VALENCIA COLLEGE

Experiment 2:
Empirical Formulas

Version 5

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Two of the basic tenets of atomic theory: Lavoisier's Law of Conservation of Mass and Proust's Law of Definite Proportions are applied to chemical reactions involving magnesium and a copper salt. The data obtained from the reactions can be used to determine the empirical formulas of the compounds and the amount of water in a hydrate.

Objectives

- To determine the empirical formulas of compounds
- To determine the percent composition of a reaction product

Learning Outcomes

- To apply the Law of Conservation of Mass to a chemical reaction.
- To apply the Law of Definite Proportions to determine the empirical formulas of compounds.
- To determine the empirical formula of a compound.
- To learn how to determine the empirical formula of a hydrate.

Definitions

- **decanting** – gradually pouring off the supernatant, leaving the solid in the original container
- **empirical formula** – chemical formula based on the lowest possible integer coefficient of elements
- **formula unit** - the ratio of atoms in an ionic compound, the chemical formula; although it does not represent the whole crystal structure of an ionic compound, it is useful for stoichiometric calculations
- **hydrate** – inorganic compound containing a determined ratio of water molecules per formula unit
- **Law of conservation of mass** – establishes that mass is preserved during the course of a chemical reaction; that is, mass is not created nor destroyed in chemical reactions
- **Law of definite proportions** – establishes that chemical compounds always have the same mass ratio of elements
- **molecular formula** – chemical formula based on the actual ratio of its elements
- **oxidation** – loss of electrons in an oxidation-reduction (redox) reaction
- **oxidizing agent** – element that causes another element to be oxidized by being reduced (accepting the other element's electron or electrons)
- **reduction** – gain of electrons in an oxidation-reduction (redox) reaction
- **reducing agent** – element that causes another element to be reduced by being oxidized (giving away its electron or electrons to the other element)
- **supernatant** – liquid lying over the solid in a chemical reaction or process

Introduction

Many metal ions exhibit bright, beautiful colors when excited by a hot flame. This excitation is the basic principle of pyrotechnic colorants or fireworks. In fireworks, the cation present determines the color shown, while the anion often can alter the temperature of the flame, helping to adjust the brightness and duration of the firework. These materials are compacted into pods called stars, consisting of a blend of oxidizing agent, reducing agent, coloring agent (metal salt), and binders. When ignited, the stars produce both sound and light effects. The appearance of a firework is determined by its stars. Blue color is often produced from the salts of the copper(I) ion, especially the chloride. Some other metals such as titanium and magnesium are often used to improve the brightness of the fireworks, or to produce bright whites. Although magnesium produces beautiful bright whites, its natural oxidation or common alloying can affect its brightness. The Light Me Up Fireworks company had to recall a batch of their fireworks as the white stars did not produce the desired results. This batch caused a major embarrassment in many Fourth of July celebrations as the white color appeared rather bleak, and the blues appeared more greenish than blue. As a chemist for the Light Me Up Fireworks Company, you were asked to assess the failure. Your colleagues believe that the materials were accidentally swapped and that different materials were used. You strongly believe that the cause of the problem was contamination with an oxidizing agent that caused the undesired oxidation of the materials used. Burning of the magnesium should generate the oxide in an oxidation-reduction reaction; and if part of the material was already oxidized, it will not produce a bright white flame. After all, magnesium can oxidize naturally to the oxide by air and in the presence of many oxidizing agents. The undesired oxidation of the copper ion in your salt can give colors that range from green to purplish. You will follow an experiment to test the formula of the materials used in the fireworks.

Chemists write formulas in different ways. The most common is the molecular formula. The molecular formula shows the actual number of atoms of each element present in the formula. A simple form of a chemical formula is an empirical formula. An empirical formula consists of the lowest possible integer ratio of the elements forming the compound. The empirical formula is not how a compound exists, and does not represent the actual structure of the compound, but it is useful since it can be determined easily by experiment. For example, the chemical compound benzene has a molecular formula of C_6H_6. The lowest possible integer ratio between the carbon and the hydrogen will be 1:1, thus benzene has an empirical formula of CH.

Two of the basic foundations of our atomic theory are Antoine Lavoisier's law of conservation of mass, and Joseph Proust's law of definite proportions (also known as law of constant composition or law of definite composition). The law of conservation of mass states that matter is not created nor destroyed, but preserved during the course of a chemical reaction. The law of definite proportions states that a chemical compound will always have the same ratios of its elements. We can observe that benzene will always consist of 92.2 % carbon by mass, and 7.8 % hydrogen by mass.

Each C_6H_6 molecule will contain six carbon atoms, and six hydrogen atoms. Using the molar masses from the periodic table, we obtain:

$$mass\ carbon = 6 \times 12.01\ g/mol = 72.06\ g/mol \text{ , and}$$

$$mass\ hydrogen = 6 \times 1.01\ g/mol = 6.06\ g/mol \text{ , thus}$$

$$\%\,C = \frac{72.06\ g\ C}{72.06\ g\ C + 6.06\ g\ H} \times 100 = 92.24\% \qquad \%\,H = \frac{6.06\ g\ H}{72.06\ g\ C + 6.06\ g\ H} \times 100 = 7.76\%$$

No matter how large or small the sample is, benzene will always have this ratio or percent. The law of definite proportions and the law of conservation of mass enable us to determine the formula of a compound if we are able to determine the masses, or the percentages of the different elements in the formula. Once we obtain the masses for the elements, we can use the elements' molar masses to determine the number of moles of each element and determine the ratio between them.

For example, a chemist found that a given compound is composed of 0.410 g Al and 1.590 g Cl. What is the empirical formula?

1. We begin by determining the number of moles of each element.
2. $n_{Al} = 0.410\ g\ \left(\frac{1\ mol}{26.98\ g}\right) = 0.0152\ mol \qquad n_{Cl} = 1.590\ g\ \left(\frac{1\ mol}{35.45\ g}\right) = 0.04485\ mol$
3. This determines a ratio of 0.0152 mol Al: 0.04485 mol Cl. We want to turn this ratio into integers. We accomplish that by dividing both numbers by the smallest one.
4. $Al_{\frac{0.0152}{0.0152}}Cl_{\frac{0.04485}{0.0152}} \quad or \quad AlCl_3$
5. We should expect that due to experimental errors, the ratios will not be exact integer numbers, but within the uncertainty, they can be rounded to the closest integers. If the ratio of any of the elements is close to 0.5 (1.5, for example), the ratios of all the elements in the compound need to be multiplied by a factor of 2 to turn them into integer ratios.

Let's consider another example. In this experiment, in addition to the law of definite proportion, we will also need to apply the law of conservation of mass to find the chemical formula of a hydrate. Hydrates are salts with a strong affinity towards water. They have a fixed ratio of water molecules. For example, $CaCl_2 \cdot 2H_2O$ (calcium chloride dihydrate) has two water molecules per formula unit. In the case of hydrates, the water of hydration will not be removed by air drying. However, these water molecules can be removed under more intense heating. The masses of the compound before and after heating to remove the water molecules allows us to obtain the mass of salt, and the mass of water. We can determine the number of moles of salt, and the number of moles of water in order to find the ratio of water molecules per formula unit.

In today's experiment, you will apply these concepts in order to determine the formulas of the products obtained by burning magnesium and dehydrating a copper salt.

Experimental Procedure

Part A Empirical formula of the product of the reaction of Mg with O_2

Record the data of Part A in Table 1.

Safety Precautions

Allow time for glassware and equipment to cool

Techniques

- Technique 2: Using a balance
- Technique 9: Using a Bunsen burner

Technique

List of Chemicals

- deionized (DI) water
- magnesium ribbon

Equipment

- two crucibles and lids
- Bunsen burner
- striker
- clay triangle
- ring support
- stand
- hot pad
- analytical balance
- disposable transfer pipet
- crucible tongs

1. Familiarize yourself with the use of the Bunsen burner. Following instructions of Technique 9 Using the Bunsen Burner, turn on and adjust height of flame and air so all three cones visible. Show flame to instructor for approval/signature on Experimental Data and Calculations sheet.

Technique 9

2. Obtain a clean crucible and lid. Check the crucible for cracks. A cracked or chipped crucible will break upon heating or cooling. Support the crucible and lid on a clay triangle and heat with an intense flame for 5 minutes (see Figure 1). NOTE: Start with low heat for about 3 minutes and gently increase to intense for another 2 minutes. Make sure that the lid is slightly opened. This “firing” process will remove any contaminants from the crucible that would affect mass change during the experiment.

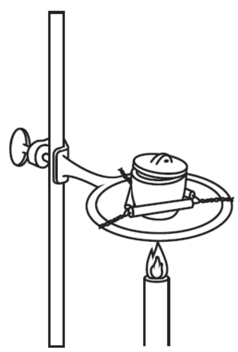
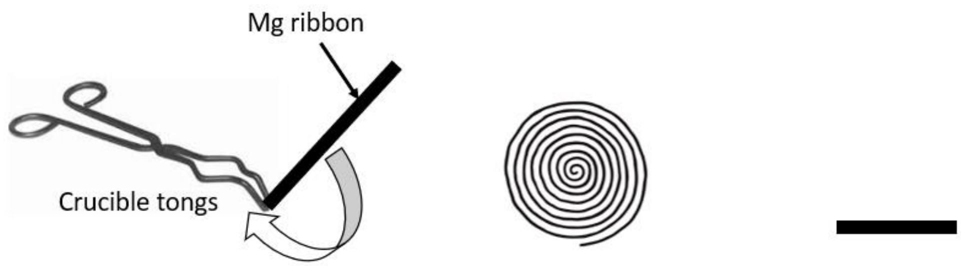


Figure 1. Set-up for heating. The burner is positioned several inches from the crucible.

3. Allow the crucible to cool on the clay triangle for 2-3 minutes. Then, place hot crucibles and hot lids on hot pads - never directly on the bench top. Allow them to cool to room temperature.
4. Measure the mass of the fired, cool crucible and lid. Use only clean, dry crucible tongs to handle the crucible and lid for the remainder of the experiment.
5. Cut a 10 to 12 cm strip of Mg. Polish it with steel wool or sand paper.

Technique 2

6. Curl the Mg ribbon (see Figure 2). The curl should be loose enough to have space between the coils but tight enough that the bottom edge lies flat on the bottom of the crucible. This will increase the rate of the reaction by increasing contact with oxygen and the hot crucible.



(a) Curling with the use of tongs. (b) Top view of curl. (c) Side view of curl.

Figure 2. Curling the magnesium ribbon.

7. Measure and record the mass of the Mg, crucible, and lid.
8. Heat over the flame for complete reaction. You will need to slightly lift the lid to allow oxygen into the reaction once per minute. Make sure that the lid is not fully removed as Mg burns very bright and may cause temporary blindness.
9. When the reaction appears complete (no visible change and sample looks like ash), allow the crucible to cool. Weigh the burned product, crucible, and lid.
10. Add a couple of drops of DI water into the crucible. There should not be standing water in the crucible. Burn again for 5 minutes without the lid. Cool the product after the second burn and weigh the product with the crucible and lid for a second time. If the difference in mass between the first and second burn is less than ±0.010 g record the second reading as the mass of the product. If the difference is larger, do a third burn without the lid and record the mass as the mass of the product.
11. Repeat the experiment with a second piece of Mg.

Part B Determination of the Amount of Water in a $CuCl_2{\cdot}nH_2O$ Compound

Record the data of this section in Table 2.

Safety Precautions

Allow time for glassware and equipment to cool

Techniques

- Technique 2: Using a balance

Technique 2

List of Chemicals

- deionized (DI) water
- copper (II) chloride hydrate crystals

Equipment

- hot plate
- two evaporating dishes
- hot pad
- analytical balance
- crucible tongs or hot hand protector

1. Weigh a clean evaporating dish. Add approximately 1 g of $CuCl_2 \cdot nH_2O$ to the evaporating dish and record the mass to the full precision of the balance.
2. Place the evaporating dish on a hot plate. Heat for approximately 5 minutes at about 2/3 of the maximum power (300 °C setting). Note: The color of the solid will gradually change from green to brown.
3. When only a small amount of the solid remains green, stir the mixture with a glass stirring rod to move the unreacted parts toward the bottom of the evaporating dish for better heating. Note: avoid stirring the crystals until most are brown since the hydrate tends to stick to the rod.
4. Once the solid is completely brown, using tongs or a hand protector, remove the evaporating dish from the heat and place it in a desiccator (minimize the time the lid is off the desiccator to increase the "life" of the desiccant in the desiccator). Let it cool for five minutes.
5. After five minutes, if the desiccator is light enough to carry, take it to the balance room. Remove the evaporating dish from the desiccator and weigh the evaporating dish.
6. Place the evaporating dish on the hot plate and heat again for five minutes at 2/3 of maximum power (300 °C).
7. Using tongs or a hand protector place the evaporating dish in the desiccator for an additional five minutes.
8. Remove the evaporating dish from the desiccator and re-weigh it.
9. If the mass difference between the two measurements is larger than 0.01 g, re-heat a third time for an additional 3 minutes. Allow it to cool in the desiccator. Re-weigh.
10. Repeat above procedure one more time with a new sample. (If both evaporating dishes fit in the desiccator, heat both trials at the same time.)
11. Determine the mass of the dehydrated samples.

Clean up/Disposal

- Dispose of all materials on the corresponding waste containers as indicated by your instructor.
- Wash all glassware with soap and water, and then rinse with deionized water (Technique 1 Cleaning Glassware). Dry the outsides of the glassware. Return all glassware to its place.

Pre-lab

1. When using a crucible and lid, why must the crucible and lid be inspected for cracks before using?
2. What is meant by “firing the crucible”? Why must we “fire” the crucible before using it?
3. A 1.30 g sample of titanium chemically combines with chlorine gas to form 5.16 g of titanium chloride. (a) What is the empirical formula of titanium chloride? (b) What is the percent by mass of titanium and the percent by mass of chloride in the sample?
4. A 0.500 g sample of tin foil reacted with oxygen to give 0.635 g of product. (a) What is the empirical formula of the tin oxide? (b) What is the percent by mass of tin and the percent by mass of oxygen in the sample?
5. Epsom salt is commonly purchased in the pharmacy for a variety of uses, anti-inflammatory, laxative, and cosmetic. Epsom salt is a hydrated salt of magnesium sulfate. If 2.000 g of Epsom salt are heated to remove the waters of hydration, 0.977 g of the anhydrous (without water) magnesium sulfate, $MgSO_4$, were obtained. Calculate the number of waters of hydration, and write the chemical formula of the Epsom salt.

Post-lab

<u>In the data/calculations section:</u>

Include the data collected in the lab and complete the calculations.
Answer the following Post-Lab Questions.

1. In part A, the crucible was not “fired” before burning the magnesium. When the magnesium was burned, volatile impurities in the crucible were burned off. Will this error increase, decrease, or not affect the ratio of magnesium to oxygen determined and the empirical formula found?
2. In part A, a student did not completely burn all the magnesium and some un-burned magnesium remained. Will this error increase, decrease, or not affect the ratio of magnesium to oxygen determined and the empirical formula found?
3. In part A, a student forgot to allow some air (oxygen) to get into the crucible while burning the magnesium. The oxygen insufficiency resulted in the formation of the magnesium nitride (Mg_3N_2) instead of the magnesium oxide. Will this error increase, decrease, or not affect the ratio of magnesium to oxygen determined and the empirical formula found?
4. In part B, a student burned the hydrate over a very intense heat, and some of the copper(II) chloride was turned into a copper(II) oxide. Will the number of waters of hydration determined be too high, too low, or remain the same?
5. In part B, a student forgot to place the heated sample in the dessicator while cooling, and some water was re-absorbed by the copper (II) chloride. Will the number of waters of hydration determined be too high, too low, or remain the same?

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Experiment 2:
Empirical Formulas
Experimental Data and Calculations

Name: ______________________________ **Date:** __________________

Lab Partner: ______________________________ **Section:** ____________

Experimental Data and Calculations

Proper use of the Bunsen Burner - Professor's signature: ____________________

Table 1. Empirical formula of the Product of the Reaction of Mg with O_2

	Trial 1*	Trial 2
mass of lid		
mass of crucible + lid		
mass of crucible + lid + Mg		
mass of Mg		
moles of Mg		
mass of burned Mg product + lid + crucible:		
1st mass recording after heating		
2nd mass recording after heating		
3rd mass recording after heating (if needed)		
mass of oxygen		
moles of oxygen		
formula of the magnesium oxide		

Name: ____

*Show calculations for Table 1 trial 1.

Table 2. Empirical Formula of a Copper Hydrate

	Trial 1*	Trial 2
mass of evaporating dish		
mass of evaporating dish + $CuCl_2{\cdot}nH_2O$		
mass of copper(II) chloride hydrate		
mass of copper(II) chloride + evaporating dish after heating:		
1st mass recording after heating		
2nd mass recording after heating		
3rd mass recording after heating (if needed)		
mass of H_2O		
moles of H_2O		
mass of anhydrous $CuCl_2$		
moles of $CuCl_2$		
formula of the copper (II) chloride hydrate		

Name: ____

*Show calculations for Table 2 trial 1.