Determination of a chemical formula: the synthesis of zinc iodide

OBJECTIVES: In this experiment zinc iodide will be prepared from its elemental components. The results will be analyzed to evaluate the composition and to determine the empirical formula of the compound.

SKILLS: weighing, stoichiometric calculations

EQUIPMENT: 125 mL Erlenmeyer flask, 250 mL beaker, watch glass, hot plate, aluminum foil

REFERENCES: Chemistry: Matter and its Changes, Brady & Senese, 5th ed, pp. 87 – 103, *Chemistry in the Laboraotry*, Postma, Roberts, Hollenberg, 7th ed.

SAFETY AND DISPOSAL: Solid iodide should be handled with care. Methanol is flammable and toxic. Any heating of methanol should be done under a fume hood. There should be no open flames in the laboratory when working with methanol.

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

$$82.4 \ g \ \text{N} \times \frac{1 \ mol \ \text{N}}{14.01 \ g \ \text{N}} = 5.89 \ mol \ \text{N} \qquad 17.6 \ g \ \text{H} \times \frac{1 \ mol \ \text{H}}{1.008 \ g \ \text{H}} = 17.6 \ mol \ \text{H}$$

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, elemental zinc will be reacted with elemental iodine to produce zinc iodide. The masses of iodide and zinc reacted will be measured along with the mass of the zinc iodide product. The weight percentages and mole ratios will be calculated from the experimental data and compared to the theoretical values.

It may be helpful to note that there are two ways to find the mole ratio between zinc and iodine:

Either use   g $Zn_{(reacted)}$ → moles Zn   and   g $I_{2(reacted)}$ → moles **I** (<u>monatomic</u> I) → mole ratio

or else   % Zn → g Zn → moles Zn   and   % I → g I → moles I → mole ratio

EXPERIMENTAL PROCEDURE

Weigh a clean, dry 125 mL Erlenmeyer flask on a milligram balance in the lab and record the mass. Next, weigh out approximately 2.0 ± 0.1 g of granular zinc metal on a plastic weigh boat. This amount of zinc will be enough to provide **excess** zinc for the reaction. Add the zinc metal to the Erlenmeyer flask and reweigh the flask. Record the mass of the flask + zinc.

Weigh out 2.0 ± 0.1 g of solid iodide crystals using a plastic weigh boat. This amount of iodine will be completely consumed by the reaction and is the **limiting reactant.** Be careful not to spill any of the iodine crystals. Solid iodine is corrosive and if left on a balance pan can lead to failure of the balance. Add the iodide to the Erlenmeyer flask containing the zinc metal and reweigh the flask. Record the mass of the flask + zinc + iodine.

Obtain 25 mL of methanol from the dispensing stations located in the large fume hoods and add it to the Erlenmeyer flask. Put an aluminum foil cap on the flask, crimping it around the edges to hold it in place. Place the flask on a hot plate positioned under your mini fume hood. Adjust the temperature setting on the hot plate to a low–medium level, sufficient to

heat the methanol to boiling. While heating, swirl the flask occasionally to dissolve the iodine. After 15 to 20 minutes, the reaction between the zinc and iodine will be complete as evidenced by the disappearance of the dark brown color of dissolved iodine. When the reaction is complete the methanol solution should be pale yellow to nearly colorless and unreacted zinc granules should be easily visible.

While waiting for the zinc–iodine reaction to finish, accurately weight a clean, dry 250 mL beaker and record the mass. The weighed beaker will be used to isolate the zinc iodide product.

When the reaction is complete, remove the flask from the hot plate. A folded strip of paper towel wrapped around the neck of the flask will protect your fingers from the heat. Remove the aluminum foil cap and under your fume hood, slowly and carefully pour the warm solution (which contains the zinc iodide) into the previously weighed 250 mL beaker. **Be careful not to let any of the solid zinc particles escape from the flask.**

It is necessary to wash the solid zinc left in your flask in order to remove all traces of the zinc iodide product. Add 5 mL of methanol to the flask and swirl for 15 seconds. Allow the solid zinc to settle then add the methanol to the solution in your 250 mL beaker. Repeat this step with two additional methanol washings of 5 mL each. Three rinses will ensure that all of the zinc iodide reaction product gets transferred to the beaker.

Place the flask containing the unreacted zinc back on the hot plate set to a medium setting to dry. Leave the foil cap off. Swirl the flask occasionally to redistribute the zinc on the bottom of the flask and speed up drying. When all the methanol has evaporated from the flask and the zinc particles look dry and move freely on the bottom the flask, remove the flask from the hot plate. Allow it to cool to room temperature for 10–15 minutes. Observe the zinc remaining in the flask. If there is a white residue on the surface of zinc then all of the zinc iodide product was not transferred and a few more rinses with methanol are needed. Be sure to transfer these rinses into your weighed 250 mL beaker containing the previous rinses. Once the flask is cool (**room temperate**), the zinc is dry, and you are sure all of the zinc iodide has been removed, reweigh the flask and record the mass. You can now calculate the mass of zinc and iodine that have reacted.

While the zinc is drying you can begin to isolate the zinc iodide product. Place your weighed 250 mL beaker containing the methanol solution of zinc iodide on the hot plate under your fume hood. Allow all of the methanol to evaporate. If the solution shows a tendency to boil irregularly (i.e. bump or splatter), stir it occasionally with a glass stirring rod until the methanol has nearly all evaporated. It normally takes about 20 minutes to evaporate all of the methanol. When the last traces of methanol have completely disappeared, allow the beaker to heat for an additional 2 minutes and then remove it from the hot plate. Allow the beaker to cool to room temperature for 15 minutes. Reweigh the beaker containing the dry zinc iodide and record the mass.

Repeat this procedure for a total of two trials. 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 and the empirical formula of zinc oxide from the class data.

From the data, it is possible to calculate the masses of zinc and iodine reacted as well as the mass of zinc iodide produced. You will 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 zinc iodide 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 zinc and iodine reacted should be converted into moles (by division with atomic weights). The number of moles of zinc and iodine in the compound can then be compared. In addition, the principle of conservation of mass can be confirmed by comparing the mass of zinc and iodine reacted with the mass of the zinc iodide product.

Notebook Template: For each trial your lab notebook should include the masses of (1) the empty flask, (2) the flask + zinc initially, (3) the flask + zinc + iodine, (4) the flask + leftover dry zinc, (5) mass of empty 250 mL beaker, and (6) mass of beaker + dry zinc iodide product. From this data, you can calculate the mass of zinc and iodine reacted used and the mass of the product. 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 values for the mass % zinc and mass % iodine 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 Zn/I 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 % zinc and mass % iodine. 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%** .