EXPERIMENT 6

Hydrates

Performance Goals

6–1 Calculate the percentage of water of hydration in a compound from experimental data.

6–2 Calculate the formula of a hydrate of a known anhydrous salt from experimental data.

CHEMICAL OVERVIEW

Hydrates are chemical compounds that contain water as part of their crystal structure. This water is quite strongly bound and is present in a definite proportion relative to other constituents. It is referred to as **water of hydration.**

The formula of a hydrate consists of the formula of the **anhydrous** (without water) **compound** followed by a dot, the number of water molecules that crystallize with one formula unit of the compound, and the formula of water. For example, $CuSO_4 \cdot 5H_2O$ indicates that 5 molecules of water—H_2O—crystallize with 1 formula unit of anhydrous copper(II) sulfate, $CuSO_4$, to form copper sulfate pentahydrate.

Generally, water of hydration can be driven from hydrates by heating, leaving behind the anhydrous salt. The process may be accompanied by physical changes, such as a change in color or physical appearance. For example, $CuSO_4 \cdot 5H_2O$ is an intense blue shiny crystal which, upon heating, turns into pale green-blue powdery anhydrous $CuSO_4$.

In this experiment you will be instructed to determine the mass of a sample of an unknown hydrate by difference, using a preweighed crucible as the container. The substance will be ''dehydrated'' by heat and weighed again. The loss of mass represents the mass of water in the original sample, which may be expressed as percentage of water of hydration, using the usual (part quantity/total quantity) $\times$ 100 relationship:

$$\text{Percentage water of hydration} = \frac{\text{grams of water}}{\text{grams of hydrate}} \times 100 \qquad (6.1)$$

To find the formula of the original hydrate, you will determine from the preceding data the mass of the anhydrous compound, the formula of which will be given to you. From this you can calculate the moles of anhydrous compound in the original sample. From the mass of water in the original sample you can calculate the moles of water. By dividing the moles of water

by the moles of anhydrous salt, you obtain the ratio of moles of water to 1 mole of anhydrous salt, as in the 5-to-1 ratio for $CuSO_4 \cdot 5H_2O$ above.

In this experiment you will be directed to ''heat to constant mass.'' Your purpose is to heat the substance until *all* of the water is driven off. After a first heating, cooling, and weighing, you cannot tell if all water has been removed or if some still remains. You therefore repeat the heating, cooling, and weighing procedure. If the same mass is reached after the second heating, you may assume that all water was removed the first time. If mass was lost in the second heating, you may be sure that all water was *not* removed in the first heating, and you are still unsure that it was all driven off in the second heating. Another heating is therefore required. The heating, cooling, and weighing sequence is repeated until two successive duplicate weighings are recorded. Two weighings within the ± uncertainty range of the balance are generally considered to be duplicate weighings; duplication within 0.005 g is satisfactory for this experiment.

Your instructor may require you to perform the experiment twice to obtain duplicate results or to run more than one unknown. If so, plan your use of time. The procedure includes some periods in which you must wait for a crucible to cool. The cooling periods in the first run of the experiment can be used for heating periods in the second run and vice versa. In this way you perform both runs simultaneously.

SAFETY PRECAUTIONS AND DISPOSAL METHODS

The safety considerations in this experiment relate to the operation of a Bunsen burner and the handling of hot items. Blue burner flames are visible, but easily lost against a laboratory background. Be careful not to reach through a flame in picking up some object behind it. If you have long hair, tie it back so it does not get into the flame. Be sure to use crucible tongs in handling hot crucibles, including the lids. Laboratory hardware gets hot, too. Goggles should always be worn when working with chemicals, and particularly while heating them, as in this experiment. Be alert also to the possibility of hot chemicals ''shooting'' out of the test tube in Part 2.

The anhydrous solids should be discarded in a container or you should follow directions given by your instructor.

PROCEDURE

NOTE: Record all mass measurements in grams to the nearest 0.001 g.

1. Percentage Water in a Hydrate: Formula of a Hydrate

A. Heat a clean porcelain crucible and its lid—the ''container'' for this experiment (see pages 9 and 10)—on a clay triangle over a direct flame for 5 minutes to drive off any surface moisture. (See Figure 5.1, page 62.) When they are cool to the touch, weigh them on a milligram balance, recording this and subsequent masses in the ''constant mass'' portion of your data sheet. Heat the container to constant mass in 5-minute heating cycles until duplicate masses (within 0.005 g) are reached. Record the final (constant) mass as the mass of the container.

NOTE: Part 2 of the experiment may be performed during the cooling cycles of Part **1**.

B. Place 1 to 1.5 g of the unknown solid hydrate into the container, and weigh again on a milligram balance. Record as the mass of the container plus hydrate.

C. With the lid almost covering the crucible, heat the container and its contents, gently at first, and then with a hot flame for 10 minutes. Cool the crucible and lid, and when they feel cool to the touch, weigh them again. Record this and subsequent weighings in the ''Constant Mass Data'' portion of the work page. Heat to constant mass in 4- to 6-minute heating cycles until duplicate masses are reached. The final (constant) mass should be recorded as the mass of the container plus anhydrous salt.

D. Set the container and its contents aside while you complete your calculations. Do not discard the residue until your calculations are finished and satisfactory; if they are not satisfactory, it is possible that you may be able to salvage your work if the material is still on hand.

2. Behavior of a Hydrate

A. Place a few small crystals of $CoCl_2 \cdot 6H_2O$ into a test tube. Holding the test tube tilted at an angle, and with its mouth pointing away from you and all others, heat the test tube gently. Record your observations.

B. After the test tube has cooled to room temperature, add a few drops of water. Hold the test tube against the back of your hand. Record your observations.

CALCULATIONS

From the masses of the container, the container plus hydrate, and the container plus anhydrous salt, identify and perform the subtractions that will yield the mass of the hydrate and the mass of the water of hydration. From these, calculate the percentage water of hydration, using Equation 6.1.

Return to the original data (masses of container, container plus hydrate, and container plus anhydrous salt), and identify and perform the subtraction that will yield the mass of the anhydrous salt. The anhydrous salts resulting from dehydration of the different unknowns in this experiment are:

Unknown	*yields*	*Anhydrous Salt*
A		$BaCl_2$
B		$MgSO_4$
C		$CuSO_4$

Convert the grams of anhydrous salt to moles. Also convert grams of water of hydration to moles. Calculate the number of moles of water per mole of anhydrous compound; enter this result in the ''Ratio'' line in your table of results. From the ratio, determine the formula of the hydrate.

Name *Date* *Section*

Experiment 6

Advance Study Assignment

1. How can you make sure that all of the water of hydration has been removed? Explain.

2. Why do you have to cool the crucible before weighing it?

3. A 2.815-g sample of $CaSO_4 \cdot XH_2O$ was heated until all of the water was removed. Calculate the percentage water of hydration and the formula of the hydrate if the residue after heating weighed 2.485 g.

Name *Date* *Section*

Experiment 6

Work Page

Data and result tables for Experiment 6 appear on pages 78 and 79.

Part 1—Percentage Water in a Hydrate; Formula of a Hydrate

Constant Mass Data (Supply identification letters or numbers for unknowns)

Item	*Container*	*Unknown*	*Unknown*	*Unknown*	*Unknown*
1st heating (g)					
2nd heating (g)					
3rd heating (g)					
4th heating (g)					
Final heating (g)					

Mass Data

Container (g)				
Container + hydrate (g)				
Container + anhydrous salt (g)				

Results

(Show full calculations for one column at the top of the next page)

Mass of hydrate (g)				
Mass of water of hydration (g)				
Percentage water of hydration				
Mass of anhydrous salt (g)				
Formula of anhydrous salt				
Moles of anhydrous salt				
Moles of water of hydration				
Ratio: $\frac{\text{moles of water}}{\text{1 mole of anhydrous salt}}$*	$\frac{\quad}{1}$	$\frac{\quad}{1}$	$\frac{\quad}{1}$	$\frac{\quad}{1}$
Formula of hydrate				

*Express this ratio as a decimal number over 1 (e.g., $\frac{2.875}{1}$).

Name *Date* *Section*

Experiment 6

Work Page

Calculations for One Full Column of Results from Previous Page

Part 2—Behavior of a Hydrate

2A. Observations when a hydrate is heated in a test tube.

2B. Observations when water is added to an anhydrous salt.

Name *Date* *Section*

Experiment 6

Report Sheet

Data and result tables for Experiment 6 appear on pages 82 and 83

Part 1—Percentage Water in a Hydrate; Formula of a Hydrate

Constant Mass Data (Supply identification letters or numbers for unknowns)

Item	*Container*	*Unknown*	*Unknown*	*Unknown*	*Unknown*
1st heating (g)					
2nd heating (g)					
3rd heating (g)					
4th heating (g)					
Final heating (g)					

Mass Data

Container (g)				
Container + hydrate (g)				
Container + anhydrous salt (g)				

Results

(Show full calculations for one column at the top of the next page)

Mass of hydrate (g)				
Mass of water of hydration (g)				
Percentage water of hydration				
Mass of anhydrous salt (g)				
Formula of anhydrous salt				
Moles of anhydrous salt				
Moles of water of hydration				
Ratio: $\frac{\text{moles of water}}{\text{1 mole of anhydrous salt}}$*	$\frac{}{1}$	$\frac{}{1}$	$\frac{}{1}$	$\frac{}{1}$
Formula of hydrate				

*Express this ratio as a decimal number over 1 (e.g., $\frac{2.875}{1}$).

Name *Date* *Section*

Experiment 6

Report Sheet

Calculations for One Full Column of Results from Previous Page

Part 2—Behavior of a Hydrate

2A. Observations when a hydrate is heated in a test tube.

2B. Observations when water is added to an anhydrous salt.