1. Hydrates and Their Thermal Decompositions

Introduction

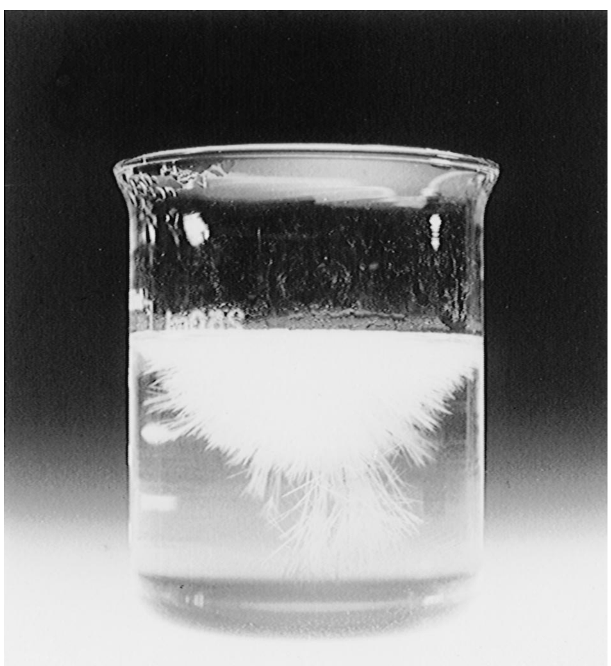
What is a *hydrate?* A hydrate is a salt that has crystallized from aqueous solution with weakly bound water molecules *contained* in the crystals (Ebbing/Gammon, Section 2.8).

Consider the crystallization of sodium acetate ($NaC_2H_3O_2$) as an example. This process is shown in Figure 1.1. The crystals that you see in the photograph can be removed from the solution by filtration. If these crystals are then allowed to sit in the open air for a few hours, all of the residual moisture on their surfaces will evaporate. They will appear to be dry. Nevertheless, water will still be present, and it will be present in a definite stoichiometric amount. Chemical analysis would show that 3 water molecules accompany every formula unit of sodium acetate. This is shown by writing the formula as $NaC_2H_3O_2 \bullet 3H_2O$.

The definite stoichiometry occurs because the water in a hydrate occupies definite sites in the crystalline lattice, just as Na^+ and Cl^- ions occupy definite positions in the NaCl lattice. Because this water occupies definite sites, it must be present in definite stoichiometric amounts. The quantity of water will not change as long as the temperature (and pressure) is not altered significantly. A substantial increase in temperature, however, will cause the loss of hydrate water.

FIGURE 1.1

The crystallization of the trihydrate of sodium acetate from an aqueous solution.



Purpose

You will use mass relationships to identify the product that you obtain from the thermal decomposition of barium chloride dihydrate ($BaCl_2 \bullet 2H_2O$). You will also have the opportunity to observe the thermal decomposition of several other hydrates.

Concept of the experiment

In the first part of this experiment, you will examine the quantitative aspects of the thermal decomposition of $BaCl_2 \bullet 2H_2O$. When this substance is heated, there are at least three possible reactions:

$$BaCl_2 \bullet 2H_2O(s) \xrightarrow{\Delta} BaCl_2 \bullet H_2O(s) + H_2O(g)$$

$$BaCl_2 \bullet 2H_2O(s) \xrightarrow{\Delta} BaCl_2(s) + 2H_2O(g)$$

$$BaCl_2 \bullet 2H_2O(s) \xrightarrow{\Delta} BaO(s) + H_2O(g) + 2HCl(g)$$

The product of the first reaction is the monohydrate, $BaCl_2 \bullet H_2O$. Alternatively, all of the water may be lost so that anhydrous (water-free) $BaCl_2$ is obtained, as shown in the second equation. Finally, the decomposition may take the course shown in the third equation. A simple loss of water has not occurred. HCl is also liberated, so the oxide, BaO, remains when the reaction is complete.

How will you know which reaction has occurred? Because you will know the mass of the reactant, you will be able to calculate the mass of each possible product. You will then be able to identify the product by comparing the measured mass of the product with the masses that you have calculated. This reaction is particularly "well behaved," so your identification will be unambiguous if you have followed the directions carefully. Moreover, you will pool your results with those of your classmates to establish the precision of the experiment. You may do the calculations by hand or, if your laboratory instructor wishes, you can use a computer and the Internet (Appendix: Mistakes, Errors, Accuracy, and Precision).

You will also have the opportunity to observe the colorful thermal decompositions of $CrCl_3 \bullet 6H_2O$, $CoCl_2 \bullet 6H_2O$, $NiSO_4 \bullet 7H_2O$, and $CuSO_4 \bullet 5H_2O$. Because these reactions are not as well behaved as the one involving $BaCl_2 \bullet 2H_2O$, you will study only their qualitative aspects. Remember that a reaction must accompany every color change. Simple loss of water may occur on gentle heating, but more extensive decomposition could occur with protracted, strong heating. For example, $CuSO_4 \bullet 5H_2O$ will lose water to give anhydrous $CuSO_4$, which, in turn, can lose gaseous SO_3 to give CuO. Moistened blue litmus paper would detect this reaction, however, because SO_3 reacts with water to give sulfuric acid (H_2SO_4), and acids cause blue litmus paper to turn pink.

Procedure

Getting started

1. Obtain a crucible and lid. Wash, rinse, and dry them. Also obtain 4 small test tubes and 8 small strips of blue litmus paper.

2. Place the covered crucible in a clay triangle on an iron ring that is attached to a ring stand. Adjust the height of the ring so that the bottom of the crucible will be in the hottest part of the flame of your laboratory burner. The first figure from Lab: The Empirical Formula of an Oxide shows the correct arrangement.
3. Obtain directions for discarding the solid products that will be produced in this experiment.

Doing the quantitative experiment

1. Heat the covered crucible strongly for about 3 min. The bottom of the crucible should attain a red-hot glow during this time.
2. Move the burner and allow the crucible to cool (5–10 min). When the crucible is cool, you should feel no heat when you place one of your fingers about 1/2 inch from the bottom of the crucible.

CAUTION: Avoid burning your fingers. Do not touch the crucible or the iron ring at any time during this experiment.

3. When the covered crucible is cool, transfer it to the pan of your most precise balance, using crucible tongs and holding a wire gauze under it. If you must wait to use the balance, do not place the crucible directly on the bench. Put it on the wire gauze or leave it in the clay triangle.
4. Obtain the record the mass of the covered crucible.
5. Repeat Steps 1 and 4 until two consecutive masses differ by nor more than ± 0.001 g (or any other range stipulated by your laboratory instructor). Use the last mass that you obtain in subsequent calculations.
6. Using a platform balance, triple beam balance, or an electronic top-loading balance, add about 1.4–1.5 g of $BaCl_2 \bullet 2H_2O$ to a piece of waxed weighing paper whose mass you have already determined.
7. Uncover the crucible using crucible tongs, add the sample of $BaCl_2 \bullet 2H_2O$ to the crucible, dispose of the weighing paper, and cover the crucible again.
8. Obtain and record the mass of the covered crucible and its contents. Calculate and record the mass of $BaCl_2 \bullet 2H_2O$ in the sample.
9. Transfer the crucible to the clay triangle, using tongs. Leave the lid slightly ajar.
10. Heat the crucible slowly by brushing it with the flame for about 2–3 min.
11. Heat the crucible strongly for about 15 min. The bottom of the crucible should be red hot during this time.
12. Repeat Steps 2 through 5.
13. Calculate and record the mass of the product. Calculate the ratio

$$\text{Mass of product/mass of } BaCl_2 \bullet 2H_2O$$

Share your value with your classmates and obtain their values for this ratio.

Doing the qualitative experiment

1. Place a pea-sized portion of $CrCl_3 \bullet 6H_2O$ in one of the test tubes.
2. With a test tube clamp, hold the test tube at an angle of about 45° and quickly move the test tube in and out of the hottest part of the flame from your burner.

3. Note and record all color changes. Immediately test with blue litmus paper the water that condenses on the upper surface of the test tube.
4. When no further color changes are apparent, hold the test tube directly in the hottest part of the flame for about 1 min. Repeat the test with blue litmus paper. Record your observations.
5. Repeat Steps 1 through 4 with, in turn, pea-sized portions of $CoCl_2 \bullet 6H_2O$, $NiSO_4 \bullet 7H_2O$, and $CuSO_4 \bullet 5H_2O$.

CAUTION: Before you leave the laboratory, make sure that your gas outlet and those of your neighbors are closed.

Hydrates and Their Thermal Decompositions

Date: .... Student name: ....

Course: .... Team members: ....

Section: ....

Instructor: ....

Prelaboratory assignment

1. Provide definitions for the following words:

   a. Hydrate

   b. Anhydrous

2. What are the differences between water on the surface of the crystals of a hydrate and hydrate water itself?

3. a. The thermal decomposition of 2.076 g of $MgSO_4 \bullet H_2O$ produces 1.812 g of a product in a well-behaved reaction. There are two possibilities:

$$MgSO_4 \bullet H_2O(s) \xrightarrow{\Delta} MgSO_4(s) + H_2O(g)$$

$$MgSO_4 \bullet H_2O(s) \xrightarrow{\Delta} MgO(s) + SO_3(g) + H_2O(g)$$

Complete the following table, and then identify the correct solid product by comparing the calculated masses of $MgSO_4$ and MgO with the observed mass of the product.

Substance	Formula Weight	Moles	Mass (g)
$MgSO_4 \bullet H_2O$	....	....	....
$MgSO_4$	....	....	....
MgO	....	....	....

Because the observed mass of the product is 1.812 g, the product is ....

b. What qualitative test could be used to substantiate this result?

4. What special safety precautions have been cited in this experiment?

Hydrates and Their Thermal Decompositions

Date: ....

Course: ....

Section: ....

Instructor: ....

Student name: ....

Team members: ....

....

....

Results

1. *Thermal decomposition of $BaCl_2 \bullet 2H_2O$*

Mass of empty crucible and lid (g) ....

....

....

Mass of crucible, lid, and $BaCl_2 \bullet 2H_2O$ (g) ....

Mass of empty crucible and lid (g) ....

Mass of $BaCl_2 \bullet 2H_2O$ (g) ....

Mass of crucible, lid, and product (g) ....

....

....

Mass of product (g) ....

Mass of product/mass of $BaCl_2 \bullet 2H_2O$ ....

Shared data (Include your own.)

2. *Observations on thermal decompositions of other hydrates*

$CrCl_3 \bullet 6H_2O$

$CoCl_2 \bullet 6H_2O$

$NiSO_4 \bullet 7H_2O$

$CuSO_4 \bullet 5H_2O$

Student name: .... Course/Section: .... Date: ....

Questions

1. a. Compute the mean value of the ratio

$$\text{Mass of product/mass of } BaCl_2 \bullet 2H_2O$$

and its standard deviation, using the shared data. Calculate the standard deviation using a computer and the Internet, as described in Appendix: Mistakes, Errors, Accuracy, and Precision, or by hand using the technique also described in Appendix: Mistakes, Errors, Accuracy, and Precision.

b. Give a mean value for the ratio that is consistent with the precision of the data.

c. Use this value of the ratio and your own mass of the $BaCl_2 \cdot 2H_2O$ to calculate a mass of the product that is consistent with the precision of the experiment.

d. Complete the following table. Then identify the correct solid product by comparing the calculated masses of $BaCl_2 \cdot H_2O$, $BaCl_2$, and BaO with the observed mass of the product (the value calculated in Question 1c).

Substance	**Formula Weight**	**Moles**	**Mass (g)**
$BaCl_2 \cdot 2H_2O$	....	....	....
$BaCl_2 \cdot H_2O$	....	....	....
$BaCl_2$	....	....	....
BaO	....	....	....

Because the observed mass of the product is .... g, the product is ....

2. Write a chemical equation that is consistent with each observation that you have made concerning the thermal decompositions of the other hydrates. More than one equation may be required for each hydrate. Explain your reasoning for each equation.