Experiment 7 – Hydrates

Most solid chemical compounds will contain some water if they have been exposed to the atmosphere for any length of time. In most cases the water is present in quite small amounts and is only *adsorbed* on the surface of the crystals. This adsorbed water can usually be removed by gentle heating. Other solid compounds will contain larger amounts of water that is bound to the compound more strongly. These compounds are called **hydrates**, and they are usually ionic salts. The water present in these salts, called the **water of hydration**, is generally bound to the *cations* in the compound.

The water in a hydrate is not merely held on the surface of the crystals. It has actually been incorporated into the crystal structure of the substance. The crystal will be able to incorporate a definite number of moles of water per mole of the anhydrous ionic substance. This number is stated in the formula of the hydrate, as in the formulas $CuSO_4 \bullet 5H_2O$ or $CoCl_2 \bullet 6H_2O$. As the formula indicates, water forms a definite percentage of the weight of any hydrate. For example, the weight of a mole of $CuSO_4 \bullet 5H_2O$ is 250 grams (this is the molar mass of $CuSO_4 \bullet 5H_2O$). A mole of $CuSO_4 \bullet 5H_2O$ contains 5 moles of water (which corresponds to 90 grams of water) as part of its structure. Therefore, the substance $CuSO_4 \bullet 5H_2O$ always consists of 90/250 or 36% water by weight. The water is a definite part of the structure of the compound, and the substance should not be considered to be "wet."

A few hydrated compounds lose water spontaneously to the atmosphere upon standing. Such compounds are called **efflorescent**. All hydrated compounds may be dehydrated by heating. As part of this experiment, the percentage of water lost by an unknown hydrated compound on heating will be determined. The number of moles of water per mole of hydrated compound may be calculated from the weight of the water lost and the formula weight of the anhydrous substance.

Some anhydrous ionic compounds will absorb water from the atmosphere to become hydrates. These substances are referred to as **hygroscopic** substances. A few ionic compounds will take up so much water from the atmosphere that they may eventually dissolve in their water of hydration; such substances are called **deliquescent**.

Safety Precautions:

- Wear your safety goggles.

Waste Disposal:

- Discard your solid waste in the container in the hood marked "Waste Hydrates".

Procedure

Part 1: Deliquescence and Efflorescence

1. Place a few crystals of sodium sulfate decahydrate, $Na_2SO_4 \bullet 10H_2O$, on a watch glass or a sheet of paper. On a separate watch glass, place a few pieces of anhydrous calcium chloride, $CaCl_2$. Observe both samples occasionally as you proceed with other parts of the experiment. Record your observations.

Part 2: The Formula of an Unknown Hydrate

2. Place a clean, dry porcelain crucible on a wire triangle, supported on a ring attached to a ring stand, and heat the crucible strongly for about 3 minutes to be sure it is perfectly dry. Allow the crucible to stand in place on the triangle long

enough to cool again, and then weigh it as accurately as possible. (Make sure to use a digital balance.) Record the mass.

3. Obtain a sample of one of the unknown hydrated salts. These will be labeled with the formula of the compound, but without specifying the number of moles of water of hydration. For example, an unknown could be designated as $MgSO_4 \bullet xH_2O$. The goal of this part of the experiment is to determine the value of x in the formula.
4. Place a sample of the hydrate weighing between 2 and 4 grams in the crucible, and weigh the crucible again as accurately as possible. Record the mass. Obtain the mass of the sample by subtraction of the mass of the empty crucible.
5. Heat the uncovered crucible and sample for about 10-15 minutes. Use a low flame at first and then heat strongly. Cover the crucible and let it cool. Then remove the cover and re-weigh it. To be sure that the dehydration process has been completed, re-heat the crucible for 5 minutes, cool, and weigh it again. This process should be repeated, if necessary, until the sample no longer loses a significant amount of mass on heating. (While you are waiting for the sample to heat and/or cool, you may want to get started on Part 3.)

Calculations for Part 2

First, calculate the mass percent water in the compound. Recall that the mass percent of a substance is the mass of that substance divided by the mass of the entire sample and then multiplied by 100. You can use the mass of water lost divided by the mass of the entire hydrated sample before heating to find the mass percent water in the hydrate.

The objective of the experiment is to find the number of moles of water per mole of the hydrated compound. This is done by calculating the number of moles of water given off on heating, and comparing this number to the number of moles of the anhydrous compound remaining after the heating.

The number of moles of water that are driven off by heating can be calculated, since the weight lost in heating should by entirely due to loss of water. The number of moles of <u>anhydrous</u> substance remaining after heating can also be calculated. The substance in the crucible after heating should be entirely the anhydrous salt. The formula of the anhydrous salt is known, so its formula weight can be calculated. The weight of the material in the crucible after heating can then be used to determine the number of moles of the anhydrous salt.

Comparing these two mole numbers gives the complete formula of the hydrated salt. Dividing the moles of water by the moles of anhydrous salt gives the ratio of <u>moles</u> of water to moles of anhydrous salt. This ratio is expressed in the formula of the compound. For example, if heating some hydrated copper (II) sulfate gave off 0.060 moles of water, and left behind 0.012 moles of anhydrous copper (II) sulfate ($CuSO_4$), then the ratio of water to anhydrous salt is 5:1, and the formula would be written as $CuSO_4 \bullet 5H_2O$. There are five moles of water in the hydrated salt for every mole of $CuSO_4$.

Part 3: Reversibility of Hydration

6. Grind a few crystals of $CuSO_4 \bullet 5H_2O$ in a mortar. Then gently heat the powder in a Pyrex test tube fastened in a horizontal position by a clamp on a ring stand. Observe whether any water can be seen (as a mist or droplets) condensing inside the test tube. Note the appearance of the solid residue after heating. When the residue seems to be completely dehydrated, allow it to cool. Then add a few drops of water to the residue in the tube. What does the water seem to have done to the anhydrous solid?
7. The same test can be repeated with crystals of cobalt chloride hexahydrate ($CoCl_2 \bullet 6H_2O$). Because of the color changes associated with hydration and

dehydration of these two substances, the two anhydrous salts ($CuSO_4$ or $CoCl_2$) can be used to detect small quantities of water.

Questions

1. Calculate the mass percent water for the hydrate $Na_2CO_3 \bullet 10H_2O$.
2. If you did not heat your hydrate sufficiently to drive off all the water, would the percent water you calculated be too high or too low? Explain.
3. How can you tell if you have heated the hydrate sample long enough? Explain.
4. If 4.68 g of $CaCl_2 \bullet xH_2O$ is heated to constant mass, the residue weighs 3.54 g. Determine the value of x and the formula of the hydrate.