

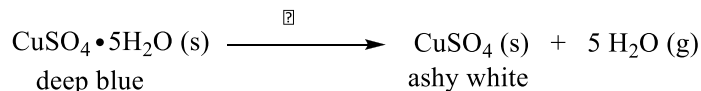
Hydrates

INTRODUCTION

Water, the most common chemical on earth, can be found in the atmosphere as water vapor. Some chemicals, when exposed to water in the atmosphere, will reversibly either adsorb it onto their surface or include it in their structure forming a complex in which water generally bonds with the cation in ionic substances. The water present in the latter case is called *water of hydration* or *water of crystallization*. Common examples of minerals that exist as hydrates are gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), Borax ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$), and Epsom salts ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$). Hydrates generally contain water in stoichiometric amounts; hydrates' formulas are represented using the formula of the anhydrous (non-water) component of the complex followed by a dot then the water (H_2O) preceded by a number corresponding to the ratio of moles of H_2O per mole of the anhydrous component present. They are typically named by stating the name of the anhydrous component followed by the Greek prefix specifying the number of moles of water present then the word *hydrate* (example: $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ is magnesium sulfate heptahydrate).

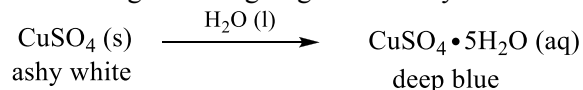
Properties of Hydrates

It is generally possible to remove the water of hydration by heating the hydrate. The residue obtained after heating, called the anhydrous compound, will have a different structure and texture and may have a different color than the hydrate.



Properties of Anhydrous compound from a hydrate

- Highly soluble in water
- When dissolved in water, the anhydrous compound will have a color similar to that of the original hydrate even if it had changed color going from the hydrate to the anhydrous compound.



Most hydrates are stable at room temperature. However, some spontaneously lose water upon standing in the atmosphere; they are said to be *efflorescent*.

Other compounds can spontaneously absorb water from the surrounding atmosphere, they are said to be *hygroscopic*. Some hygroscopic substances, such as P_2O_5 and anhydrous CaCl_2 , are widely used to “dry” liquids and gases; they are referred to as desiccants. Other hygroscopic substances, such as solid NaOH , absorb so much water from the atmosphere that they dissolve in this water; these substances are said to be *deliquescent*.

Some compounds like carbohydrates release water upon heating by decomposition of the compound rather than by loss of the water of hydration. These compounds are NOT considered true hydrates as the hydration process is not reversible.

Formula of a Hydrate (Anhydrous Solid $\cdot x\text{H}_2\text{O}$)

The formula of a hydrate can be determined by dehydrating a known mass of the hydrate, then comparing the masses of the original hydrate and the resulting anhydrous solid. The mass of water evaporated is obtained by subtracting the mass of the anhydrous solid from the mass of the original hydrate (Eqn. 1).

$$m_{\text{H}_2\text{O}} = m_{\text{Hydrate}} - m_{\text{AnhydrousSolid}} \quad (1)$$

From the masses of the water and anhydrous solid and the molar mass of the anhydrous solid (the formula of the anhydrous solid will be provided), the number of moles of water and moles of the anhydrous solid are calculated as shown below (Eqns. 2 , 3):

$$n_{H_2O} = \frac{m_{H_2O}}{MM_{H_2O}} \quad (2)$$

$$n_{AnhydrousSolid} = \frac{m_{AnhydrousSolid}}{MM_{AnhydrousSolid}} \quad (3)$$

where n is number of moles, m the mass, and MM the molar mass of each substance.

In order to determine the formula of the hydrate, $[Anhydrous\ Solid \cdot xH_2O]$, the number of moles of water (x) per mole of anhydrous solid will be calculated by dividing the number of moles of water by the number of moles of the anhydrous solid (Eqn. 4).

$$x = \frac{n_{H_2O}}{n_{AnhydrousSolid}} \quad (4)$$

Name _____

Date _____

Pre-lab Assignment: Hydrates

MUST be completed before an experiment is started. The COPY pages will be collected as you enter the lab. Show all your work. Report to correct number of significant figures and include appropriate units!

Q1. In an experiment, 2.3754 g of copper(II) sulfate pentahydrate is heated to drive off all the water of crystallization.

a) Write a balanced equation.

b) Determine the mass of anhydrous salt that remains. Show your calculations.

Q2. Cobalt (II) chloride is commonly obtained from chemical supply houses as a hydrate with the formula $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$. What is the percent water by weight in this hydrate?

Q3. A hydrate of sodium phosphate, Na_3PO_4 , contains 49.7% water by weight.

a) How many grams of water and how many grams of anhydrous Na_3PO_4 are in 1000. grams of this sample?

b) In this same 1000.-gram sample, how many moles of water and how many moles of anhydrous Na_3PO_4 , are present?

c) What is the formula of the hydrate?

EXPERIMENTAL PROCEDURE

Safety: Use caution when heating the crucible and cover. A hot crucible looks like a cold one, avoid direct contact with the crucible, clay triangle and ring stand until you are sure they are cooled.

A) Reversibility of hydration (Demonstration by the Instructor)

In this section we will demonstrate the dehydration and re-hydration of cobalt(II) chloride hexahydrate. When heated gently, the red burgundy $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ will decompose into the violet $\text{CoCl}_2 \cdot 2\text{H}_2\text{O}$ then to the blue anhydrous CoCl_2 . When this anhydrous compound is dissolved in water it will go back to the original red burgundy color.

- 1) In an evaporating dish, gently heat a small amount (0.1 – 0.2 g) of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ crystals until its color changes to violet then to blue.
- 2) When this color change appears to be complete, add 3 to 5 mL of water and observe the color of the dissolved substance.
- 3) Then reheat the solution to dryness.

B) Hygroscopic and Efflorescent Solids (Demonstration by the Instructor)

In this section you will observe the changes in the physical properties of compounds, including wetness, color, structure, texture and mass. You should then decide if the compound is hygroscopic, efflorescent, or neither using the change in the mass of the substance.

- 1) On an analytical balance, weigh a pea-sized sample of each of the compounds below on separate weighing paper. Record the values as initial masses of containers and samples.
- 2) Label and place all samples at the same location in the room, well out of the way so they won't be spilled. After one hour, note any change in the physical appearance of each sample.
- 3) Weigh the samples and record the masses as final masses.
- 4) Calculate the change in mass for each sample. A substance is classified as efflorescent if its mass decreases by 0.005 g or more; and it is classified as hygroscopic if its mass increases by 0.005 g or more.

Compounds to be tested: $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$, FeCl_3 , $\text{KAl}(\text{SO}_4)_2$, CaCl_2 , CuSO_4 .

C) Identification of hydrates

In this section you will try to determine through the testing of a series of compounds, which ones are true hydrates. Some compounds may possess some of the properties of hydrates without being true hydrates. For a compound to be a true hydrate, it has to show all properties of true hydrates, *including evolution of water upon heating, solubility of its anhydrous residue in water and reversibility in the color of the residue back to the color of the hydrate when dissolved in water.*

- 1) For each of the chemical compounds below, place a pea-sized (≈ 30 mg) amount of the compound (just enough to cover the bottom of the test tube) in a dry test tube and note its color.
- 2) Heat the test tube and note any condensation that may appear at the mouth of the test tube as evidence of dehydration, note the color of the residue.
- 3) Let the residue cool down (put the test tube in a beaker not on a plastic test tube rack) then try to dissolve in about 3 mL of water (about 1/3 of the small test tube), warming gently if necessary to dissolve the residue (dissolve only substances that have shown condensation).
- 4) Note the color of the dissolved residue. If the compound possesses all three of the above-mentioned properties, it is a true hydrate; if at least one of them is not present, the compound is not.

Compounds to be tested: Nickel(II) chloride, Cobalt(II) chloride, Calcium Carbonate, Barium

chloride, Sodium tetraborate, Potassium chloride.

D) Determination of the formula of a hydrate

In this section you will determine the number of moles of water present per mole of anhydrous solid in a given hydrate.

- 1) Clean your crucible and its cover by rinsing them with distilled water and drying them with a towel.
- 2) Place the crucible with its cover slightly open on a clay triangle.
- 3) Heat gently with a Bunsen burner flame for a minute or two, then strongly to redness for three minutes.
- 4) Place the crucible and cover on the fiberglass pad to cool down to room temperature (do not set the hot crucible on the bench top). Weigh the crucible with its cover.
- 5) Handling the crucible and its cover with clean tongs, you add about one gram of the unknown hydrate. Record the mass of the crucible, cover and sample.
- 6) Place the crucible containing the hydrate with its cover slightly open to allow the water of hydration to escape. Heat the crucible gently for 10 minutes. *You should fan the burner (i.e., move or wave the flame under the crucible using the cooler outer core of the blue flame to slowly start heating the hydrate.*
- Do NOT use a yellow flame or it will deposit soot on your crucible, affecting your measurements.**
- 7) After 10 minutes of slowly increasing the heating, start heating the crucible to red glow for 5 minutes. Place the crucible and cover on the fiberglass pad.
- 8) Center the cover on the crucible and let it cool to room temperature. (It takes about 15 minutes). Weigh and record the mass of the cooled crucible with its cover and content (anhydrous residue).
- 9) Reheat the crucible, with the cover on, to red hot for 5 minutes. This is to make sure that you have driven off all the water.
- 10) Let the crucible cool to room temperature. Weigh and record the mass of the cooled crucible with its cover and content. If you get consistent mass value as the first weighing, all water has been driven off the sample. If the results of your calculations suggest that you have some water left in the residue, reheat your sample for an additional 5 minutes, allow it to cool down and weigh it again.
- 11) *When you have completed the experiment, dissolve all your heated residues in small amount of tap water, put all solids and liquids in the waste container.*

Hydrates

Name _____

Date _____

Partner's Name _____

A) Reversibility of Hydration

Summarize your observations on $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$:

Is the dehydration and hydration of CoCl_2 reversible?

B) Hygroscopic and Efflorescent Solids

Substance	Mass (sample + watch glass)		Observations	Conclusion
	Initial	Final		
$\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$	_____	_____	_____	_____
FeCl_3	_____	_____	_____	_____
$\text{KAl}(\text{SO}_4)_2$	_____	_____	_____	_____
CaCl_2	_____	_____	_____	_____
CuSO_4	_____	_____	_____	_____

C) Identification of hydrates

Substance	H_2O evolved in test tube?	Color of residue	Water soluble?	Hydrate or not?
NiCl_2	_____	_____	_____	_____
CoCl_2	_____	_____	_____	_____
CaCO_3	_____	_____	_____	_____
BaCl_2	_____	_____	_____	_____
$\text{Na}_3\text{B}_4\text{O}_7$	_____	_____	_____	_____

KCl _____

D. Determination of the formula of a hydrate

- 1) Mass of crucible and cover _____ g
- 2) Mass of crucible, cover, and solid hydrate _____ g
- 3) Mass of crucible, cover, and anhydrous solid
Heat #1 _____ g Heat #2 _____ g Heat #3 _____ g
- 4) Formula of compound: _____ (given on the label)

Calculations (Show your work)

- 5) Mass of solid hydrate _____ g
- 6) Mass of anhydrous residue _____ g
- 7) Mass of water lost _____ g
- 8) Mass percent of water in the unknown hydrate _____ g
- 9) Moles of anhydrous residue _____ moles
- 10) Moles of water of hydration _____ moles
- 11) Number of moles of water per mole of anhydrous residue _____
- 12) Formula of the hydrate: _____

Write the balanced equation to show the process of dehydration:

Unknown number: _____

Post-Lab Questions And Exercises
MUST be completed after the completion of the experiment
(Show all your work. Report to correct number of significant figures and include units!)

- Q1. In an experiment, 5.367 g of $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ is heated to drive off all the water of crystallization.
- Write a balanced equation.
 - Determine the mass of anhydrous salt that remains. Show your calculations.
- Q2. What is the percent water by weight of lithium perchlorate trihydrate?
- Q3. A hydrate of magnesium sulfate, contains 51.1% water by weight.
- How many grams of water and how many grams of anhydrous MgSO_4 are in 127.2 grams of this sample?
 - In this same 127.2 grams sample, how many moles of water and how many moles of anhydrous MgSO_4 , are present?
 - What is the formula of the hydrate?
- Q4. In what way does this experiment illustrate the difference between a hydrate and a compound that is simply wet?