Determination of the Empirical Formula of Magnesium Oxide

Fundamental stoichiometric relationships relating macroscopic quantities easily measured by experiment (masses) and to microscopic amounts (counts of atoms in moles) will investigated. At high temperature, magnesium reacts with oxygen present in air. Masses before and after the oxidation will measured, allowing the experimental empirical formula of magnesium oxide to be calculated and compared to the theoretical empirical formula, MgO. By assuming the product is strictly composed of only magnesium and oxygen, a percent yield of the reaction can also be found.

Objectives and Science Skills

Determine the empirical formula and percent yield of the ionic oxide produced by the reaction of Mg with O_2 based on experimental data.
Quantitatively and qualitatively evaluate experimental results relative to those theoretically predicted based on known chemical principles and stoichiometric calculations.
Identify and discuss factors or effects that may contribute to deviations between theoretical and experimental results and formulate optimization strategies.

Suggested Review and External Reading

Data analysis introduction (online), reference materials, textbook information on ionic compounds and empirical formulas

Introduction

A great deal of chemical knowledge has been amassed by using simple combustion experiments conducted with crucibles, burners, and balances. In this experiment, you are using this technique to experimentally determine the empirical formula of magnesium oxide. This experiment also illustrates the law of conservation of mass and the law of constant composition.

Magnesium metal reacts vigorously when heated in the presence of air (~79% N_2 and ~21% O_2 by moles). Although oxygen is more reactive than nitrogen, a small amount of magnesium nitride (and magnesium oxynitride) is produced and must be removed. This can be accomplished by adding water, which converts nitride to magnesium hydroxide and ammonia gas. Further heating of the mixed magnesium oxide and hydroxide drives off the hydrogen in the form of water vapor, leading to the conversion of the hydroxide to oxide.

The *unbalanced* equations summarizing the steps in the magnesium oxide synthesis are:

$$Mg(s) + O_2(g) + N_2(g) \xrightarrow{\text{heat}} \text{"}MgO, Mg_3N_2\text{"}(s)$$

$$\text{"}MgO, Mg_3N_2\text{"}(s) + H_2O(l) \rightarrow \text{"}MgO, Mg(OH)_2\text{"}(s) + NH_3(g)$$

$$\text{"}MgO, Mg(OH)_2\text{"}(s) \xrightarrow{\text{heat}} Mg_xO_y(s) + H_2O(g)$$

Balanced equations are not needed. The amount of Mg metal available to react limits the reaction yield, and the expected mole ratio of Mg-to-O is one-to-one based on the theoretical empirical formula, MgO.

Equipment list

Safety goggles
Magnesium ribbon, Mg
Crucible and lid
Analytical balance (reads to four decimal places, ±0.0001g)
Ring stand and support ring
Bunsen burner and striker
Clay triangle and clay tile
Tongs

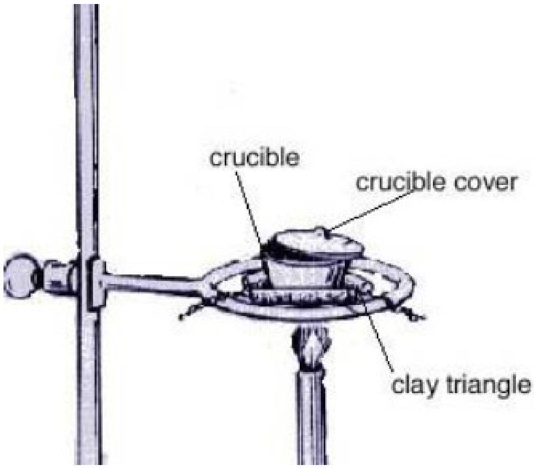
CAUTION

Eye protection and correct attire are essential.
Open flame will be present. Keep all non-essential materials away from it and pay attention to sleeves, papers, etc.
Once any burner is lit, assume ALL equipment is hot (burn risk).
Do not touch the crucible, lid, triangle, ring, or stand during or after they have been heated.
Do not look into the crucible when it is over flame (or very hot).
Do not place the hot crucible on the lab bench or any surface other than the clay tile.
Do not breathe any fumes emanating from the crucible.

Although the crucible is ceramic designed to withstand high temperatures, they can become brittle. If your crucible cracks or breaks, please get your TA to help you clean it up safely and correctly.

Procedure

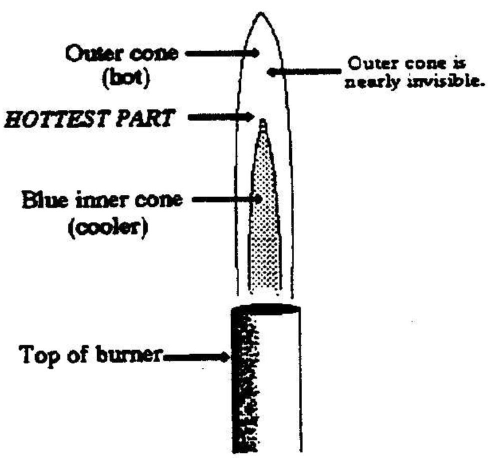
Please pay close attention to the TA demonstration. If you have any questions, please ask.



1. Practice handling the crucible and lid with the tongs.

Do not touch the crucible or lid with your hands. Use the tongs and clay tile.
Practice using the tongs to pick up and set down the crucible and the lid on the clay triangle and the clay tile.
When the crucible is in the clay triangle, practice placing the lid partially over the crucible so that there is a gap of about 0.5 cm (the lid should rest on the crucible edge and on two legs of the triangle).
Always hold the crucible steady with tongs while carrying it on the clay tile.

2. Adjust the ring height before heating/exposing to flame.



Start the burner and adjust the vent at the base. The flame should be blue and steady, and you should see two “cones”. The top of the inner cone, which is the hottest part of the flame, must be in contact with the bottom of the crucible for the reaction to occur.
You must adjust the height of the ring BEFORE putting the flame under the crucible.

Hold the burner by the base to the SIDE of the ring stand and adjust the ring to the correct height.
DO NOT ADJUST THE RING AFTER YOU HAVE EXPOSED IT TO FLAME (it will be hot).

3. Pre-fire your crucible – "gentle" versus "strong" heating (or "firing").

You should always gently warm up the cool crucible and lid before subjecting them to high heat.
Hold the burner by its base and pass it back and forth under the crucible and lid for 1-2 minutes.
After gently warming the crucible and lid, set the burner under them to "fire" them for about 3 minutes. The bottom of the crucible should glow red hot.

4. After the crucible and lid have cooled, record their mass to four decimal places on the analytical balance. Never place anything hot on a balance.

5. Obtain about 0.3000 g (~35 mm) Mg ribbon from the container on the reagent counter (please close the lid on the jar). Loosely fold the ribbon so that it fits into your crucible. Use your forceps; do not touch the Mg ribbon with your hands.

6. Record the mass of the crucible, lid, and Mg ribbon to four decimal places on the analytical balance.

7. Place the crucible securely on the clay triangle. Set the lid slightly off-center so that there is an approximately 0.5 cm gap between the lid and the edge of the crucible.

8. Gently heat the crucible, lid, and Mg for ~1-2 minutes and then fire them for about 15 minutes. There should be a gray-white powdery solid inside your crucible.

9. Allow the crucible, lid, and contents to cool for several minutes. Using a disposable pipet, add ~1 mL (10-15 drops) of deionized water to the contents of the crucible. While adding the water, use your tongs to hold a piece of pH paper moistened with deionized water over the crucible. Note any color change in the paper and determine if the vapors are acidic, basic, or neutral.

10. Set the lid slightly off-center so that there is an approximately 0.5 cm gap between the lid and the edge of the crucible. Gently heat the crucible, lid, and contents for ~1-2 minutes and then fire them for about 10 minutes.

11. Allow the crucible, lid, and oxide product to cool and then record the mass to four decimal places.

12. Follow your TA's instructions for disposing of the oxide product. Clean everything well and return anything borrowed to the reagent bench. The used disposable pipet goes into a broken glass box.

You may be asked to place the used crucible base into a waste container. Please do not dispose of the lid; return it clean and dry to the correct container on the reagent bench.

13. Calculate the mass of Mg used, the mass of oxide product, and, assuming that the increase in mass is due to oxide ions the ionic solid product, and the mass of O incorporated during the reaction. Your masses should be reported to four decimal places.

$$\text{mass Mg used} = (\text{mass crucible} + \text{lid} + \text{Mg ribbon}) - (\text{mass crucible} + \text{lid})$$
$$\text{mass oxide product} = (\text{mass crucible} + \text{lid} + \text{oxide products}) - (\text{mass crucible} + \text{lid})$$
$$\text{mass O incorporated} = (\text{mass oxide products}) - (\text{mass crucible Mg})$$

14. Calculate the moles of Mg and of O to four significant figures.

$$\text{mol Mg} = \text{mass Mg used} \times \frac{1\ \text{mol Mg}}{24.31\ \text{g Mg}}$$
$$\text{mol O} = \text{mass O incorporated} \times \frac{1\ \text{mol O}}{16.00\ \text{g O}}$$

15. Calculate the ratio of moles-Mg-to-moles-O in the product by dividing both mole values by the moles of O (first to four significant figures; then, rounded to two decimal places).

16. The values calculated in #15 may or may not be within 0.10 of a whole number. If they are, they are the subscripts in the empirical formula. If not, multiply both values by the appropriate integer as specified by the multiplier table in the inlab assignment to obtain the subscripts to use in your experimental empirical formula.

Examples:

Case 1) 0.3000 g Mg + 0.1962 g O
0.01234 mol Mg + 0.01226 mol O
1.007 mol Mg : 1.000 mol O $\rightarrow$ 1.01 mol Mg : 1.00 mol O
$Mg_{1.01}O_{1.00} \rightarrow MgO$

Case 2) 0.3000 g Mg + 0.1580 g O
0.01234 mol Mg + 0.009875 mol O
1.250 mol Mg : 1.000 mol O $\rightarrow$ 1.25 mol Mg : 1.00 mol O
$(Mg_{1.25}O_{1.00})_{\times 4} \rightarrow Mg_5O_4$

17. Calculate the percent by mass composition of the oxide product to four significant figures.

$$\% \text{ by mass Mg} = \frac{\text{mass Mg used}}{\text{mass oxide product}} \times 100\%$$
$$\% \text{ by mass O} = \frac{\text{mass O incorporated}}{\text{mass oxide product}} \times 100\% = 100.00\% - \% \text{ by mass Mg}$$

18. Calculate the theoretical yield and percent yield of the net oxidation reaction to four significant figures. $Mg(s) + \frac{1}{2} O_2(g) \rightarrow MgO(s)$

$$\text{theoretical yield of MgO} = \text{mol Mg} \times \frac{1 \text{ mol MgO}}{1 \text{ mol Mg}} \times \frac{40.31 \text{ g MgO}}{1 \text{ mol MgO}}$$
$$\% \text{ yield} = \frac{\text{experimental yield}}{\text{theoretical yield}} \times 100\%$$

Results / Sample Calculations

Complete the online inlab assignment or write a lab report as directed by your TA.

Masses Mg used, oxide product, O incorporated
Moles Mg and O
Mole ratio Mg-to-O and empirical formula
Percent by mass Mg and O
Theoretical yield and % yield

Discussion Questions and Review Topics

What did you find and how did you do it?
How does your experimental empirical formula compare to the theoretical? How does the % yield reflect this?
What were the major experimental sources of error? How would factors such as 1) incomplete oxidation of the Mg metal; 2) incomplete conversion of magnesium nitride (Mg_3N_2) to oxide; and/or 3) residual $Mg(OH)_2$ in the product affect your results?
Does this method appear to be an appropriate and valid way to experimentally determine the empirical formula of metal oxides such as MgO?