Experiment 5 -Determination of the Empirical Formula of Magnesium Oxide

 When magnesium and oxygen are heated together, they readily undergo a chemical change (reaction):

magnesium + oxygen **→** magnesium oxide (Rxn.1)

From the masses of magnesium and oxygen that combine, we can calculate the empirical formula of magnesium oxide. We will weigh the magnesium before it combines with the oxygen, and we will also weigh the product of the reaction, magnesium oxide. The final weighing is necessary because we need to subtract the original weight of magnesium from this weight of product. We "weigh" the oxygen in this indirect fashion because it is easier to do than to try to weigh the oxygen gas before it combines with the magnesium.

 If magnesium is heated in open air, its reaction with oxygen is rapid and spectacular. The metal catches fire, burning with an intense white flame and with production of white smoke. (The metal has been used in flares to provide light for night-time military operations.) We can slow down the reaction by limiting the supply of oxygen that reaches the magnesium; we do that by putting a cover on the crucible that contains the magnesium sample.

 Our procedure in this experiment is complicated by another fact. Magnesium is such an active metal that it reacts with the relatively inactive element nitrogen:

magnesium + nitrogen **→** magnesium nitride (Rxn. 2)

This occurs in competition with the reaction of magnesium with oxygen, so it is called a "side reaction." You should realize that this side reaction uses up some of the magnesium that is supposed to be combining with oxygen. Fortunately it is possible to "undo" the reaction in this way:

magnesium nitride + water  **→** magnesium hydroxide + ammonia (Rxn. 3)

 *and*

magnesium hydroxide **→** magnesium oxide + water vapor (Rxn. 4)

To get reactions (3) and (4) to occur, we add water to the crucible contents at the end of the first heating period. We then heat again to speed up reactions (3) and (4) and to evaporate any excess water.

**Safety Precautions:**

* Wear your safety goggles.
* When the magnesium is burning, do not look directly at the flame. It is bright enough to damage your eyes.

**Waste Disposal:**

* The solid product of this reaction can be discarded in the regular garbage can after the experiment.

Procedure

1. Heat a clean, dry porcelain crucible with cover on a clay triangle (supported on a ring stand), using a direct flame, for about 5 minutes. This is to make certain that the crucible is dry.
2. If your crucible is not clean, wash it. Follow step 1 to remove all water from the crucible and lid.
3. Turn off the burner, and let the crucible and cover cool. Leave the crucible and cover resting on the clay triangle.
4. Weigh the empty, dry crucible and cover on the digital balance.
5. Obtain a strip of magnesium ribbon that is 10-15 cm long.
	1. **NOTE:** If it is not shiny, polish it briefly with steel wool to remove any oxide coating.
6. Cut the magnesium ribbon into 0.5 cm strips so that it will fit inside the crucible.
7. Carefully weigh the crucible, cover, and magnesium ribbon together. Record this total mass, from which the mass of the ribbon will be calculated.
8. Start heating the crucible with the magnesium in it and the cover on.
9. After heating for about 1 min, open the lid slightly to let air into the crucible. The magnesium will ignite.
	1. **NOTE:** Magnesium is used in flares. It burns hot and white. **DO NOT LOOK AT THE LIGHT-IT CAN BURN YOUR RETINA!**
10. Continue heating the covered crucible for a minute or so, then take off the cover again. Wait for the magnesium to catch fire again, and then quickly re-cover the crucible.
11. Repeat steps 9 and 10 until the magnesium no longer catches fire when the cover is removed. You need to do this step several times.
12. Heat the sample strongly for 3-5 minutes, with the cover slightly on the crucible. Make sure the bottom of the crucible becomes red hot.
13. Turn the burner off and let the crucible cool until it is only barely warm to the touch.
14. To the **cooled crucible**, add about 2-5 drops of distilled water. Make sure to wet the entire surface of the sample, not just one spot.
15. Warm the crucible with the damp sample using a gentle flame for a minute or so, then heat it moderately strongly for about 5 minutes. (The crucible need not become red hot for this phase of the experiment, and the cover is not needed for this heating.)
16. Let the crucible and contents cool until the crucible is barely warm to the touch.
17. Weigh the cooled crucible, cover, and contents of the crucible, recording this total mass.
18. Heat the crucible again for 1-3 min.
19. Weigh the cooled crucible, cover, and contents of the crucible, recording this total mass. You are bringing the crucible and its contents to constant mass.
20. When the masses agree to ± 0.02g, you are done.

Calculations

1. From the total mass recorded in step 16 and the mass of the empty crucible and cover, you can calculate the mass of the crucible contents. These contents should be fairly pure magnesium oxide.
2. Calculate the mass of oxygen that combined with your mass of magnesium in this experiment.
3. Calculate the number of moles of magnesium you started with.
4. Calculate the number of moles of oxygen that reacted with the magnesium.
5. Calculate the mole ratio of magnesium to oxygen. To do this, divide the moles of magnesium by the moles of oxygen. Express the result to the correct number of significant figures.
6. Using the above ratio, write the empirical formula of magnesium oxide. Round off the ratio to the nearest whole number.

Questions:

Questions 1 & 2 are typed.

1. In this experiment you determined the mass of oxygen (that combined with a weighed mass of magnesium) by subtraction: mass of product minus mass of original magnesium = mass of oxygen that combined.

 As a result of this procedure, anything that was in the crucible at the end of the experiment, along with the magnesium oxide product, would cause an error in the figure that is recorded as "mass of oxygen". Would extra mass in the crucible cause the "mass of oxygen" to come out too high or too low? Explain.

2. The correct formula for magnesium oxide is MgO, a 1.0 to 1.0 ratio. But sometimes in this experiment the ratio of Mg to O comes out too low. (Example: 0.9 Mg to 1.0 O) In that case, it means that there was too much oxygen relative to the mass of magnesium. At other times it comes out that the ratio is too large. An example would be: 1.2 to 1.0 (Mg to O). In such a case it must be that there has been too little oxygen (or too little weight at the end of the experiment, which registers as too little oxygen.)

 In each case below, decide whether the situation described would lead to a calculated ratio of too much oxygen, or too little oxygen, and explain your reasoning. (The calculated ratio contains the mistake.)

 a. Putting in more water than is needed for reaction 3, and then not drying out this excess water.

 b. Having some Mg(OH)2 left in the crucible instead of converting it to MgO.

 c. Having some Mg3N2 in the crucible as product, instead of pure MgO.

 d. Forgetting to weigh the cover along with the crucible and contents at the end.

 e. Letting a lot of the dense white smoke escape from the crucible during the burning.

3. Here are some data from a similar experiment, to determine the empirical formula of an oxide of tin. Calculate the empirical formula according to these data.

 **mass of crucible, cover, and tin sample** 21.76 g

 **mass of empty crucible with cover** 19.66 g

 **mass of crucible and cover and sample,**

 **after prolonged heating gives constant weight** 22.29 g