Chemical Formula Detective:
Determining the empirical formula of a hydrate

Background

Different elements can form chemical bonds to create compounds. For example, sodium and chlorine combine to form sodium chloride, NaCl. In the chemical formula NaCl, there is a 1:1 ratio of sodium ions:chloride ions. However, not all compounds form in a 1:1 ratio of their constituent elements. If they did, John Dalton would have been correct in 1803 when he proposed the chemical formula of water as HO[*]. Of course, we now know that the correct chemical formula of water is H_2O, in which there is a 2:1 ratio of hydrogen atoms to oxygen atoms. Since a mole is Avogadro's number of atoms, H_2O is also a 2:1 ratio of moles of hydrogen to moles of oxygen. Thus, the atom ratio is equivalent to the ***mole*** ratio (not a mass ratio) in a given chemical formula.

As chemistry students you have learned how to predict chemical formulas of ionic compounds based on periodic trends and nomenclature rules, but it hasn't always been that way. For hundreds of years, the chemical composition of compounds was studied experimentally, and the results generalized into the nomenclature rules used today. These rules allow the accurate prediction of chemical formulas for many ionic compounds without doing any experimentation. For example, the nomenclature rules can be used to correctly predict the formula of magnesium iodide as MgI_2 rather than MgI. The curious student of chemistry will wonder how such a prediction could be verified by experimental means.

Your task is to determine the chemical formula of an unknown copper chloride hydrate by experiment. An ionic **hydrate** is an ionic compound that has water molecules trapped within its crystal lattice (refer to the index/glossary of your textbook for more information). For example, Epsom salt ($MgSO_4 \cdot 7H_2O$) is a heptahydrate of magnesium sulfate: within one mole of magnesium sulfate heptahydrate there are seven moles of water. This water can be driven off by heat to form the anhydrous (dehydrated) ionic compound, magnesium sulfate ($MgSO_4$).

The chemical formula of your unknown copper chloride hydrate is in the general form of $\mathbf{Cu_{\mathit{x}}Cl_{\mathit{y}} \cdot \mathit{z}H_2O}$. Your objective is to determine what the actual formula is (what are the integers x, y, and z?) You will be required to make careful mass measurements and make calculations based on these.

The Overall Strategy

[*] John Dalton (1766-1844) made an assumption that when only one compound was formed from two elements, they did so in the simplest ratio, 1:1. (Water was the only known compound formed from hydrogen and oxygen at the time. Hydrogen peroxide, H_2O_2, was not discovered until 1815.) Since the mass ratio of oxygen to hydrogen in water is 8:1, he assigned the mass of hydrogen (the lightest element) to be 1 and , assuming the formula HO, assigned the value 8 to oxygen. The correct formula of water and the relative atomic mass of oxygen as 16 was a puzzle that would not be solved for another fifty years, despite evidence on the combining volumes of hydrogen and oxygen gas in a 2:1 ratio. Avodgadro's hypothesis would later be used to interpret this evidence correctly.

The formula will be determined by careful mass measurements. Remember, you are starting with $Cu_xCl_y{\cdot}zH_2O$. You will decompose this into several components, taking mass measurements along the way.

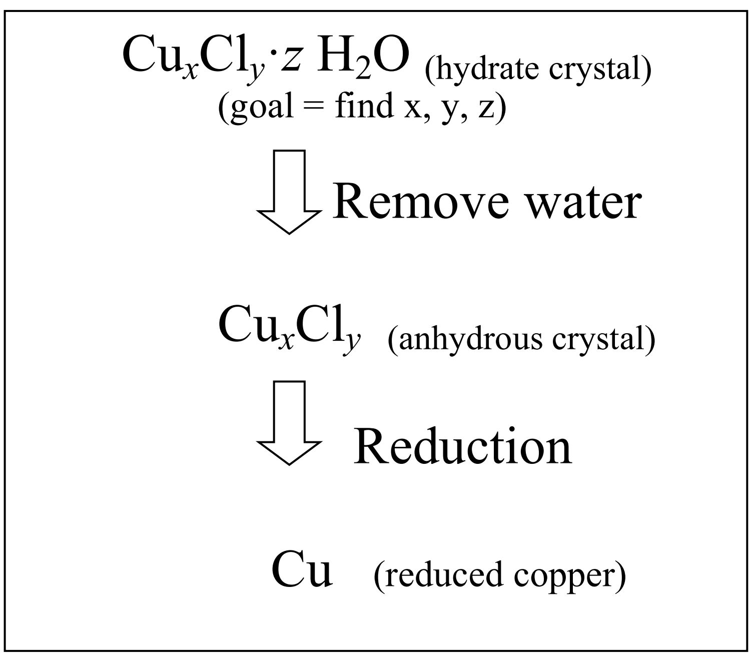
The first step is to gently dehydrate a known mass of your sample. The resulting dehydrated sample will be weighed to determine the amount of water lost (this is the zH_2O part).

The dehydrated copper chloride (now just Cu_xCl_y) will be made into a solution, dissolving the sample into water, making a mixture of copper ions and chloride ions. The copper ions will be reduced[*] to copper metal, which will be collected, dried, and weighed (now just Cu_x).

The remaining task is to determine the mass of chloride[**] in the compound, which can easily be done by mass difference. (The masses of the initial sample, water lost, and copper were determined in the previous steps.)

These steps should give you enough data to figure out the chemical formula of the unknown copper chloride hydrate.

A flow chart for today's experiment:



[*] Reduction of copper means that copper ions gain electrons to form copper metal. These electrons will be provided by the oxidation (loss of electrons) of an aluminum wire in the solution.

[**] The mass of chloride (Cl^- ion) is being determined. The difference between the mass of chlorine (Cl) and chloride (Cl^- ion) is negligible. (Why?)

Safety Precautions

HCl is corrosive. In case of contact with skin, rinse with plenty of water and notify your instructor. Wear goggles at all times in the chemistry laboratory.

Chemical Waste

The aluminum wire, liquid waste and copper produced in the experiment should be put into the appropriately labeled waste containers in the hood. Never pour chemical waste into the sink unless directed by your instructor. **Discard the filter papers in the garbage (put as much of the solids in the chemical waste as possible before throwing the paper in the garbage).**

Procedure

NOTE: **Keep and label everything**! Do not throw anything away until you are finished with the experiment, calculations, and have arrived at a reasonable compound formula!

NOTE (for instructors): At the beginning of the lab period, turn on the oven to 110 °C.

1. Weigh an empty, dry crucible and put about one gram of your unknown copper chloride hydrate into it, breaking up any clumps that are present. **Record the precise mass of your sample using significant figures.**

2. Place the uncovered crucible on a clay triangle supported by an iron ring clamp. Holding the Bunsen burner in your hand, move it back and forth under the crucible to **GENTLY** heat the sample. **Do not overheat it**. The hydrated crystals change color and will look like tobacco when dehydrated (do not allow the crystals to turn black). *Record your observations of the color change*. Continue heating for two minutes after all the crystals turn color. Cover and cool the crystals for 15 minutes.

   Check to see if any green crystals remain after this time by gently rolling the crystals around the crucible (do not touch the crystals or use anything to stir them!). Repeat the heating step if green crystals remain. **Record the mass of crucible and anhydrous (dried) Cu_xCl_y.**

   Safety: The crucible will be very hot. Handle it only with tongs or oven mitts!

3. Transfer the dehydrated sample to an empty 50-mL beaker. To ensure all the crystals have been transferred from the crucible to the beaker, use two 5 mL portions of distilled water to rinse the crucible contents into the beaker. Swirl the beaker to dissolve the crystals. The solution will turn color, signifying the presence of hydrated copper ions. *Record your obervations*.

4. Obtain a piece of aluminum wire approx. 10 cm long. Wind it into a loose coil. Completely submerge it into the 50-mL beaker containing your copper solution. *Record your obervations*.

5. The reaction will slow down as the surface of aluminum is reduced. Use a glass stirring rod to scrape the copper from the wire as completely as possible, exposing more of the surface for reaction.
   Record your observations. What changes do you observe as the reaction slows down? How will you know when it is over? With your partner, determine when the reaction is finished.

6. After the reaction is finished, remove the aluminum wire from the beaker with forceps and dispose of it in the waste container. Add 5 drops of 6M HCl to dissolve any insoluble aluminum salts and clear up the solution.

7. In the next steps, you will be collecting the copper by filtration. Secure a filtration flask (Erlenmeyer flask with a side port) to a ring stand using a large clamp. Attach the side port of filtration flask to the vacuum line using a thick-walled rubber hose. Obtain a 7.5-cm diameter circular filter paper and write your name on it using a pencil. **Record the mass of the filter paper**. The filter paper collects any solid material and allows liquid to pass through, thus separating a solid from a liquid. It will be difficult to scrape the solid copper off the filter paper without losing some of it. (In a later step you will weigh the filter paper with your copper, once it has dried, and then calculate the mass difference to determine the mass of copper. This method is called "weighing by difference".)

8. Place your filter paper in a Büchner funnel. Place a rubber gasket on top of the filter flask. Insert the Büchner funnel. Pour some distilled water onto the filter paper to make the filter paper lay flat on the bottom of the Büchner funnel. Open the vacuum valve and adjust to obtain light suction. Pour all of the copper mixture into the center of the funnel. Use distilled water as necessary for the transfer and also to rinse the solid copper. Turn off the suction. Add 10 mL of acetone (a drying agent) to the funnel. Turn on the suction again and leave it on for 5 minutes. **Sketch the setup with labels in your notebook.**

9. **Record the mass of a watch glass.** Use forceps to carefully transfer the filter paper with the copper to the weighed watch glass. Dry the copper by placing it (on the filter paper on the watch glass) in the oven, at 110°C, for about 15 minutes. **Record the total mass (mass of the watch glass + filter paper + dried Cu).**

Keep your copper until your calculations are finished!

Complete these calculations in your notebook.

It is crucial that you carry extra digits through each step and only round at the end.

1) Determine the number of moles of water lost from the hydrated unknown.

2) Determine the number of moles of copper collected.

3) Determine the number of moles of chloride there must have been in the compound.

4) Use the answers from above to determine the chemical formula of the copper chloride hydrate. (Note: It is possible that your chemical formula may look strange due to experimental error.)

5) Show your calculations to your instructor BEFORE you discard your copper.

Report Sheets
Chemical Formula Detective

Name________________________
Lab partner________________ Section______

Data

Mass of crucible	
Mass of crucible + $Cu_xCl_y{\bullet}zH_2O$	
Color of $Cu_xCl_y{\bullet}zH_2O$ crystals	
Mass of crucible + Cu_xCl_y	
Color of Cu_xCl_y solid	
Initial color of Cu_xCl_y solution (before adding Al)	
Final color of Cu_xCl_y solution (after reaction with Al)	
Mass of watchglass + filter paper	
Mass of watchglass + filter paper + dried copper	

Results

	mass (g)	moles
$Cu_xCl_y{\bullet}zH_2O$		
Cu_xCl_y		
water released		
copper		
chlorine		

Empirical formula of the copper chloride hydrate: ______________________

Post Lab Questions

1. a) Ask your instructor for the true (correct) formula of the unknown hydrate:

b) Write the systematic (use nomenclature rules) name for this compound.

c) Compare this formula with the one you obtained. Are they the same or different?

2. What colors did you observe for the following?

a) Copper chloride hydrate_______________

b) Copper chloride dehydrated solid_______________

c) Copper chloride aqueous solution before reaction with aluminum_______________

d) Copper chloride aqueous solution after reaction with aluminum_______________

3. In this experiment, you worked with all types of matter: elements, compounds, homogeneous mixtures, heterogeneous mixtures. Give examples of each type **that were encountered in this lab.**

a) Element(s) _____________________

b) Compound(s) _____________________

c) Homogeneous mixture(s) ______________________

d) Heterogeneous mixture(s) ______________________

4. a) What ions are present in the correct copper chloride hydrate? Give the sign (+ or –) and magnitude of their charges.

   b) Does water exist in the crystal lattice predominately as molecules of H_2O or as ions of H^+ and OH^-?

5. What effect would each of the following situations have on the calculation of the number of moles of copper in this experiment? Would the moles of copper increase, or decrease, or stay the same? **Give a very brief explanation for each.**

   a) You removed the aluminum wire while the mixture was still blue or contained bubbles.

   b) You added twice as much aluminum wire as necessary to the copper chloride. (Hint: Is aluminum a limiting reactant or in excess?)

   c) You couldn't scrape all of the copper off the aluminum wire.

   d) The dehydration was not complete because your crystals were still green. You did not reheat to complete the dehydration.

6. List at least two reasonable sources of error in this experiment. DO NOT list human error (*e.g.* spilling chemicals), miscalculations, or significant figures/rounding errors.

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Pre-Lab Assignment: Name____________________
Chemical formula detective Section______

Refer to the sections on moles and empirical formula in your textbook.

1. Read the procedure. Based on the flow chart, list all masses that are absolutely critical in obtaining during this experiment.

2. Epsom salt is $MgSO_4 \cdot 7\ H_2O$. What does the chemical formula become when this hydrate is gently heated?

3. A 1.000 g sample of an unknown hydrate of cobalt chloride is gently dehydrated. The resulting mass is 0.546 g. The cobalt is isolated and weighs 0.248 g. What is the empirical (experimentally determined) formula of the hydrate? Report each answer to the proper number of significant figures. Show your work.

    a. Determine the number of moles of water lost from the hydrated unknown. ______

    b. Determine the number of moles of cobalt collected. ______

    c. Determine the number of moles of chloride in the compound. ________

    Now, determine the empirical formula of the compound by finding the smallest set of integers for each component of the formula. (Divide the number of moles of each component (in parts a, b, c) by the smallest of the values. In this particular case, the number of moles of cobalt should be the smallest. If decimal values are obtained, round each value to arrive at whole numbers.)

    d. What is the mole ratio of cobalt to chloride (set cobalt equal to 1)? 1 : ___

    e. What is the mole ratio of cobalt to water lost (set cobalt equal to 1)? 1 : ___

    f. Write these mole ratios (parts d and e) to write the empirically determined chemical formula of the cobalt chloride hydrate. It will have the form $\mathbf{Co_xCl_y \cdot z\ H_2O}$, where x, y, and z are the integers that you determined.

    Empirical formula of the unknown cobalt chloride hydrate: ______________

4. In this experiment, you will determine the formula of a copper chloride. Copper usually exists as 1+ or 2+ charged ions. Give the two possible chemical formulas and the corresponding chemical names you expect for Cu_xCl_y, where *x* and *y* are integers.