Properties of Hydrates

Objectives

The objectives of this laboratory are as follows:

- To learn how to identify any hydrates in a group of compounds,
- To investigate some of the properties of hydrates, and
- To experimentally determine the number moles of water of hydration in an unknown hydrate.

Background

Water, the most common chemical on earth, can be found in the atmosphere as water vapor. When exposed to this water vapor, some chemicals will reversibly either adsorb it onto their surface, or include it in their structure forming a complex. In the latter case, the complex is formed when water molecules bond with the cation in ionic substances, yielding a hydrate. This water is called the water of hydration (or water of crystallization). Common examples of compounds that exist as hydrates include minerals such as gypsum (CaSO$_4$•2H$_2$O), Borax (Na$_3$B$_4$O$_7$•10H$_2$O) and Epsom salts (MgSO$_4$•7H$_2$O).

Hydrates generally contain water in fixed stoichiometric amounts. The formulae of hydrates are represented using the formula of the anhydrous (non-water) component of the complex, followed by a dot, then finally the water (H$_2$O) preceded by a number corresponding to the ratio of H$_2$O moles per mole of the anhydrous component present. They are named systematically by stating the name of the anhydrous component, followed by the appropriate Greek prefix specifying the number of moles of water present, then followed by the word hydrate. For example, MgSO$_4$•7H$_2$O is named magnesium sulfate heptahydrate.

Properties of Hydrates

It is usually possible to remove the water of hydration by heating the hydrate. The residue obtained after heating is called the anhydrous salt, and will have a different structure, texture and possibly different color than the original hydrate:

hydrate $\xrightarrow{\text{anhydrous salt}}$ anhydrous salt

CuSO$_4$•5H$_2$O (s) $\xrightarrow{\Delta}$ CuSO$_4$ (s) + 5 H$_2$O (g)

blue solid crystals white solid powder

Anhydrous salts derived from hydrates are all highly soluble in water. Additionally, when dissolved in water the anhydrous salt will have a color similar to that of the original hydrate, even if it had changed color going from the hydrate to the anhydrous salt:

CuSO$_4$ (s) $\xrightarrow{\text{water}}$ CuSO$_4$ (aq)

white solid powder blue solution

Most hydrates are stable at room temperature. However, some spontaneously lose water upon standing in the atmosphere; they are said to be efflorescent. Others can spontaneously absorb water from the surrounding atmosphere; they are said to be hygroscopic. Some hygroscopic compounds, such as P$_2$O$_5$ and CaCl$_2$, are widely used to “dry” liquids and gases; they are
referred to as *desiccants*. Other hygroscopic compounds, such as solid NaOH, absorb so much water from the atmosphere that they dissolve in this water; they are said to be *deliquescent*. Finally, it should be noted that some compounds, such as 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 process is not reversible.

**Formula of a Hydrate**

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 (1):

\[ m_{\text{water}} = m_{\text{hydrate}} - m_{\text{anhydrous solid}} \] (1)

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

\[ n_{\text{water}} = \frac{m_{\text{water}}}{MM_{\text{water}}} \] (2)
\[ n_{\text{anhydrous solid}} = \frac{m_{\text{anhydrous solid}}}{MM_{\text{anhydrous solid}}} \] (3)

Finally, in order to determine the formula of the hydrate, \([\text{Anhydrous Solid} \cdot x \text{H}_2\text{O}]\), 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 (4):

\[ x = \frac{n_{\text{water}}}{n_{\text{anhydrous solid}}} \] (4)

**Procedure**

**Chemicals/Materials**

Nickel(II) chloride, cobalt(II) chloride, sucrose, calcium carbonate, barium chloride, sodium tetraborate, potassium chloride, copper(II) sulfate, sodium sulfate, iron(III) chloride, potassium aluminum sulfate, calcium chloride, and an unknown hydrate.

**Equipment**

Tests tubes (8 small), test tube holder, clay triangle, 4 watch glasses, crucible tongs, crucible and cover*, Bunsen burner, analytical balance, stand and ring clamp.

*Items with an asterisk may have be checked out from the stockroom

**Safety**

In Part C – note that a hot crucible looks like a cold one, so avoid direct contact with the crucible, clay triangle and ring stand until you are sure they have completely cooled after heating in the Bunsen burner flame.
Part A: Identification of Hydrates

Compounds to be tested: nickel(II) chloride, cobalt(II) chloride, sucrose, calcium carbonate, barium chloride, sodium tetraborate, potassium chloride, copper(II) sulfate.

1. Place a pea-sized amount (≈ 30 mg, just enough to cover the bottom of the test tube) of each of the compounds above in a small dry test tube and note its color. Heat the test tube and sample gently. Look for condensation appearing at the mouth of the test tube as evidence of dehydration, and note the color of the residue.

2. For only those substances that show condensation: Allow the residue cool down (put the test tube in a beaker, not on a plastic test tube rack), then try to dissolve it in about 5 mL of water (fill about ½ of the test tube). It may be necessary to gently warm the test tube to dissolve the residue. Note the color of the dissolved residue.

3. Based on your observations, determine which of the compounds tested are hydrates. Note that for a compound to be a hydrate, it must display all the properties of hydrates.

Part B: Hygroscopic and Efflorescent Solids

Compounds to be tested: sodium sulfate, iron(III) chloride, potassium aluminum sulfate, and calcium chloride.

1. On an electronic balance, weigh a pea-sized sample of each of the compounds above on separate clean, 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 (including wetness, color, texture) of each sample. Weigh the samples and record the masses as final masses. Calculate the change in mass for each sample.

3. Based on your results, determine whether each compound investigated is hygroscopic, efflorescent or neither. A substance is classified as efflorescent if its mass decreases by 0.005 g or more; it is classified as hygroscopic if its mass increases by 0.005 g or more.

Part C: Determination of the Formula of a Hydrate

1. Your instructor will provide you with a sample of an unknown hydrate to analyze. Record its identification code on your report form.

2. Obtain a crucible and lid from the stockroom. Make sure that no hair-line cracks are visible in these items, and then rinse them thoroughly with distilled water.

3. Using your crucible tongs, hold the crucible (right-side up) in a Bunsen burner flame and heat it strongly for 6 minutes. Then set it down on the metal base of the ring stand to cool. Do not set the hot crucible down on the bench top – it will get scorched! Repeat this process for the crucible lid. Once cooled down to room temperature, weigh the crucible and lid using an electronic balance to the nearest 0.001 g.
4. Add approximately 1 g (weighed to the nearest 0.001 g) of the unknown hydrate to your crucible. **Do not do this over the balance – the sample may spill on the balance and destroy it!** Record the mass of the crucible, lid and hydrate.

5. Assemble your stand, ring clamp, clay triangle and crucible as shown in the figure below. Heat the crucible and hydrate with the **lid slightly open** to allow the water of hydration to escape – first gently (about 10 minutes), then strongly (about 6 minutes). Then **close the lid** (completely covering the crucible) and let it cool down to room temperature. Weigh and record the mass of the cooled crucible, lid and contents (anhydrous residue).

![Diagram of crucible, lid, ring clamp, and clay triangle with a burner and gas outlet.]

6. Now reheat your sample strongly for an additional 6 minutes (lid slightly open), then allow it to cool down (lid closed) and weigh it again. The mass after this second heating should agree to within 0.050 grams of the first heating. If it does not, you must reheat your sample a third time for another 6 minutes.

7. Use your measurements to determine the number of moles of water present per mole of anhydrous solid in your given hydrate.

**Clean-Up**

When you have completed the experiment, dissolve all your residues in water and pour them into the waste container provided. Then rinse your equipment thoroughly with distilled water.