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.

Chemistry students should appreciate the ability to predict chemical formulas based on nomenclature rules. For hundreds of years, the chemical composition of many compounds has been studied, 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 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 are seven moles of water. This water can be driven off by heat to form the anhydrous (dehydrated) ionic compound, magnesium sulfate.

Thus, the chemical formula of your unknown copper chloride hydrate will be in the general form **$Cu_xCl_y \cdot zH_2O$**. 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.

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[*] 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 Overall Strategy

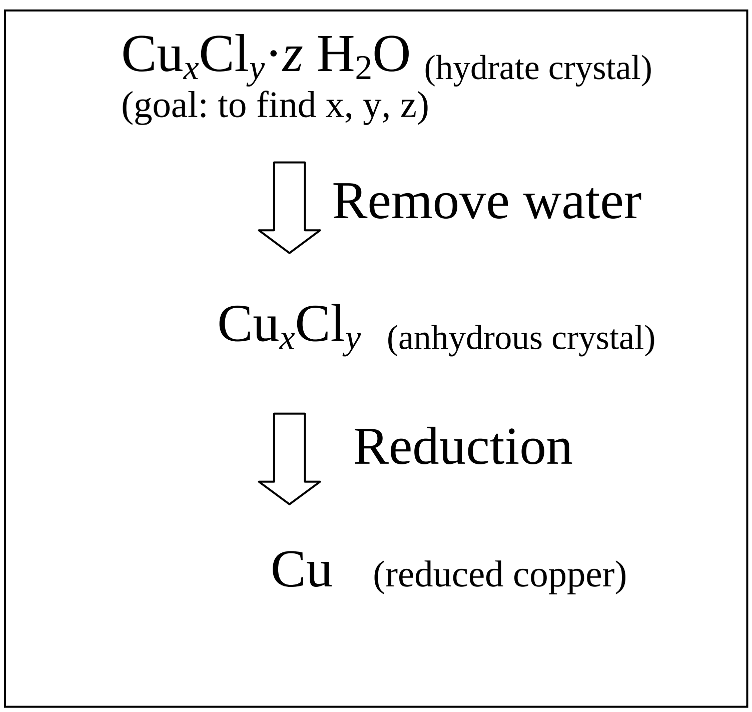
The formula will be determined by careful mass measurements. Remember, you are starting with $Cu_xCl_y{\cdot}zH_2O$. You will decompose this into several components. The first step is to gently dehydrate a known mass of your sample. The resulting dehydrated sample will be weighed to determine the amount of water lost (which is the zH_2O part).

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

The remaining task is to determine the mass of chloride[**] in the compound $Cu_xCl_y{\cdot}zH_2O$, 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?)

Experimental Procedure

NOTE: **Keep and label everything**!
Do not throw anything away until you are finished with the lab experiment and calculations!

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 (but should not be allowed to turn black). *Record 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 heating if green crystals remain.

Record the mass of crucible and dried Cu_xCl_y

Use this space to record your observations and/or data

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 the color change*.

4. Obtain a piece of aluminum wire approx. 20 cm long. Wind it into a loose coil. Completely submerge it into the 50-mL beaker containing your copper solution. *Record what you observe*.

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.

What changes do you observe as the reaction slows down? How will you know when it is over? Record your observations. 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.

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.

Procedure, continued…

7. In the next steps, you will be collecting the copper by filtration. You will use a Büchner funnel and a filter paper.

Remember it will be difficult to scrape the copper off the filter paper without losing some of it. Therefore, record the mass of the filter paper with the watch glass (enter this data in the table). In a later step you will weigh it again with your copper (once dried), and then use the mass difference to determine the amount of copper. The filtration setup is discussed in the next step.

8. Set up a small Büchner funnel fitted with a moistened piece of filter paper (sketch the setup in the space below with labels) and attach it to the vacuum. With light suction, transfer all of the copper to the funnel. Use distilled water as necessary for the transfer and also to rinse the copper. Turn off the suction and add 10mL of 95% ethanol to the funnel. After a minute, rinse the copper with 5 mL of acetone. Turn on the suction again and leave it on for 5 minutes.

Make a detailed sketch of your setup here for future reference.

9. The remaining copper is best collected on a watch glass, with the filter paper. **Record the mass of watch glass.** Dry the copper by placing it (on the filter paper and watch glass) under a heat lamp for about 5-10 minutes. **Record the mass of the watch glass, filter paper and dried Cu**.

<u>Do not clean up until your calculations are finished!</u>

Waste Disposal

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.**

Report Sheets

Chemical Formula Detective

Name____________________

Lab partner_______________

Section______

Data table: Record all data in ink.

Mass of crucible	
Mass of crucible + Cu_xCl_y•zH_2O	
Color of Cu_xCl_y•zH_2O	
Mass of crucible + Cu_xCl_y	
Color of Cu_xCl_y	
Mass of watchglass + filter paper	
Mass of watchglass + filter paper + dried copper	

Calculations:

Mass of Cu_xCl_y•zH_2O	
Mass of Cu_xCl_y	
Mass of water released	
Mass of copper	
Mass of chlorine	

	Copper	Chlorine	Water
Mass in grams			
Number of moles			
Mole ratio (round to whole number)			
Empirical formula of compound			
Systematic name of compound			

Calculations

Show your work. It is crucial that you use the proper number of significant figures.

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

5) What is the mole ratio of copper to water lost (set copper equal to 1)? 1 : ___

6) Use the answers from 4 and 5 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.)

7) Get your calculations checked by your instructor before you leave the lab BEFORE you discard your copper.

Post Lab Questions

1) Ask your instructor for the correct formula of the unknown hydrate: ____________

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

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

(a) Element(s) ______________________

(b) Compound(s) ___________________

(c) Homeogeneous mixture(s) _______________________

(d) Heterogeneous mixture(s) _______________________

3) What ions are present in the correct copper chloride hydrate? Give the sign and magnitude of their charges.

4) What color do you associate with the hydrated copper ion in the hydrate (what color are the crystals)? What happens to the color when the copper ions become dehydrated?

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 choice.

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 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.

Pre-Lab Assignment:
Chemical formula detective

Name____________________
Section______

Study sections on moles and empirical formula in your textbook.

1. In your own words, what is the purpose of this experiment?

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

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

4. 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 formula of the hydrate? **Make sure you use correct number of significant figures, or your answer could be incorrect. Show your work on all calculations.**

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. ________

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) Use the answers from parts d and e to determine the 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 integers that you determined from parts a through e.

Formula of the unknown cobalt chloride hydrate: ______________

5. In this experiment, you will determine the formula of a copper chloride. Copper usually exists as +1 and +2 oxidation states. **Give at least two possible chemical formulas** you expect for Cu_xCl_y, where *x* and *y* are integer.