Experiment 5 Name: ____________________________

ANAlYSIS OF HYDRATES

In this experiment, you will learn about hydrates and how to determine the percent of water in a hydrate.

Hydrates are compounds that contain water molecules chemically bonded in their structure. The water adsorbs on the compound's surface and becomes chemically bonded as illustrated in Figure 1. The formula of hydrates includes the formula of the compound followed by a dot and then by the number of water molecules per unit and the formula of water. The hydrate illustrated has the formula $CuSO_4{\bullet}5H_2O$, which has five waters of hydration attached to a unit of the compound. Hydrates are named by naming the compound and then adding the word hydrate with the Greek prefix specifying the number of molecules of water per unit of the compound.

Table 1. Prefixes used to specify the number of water molecules in a hydrate

Number	Prefix
1	mono-
2	di-
3	tri-
4	tetra-
5	penta-

Number	Prefix
6	hexa-
7	hepta-
8	octa-
9	nona-
10	deca-

The name of $CuSO_4{\bullet}5H_2O$ is copper (II) sulfate pentahydrate.

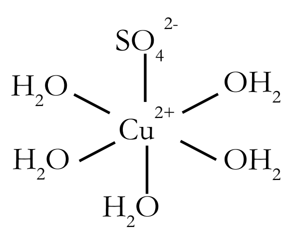

Figure 1. Chemical structure of $CuSO_4{\bullet}5H_2O$.
Note the water molecules are chemically bonded to the Cu^{2+}.

Heating a hydrate generally removes its waters of hydration. When heating $CuSO_4{\bullet}5H_2O$ to produce anhydrous $CuSO_4$ and water by breaking the chemical bonds between $CuSO_4$ and H_2O, a colour change is observed because the hydrate is blue, and the anhydrous compound is white. The liberated water then evapourates.

$$\underset{\text{blue solid}}{CuSO_4{\bullet}5H_2O} \rightarrow \underset{\text{white solid}}{CuSO_4} + 5\ H_2O$$

The process can be reversed by adding water to the anhydrous $CuSO_4$:

$$\underset{\text{white solid}}{CuSO_4} + 5\ H_2O \rightarrow \underset{\text{blue solid}}{CuSO_4{\bullet}5H_2O}$$

The percent of water in a hydrate can be determined experimentally. A known mass of hydrate is heated in a crucible until it is completely dehydrated, and then the mass of the residue (the anhydrous compound) is determined. **Complete dehydration cannot be detected by visual inspection. The hydrate is thus reheated until the successive weighings of the residue agree very closely**, indicating that all the water of hydration has been driven off and only the anhydrous compound remains in the crucible. This is called heating to a constant weight.

The mass of the water is calculated by subtracting the mass of the anhydrous compound from the mass of the hydrate. The percent of water is calculated by dividing the mass of the water by the mass of the hydrate, then multiplying by 100.

As an example, the following data is obtained by completely dehydrating a sample of $CuSO_4{\cdot}5H_2O$:

$$m_{\text{crucible+cover}} = 14.858\text{ g}$$
$$m_{\text{crucible+cover +hydrate}} = 17.537\text{ g}$$
$$m_{\text{crucible+cover+anhydrous solid}} = 16.605\text{ g}$$

To calculate for the percent of water from the experimental data:

$$m_{\text{water}} = (m_{\text{crucible+cover+hydrate}}) - (m_{\text{crucible+cover+anhydrous solid}})$$
$$= 17.537\text{ g} - 16.605\text{ g} = 0.932\text{ g}$$

$$m_{\text{hydrate}} = (m_{\text{crucible+cover+hydrate}}) - (m_{\text{crucible+cover}})$$
$$= 17.537\text{ g} - 14.858\text{ g} = 2.679\text{ g}$$

$$\%\text{ water} = \frac{m_{\text{water}}}{m_{\text{hydrate}}} \times 100$$

$$\%\text{ water} = \frac{0.932\text{ g}}{2.679\text{ g}} \times 100 = 34.8\%$$

The molecular formula of the hydrate can be used to determine the theoretical value of the percent of water. From the molecular formula $CuSO_4{\cdot}5H_2O$, the percent of water can be calculated as follows:

$$\%\text{ water} = \frac{x\,(mw_{\text{water}})}{mw_{\text{hydrate}}} \times 100$$

where x = number of water of hydration
(how many H_2O are attached)
mw_{water} = molecular weight of water
$mw_{\text{hydrate}} = mw_{\text{compound}} + x\,(mw_{\text{water}})$

Note that when calculating the molecular weight of the hydrate, the molecular weight of H_2O is added, not multiplied, to that of the compound.

$$mw_{water} = 2(1.0) + 1(16.0) = 18.0\text{ g/mol}$$

The molecular weight (formula weight) of $CuSO_4 \cdot 5H_2O$ is:

$$mw_{hydrate} = 1(63.5) + 1(32.1) + 4(16.0) + 5(18.0) = 249.6 \text{ g/mol}$$

Alternatively, the mass of hydrogen and oxygen can be added separately.

$$mw_{hydrate} = 1(63.5) + 1(32.1) + 4(16.0) + [10(1.0) + 5(16.0)] = 249.6 \text{ g/mol}$$

The percent of water in $CuSO_4 \cdot 5H_2O$ is:

$$\%\text{ water} = \frac{5(18.0\text{ g/mol})}{249.6\ g/mol} \times 100 = 36.1\%$$

PROCEDURE

I. Determination of Percent of Water

1. Obtain a clean, dry crucible with cover. Prepare the experimental set-up as shown in Figure 2. Before lighting the burner when heating on a ring stand, adjust the ring so that it is ~3 cm above the top of the burner (approximately the width of your index and middle fingers held together). This ensures you use just the right size of flame.

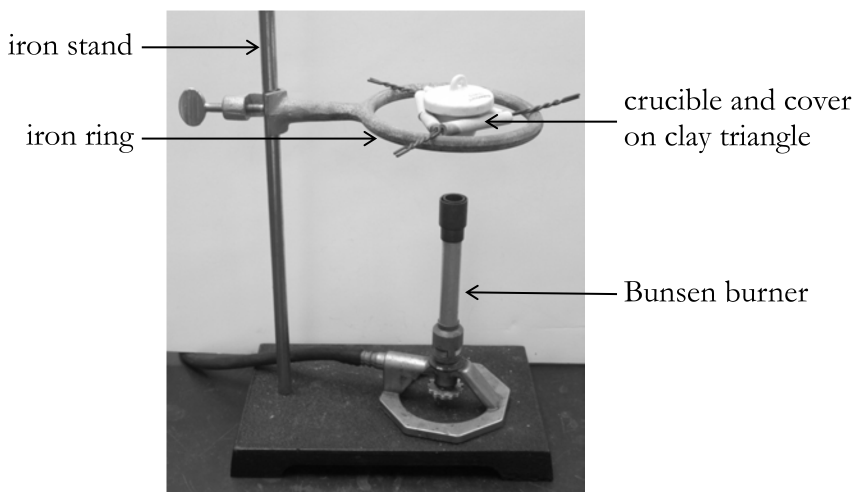

Figure 2. Experimental set-up for analysis of hydrates

Variations in the intensity of heat (gentle, moderate or strong) can be achieved by adjusting the size of the flame. Remember that the hottest part of the flame is the tip of the inner blue cone. The intensity of heat is related to the distance of the tip of the inner blue cone to the crucible – strong heat means that the tip of the inner blue cone touches the bottom of the crucible.

2. Heat the crucible strongly for 5 minutes to remove moisture, oils and other contaminants. Using a clean crucible tongs, transfer the crucible to a wire gauze on your bench. **From here on, handle the crucible and cover, hot or not, only with crucible tongs, to avoid accidental burns and reintroduction of contaminants.** Allow the crucible to cool to room temperature; check for coolness by placing your hand close to the crucible without touching it. Weigh the crucible with the cover.

CAUTION: As a rule, objects weighed on the balance must be the same temperature as the balance. Do not place hot or warm objects on the balance

CAUTION: Hot crucibles and cold crucibles look identical. Do not touch them with your hand and risk burning yourself.

3. Obtain a sample of $MgSO_4 \cdot 7H_2O$ (Bottle A) or $ZnSO_4 \cdot 7H_2O$ (Bottle B). Record the identity/formula of your sample. Place ~1.5 g of the hydrate into the crucible then cover. Weigh the covered crucible with hydrate and record.

4. Place the crucible back on the clay triangle. To avoid decomposition and producing a liquid, gently heat for 5 minutes.

5. Then, heat over a medium flame (the tip of the inner blue cone should NOT be touching the crucible) for 15 minutes. It's okay if the bottom of the crucible or clay triangle glows orange, but not the whole crucible. Be careful not to heat vigorously because if the temperature is too high, the compound may decompose.

6. Allow the crucible to cool to room temperature. Weigh the covered crucible with the residue.

7. Reheat in medium flame for another five minutes. Cool and reweigh the covered crucible with residue. A difference of less than 0.01 g between the first and second weighings indicate that the salt is completely dehydrated. If the difference is more than 0.01 g, repeat the heating, cooling and weighing process until a constant weight is obtained. **Use the last recorded weight for your calculations.**

II. Dehydration of Hydrates

1. In a clean, dry test tube, add a tiny amount (tip of the spatula) of $CoCl_2 \cdot 6H_2O$. Record the colour of the crystals.

2. Using a Bunsen burner, heat the test tube gently until a change in colour is observed. Record the new colour of the crystals.

3. Allow the test tube to cool. Add two drops of distilled water to the residue. Record the new colour.

CLEAN-UP

- Dispose of wastes in designated containers in the front hood.
- Wash the crucible, cover and test tube. Return them to your workstation.
- Make sure the Bunsen burner and gas valve are off, disconnect the tubing from the gas valve.

Name: ________________________________ **Date:** ____________________

Partner's Name: _________________________

ANALYSIS OF HYDRATES

DATA AND OBSERVATIONS

I. Percent of Water

Formula of hydrate ____________________

$m_{crucible+cover}$ ____________________

$m_{crucible+cover+hydrate}$ ____________________

$m_{crucible+cover+anhydrous\ solid}$ (first heating) ____________________

$m_{crucible+cover+anhydrous\ solid}$ (second heating) ____________________

$m_{crucible+cover+anhydrous\ solid}$ (third heating) ____________________

II. Dehydration of Hydrates

Original colour of hydrate ____________________

Colour after heating ____________________

Colour after adding water ____________________

POST-LAB QUESTIONS AND CALCULATIONS

Show clearly the complete calculations with correct number of significant figures and units.

1. a. Name the following hydrates:

   $CoCl_2 \cdot 6H_2O$ ____

   $BeSO_4 \cdot 4H_2O$ ____

b. Write the formulas for the following hydrates

sodium sulfite heptahydrate ______________________________

barium chloride dihydrate ______________________________

2. Use your **experimental data** to calculate the percent of water in the hydrate.

3. Use the **molecular formula** (from the periodic table) of the hydrate to calculate the percent of water. (Note: Theoretically, this is the percent of water you should get experimentally).

   Formula of Hydrate: ____________________

4. Compare the percent of water determined from the experimental data (question 2) to that calculated using the molecular formula (question 3). Which is higher and why? (Hint: in which experimental steps could you have added/lost mass from your sample, how does this affect the apparent mass of the water measured?)

5. In Part II, why does the colour change when the compound is heated? Why does the colour change when water is added?