MAGNESIUM OXIDE SYNTHESIS AND ANALYSIS

OBJECTIVES: In this experiment magnesium oxide will be prepared from its elemental components. The results will be analyzed to evaluate the composition of the compound.

SKILLS: weighing, stoichiometric calculations

EQUIPMENT: ring stand, ring, clay triangle, Bunsen burner

REFERENCE: Chemistry: The Molecular Nature of Matter, Jespersen et al., 7th edition, Sections 3.1–3.4, Safety video (course website)

SAFETY AND DISPOSAL: Magnesium burns with an extremely bright flame that should not be viewed directly. Care should be taken to avoid burns when checking the hot crucible. Solids from the experiment can be disposed of directly into the trash after they have cooled.

INTRODUCTION: When new materials are discovered or synthesized by scientists, it is important to determine their composition. In particular, the chemical formula tells the how many atoms of each kind are present. For example, there are two compounds made of hydrogen and oxygen: water (H_2O) and hydrogen peroxide (H_2O_2). Each has different properties. By measuring the mass of each element that is used to form a compound, it is possible to determine the mass percentage of each element (which is independent of sample size) and from that, to determine the chemical formula. This can be used to demonstrate the law of definite composition (showing that there is a fixed ratio of elements in a compound). If the formula of the compound is known, then theoretical weight percentages can be calculated and a comparison made to the experimental data.

Using ammonia as an example, if we find experimentally that it is composed of 82.4% nitrogen and 17.6% hydrogen, then we can find its formula NH_3 in the following way: From the weight percentages, a 100 g sample would contain 82.4 g N and 17.6 g H or

$$5.89 \text{ moles of N} = (82.4\text{g N})*\left(\frac{1 \text{ mol N}}{14.01\text{g N}}\right) \quad \text{and} \quad 17.6 \text{ moles of H} = (17.6\text{g H})*\left(\frac{1 \text{ mol H}}{1.008\text{g H}}\right)$$

The chemical formula of ammonia shows the ratio of H to N atoms. To find the ratio for this example, the 17.6 moles of H is divided by the 5.89 moles of N. The answer (2.99) shows that the ratio is 3 H to 1 N (within the experimental uncertainty), and that therefore the correct formula in NH_3.

The formula NH_3 leads to the theoretical weight percent composition in the following way:

$$\text{Wt \% N in } NH_3 = \left(\frac{\text{mass of N in 1 mole } NH_3}{\text{mass of 1 mole of } NH_3}\right) \text{ X } 100\% = \left(\frac{14.0 \text{ g N}}{17.0 \text{ g } NH_3}\right) \text{ X } 100\% = 82.4\% \text{ N}$$

Similarly, the result leads to 17.6% H in NH_3 .

In this experiment, the masses of magnesium and oxygen in magnesium oxide will be measured. The weight percentages and mole ratios will be calculated from the experimental data and compared to the theoretical values. Note that in the discussion above, the starting point for the calculations uses elements in their monatomic form, even when they naturally occur as diatomic molecules.

It may be helpful to note that there are two ways to find the mole ratio between oxygen magnesium:

Either use g $Mg_{(xpt)}$ → moles Mg and g $O_{(xpt)}$ → moles O → mole ratio

or else % Mg → g Mg → moles Mg and % O → g O → moles O → mole ratio

EXPERIMENTAL PROCEDURES

Set up the apparatus as described during the lab demonstration and heat the crucible for 5 minutes or until its bottom is a dull red color. Turn the burner off and allow the crucible to cool. This process will burn off any combustible and volatile materials and make the experimental weights more accurate. Do not move the crucible until it cools and be careful not to get burned by touching it while it is still hot. **<u>Do not place a hot crucible on paper towels</u>, use only a heat-resistant wire gauze square.** While waiting for the crucible to cool, obtain a piece of magnesium ribbon about 15 cm long. Polish the ribbon with steel wool to remove the oxide that sometimes forms on the surface of the magnesium. When the crucible has cooled, weigh it to the nearest $\pm$ 0.001 g. Place the magnesium ribbon in the crucible and re-weigh the crucible and magnesium together to determine the combined mass of the crucible and magnesium ribbon.

Place the magnesium ribbon, loosely coiled, into the crucible, cover it, and heat until the bottom of the crucible again glows with a dull red color. After about a minute at this temperature, momentarily lift the crucible cover. This allows oxygen in and should result in the ignition of the magnesium. (A bright light, visible inside the crucible, confirms this.) Return the crucible cover quickly and continue heating for 10 minutes. If there was no sign of ignition the first time the cover was removed, continue heating for another 2 minutes and then lift the cover again. At

the end of ten minutes, the crucible should be heated for 2 to 3 minutes while holding the cover above the crucible or allowing it to rest tilted to one side of the crucible. (A bright glow and white "smoke" at this time indicates that the ignition was not complete; if this occurs, the cover should be replaced and heating should continue for an additional 5 minutes.) Allow the crucible to cool to room temperature and reweigh it to the nearest $\pm$ milligram ($\pm$0.001 g).
From the data, it is possible to calculate the masses of magnesium, oxygen, and magnesium oxide and use this information to determine the percent composition of the compound. This data can be understood in a variety of ways. If the formula of magnesium oxide is already known, then the results can be compared with a prediction of percent composition from known atomic weights. The data can also be used to determine the empirical formula from the percent composition. To do this, the masses of oxygen and magnesium should be converted into moles (by division with atomic weights). The number of moles of oxygen and magnesium in the compound can then be compared. If they are equal, then the formula is 1: 1 yielding MgO. If they are unequal, then the relative number of moles will give insight into writing the formula.

Repeat the experiment a second time to test the reproducibility of the data. When you have finished, enter your data into the lab computer. As part of your lab report, you will use Excel to calculate the average mole ratio of magnesium to oxygen and the empirical formula of magnesium oxide from the class data.

Notebook Template: Your lab notebook should include the masses of (1) the empty crucible, (2) the crucible with magnesium, and (3) the crucible with magnesium oxide for each of the two trials. From this data, you can calculate the mass of magnesium used and the mass of oxygen that combined with the magnesium. From these masses, you can calculate the number of moles of each of these elements, and then compare their values. Be careful to use the correct number of significant figures in reporting your measurements and in your calculations. In addition, your lab notebook should include observations of what you saw during the experiment.

LAB REPORT

Class data for this experiment will be available on Blackboard. Use Excel to calculate the empirical values for the mass % magnesium and mass % oxygen for each student in the class. If any points are to be excluded, note them in your lab report. Determine the average values and use these average values to find the mole ratio of Mg/O for your class. For the lab report, you will need to prepare a table of results in Excel that has 2 columns and 5 rows. Label the two columns as mass % magnesium and mass % oxygen. Label the rows as (1) your average value, (2) the class average, (3) the theoretical value, (4) your difference, relative to the class value and (5) the class relative error.

Your difference, relative to the class value, gives insight into how your results compared to those of others in the class. It is defined as **[(your value – class average value) / (class average value)] x 100%** . A positive (negative) relative error means that your value was higher (lower) than the class average. The class relative error is a measure of how close the class result is to the theoretical value. It can be used to evaluate the experiment itself. The class relative error is defined as **[(class average - theoretical value) / (theoretical value)] x 100%** .