

Determining the Empirical Formula of an Oxide Laboratory Experiment

by W. Lee with additional information from M. Horton ~ Revised J. Flint Baumwirt 2005

In this experiment, you will be able to determine the empirical formula for magnesium oxide from results you will obtain by burning magnesium in air.

Introduction

Whether it is alone or in air, molecular oxygen, O_2 , is a very reactive substance when it is heated and many elements will react with it. When an element reacts with molecular oxygen, it chemically combines with the oxygen and forms an oxide.

Burning the magnesium in the presence of air, will not only form magnesium oxide, but will also form magnesium nitride. Since magnesium is an active metal, it will react with both molecular oxygen and nitrogen in the air. The amount of magnesium oxide produced will be much greater than the amount of magnesium nitride. However, it is important to convert that small amount of magnesium nitride to magnesium oxide. This can be done by adding water and then heating. The water will convert the magnesium nitride to magnesium oxide and the nitrogen will be liberated as ammonia. Heating will convert magnesium hydroxide to magnesium oxide by losing water vapor thus the product will consist solely of magnesium oxide. (*Note In this lab we will NOT add water to a hot crucible as it will break.)

One can determine the amount of oxygen that is present in the oxide by taking the mass of magnesium oxide present and the original mass of the magnesium that was weighed at the start of the experiment.

As an example, review the reaction of phosphorous with molecular oxygen to form some phosphorous oxide. In this example, 0.1920g of phosphorous burns in the presence of oxygen and produces 0.4400g of the oxide. The first step is to determine the mass of the oxygen in the oxide. Obtain this value by subtracting the mass of the phosphorous in the beginning from the mass of the oxide.: $0.4400g - 0.1920g = 0.2480g$ of oxygen

The next step is to convert both the mass of the phosphorous and oxygen to moles.

$$0.1920g \text{ P} \times \frac{1 \text{ mole P}}{30.974g \text{ P}} = 6.199 \times 10^{-3} \text{ moles of P}$$

$$0.2480g \text{ O} \times \frac{1 \text{ mole O}}{16.00g \text{ O}} = 1.550 \times 10^{-2} \text{ moles of O}$$

Next, divide each value you have obtained by the smallest number of moles.

$$\frac{6.199 \times 10^{-3}}{6.199 \times 10^{-3}} = 1.000 \qquad \frac{1.550 \times 10^{-2}}{6.199 \times 10^{-3}} = 2.500$$

Since the empirical formula is a ratio of whole positive numbers, you must multiply these numbers by two. This gives you the empirical formula for the phosphorous oxide produced as P_2O_5 .

Laboratory Procedure and Write up:

This lab shall be written up in its entirety in your laboratory comp book. Include the standard **Title, Purpose, Prelab Questions, Equipment**, a fully labeled **Diagram of Equipment** and set up, **Procedure** (short summary of what YOU did to complete this lab), fully labeled **Data Table, Calculation Section**, showing one sample of each type of calculation with answers boxed and labeled, including percent error, **Error Discussion** which includes a discussion of influences that may have affected your data and a **Conclusion** statement. This shall be followed by the completed **Post Lab** questions.

Determination of the Empirical Formula of a Compound

Objective: To determine the empirical formula of magnesium oxide (Mg_xO_y).

Method: Read through these instructions before doing anything.

1. A crucible must be close to room temperature before you weigh it. A hot or very warm crucible will induce air currents in the analytical balance chamber and this will cause a significant error in weighing. In addition, a hot crucible could damage the balance.
2. Cut off a piece of magnesium ribbon about 1.5 cm in length.
3. Weigh the empty crucible together with the lid on the analytical balance. It is most convenient to place the lid on upside down on top of the crucible at this point
4. Open balance door and drop the piece of magnesium into the inverted crucible lid. Record the mass of crucible with the magnesium ribbon.
5. Make a hook on the end of large paperclip. Fasten the hook to the crucible lid and extend outward so that you can use this to lift the lid during the reaction without burning your fingers. Make sure it doesn't touch the ring clamp as the crucible heats.
6. Press the magnesium ribbon into the bottom of the crucible so that it is in contact with the base of the crucible and then place the crucible on a clay triangle and heat it over a Bunsen burner. The tip of the inner blue flame from the burner should touch the bottom of the crucible. **BE SURE TO WEAR GOGGLES.** A chemical reaction should occur. When the magnesium just begins to spark or flame completely cover the crucible with the lid. You need to let some oxygen in by lifting the lid slightly but you do not want any of the product (in the form of smoke) to escape. Continue to heat until there is no more evidence of a chemical reaction taking place and the magnesium no longer flames up or glows brightly when you lift the lid. The reaction is complete when all of the magnesium ribbon has turned into a white ash though the ash will glow red even after the reaction is completed only because the ash is hot not because it still reacting. It will not damage your results and may be of benefit to continue heating your product 1 to 2 minutes after the reaction appears to be complete.
7. Turn off the gas to the burner and allow the crucible containing the newly formed compound to cool without removing it from the clay triangle.
8. *After the crucible has cooled to room temperature*, weigh it on the analytical balance.
9. Describe the appearance of the product.
10. Add a few drops of water to the product, mix very thoroughly, and try to detect the odor of ammonia gas (NH_3).
11. Wipe out the crucible with a paper towel and discard the product (and paper towel) in the trash receptacle. Return the crucible to your lab station and clean and arrange your equipment and materials as you found them.

Prelab Questions: Read the entire lab thoroughly FIRST

Part A:

1. What is the purpose of this experiment?
2. Write the balanced molecular equation for the reaction of calcium with molecular oxygen to form calcium oxide:
3. You start out with an empty crucible weighing 19.3350g. After adding some calcium to the crucible, the mass is now 22.1156g. After heating the calcium and completely converting it to the oxide, the mass of the crucible is now 25.7701g. Determine the mass of the calcium and oxygen in the oxide.

Part B:

1. What measurements must be made and what data must be recorded for you to determine the empirical formula of magnesium oxide? Read through the method a second time to make sure you have listed all of the measurements you will need to make.
2. What element will the magnesium combine with and where will that element come from?
3. Which would you expect to be greater, the mass you weighed in step 4, or the mass you weighed in step 8? Explain your answer.
4. Why is the crucible lid needed in determination of the empirical formula of magnesium oxide?
5. Why does the crucible have to be at room temperature before you weigh it?

Data: You must make a data chart before you begin the lab.

Make a list of all the measurements you will need to make. Set up a data chart of your own design to record all of the measurements you listed including the following:

1. Mass of fired empty crucible _____ g
2. Mass of crucible and magnesium _____ g
3. Mass of crucible and newly formed compound _____ g

Calculations: You must make a list of calculations before you begin the lab.

Make a list of all the calculations of your own design to record all of the computations you listed above.

1. Mass of magnesium _____ g Mg
2. Mass of magnesium oxide _____ g Mg?O?
3. Mass of oxygen in compound _____ g O
4. Moles of magnesium in compound _____ mol Mg
5. Moles of oxygen in compound _____ mol O
6. Ratio of moles of magnesium to moles of oxygen ____ mol Mg : ____ mol O
7. Empirical formula for magnesium oxide _____

Post-Lab Questions:

1. Write the balanced molecular equation for the reaction of magnesium with molecular oxygen to form magnesium oxide.
2. What evidence that a chemical reaction took place did you observe?
3. Did you smell ammonia after adding water to the product? The odor of ammonia might suggest the formation of a product containing what element? The product formed might have been magnesium _____ (Mg__ __ __)
4. What are possible sources of error?
5. How would any of the errors you listed in question 3 affect the results of your determination? In other words would the error cause an over-estimate or would it cause an under-estimate of the amount of oxygen that combined with the magnesium? Explain your answer.
6. Did you include the formation of the product suggested in question B-2. as one of the sources of error? How would the formation of this product affect your estimate of the amount of oxygen that combined with the magnesium?

