9.1 Dehydration of Hydrates – Pre-Lab Questions

Name: ____ Date: ___________________

Instructor: ______________________________________ Section/Group: ____________

Show all work for full credit.

1. Complete the following table. For each of the hydrates: 1) write the chemical formula; 2) calculate the formula mass; and 3) calculate the mass percentage of water.

Hydrate	**Chemical Formula**	**Fomula Mass**	**Mass % Water**
Nickel(II)chloride hexahydrate			
Cobalt(II)chloride hexahydrate			
Copper(II)sulfate pentahydrate			

2. A student dehydrated an unknown hydrate. The mass of the compound was initially 4.13 g. After heating, the mass was 3.52 g.

   a. Calculate the mass percentage of water in the hydrate.

   b. The table below lists the possible hydrates that could be your unknown. Complete the table.

   c. Compare the mass% values to determine the identity of your unknown.

   d. Unknown Identity__________________________.

Hydrate	Mass Salt (g)	Mass Water (g)	Mass Percent (%)
$CuSO_4 \cdot 5H_2O$			
$BaCl_2 \cdot 2H_2O$			
$MgSO_4 \cdot 7H_2O$			

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9.2 Dehydration of Hydrates – Introduction

Hydrates are compounds that have one or more water molecules integrated into their structure. They provide a good way to study the concepts of Mass % and Molar Ratios.

Relevance

Hydrates and desiccants have many uses in our day-to-day lives. You probably have several desiccants at home right now. Those little white packets that feel like a bag of rocks that you find in medicine bottles or new shoe boxes contain desiccants that re-absorb water from the air. These packets are also added to electronics, optical devices (such as camera lenses), and many other moisture-sensitive items. In chemistry, we often use cheap desiccants to keep other more expensive or volatile chemicals from absorbing moisture from the air. This is particularly important for chemicals that undergo explosive reactions when coming into contact with water. So, what are desiccants and how are they formed?

Background

Hydrates and Desiccants

Hydrates are ionic crystalline solids (salts) that contain a fixed number of water molecules integrated into their crystalline structure. Several examples include sodium carbonate decahydrate ($Na_2CO_3•10H_2O$), also known as washing soda; magnesium sulfate heptahydrate ($MgSO_4•7H_2O$), commonly called Epsom salt; and calcium sulfate dihydrate ($CaSO_4• 2H_2O$), which is known as gypsum. Another common hydrate is copper(II) sulfate pentahydrate, which contains 5 moles of water per 1 mol of copper(II) sulfate, written as $CuSO_4•5H_2O$.

When writing the formula of a hydrate, a dot (•) is placed between the ionic compound and the water molecules associated with it. The dot should be read as an addition sign when calculating the formula mass of the compound.

For example: The formula mass of $CuSO_4$ is 159.61 g/mol and the total molar mass of 5 water molecules is 90.08 g/mol. The formula mass of $CuSO_4• 5H_2O$ is therefore 159.61 g/mol + 90.08 g/mol = 249.69 g/mol. The formula mass of a hydrate always includes the mass of the water molecules that are an integral part of the formula.

Desiccants

When the water molecules found in a hydrate are removed either by exchange with a drier atmosphere or by heating, an **anhydrous salt** is formed. (*Anhydrous* means "without water".) The formula of the salt remains the same; it has simply lost the water portion of its formula. Some anhydrous salts are capable of becoming re-hydrated with exposure to the moisture in their surroundings. These salts are called **hygroscopic** and can be used as chemical drying agents or **desiccants**. Some salts are extremely hygroscopic and can absorb so much moisture from their surroundings that they can eventually dissolve themselves. These salts are called **deliquescent**.

The process of removal of water from a hydrate and addition of water to a desiccant can be written in the form of a reversible reaction:

$$CuSO_4•5H_2O(s) + heat \quad CuSO_4(s) + 5H_2O(l)$$

Copper(II)sulfate pentahydrate has a blue crystalline structure and the anhydrous or desiccant form is a white powder.

Figure 1. Copper(II)sulfate pentahydrate.

If water is added to the white anhydrous copper sulfate, the blue color returns indicating that the pentahydrate has been regenerated. True hydrates display this property of reversibility. Other compounds that react when heated to produce water are not hydrates if they are unable to reabsorb the water they have lost.

Sample Problem for Calculating the Formula of an Unknown Hydrate

Sample Problem

A 13.22 g sample of an unknown copper(II)hydrate, $CuSO_4 \cdot xH_2O$, was heated in a crucible to drive off the water. The mass was reduced to 8.44 g. What is the formula of the hydrate?

Problem Solution

Step 1. Determine mass of water driven off.

13.22 g initial mass – 8.44 g final mass = 4.78 g of water released

Step 2. Determine moles of $CuSO_4$ and water:

Moles of $CuSO_4$: $8.44\ g \times \frac{1\ mole}{159.61\ g} = 0.0529\ mol$

Moles of H_2O: $4.78\ g \times \frac{1\ mole}{18.02\ g} = 0.265\ mol$

Step 3. Find a whole number molar ratio by divide by the smallest number of moles (0.0529 mol $CuSO_4$).

$CuSO_4$ 0.0529 mol / 0.0529 mol = 1

H_2O 0.265 mol / 0.0529 mol = 5.01 (round to 5)

Therefore, x = 5 and the formula is $CuSO_4 \cdot 5\ H_2O$

Mass Percent

In order to determine the number of water molecules integrated into a hydrate's structure, a unit of concentration that is independent of temperature must be used. Mass percent does not change with temperature or pressure so it is the ideal unit of concentration for the dehydration-rehydration process. **Mass percent** is defined as the mass of a species (atom, compound, solute, etc.) divided by the total mass of the system (compound, hydrate, solution, etc.) multiplied by 100%.

$$\text{mass\%} = \frac{\text{mass of species for consideration}}{\text{total mass}} \times 100\%$$

For example, in the compound $Mg(BH_4)_2$ the mass percentage of Mg would be:

$$\text{mass\% (Mg)} = \frac{\text{mass of Mg}}{\text{mass of Mg(BH}_4)_2} = \frac{24.31\text{g}}{54.01\text{g}} = 0.45 \times 100\% = 45\%$$

The Experiment

A calculation similar to the one above will enable you to determine the identity of an unknown hydrate by determining the mass % of water contained in its formula. The formulas for several known hydrates are given in your Pre-lab. You will need to calculate the mass %of water in each of these hydrates to act as a reference guide for your experiment. In lab, you will be issued an unknown hydrate and will be required to remove all of the water by heating. By measuring the initial mass of the fully hydrated compound and then re-weighing the fully dehydrated desiccant, the mass of water in the hydrate can be determined. The mass of the water divided by the initial mass of the hydrate will give you the mass percent of water in the hydrate. Comparison of your experimental results to the reference information you produced in your Pre-lab should allow you to identify the unknown.

9.3 Dehydration of Hydrates – Procedure

Safety Notes: Some of the unknown hydrates can discolor skin, so avoid contact. Others can be eye irritants, so be sure to wear your goggles at all times. Remember that hot objects look just like cold objects, so be very careful picking up heated objects. If there is any trouble with the Bunsen burners, ASK YOUR INSTRUCTOR FOR HELP. It is better to be absolutely sure than cause an accident. Be sure to wash your hands thoroughly before leaving.

Part I: Preparing the Crucible

1. Obtain a crucible and lid.
2. Clean and dry the crucible and inspect for any stress cracks. *Note: Replace any crucible that displays a crack, as it will be unsafe to use*.
3. If the crucible is dirty, clean it with distilled water and gently evaporate to dryness.
4. Obtain a ring stand, ring clamp, and clay triangle and assemble as shown in Figure 2.

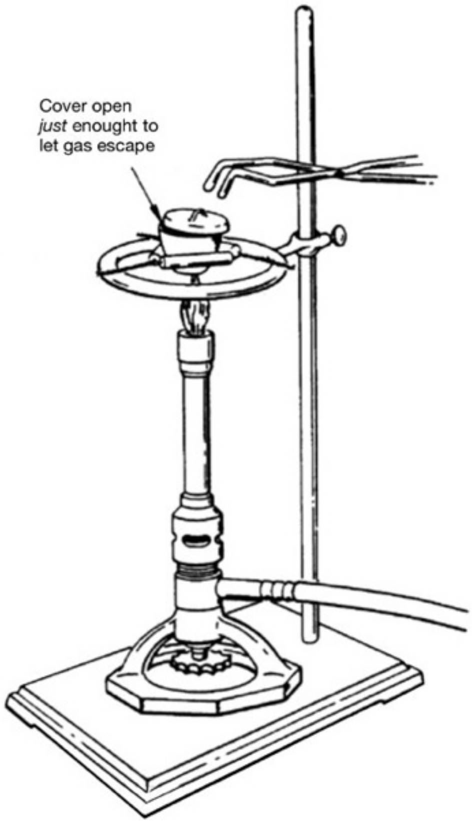

Figure 2. Dehydration set-up.

5. Record an initial mass of the crucible and lid.

6. Set the crucible in the triangle as shown; add the lid and gently heat the crucible for 5 minutes.

7. Increase the flame's intensity and heat the crucible for ~10 minutes once the bottom glows red.

8. After 10 minutes, turn off the heat and allow the crucible to cool, while remaining on the clay triangle. *Caution: Do not set the crucible on the bench as it may cause the crucible to crack or pick up contaminants*.

Part II: Initial Mass Measurements

1. Weigh the cooled crucible and lid on an analytical balance and record the mass to 0.001 g.

2. Collect an unknown hydrate sample. *Note: Be sure to record the code of the unknown in your lab notebook.*

3. Add between 1.50 and 2.50 grams of your unknown to your crucible. Record the total mass to 0.001 g.

4. Record any initial observations of the structure, color, etc. of the hydrate in your lab notebook.

Part III: Dehydrating the Hydrate

1. Return the crucible with the unknown sample to the clay triangle support.

2. Position the crucible lid off to the side to allow the evolving water molecules to escape during heating.

3. Start warming the crucible at a low heat and gradually intensify to a high-intensity flame. Heat the crucible for at least 20 minutes on high.

4. Cover the crucible once the heat is removed and allow it to cool to room temperature.

5. Reweigh the crucible, lid, and anhydrous salt using the same balance as used in the earlier steps. Record the mass to 0.001 g.

6. Reheat the sample for an additional 5 minutes with medium/high heat, cool, and re-weigh.

7. Repeat this process until you obtain two concurrent masses that are within 0.01 g of each other.

Part IV: Rehydrating the Hydrate

1. Prior to disposing of the now anhydrous salt, sprinkle a few drops of water onto the "powder" and record your observations in your lab notebook.

Part V: Repeat the Experiment

1. If time allows, repeat this procedure with a new sample of the original hydrated unknown.

Clean-Up

The dried hydrates should be disposed of in the waste container labeled hydrates. Rinse the emptied crucible with 2-3 mL of 1 M HCl and discard in the appropriate waste container. Rinse the crucible three times with tap water, once with deionized water, and return to the appropriate location. Wash your hands before leaving as usual.

9.4 Dehydration of Hydrates – Data Sheet

Name: ______________________________ Date: ______________

Instructor: ______________________________ Section/Group: __________

Unknown Hydrate Code	________________

Initial observations of your unknown:

Initial mass crucible + lid	________________
Initial mass crucible + lid + unknown	________________
1st heating: mass crucible + lid + unknown	________________
2nd heating: mass crucible + lid + unknown	________________
3rd heating: mass crucible + lid + unknown	________________
4th heating: mass crucible + lid + unknown	________________
5th heating: mass crucible + lid + unknown	________________
6th heating: mass crucible + lid + unknown	________________
Final mass of unknown	________________
Mass % water in unknown	________________
Final mass of unknown	________________

Data Collection for Part V: Repeat of the Experiment (if necessary)

Unknown Hydrate Code	______________________

Initial observations of your unknown:

Initial mass crucible + lid	______________________
Initial mass crucible + lid + unknown	______________________
1st heating: mass crucible + lid + unknown	______________________
2nd heating: mass crucible + lid + unknown	______________________
3rd heating: mass crucible + lid + unknown	______________________
4th heating: mass crucible + lid + unknown	______________________
5th heating: mass crucible + lid + unknown	______________________
6th heating: mass crucible + lid + unknown	______________________
Final mass of unknown	______________________
Mass % water in unknown	______________________
Final mass of unknown	______________________

9.5 Dehydration of Hydrates – Post-Lab Questions

Name: ______________________________ Date: ________________

Instructor: ______________________________ Section/Group: ____________

1. If too much heat was applied to your hydrate and the salt began to decompose, how might this affect your results?

2. You added water to your desiccant salt once your experiment was concluded. Why? What did the results of that test tell you?

3. In order for your mass measurements to be accurate, the crucible must be completely cooled. Why? What effect would a slightly warm crucible have on your measurements?