7B. EMPIRICAL FORMULA

(Spectrometer version)

Initial Question

A major emphasis of laboratory work for a chemist is determining the composition of a compound. There are many tools, such as chromatographic separation and spectroscopy, that aid the chemist in determining chemical composition. By keeping track of mass and breaking a compound into its component pieces, the pieces can be measured and the composition determined.

How do you discover the formula for an unknown substance?

Materials and Equipment

Model 1

- Hot plate, 1 per group, or an oven for the class
- Crucible and cover
- Crucible tongs

- Unknown copper hydrate, 1.0–1.5 g
- Balance (1–2 per class)

Model 2

- Spectrometry Application
- Spectrometer
- Graduated cylinder, 25-mL
- Volumetric flask, 100-mL

- Unknown copper hydrate, 1.0–1.5 g
- 0.10 M Copper(II) chloride ($CuCl_2 \cdot 2H_2O$), 60 mL
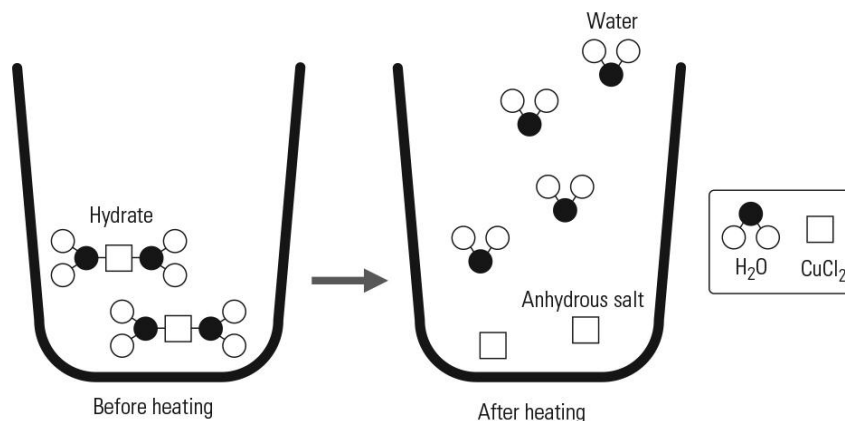- Distilled water, 25 mL

Safety

Add these important safety precautions to your normal laboratory procedures:

- Do not look into a hot crucible. Hot material may be ejected.

- Do not touch chemicals with your hands.

Getting Your Brain in Gear

1. In this lab, you will be heating a hydrate to remove the water. Label the appropriate molecules in the before-heating and after-heating diagrams below with "Hydrate," "Anhydrous salt," and "Water."

2. Write a mathematical equation that would show the relationship between the mass of the hydrate, the mass of water lost, and the mass of the anhydrous salt.

    _____

3. There are two possible ionic compounds that can be formed by copper and chlorine. Write their chemical formulas.

    _____

4. Recall that copper ions have a blue to green color in solution. Propose a lab technique that could be used to determine the concentration of copper ion in a solution prepared with your hydrate sample.

    _____

    _____

    _____

MODEL 1

Building Model 1 – Percentage of Water

1. Clean and dry a small ceramic crucible and cover.

2. Measure the mass of the crucible and cover. Record this in the Model 1 Data Table.

3. Measure between 1.0 and 1.5 grams of the unknown in the crucible (the cover should be on the balance, as well). Record the total mass of the sample, crucible, and cover in the Model 1 Data Table.

❷ 4. In this lab procedure, you obtain the mass of the unknown in the crucible. Explain why this method is preferred over finding the mass of the unknown in a weighing container and then pouring the sample into the crucible.

_____

_____

_____

5. Set up a hot plate to heat the uncovered crucible. Use a medium setting.

   NOTE: Heating the hydrate too hot (>300 °C) will result in the production of poisonous chlorine gas.

6. As water is released from the sample, the color will change from blue to brown. Use crucible tongs to gently shake the crucible occasionally to expose the blue hydrate in the middle. Continue heating until the blue color is gone; this will take ten to fifteen minutes. Work on the problem below while you wait.

❷ 7. In Model 2 you will use a spectrophotometric technique to determine the percentage of copper ion in your sample. To do this, you will need standard solutions of 0.10 M, 0.08 M, 0.06 M and 0.04 M copper ion. You will be given a stock solution of 0.10 M copper ion. Perform the calculations and write a procedure for how you will make at least 20.0 mL of each of the standard solutions.

_____

_____

_____

8. Once all the crystals are brown, remove the crucible from the hot plate with crucible tongs. Let the sample cool with the cover in place. After cooling for five minutes, find and record the total mass of the sample, crucible, and cover.

   NOTE: You cannot measure the mass of a hot object on the balance.

❷ 9. Why is it necessary to heat the sample with the cover removed?

_____

_____

❷ 10. Why is it necessary to let the substance dry with the cover on?

_____

_____

11. To ensure that all the water has been removed, reheat the crucible with the unknown for five minutes (uncovered). Let the sample cool on the desk (covered) and then obtain the mass as you did previously. Continue to heat in five-minute intervals until all of the water has been removed from the hydrate sample.

❷ 12. How will you know if you have heated the hydrate sufficiently to remove all of the water?

_____

_____

13. Transfer the brown anhydrous sample from your crucible to the solid waste jar in the hood. Rinse and dry the crucible.

Model 1 – Percentage of Water

Table 1: Model 1 Data Table—Determine the percentage of water in the hydrate

Parameters	Mass (g)
Crucible and cover	
Hydrate sample, crucible and cover	
After 1st heating	
After 2nd heating	
After 3rd heating (if necessary)	

Analyzing Model 1 – Percentage of Water

14. Calculate the mass of water lost from the hydrate.

_____

15. Calculate the percentage of water in the original sample.

16. Complete Table 2 using data from your classmates to compare different sized samples of hydrate. Compute the average percentage of water in the sample.

Table 2: Compare class results for the percentage of water in the hydrate

Mass of Hydrate Sample (g)	Mass of Water Lost (g)	Percentage of Water in Hydrate (%)

Average percentage of water in the hydrate: _____

17. Consider the class data above.

    a. How does the mass of water lost relate to the mass of the hydrate sample?

    _____

    _____

    b. How does the percentage of water lost relate to the mass of the hydrate sample?

    _____

    _____

18. Explain how the class data above supports the Law of Definite Proportions.

    _____

    _____

    _____

MODEL 2

Building Model 2 – Moles of Copper

1. Add a 1.0 to 1.5 gram sample of hydrate to a 100.0 mL volumetric flask and fill it to the mark with distilled water.

    Mass of copper hydrate sample: _____1.21 g_____

2. For obtaining a graph exhibiting Beer's Law, prepare the four copper ion standards (0.10 M, 0.08 M, 0.06 M, 0.04 M) as you described in the Building Model 1 section.

❷ 3. What color is the copper ion when dissolved in water?

    _____

❷ 4. Which color of light do you anticipate will give the highest absorbance reading on the spectrometer?

_____

❷ 5. You will be using the linear regression of the line to determine the number of moles of copper ion present in your sample. Would it be better to fit the absorbance or transmittance data? Explain your reasoning.

_____

_____

6. Connect the spectrometer to the data collection system using a USB cable connection, or wirelessly connect to the system through Bluetooth pairing.

7. Open the Spectrometry application.

8. Select Analyze Solution from the menu at the top of the screen.

9. Select Calibrate Dark from the Menu at the bottom of the screen. The Spectrometer will turn off all of its lights and perform the calibration. A check mark will appear when the calibration is finished.

10. Add distilled water to a cuvette. This should be the same distilled water that was used as a solvent for the solutions being analyzed. Place the cuvette into the spectrometer. Follow the cuvette handling guidelines listed below for the remainder of the investigation.

• Always handle the cuvette by the ridged sides.

• Wipe off any fingerprints using a lint free wipe (such as Kimwipes).

• Place the cuvette into the spectrometer so that the ridged sides are facing the violet and green light icons and the clear sides face the white light and absorbance spectrum icons. The white light is what will pass through the samples.

11. Select Calibrate Reference to calibrate the spectrometer with the distilled water (the water sample is called a "blank"). A check mark will appear when the calibration is complete.

12. Place ~2 mL of each test solution into separate cuvettes. Wipe each cuvette and handle it only from the ridged side.

13. Place a cuvette in the spectrometer chamber and start recording. Use the Add Coordinate feature to find the maximum absorbance for the sample. Use Scale to Fit to see greater detail. A pop up menu appears with a check mark on the left. Select the check mark when you have found the greatest absorbance. Record the Selected Wavelength in Model 2 Data Table. Stop Data collection.

14. Select Concentration from the menu at the top of the screen. In the table on the left type over the given concentration with the value you calculated. Start recording data. Once the absorbance has stabilized select the check mark next to the absorbance value to record it. 15. Record the absorbance in the Model 2 Data Table for each solution, sketch or attach a copy of your graph of concentration versus absorbance, and use the linear regression of the line to acquire and record the equation for the line.

CALIBRATE DARK

CALIBRATE REFERENCE

START RECORDING

STOP RECORDING

EXPERIMENT OPTIONS

ANALYZE SOLUTION

CONCENTRATION

TIME

SCALE TO FIT

ADD COORDINATE
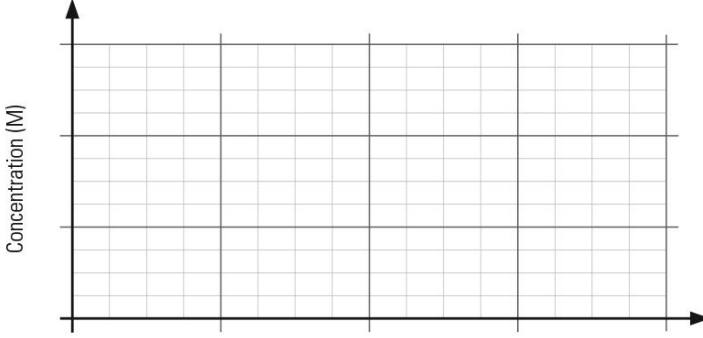
SHOW LINEAR FIT

SHOW COMPARISON

SHOW SELECTION

SHOW DUAL Y-AXES

Model 2 – Moles of Copper

Measured Wavelength _____

Table 3: Model 2 Data Table—Using a standard curve to determine the concentration of the unknown

Copper Ion Concentration	Absorbance	Graph
0.10 M		Concentration vs Absorbance
0.08 M		
0.06 M		
0.04 M		
Unknown		
		Equation for the line:

Analyzing Model 2 – Moles of Copper

16. Use the equation obtained from the absorbance data from the copper ion standards to find the concentration of copper ion in the solution made with your hydrate sample.

17. Use the Determine Unknown Concentration table to verify the concentration of copper ion.

18. Determine the number of moles of copper in the unknown.

19. Calculate the number of moles of water in the unknown using the percentage of water determined in Model 1.

20. Calculate the ratio of the number of moles of water to that of copper in your hydrate. Reduce the ratio to whole numbers.

21. Complete Table 4 using data from your classmates to compare the results when using different sized samples of hydrate.

    Table 4: Compare class results for the ratio of the number of moles of water to those of copper

Mass of Hydrate Sample (g)	Moles of Water (mol)	Moles of Copper Ion (mol)	Moles H_2O : Moles Cu

22. Consider the class data above. How does the ratio of moles of water to moles of copper relate to the mass of the hydrate sample?

_____

_____

23. Explain how the class data above supports the Law of Definite Proportions.

_____

_____

24. Determine the mass of chlorine present in your hydrate sample. (Hint: The hydrate contains only copper atoms, chlorine atoms and water molecules.)

25. Calculate the ratio of the number of moles of copper to the number of moles of chlorine in your hydrate. Reduce the ratio to whole numbers.

26. The formula for your hydrate has the form $Cu_xCl_y \cdot zH_2O$. Determine x, y and z in the formula from your answers above and identify your hydrate sample.

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Connecting to Theory

John Dalton was an Englishman, a teacher, and an exceptional theoretical chemist. He developed and wrote many postulates of the modern atomic theory at the turn of the 19th century (circa 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 (the 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 the possibility of more than one plausible mole ratio for the elements in that compound. Dalton called this the Law of Multiple Proportions. For example, when testing a compound that contained iron and sulfur, the plausible chemical formula could be FeS or Fe_2S_3. However, once the mass of iron and the mass of sulfur present in a given mass of the compound are determined, the true chemical formula of the compound can be established.

Applying Your Knowledge

1. The student has a combination of iron and oxygen.

   a) What are the possible formulas for the compound?

   _____

   b) The student obtained the following information regarding the compound. Based on this information, which compound is it?

   Item

Total mass of sample	1.50 g
Grams of iron	1.05 g
Grams of oxygen	0.45 g