

53 **EMPIRICAL FORMULA OF MAGNESIUM OXIDE** 92 **U**

In this experiment, you will synthesize magnesium oxide via the reaction pathways summarized in Figure 1. Note that [1] is the main reaction and [2] is a side reaction.

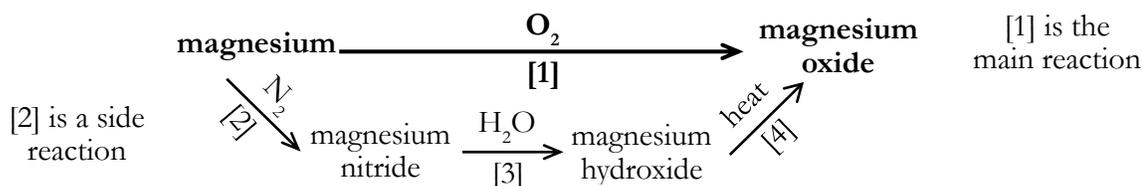


Figure 1. Pathways to form magnesium oxide from magnesium

When magnesium is heated in the presence of oxygen in air, magnesium reacts with oxygen to produce magnesium oxide [1]. The nitrogen in air may also react with magnesium to form magnesium nitride [2], but oxygen is much more reactive so there is a greater amount of the oxide formed than the nitride. The nitride that forms is removed by the addition of water which produces magnesium hydroxide [3], which is then converted to magnesium oxide by heating [4]. Thus, all the magnesium used as starting material ends up as magnesium oxide. These reactions are summarized in Table 1.

Table 1. Reaction summary

Reaction	Reactants	Products
[1]	$\text{Mg(s)} + \text{O}_2\text{(g)}$	$\text{Mg}_x\text{O}_y\text{(s)}$
[2]	$\text{Mg(s)} + \text{N}_2\text{(g)}$	$\text{Mg}_3\text{N}_2\text{(s)}$
[3]	$\text{Mg}_3\text{N}_2\text{(s)} + \text{H}_2\text{O(g)}$	$\text{Mg(OH)}_2\text{(s)} + \text{NH}_3\text{(g)}$
[4]	$\text{Mg(OH)}_2\text{(s)}$	$\text{Mg}_x\text{O}_y\text{(s)} + \text{H}_2\text{O(g)}$

By allowing these reactions to occur, the empirical formula of magnesium oxide can be determined. The empirical formula is the simplest chemical formula; the subscripts indicate the lowest whole number ratio of the elements in the compound. To demonstrate, consider the following experimental data in the synthesis of an oxide of chromium and the determination of its empirical formula, Cr_xO_y . The task is to find the values of x and y , where $x : y$ is the Cr : O molar ratio.

$$\begin{array}{rcl}
 m_{\text{chromium}} \text{ (mass of the starting material)} & = & 14.81 \text{ g} \\
 m_{\text{chromium oxide}} \text{ (mass of the product)} & = & 21.33 \text{ g}
 \end{array}$$

To work this out, the masses of chromium and oxygen in the chromium oxide must be known to calculate the number of moles for each element and thus the molar ratio. The mass of the chromium in the oxide product is equal to the mass of the chromium used in the reaction. The mass of oxygen can be deduced by subtracting the mass of chromium from the mass of the oxide product.

$$m_{\text{oxygen}} = m_{\text{chromium oxide}} - m_{\text{chromium}} = 21.33 \text{ g} - 14.81 \text{ g} = 6.52 \text{ g O}$$

The masses can be converted to moles using the molar masses, which leads to the Cr : O molar ratio, and thus the empirical formula:

$$\begin{aligned} \text{mol Cr} &= 14.81 \text{ g} \left(\frac{\text{mol}}{52.0 \text{ g}} \right) = 0.2848 \text{ mol Cr} \\ \text{mol O} &= 6.52 \text{ g} \left(\frac{\text{mol}}{16.0 \text{ g}} \right) = 0.4075 \text{ mol O} \end{aligned}$$

The calculated moles are first used as subscripts, but keeping in mind that the subscripts in the empirical formula should be expressed in the smallest whole number ratio. Both subscripts are then divided by the smaller subscript:

$$\text{Cr}_{0.2848}\text{O}_{0.4075} = \text{Cr}_{\frac{0.2848}{0.2848}}\text{O}_{\frac{0.4075}{0.2848}} = \text{Cr}_1\text{O}_{1.431}$$

In this case, since the resulting subscripts are not whole numbers, multiply by an appropriate factor

Ending close to 0.25	1/4	Multiply by 4
Ending close to 0.33	1/3	Multiply by 3
Ending close to 0.50	1/2	Multiply by 2
Ending close to 0.66	2/3	Multiply by 3
Ending close to 0.75	3/4	Multiply by 4

$$(\text{Cr}_1\text{O}_{1.431})_2 = \text{Cr}_2\text{O}_{2.862} = \text{Cr}_2\text{O}_3$$

The percent of oxygen from the data and from the resulting formula can be compared.

From the experimental data:

$$\% \text{O} = \frac{m_{\text{oxygen}}}{m_{\text{chromium oxide}}} \times 100 = \frac{6.52 \text{ g}}{21.33 \text{ g}} \times 100 = 30.6\%$$

and theoretically, from the molecular formula:

$$\% \text{O} = \frac{3(mw_{\text{oxygen}})}{mw_{\text{chromium oxide}}} \times 100 = \frac{3(16.0)}{152.0} \times 100 = 31.6\%$$

Why might the percent oxygen calculated from the experimental data (30.6%) be less than the expected theoretical percent oxygen (31.6%)? In this case, there is less oxide than expected. Keep this in mind while performing the experiment. Think about how oxygen is being added. What might affect this?

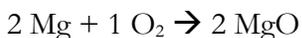
The percent yield for a reaction can be calculated as follows:

$$\% \text{ yield} = \frac{\text{actual yield}}{\text{theoretical yield}} \times 100$$

The actual yield is the amount of product actually recovered after doing the experiment, which in this case is the mass of the magnesium oxide product. The theoretical yield is the amount of product expected to be recovered based on the initial amount of reactant. In this case, the amount of magnesium oxide product theoretically calculated from the mass of the magnesium.

For example, if 1.69 g of magnesium was used for the experiment and 2.76 g of magnesium oxide was recovered after the experiment, then:

Actual yield = 2.76 g



$$\text{Theoretical yield} = 1.69 \text{ g Mg} \left(\frac{1 \text{ mol Mg}}{24.3 \text{ g}} \right) \left(\frac{2 \text{ mol MgO}}{2 \text{ mol Mg}} \right) \left(\frac{40.3 \text{ g}}{\text{mol MgO}} \right) = 2.80 \text{ g MgO}$$

$$\% \text{ yield} = \frac{\text{actual yield}}{\text{theoretical yield}} \times 100 = \frac{2.76 \text{ g}}{2.80 \text{ g}} \times 100 = 98.6\%$$

PROCEDURE

1. Obtain a clean, dry crucible with cover. Prepare the experimental set-up as shown on Figure 2.

Heat the crucible strongly for 5 minutes to remove moisture, oils and other contaminants. Using a clean crucible tongs, transfer the crucible to a wire gauze on your bench. **From here on, handle the crucible and cover, hot or not, only with crucible tongs, to avoid accidental burns and reintroduction of contaminants.** Allow the crucible to cool to room temperature; check for coolness by placing your hand close to the crucible without touching it. Weigh the crucible with the cover.

CAUTION: As a rule, objects weighed on the balance must be the same temperature as the balance. Do not place hot or warm objects on the balance pan.

CAUTION: Hot crucibles and cold crucibles look identical. Do not touch them with your hand and risk burning yourself.

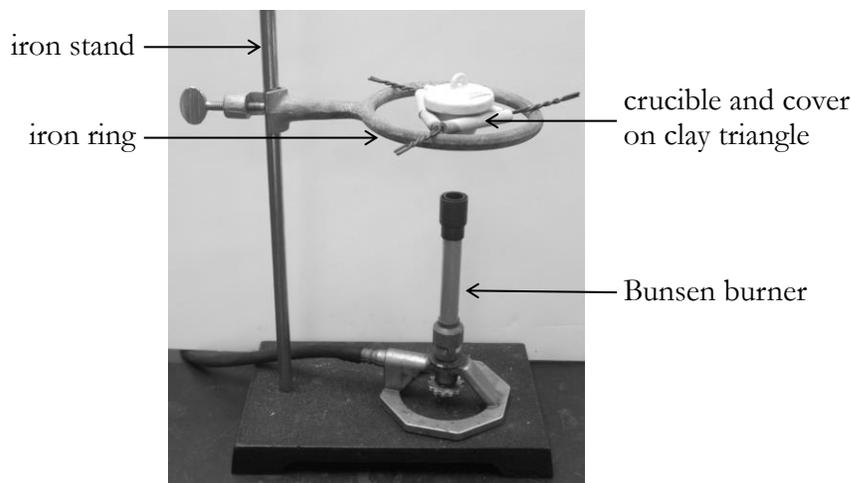


Figure 2. Experimental set-up for empirical formula determination

2. Obtain a piece of magnesium ribbon ~10 cm in length.
3. Coil the magnesium around a stirring rod or pencil. Slide the ribbon off and place it in the crucible. Weigh the covered crucible with magnesium.
4. Place the crucible on the clay triangle with the lid slightly offset (open ~ $\frac{1}{4}$ of the way) to allow air in while preventing contents from escaping, as shown in Figure 3. Heat the crucible strongly. If the heat is not strong enough, the reaction will not proceed. The instant the magnesium starts to burn, place the cover back on using crucible tongs in order to put out the fire. After 30 seconds, offset the cover slightly using crucible tongs and cover when the magnesium starts to burn. Continue repeating this until the magnesium no longer catches fire when the cover is lifted.

Note: If the magnesium is smoldering (glowing orange), leave the lid cracked. If the magnesium begins to smoke, burn (bright light), or spark place the cover back on using the tongs. After 30 seconds, offset the cover slightly using crucible tongs and cover when the magnesium starts to burn. Continue repeating this until the magnesium no longer catches fire when the cover is lifted.

CAUTION: The burning magnesium emits a very bright light that can be damaging to the eyes. Do not look at it directly.

5. When the magnesium stops glowing, cover the crucible and heat strongly for 10 minutes. Allow the crucible to cool.
6. Lift the cover to determine if **all of the magnesium ribbon** has turned into gray-white ash. (If some of it has not turned into ash, reheat strongly for another 10 minutes, then allow to cool.)



Figure 3. Letting air into the crucible

7. When the crucible has cooled, wet the ash with 10 drops of distilled water.
8. Cover the crucible and heat gently for 2 minutes, then strongly for 10 minutes. Allow the crucible to cool to room temperature on a wire gauze on your bench. Weigh the crucible with cover and product.

CLEAN-UP

- Dispose of the product in the designated container in the front hood.
- Wash the crucible and cover and leave them on your workstation.
- Make sure the Bunsen burner and gas valve are off, disconnect the tubing from the gas valve.

Name: _____

Date: _____

Partner's Name: _____

EMPIRICAL FORMULA OF MAGNESIUM OXIDE

DATA

mass of empty crucible and cover _____

mass of crucible, cover and magnesium _____

mass of crucible, cover and magnesium oxide product _____

POST-LAB QUESTIONS

Show clearly the complete calculations with correct number of significant figures and units.

1. Calculate the mass of the magnesium you started with.

2. Calculate the mass of the magnesium oxide product.

3. Calculate the mass of oxygen in the product.

8. Theoretically, what is the empirical formula of magnesium oxide?
From this formula, calculate the percent of oxygen in magnesium oxide.
9. Compare the percent of oxygen determined experimentally (Question 4) to that determined from the molecular formula (Question 8). Which is higher and why?
(Hint: What would affect the amount of oxygen measured in your experiment?)
10. What is the percent yield of your magnesium oxide product?
(Note: $2 \text{ Mg} + 1 \text{ O}_2 \rightarrow 2 \text{ MgO}$)