Lab 3. Analysis of Hydrated Sulfate Salts

Prelab Assignment

Before coming to lab:

- Use the handout "Lab Notebook Policy" as a guide to complete the following sections of your report for this lab exercise *before* attending lab: Title and Date of Lab, Introduction, Materials/Methods *and Data Tables*. Ensure that the table of contents of your lab notebook is current.
- Complete the Prelab questions on the last page and hand in on the instructor's table at the start of lab.

Part 1. Memo from Hydrates 'R Us Industries

To: Chem 161 Laboratories Inc, Auburn, WA
From: Hydrates 'R Us Industries
Re: Sample Analysis for Chemical Analysis Contract

As we discussed in our telephone conversation last week, our company is interested in contracting for quality control analysis of various materials and chemicals which we will be producing. It is of the utmost importance that we have confidence in the abilities of the independent laboratory undertaking these analyses to perform them at the highest levels of accuracy and precision. Therefore, before awarding this large and lucrative contract, we are asking each of the potential contractors to demonstrate their analytical and scientific aptitude by performing analyses on three samples provided by us. *All of the samples are hydrated sulfate salts, with the cationic species being an alkali metal in group 1A, an alkaline earth metal in group 2A, or a transition metal from the 4th period of the periodic table*.

As a measure of your analytical prowess, we ask that your laboratory determine, as accurately and precisely as possible, the mass percent of water present in each of the three samples we provide. Our assessment of your performance will be based, for the most part, on this determination.

We should note, however, that we are particularly impressed by the demonstration of initiative and capable scientific reasoning. Therefore, if your laboratory is able to determine the name and formula of any (or all) of the three compounds you are given, including a clear and cogent explanation of your determination, we will consider that to be worth some additional consideration in evaluating your overall performance.

We would be happy to receive your report on this project at your earliest convenience, but no later than in one week time.

Thank you,

A. Little Nass, C.E.O.
Hydrates 'R Us Industries

Part 2. InterLab Memo to Chem 161 Industries Inc, Auburn, WA

InterLab Memo:

As you can see from the attached letter, we have been given the task of determining the mass % of water in three unknown hydrate salts and to determine the name and formula of each.

Many salts that crystallize in nature do so with the accompaniment of a fixed number of water molecules to form a substance known as a hydrate. ***Hydrates*** are ionic compounds that contain a fixed percent by mass of water as part of their crystal structure. When most hydrated salts are heated, the water molecules escape and the brilliance of the crystal is lost as the salt lattice crumbles turning it into a powder.

The ***percent by mass of water*** in a hydrate is a fixed quantity and can be determined through a simple experiment. For example, if sufficient heat is added to the hydrate of sodium carbonate, $Na_2CO_3 \bullet 10H_2O$, the water of hydration will be driven off:

$$\mathbf{Na_2CO_3 \bullet 10H_2O_{(s)} + Heat \rightarrow Na_2CO_{3\,(s)} + 10\ H_2O_{(g)}}$$

In sodium carbonate decahydrate, 10 moles of water (i.e. 180.0 g H_2O) are bound per one mole of Na_2CO_3 (i.e. per 106 g Na_2CO_3). Hence, if the formula of the hydrate is known, the theoretical percent water in the hydrate can be calculated:

$$\frac{180.gH_2O}{180.gH_2O + 106.gNa_2CO_3} x100 = 62.9\%H_2O$$

In some hydrates, the water molecules are so tightly bound to the ions within the crystalline lattice they cannot be easily removed, no matter how intense the heat, e.g. $FeCl_3 \bullet 6H_2O$. This lab investigation involves hydrates where the water molecules are weakly associated within the crystalline lattice.

I would like each of you to get into your work groups and brainstorm on the following:

1. Prelab question #1 on page 5: Fill in the table for $CuSO_4 \cdot 5H_2O$. Explain your calculations and answers with your group until you have confirmed your answers and understand the concepts involved. Then complete the rest of the table and confirm your answers with your team members.
2. How can we experimentally determine the percent by mass water in each hydrate? The following points are some considerations to keep in mind as you go about devising a procedure.
3. What specific lab method can you devise to implement your proposal?
   - How much of the unknown should you use? What are the advantages and/or disadvantages of using a large sample? A small sample?
   - What apparatus should you use to heat the hydrate in?
   - How many times should you repeat the experiment?
   - How can you be certain that all of the water has been driven off?
   - What can you do to prevent the compound from reabsorbing water after it has been heated?

Notes:

- Each unknown will have a number or letter on it. Be sure to record each number or letter in your lab notebook.
- The amount of the unknown you receive will be all you will have to work with. Plan accordingly!
- NEVER weigh objects while they are hot. This will give an incorrect reading, and may also damage the scale.

After each group comes up with a working lab protocol please show it to me. At that point your group will be given an adequate sample of each salt to collect your data. Feel free to compare your data with other work groups. However, although each group will be given the same three samples, they may be labeled differently☹.

Written Report (in your lab notebook)

Each person will submit a report (about one week from today—the exact deadline T.B.A.) which should include the following sections:

1. **Introduction:** Indicate your goals and give an introduction to the project.
2. **Materials and Methods**
   - Note: This should be essentially what you have shown me from above and then refined to express what you actually did do in the lab.
3. **Results**
   - Please show ALL measurements and calculations clearly in a neat and well organized manner. Display all data in a ruled data table (numbered with a caption!). Use correct units and significant figures for all numerical data and calculations.
4. **Analysis of the Data.**
   - Calculate the % H_2O in each of the three hydrates to the maximum number of significant figures allowable. Show only *one sample calculation* in your notebook.
   - Use the % water for each hydrate, the information given in the memo from Hydrates 'R Us Industries and the *CRC handbook of Chemistry and Physics* to identify the name and formula of each hydrate. *Quote specific numerical data* to support all claims that are made and explain fully how you identified each of the three unknown hydrates.
   - Do you have any reason to suspect that some of your data is inaccurate? Why? Calculate the % Error for each of the three hydrates. What is(are) the most likely reason for the error(s) and how could these errors be minimized in the future?
5. **Conclusion**
   - Use bullets to summarize what you found, the % Error, the possible sources of error and how this addresses the question proposed by the client, *Hydrates 'R Us Industries*.

Error Analysis Questions

Consider the following questions when analyzing the results of this lab for sources of experimental error, but record your answers to these questions after the conclusion section of your report. Title this section of your report "***Error Analysis Questions***" and number your responses as the questions are numbered below. It should be clear from each answer what the question is you are answering—hence, "*incorporate the question into each answer*."

1. If the hydrated salt is not heated to a high enough temperature for a long enough period of time, will the *calculated* moles of water in the hydrated salt be too high, too low or unaffected? *Explain your reasoning*.
2. If the *salt* decomposes to yield a volatile product (other than water!), will the *calculated* value for the percent water be too high, too low or unaffected? *Explain your reasoning*.
3. Suppose you were in a hurry and weighed the crucible and the anhydrous salt while still very hot. Will the *calculated* value for the percent water be too high, too low or unaffected? *Explain your reasoning*. Hint: hot air is more buoyant than cooler air.

Sample Calculations

Rusty with moles?? Recall from your previous chemistry classes….

- A mole in chemistry is simply a number, albeit a very large one!!

  1 mole = 6.022 x 10^{23}

- The mass of one mole of a substance is the substance's formula mass expressed in grams. For example…
  - 1 mole of water molecules, H_2O, has a mass of 18.0 grams or 18.0 g H_2O/mole. i.e. **18.0 g H_2O = 1 mole H_2O = 6.022 x 10^{23} water molecules**

Formula of a Hydrate: Sample Calculation for a Hydrate of Magnesium Sulfate: $MgSO_4 \cdot ?H_2O$

A clean and empty crucible has a mass of 12.770 g. The crucible and hydrate have a mass of 15.895 g. After heating, the crucible and anhydrous salt have a mass of 13.889 g. What is the formula of this hydrate of magnesium sulfate, $MgSO_4 \cdot ? H_2O$?

Mass of hydrate =
(mass of crucible and hydrate) – (mass of crucible)
= 15.895 g – 12.770 g = **3.125 g hydrate**

Mass of anhydrous salt, $MgSO_4$ =
(mass of crucible and salt after heating) – (mass of crucible)
= 13.889 g – 12.770 g
= **1.119 g anhydrous salt, $MgSO_4$**

Mass of water, H_2O =
(mass crucible + salt before heating) – (mass crucible + salt after heating)

= 15.895 g – 13.889 g = **2.006 g H_2O**

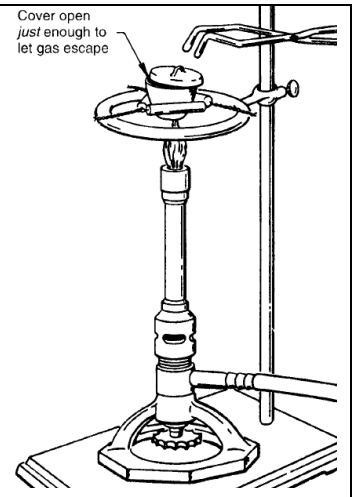

Figure 1. Method for heating a crucible

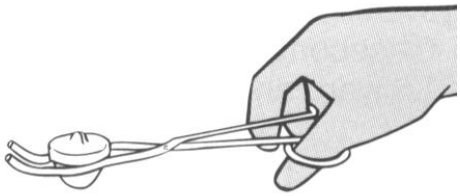

Figure 2. Using crucible tongs to hold a crucible.

Moles of anhydrous salt, $MgSO_4$ = 1.119 g $MgSO_4$ x $\frac{1 \text{ mol } MgSO_4}{120.37 \text{ g } MgSO_4}$ = **0.0092963 mole $MgSO_4$**

Moles of water = 2.006 gH_2O x $\frac{1 \text{ mol } H_2O}{18.0148 \text{ g } H_2O}$ = **0.111353 moles H_2O**

Ratio of moles H_2O to moles of $MgSO_4$ = $\frac{\mathbf{0.111353} \text{ mol } H_2O}{\mathbf{0.009296}3 \text{ mol } MgSO_4} = \frac{\mathbf{11.97}8 \text{ mol } H_2O}{1 \text{ mol } MgSO_4}$

This means that for every one mole of $MgSO_4$ there are 12 moles of H_2O. Therefore the formula of the hydrate of magnesium sulfate that was heated is **$MgSO_4 \cdot 12H_2O$**

Lab 3 Prelab Questions Name_____

Analysis of Hydrated Sulfate Salts Team ___Date _____Section ___

Instructions: Complete the following questions and hand in at the start of your lab period. Show your work with units and correct significant figures for all questions that involve a calculation. *Circle all numerical answers*.

1. Fill in the table below for $CuSO_4 \cdot 5H_2O$. Explain your calculations and answers with your group until you have confirmed your answers and understand the concepts involved. Then complete the rest of the table and confirm your answers with your team members.

Compound	Number of waters of hydration per formula unit	Molecular Weight of Compound (g/mol)	Mass of water in one mole of compound (g)	Mass NOT water in one mole of compound (g)	Mass percent of water in compound
$CuSO_4 \bullet 5H_2O$					
$Cu(NO_3)_2 \bullet 3H_2O$					
$NaH_2PO_4 \bullet H_2O$					
$Ca(NO_3)_2 \bullet XH_2O$					30.50

2. A 3.550 g sample of a hydrate of nickel (II) chloride was heated in a crucible until a constant mass was obtained. The mass of the anhydrous salt (i.e. the salt without water) after heating was 1.937g $NiCl_2$.
   a) Calculate the percent by mass of water in the hydrate. *Circle your answer.*

   b) Calculate the moles of H_2O removed by heating. *Circle your answer.*

   c) Calculate the moles of $NiCl_2$ left behind in the crucible after heating. *Circle your answer.*

   d) Use your answers to parts b and c to determine the formula of hydrate of nickel (II) chloride. *Circle your answer.*

2. Calculate the % water in cobalt (II) sulfate heptahydrate, $CoSO_4 \bullet 7H_2O$. *You must show your work clearly and with units to receive credit*. *Circle your answer.*