Name: _____ Section: _____

Experiment 4 – Hydrates and Anhydrous Salts

Discussion

When a salt incorporates a certain number of water molecules for each formula unit in order to create a stable complex substance, we have a hydrate. These compounds are not "wet"; the water molecules are incorporated into the solid structure in a fixed pattern. The following are some representative examples:

$MgCl_2 \cdot 2H_2O$, $Na_2SO_4 \cdot 10H_2O$, $CoCl_2 \cdot 6H_2O$, $BaCl_2 \cdot 2H_2O$, $CuSO_4 \cdot 5H_2O$

Notice that the formulas all begin with the salt and a dot separates the number of water molecules attached per formula unit. The water is chemically bound to the substance with weaker bonds than the ionic attraction within the salt or the covalent bonds within the water molecule.

In naming hydrates, you use the nomenclature rules you have learned to name the salt and add an appropriate prefix (mono = 1, di = 2, tri = 3, tetra = 4, penta = 5, hexa = 6…) to identify the number of water molecules ending the name with the word hydrate. For example, $CuSO_4 \cdot 5H_2O$ has the name copper(II) sulfate pentahydrate.

The water chemically bound in hydrates can generally be removed by heating without destroying the salt or the molecule of water. When the water has been completely removed, we call the remaining compound the anhydrous salt (*salt without water*). Anhydrous salts will readily absorb water from its surroundings and may be used as moisture detectors. A common example is anhydrous cobalt(II) chloride, $CoCl_2$ (blue in color). When in contact with moisture, this substance absorbs water to create cobalt(II) chloride hexahydrate, $CoCl_2 \cdot 6H_2O$ (pink in color).

In this experiment, hydrated salts will be heated. First, we will perform a qualitative test with the hydrate $CuSO_4 \cdot 5H_2O$ (blue in color), whereby heat is applied until the hydrate becomes anhydrous $CuSO_4$ (white in color). Next, the resulting liquid will be tested with anhydrous $CoCl_2$ to detect if that liquid is water. Finally, we will also perform a quantitative experiment with an unknown hydrate to determine the mass percent of water lost and solve for the formula and name of the unknown hydrate.

Procedure

A. Qualitative Determination of the Released Liquid after Heating $CuSO_4 \cdot 5H_2O$

1. Weigh out approximately 3 grams of copper(II) sulfate pentahydrate into a clean, dry large ignition tube (25 x 200 mm). Make sure the hydrate is not sticking to the sides; it should all be near the bottom of the tube.

2. Clamp the ignition tube at a 15–20 degree downward from the horizon so that the mouth of the tube is lower than the bottom of the tube where the hydrate is located. The clamp should be near the mouth of the test tube.

3. To collect a sample of the liquid that will be removed from the hydrate, place a medium test tube held upright in an Erlenmeyer flask just below the mouth of the ignition tube containing the hydrate. Your instructor will demonstrate this set-up.

4. Heat the hydrate with a Bunsen burner gently at first, and gradually increase the intensity of the heat. Note the changes that occur. Collect some liquid that condenses in the lower, cooler mouth of the tube. Continue heating until most of the blue color has disappeared. Your residue should now be pale blue or white (*not black, see note*). To remove the remaining liquid still inside the ignition tube, gently warm the entire tube to vaporize. Allow tube and contents time to cool. Observe and record the appearance and odor of the collected liquid. NOTE: at temperatures above 600 °C, the copper(II) sulfate decomposes; sulfur trioxide is driven off and black copper(II) oxide remains as residue.

5. Obtain a couple pieces of anhydrous cobalt(II) chloride test strips. Place distilled water on one and your experiment liquid on the other. Compare and record observations.

6. Empty the anhydrous salt from your cooled ignition tube onto a watch glass and divide it into two sections. To one section, add a few drops of distilled water; to the other, add your experiment liquid. Compare and record observations. Dispose of residue in the waste container provided.

B. Quantitative Determination of Mass Lost in a Hydrate

1. Obtain an unknown hydrate sample. Record the unknown number.

2. Weigh a clean dry crucible with its cover to the highest precision. Be sure that you use the same balance for all mass measurements within the same experiment to cancel out systematic errors.

3. Add between 2–3 grams of the unknown into the crucible with cover and weigh to the highest precision.

4. To a ring stand, attach a ring and place a clay triangle on the ring. Transfer the crucible onto the clay triangle and adjust the cover so it is slightly ajar to allow liquid vapor to escape. Very gently heat with a Bunsen burner for about 5 minutes. Readjust the flame to continue heating, more intensely now so that the bottom of the crucible glows red, for another 12 minutes.

5. After this two-step heating process (1^{st} heating) is complete, turn off the burner, close the crucible lid, and allow the crucible to cool for about 10 minutes before weighing. It is important to have all mass measurements made at or near room temperature. Do not transfer the hot crucible to the tabletop; it is too hot and will permanently mark the table. Use the crucible cooling plates that are provided. Weigh your sample.

6. Heat the covered crucible and contents another 6 minutes at maximum temperature; cool and reweigh (2^{nd} heating). If the results agree within $\pm$0.050 g, you are done and will not need to have a 3^{rd} heating. If the difference is greater than $\pm$0.050 g, repeat the heating for another 6 minutes (3^{rd} heating).

Name: _____ Section: _____

Data and Calculations for Experiment 4

A. Qualitative Determination of the Released Liquid

1. Record observations regarding the solid before, during, and after heating the copper(II) sulfate pentahydrate.

2. Compare and record observations after adding liquid to the anhydrous cobalt(II) chloride test strips.

3. Compare and record observations after adding liquid to the residue on the watch glass.

4. What conclusions can you draw from the above observations?

5. Write the balanced chemical equation for the decomposition of copper(II) sulfate pentahydrate, include phases.

B. Quantitative Determination of Mass Lost in a Hydrate

Sample number: _____

1. Mass of crucible and cover _____
2. Mass of crucible, cover and sample _____
3. Mass of crucible, cover and sample after 1st heating _____
4. Mass of crucible, cover and sample after 2nd heating _____
5. Mass of crucible, cover and sample after 3rd heating _____
6. Mass of sample after final heating _____
7. Mass of original sample _____
8. Total mass lost by sample _____

9. Percentage of water in sample _____

10. Ask your instructor for the name of the anhydrous salt of your residue and solve for the formula and name of your original unknown hydrate.

Formula: _____ Name: _____

11. Is it possible that the decrease in mass from heating is something other than water?

Yes or *No* Explain and include an example.

Pre-Lab (to be completed before coming to lab)

A student heated a hydrated salt sample with an initial mass of 2.244 grams. After the second heating, the mass had decreased to 1.798 grams. Make the assumption that all the lost mass is water.

a) Solve for the mass lost.

b) Calculate the percentage of water in the original hydrated salt sample.

c) The instructor has informed you that the sample is a hydrate of $ZnSO_3$. Use the information above and molar masses from the periodic table to solve for the moles of $ZnSO_3$ in the residue and the moles of H_2O lost.

d) Solve for the *X* in the formula of the hydrate of $ZnSO_3 \cdot XH_2O$ and name it.

Moles of water / moles of $ZnSO_3$ = *X*

Formula: _____ Name: _____