# **Experiment 5 -Determination of the Empirical Formula of Magnesium Oxide**

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

 $magnesium + oxygen \rightarrow magnesium oxide$  (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  $\rightarrow$  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. Turn off the burner, and let the crucible and cover cool. Leave the crucible and cover resting on the clay triangle.
- 3. Weigh the empty, dry crucible and cover on the digital balance.
- Obtain a strip of magnesium ribbon that is about 60 to 70 cm long.
   NOTE: If it is not shiny, polish it briefly with steel wool to remove any oxide coating.
- 5. Fold or coil the magnesium ribbon so that it will fit inside the crucible. **NOTE:** Don't coil it so tightly that it will be difficult for the magnesium to react completely. Wrapping it around a pencil works well.
- 6. Carefully weigh the crucible, cover, and magnesium ribbon together. Record this total mass, from which the mass of the ribbon will be calculated.
- 7. Put the cover aside for a moment, and start heating the crucible with the magnesium in it. Have the cover close by, with some crucible tongs ready to handle it with.
- 8. The instant that the magnesium starts to burn, put the cover on the crucible (using the crucible tongs) to put out the fire.
- 9. Continue heating the covered crucible for a minute or so, then take off the cover again. Wait for the magnesium to catch fire again, then quickly re-cover the crucible.
- 10. Repeat steps 9 and 10 until the magnesium no longer catches fire when the cover is removed.
- 11. Heat the sample strongly for 5 minutes, with the cover <u>on</u> the crucible. Make sure the bottom of the crucible becomes red hot.
- 12. Turn the burner off and let the crucible cool until it is only barely warm to the touch.
- 13. To the <u>cooled crucible</u>, add about 10 drops of distilled water. Make sure to wet the entire surface of the sample, not just one spot.
- 14. Warm the crucible with the damp sample using a gentle flame for a minute or so, then heat it moderately strongly for about 10 minutes. (The crucible need not become red hot for this phase of the experiment, and the cover is not needed for this heating.)
- 15. Let the crucible and contents cool until the crucible is barely warm to the touch.
- 16. Weigh the cooled crucible, cover, and contents of the crucible, recording this total mass.
- 17. Perform the calculations, and write your mole ratio of Mg to O on the chalkboard along with your name.
- 18. Write down the results of the class into your notebook.

### **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.
- 7. Using student results that have been listed on the chalkboard, calculate the standard deviation in the mole ratio for the class. You may either:

  a. Calculate the standard deviation, showing all of your work, for 5 student results, or b. Calculate the standard deviation for the results of the entire class on your calculator.

## Questions

- 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 <u>calculated</u> ratio of too much oxygen, or too little oxygen, and <u>explain your</u> 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)<sub>2</sub> left in the crucible instead of converting it to MgO.
- c. Having some Mg<sub>3</sub>N<sub>2</sub> 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

4. Once you have calculated the standard deviation of the student results for the class, explain its significance by stating the 68% and 95% confidence intervals. (We have 95% confidence that the true value of the mole ratio lies between and assuming only random errors.)