Experiment 2 – Formula of a Hydrate

Overview

In this experiment, you will determine the chemical formula for a compound that has water entrained in the crystal structure. This water is "driven off" easy by heating a sample of the compound. Then, using the difference in mass, and the chemical formula of the anhydrous residue, one can find the chemical formula of the hydrate. In the case of titanium (III) oxalate decahydrate, the reaction is represented as follows:

$$Ti_2(C_2O_4)_3 \cdot 10\ H_2O \xrightarrow[\Delta]{} Ti_2(C_2O_4)_3 + 10\ H_2O$$

Hydrate Salts

Hydrate salts have chemical formulae such as $OsCl_3 \cdot 3\ H_2O$ (osmium (III) chloride trihydrate) or $Cs_2S \cdot 4\ H_2O$ (cesium sulfide tetrahydrate). In these formulae, the dot indicates a **weak chemical bond** that is easily broken by heating the sample. The mass loss from the heating of the sample can be used to determine the **percent composition** of the compound due to water, and from this, the mole ratio of water to **formula units** of the salt.

Typically, this experiment is performed in a **crucible**, a piece of laboratory equipment that can hold a small sample of matter and is made to be heated and reheated many times. One determines the mass of the crucible, the crucible plus the original sample of hydrate, and the mass of the crucible

Example: In order to determine the number of waters of hydration for a hydrate of CrF_3, Lelani records the following data:

	Mass (g)
Crucible and Lid	43.8629
Crucible and Lid + hydrate salt	45.4201
Crucible and Lid + anhydrous residue	44.8012

What is the formula of the hydrate salt as determined from Lelani's data?

Solution: First, let's find the percent by mass due to water in the sample. This is done by finding the mass of the hydrate sample ($CrF_3 \cdot X\ H_2O$) and that of the anhydrous residue (CrF_3). The difference is the mass of water.

$$mass\ hydrate = 45.4201\ g - 43.8629\ g = 1.5572\ g$$

$$mass\ anhydrous\ residue = 44.8012\ g - 43.8629\ g = 0.9383\ g$$

$$mass\ water = 45.4201\ g - 44.8012\ g = 0.6189\ g$$

The percent by mass due to water is then given by

$$\%\ water = \left(\frac{0.6189\ g}{1.5572\ g} \cdot 100\%\right) = 39.7\underline{4}44\% = 34.74\%$$

Now, to get the value of X (the number of waters of hydration per formula unit) is basically an empirical formula problem. To solve this,, we need to know the formula weight (FW) of the anhydrous residue and the molar mass of water:

CrF_3:	108.991 g/mol
H_2O:	18.02 g/mol

Using this data, we can find the mole ratio of anhydrous formula units to water molecules.

$$0.9383\ g \cdot \frac{mol\ CrF_3}{108.991\ g} = 0.0086090\ mol\ CsF_3$$

$$0.6189\ g \cdot \frac{mol\ H_2O}{18.02\ g} = 0.034345\ mol\ H_2O$$

The mole ratio is given by

$$\frac{0.034345\ mol\ H_2O}{0.0086090\ mol\ CrF_3} = 3.99\frac{H_2O}{CrF_3}$$

This can be rounded to 4 waters per CrF_3 and the formula of the hydrate is therefore

$$CrF_3 \cdot 4\ H_2O$$

Experimental Procedure

Be sure to wear goggles throughout this experiment.

1. Measure and record the mass of a clean, dry crucible with cover.
    a. Heat the crucible and lid with a Bunsen burner for 5 minutes using the procedure described by your laboratory instructor.
    b. After cooling (about 5-10 minutes), record the mass.
    c. Repeat this procedure until two consecutive weighings produce a mass that agrees within ± 0.0050 g.

2. Obtain about 1-1.5 g of the selected compound and place it in the crucible. Use a spatula to break up any large pieces of the substance by pressing the pieces against the wall of the crucible. Measure and record the mass of the crucible, cover, and compound.

    a. Heat the crucible, lid, and sample with a Bunsen burner for 10 minutes as before, being careful to avoid the "popcorn effect" or other problems that cause you to lose sample.

    b. After cooling (about 5-10 minutes), record the mass.

    c. Repeat this procedure until two consecutive weighings produce a mass that agrees within ± 0.050 g.

Finally, dispose of your sample as directed.

Vocabulary and Concepts

References

Klingshirn, M. A., Wyatt, A. F., Hanson, R. M., & Spessard, G. O. (2008). Determination of the Formula of a Hydrate: A Greener Alternative. *Journal of Chemical Education*, *85*(6), 819. doi:10.1021/ed085p819

Randall, J. (2013). *Advanced Chemistry with Vernier* (3rd ed.). Beaverton, OR: Vernier Software and Technology, Inc.

Pre-Laboratory Assignment – Formula of a Hydrate

Name _____ Section _____

Ezekiel is determining the formula of a hydrate of calcium chloride ($CaCl_2 \cdot X\ H_2O$). He records the following data:

	Mass (g)
Crucible and Lid	28.4735
Crucible and Lid + hydrate salt	33.1938
Crucible and Lid + anhydrous residue	32.0339

1. What is the mass of the hydrate? _____
2. What is the mass of the anhydrous residue? _____
3. What is the mass of water lost on heating? _____
4. How many moles of water were lost from the hydrate? _____
5. How man moles of anhydrous residue ($CaCl_2$) were present? _____
6. What is the mole ratio of H_2O : $CaCl_2$? _____
7. What is the formula of the hydrate? _____

Report Sheet – Formula of a Hydrate

Name _____ Date _____

Lab Partner(s) _____

Data Table

Compound selected for analysis	
Mass of crucible and cover (g)	
Mass of crucible, cover, and hydrated sample (g)	
Mass of hydrated sample (g)	
Mass of crucible, cover, and dehydrated sample – 1st weighing (g)	
Mass of crucible, cover, and dehydrated sample – 2nd weighing (g)	
Mass of crucible, cover, and dehydrated sample – 3rd weighing (g)	
Mass of crucible, cover, and dehydrated sample – 4th weighing (g)	
Mass of dehydrated sample (g)	
Mass of water evolved (g)	

In the space below, determine the formula for the hydrate. Show your work!