Investigating Hydrates: Formulas and Mass Percent Composition

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Purpose of the Experiment

- Understand how to calculate the mass percent of components of a chemical formula
- Experimentally determine the mass percent water in an unknown hydrate
- Be able to calculate percent error with correct significant figures

PreLab Preparation:

Complete the online prelab assignment and Lab Procedure Outline as your instructor assigned.

Background Information:

Formulas, Molar Mass and Mass Percent Composition

Chemical formulas tell us which elements are in a compound and how many atoms of each element are present. For example, the formula for copper (II) sulfate is $CuSO_4$.

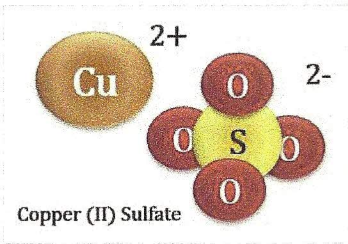
The formula tells us that each formula unit of $CuSO_4$ contains 1 copper atom, 1 sulfur atom and 4 oxygen atoms. The formula also tells us that *1 mole* of $CuSO_4$ contains *1 mole* of copper atoms, *1 mole* of sulfur atoms and *4 moles* of oxygen atoms. We can calculate the mass of one mole of $CuSO_4$ (called the molar mass) by multiplying the atomic mass of each element by the number of moles of that element in the compound and adding the results together:

$$1 \text{ mol Cu} \times \frac{63.55 \text{ g Cu}}{1 \text{ mol Cu}} = 63.55 \text{ g Cu}$$

$$1 \text{ mol S} \times \frac{32.07 \text{ g S}}{1 \text{ mol S}} = 32.07 \text{ g S}$$

$$4 \text{ mol O} \times \frac{16.00 \text{g O}}{1 \text{ mol O}} = \underline{64.00 \text{ g O}}$$

Molar Mass of $CuSO_4$ 159.62 g



Copper (II) Sulfate

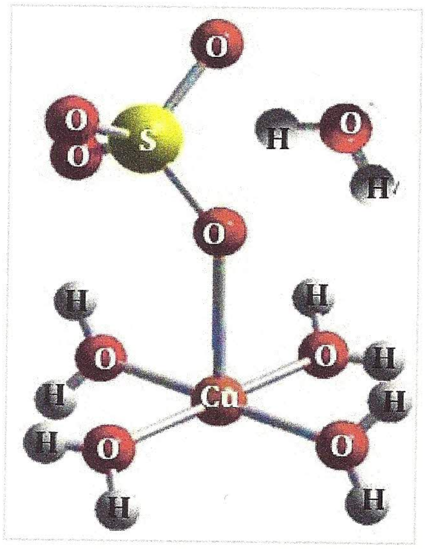
The percent by mass (or mass percent) of any component in a compound can be calculated by dividing the mass of the component in 1 mol of the compound by the molar mass of the compound, then multiplying the result by 100%. For example, there are 4 mol of oxygen in every mol of $CuSO_4$. The mass percent of oxygen in $CuSO_4$ is calculated by first determining the mass of oxygen in 1 mol of $CuSO_4$, then dividing the mass of oxygen by the molar mass of $CuSO_4$ and finally, multiplying the result by 100%:

$$4 \text{ mol O} \times \frac{16.00 \text{ g O}}{1 \text{ mol O}} = 64.00 \text{ g O} \qquad \frac{64.00 \text{ g O}}{159.62 \text{ g } CuSO_4} \times 100\% = 40.09\% \text{ oxygen}$$

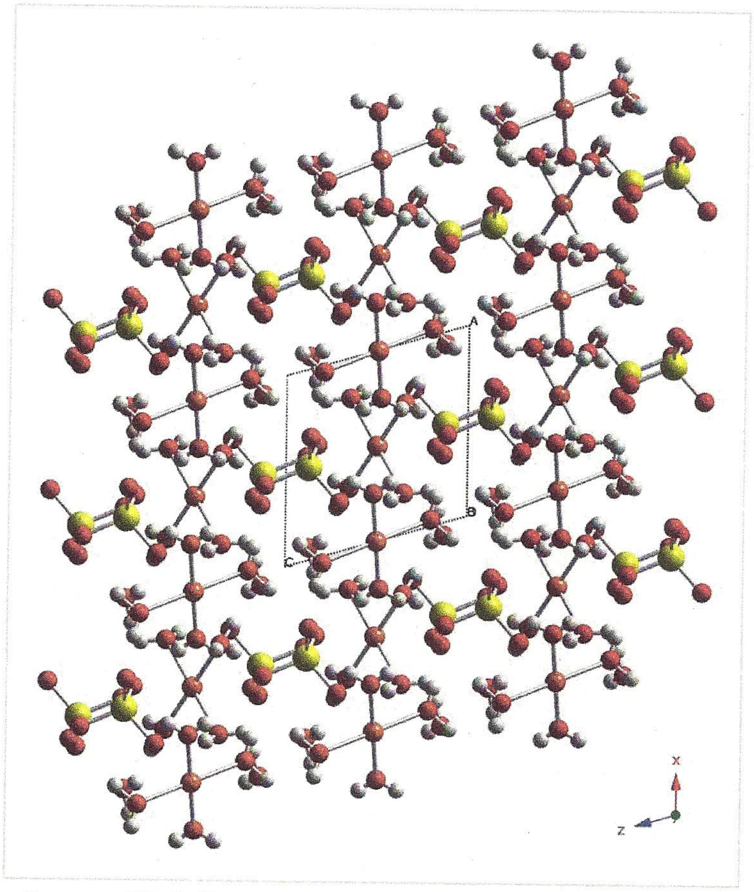
Remember, percent means "out of 100" or "parts per 100", so our mass percent calculation tells us that every 100.00 g of $CuSO_4$ contains 40.09 g oxygen. We will use this approach to calculating mass percent for determining the theoretical value for the mass percent water in a hydrate formula.

Ionic compounds and Hydrates

Many ionic compounds are found in crystalline form with a fixed mass percent of water molecules (H_2O) that are weakly bound in their crystalline lattice. The fixed percentage by mass also translates to a fixed number of water molecules per formula unit of the ionic compound. We call these compounds hydrates, to reflect the added water. Copper II sulfate forms a common hydrate by incorporating 5 water molecules for every $CuSO_4$ formula unit into its crystalline lattice as shown in the following figures.



Copper (II) Sulfate with 5 H_2O



Copper (II) Sulfate Pentahydrate Crystalline Lattice[1]

Hydrates of ionic compounds are named by using the name of the ionic compound, followed by a prefix to indicate how many water molecules per formula unit are incorporated into the lattice and the word hydrate. Thus, the hydrate formed by copper (II) sulfate and five water molecules is named copper (II) sulfate *penta*hydrate. The prefixes are the same counting prefixes used in naming molecular compounds (di, tri, tetra, penta, hexa, hepta, octa, nona, deca and so on). When writing the formula of a hydrate, we write the formula of the ionic compound and show the number of water molecules separately, separated by a dot ("·"). Using this approach, the formula for copper (II) sulfate pentahydrate is written as $CuSO_4 \cdot 5H_2O$.

The water molecules in a hydrate are only weakly bound within the crystalline lattice and can be easily removed by heating. This allows us to determine the mass of water in a sample of an unknown hydrate in a simple experiment. We first determine the mass of a sample of the hydrate, heat the sample to remove all of the water molecules and then weigh the remaining residue. We call this process "dehydration" and the $CuSO_4$ residue after heating is called the anhydrous or "without water" form of the compound. The difference between the starting mass of the hydrate sample and the remaining solid residue is the mass of the water lost during heating. The mass percent of the water in the hydrate is calculated as the mass of the water divided by the mass of the hydrate sample, then multiplied by 100%:

1. Start oven
2. Give keys to their drawer
3. Ask each partner to put their crucible in oven

$$\text{Mass percent water in the hydrate sample} = \frac{\text{mass of water}}{\text{mass of hydrate sample}} \times 100\%$$

We can show this dehydration process as a chemical reaction in which solid copper (II) sulfate pentahydrate is heated, releasing the water as vapor and leaving the anhydrous (*without water*) residue:

$$CuSO_4 \cdot 5H_2O(s) \rightarrow CuSO_4(s) + 5H_2O(g)$$

In this lab, we will use top-loading balances to measure the mass of our hydrate sample before and after heating and employ a Bunsen burner to heat the sample. By the end of this lab, you should be able to: record mass measurements with the correct number of digits, properly light a Bunsen burner, calculate the mass percent water in your hydrate sample from your experimental data, determine the theoretical mass percent of your hydrate from the given formula, and calculate percent error with correct significant figures.

Equipment:

Small ceramic crucible and lid	Ring stand with ring
Bunsen burner	Clay triangle
Top loading balance	Wire gauze
Crucible tongs	Unknown hydrate samples

Safety: Always wear your safety goggles while in the lab room. Use extreme caution with the Bunsen burners and only handle your hot crucible with *crucible tongs*. Avoid skin contact with the hydrate samples and wash your hands before leaving lab. Keep balance areas clean and carefully wipe up any spills with a damp paper towel.

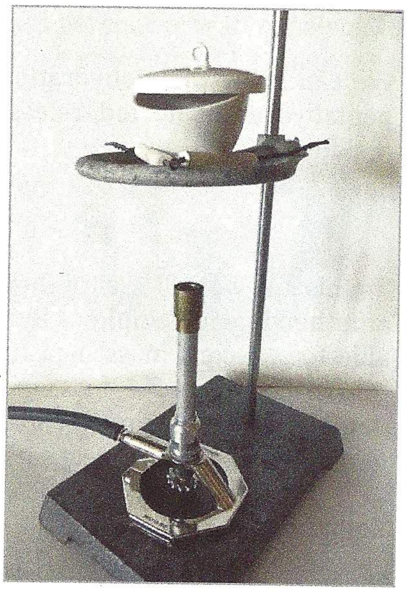
Procedure: (Work with a partner for data sharing, but each partner will perform their own dehydration experiment on their own sample of the same unknown hydrate.)

Before Lab Lecture:
Place a clean dry crucible (without the lid) into the drying oven to remove any residual moisture.

After Lab Lecture:
Preparation of Hydrate Sample

5. Retrieve your crucible from the drying oven using crucible tongs. Place crucible on a wire gauze and allow to cool to room temperature. The crucible is cool enough to weigh if you feel no heat when your hand is ~1 cm away from the crucible.
6. Once your crucible is cool, take your crucible (use tongs to avoid getting oils from your hands on the crucible) and lab manual to a top loading balance, zero the balance and weigh the empty crucible. Record this mass (all digits) as the "mass of empty crucible".
7. Leave your crucible on the balance and obtain an unknown. Using a metal scoop, slowly add small amounts of hydrate to your crucible until your crucible + hydrate mass is approximately 2 g higher than the mass of the empty crucible. Record this mass (all digits) as "mass of crucible and hydrate".

Heating the Hydrate



1. Obtain a ring support pole, ring, clay triangle and Bunsen burner.
2. Set up the equipment as shown in the figure. Your ring should be approximately 3.5 to 4 inches (use a ruler to estimate) from the top of the Bunsen burner (Check *before* lighting your Bunsen burner – the metal ring is too hot to adjust after your burner has been on!).
3. Using crucible tongs, place your crucible with hydrate sample on the clay triangle at a slight angle and gently put the lid in place.
4. Carefully adjust and then light your Bunsen burner as demonstrated by your instructor. Do not light the burner under the apparatus – always light the burner away from the apparatus and then move the burner under the ring. Adjust your flame to have a blue cone at its base and only a gentle "roar".
5. Heat crucible and sample gently for 5 minutes to avoid splattering as the water is vaporized.
6. After 5 minutes, adjust your burner by adding slightly more gas and oxygen, which will increase the flame temperature (slightly louder roar, tighter blue cone as the base of the flame). Heat for an additional 15 minutes.
7. While your sample is heating, obtain the formula for your unknown from your instructor and calculate the theoretical mass percent water based on the formula. You should also have time to work on Discussion Questions 2 – 6.
8. Once your heating is complete, turn off the gas to your burner and let the crucible cool on the clay triangle for 5 minutes. After 5 minutes, remove the crucible lid using tongs and transfer the crucible using crucible tongs to the wire gauze, replacing the lid completely onto the crucible. Let the crucible cool to room temperature. Do not touch the crucible to determine if it is cool. Instead, cup your hands around the crucible (but not touching it) to feel if heat is still being radiated.
9. Using crucible tongs, transfer the cooled crucible to the *same balance* you used before. Weigh the crucible and residue and record this mass (all digits) as "mass of crucible and residue".
10. Discard the crucible contents into the labeled waste container. The residue should come out easily. Clean your crucible and lid thoroughly.

Data, Calculations and Discussion

Record all data in the indicated spaces in the Data Table. Complete all calculations on the table and answer the discussion questions with your partner before leaving. Follow your instructor's directions for turning in the Data, Calculations and Discussion Questions pages.

Reference 1 (copper sulfate three-dimensional model figures):
https://commons.wikimedia.org/wiki/File:Copper%28II%29-sulfate-pentahydrate-CuOSOCu-chain-from-xtal-2007-CM-3D-balls.png (released into common domain by Benjah-bmm27):

Name____________________ **Partner**____________________ **#**_____

Data and Calculations

Identification Letter of Unknown Hydrate _____	**My value (units)**	**Partner's value (units)**
Mass of empty crucible (g)		
Mass of crucible and hydrate (g)		
Mass of crucible and residue (g)		
Calculation of <u>Experimental</u> Mass Percent Water (***Show set-up: numbers and units***)		
Mass of hydrate sample: (Mass of crucible and hydrate – mass of empty crucible)		
Mass of water: (mass of crucible and hydrate - mass of crucible and residue)		
Experimental mass percent water: $\left(\frac{\text{mass of water}}{\text{mass of hydrate sample}} \times 100\%\right)$		

Calculation of <u>Theoretical</u> Mass Percent Water from Formula (***Show set-up: numbers & units***)	
Formula of Hydrate (from instructor): $MX_a \cdot nH_2O$	
Calculation of molar mass of hydrate: *<u>Round atomic masses to 2 decimal places first</u>* *Molar mass = ∑(atomic mass of each element x # of atoms of element in compound)* $M + a \cdot X + n(2 \times 1 + 1 \times 16) =$ $M + a \cdot X + n \times 18$	
Theoretical mass percent water in the hydrate: $\left(\frac{\text{mass of } H_2O \text{ in 1 mol hydrate}}{\text{molar mass of hydrate}} \times 100\%\right)$ ~~wt of nH_2O~~ $= \frac{n \times 18}{[M + a \cdot X + n(18)]} \times 100$	

Calculation of Percent Error (Show set-up; *check sig figs*)	**My value**	**Partner value**
$\frac{(\text{experimental mass \% water} - \text{theoretical mass \% water})}{\text{theoretical mass \% water}} \times 100\%$		

Discussion Questions:

1. How does your mass percent water compare to your partner's result? Is their value close to yours (within +/- 1 %)? If not, discuss what happened during <u>your experiment</u> that may have caused different results.

2. Theoretically, should the experimental mass percent water values for everyone who used the same hydrate be approximately the same value? Why or why not?

3. <u>Error analysis</u>: A student spilled some of the hydrate when they set their crucible on the clay triangle. When the student calculates their mass percent water, will their value be higher or lower than the theoretical value? Explain your answer.

4. Name this hydrate: **$Fe(NO_3)_3 \cdot 9H_2O$**

5. Write the formula for this hydrate: **Cobalt (II) chloride hexahydrate**

6. **Lab Quiz Preview Question:** Calculate the mass percent water in an unknown hydrate using the data below. Show your set-up and express your answer with the correct significant figures.

Mass of empty crucible:	23.750 g
Mass of crucible and hydrate:	25.730 g
Mass of crucible and residue:	24.518 g