

**CHM-201 General Chemistry and Laboratory I**  
**Laboratory 6 – Empirical Formula of Magnesium Oxide**  
**Sections CHM201-03/T1/T2 – Spring, 2021**

*Note: Be sure to download and print the timetable for your section.*

### **Purpose**

In this experiment, you will determine the empirical formula of a compound – magnesium oxide. Since you already possess the knowledge to determine the formula by simply looking at the periodic table, this may not seem like much of a challenge. However, “knowing” the formula and proving it are two different things. This laboratory will provide you with experience calculating the empirical formula from percent composition and give you a greater appreciation of stoichiometry. It should also reinforce the importance of accurate measurement, i.e., significant figures, in experimental work.

### **Introduction**

Antoine Lavoisier (1743-1794) experimentally determined that mass is conserved in the course of a chemical reaction, and that a compound is made up of elements in fixed proportions. You will use both of these fundamental concepts of chemistry to determine the empirical formula of magnesium oxide. Lavoisier arrived at his conclusions by accurately measuring the masses of materials before and after a chemical reaction. Although the instruments he used were crude by today’s standards, he was able to uncover two basic truths. In order to determine the formula of magnesium oxide when magnesium reacts with oxygen, you will also have to make accurate measurements of mass. With these data, you will calculate the amount of oxygen that combines with magnesium.

Magnesium is a shiny, silver-colored metal that burns with an intense white flame when ignited in air. One possible method of determining the amount of oxygen present in magnesium oxide is to determine the mass of a piece of magnesium, burn it, and determine the mass of the resulting white material. Since this material is magnesium oxide (theoretically), the difference in mass would be the mass of oxygen in the oxide. One problem with this method is that some of the magnesium oxide would escape into the environment, looking like white smoke. Although this amount is small, it cannot be ignored. In order to obtain usable data, you must contain and control the combustion.

Even when the experiment is carefully controlled, you must consider the possibility that other reactions take place during the combustion in air. After all, air is only about 20% oxygen. The primary component of air, the solvent, is nitrogen, so some magnesium nitride,  $Mg_3N_2$  may also be formed.

After burning the magnesium, it will be necessary to perform one more step to remove the magnesium nitride by reacting it with water to form magnesium hydroxide,  $Mg(OH)_2$ , and ammonia,  $NH_3$ . The ammonia, a gas, will escape to the atmosphere where you will be able to confirm its presence. (How?) Finally, you will heat the resulting mixture one more time to decompose the magnesium hydroxide into magnesium oxide and water.

In this lab, participants will combine data to help ensure the precision (repeatability) of the measurements. After the experimental procedure is done, bring me your data sheet to be scanned. Your results will be posted on Moodle. Your final calculation of the empirical formula of magnesium oxide will be based on the results obtained by the entire class.

### **Procedure**

#### **1. Preparation.**

- 1.1 Obtain a crucible and crucible lid, clay triangle, iron ring, ring stand and Bunsen burner.
- 1.2 Attach the iron ring to the ring stand and adjust the height of the ring stand so that the top of the Bunsen burner is 2-3 cm from the bottom of the crucible when it sits in the clay triangle.
- 1.3 Wash the crucible and lid to make sure that they are not contaminated, and dry them with paper towels.

- 1.4 Prior to heating the crucible and lid, **ask** me to demonstrate the method to be used for heating it. **Do not proceed until you have done so.**
- 1.5 To remove all the water from the crucible and lid, heat them in the clay triangle directly in the flame for 3-4 minutes. Heat both the sides and the bottom of the crucible. It should become red hot if you are doing it correctly.
- 1.6 Allow the crucible and lid to cool to room temperature. After an initial cooling period, they will cool faster on wire gauze on the desk. **Transfer the crucible and lid with tongs to avoid burns or transferring any moisture or oils from your fingers to the them.**
- 1.7 Record the mass of the **cooled** crucible and lid on an analytical balance to 0.1 mg (four decimal places – **RECORD ALL THE DIGITS**).
- 1.8 Repeat procedures 1.5–1.7 until the mass has stabilized. This has been achieved when the difference between two consecutive measurements is 2 mg (0.002g) or less. When you believe you have achieved this, see me for confirmation. Only two measurements should be needed, but you have spaces for up to four on the data sheet. If it takes more than two measurements, you have almost certainly done something wrong. **Consult me!**
- 1.9 Determine the mass of the dry crucible and lid (tare). There is no shortcut, as in previous labs. You must determine and record the actual mass every time.
- 1.10 Obtain a few pieces of magnesium ribbon and confirm that its mass is between 0.2-0.3 grams on the top loading balance. **Do not spend time trying for an exact mass, but do not go above or below these masses.** This value does not have to be recorded.
- 1.11 The magnesium ribbon should be clean (shiny). If it has gray spots on it, you must remove them with steel wool or sandpaper before the next step. Wipe the cleaned magnesium with a dry paper towel. The magnesium should be shiny. If you are unsure whether to proceed, **ask**.
- 1.12 Roll the magnesium ribbon into a loose spiral (see the model setup on my desk), and place it in the crucible using tongs. Be sure that it is exposed to air on both sides (is not compressed into a ball).
- 1.13 Determine and record the mass of the magnesium, crucible and lid on an *analytical* balance to within 0.1 mg (four decimal places).
- 2. Reaction of magnesium**
- 2.1 Return the crucible which now contains the magnesium to the clay triangle using **tongs (be sure the cover is on)**.
- 2.2 Gently heat the bottom of the crucible with the flame, moving the flame back and forth for 2-3 minutes. **This step reduces the danger of the crucible cracking from the intense heat of the combustion reaction. It is absolutely necessary to heat the crucible gently before allowing the reaction to commence.**
- 2.3 Next, place the burner directly below the crucible and heat strongly for an additional 5 minutes. After you have completed this step, **ask** me to demonstrate the next step.
- 2.4 **Ask me to help with this step.** Use the tongs to lift the lid slightly to allow additional air into the crucible. If you look carefully, you should be able to see the magnesium glow, but you do not want it to flame. Replace the cover and heat for another 2 minutes. Repeat opening the cover of the crucible while heating until no glow is evident in the sample. The total heating time should be about 10-15 minutes.
- 2.5 When you have completed step 2.4, consult me before continuing.

- 2.6 After I tell you to continue, allow the crucible to cool to room temperature. The contents of the crucible should be a white or gray powder without any metallic luster. If you think you see any unreacted magnesium, consult me.
- 2.7 **After notifying me that you are on this step**, add a few (5 – 10) drops of distilled water to the **room temperature** crucible. Do you detect any ammonia smell? **Note: Do not place your nose directly over the crucible and sniff.** Record your observations.
- 2.8 Place the cover on top of the crucible, but leave it slightly open to the air so that any water or ammonia can escape. Heat the crucible gently until the contents appear to be dry.
- 2.9 Heat strongly for 10 minutes (this removes any excess water or ammonia and converts the hydroxide to the oxide).
- 2.10 Allow the crucible to cool to room temperature.
- 2.11 Determine and record the mass of the crucible, cover and magnesium oxide on the analytical balance (to 0.1 mg accuracy).
- 2.12 Heat the crucible again for 2-3 minutes and allow it to cool.
- 2.13 Measure the mass on the analytical balance again, and record
- 2.14 Repeat steps 2.12 and 2.13 until the mass stabilizes to within 5 mg (0.005 g). Record each mass as it is measured. If your mass does not stabilize after three reheatings, consult me.
- 2.15 Determine and record the mass.
- 2.16 Clean the crucible, and return the laboratory equipment to the appropriate locations. The rest of the work is done later, so submit your data sheet and leave the lab.
- 3. Calculation of the empirical formula (need not be done in the lab)**
  - 3.1 Convert the number of grams of magnesium used in this experiment to moles.
  - 3.2 Calculate the amount of oxygen present in the oxide and convert it to moles.
  - 3.3 Using the number of moles from 3.1 and 3.2 above determine the empirical formula of the oxide to the nearest whole number.
- 4. Calculation of classwide results (need not be done in the lab)**
  - 4.1 Fill in the group data table using the results obtained by the other groups. The simple calculations are the same as the calculations you did with your own group's data. Do not forget to include your own group in the class results. **I may post a list of lab groups whose data should not be used for this calculation. Check the website for this information.**
  - 4.2 Complete the table by computing the total masses.
  - 4.3 Calculate the empirical formula again, this time using the **totals** from the class data.