand or to the lab scaffolding.
19) Secure the rubber stopper containing the hollow glass tube into the test tube.
20) Run a secure portion of rubber tubing onto the end of the glass tube and run into the pan of water.
21) One at a time, fill up each glass bottle to the brim with tap water.
a. Slide the glass plate onto the lip, invert into the pan, and then remove the glass plate.
b. Try not to allow any air to enter the inverted glass bottles.
22) Begin heating the test tube with a Bunsen burner flame moderately by fanning the flame at the bottom of the tube.
a. Do NOT stop heating the sample during this process; a reverse vacuum can be created producing a potentially explosive situation.
23) Place the end of the rubber tubing into the mouth of one of the glass bottles and collect the oxygen.
a. The oxygen should displace out the water that is inside the glass bottle.
24) Once full of oxygen and all of the water has been displaced, slide the glass plate over the mouth of the bottle and set up-right onto the lab bench.
a. Label this container \#1.
25) Fill up the remaining five bottles with oxygen in the same fashion.
26) Remove the delivery tube from the tray BEFORE discontinuing the heating of the sample.

## Part 2: Procedure for Generating Oxides \& Reacting with Water:

1) Add approximately 25 mL of distilled water to each bottle by slightly removing the glass plate and quickly re - covering.
a. Ensure that all collected oxygen gas does not escape!
2) Bottle \#1: The glowing splint test
a. Insert a wooden splint into a Bunsen burner flame until it ignites and then blow out.
b. Insert the glowing splint into the first bottle, still keeping it slightly capped. Note observations.
3) Bottle \#2: Generation of carbon dioxide
a. Load a deflagrating spoon with carbon charcoal.
b. Hold the sample into a Bunsen burner flame for a few minutes or until the spoon begins to glow.
c. Slightly slide off the glass plate from the second oxygen bottle and lower the hot carbon to a depth above the water level. Note observations.
d. Keep the bottle as covered as possible.
e. As soon as the carbon ceases burning, remove spoon but keep the glass plate in place. Swirl.
i. Do not allow unreacted carbon to fall into water.
f. Test the pH by dropping a piece of pH paper into the solution. If the pH is neutral, cool bottle in an ice-water bath until a change is present.
i. If there is still no change, obtain a small quantity of dry ice (solid $\mathrm{CO}_{2}$ ) and drop into the water. Retest pH and note in observations.
4) Bottle \#3: Generation of magnesium oxide
a. Hold an 8 cm strip of magnesium ribbon with a pair of crucible tongs into a Bunsen burner flame.
b. As soon as it ignites, quickly slide off the glass plate from the third oxygen bottle and lower the burning magnesium to a depth above the water level. Note observations.
c. Keep the bottle as covered as possible.
d. After it has completely combusted, allow ash to drop into the water. Swirl.
e. Test the pH by dropping a piece of pH paper into the solution. If the pH is neutral, warm bottle in a water bath until a change is present.
5) Bottle \#4: Generation of aluminum oxide
a. Ball up a piece of aluminum foil and load into a deflagrating spoon.
b. Hold the sample into a Bunsen burner flame until spoon begins to glow. As soon as you see it glowing, quickly proceed to the next step.
c. Slightly slide off the glass plate from the fourth oxygen bottle and lower the hot aluminum to a depth above the water level. Note observations.
d. Keep the bottle as covered as possible.
e. Allow ash to drop into the water. Swirl.
f. Test the pH by dropping a piece of pH paper into the solution. Warm bottle in a water bath and then repeat pH test.
6) Bottle \#5: Generation of sulfur dioxide
a. Load sulfur into a deflagrating spoon and take to the hood with the fifth oxygen bottle.
b. Hold the sample into a Bunsen burner flame until it ignites.
c. Slightly slide off the glass plate from the fifth oxygen bottle and lower the burning sulfur to a depth above the water level. Note observations.
d. Keep the bottle as covered as possible.
e. As soon as the sulfur ceases burning, remove spoon but keep the glass plate in place. Swirl.
f. Test the pH by dropping a piece of pH paper into the solution. If the pH is neutral, cool bottle in an ice-water bath until a change is present.
7) Bottle \#6: Generation of diphosphorus pentoxide
a. Load a small amount of red phosphorus into a deflagrating spoon in the hood.
b. Hold the sample into a Bunsen burner flame until it ignites.
c. Slightly slide off the glass plate from the sixth oxygen bottle and lower the burning phosphorus to a depth above the water level. Note observations.
d. Only keep bottle partially covered by glass plate; phosphorus burns very hot and may break glass plate if bottle is completely covered.
e. As soon as the phosphorus ceases burning, remove spoon but recover with the glass plate. Swirl.
f. Test the pH by dropping a piece of pH paper into the solution. If the pH is neutral, warm bottle in a water bath until a change is present.
i. If there is still no change, obtain a small quantity of diphosphorus pentoxide powder and add to the water. Swirl, retest pH , and note in observations.

## Part 3: Procedure for Reacting Provided Oxides With Water:

1) Bottle \#7: Generating aqueous solution of calcium oxide.
a. Obtain an empty glass bottle and place 25 mL of deionized water into bottle.
b. Place $\sim 0.5$ grams of calcium oxide into the bottle and swirl. Note the evolution of heat.
c. Test the pH by dropping a piece of pH paper into the solution. If the pH is neutral, warm bottle in a water bath until a change is present.
2) Bottle \#8: Generating aqueous solution of silicon dioxide.
a. Obtain an empty glass bottle and place 25 mL of deionized water into bottle.
b. Place $\sim 0.5$ grams of silicon dioxide* into the bottle and swirl.
ii. *A fine powder of silicon dioxide is called silica.
c. Test the pH by dropping a piece of pH paper into the solution. If the pH is neutral, warm bottle in a water bath until a change is present.
3) Bottle \#9: Generating aqueous solution of sodium oxide.
a. Obtain an empty glass bottle and place 25 mL of deionized water into bottle.
b. Place $\sim 0.5$ grams of sodium oxide into the bottle and swirl. Note the evolution of heat.
c. Test the pH by dropping a piece of pH paper into the solution. If the pH is neutral, warm bottle in a water bath until a change is present.
4) Bottle \#10: Generating aqueous solution of diiodine pentoxide (AKA iodine pentoxide).
a. Obtain an empty glass bottle and place 25 mL of deionized water into bottle.
b. Place $\sim 0.5$ grams of iodine pentoxide into the bottle and swirl. Note the evolution of heat.
c. Test the pH by dropping a piece of pH paper into the solution. If the pH is neutral, warm bottle in a water bath until a change is present.

## Waste Disposal and Lab Clean - Up:

1) Place all used pH papers into the trash can.
2) Place all aqueous oxide products into the same waste beaker.
3) Place reacted potassium chlorate into the trash can.
4) Make sure that all gas valves are turned off!

## The Lab Write - Up:

1) Determine all values in your data table.
2) Provide all balanced reactions for formations of oxides.
3) Provide all balanced reactions for oxides with water.
4) In conclusion, explain data collected \& discuss reasons for error.
5) Discuss the acidic, basic, and amphoteric oxide trends in the periodic chart in reference to your data.
6) Which oxides are ionic and which are molecular? How does this affect the Lewis-dot diagram?

## Balanced Equations for Oxide Formation:

1) Balanced Equation for Aluminum Oxide Formation: $\qquad$
2) Balanced Equation for Carbon Dioxide Formation: $\qquad$
3) Balanced Equation for Diphosphorus Pentoxide Formation: $\qquad$
4) Balanced Equation for Magnesium Oxide Formation: $\qquad$
5) Balanced Equation for Sulfur Dioxide Formation: $\qquad$

## Balanced Equations of Reactions of Oxides with Water:

1) Balanced Equation for Aluminum Oxide + Water: $\qquad$
2) Balanced Equation for Calcium Oxide + Water: $\qquad$
3) Balanced Equation for Carbon Dioxide + Water: $\qquad$
4) Balanced Equation for Diphosphorus Pentoxide + Water: $\qquad$
5) Balanced Equation for Iodine Pentoxide + Water: $\qquad$
6) Balanced Equation for Magnesium Oxide + Water: $\qquad$
7) Balanced Equation for Silicon Dioxide + Water: $\qquad$
8) Balanced Equation for Sodium Oxide + Water: $\qquad$
9) Balanced Equation for Sulfur Dioxide + Water: $\qquad$

## Sample Data Table for Oxide Results - 1:

| Name of <br> Oxide | Formula | pH of <br> Water | Acid, Base, or <br> Amphoteric | Lewis Dot <br> Structure of <br> Oxide |
| :---: | :---: | :---: | :---: | :---: |
| Aluminum <br> Oxide |  |  |  |  |
| Calcium <br> Oxide |  |  |  |  |
| Carbon <br> Dioxide |  |  |  |  |
| Diphosphorus <br> Pentoxide |  |  |  |  |
| Diiodine <br> Pentoxide |  |  |  |  |

## Sample Data Table for Oxide Results - 2:

| Name of <br> Oxide | Formula | pH of <br> Water | Acid, Base, or <br> Amphoteric | Lewis Dot <br> Structure of <br> Oxide |
| :---: | :--- | :--- | :--- | :--- |
| Magnesium <br> Oxide |  |  |  |  |
| Silicon <br> Dioxide |  |  |  |  |
| Sodium <br> Oxide |  |  |  |  |
| Sulfur <br> Dioxide |  |  |  |  |

## Experiment \#12: Electron Pair \& Molecular Geometry

## Introduction to VSEPR:

1) The valence - shell electron - pair repulsion model (VSEPR) can predict the shape of molecules and ions by proposing that valence-shell electron pairs are arranged about each atom such that these pairs are as far away from one another as possible to minimize electron-pair repulsions.
2) Electron pair geometry is the shape that is obtained by assuming the electron pairs are groups around the central atom.
a. There are only five types of electron pair geometry:
i. Linear - two groups around central atom
ii. Trigonal planar - three groups around central atom
iii. Tetrahedral - four groups around the central atom
iv. Trigonal bipyramidal - five groups around the central atom
v. Octahedral - six groups around the central atom
3) Molecular geometry is the shape that is obtained by assuming the electron pairs are absent around the central atom.
a. There are multiple types of molecular geometries depending on the number of bonding groups (or substituents) and the number of loan pairs.
b. The best way to determine molecular geometry is by following the following steps:
i. Step 1: Draw the Lewis dot structure
ii. Step 2: Determine how many groups (or electron pairs) are around the central.
1. Note: Multiple bonds (i.e. double or triple bonds) are considered one group (or pair).
iii. Step 3: With the number of groups (or electron pairs) designated, select the appropriate electron pair geometry.
iv. Step 4: Remove lone electron pairs and assess new geometry.
c. All possible molecular geometries, with examples, are provided in Table A below.
4) Molecular polarity is dependent upon the generation of dipole moments due to differences in electronegativity between bonding substituents and the geometry of the resultant molecule.
a. Nonpolar molecules have no net dipole.
b. Polar molecules have net nonzero dipole moments.
c. Dipole moment information is provided in Table B below.

Table A: Molecular Geometries Predicted by the VSEPR Model

| Total <br> Groups <br> on <br> Central <br> Atom | Bonded <br> Groups to <br> Central <br> Atom | Nonbonding <br> Electron <br> Pairs on <br> Central <br> Atom | VSEPR <br> Formula | Approx. <br> Bond <br> Angles | Electron <br> Pair <br> Geometry | Molecular <br> Geometry | Examples |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 2 | 2 | 0 | $\mathrm{AX}_{2}$ | $180^{\circ}$ | linear | linear | $\mathrm{BeF}_{2}$ |
| 3 | 3 | 0 | $\mathrm{AX}_{3}$ | $120^{\circ}$ | trigonal <br> planar | trigonal <br> planar | $\mathrm{BCl}_{3}$ |
| 3 | 2 | 1 | $\mathrm{AX}_{2} \mathrm{E}$ | $<120^{\circ}$ | trigonal <br> planar | angular, <br> bent | $\mathrm{SnCl}_{2}$ |
| 4 | 4 | 0 | $\mathrm{AX}_{4}$ | $109.5^{\circ}$ | tetrahedral | tetrahedral | $\mathrm{CH}_{4}$ |
| 4 | 3 | 1 | $\mathrm{AX}_{3} \mathrm{E}$ | $<109.5^{\circ}$ | tetrahedral | trigonal <br> pyramidal | $\mathrm{PH}_{3}$ |
| 4 | 2 | 2 | $\mathrm{AX}_{2} \mathrm{E}_{2}$ | $<109.5^{\circ}$ | tetrahedral | angular, <br> bent | $\mathrm{H}_{2} \mathrm{O}$ |
| 5 | 5 | 0 | $\mathrm{AX}_{5}$ | $90^{\circ} / 120^{\circ}$ | trigonal <br> bipyramidal | trigonal <br> bipyramidal | $\mathrm{PI}_{5}$ |
| 5 | 4 | 1 | $\mathrm{AX}_{4} \mathrm{E}$ | $>90^{\circ} />120^{\circ}$ | trigonal <br> bipyramidal | irregular <br> tetrahedral | $\mathrm{SCl}_{4}$ |
| 5 | 3 | 2 | $\mathrm{AX}_{3} \mathrm{E}_{2}$ | $<90^{\circ}$ | trigonal <br> bipyramidal | $\mathrm{T}-$ shaped | $\mathrm{ClF}_{3}$ |
| 5 | 2 | 3 | $\mathrm{AX}_{2} \mathrm{E}_{3}$ | $180^{\circ}$ | trigonal <br> bipyramidal | linear <br> octahedral | $\mathrm{XeF}_{2}$ |
| 6 | 6 | $\mathrm{AX}_{6}$ | $90^{\circ}$ | octahedral | $\mathrm{SF}_{6}$ |  |  |
| 6 | 5 | 1 | $\mathrm{AX}_{5} \mathrm{E}$ | $>90^{\circ}$ | octahedral | square <br> pyramidal | $\mathrm{BrF}_{5}$ |
| 6 | 4 | 2 | $\mathrm{AX}_{4} \mathrm{E}_{2}$ | $90^{\circ}$ | octahedral <br> pquare | $\mathrm{ICl}_{4}^{-}$ |  |

${ }^{\ddagger}$ A double or triple bond counts as a single group.

* $\mathrm{A}=$ central atom, $\mathrm{X}=$ substituent/bonding group, $\mathrm{E}=$ lone electron pair

Table B: Net Dipoles of Predicted Molecular Geometries

| VSEPR* Formula | Molecular Geometry | Net Dipole ${ }^{\dagger}$ |
| :---: | :---: | :---: |
| AX | Linear | Can be nonzero |
| $\mathrm{AX}_{2}$ | Linear | Zero |
| $\mathrm{AX}_{2}$ | Bent | Can be nonzero |
| $\mathrm{AX}_{3}$ | Trigonal planar | Zero |
| $\mathrm{AX}_{3}$ | Trigonal pyramidal | Can be nonzero |
| $\mathrm{AX}_{3}$ | T-shaped | Can be nonzero |
| $\mathrm{AX}_{4}$ | Tetrahedral | Zero |
| $\mathrm{AX}_{4}$ | Square planar | Zero |
| $\mathrm{AX}_{4}$ | Irregular tetrahedral | Can be nonzero |
| $\mathrm{AX}_{5}$ | Trigonal bipyramidal | Zero |
| $\mathrm{AX}_{5}$ | Square pyramidal | Can be nonzero |
| $\mathrm{AX}_{6}$ | Octahedral | Zero |

## Experimental Procedure:

1) Draw out the appropriate Lewis structure for all of the molecules or ions in the two charts below.
2) Using the molecular model kits provided in the lab, construct ball and stick models of these molecules or ions.
a. Ensure that you include all groups around the central atom; that includes both bonding atoms and nonbonding electron pairs.
3) Following construction, determine the following information about each:
a. The number of total groups (electron pairs) on the central atom, the number of bonding groups (or substituents), and the number of lone (nonbonding) electron pairs.
b. The electron pair geometry
c. The molecular geometry
d. The approximate bond angles between the central atom and its substituents
e. The polarity of the resultant molecule or ion

## Waste Disposal and Lab Clean-Up:

1) Place all materials for the molecular model kits back in the original box and store as indicated by your instructor.

## The Lab Write - Up:

1) Determine all values or structures in your data table. Discuss relevant data in conclusion
2) Discuss the factor(s) that govern(s) the polarity of molecules.
3) What is meant by hybrid orbitals? Give two examples of the process of orbital hybridization.
4) What is formal charge? Explain the formula and how this can assist at determining correct Lewis structures.

## Molecules/Ions that Obey Octet Rule

| Molecule/ Ion | Lewis Structure |  | Bonding <br> Groups on <br> Central Atom <br> Nonbonding <br> Electron Pairs <br> on Central <br> Atom | Electron Pair Geometry | Molecular Geometry | Approx. Bond Angles | Polar or Nonpolar |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| $\mathrm{CH}_{4}$ | H |  | 4 | Tetrahedral | Tetrahedral | $109.5^{\circ}$ | Nonpolar |
|  | $\ddot{H}$ |  | 0 |  |  |  |  |
| $\mathrm{OF}_{2}$ |  |  |  |  |  |  |  |
| $\mathrm{NH}_{3}$ |  |  |  |  |  |  |  |
| $\mathrm{SF}_{2}$ |  |  |  |  |  |  |  |
| $\mathrm{H}_{3} \mathrm{O}^{+}$ |  |  |  |  |  |  |  |
| $\mathrm{BrF}_{2}{ }^{+}$ |  |  |  |  |  |  |  |
| $\mathrm{CO}_{3}{ }^{2-}$ |  |  |  |  |  |  |  |
| $\mathrm{BF}_{4}{ }^{-}$ |  |  |  |  |  |  |  |
| $\mathrm{BeCl}_{2}$ |  |  |  |  |  |  |  |

Note: $\mathrm{BeCl}_{2}$ does not satisfy the octet.

## Molecules/Ions that Do Not Obey Octet Rule

| Molecule/ Ion | Lewis Structure | Total Groups on Central Atom | Bonding Groups on Central Atom Nonbonding Electron Pairs on Central <br> Atom | Electron Pair Geometry | Molecular Geometry | Approx. <br> Bond <br> Angles | Polar or Nonpolar |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| $\mathrm{SO}_{4}{ }^{2-}$ |  | 4 | 4 0 | Tetrahedral | Tetrahedral | $\sim 109.5^{\circ}$ | Polar |
| $\mathrm{AsF}_{5}$ |  |  |  |  |  |  |  |
| $\mathrm{BrF}_{4}^{-}$ |  |  |  |  |  |  |  |
| $\mathrm{XeF}_{4}$ |  |  |  |  |  |  |  |
| $\mathrm{I}_{3}{ }^{-}$ |  |  |  |  |  |  |  |
| $\mathrm{BrF}_{3}$ |  |  |  |  |  |  |  |
| $\mathrm{SnF}_{6}{ }^{2-}$ |  |  |  |  |  |  |  |
| $\mathrm{PF}_{5}$ |  |  |  |  |  |  |  |
| $\mathrm{ICl}_{2}{ }^{-}$ |  |  |  |  |  |  |  |


[^0]:    *To determine the mole to mole ratio in whole numbers, divide the number of moles for both Mg and O by the smallest number of moles from the two. Round values to the nearest whole number.

[^1]:    ${ }^{\dagger} \mathrm{q}=(\mathrm{c}+\mathrm{d})\left(4.184 \mathrm{~J} / \mathrm{g} \bullet{ }^{\circ} \mathrm{C}\right)(\mathrm{i})$
    ${ }^{\dagger} \mathrm{q}_{\mathrm{H} 2 \mathrm{O}}=-\mathrm{q}_{\text {salt }}(\mathrm{NH} 4 \mathrm{NO} 3$ or CaCl 2$)$

[^2]:    *Select from "Via Calorimetry" or "Via Bond Energy"

