1. Hydrates

Objective

Chemical compounds that contain discrete water molecules as part of their crystalline structure are called **hydrates**. Hydrates occur quite commonly among chemical substances, especially among ionic substances. More often than not, such compounds are either prepared in, or are recrystallized from, aqueous solutions. Hydrates exist for ionic compounds most commonly, but hydrates of polar and nonpolar covalent molecules are also known. In this experiment, you will study some of the properties and characteristics of several ionic hydrates.

Introduction

Hydrates are most commonly encountered in the study of metal salts, especially those salts of the *transition metals.* Water is bound in most hydrates in definite, *stoichiometric* proportions, and the number of water molecules bound per metal ion is often *characteristic* of a particular metal ion.

A very common hydrate often encountered in the general chemistry laboratory is copper(II) sulfate pentahydrate, $CuSO_4 \cdot 5H_2O$. The word "*penta*hydrate" in the name of this substance indicates that *five* water molecules are bound in this substance per copper sulfate formula unit. Hydrated water molecules are generally indicated in formulas as shown above for the case of the copper sulfate, using a *dot* to separate the water molecules from the formula of the salt itself.

Many hydrated salts can be transformed to the **anhydrous** (without water) compound by heat. For example, if a sample of copper sulfate pentahydrate is heated, the bright blue crystals of the hydrate are converted to the white, powdery, anhydrous salt.

$$\underset{\text{blue}}{CuSO_4 \cdot 5H_2O(s)} \rightarrow \underset{\text{white}}{CuSO_4(s)} + 5H_2O(g)$$

During the heating of copper sulfate pentahydrate, the water of crystallization is clearly seen escaping as steam from the crystals.

It is also possible to *reconstitute* the hydrate of copper sulfate: if water is added to the white anhydrous salt, the solid will reassume the blue color of the hydrated salt. Anhydrous salts are sometimes used as chemical drying agents, or **desiccants**, because of their ability to combine with and remove water from their surroundings. For example, most electronic equipment (and even at least one brand of ground coffee) comes packed with small envelopes of a desiccant to protect the equipment from moisture. Substances that absorb moisture and are able to be used as desiccants are said to be **hygroscopic.**

Not all hydrated salts are converted simply into the anhydrous compound when heated, however. Some hydrated metal salts will *decompose* upon losing the water of crystallization; subsequently they are usually converted to the metal *oxide* if the heating is carried out in air. Most covalent hydrates decompose rather than simply lose water when heated.

The water molecules contained within the crystals of a hydrate may be bound by several different means. For the case in which water molecules are bound with a metal salt, generally a nonbonding pair of electrons on the oxygen atom of the water molecule forms a coordinate covalent bond with empty, relatively low energy *d*-orbitals of the metal ion. In the case of copper sulfate pentahydrate, for example, four of the five water molecules form such coordinate bonds with the copper(II) ion. In other situations, the water molecules of the hydrate may be hydrogen-bonded to one or more species of the salt. This is especially common for covalently bonded hydrates.

SAFETY PRECAUTIONS

- **Wear safety glasses at all times while in the laboratory.**
- **Copper, cobalt, nickel, chromium, and barium compounds are all highly toxic. Wash hands after use.**
- **When you are heating the hydrated metal salts, they may *spatter* if heated too strongly. To avoid this, heat the solids with as *small* a flame as possible at first, and do not heat strongly with the full heat of the burner until most of the water has been driven from the hydrate. Make certain that the mouth of the test tube used to heat the hydrate is not pointed at yourself or anyone else.**
- **Dispose of the metal salts as directed by the instructor. Do not wash the salts down the drain, and do not place them in the wastebasket.**

Apparatus/Reagents Required

Nickel(II) chloride hexahydrate, cobalt(II) chloride hexahydrate, copper(II) sulfate pentahydrate, chromium(III) chloride hexahydrate, anhydrous calcium chloride

Procedure

Record all data and observations directly in your notebook in ink.

Determine the mass of a clean, dry casserole or small evaporating dish to the nearest milligram (0.001 g). Add to the casserole or evaporating dish a spatula tipful of copper(II) sulfate pentahydrate, $CuSO_4 \cdot 5H_2O$, and reweigh. Calculate the mass of $CuSO_4 \cdot 5H_2O$ taken. Record the appearance of the crystals.

Based on the mass of $CuSO_4 \cdot 5H_2O$ taken and its formula, calculate the *theoretical mass* of water that should be lost from the crystals if all the water were driven off.

Set up a wire gauze on a metal ring, and prepare to heat the casserole/evaporating dish in the burner flame. Begin the heating with a very small flame. If there is any evidence that the material is about to spatter, remove the heat immediately. Record any changes in appearance/color as the hydrate is heated.

When it is apparent that most of the water has been driven from the sample, increase the size of the flame. Stir the salt with a clean stirring rod until the sample is uniform in texture and appearance.

Remove the heat and allow the casserole to cool completely to room temperature. When the casserole has cooled completely, reweigh and calculate the mass of water driven off from the crystals. Using the theoretical mass loss calculated above, along with the experimentally determined mass loss, determine the percent error in your experiment.

After all mass determinations for the $CuSO_4 \cdot 5H_2O$ sample have been completed, add water dropwise to the sample. Record any changes in appearance/color.

For the hydrates listed below, first record the appearance of the crystals. Then transfer tiny amounts of the hydrates each to separate, clean borosilicate test tubes.

Using a test tube clamp to protect your hands, and making sure that the mouth of the test tube is not pointed at yourself or anyone else, heat each hydrate sample in turn and record any color changes or other changes in appearance that take place on heating. Allow the test tubes to cool completely to room temperature and then add a few drops of water to each test tube. Record any changes that take place on adding water. The hydrate samples to be used are: $NiCl_2 \cdot 6H_2O$; $CoCl_2 \cdot 6H_2O$; $CrCl_3 \cdot 6H_2O$.

As a vivid demonstration of the ability of anhydrous salts to absorb moisture, do the following: weigh an empty clean watch glass (to the nearest 0.01 g), then add about a teaspoon of *anhydrous* calcium chloride to the watch glass and reweigh. Examine the salt from time to time during the remainder of the lab period, and reweigh the watch glass and contents before leaving lab. Calcium chloride is an excellent desiccant and is able to absorb so much moisture from the air that it usually forms a solution of itself. A salt that absorbs such a great deal of water is said to be **deliquescent**. Calculate the mass of water absorbed by the anhydrous calcium chloride. Calculate what percentage of its own weight the $CaCl_2$ sample was able to absorb in moisture during the lab period.

Hydrates

Date:	....	Student name:	....
Course:	....	Team members:	....
Section:	....		....
Instructor:	....		....

Prelaboratory Questions

1. Use a handbook of chemical data to find the number of water molecules bound per formula unit in the common hydrates of the following salts:

   Strontium chloride, $SrCl_2$ ....

   Sodium chromate, Na_2CrO_4 ....

   Nickel(II) nitrate, $Ni(NO_3)_2$ ....

   Iron(II) ammonium sulfate, $Fe(NH_4)_2(SO_4)_2$ ....

2. As described in the introduction to this experiment, when copper(II) sulfate pentahydrate is heated, the deep *blue* color of the hydrate changes to the *white* color of the anhydrous salt. Use the sections of your textbook discussing the chemistry of the transition elements to determine why such a vivid change in color is common when such elements' hydrated compounds are heated.

3. Suppose 2.3754 g of copper(II) sulfate pentahydrate is heated to drive off the water of crystallization. Calculate what weight of anhydrous salt will remain.

4. In $CuSO_4 \cdot 5H_2O$, it was mentioned that four of the five water molecules held per formula unit of the salt were attached by coordinate covalent bonds to the copper ion. The fifth water molecule is attached to the sulfate ion, but by a different mechanism. Use your textbook or a chemical encyclopedia to determine how a water molecule might be bonded to a sulfate ion.

Hydrates

Date: ....
Course: ....
Section: ....
Instructor: ....

Student name: ....
Team members: ....
....
....

Results/Observations

Copper(II) sulfate pentahydrate

Mass of empty casserole/evaporating dish, g ....

Mass of casserole/evaporating dish plus $CuSO_4 \cdot 5H_2O$, g ....

Mass of $CuSO_4 \cdot 5H_2O$ taken, g ....

Appearance of $CuSO_4 \cdot 5H_2O$ ....

Theoretical mass loss expected on heating $CuSO_4 \cdot 5H_2O$ ....

Mass of casserole/evaporating dish after heating, g ....

Mass of water lost from $CuSO_4 \cdot 5H_2O$ crystals, g ....

% error in mass water lost from $CuSO_4 \cdot 5H_2O$, g ....

Appearance of $CuSO_4$ after heating ....

Appearance of $CuSO_4$ on adding water ....

Nickel(II) chloride hexahydrate

Observation before heating ....

Observation after heating ....

Observation on adding water ....

Cobalt(II) chloride hexahydrate

Observation before heating ....

Observation after heating ....

Observation on adding water ....

Chromium(III) chloride hexahydrate

Observation before heating ....

Observation after heating ....

Observation on adding water ....

Barium chloride dihydrate

Observation before heating ....

Observation after heating ....

Observation on adding water ....

Calcium chloride (anhydrous)

Observation on absorbing moisture from air ....

Mass of empty watch glass, g ....

Mass of watch glass plus anhydrous $CaCl_2$, g ....

Mass of watch glass plus $CaCl_2$ on standing, g ....

Mass of water absorbed, g ....

Percent water absorbed ....%

Questions

1. Use a chemical dictionary or your textbook to distinguish between the terms *desiccant, hygroscopic,* and *deliquescent.*

2. Sugars and starches belong to a class of biological compounds called *carbohydrates,* indicating that the general formula for such compounds is of the sort $(CH_2O)_n$. Use your textbook to find out why such compounds are not really hydrates of carbon as the family name suggests and record your findings here.