Percent Water in a Hydrate

Cautions

Unknown hydrated salts, Nitric Acid, Hydrochloric Acid, hot porcelain, flames

Purpose

The purpose will be to determine the percent of water by weight in a hydrated salt along with establishing the formula of the hydrated compound.

Introduction

Many natural and commercial salts are hydrated salts. A ***hydrated salt*** (or hydrate) is a salt which has a number of water molecules associated with the ions within its crystalline structure. These water molecules maybe referred to as the waters of crystallization or water of hydration.

In most cases there is a fixed number of water molecules associated with each mole of salt. The formula for a hydrated salt is written as the formula for the anhydrous salt followed by a dot and the appropriate number of water molecules. For example, the formula for cobalt(II) chloride hexahydrate is $CoCl_2 \bullet 6H_2O$, which indicates that 6 waters of hydration are present for each 1 mole of $CoCl_2$ salt.

Some hydrated salt compounds lose their waters of hydration easily when exposed to the atmosphere, a process known as ***efflorescence***. Epsom salt (magnesium sulfate heptahydrate, $MgSO_4 \bullet 7H_2O$ (s)) will effloresce when placed on a bench top. Certain anhydrous salts will gain waters of hydration easily by simple exposure to the atmosphere. ***Hygroscopic substances*** readily absorb water from the atmosphere. Finally, ***deliquescent salts*** are hygroscopic salts that can absorb so much water from the atmosphere a solution forms. Calcium chloride is an example of a deliquescent salt.

$$\mathbf{CaCl_2\ (s) + 6H_2O\ (l) \rightarrow CaCl_2 \cdot 6H_2O\ (l)}$$

In some cases the water is loosely bound to the salt allowing the water to be easily removed by applying heat to form the ***anhydrous salt***, meaning without water.

$$\mathbf{CoCl_2 \cdot 6H_2O\ (s) \rightarrow CoCl_2\ (s) + 6H_2O\ (g)}$$

In other cases, the salt binds the waters so tightly that decomposition occurs before the water is removed. Nitrate salts have this tendency; if too much heat is applied to the hydrate, the nitrate portion decomposes into nitrogen oxides before the waters are removed. In this experiment, the salts investigated will not decompose before the waters are removed.

The mass percentage of water in a hydrate can be determined by heating a known amount of a sample until complete dehydration is accomplished.

Hydrated Salt + Heat → Anhydrous Salt + Water

$$\mathbf{CoCl_2 \cdot 6H_2O\ (s) + Heat \rightarrow CoCl_2\ (s) + 6H_2O\ (g)}$$

Upon heating, the mass of solid will decrease as the water is liberated. This makes it possible to determine the amount of water that was present in the initial salt sample.

Total mass of hydrated salt = mass of the anhydrous salt + mass of waters of hydration **(*Eqn 1*)**

Once the mass of water is known, the percent mass of water in the hydrate may be calculated using the equation:

$$\%\,water = \frac{mass\ of\ waters\ of\ hydration}{total\ mass\ of\ hydrated\ salt} \times 100 \qquad \textbf{(}\textit{Eqn 2}\textbf{)}$$

In this experiment, the formula of a hydrated salt will be determined by heating a hydrate in a crucible until the mass remains constant. Once the formula is known, the mass percent water in the compound will be calculated.

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Procedure

To complete 2 trials in one lab period, your instructor may require you to perform both trials simultaneously.

Figure 1: Set-up for heating a crucible.
web.centre.edu/che/ che131_lab/Exp3crucible.jpg

1. Obtain a crucible and lid. Clean the crucible and check for any stress cracks, fractures, or fissures. These types of defects are common in used crucibles. If the crucible is dirty then move the apparatus to a hood and add 1-2 mL of 6M HNO_3 and gently evaporate to dryness then inspect the crucible after cooling for any defects. If no defects are found the crucible and lid should be supported on a clay triangle. See Figure 1.
2. Record an initial mass of the crucible and lid.
3. Heat the crucible gently for 5 minutes before heating with an intense flame. Continue heating for 10 to 12 minutes with an intense flame after the bottom of the crucible has become red. Allow the crucible to cool on the clay triangle (do not set the crucible on the bench top due to contamination possibility or cracking.).
4. Determine the mass of the "fired" cool crucible and lid and record. Repeat step 3 until you have two crucible and lid mass readings that differ by no more than 10 mg or 0.010g.
5. Add between 1.75 and 2.25 grams of your hydrated salt to the crucible and measure the combined mass of the crucible, lid, and salt. Determine the mass of the hydrated salt.
6. Return the crucible with the sample to the clay triangle support. Position the crucible lid off to the side to allow the evolving water molecules to escape during heating.
7. Initially heat the sample slowly and gradually intensify the heat. Heat the sample at a high temperature for 15 to 17 minutes. Cover the crucible once the heat is removed and allow cooling to room temperature. Determine the mass of the crucible, lid, and anhydrous salt using the same balance as used in the earlier steps.
8. Reheat the sample for an additional 5 minutes with medium/ high heat. Remeasure the combined mass of the crucible, lid, and anhydrous salt and continue repeating this process until you obtain 2 concurrent readings within 10 mg of each other.
9. If time allows, repeat this procedure with a new sample of the original hydrated salt.

CALCULATIONS for this lab are summarized on page 5.

Waste Disposal

Dispose of all waste anhydrous salts in the appropriate "ANHYDROUS SALT WASTE."

Clean-Up

Rinse the crucible with 2-3 mL of 1 M HCl and discard in the "WASTE ACID" container. Rinse the crucible 3 times with tap water, and once with deionized water.

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Data Sheet

Name: _____ **Lab Partner:** _____

Identity of Anhydrous Salt: _____

Formula Mass of Anhydrous Salt: _____

	TRIAL 1	TRIAL 2
Initial Mass of Empty Crucible and Lid		
After 1st Heating		
After 2nd Heating		
After 3rd Heating		
After 4th Heating		
Initial Mass of Crucible, Lid, and Hydrated Salt		
After 1st Heating		
After 2nd Heating		
After 3rd Heating		
After 4th Heating		

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Data Sheet 2

Show all calculations on a separate sheet of paper.

Name: _____

	TRIAL 1	TRIAL 2
Mass of Hydrated Salt (Initial)		
Mass of Anhydrous Salt (After last heating)		
Mass of Water Lost		
Mass Percent Water in Hydrated Salt		
Average Percent Water in Hydrated Salt		
Moles of Anhydrous Salt		
Moles of Water Lost		
Mole Ratio of Water Lost to Anhydrous Salt		
Formula of Hydrated Salt		

Full Name of Hydrated Salt _____

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Calculations:

Show all work in performing calculations.

1. Determine the mass of hydrated salt using the masses after the last heating of the empty crucible and the initial mass of crucible, lid and hydrated salt.
2. Determine the mass of water in the hydrated salt using Eqn 1 of the introduction.
3. Calculate the mass percent water in the hydrate using Eqn 2 of the introduction.
4. Calculate moles of salt and water using masses and formula masses.
5. Use the mole ratio of water lost to anhydrous salt to determine the formula of the hydrated salt. Make sure to use whole numbers in expressing the formula.

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Post-Lab Questions

Name: _____

1. Why was it important to heat the empty crucible to a constant mass before adding the hydrated salt?

2. What experimental sources of error were observed in your experiment?

3. Suppose Hasty Harry was in a hurry during this experiment and he did not heat his hydrated salt sample slowly at first. Some material splattered out of the crucible.
    a. How would this error affect the final mass in the crucible?

    b. How would the formula of the hydrate be affected by this error? Explain your answer using the calculations you performed as a guide.

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Pre-lab Assignment

Name: _____

1. In today's lab, what change will the starting material undergo when heated strongly?

2. A sample of an unknown hydrate weighs 0.543 g and after heating it weighs 0.4567 g. What is the percent of water in the hydrate