Determination of the Percent Water in a Compound and its Empirical Formula

The polarity of the water molecule, which makes it a great solvent for ionic compounds, causes water molecules to cling to the structure of solid substances. When this occurs, the trapped water molecules are called water of hydration and they become an integral part of the crystal structure.

There are many compounds that have a tendency to absorb water vapor from the air. These compounds are said to be *hygroscopic*, and can be used as moisture-reducing agents. Other compounds absorb such large quantities of water vapor that they will actually dissolve in their own water of hydration, a property known as *deliquescence*.

In this experiment, you will test a hygroscopic ionic compound containing copper, chlorine, and water molecules locked in the crystal structure of the solid to determine its water of hydration. The general formula for the compound is $Cu_xCl_y{\cdot}zH_2O$. The letters *x*, *y*, and *z* represent integers that will establish the proper chemical formula for this substance. Although the water molecules are securely attached to the ionic solid that you will test, they are susceptible to removal by heat. You will gently heat a sample of the compound to drive off the water of hydration. By measuring the mass of the sample before and after heating, you can determine the amount of water in the sample and calculate its water of hydration.

In the second part of this experiment you will conduct a chemical reaction with the dried sample, which will produce elemental copper. By measuring the mass of copper that forms, you will have the necessary information to determine the moles of copper and chlorine in your sample, and you will be able to establish the proper empirical formula.

John Dalton was an Englishman, a teacher, and an exceptional theoretical chemist. He developed and wrote the modern atomic theory at the turn of the 19^{th} century (documents point to 1803). He was influenced by the experiments of two Frenchmen, Antoine Lavoisier and Joseph Louis Proust. A fundamental component of the modern atomic theory is that the mole ratios of elements in a compound will be small whole numbers (*law of definite proportions*). The whole number mole ratio is commonly referred to as the empirical formula of a compound.

One of the challenges in finding the proper chemical formula for a compound is that there may be more than one plausible mole ratio for the elements in that compound. Dalton called this the law of multiple proportions. For example, if you were testing a compound that contained iron and sulfur, the plausible chemical formula could be FeS or Fe_2S_3. However, if you determine the mass of iron and the mass of sulfur present in a given mass of the compound, you will be able to establish the true chemical formula of the compound.

OBJECTIVES

In this experiment, you will

- Carefully heat a measured sample of a hygroscopic ionic compound.
- Determine the water of hydration in a copper chloride hydrate sample.
- Conduct a reaction between a solution of copper chloride and solid aluminum.
- Use the results of the reaction to determine the mass and moles of Cu and Cl in the reaction.
- Calculate the empirical formula of the copper chloride compound.

MATERIALS

crucible with cover
crucible tongs
spatula
ring stand, ring, and clay triangle
lab burner
50 mL beaker
Büchner funnel and filter flask
filter paper to fit Büchner funnel
watch glass
unknown solid copper chloride hydrate
aluminum wire, 20 gauge
6 *M* hydrochloric acid, HCl, solution
95% ethanol solution
distilled water
wash bottle
balance
glass stirring rod
heat lamp or drying oven

PROCEDURE

1. Obtain and wear goggles.

2. Measure and record the mass of a clean, preheated crucible and cover. Obtain about 1 g of the unknown copper chloride hydrate and place it in the crucible. Use a spatula to break up any large pieces of the substance by pressing the pieces against the wall of the crucible. Measure and record the *exact* mass of the crucible with compound and its cover.

3. Set up a ring stand, ring, and clay triangle for heating the sample. Rest the crucible on the clay triangle with the cover slightly tilted so that vapor may escape. Set up a lab burner and ignite the burner away from the crucible. Adjust the burner to get a small flame.

4. Hold the burner in your hand and move the flame slowly back and forth underneath the crucible to gently heat the sample. Do not overheat the compound. Note the color change, from blue-green to brownish, as the water of hydration is driven out of the crystals. When the sample has turned brown, gently heat the crucible for two more minutes.

5. Remove and turn off the burner. Cover the crucible and allow the sample to cool for about ten minutes. Mass the crucible, crucible lid and sample.

6. Reheat the crucible, crucible lid and sample until constant mass is achieved. Record the final mass.

7. Once the sample has achieved constant mass, transfer the brown solid to a clean and empty 50 mL beaker. Rinse out the crucible with two 8 mL aliquots of distilled water and pour the water into the 50 mL beaker. Gently swirl the beaker to completely dissolve the solid. Note that the color of the solution is green as the copper ions are rehydrated.

8. Measure out about 20 cm of aluminum wire, coil the wire, and place the wire in the beaker of solution so that it is completely immersed in the copper chloride solution. Note that the reaction produces a gas, elemental copper is forming on the surface of the aluminum wire, and the color of the solution is fading. The reaction will take about 30 minutes to complete. Go to Step 12 and set up parts a and b while you wait.

9. When the reaction is done, the solution will be colorless. Most of the elemental copper will be on the aluminum wire. Add 5 drops of 6 *M* HCl solution to dissolve any insoluble aluminum salts in the mixture, which should make the solution clear. **CAUTION:** *Handle the hydrochloric acid with care. It can cause painful burns if it comes in contact with the skin.*

10. Use a glass stirring rod to scrape off as much copper as possible from the Al wire. Slide the wire up the wall of the beaker and out of the solution with the glass stirrer and rinse off any remaining copper with distilled water. If any of the copper refuses to wash off the aluminum wire, wash it with one or two drops of 6 M HCl solution. Put the Al wire aside leaving the solid copper in the beaker.

11. Collect and wash the copper produced in the reaction.
    a. Set up a Büchner funnel for vacuum filtration.
    b. Obtain a piece of filter paper. Measure and record its mass, and then place the filter paper on the funnel. Start the vacuum filtration.
    c. Use small amounts of distilled water to wash all of the copper onto the filter paper on the Büchner funnel. Use the glass stirring rod to break up the larger pieces of copper.
    d. Wash the copper twice more with small amounts of distilled water.

12. Turn off the suction on the vacuum filtration apparatus. Add 10 mL of 95% ethanol to the copper on the filter paper and let it sit for about 1 minute. Turn the suction back on and let the vacuum filtration run for about five minutes.

13. Measure and record the mass of a clean, dry watch glass. Transfer the copper to the watch glass. Make sure that you have scraped *all* of the copper onto the watch glass.

14. Dry the watch glass of copper under a heat lamp or in a drying oven for five minutes. Remove the watch glass and allow it to cool. When the watch glass is cool enough to touch, measure the mass of the watch glass plus copper. Repeat the drying and weighing of the copper until you achieve a constant mass. Record the final mass.

15. Dispose of the copper, aluminum wire, and filtered liquid as directed.

DATA TABLE

Mass of crucible (g)	
Mass of crucible and hydrated sample (g)	
Mass of hydrated sample (g)	
Mass of crucible and dehydrated sample (g)	
Mass of dehydrated sample (g)	
Mass of water evolved (g)	
Mass of empty watch glass (g)	
Mass of watch glass and copper (g)	
Mass of copper (g)	

PRE-LAB QUESTIONS

1. What is the purpose of preheating the crucible and its cover prior to measuring its mass?

2. Washing soda is a hydrated compound whose formula can be written $Na_2CO_3 \bullet zH_2O$, where *z* is the number of moles of H_2O per mole of Na_2CO_3. When a 2.123 g sample of washing soda was heated at 130°C, all of the water of hydration was lost, leaving 0.787 g of anhydrous sodium carbonate. Calculate the value of *z*.

3. A piece of iron weighing 85.65 g was burned in air. The mass of the iron oxide produced was 18.37 g.

   (a) Use the molar mass of iron to convert the mass of iron used to moles.

   (b) According to the law of conservation of mass, what is the mass of oxygen that reacted with the iron?

   (c) Calculate the number of moles of oxygen in the product.

   (d) Use the ratio between the number of moles of iron and number of moles of oxygen to calculate the empirical formula of iron oxide. *Note:* Fractions of atoms do not exist in compounds. In the case where the ratio of atoms is a fractional number, such as ½, the ratio should be simplified by multiplying all the atoms by a constant to give whole number ratios for all the atoms (e.g., $HO_{½}$ should be H_2O).

POST-LAB QUESTIONS AND DATA ANALYSIS

1. Why must objects be cooled before their mass is determined on a sensitive balance?

2. How many moles of water were in your sample of copper chloride hydrate?

3. How many moles of copper were in your sample of copper chloride?

4. How many moles of chlorine were in your sample of copper chloride?

5. Write the proper chemical formula and name for the compound that you tested.

6. Use stoichiometry to calculate the theoretical yield of copper in this experiment based on the initial mass of your sample.

7. Calculate the percent yield of copper actually produced in this experiment.

8. A student fails to place the lid on the crucible during the initial heating of the hydrated sample and some of the solid spatters out. What effect does this error have on the calculated mass of the water lost by the hydrate? Justify your answer.