Objectives

- Identification of hydrates in a group of compounds
- Investigation of the properties of hydrates
- Determination of the number moles of water of hydration in a hydrate

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 (\(\ce{CaSO4*2H2O}\)), Borax (\(\ce{Na3B4O7*10H2O}\)) and Epsom salts (\(\ce{MgSO4*7H2O}\)). Hydrates generally contain water in stoichiometric amounts; hydrates' formulae are represented using the formula of the anhydrous (non-water) component of the complex followed by a dot then the water (\(\ce{H2O}\)) preceded by a number corresponding to the ratio of \(\ce{H2O}\) moles 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: \(\ce{MgSO4*7H2O}\): magnesium sulfate heptahydrate).

Properties of Hydrates

It is generally possible to remove the water of hydration by heating the hydrate. Le Chatelier’s principle predicts that an addition of heat to an endothermic reaction (heat is a "reactant") will shift the reaction to the right (product side). Heating will shift the equation of dehydration below to the right since it is an endothermic reaction. 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.

Example:

\[
\text{\underbrace{\ce{CuSO4*5H2O (s)}}_{	ext{Deep Blue}}} \rightarrow \text{\underbrace{\ce{CuSO4 (s)}}_{	ext{Ashy White}}} + 5 \text{\ce{H2O (g)}} \label{1}
\]

\[
\text{\underbrace{\ce{CuSO4 (s)}}_{	ext{Ashy White}}} \rightarrow \text{\underbrace{\ce{CuSO4 (aq)}}_{	ext{Deep Blue}}} \label{2}
\]

Any anhydrous compound from a hydrate generally has the following properties:

- 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 \(\ce{P2O5}\) and anhydrous \(\ce{CaCl2}\), are widely used to "dry" liquids and gases (see experiment on the Molecular Weight of \(\ce{CO2}\)); they are referred to as desiccants. Other hygroscopic
substances, such as solid $\text{NaOH}$, 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 ($\text{Anhydrous Solid} \cdot x \cdot \text{H}_2\text{O}$)**

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 ($\ref{3}$):

$$m_{\text{H}_2\text{O}} = m_{\text{Hydrate}} - m_{\text{Anhydrous Solid}} \label{3}$$

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 ($\ref{4}$, $\ref{5}$):

$$n_{\text{H}_2\text{O}} = \frac{m_{\text{H}_2\text{O}}}{MM_{\text{H}_2\text{O}}} \label{4}$$

$$n_{\text{Anhydrous Solid}} = \frac{m_{\text{Anhydrous Solid}}}{MM_{\text{Anhydrous Solid}}} \label{5}$$

In order to determine the formula of the hydrate, \[
(\text{Anhydrous Solid} \cdot x \cdot \text{H}_2\text{O})\], the number of moles of water per mole of anhydrous solid ($x$) will be calculated by dividing the number of moles of water by the number of moles of the anhydrous solid (Equation $\ref{6}$).

$$x = \frac{n_{\text{H}_2\text{O}}}{n_{\text{Anhydrous Solid}}} \label{6}$$

**Procedure**

**Materials and Equipment**

*Solutions:* Nitric Acid (6M)
*Solids:* Nickel (II) chloride, Cobalt (II) chloride, Sucrose, Calcium Carbonate, Barium chloride, Sodium tetraborate, Potassium chloride, Sodium sulfate hydrate, Iron (III) chloride, Potassium aluminum sulfate, Calcium chloride, Copper sulfate, Unknown.

*Materials:* Tests tubes (small or medium size), watch glasses, crucible* and cover*, crucible tongs, clay triangle.

**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. Use crucible tongs when cleaning the crucible with concentrated nitric acid.
Part A: Reversibility of hydration (optional, by Instructor)

In this section we will demonstrate the dehydration and re-hydration of cobalt (II) chloride hexahydrate. When heated gently, the red burgundy $\ce{CoCl2*6H2O}$ will decomposes into the violet $\ce{CoCl2*2H2O}$ then to the blue anhydrous $\ce{CoCl2}$). 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 (0.3 – 0.5 g) of $\ce{CoCl2*6H2O}$ 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.

Part B: Hygroscopic and Efflorescent Solids

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 clean and dry watch glasses. Record the values as initial masses of containers and samples. Label and place all samples at the same location in the room, well out of the way so they won't be spilled.
2. After one hour, note any change in the physical appearance of each sample. Weigh the samples and record the masses as final masses. 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: $\ce{Na2SO4*10H2O}$, $\ce{FeCl3}$, $\ce{KAl(SO4)2}$, $\ce{CaCl2}$, $\ce{CuSO4}$.

Part 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). 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, Sucrose, Calcium Carbonate, Barium chloride, Sodium tetraborate, Potassium chloride.

Part D: Determination of the formula of a hydrate

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

1. Using crucible tongs, clean a porcelain crucible and its cover using concentrated nitric acid (6 M). Pour the used nitric acid in the waste container provided. Rinse the crucible and its cover with distilled water.

2. Set the crucible with its cover slightly open on a clay triangle and heat strongly for at least 10 minutes. Allow the crucible to cool down to room temperature (do not set the hot crucible on the bench top). Weigh the crucible with its cover to the nearest 0.001 g.

3. Making sure to handle the crucible and its cover with clean tongs, add about 1 g (weighed to the nearest 0.001 g) of the unknown hydrate. Record the mass of the crucible, cover and sample. Heat the content of the crucible with its cover slightly open to allow the water of hydration to escape, first gently (about 10 minutes), then strongly (about 5 minutes).

4. Center the crucible’s cover and let it cool down to room temperature. Weigh and record the mass of the cooled crucible with its cover and content (anhydrous residue).

5. Save the residue and perform your calculations. 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.

When you have completed the experiment, dissolve all your heated residues in water, put all your solids and liquids in the waste crock.
Pre-laboratory Assignment: Properties of Hydrates

1. A student trying to determine if a white solid is a true hydrate heats the sample and finds that there is evolution of water, that the residue obtained is soluble in water and that the solution is colorless. Is this a true hydrate? Explain.

2. A student is given a cobalt (II) chloride hydrate. He weighs a clean and dry crucible with its cover and records a mass of 18.456 g. He then weighs the sample in the crucible and cover and obtains a mass of 19.566 g. He heats the sample, allows it to cool to room temperature and reweighs it to obtain a mass of 19.062 g. In the process, the sample’s color changed from red-burgundy to blue.

   • Mass of hydrate:
   • Mass of anhydrous \(\ce{CoCl2}\):
   • Mass of water driven off:
   • Moles of water:
   • Moles of anhydrous \(\ce{CoCl2}\):
   • Moles of water per mole of \(\ce{CoCl2}\):
   • Formula of hydrate:

3. Why did the color of the hydrate change?

4. What color would you expect to see when this student dissolves the blue residue in water at the end of the experiment?
Lab Report: Properties of Hydrates

Part A: Reversibility of Hydration (Optional)

Record your observations:

Part B: Hygroscopic and Efflorescent Solids

<table>
<thead>
<tr>
<th>Substance</th>
<th>Initial mass of container and sample</th>
<th>Final mass of container and sample</th>
<th>Change in mass</th>
<th>Observations on structure, texture, wetness, etc. ...</th>
<th>Conclusion</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 (\text{CaCl}_2)</td>
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<tr>
<td>2 (\text{Na}_2\text{SO}_4*10\text{H}_2\text{O})</td>
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<tr>
<td>3 (\text{KAl(SO}_4)_2*12\text{H}_2\text{O})</td>
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<td>4 (\text{CuSO}_4)</td>
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<td>5 (\text{FeCl}_3)</td>
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Part C: Hydrates

<table>
<thead>
<tr>
<th>Substance</th>
<th>Initial color</th>
<th>Water upon heating (Y/N)</th>
<th>Color Residue</th>
<th>Residue soluble (Y/N)</th>
<th>Color of residue dissolved</th>
<th>Hydrate (Y/N)</th>
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</thead>
<tbody>
<tr>
<td>1 Nickel (II)</td>
<td>chloride</td>
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<tr>
<td>2 Cobalt (II)</td>
<td>chloride</td>
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<tr>
<td>Substance</td>
<td>Initial color</td>
<td>Water upon heating (Y/N)</td>
<td>Color Residue</td>
<td>Residue soluble (Y/N)</td>
<td>Color of residue dissolved</td>
<td>Hydrate (Y/N)</td>
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<tr>
<td>3 Sucrose</td>
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<td>4 Calcium carbonate</td>
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<td>5 Barium chloride</td>
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<td>6 Sodium tetraborate</td>
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<tr>
<td>7 Potassium chloride</td>
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Part D: Determination of the formula of a hydrate

Data
1. Mass of crucible and cover:
2. Mass of crucible, cover and solid hydrate:
3. Mass of crucible, cover and anhydrous solid:

Calculations
1. Mass of hydrate:
2. Mass of anhydrous solid:
3. Mass of water lost:
4. Formula of anhydrous solid (from Instructor):
5. Molar mass of anhydrous solid:
6. Moles of $\text{H}_2\text{O}$ present in the hydrate:
7. Moles of anhydrous solid present:
8. Ratio of moles $\text{H}_2\text{O}$:Anhydrous solid $= \langle x \rangle$:
9. Formula of hydrate $[\langle \text{Anhydrous solid} \rangle \text{ce}^{\langle x \rangle} \text{ce}\{\text{H}_2\text{O}\}]$:
10. Name this compound:
11. Unknown ID:

**Questions:**

1. Did the compound(s) that appeared wet in section B lose or gain water? Explain what may have happened.

2. What will be the effect, on the mass of the residue, of overheating the hydrate so that the compound decomposes. Will this likely lead to a higher or lower value of \(x\) than the actual value?

3. What will be the effect, on the mass of the residue, of not heating the hydrate enough to drive off all the water of hydration in the hydrate. Will this likely lead to a higher or lower value of \(x\) than the actual value?