EXPERIMENT 18: Formula of a Hydrate

Materials:

- Ring stand
- Crucible tongs
- Large evaporating dish
- Ring
- Hot plate
- Balance
- Stirring rod
- Small dropper for water

CuSO₄•5H₂O

Unknowns: MgSO₄•XH₂O, FeSO₄•XH₂O

Objective:

a) Confirms the formula of a hydrate with known formula.
b) Second, finds the formula of a hydrate in which the salt formula is known but not the molar amount of water.

INTRODUCTION. Some chemical compounds, especially inorganic salts, incorporate water into their crystalline structures. Water has a polar structure; it has positively and negatively charged parts within each molecule. This gives it a strong attraction toward ions. The ions in some salts attract and form strong bonds with water molecules. These salts, when they have absorbed water, are called hydrates. Anhydrous salts are salts that can form hydrates but which have had all the water driven off, usually by heat. Hydrated salts are characterized by the number of moles of water molecules per mole of salt. The so-called water of hydration of nickel(II) chloride (NiCl₂) is six moles H₂O for every one mole of NiCl₂. The hydration reaction is shown below. The hydrate in this reaction is called nickel(II) chloride hexahydrate.

\[
\text{NiCl}_2 + 6\text{H}_2\text{O} \rightarrow \text{NiCl}_2\cdot6\text{H}_2\text{O}
\]

The formula of this hydrate shows the molar amount of water incorporated into the crystal matrix. For most hydrates the amount of water included in the formula is only important when trying to measure molar amounts of the salt. You need to know the true formula weight (molar mass) in order to measure out the mass needed to give a certain number of moles. The chemical importance of the water of hydration is minimal since it can be driven off by heat or simply dissolve away if the salt is dissolved in water. From the reaction above, nickel(II) chloride hexahydrate (NiCl₂•6H₂O) the molar mass is 237.69 g/mol not 129.60 g/mol. The figure 129.60 g/mol is the molar mass of the anhydrous salt.

Greek Prefixes: used to indicate the molar amount of water in hydrated salt.

- 1 (mono-)
- 2 (di-)
- 3 (tri-)
- 4 (tetra-)
- 5 (penta-)
- 6 (hexa-)
- 7 (hepta-)
- 8 (octa-)
- 9 (nona-)
- 10 (deca-)

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Formulas for hydrates are written using a dot convention: a dot is used to separate the formula of the salt from the formula of the water of hydration. A numerical coefficient gives the molar amount of water included in the hydrate. Hydrates are named using prefixes for the word hydrate (at right). For example, CuCl$_2$$\cdot$2H$_2$O is copper(II) chloride dihydrate and CuSO$_4$$\cdot$5H$_2$O is copper(II) sulfate pentahydrate. One key point: the dot is not a multiplication sign. When calculating the molar mass you add the molar mass of water (multiplied by the coefficient).

An everyday example of hydration is concrete. Concrete is made by mixing Portland cement with water and aggregate materials. The aggregate materials are the gravel and sand that add strength to the final concrete. The Portland cement is a mixture of calcium silicates, calcium aluminate, calcium aluminoferrite and gypsum. All of these chemicals absorb water by hydration. This means that concrete does not ‘dry’ in a conventional sense. Instead the water mixed with the concrete combines chemically with the materials in the cement and the resulting hydrates form a strong matrix that holds the concrete together and makes it strong.

Another interesting example of the value of hydration is the incorporation of hydrated building materials (such as concrete, gypsum wall board and plaster). The building materials will not rise above the 100°C boiling point of water until all of the water of hydration has been driven off. This can help keep damage to a minimum until the fire can be put out. In the construction business this is known as passive fire protection.

**In this experiment**, you will be instructed to determine the mass of a sample of an unknown hydrate by "difference", using a pre-weighed crucible and cover as the container. The substance will be "dehydrated" by heat and weighed again. The loss of mass represents the mass of water in the original sample, which may be expressed as percentage of water of hydration, using the following relationship:

\[
\text{% water of Hydration} = \frac{g \text{ (water)}}{g \text{ (hydrated salt)}} \times 100
\]

To find the formula of the original hydrate, you will determine from the preceding data the most anhydrous compound for the formula given to you. Form this you can calculate the moles of anhydrous compound in the original sample. From the mass of the water in the original sample, you can calculate the moles of water to one mole of anhydrous salt, as in the 5-to-1 ratio for CuSO$_4$$\cdot$5H$_2$O above.

**In this experiment** you will be directed to "heat to constant mass." Your purpose is to heat the substance until all of the water is driven off. After the first heating, cooling, and weighing, you cannot tell if all water has been removed or if some still remains. You therefore must repeat the heating, cooling, and weighing procedure. If the same mass is reached after the second heating, you may assume that all water was removed the first time. If mass is lost in the second heating, you may be sure that all water was not removed in the first heating, and you are still unsure whether all water was driven off in the second heating. Another heating is therefore required.
The heating, cooling, and weighing sequence is repeated until two successive duplicate weighing are recorded. Duplicate weighing within 0.02g is satisfactory for this experiment.

Example 1

Assume that heating of a 3.50g sample of the hydrate of copper sulfate yields 2.10g of anhydrous copper sulfate. The mass percent of water in the hydrate would be:

\[
\% \text{ water of Hydration} = \frac{(3.50 - 2.10) \times 100}{(3.50)} = 40.0 \%
\]

Now the ratio of the number of water molecules per formula unit copper sulfate can be determined.

\[
(2.50 \text{ g CuSO}_4) \left( \frac{1 \text{ mol CuSO}_4}{159.60 \text{ g}} \right) = 0.0157 \text{ mol CuSO}_4
\]

\[
(1.40 \text{ g H}_2\text{O}) \left( \frac{1 \text{ mol H}_2\text{O}}{18.02 \text{ H}_2\text{O g}} \right) = 0.0777 \text{ mol H}_2\text{O}
\]

<table>
<thead>
<tr>
<th>Compound</th>
<th>Mole</th>
<th>Ratio</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>CuSO₄</td>
<td>0.0157</td>
<td>1</td>
<td>CuSO₄•5H₂O</td>
</tr>
<tr>
<td>H₂O</td>
<td>0.0777/0.0157 = 4.95</td>
<td>5</td>
<td></td>
</tr>
</tbody>
</table>
Example 2

A 140.5-g sample of NiSO₄•XH₂O is heated until no further decrease in mass. The mass of the anhydrous salt is 77.5 g. Find the number of water molecules in the formula of this hydrate of nickel(II) sulfate and calculate the percentage of water.

\[
\text{Reaction: } \quad \text{NiSO}_4\cdot\text{XH}_2\text{O} \rightarrow \text{NiSO}_4 + \text{XH}_2\text{O}
\]

\[
\frac{1 \text{ mol}}{(77.5 \text{ g})} \left( \frac{154.76 \text{ g}}{1 \text{ mol \ NiSO}_4} \right) = 0.50 \text{ mol NiSO}_4
\]

\[
(140.5 \text{ g} - 77.5 \text{ g}) = 63 \text{ g H}_2\text{O}
\]

\[
\frac{1 \text{ mol}}{(63 \text{ g H}_2\text{O})} \left( \frac{18.02 \text{ g}}{1 \text{ mol H}_2\text{O}} \right) = 3.5 \text{ mol H}_2\text{O}
\]

\[
\text{mol ratio} = \frac{3.5 \text{ mol H}_2\text{O}}{0.50 \text{ mol NiSO}_4} = 7 \text{ mol H}_2\text{O per mol anhydrous salt}
\]

Therefore, the hydrate formula is \text{NiSO}_4 \cdot 7\text{H}_2\text{O}, \text{nickel(II) sulfate heptahydrate}.

The percentage of water in this hydrated salt is calculated as follows:

\[
\% \text{ H}_2\text{O} = \left( \frac{63.0}{77.0} \right) \times 100 = 81.8
\]
Procedure

PART I

Dehydration of a hydrate

a) Place a few small crystals of copper sulfate pentahydrate, CuSO$_4$•5H$_2$O into an evaporating dish. Hold the evaporating dish with crucible tong and plate it on a hotplate. Heat the evaporating dish gently while stirring for about 10 minutes. Record your observation.

b) After the evaporating dish has cooled to room temperature, add a few drops of water. Record your observations.

PART II

Weight a clean, dry crucible and the cover to at least the nearest 0.01g. Place 2.0 to 2.5g of the solid unknown hydrate crystals in the crucible and weigh again to the nearest 0.01g. Place the crucible and cover in a triangle over a Bunsen burner. Heat the container and its contents vigorously with the top slightly open for about 15-20 minutes. Allow the crucible to cool (to the touch) and weigh it to the nearest 0.01g. Record this and subsequent weighing in the "constant mass" portion of the report form. If the mass difference between the first and second heating is greater than 0.02g, perform a third heating, cooling, and weighing cycle until duplicate masses are reached. The final (constant) mass should be reported as the mass of the container (crucible and cover) plus anhydrous salt.

Do not discard the anhydrous salt in the crucible until your calculations are finished and satisfactory. If they are not satisfactory, it is possible that you may need to repeat the experiment.

Calculate the mass percentage of water and the empirical formula of the hydrate.
EXPERIMENT 18: Formula of a Hydrate

REPORT FORM

Name ___________________________

Instructor ________________________

Date ______________________________

PART I

a) Write your observations when the copper hydrate is heated in evaporating dish.


b) Write your observations when water is added to copper anhydrous salt.


PART II

Unknown Number __________

1. Mass of evaporating dish

   _______ g

2. Mass of evaporating dish + hydrated salt

   _______ g


   _______ g
4. Mass of evaporating dish + anhydrous salt (after heating)

        first ________ g , second ________ g , third ________ g , fourth ________ g (if necessary)


        ________ g

6. Mass percentage of water in hydrated salt

        (show calculation)  

        ________ %

7. Formula mass of anhydrous salt

        ________ g/mol


        ________ mol

9. Formula mass of water

        ________ g/mol


        ________ mol


        ________ ratio

12. Empirical formula of hydrated salt

        ___________________________  

        (hydrated salt formula)
**Pre-laboratory Questions and Exercises**

Due before lab begins. Answer in space provided.

1. Define the following terms.
   a. Water of hydration
   b. Constant mass

2. A 2.815 g sample of CuSO$_4$$\cdot$XH$_2$O was heated until all of the water was removed. Calculate the percentage of water of hydration and the formula of the hydrate if the residue after heating weighed 2.485 g.

3. Give the correct name for the following:
   a. CaCl$_2$$\cdot$2H$_2$O  __________________________________________
   b. Cu(NO$_3$)$_2$$\cdot$H$_2$O  __________________________________________

4. The mass percent of water in a hydrate of MnCl$_2$ is 36.41%. What is the empirical formula of the hydrate?
Post-laboratory Questions and Exercises

Due after completing lab. Answer in space provided.

1. A student had a sample of BaCl$_2$$\cdot$2H$_2$O and an inert material. He heated a sample until the mass did not change. From this data, calculate the percentage of BaCl$_2$$\cdot$2H$_2$O in the original sample.
   Mass of crucible + cover = 26.39 g
   Mass of crucible + cover + unknown sample = 28.20 g
   Mass of crucible + cover + residue = 28.03 g

2. In the dehydration of hydrated salt, the sample was slightly moist because of high humidity. Briefly explain how the moisture affects the percentage calculation.

3. How can you make sure that all of the water of hydration has been removed? Explain.

4. What are the formulas for the following hydrated salts?
   a. Cobalt(II) chloride hexahydrate
   _______________________

   b. Iron(II) sulfate heptahydrate
   _______________________

5. Given that a 40.14 g sample of hydrated NiSO$_4$$\cdot$XH$_2$O is reduced in mass to 2.14 g upon heating. Determine the value of X.