

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 H₂O, in which there is a 2:1 ratio of hydrogen atoms to oxygen atoms. Since a mole is Avogadro's number of atoms, H₂O 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 MgI₂ 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 (MgSO₄·7H₂O) 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 (MgSO₄).

The chemical formula of your unknown copper chloride hydrate is in the general form of **Cu_xCl_y·zH₂O**. 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.

* 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, H₂O₂, 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. Avogadro'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 $\text{Cu}_x\text{Cl}_y \cdot z\text{H}_2\text{O}$. You will decompose this into several components, taking mass measurements along the way.

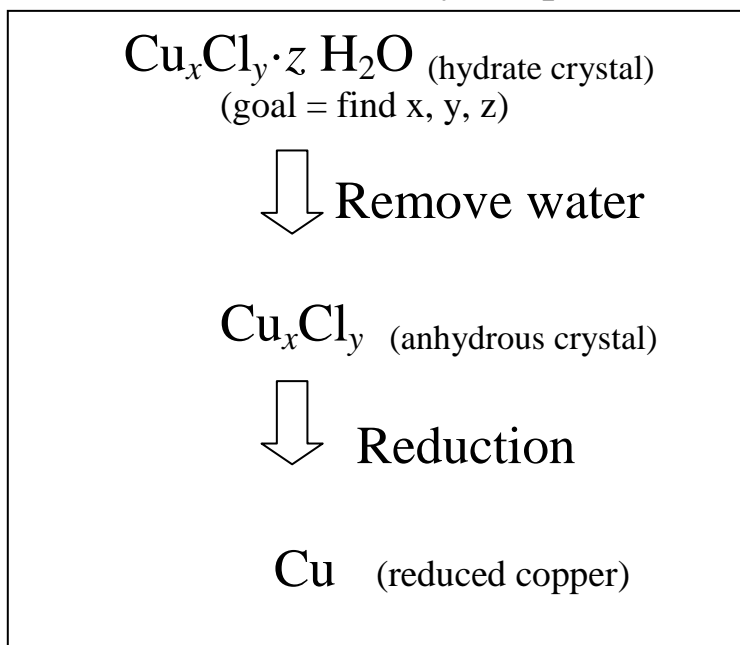
The first step is to gently dehydrate a known mass of your sample. The resulting dehydrated sample will be weighed to determine the amount of water lost (this is the $z\text{H}_2\text{O}$ part).

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!

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 observations.*
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 your observations.*

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. You will use a Büchner funnel placed on top of a filtration flask (Erlenmeyer flask with a side port) that is attached to the vacuum line using a thick-walled rubber hose. A piece of circular filter paper sits inside the Büchner funnel and 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; therefore, you will need to obtain the mass of copper using a “weighing by difference” method.

Record the mass of the filter paper. 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 with labels in your notebook) 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 acetone to the funnel. Turn on the suction again and leave it on for 5 minutes.

9. **Record the mass of a watch glass.** 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 10 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 _____ Date _____
 Lab partner _____ Section _____

Data

Mass of crucible	
Mass of crucible + $\text{Cu}_x\text{Cl}_y \cdot z\text{H}_2\text{O}$	
Color of $\text{Cu}_x\text{Cl}_y \cdot z\text{H}_2\text{O}$ 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
$\text{Cu}_x\text{Cl}_y \cdot z\text{H}_2\text{O}$		
Cu_xCl_y		
water released		
copper		
chlorine		

Empirical formula of the copper chloride hydrate: _____

Post Lab Questions

- Ask your instructor for the true (correct) formula of the unknown hydrate:
 - Write the systematic (use nomenclature rules) name for this compound.
 - Compare this formula with the one you obtained. Are they the same or different?
- What colors did you observe for the following?
 - Copper chloride hydrate _____
 - Copper chloride dehydrated solid _____
 - Copper chloride aqueous solution before reaction with aluminum _____
 - Copper chloride aqueous solution after reaction with aluminum _____
- 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.**
 - Element(s) _____
 - Compound(s) _____
 - Homogeneous mixture(s) _____
 - Heterogeneous mixture(s) _____
- What ions are present in the correct copper chloride hydrate? Give the sign (+ or -) and magnitude of their charges.
 - Does water exist in the crystal lattice predominately as molecules of H_2O or as ions of H^+ and OH^- ?

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Pre-Lab Assignment:
Chemical formula detective

Name _____

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 $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$. 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? **Make sure you use a sufficient number of significant figures, carry extra digits at each step, and only round at the end, 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. _____

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, multiply each by a common factor 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 $\text{Co}_x\text{Cl}_y \cdot z \text{H}_2\text{O}$, 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.