CHM 1045 Lab Midterm Review

Density Measurements

Density = mass/volume

Density of Solids

- Measure the mass of the metal by using analytical balance
- Determine the volume by placing it in a graduated cylinder containing measured volume of water

Volume by Displacement

1) Volume of water before adding metal
2) Volume of water after adding metal

Take the difference between the two and that is the volume of the metal.

Density of Liquids

- Use an analytical balance pour water into graduate cylinder
- Use solutions of NaCl (4,8,6 and 12% plus unknown)

1) Measure mass using balance
2) Measure volume using cylinder
3) Record values for each solution
4) Divide the mass of NaCl solution by the volume to get the density
5) Make a line graph to show the density vs. % NaCl. Use this graph to find unknown solutions concentration

Percent Composition of MgO

- Heat Mg ribbon to produce MgO by burning
- Some may be lost as white smoke unless crucible is covered
- Mg can react w/O2 but also with N2 in the air. Reheat to get rid of N2
  $Mg_3N_2 + 3H_2O \rightarrow 3MgO + 2NH_3$

- MW MgO = 40.3 g
- % Mg in MgO = mass Mg = 0.601g, mass O = 0.406g
- Mass of MgO product result = 1.007 g MgO
- 0.601g/1.007g = 59.7%, 0.406g/1.007g = 40.3%
- % Expected (from the formula) = 24.3g Mg/40.3g MgO = 0.6029 or 60.3%

The percent error is calculated using the following formula

% error = observed- expected/expected *100

% error = 0.601 – 0.603/0.603 is 0.33%

Water of Hydration

- Composed of a solid and water
- The idea is that they are dehydrated or that they lose water

- Inorganic salts = hydrates
- Lose water = efflorescence
- Absorb water = hygroscopic, dessicants/drying agents
- Water is combined to cations and anions in the salt
- Remove water by heating to Bp of water
- Calculate the amount of water lost after heating and compare experimental values for % H_2O to theoretical values based on formula
- Procedure is to weigh first and then re-weigh to determine the % water in unknown hydrate

% KClO3 in Unknown

- Chemicals when heated break down into other compounds/gases
- Use the mole ratio and L.R.

$$2KClO_3(s) \rightarrow 2KCl(s) + 3O_2(g)$$

1.063g 0.637g 0.426g

Pure $KClO_3$ = 1.063g
Residue = 0.637g

[Mass of test tube, catalyst + pure $KClO_3$] – [Mass of test tube + catalyst] = Mass of Pure $KClO_3$
[Mass test tube, catalyst + residue] – [Mass test Tube and catalyst] = Mass of residue

Synthesis of Aspirin

- Use the given values in the lab to determine the moles of each based on the concept that M.W. =g/mol
- Re-arranging the equation allows you to calculate for both grams and the number of moles
- Theoretical yield is determined by the numbers that are given and Actual yield is a measure of what you obtained in the experiment

Water of Hydration

Objective:

To calculate the percent water by mass in several potential unknowns; to dehydrate a solid sample and identify it by comparison to the possible unknowns.

Background:

Many solids, especially inorganic salts, occur naturally as *hydrates*. This means that H_2O molecules are incorporated into the crystalline lattice in specific ways, that is, they are chemically combined. The materials may or may not look or feel "wet." They may spontaneously tend to lose water molecules in dry air, a process called *efflorescence*. Other hydrates absorb water molecules from humid air, are classified as *hygroscopic*, and are useful as *desiccants* (drying agents.)

Note that hydrates are not solids that are simply wet: most materials will absorb some water onto their surfaces. Hydrates, however, contain water molecules that are chemically combined to the cations and anions in the salt, and are present in specific molar ratios.

The common notation for hydrates is to use a raised dot (•) between the formula of the salt and the number of moles of water molecules per mole of salt. For example

$$\mathbf{CuSO_4 \cdot 5H_2O}$$

Hydrates are named by adding the number of water molecules to the end of the standard name of the salt. The Greek prefixes mono, di, tri, tetra, penta, etc., are used to indicate the number of moles of water. Thus

$\mathbf{CuSO_4 \cdot 5H_2O}$	**"copper (II) sulfate pentahydrate"**
$\mathbf{CaCl_2 \cdot 2H_2O}$	**"calcium chloride dihydrate"**

Water of hydration can be removed usually by simple heating to just above 100°C, the boiling point of water. In this experiment, you will identify an unknown salt from a list of potential unknowns by calculating the amount of water lost after heating and comparing the experimental value for percent water to the theoretical values based on the formulas.

Procedure:

1. Strongly heat, cool and weigh a crucible with lid.

2. Add about one gram of the unknown hydrate to the crucible and reweigh.

3. Support the crucible and cover (slightly ajar to allow steam to escape) on a wire triangle and heat gently for 5 minutes. Best results are obtained with gentle heat. Do not fry or melt the solid as this can lead to decomposition of the salt itself. You may wish to lift the cover occasionally to observe any changes in the material. When heating is finished, place the cover completely on the crucible (this prevents reabsorption of the water) and allow the crucible to cool completely.

4. Weigh the crucible and *anhydrous* salt.

5. Return the crucible to the wire triangle and heat gently for 5 minutes. Cool and re-weigh. Record the weigh. If the weigh after the second heating agrees with the first to the nearest 0.007 g, then no further heating is necessary. If the second heating appeared to drive off more water, heat for a third time, cool and re-weigh. Consult with your instructor if you think a fourth heating is necessary.

6. From your data calculate the percent H_2O in your unknown hydrate.

7. From the formula of the following hydrates calculate the percent H_2O in each.

Possible unknowns	Percent H_2O
$BaCl_2 \cdot 2H_2O$ 208.33g + 36g = 244.33g	14.7%
$CaSO_4 \cdot 2H_2O$ 136.144g + 36g = 172.144g	20.9%
$CuSO_4 \cdot 5H_2O$ 159.546g + 90g = 249.546g	36.06%
$NiCl_2 \cdot 6H_2O$ 129.693g + 108g = 237.693g	45.4%
$MgSO_4 \cdot 7H_2O$ 120.305g + 126g = 246.305g	51.15%

8. Identify your unknown by making the closest match.

Data:	Unknown A	Unknown B	Unknown C
1) Mass crucible and cover	79.376 g	g	g
2) Mass crucible, cover and hydrated salt *before heating*	80.394 g	g	g
3) Mass crucible, cover and Anhydrous salt (First weighing) *after heat*	79.988 g	g	g
4) Mass crucible, cover and Anhydrous salt (Second weighing) *after heat*	79.987 g	g	g
Mass crucible, cover and Anhydrous salt (Third weighing) *only if necessary*	N/A g	g	g

lose H_2O [beside items 3–4]

Results:			
Mass hydrated salt *#2 - #1*	1.018 g	g	g
Mass anhydrous salt (after final heating) *#4 - #1*	0.611 g	g	g
Mass H_2O lost *#2 - #4*	0.407 g	g	g
% H_2O in unknown hydrate	40.0%		
Formula of unknown hydrate	$FeCl_3 \cdot 6H_2O$		

mass anhydrous salt / mass hydrated salt (crossed out)

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$$\frac{\text{mass } H_2O \text{ lost (lost water)}}{\text{mass hydrated salt } (\text{w}/H_2O)} = 39.98\%$$

Density Measurements

Objective:

Determine densities of solids and liquids. Determine the concentration of an unknown NaCl solution.

Procedure: Density of Solids.

1. Measure the mass of a metal sample using an analytical balance.

2. Determine the volume of the metal by placing it in a 50-mL graduated cylinder containing a measured volume of water. Determine the new volume in the cylinder with the added metal sample. Make sure that the metal is completely covered with water. Record the volume measurements to the nearest 0.1 mL.

3. Calculate the density of the solid.

Procedure: Density of Liquids.

1. Measure the mass of water (use about 8 to 10 g) in a 10-mL graduated cylinder using an analytical balance.

2. Determine the volume of the water sample in the graduated cylinder to the nearest 0.1 mL.

3. Dry the 10-ml cylinder with a paper towel and then repeat steps 1 and 2 above using a 4.0%, 8.0%, 12.0%, 16.0% and unknown NaCl solution.

4. Calculate the densities of the liquid samples.

5. Plot density versus percent NaCl (note water will be 0% NaCl). From the graph estimate the concentration of the unknown NaCl solution.

Useful Information: Density = mass/volume

Data:
Density of solids

Mass of metal	________ g	________ g
Volume of water	________ mL	________ mL
Volume of water plus metal	________ mL	________ mL

Data:
Density of liquids

Mass of water (0% NaCl)	________ g
Volume of water (0% NaCl)	________ mL
Mass of 4.0% NaCl	________ g
Volume of 4.0% NaCl	________ mL
Mass of 8.0% NaCl	________ g
Volume of 8.0% NaCl	________ mL
Mass of 12.0% NaCl	________ g
Volume of 12.0% NaCl	________ mL
Mass of 16.0% NaCl	________ g
Volume of 16.0% NaCl	________ mL
Mass of unknown NaCl	________ g
Volume of unknown NaCl	________ mL

Useful Information: Density = mass/volume

calculate using Volume by Displacement

Data:
Density of solids

Mass of metal	18.256 g	____ g
Volume of water	20 mL	____ mL
Volume of water plus metal	26.8 mL	____ mL

Data:
Density of liquids

Mass of water (0% NaCl)	measured in class using analytical balance	7.685 g
Volume of water (0% NaCl)		8 mL
Mass of 4.0% NaCl	measured in lab class using scale use to make graph	7.799 g
Volume of 4.0% NaCl		8 mL
Mass of 8.0% NaCl		8.123 g
Volume of 8.0% NaCl		8 mL
Mass of 12.0% NaCl		8.251 g
Volume of 12.0% NaCl		8 mL
Mass of 16.0% NaCl		8.530 g
Volume of 16.0% NaCl		8 mL
Mass of unknown NaCl	use graph to determine which concentration the unknown is	7.986 g
Volume of unknown NaCl		8 mL

Results Solids:

Volume of metal	6.8 mL	mL
Density of metal	2.685 g/mL	g/mL
Average density of metal	g/mL	

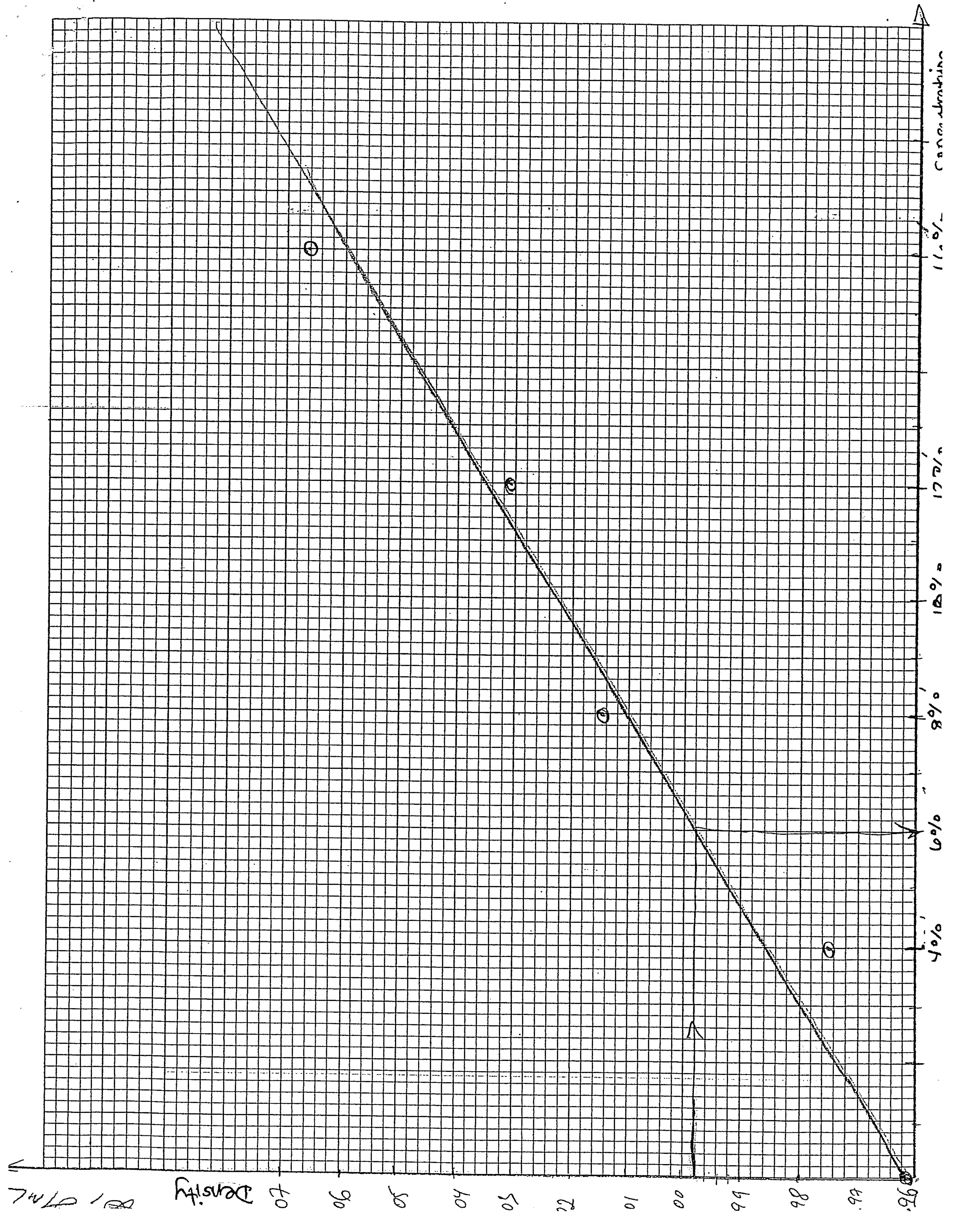
Results Liquid:

× **Densities:** $D = \frac{m}{v}$

Water (0%Nacl)	$D = \frac{7.685g\ H_2O}{8g} = 0.961$	0.961 g/mL
4.0% NaCl		0.975 g/mL
8.0% NaCl		1.015 g/mL
12.0% NaCl		1.031 g/mL
16.0% NaCl		1.066 g/mL
Unknown NaCl		0.998 g/mL

Concentration of unknown NaCl solution 6.0% %

[ ] is determined by graphing known plots of Density vs. Concentration. Line is determined and relative concentration of unknown is determined by plotting density and matching to determine concentration using line graph



Concentration
14%
12%
10%
8%
6%
4%
Density

Percentage Composition of MgO

Objective:

Prepare magnesium oxide and measure the percent magnesium and oxygen in the compound.

Procedure:

1. Strongly heat, cool, and weigh a crucible with lid.

2. Obtain about 0.4 g to 0.6 g of magnesium ribbon. Roll the magnesium into a loose ball. Place the magnesium ball in the crucible and measure the mass of the crucible, lid and magnesium.

3. Strongly heat the crucible and magnesium while holding the lid nearby with forceps.

4. At the instant the magnesium ignites, put the lid on the crucible momentarily. Although the magnesium oxide that is formed by the burning is a solid, it is very finely divided, and some will be lost as a white smoke unless the crucible is kept covered as much as possible.

5. After about 10-15 seconds, raise the cover about an inch to start the magnesium burning again, and then replace the cover.

6. Continue admitting air in this way at short intervals. The objective is to keep the magnesium burning at a slow rate and in such a manner that no MgO will be lost as white smoke. ~~When the contents no longer burn when the lid is raised,~~ cover about seven-eights of the opening of the crucible with the lid, and then heat the bottom of the crucible with a hot flame for five minutes.

7. If the combustion had been carried out in pure oxygen, only magnesium oxide would be produced.

$$2Mg + O_2 \rightarrow 2MgO$$

However, magnesium also reacts with nitrogen gas in the air producing a small amount of magnesium nitride.

$$3Mg + N_2 \rightarrow Mg_3N_2$$

In order to convert the magnesium nitride into magnesium oxide, proceed as follows. After the crucible has cooled, carefully crush the product into a powder with the end of a glass stirring rod, taking care to avoid loss. Any powder adhering to the rod may be tapped back into the crucible. Now moisten the product with about 20 drops of distilled water.

8. Reheat the crucible with lid in place, at first gently to avoid spattering, then strongly for five minutes. The overall reaction is:

$$\mathbf{Mg_3N_2 + 3H_2O \rightarrow 3MgO + 2NH_3}$$

The NH_3 and excess H_2O escape as a gas leaving only MgO in the crucible.

9. Cool and weigh the crucible, lid, and magnesium oxide product.

Useful Information:

Molar Mass Mg = 24.3 g
O = 16.0 g

Data:

1. Mass crucible and lid ____________g
2. Mass crucible, lid and Mg ____________g
3. Mass crucible, lid and MgO ____________g

Results:

4. Mass MgO product ____________g
5. Mass Mg in MgO product ____________g
6. Mass O in MgO product ____________g
7. Percent Mg in MgO product ____________
8. Percent O in MgO product ____________
9. Percent Mg expected from formula MgO ____________

10. Percent error ____________

In order to convert the magnesium nitride into magnesium oxide, proceed as follows. After the crucible has cooled, carefully crush the product into a powder with the end of a glass stirring rod, taking care to avoid loss. Any powder adhering to the rod may be tapped back into the crucible. Now moisten the product with about 20 drops of distilled water.

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$$Mg_3N_2 + 3H_2O \rightarrow 3MgO + 2NH_3$$

The NH_3 and excess H_2O escape as a gas leaving only MgO in the crucible.

9. Cool and weigh the crucible, lid, and magnesium oxide product.

Useful Information:

Molar Mass Mg = 24.3 g
O = 16.0 g

Data:

1. Mass crucible and lid 23.697 g
2. Mass crucible, lid and Mg 24.298 g
3. Mass crucible, lid and MgO 24.704 g

Results:

4. Mass MgO product 1.007 g #3 - #1
5. Mass Mg in MgO product 0.601 g #2 - #1
6. Mass O in MgO product 0.406 g #3 - #2
7. Percent Mg in MgO product 59.7% #5 ÷ #4
8. Percent O in MgO product 40.3% #6 ÷ #4
9. Percent Mg expected from formula MgO 60.3% mass of Mg ÷ mass of MgO — from Periodic Table

10. Percent error (-0.99%)

$$\frac{59.7\%\,Mg - 60.3\%\,Mg}{60.3\%\,Mg} \times 100\%$$

$$\frac{\#7 - \#9}{\#9} \times 100\%$$

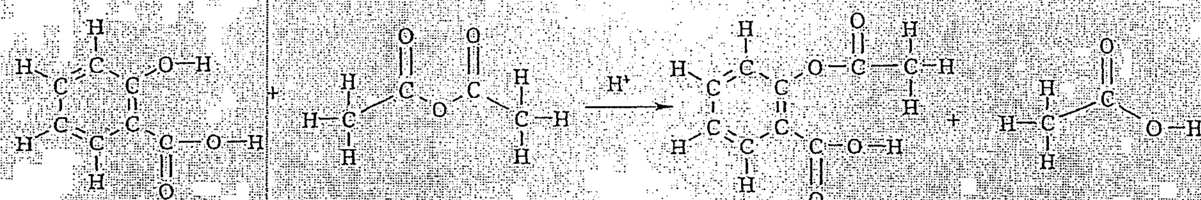
Synthesis of Aspirin

Objective:

To practice techniques in the preparation of Aspirin.

Background:

Aspirin can be made by treating salicylic acid with acetic anhydride in the presence of a catalyst. An excess of acetic anhydride will be used. This reaction is:



salicylic acid $C_7H_6O_3$ + excess acetic anhydride $C_4H_6O_3$ → aspirin $C_9H_8O_4$ + acetic acid $C_2H_4O_2$

Procedure:

1. Add 2 g of salicylic acid to a 250 mL beaker.
2. Add 6 mL of acetic anhydride followed by 5 to 10 drops of 85% phosphoric acid.
3. On a hot plate, warm the mixture gently for 10 minutes.
4. Remove the mixture from the hot plate and while still warm, slowly and cautiously add dropwise about 3 mL of water (~ 60 drops) to decompose the excess acetic anhydride.
5. Add 30 mL of water to the mixture, heat until clear, and then allow to cool in an ice bath.
6. Filter the aspirin crystals and wash twice with small amounts (5-10 mL) of cold water.

7. Dry the aspirin in an oven weigh and calculate the percent yield. Take melting point (optional).

Data:

Mass of salicylic acid	1.893 g
Mass of aspirin (actual yield, measured in lab)	2.094 g
Melting point	135 °C

Results:

Moles of salicylic acid	0.0137 moles
Moles of aspirin	0.0116 moles
Percent yield (aspirin $C_9H_8O_4$)	84.8%

$$\frac{2.094\text{g actual yield}}{2.469\text{g theoretical yield}} = 0.848 \text{ or } 84.8\%$$

$$\frac{\text{actual}}{\text{theoretical}} \times 100\%$$

Since the equation is 1:1 then the mole value should be close together for the results

Using chemical equation

$$1.893\text{g salicylic acid} \times \frac{1\text{mol salicylic acid}}{138\text{g salicylic acid}} = 0.01372 \text{ mol Salicylic acid}$$

$$0.01372 \text{ mols salicylic acid} \times \frac{1\text{mol aspirin}}{1\text{mol salicylic acid}} = 0.01372 \text{ mol aspirin} \times \frac{180\text{g aspirin}}{1\text{mol aspirin}} =$$

2.469 g aspirin (theoretical)

You can calculate % yield using these values

$$\frac{0.0116 \text{ moles Aspirin}}{0.0137 \text{ moles salicylic acid}} = 84.67\%$$

Percent $KClO_3$ in an Unknown Mixture

Objective:

A. To investigate the stoichiometry of potassium chlorate.

B. To determine the percent potassium chlorate in an unknown mixture containing potassium chlorate and sodium chloride.

Background:

In this experiment we will investigate the stoichiometry of the thermal decomposition of potassium chlorate.

Some chemical compounds, when heated, break down into other compounds and/or gases. One example is sodium hydrogen carbonate (bicarbonate) which breaks down to sodium carbonate, H_2O and CO_2 and is responsible for bread rising. Another is potassium chlorate which forms potassium chloride and O_2 gas, as shown by the following equation:

$$2KClO_3(s) \rightarrow 2KCl(s) + 3O_2(g)$$

From the balanced equation one sees that when 3 moles of oxygen gas escapes, 2 moles of potassium chlorate must have reacted.

To speed up the rate of the reaction, a small amount of manganese dioxide is used as a catalyst.

Procedure A:

1. Add a "pinch" of catalyst to a dry test tube.

2. Obtain the mass of a test tube plus catalyst.

3. Add about 1.5 g of pure potassium chlorate to the test tube and re-weigh.

4. Mix the catalyst and pure potassium chlorate, by shaking.
   Note: You will use the unknown mixture for this step in Procedure B.

5. Clamp the test tube to a stand at a 45° angle and gently heat for ~2 minutes.

6. Heat strongly for 5 minutes.

7. Re-weigh the test tube and residue.

Procedure B:

Repeat the above procedure using the unknown mixture of potassium chlorate and sodium chloride.

Data: Part A – Pure $KClO_3$

1. Mass test tube and catalyst — 22.183 g
2. Mass test tube, catalyst and pure $KClO_3$ sample — 23.246 g
3. Mass test tube, catalyst and residue after heating — 22.820 g

(KCl)

Results: Part A

1. Mass O_2 gas produced (#2 − #3) — 0.426 g
2. Moles O_2 gas produced $0.426 g\ O_2 \times \frac{1\ mol}{32 g\ O_2}$ — 0.0133 moles

} with heat

3. Moles O atoms in original sample $0.426 g\ O \times \frac{1\ mol}{16 g\ O}$ — 0.0266 moles
4. Mass KCl (residue) (#3 − #1) — 0.637 g
5. Moles KCl (residue) $0.637 g\ KCl \times \frac{1\ mol}{74.45 g\ KCl}$ — 0.00854 moles
6. Mole ratio of O atoms to KCl — 3 : 1
7. Simplest formula of potassium chlorate — $KClO_3$

$KClO_3$

$\frac{0.0266\ \text{moles Oxygen}}{0.00854\ \text{moles KCl}}$ → limiting reactant = 3.11 mol Oxygen

$\frac{0.00854\ \text{moles KCl}}{0.00854\ \text{moles KCl}}$ = 1 mol KCl

Data: Part B – $KClO_3$ Mixture

1. Mass test tube and catalyst ________ g

2. Mass test tube, catalyst and unknown mixture ________ g
   (unknown + $KClO_3$)

3. Mass test tube, catalyst and residue after heating ________ g
   (w/O Oxygen) → (KCl)

Results: Part B

1. Mass unknown (#2 − #1) 1.004 g

2. Mass O_2 gas produced (#2 − #3) 0.143 g

3. Moles O_2 gas in produced $0.143g \times \frac{mol}{32g\ O}$ 0.00447 moles

4. Moles $KClO_3$ in unknown $0.00447 mol\ O_2 \times \frac{2 KClO_3}{3 mol\ O_2}$ 0.00298 moles

5. Mass $KClO_3$ in unknown $0.00298 mol\ KClO_3 \times \frac{123.45 g\ KClO_3}{1 mol}$ 0.365 g

6. Percent $KClO_3$ in unknown 36.4%
   $\frac{\#5}{\#1} \times 100\%$

$$\frac{0.365\ g\ KClO_3}{1.004\ g\ unknown} \times 100 = 36.354\%$$