Determination of the Empirical Formula of Magnesium Oxide – Background

During the latter half of the 18th century, the French chemist Antoine Lavoisier performed numerous combustion experiments, meticulously measuring the masses of all reactants and all products, including those which were gases. Time after time, Lavoisier observed that while the physical and chemical properties of the products and reactants differed, the total mass of the products was always the same as the total mass of the reactants. Lavoisier summarized his discoveries in the law of conservation of mass which states "In a chemical reaction, matter is neither created nor destroyed." In today's experiment, you will measure the mass of your reactant magnesium, chemically transform it to magnesium oxide, and measure the mass of your product. Will the mass of magnesium atoms change from reactant to product? If your product has a greater mass than your reactant, where did the extra mass come from?

Today's experiment will demonstrate the law of conservation of mass, and more specifically, how the law can be used to determine the empirical formula of our intended product, magnesium oxide. While the molecular formula of a substance gives the actual number of atoms of each element in the substance, the empirical formula is the simplest, whole number ratio of atoms of each element in the substance. For instance, the empirical formula of glucose (molecular formula = C₆H₁₂O₆) is CH₂O, and the empirical formula of hydrogen peroxide (molecular formula = H₂O₂) is HO.

To determine the empirical formula of magnesium oxide, you will react elemental magnesium with elemental, atmospheric oxygen, to generate magnesium oxide.

\[ \text{Mg}(s) + \text{O}_2(g) \rightarrow \text{Mg}_x\text{O}_y(s) \]  \hspace{1cm} (1)

The lowest whole number ratio of moles of magnesium atoms to moles of oxygen atoms present in magnesium oxide will give the empirical formula of magnesium oxide.

\[
\frac{\text{moles of Mg}}{\text{moles of O}} = \frac{x}{y}
\]

You will have recorded the mass of magnesium used as a reactant, and the mass of the magnesium oxide product, but how can you know the moles of magnesium in the product? Worse yet, how can you know the moles of oxygen in the product when you never measured the mass of oxygen at any point? Use your knowledge of mass conservation, and a little logic!
References
(1) Roonguthai, W. *A chunk of vapor-deposited magnesium crystals produced by the Pidgeon process at a refinery in China. Its maximum dimension is about 24 cm long;* 2007. Used with permission under the GNU Free Documentation License.
(2) Hillier, W. *Liquid Oxygen;* Australian National University; 2006. Used with permission under the GNU General Public License.
(3) Public domain.
Determination of the Empirical Formula of Magnesium Oxide – Procedure

**BUNSEN BURNER SAFETY AND USE**

You must read and understand all guidelines presented herein. Failure to responsibly use a Bunsen burner can result in catastrophic harm to yourself, your classmates, the Chemistry Department, and the University.

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I. General Safety Guidelines

A. Preparation
   1. Goggles on!
   2. Long hair should be pulled back and secured. Use a ponytail or another reasonable solution.
   3. Roll up long sleeves, especially if they are loose and baggy.
   4. Clear all chemicals, other than those needed for the experiment, from the fume hood. This is critically important for flammable chemicals such as squeeze bottles of acetone.
   5. Identify the location of the nearest fire extinguisher.

B. During Use
   1. Once the burner has been lit, you cannot allow yourself to be distracted by anyone or anything. This includes your lab partner, neighbors, and your cell phone. A lack of focus leads to accidents.
   2. Never leave a lit burner unattended. If you must walk away, turn the burner off and relight it when you return.
   3. If you must move the burner to raise it, or change its position, always hold the burner by its base. The only part that will get hot is the barrel. Never touch the barrel when lit!
   4. Never move a lit burner from inside the fume hood to outside the fume hood. There is absolutely no reason for why you should ever need to do this!
   5. Keep the fume hood sash at, or below the OSHA designated maximum height.

C. In Case of an Accident
   1. Immediately get the attention of your laboratory instructor by any means necessary.
   2. Immediately turn off the main gas valve(s) in the fume hood.
   3. If a fire has taken hold in the fume hood, immediately pull down the hood sash and back away.
   4. *Never, ever* throw any chemical, including water, onto a lab fire. Chemical fires are not wood fires, and require the use of a properly rated fire extinguisher.

D. USE COMMON SENSE!
II. Know the Parts and What They Do

![Figure 1. Bunsen burner](image)

(a) barrel: Natural gas and air are directed up through the barrel. At the top, the gas undergoes combustion, producing a flame.

(b) collar: Air, specifically oxygen, is necessary for the combustion of the gas. The amount of air that mixes with the gas is adjusted via the collar.

more air = hotter flame
less air = cooler flame.

The collar for the model of burner shown in Figure 1 adjusts by twisting counterclockwise (open) or clockwise (close) like a screw, moving the collar up or down. The burner in Figure 1 is completely open.

(c) needle valve: This adjusts the flow of gas. Again, it works like a screw. Turn the wheel clockwise for more gas, counterclockwise for less gas (from the top perspective). Do not adjust gas flow with the main gas valve.

(d) gas inlet: Connect the hose-barb inlet to the main gas valve outlet in the fume hood using the appropriate designated tubing. Do not use the rubber hoses in your lab drawer for this purpose. The rubber hoses can melt and often have cracks and holes which can lead to accidents.

III. Lighting the Burner

Follow the steps:
1. Connect your burner to a gas outlet in a fume hood. Do not use the benchtop outlets, if available.
2. Close the needle valve and open the collar for medium air intake.
3. Turn on the main gas valve in the fume hood all the way.
4. Open the needle valve until you hear the hissing sound of gas.
5. Place the burner upright on the fume hood's benchtop. Place your spark lighter/striker over the top of the barrel and squeeze to light the burner. Repeat until the burner is lit.
6. Adjust both the air intake collar and needle valve for the desired flame.
**IV. Adjusting the Flame**

Using the proper flame for your needs is critically important to both the success of your experiment and your safety. A controlled flame is a safe flame. A flame that is out of control is an extreme safety hazard. Always hold the base of the burner steady while adjusting the air intake and/or the needle valve.

**A. Safe, Controlled Flames**

<table>
<thead>
<tr>
<th>hard flame (Figure 2a)</th>
</tr>
</thead>
<tbody>
<tr>
<td>• air intake collar = wide open</td>
</tr>
<tr>
<td>• needle valve = medium gas</td>
</tr>
<tr>
<td>This is an extremely hot flame, with the hottest part being the top of the clearly visible inner blue cone. This flame is also very noisy.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>soft flame (Figure 2b)</th>
</tr>
</thead>
<tbody>
<tr>
<td>• air intake collar = nearly closed</td>
</tr>
<tr>
<td>• needle valve = nearly closed</td>
</tr>
<tr>
<td>Adjust both the air intake collar and the needle valve until the blue cone is just barely visible. This flame is cooler and much less intense than the hard flame.</td>
</tr>
</tbody>
</table>

**B. Unsafe, Out of Control, Ridiculous Flames**

<table>
<thead>
<tr>
<th>Figure 3a</th>
</tr>
</thead>
<tbody>
<tr>
<td>• air intake collar = wide open</td>
</tr>
<tr>
<td>• needle valve = maximum gas</td>
</tr>
<tr>
<td>This flame is too large and too intense to safely use for any purpose. Close down the needle valve and you will get the hard flame of Figure 3a.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Figure 3b</th>
</tr>
</thead>
<tbody>
<tr>
<td>• air intake collar = nearly closed</td>
</tr>
<tr>
<td>• needle valve = maximum gas</td>
</tr>
<tr>
<td>This orange, dirty flame results from too much gas and not enough oxygen. Although it is the coldest flame, it is too large, unwieldy, and unpredictable to use safely. Closing the needle valve will give the soft flame of Figure 2b.</td>
</tr>
</tbody>
</table>

**IV. Shutdown**

- Always turn off a Bunsen burner by **completely** closing the main gas valve in the fume hood.
- Do not use the needle valve to turn off a burner.
- Absolutely do not attempt to "blow out" the burner like a candle!
**Procedure:**

Obtain a nickel crucible, crucible lid, and a clay triangle. Clean the crucible, removing any loose particulate matter. It is not necessary to remove all debris from the crucible as most of it has been fused to the nickel and cannot be removed. Visually inspect the crucible and lid for structural defects or flaws like cracks and holes. Give any such defective crucible to your instructor. In your fume hood, set up the apparatus as shown in Figure 4.

![Figure 4. Crucible setup](image)

(a) ring stand  
(b) crucible lid  
(c) iron ring  
(d) clay triangle  
(e) crucible

Using your crucible tongs, practice the following techniques on the cool crucible:

- lifting the lid from the crucible  
- placing the lid ajar so that the crucible is slightly open, but the lid will not fall off, as in Figure 4.  
- quickly, but gently pushing the lid from its ajar position to completely cover the crucible  
- lifting the crucible, with its lid, from the clay triangle  
- carrying the crucible and lid while supporting from underneath with your wire gauze (Figure 5)

![Figure 5. Proper technique for carrying a cool crucible. Never, ever attempt to carry a hot crucible!](image)

(a) crucible tongs  
(b) wire gauze

Practicing these techniques may seem silly, but it is worth your time to do so. Would you rather repeat a trial because you dropped a crucible?

With your covered crucible in the clay triangle, fire the crucible for 3-5 minutes. "Firing a crucible" is the process of heating the crucible with a Bunsen burner to remove water, oils from your hands, and any other potentially volatile contaminants. Use a soft flame; hot enough to turn the bottom of the crucible orange, but not so hot that you risk melting the nickel crucible. Turn off your burner and allow the crucible and lid to completely cool to room temperature. You should allow the crucible to cool for at least 15 minutes. You can test whether the crucible is cool by placing your hand one inch over the crucible and sensing for radiant heat. At no point after firing the crucible should you touch the crucible with your hands, even after it has cooled.
As the crucible is cooling, obtain 0.3 g of magnesium (~35 cm of ribbon). Wear gloves and use your spatula to scrape the surface oxidation from both sides of the magnesium ribbon to expose the shiny metal. Coil the magnesium ribbon into a tight spiral, capable of sitting flat on the bottom of the crucible, and record its exact mass. Once the crucible and lid have cooled to room temperature, lift them from the clay triangle and record their combined mass. Use your crucible tongs to lift and hold the crucible, and use your wire gauze to support it from underneath as in Figure 5.

Return the crucible and lid to the clay triangle and place the coiled magnesium ribbon into the crucible. The magnesium will burn most efficiently if the coil is sitting flat on the bottom of the crucible. Adjust the crucible lid so that it is ajar, but not at risk of falling off (Figure 4). This will allow atmospheric oxygen to enter the crucible. Heat the bottom of the crucible with the Bunsen burner until flashes of light from inside the crucible are visible. You may want to turn off the lights in the fume hood to see the flashes of light more clearly. Do not look at the burning magnesium. In fact, as soon as you see flashes of light, quickly push the crucible lid to completely cover the crucible, and remove the flame. This prevents the fluffy magnesium oxide from floating away.

After the initial flashes of light, allow the covered crucible to sit without heating for 2-3 minutes. After 2-3 minutes, use your crucible tongs and reposition the lid so that it is once again ajar. If "white smoke" appears when you open the lid, immediately close the lid and wait another minute. This "smoke" is your product and allowing it to escape will greatly affect your calculated empirical formula. Once the "smoke" is no longer apparent, keep the lid ajar and repeat the heat – flash of light – cover – wait 3 minutes process 4 or 5 more times. You must repeat the process until, when heated, there are no more flashes of light emitted from the crucible. This absence of light signals the complete reaction of the magnesium. Once complete, allow the crucible and lid to cool for 15 minutes.

To this point, we have assumed that magnesium has reacted with atmospheric oxygen according to equation 1 (unbalanced).

\[ \text{Mg(s)} + \text{O}_2(g) \rightarrow \text{Mg}_x\text{O}_y(s) \]  \hspace{1cm} (1)

However, the atmosphere is composed of 21% O\(_2\), 78% N\(_2\), and 1% trace gases. Although oxygen is more reactive, there is almost four times more nitrogen present, and the extreme temperatures to which the magnesium is subjected, allow for some magnesium to react with atmospheric nitrogen (equation 2).

\[ 3\text{Mg(s)} + \text{N}_2(g) \rightarrow \text{Mg}_3\text{N}_2(s) \]  \hspace{1cm} (2)

Any magnesium nitride which forms can be converted to magnesium oxide by addition of water, to initially form magnesium hydroxide and ammonia (equation 3),

\[ \text{Mg}_3\text{N}_2(s) + 6\text{H}_2\text{O}(l) \rightarrow 3\text{Mg(OH)}_2(s) + 2\text{NH}_3(g) \]  \hspace{1cm} (3)

followed by heating the intermediate magnesium hydroxide, driving off water, and generating the intended magnesium oxide (equation 4, unbalanced).

\[ \text{Mg(OH)}_2(s) \rightarrow \text{Mg}_x\text{O}_y(s) + \text{H}_2\text{O}(g) \]  \hspace{1cm} (4)
Once the crucible has cooled for 15 minutes, add 1 mL of deionized H₂O to the crucible. Try to wet the majority of the solid. If you do not allow your crucible to cool, the addition of water may result in significant splattering of your product and hot water. Depending on the amount of Mg₃N₂ present, the addition of water may result in a bubbling reaction, releasing ammonia gas (equation 3). Do not breathe this gas! Replace the lid, completely covering the crucible and heat gently with your Bunsen burner for 3-5 minutes. Turn off your burner and allow the crucible and lid to cool to room temperature (15 minutes or longer). You must perform at least two trials, so while the crucible cools, you can prepare a second crucible for trial two. Once the crucible has cooled to room temperature, record the combined mass of the crucible, lid, and magnesium oxide product.

Waste Disposal:
• Dispose of the magnesium oxide in the proper solid waste container, brushing out any loose particulate matter. Return the crucible, lid, and clay triangle.

Calculations:
For each trial
1. calculate the moles of magnesium present in your magnesium oxide.
2. calculate the mass of magnesium oxide formed.
3. calculate the mass of oxygen present in your magnesium oxide.
4. calculate the moles of oxygen present in your magnesium oxide.
5. calculate the lowest whole number ratio of \( \frac{\text{moles of Mg}}{\text{moles of O}} \), rounding appropriately.

Determine the average ratio for your trials (a ratio can be expressed as a single number, right?). Calculate the percent error in your average ratio, using the accepted molar ratio for magnesium oxide. From your average ratio, report the experimentally determined, average empirical formula for magnesium oxide.

Conclusion:
Report your average empirical formula for magnesium oxide and compare it to the accepted empirical formula. Report your percent error in the molar ratio and discuss whether it is a good or bad result. If it is a poor result, identify some specific sources of error which might have affected your result. Even if your result is good, still list some potential sources of error. How might you fix these sources of error if you were to repeat the experiment? Include the complete balanced chemical equation for the formation of magnesium oxide from elemental magnesium and elemental oxygen.