1. The Empirical Formula of an Oxide

Introduction

Every chemical compound has a chemical formula that can be determined by a combination of experiments and calculations. For most ionic compounds, such as magnesium oxide, the *empirical formula* (the simplest formula) is the formula that is used for the compound. An empirical formula is the formula with the smallest integer (whole number) subscripts (Ebbing/Gammon, Section 3.5).

Purpose

You will be able to determine the empirical formula for magnesium oxide from the results that you will obtain by burning magnesium in air.

What happens when an element is burned in air?

Molecular oxygen, alone or in air, is a very reactive substance when it is heated. Many elements will react with it. When an element reacts and combines chemically with molecular oxygen, an oxide (a compound of the element with oxygen) is formed.

Molecular nitrogen, the chief component of air, is a rather unreactive substance, even at a high temperature. Only the more active metals will react and combine chemically with molecular nitrogen during heating. When nitrogen does react with an active metal, a nitride (a compound of the element with nitrogen) is formed.

Although the amount of molecular nitrogen in the air is approximately four times the amount of molecular oxygen, more oxide than nitride is formed when an active metal is burned in air. The reason is the superior reactivity of molecular oxygen.

Concept of the experiment

In this experiment, after a known mass of magnesium is burned, the product will consist of magnesium oxide and smaller amounts of magnesium nitride (Mg_3N_2). Water will convert the nitride to magnesium hydroxide [$Mg(OH)_2$] with the liberation of ammonia (NH_3). Heat will cause conversion of the hydroxide to the oxide with the loss of gaseous water.

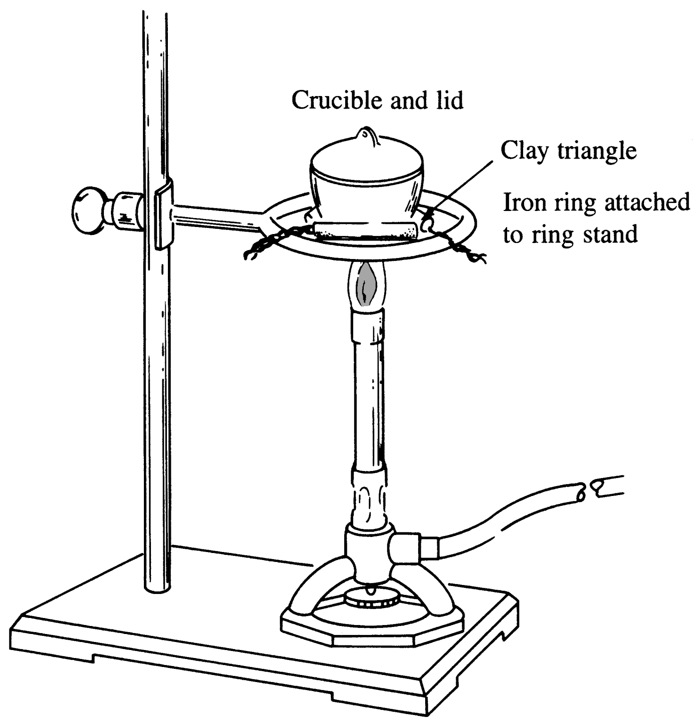
Because the product will consist solely of the oxide after this treatment, you can determine the mass of oxygen that is present in the oxide from its mass and the original mass of the magnesium. The laws of conservation of mass, as well as the concept of a mole, will lead you to the method by which you can determine the empirical formula of this oxide (Ebbing/Gammon, Sections 1.3 and 3.2).

Procedure

Getting started

1. Your laboratory instructor may wish to provide special safety precautions concerning the bulk supply of magnesium ribbon.
2. Ask your laboratory instructor about discarding the magnesium oxide that you will prepare in this experiment.
3. Obtain a crucible and lid. Wash, rinse, and dry them.
4. Obtain about 0.2 g of magnesium ribbon. If it is not bright, clean the surface with sandpaper.
5. Place the covered crucible in a clay triangle on an iron ring that is attached to a ring stand. Adjust the height of the ring so that the bottom of the crucible will be in the hottest part of a properly adjusted laboratory burner. (The Introduction to this manual discusses burners and their use.) The correct arrangement of the equipment, crucible, and burner is shown in Figure 1.1.

FIGURE 1.1
The correct arrangement of the ring stand, the clay triangle, the crucible with its lid, and the burner.

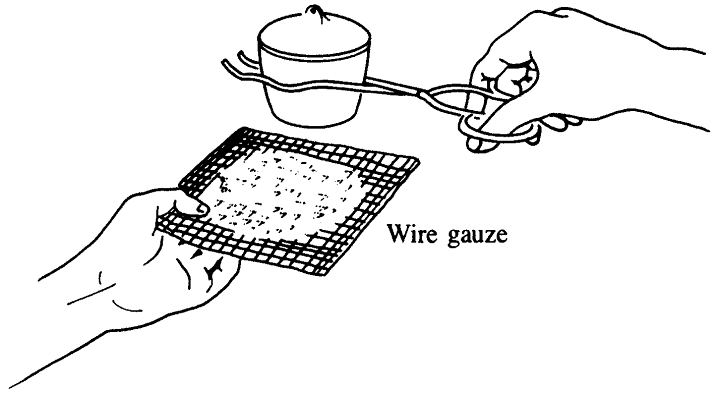


Doing the experiment

1. Heat the covered crucible for about 3 min. The bottom of the crucible should attain a red-hot glow during this time. Move the burner and allow the crucible to cool (5–10 min). When the crucible is cool, you should feel no heat when you place one of your fingers about 1/2 inch from the bottom of the crucible.

CAUTION: Avoid burning your fingers. Do not touch the crucible or the iron ring at any time during this experiment.

FIGURE 1.2
The proper method for carrying the crucible, with crucible tongs over wire gauze. A watch glass may be used instead of the wire gauze.



2. When the covered crucible is cool, transfer it to the pan of a balance using crucible tongs while holding a wire gauze under the crucible (Figure 1.2), but do not put the wire gauze on the pan. If you must wait to use the balance, do not place the crucible directly on the bench. Put it on the wire gauze or leave it in the clay triangle.
3. Obtain and record the mass of the covered crucible.
4. Repeat Steps 1, 2, and 3 until two consecutive masses differ by no more than ±0.001 g or any other precision that is stipulated by your laboratory instructor. Record the mean or average value of these masses. You will use this result in subsequent calculations.
5. Fold the magnesium ribbon into a loose ball that will fit completely inside the crucible. Do not fold the ribbon too tightly. The best results will be obtained when as much of the surface of the ribbon as possible is exposed.
6. Cover the crucible, obtain the mass, and record it once again.
7. Return the crucible to the clay triangle, using crucible tongs and the wire gauze. The lid should be completely in place. Brush the bottom of the crucible with the flame for about 2 or 3 min. Next place the burner on the ring stand and heat the crucible in the hottest part of the flame for another 3 min.
8. Use crucible tongs to lift the lid carefully by a slight amount to allow more air to enter the crucible. Do not open the lid too far because doing so will allow the metal to enflame. The metal should glow brightly without flames. Flames from the metal must be avoided because they will carry part of the solid oxide out of the crucible and into the air as a smoke.
9. Repeat Step 8 every few minutes until no metal is evident and no glow occurs when the lid is lifted.
10. Allow the covered crucible and its contents to cool. The contents should be white or slightly gray.
11. Remove the lid and place it on the wire gauze. Add a few drops of distilled water from a medicine dropper directly on the contents. The smell of ammonia may be evident at this point.
12. Recover the crucible so that the lid is slightly ajar. Heat the crucible by brushing it with the flame until the contents are dry. Next heat the crucible strongly for 8–10 min to convert the hydroxide to the oxide.
13. Allow the covered crucible and its contents to cool to the same point as in Step 1.
14. Obtain the mass of the covered crucible and record it.

15. Heat the covered crucible strongly again for about 3 min. Obtain and record the mass after the crucible has cooled.
16. You will have obtained a "constant" mass if the two measurements agree to the precision specified in Step 4. If they do not, repeat Step 15 until two successive measurements differ by no more than this amount.
17. Clean the crucible and lid carefully.

CAUTION: Before leaving the laboratory, make sure that your gas outlet and those of your neighbors are closed.

The Empirical Formula of an Oxide

Date:	....	Student name:	....
Course:	....	Team members:	....
Section:	....		....
Instructor:	....		....

Prelaboratory assignment

1. a. What is the law of definite proportions, and how does it apply to this experiment?

   b. What is a mole and what is a molar mass? What are the molar masses of magnesium and atomic oxygen?

   c. Why is magnesium the limiting reactant (Ebbing/Gammon, Section 3.8) in this experiment?

2. What safety precautions are cited in this experiment?

3. When 0.848 g of finely divided iron is burned, 1.212 g of a reddish brown oxide is obtained.

   a. Determine the empirical formula of the oxide.

   b. Write a balanced chemical equation for the reaction of iron and molecular oxygen based on this empirical formula.

The Empirical Formula of an Oxide

Date:	....	Student name:	....
Course:	....	Team members:	....
Section:	....		....
Instructor:	....		....

Results

Mass of empty crucible and lid (g)	....	....
	....	....
	....	....
Average mass of empty crucible and lid (g)	....	
Mass of crucible, lid, and Mg (g)	....	
Average mass of empty crucible and lid (g)	....	
Mass of Mg (g)	....	
Mass of crucible, lid, and oxide (g)	....	....
	....	....
	....	....
Average mass (g)	....	
Average mass of empty crucible and lid (g)	....	
Mass of oxide (g)	....	

Questions

1. Determine the empirical formula of the oxide of magnesium. (Think carefully about the precision of each mass that you measured before you do this calculation. How many significant figures are justified for each mass?)

2. Write correctly balanced chemical equations for the following reactions:

   a. Magnesium with molecular oxygen

   b. Magnesium with molecular nitrogen

   c. Magnesium nitride with water

   d. Heating magnesium hydroxide