Experiment 14
Water in Hydrates

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Name:

Date:

Key Objectives

1. Calculate molecular weight of compounds.
2. Calculate percent composition of compounds.

Discussion

Certain ionic compounds form with large spaces in their crystalline structures as shown in Figure 14.1. These spaces are a perfect fit for many small molecules which are trapped there. When water is trapped in the lattice these compounds are often referred to as **hydrates**. The water which is chemically bonded to the hydrate is often called the **water of crystallization** or water of hydration. In a hydrate the water molecules are a distinct part of the compound and are joined to it by bonds that are significantly weaker than those forming the ionic lattice. To indicate this, the formula for hydrates is written with a dot used to separate the formula of the ionic salt from that of the trapped water molecules. The name of the compounds are slightly different also, the ionic part is named as usual, and we use indicate the additional water by giving the number of water molecules (using di, tri, etc.) and the word hydrate. For example $CaSO_4 \cdot 2\,H_2O$ would be called calcium sulfate dihydrate.

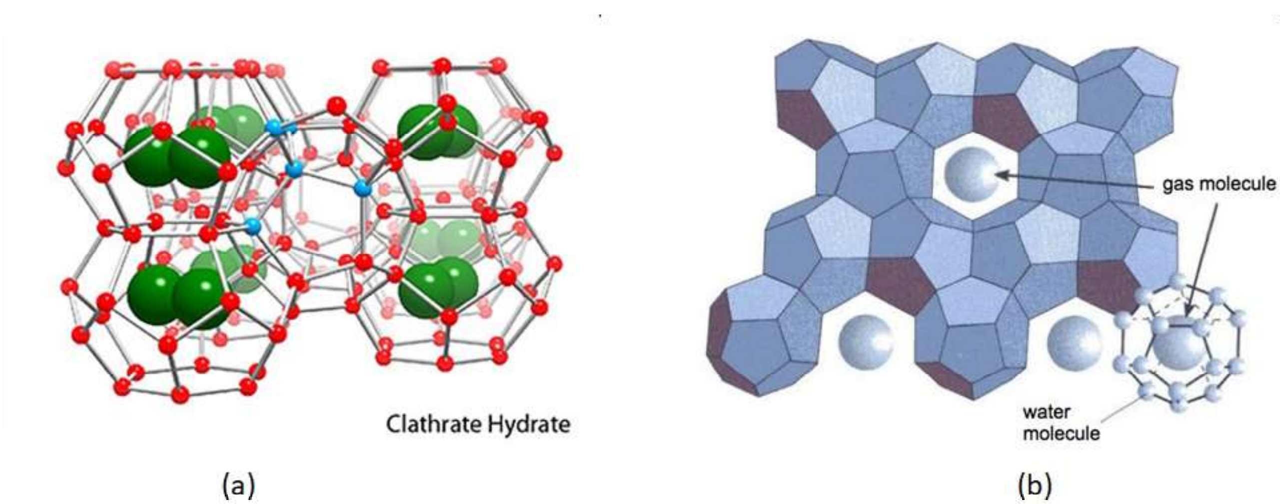

Figure 14.1: (a) and (b) Hydrates are crystalline compounds that have water trapped in the empty space within the crystalline lattice. Similar compounds shown here are chlathrate hydrate structures which are crystalline compounds in gas molecules are trapped in the spaces inside the crystal structure of frozen water. credit: (a) `https://ps.uci.edu/group/kcjanda/research/gas-hydrate-structure` (b) `http://dusk.geo.orst.edu/oceans/deep_currents.html`

These compounds have many industrial uses, as well possibly playing an important role in environmental issues such as global warming. We will briefly discuss two of the many uses for these types of compounds.

Natural gas hydrates (also commonly referred to as: clathrates, gas hydrates, methane hydrates) are solid compounds formed from small gas molecules (natural gas/methane), and water that are formed at low temperatures and high pressures. An example of a clathrate is shown in Figure 14.1. Some estimates predict that the total amount of gas hydrates could be as large as all the known reserves of coal, oil and gas combined, and could last for 1,000 years at current rates of use. Clathrates are crystalline water based solids physically resembling ice, in which small non polar molecules (typically gases) are trapped inside "cages" of hydrogen bonded water molecules. Without the support of the trapped molecules, the lattice structure of hydrate clathrates would collapse into conventional ice crystal structure or liquid water. Most low molecular weight gases (including O_2, H_2, N_2, CO_2, CH_4, H_2S, Ar, Kr, and Xe), as well as some higher hydrocarbons and freons will form hydrates at suitable temperatures and pressures. Clathrate hydrates are not chemical compounds as the sequestered molecules are never bonded to the lattice.

Hand warmers also make use of crystalline compounds with trapped molecules in them. Solid sodium acetate trihydrate ($NaC_2H_3O_2 \cdot 3\,H_2O$) when heated forms a solution of sodium acetate dissolved in the water which was trapped in the solids lattice. This solution is super-saturated and when disturbed will reform the solid compound liberating large quantities of heat. The compound makes excellent, reusable hand warmers because the reaction can be cycled many times. We can write a chemical equation for the reaction as:

$$\mathrm{NaC_2H_3O_2 \cdot 3\,H_2O(s) + Energy \xrightarrow[\text{Boiling Water}]{\text{Heator}} NaC_2H_3O_2 + 3H_2O(l)} \tag{40}$$

The reaction is of course reversible if water is added to the anhydrous sodium acetate, crystalization occurs as the water is absorbed into the solid lattice resulting in an exothermic reaction occurring which releasing energy/heat (i.e. a hand warmer).

$$\mathrm{NaC_2H_3O_2 + 3H_2O(l) \xrightarrow{\text{Crystallization}} NaC_2H_3O_2 \cdot 3\,H_2O(s) + Energy} \tag{41}$$

(a) (b)

Figure 14.2: (a) Cold hands? (b) Get some mittens and a reusable hot pack! credit: (a) `https://www.pxfuel.com/en/free-photo-qtjlg` (b) `https://commons.wikimedia.org/wiki/File:Handwaermer12.jpg`

Calculating Molecular Weight and Percent Composition

In the quantitative section of the lab you will measure the mass of water lost by a hyrdated sample upon heating. Using this information you will be able to determine the percentage of water in the sample, and compare it to several known hydrates to determine the identity of an unknown hydrate. In order to do so we will need to calculate the molar mass or molecular weight of each compound and the percentage of water in each compound.

The molar mass/molecular weight of a compound is the atomic mass of all the elements in the compound expressed in grams/mol. For example, the molecular weight of NaCl is the sum of the molar masses of Na and Cl which is 22.99 g/mol and 35.45 g/mol for a total of 58.44 g/mol. For a more complicated example we can calculate the molecular weight of $CaSO_4 \cdot 2\,H_2O$.

Element	# Atoms	Molar Mass (g/mol)	Total Mass (g/mol)
Ca	1	40.08	1(40.08) = 40.08
S	1	32.07	1(32.07) = 32.07
O	6	16.00	6(16.00) = 96.00
H	4	1.008	4(1.008) = 4.032
$CaSO_4 \cdot 2\,H_2O$	-	-	172.2

Figure 14.3: Example of calculating the Molecular Weight (MW) of a compound.

The percent composition of a compound is found by dividing the total mass of each element by the total mass of the compound and multiplying by 100. An example calculation for $CaSO_4 \cdot 2\,H_2O$ in Figure 14.4.

$$\frac{\text{MW of element}}{\text{MW compound}} \times 100$$

Element	# Atoms	Molar Mass (g/mol)	Percent Composition
Ca	1	40.08	$\frac{1(40.08)}{172.2} \times 100 = 23.28\%$
S	1	32.07	$\frac{1(32.07}{172.2} \times 100 = 18.63\%$
O	6	16.00	$\frac{6(16.00)}{172.2} \times 100 = 55.76\%$
H	4	1.008	$\frac{4(1.008)}{172.2} \times 100 = 2.341\%$
$CaSO_4 \cdot 2\,H_2O$	-	-	100.0%

Figure 14.4: (a) Formula for calculating percent composition of an element in a compound, (b) Table showing example calculation for $CaSO_4 \cdot 2\,H_2O$.

If we want to know the percentage of water in the compound than we need to take the molar mass of the water in the compound divided by the total molar mass as shown in the equation below.

$$\frac{\text{total mass of the } H_2O}{\text{total molar mass}} \times 100$$

For $CaSO_4 \cdot 2\,H_2O$ the equation would be:

$$\frac{36.03 \text{ g/mol } H_2O}{172.2 \text{ g/mol } CaSO_4 \cdot 2\,H_2O} \times 100 = 20.92\ \%\ H_2O$$

Procedure

This experiment will consist of two parts, heating a hydrated compound to remove the trapped molecules inside from the lattice leaving us with an anhydrous salt (the compound without water) and an unknown liquid. We will then attempt to characterize the liquid component of the hydrate and prove that it is indeed water. In the second part of the experiment we will quantitatively determine the percentage of water trapped in an unknown hydrate.

Part 1 - Qualitative Determination of Water

In this part of the experiment our goal is to place a finely ground sample of copper (II) sulfate pentahydrate in the bottom of a test tube without having any stick to the sides of the test tube. We will then heat the bottom of the test tube slowly in order to free the molecules trapped in the lattice of the copper (II) pentahydrate. The resulting gas will condense on the sides of the test tube and drip into the receiving test tube. We will then perform a series of tests on the collected liquid to prove it is water.

1. Obtain a medium sized test tube, a ring stand, clamp, erlenmeyer flask, and a bunsen burner.
2. Fold a 2.5 by 20 cm strip of paper lengthwise to form a V-shaped trough or chute.
3. Weigh about 3 grams of copper (II) sulfate pentahydrate and spread it evenly along the length of the trough. Assemble the apparatus as shown below in Figure 14.7. Clamp the test tube so that its mouth is 15-20 degrees above the horizontal. Carefully place the loaded trough containing the copper (II) sulfate pentahydrate into the bottom of the test tube. Tap the paper chute gently if needed, being careful not allow any copper (II) sulfate pentahydrate to adhere to the sides of the test tube.
4. Remove the chute and turn the test tube until it slants downward at an angle of 15-20 degrees below the horizontal (Figure 14.7c). Make sure the copper (II) sulfate pentahydrate does not slide downward, and remains at the bottom of the tube. Place a test tube in an erlenmeyer flask t0 catch the liquid that will condense in the test tube as it is heated.
5. Heat the hydrate gently at first, to avoid excessive splattering, by waving the bunsen burner underneath the solid sample. Gradually increase the rate of heating. Note any changes that occur in the results section.
6. Continue heating and collect the liquid which will condense in the cooler part of the test tube.
7. Heat the sample until the original blue color has disappeared, but **do not** overheat the sample causing the residue to turn black. (At excessively high temperatures (above 600℃) copper (II) sulfate decomposes into sulfur trioxide which is driven off as a gas, and copper (II) oxide which is a black solid.)
8. It may be necessary to heat the entire length of the test tube for a minute or two to drive most of the condensing liquid down the test tube and into the collecting test tube. Be sure to not heat the clamp holding the test tube in place.
9. Allow the test tube and its contents to cool.

(a) (b)

Figure 14.5: (a) Use a folded sheet of paper to place the sample in the test tube being careful not to spill. (b) Heating the test tube to drive off the trapped water, be sure to not heat by the clamp. credit: author.

10. Observe and record the odor of the liquid that has been collected.

11. While the tube is cooling, dry a piece of cobalt chloride test paper by holding it about 25 cm above the bunsen burner. Heat the paper but do not char or ignite it. When properly dried the test paper should be a blue color. Using a clean stirring rod, place a drop of the collected liquid on the dried cobalt chloride test paper. Record your observations

12. For comparison, place a drop of distilled water on a second piece of dried cobalt chloride test paper. Record your observations.

13. Empty the anhydrous salt residue onto a watch glass, and divide it into two portions.

14. Place one portion in a test tube, and carefully add 3-4 drops of the liquid collected earlier to the anhydrous salt. Record your observations.

15. Place the second portion into a test tube, and carefully add 3-4 drops distilled water to the anhydrous salt. Record your observations.

16. Dispose of the solid residue in the waste container labeled “Copper Waste” or “Cu Waste”.

Part 2 - Quantitative Determination of Water in a Hydrate

To insure that all of the water in the sample has been driven off, we will use a technique known as **heating to constant weight**. Essentially, we will heat and weight the sample until the mass of the sample does not change between heatings indicating that all of the molecules trapped in the lattice have been driven off. Due to limited time, constant weight in this experiment will be achieved when the sample is heated and weighed in successive trials until the weight differs by no more than 0.05 grams between two weighings. This may take 2-3 trials to occur.

An example would be if a student made several heatings as shown in the table below. He would only be able to stop at the last heating because the difference between the 3rd and 4th trials was less than 0.05 grams.

Trial	Initial Mass of Sample	Difference in Mass	Conclusion
Initial Weight	4.000 g	-	Start heating
Mass after 1st Heating	3.500 g	-	Always do a 2nd heating
Mass after 2nd Heating	3.250 g	0.250 g	$0.250 > 0.05$ therefore keep heating
Mass after 3rd Heating	3.150 g	0.100 g	$0.100 > 0.05$ therefore keep heating
Mass after 4th Heating	3.125 g	0.025 g	$0.025 < 0.05$ therefore we are done!

Figure 14.6: An example of heating to a constant mass. Only when the difference in mass between two trials is less than 0.05 is the sample considered dry.

1. Obtain a sample of an unknown hydrate from your instructor. Record its identity in the results section.

2. Obtain a crucible and cover from your instructor. Clean and dry it. Record the mass of the crucible and cover.

3. Place between 2 and 3 grams of your unknown into the crucible. Place the cover on top and weigh the crucible + sample.

4. Obtain a bunsen burner, ring stand, ring clamp, clay triangle, tongs, and construct the apparatus shown in Figure **??**.

5. Place the covered crucible on the clay triangle and adjust the cover so that it is slightly ajar to allow the water vapor to escape.

6. Heat the crucible very gently for around 5 minutes by waving the flame gently underneath. Be careful not to splatter the sample.

7. After 5 minutes readjust the flame so that a sharp inner-blue cone is formed. Place the bunsen burner underneath the sample and heat for 12 minutes with the tip of the inner-blue cone just touching the bottom of the crucible. The crucible bottom should be come a dull red color during this period.

8. Recall "Hot things are hot!" Handle the crucible with the crucible tongs. It will prove necessary to remove the cover first and place it on a ceramic pad. Then place the crucible on the pad, and place the cover on it again.

9. After the first heating is completed, close the cover and allow the crucible to cool for 10 minutes (or longer if required).

10. **The crucible should be cool to the touch before placing it on the scale to weigh it.**

11. Weigh the cooled crucible.

12. To determine if all of the water in the sample was removed during the initial heating, reheat the covered crucible and sample for an additional 5 minutes at maximum heat. Allow the sample to cool for 10 minutes (or longer if required) and weight the sample. The results of the last two

weighing should agree within 0.05 grams. If the decrease in mass is larger than this, repeat the heating and weighing until the results of two successive weighings are within 0.05 grams of each other.

13. Calculate the percentage of water in your sample on the basis of the final weight measured.

14. Perform 2 trials if time permits (it should) and average the results.

15. Dispose of the solid residue in the waste container labeled "Unknown Heavy Metal Waste" or ''Unknown Hydrate Waste".

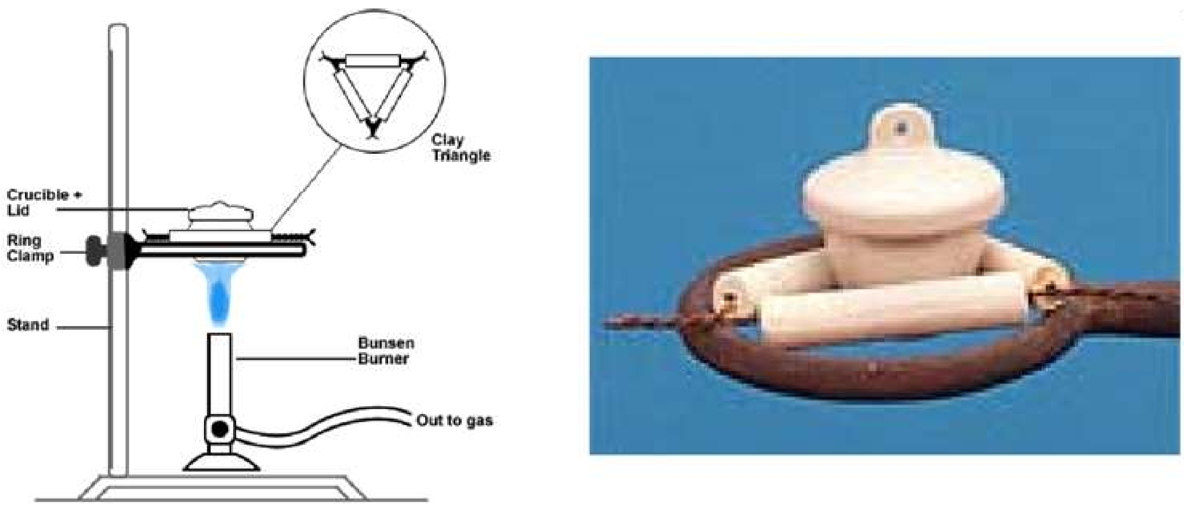

Figure 14.7: Setup for heating a crucible. Leave the cover ajar, to allow gas to escape but larger particles can not. credit: " The Composition of Potassium Chlorate (Experiment)" by Santa Monica College, LibreTexts is licensed under CC BY-NC .

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Name: ______________________ Class: ________________ Date: ________________

Results

A. Qualitative Determination of Water in a Hydrate

Property Observed	Before Heating	After Heating
Describe the appearance of the solid:		
Property Observed	**Unknown (Collected) Liquid**	**Water**
Describe the appearance of the liquid:		
Describe the odor of the liquid:		
Property Observed	**Unknown (Collected) Liquid**	**Water**
Original Color of Cobalt Chloride Paper:		
Color after adding liquid:		
Temperature change of anhydrous solid:		
Color change in anhydrous solid after adding liquid:		

1. What evidence did you see that indicated the liquid obtained above from the copper (II) sulfate pentahydrate was water. (2 answers)

2. What evidence of a chemical reaction occurring was observed when the unknown liquid, and distilled water were added to the anhydrous salt. (2 answers)

B. Quantitative Determination of Water in a Hydrate

		Trial 1	Trial 2
1.	Unknown Identity:	______	______
2.	Mass of crucible and cover:	______	______
3.	Mass of crucible, cover and sample:	______	______
4.	Mass of crucible, cover, and sample after 1^{st} heating:	______	______
5.	Mass of crucible, cover, and sample after 2^{nd} heating:	______	______
6.	Mass of crucible, cover, and sample after 3^{rd} heating:	______	______
7.	Mass of the original sample (show calc.):	______	______
8.	Total mass lost by the sample during heating (show calc.):	______	______
9.	Percentage of water in the sample (show calc.):	______	______
10.	Average percentage of water in the sample (show calc.):	______	
11.	What is the chemical formula of your unknown:	______	

Explain how you identified your unknown:

Post Lab Questions

1. Why do we want to avoid getting any copper (II) sulfate pentahydrate onto the sides of the test tube when we heated the sample to collect the molecules trapped inside the lattice?

2. Write a complete (balanced, states etc) chemical equation for the decomposition of copper (II) sulfate pentahydrate assuming it decomposes into copper (II) sulfate and dihydrogen monoxide.

3. Write the complete (balanced, states etc) chemical reaction (including states) for the decomposition of copper (II) sulfate at high temperatures.

4. When the unknown was heated, could the decrease in mass have been partly due to the loss of some other substance than water? Explain.

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Name: ______________________________ Class: ______________________ Date: ______________________

Prelab Questions

1. Complete the following table by filling in the missing name, formula, and molecular weight of the hydrates. Show work for one of the compounds on the back of the page.

Name	**Formula**	**Molecular Weight (g/mol)**	**% Water**
Calcium sulfate dihydrate		172.1816	
	$CoCl_2 \cdot 6\,H_2O$		
	$MgSO_4 \cdot 7\,H_2O$		
Sodium carbonate decahydrate		286.148	
Barium chloride dihydrate		244.2616	
	$CuSO_4 \cdot 5\,H_2O$		
	$SrCl_2 \cdot 6\,H_2O$		

2. A student heated a hydrated salt sample with an initial mass of 4.8702 grams. After the first heating the mass had decreased to 3.0662 grams. If the student is using the method of **heating to a constant weigh** described in this experiment, what is the minimum mass that the sample can have after the second heating to avoid having to heat it a third time? Explain.

2. ______________

3. The student in the previous problem determined that the mass lost by the sample was 1.8053 grams. What was the percent water in the original hydrated sample? Explain.

3. ______________

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