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Determining the Empirical Formula of Copper Chloride

Overview

Determining the chemical formula of a compound is at the heart of what chemists do in the laboratory everyday. Many tools are available to aid in this determination, but one of the simplest (and most accurate) is the determination of the empirical formula. Why is this so useful? Because of the Law of Conservation of Mass, any reaction can be followed gravimetrically, or by change in mass. The empirical formula gives us the smallest whole number ratio among elements (or compounds) within a molecular compound. In this experiment, we are going to use gravimetric analysis to determine the empirical formula of copper chloride hydrate, $\text{Cu}_x\text{Cl}_y \cdot n\text{H}_2\text{O}$.

Principles

Hydrates are chemical compounds that have water molecules attached, not covalently bonded, to the compound. Formulas that are hydrated are symbolized by a dot (“ \cdot ”) between the compound and the water molecule. Hydrates easily lose water molecules upon heating, leaving behind the anhydrous (without water) compound. In this case, it would be copper chloride, Cu_xCl_y . The difference in mass between the anhydrous and hydrated forms of the salt corresponds to the mass (and moles) of water in the chemical compound $\text{Cu}_x\text{Cl}_y \cdot n\text{H}_2\text{O}$. The anhydrous copper chloride is then dissolved in water and copper is removed through a redox reaction with aluminum to form solid copper. The difference in mass between the total copper chloride hydrate and the sum of the reduced copper metal and water molecules corresponds to the mass of chloride in the sample. The mass of each component (Cu, Cl, H_2O) is converted to moles whereby the Law of Multiple Proportions allows us to use a ratio to determine the empirical formula of the compound. We cannot determine the true chemical formula of the compound without knowing its molecular mass, but the ratio will always remain the same.

Procedure

1. Dehydrating the hydrate
 - 1.1. Accurately weigh a sample of copper chloride hydrate and place it into a pre-dried and tared crucible. It is important that the crucible is dried above 120°C to drive off any adsorbed moisture. Typically, 1-2 grams of compound will suffice.
 - 1.2. Heat the sample using a Bunsen burner or other flame source until it changes color from greenish-blue to a reddish-brown (**figure 1**). This color change is indicative of the anhydrous form of copper chloride. The cover can stay on the crucible to prevent splattering, but can be cracked slightly to allow water vapor to escape.

- 1.2.1. Stir the sample to be sure the water is driven off the entire sample and the color is constant throughout.
- 1.2.2. As an alternative, the sample can be placed into a drying oven above 110 °C.
- 1.3. Cool the sample in a desiccator. This prevents water from re-hydrating the sample.
- 1.4. Measure the mass of the anhydrous sample. The difference should correspond to the water from the hydrate that was lost upon heating.
2. Isolating copper
 - 2.1. Transfer the sample to a 100-mL beaker and dissolve it in 50-mL of deionized water. The solution should turn blue once again, typically more blue than the hydrated solid.
 - 2.2. Add a small quantity (~0.20 g) of aluminum metal to the beaker. This will cause the copper to reduce to a reddish metal and the aluminum will oxidize to colorless Al^{3+} . The blue color of the solution should disappear as the Cu^{2+} ions form Cu^0 . After 30 minutes, add additional small pieces of aluminum to ensure all of the copper is reduced to solid copper.
 - 2.2.1. The solution now contains Al^{3+} ions, solid copper, and a small amount of solid aluminum.
 - 2.3. Dissolve any excess aluminum by adding ~5mL of 6M HCl. Aluminum is amphoteric, meaning it can react and dissolve in the presence of an acid or a base.
 - 2.4. Vacuum filter the colorless solution by tearing a piece of filter paper. Wash it twice with 10 mL of deionized water and then rinse with absolute ethanol. Air dry (not oven dry) the sample to prevent the formation of copper (II) oxide.
 - 2.5. Measure the mass of the copper solid to determine the mass of the chloride ion by difference.
3. Calculations
 - 3.1. Determine the mass of the chloride ion by difference:

$$\begin{aligned} \text{Total sample mass} &= \text{Cu}_{\text{mass}} + \text{H}_2\text{O}_{\text{mass}} + \text{Cl}^-_{\text{mass}} \\ \text{Total sample mass} - (\text{Cu}_{\text{mass}} + \text{H}_2\text{O}_{\text{mass}}) &= \text{Cl}^-_{\text{mass}} \end{aligned}$$
 - 3.2. Use the molar mass of each component of the compound to determine the moles of each component.
 - 3.3. Divide the moles of each component by the moles of the smallest component to give the smallest whole-number ratio of components, also known as the empirical formula of the compound.

Representative results

1. Experiment

- 1.1. Heat 1.25 g of copper chloride hydrate in a crucible. After heating and then cooling, the final mass is 0.986 g of copper chloride, Cu_xCl_y .
- 1.2. Dissolve the Cu_xCl_y sample in 50 mL of deionized water and add 0.2 g of fine aluminum mesh to the beaker.
- 1.3. After reacting and dissolving the excess aluminum, 0.198 g of dried copper metal is recovered.
- 1.4. Subtracting the mass of both copper and water from the initial copper chloride hydrate and yields the mass of chloride ion in the sample:
$$1.25 \text{ g} = 0.198 \text{ g copper} + 0.264 \text{ g water} + x \text{ g chloride}$$
$$x = 0.521 \text{ g Cl}^-$$

2. Data

- 2.1. To determine the smallest whole-number ratio of components in the compound, the mass of each component is converted to moles and then each is divided by the smallest number of moles in the sample, copper in this case:

Table 1

- 2.2. The resulting smallest whole-number ratio yields a formula of $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$.

2.2.1. In the event the final ratio yields decimal values, the whole formula would be multiplied by a constant to give whole number values. Common fractional values are 0.25, 0.333, 0.50, 0.667, and 0.75. For example, if a smallest whole number ratio yielded the formula yielded $\text{C}_7\text{H}_9\text{NO}_{2.5}$, the entire formula would be multiplied by 2 to give the empirical formula $\text{C}_{14}\text{H}_{18}\text{N}_2\text{O}_5$.

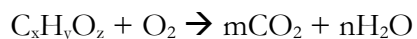
- 2.3. A molecular formula cannot be determined from the empirical formula without knowing the molecular mass of the compound. The reason for this is demonstrated in the example below:

Table 2

All three compounds have the same empirical formula, but very different molecular formulas.

Applications

In one example, suppose an unknown biomolecule containing only C, H, and O is found to act well as a new fuel. One way to determine the formula of the fuel would be to combust it in air and analyze the products:



While O_2 is in excess, we would know all the carbon in CO_2 originated from the biomolecule and all the hydrogen would be present in H_2O . The difference between that total mass and

the mass of initial sample would be the mass of oxygen in the molecule. We could then convert to moles and determine the empirical formula.

In another example, a hydrate sample of $\text{Mg}_x\text{Cl}_y \cdot n\text{H}_2\text{O}$ is given. The mass of the water molecules would again be easily determined by heating. Using some solubility rules, chloride is then precipitated with silver ion, Ag^+ , to form $\text{AgCl}(s)$. Once the mass of $\text{AgCl}(s)$ is found, the moles of Cl^- are determined using the molar mass of $\text{AgCl}(s)$ and then converted to grams of Cl^- . This would allow us to determine the mass of magnesium in the sample followed by the empirical formula.

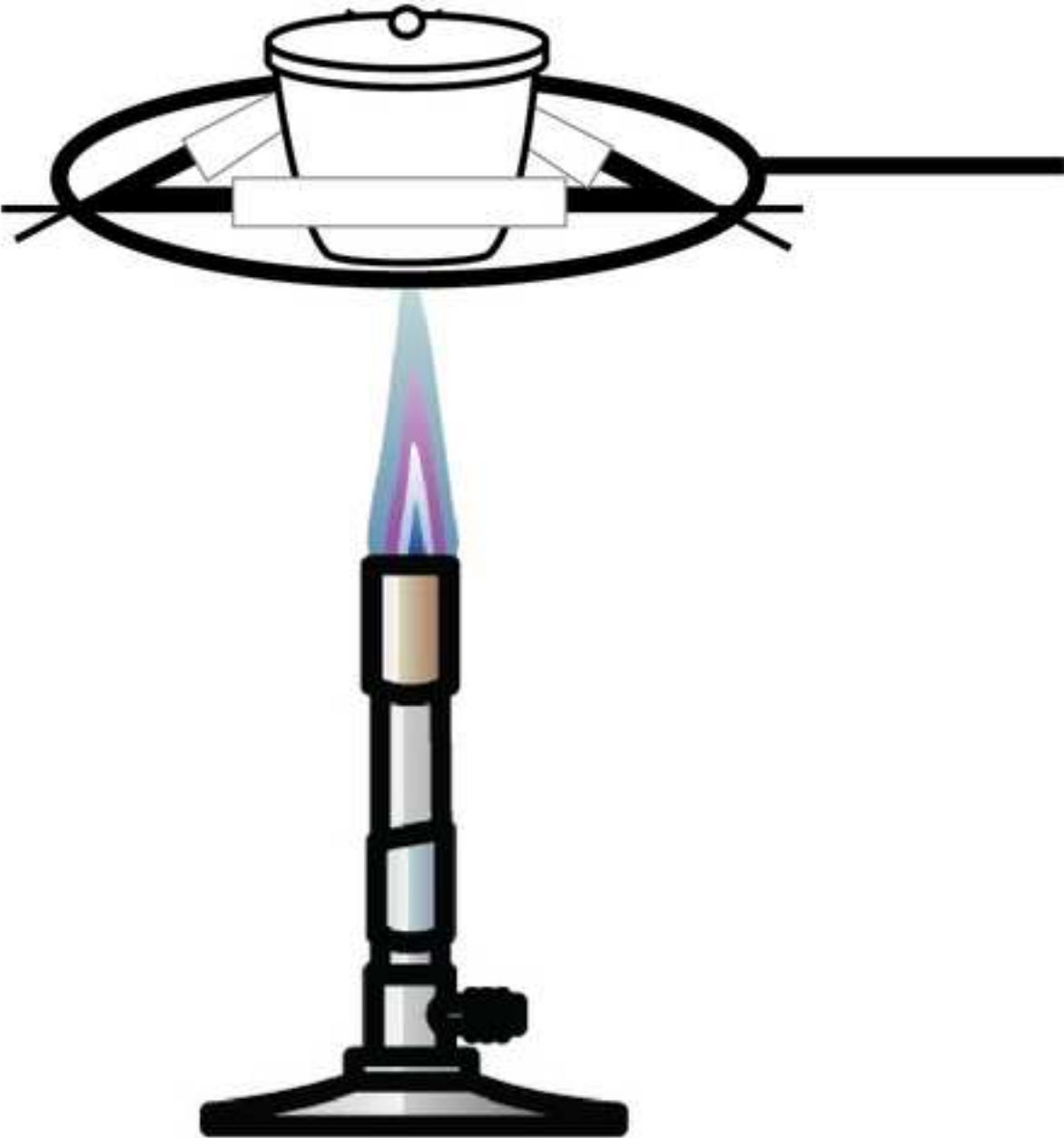
Determining an empirical formula is at the center of identifying the formula of the actual molecule. From pharmaceuticals to forensics, determination of a molecular formula is key to identifying an unknown compound which means taking the empirical formula to the next step. Typically, the determination of an empirical formula is coupled with elemental analysis to obtain elemental weight percent information. From these data, the molar ratios are calculated and the empirical formula is determined. We can determine the mass of molecule using another analytical tool like a mass spectrometer. Then, the ratio between the molecular mass and empirical mass is calculated to determine the true molecular formula.

Conclusion

While there can be some arithmetic and stoichiometry involved, determining the empirical formula of a compound is a bit like solving a jigsaw puzzle. All of the pieces are there! Because all things must obey the Law of Conservation of Mass and Law of Multiple Proportions, empirical formulas are at the heart of identifying a chemical compound.

Legend:

Figure 1. Bunsen burner with ceramic crucible.



Component	Mass (g)	Molar mass (g/mol)	Moles	Ratio	Calculated whole number ratio
Copper	0.198	63.55	7.332×10^{-3}	$\frac{7.332 \times 10^{-3}}{7.332 \times 10^{-3}}$	1
Chloride	0.521	35.45	1.469×10^{-2}	$\frac{1.469 \times 10^{-2}}{7.332 \times 10^{-3}}$	$1.998 \approx 2$
Water	0.264	18.01	1.466×10^{-2}	$\frac{1.466 \times 10^{-2}}{7.332 \times 10^{-3}}$	$2.003 \approx 2$

Name	Molecular Formula	Empirical Formula
Acetic acid	CH ₃ COOH	CH ₂ O
Formaldehyde	CH ₂ O	CH ₂ O
Glucose	C ₆ H ₁₂ O ₆	CH ₂ O