**CHEM11 - DETERMINATION of the EMPIRICAL FORMULA of a HYDRATE**

**Purpose:**

Objective: To determine the empirical formula of hydrate in which the salt formula is known but not the molar amount of water. We will be working with the salt *bluestone*.

Introduction:Some chemical compounds, especially inorganic salts, incorporate water into their crystalline structures. 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:

NiCl2 + 6H2O 🡪 NiCl2·6H2O

The hydrate in this reaction is called **nickel (II) chloride hexahydrate**

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-)

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, **CuCl2•2H2O** is **copper (II) chloride dihydrate** and **Na3PO4•8H2O** is **sodium phosphate octahydrate**.

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.

**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. 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 8-to-1 ratio for Na3PO4**•**8H2O 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.

**Pre-lab Questions:** *(these questions can be answered after your purpose and must be included in the pre-lab!)*

1. Define the following terms: **(a) water of hydration** and **(b) constant mass**
2. Name the following: **(a) Cr(NO3)3•4H2O** and **(b) FeCO3•H2O**
3. What does the “**•**” mean in the chemical formula of a hydrate?

**Materials:**

Apparatus:

|  |  |  |  |
| --- | --- | --- | --- |
| Ring stand | Ring clamp (small) | Crucible | Clay triangle |
| Heat pad | Bunsen burner + striker | Electronic balance | Tongs + Spatula |

Reagents:

 *Note: for this lab you are NOT required to complete safety hazards, as the only reagent is an unknown. The safety concerns are minimal and this compound can be handled using your hands.*

Unknown hydrated salt

**Procedure:**

1. Collect your safety on and obtain the apparatus.
2. Set-up the Ring stand and clamp. Place the Bunsen burner on the ring stand and make sure the ring clamp is about 5 – 10 cm above the Bunsen burner.
3. Weigh a clean, dry crucible in the electronic balance. Place 2.0 to 2.5g of the solid unknown hydrate crystals in the crucible and find the mass.
4. Place the crucible on the clay triangle and then place this on top of the ring clamp and prepare to heat the salt.
5. Heat the crucible and contents, gently at first, then more strongly, until no more change in the bluestone can be observed (this may take anywhere from 5 – 20 minutes).
6. Using the tongs, place the crucible on top of the heat pad to allow it to cool (to the touch). Find the mass of the *anhydrous* salt.
7. Repeat steps 4 – 6 to make sure your group has obtained a ‘constant mass’ (heat for about 5 minutes). 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 crucible plus anhydrous salt.
8. 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.
9. Once the lab is complete, ask your teacher how to dispose of the salt. ***What would be fun to do, is to add water and notice the effects!***
10. Calculate the mass percentage of water and the empirical formula of the hydrate.

**Data & Observation:**

*You* ***MUST*** *come prepared with a DATA TABLE to include all pertinent information mentioned in the above procedure. Please keep in mind that you may have to collect data for more than 2 heatings!*

***Note: the calculations and question sheet will be given to you next day. You have all the info needed to complete the pre-lab portion!***

**Questions & Calculations:**

1) How much mass was **lost** when bluestone was dried?

1. What was the mass that was lost? (note: I am not asking for a numerical answer)
2. Calculate the % of mass lost.
3. The final white solid was analyzed and found to contain 39.81% Cu, 40.10% O, and 20.09% S. What is the empirical formula of the **anhydrate**?
4. Using the information above, what is the formula of the **hydrate**? *Note: you are not given the molar mass of the hydrated molecule… you must deduce this from your lab results!!!*

**Conclusion**:

Write a brief summary of your findings and any sources of error that are associated with this experiment. Also, explain if your hypothesis was found to be correct.