Laboratory Experiment No. 3

The Empirical Formula of a Compound

Introduction

An initial look at mass relationships in chemistry reveals little order or sense. Mass ratios of elements in a compound, while constant, do not immediately tell anything about a compound's chemical formula. For instance, water always contains the same proportions of hydrogen (11.11% by mass) and oxygen (88.89% by mass) but these figures do not tell how the formula H_2O is obtained.

A chemical formula is a usually a whole number ratio showing the relative numbers of moles of each element present. A chemical formula can be determined from the mass of each element present in a sample of compound by taking into account their relative atomic masses. The unit of chemical quantity is not mass but the gram-molecular weight, or mole, which is the formula mass of a substance expressed in grams. As a result, a mole of any substance can be weighed out. Moreover, a mole of any substance contains the same number (Avogadro's number, 6.022×10^{23}) of formula units. For example, a mole of U weighs 238.029 g and contains Avogadro's number of uranium atoms. One mole of H_2O weighs 18.015 g and contains Avogadro's number of H_2O molecules. One mole of NaC1 weighs 58.443 g and contains Avogadro's number of NaC1 formula units, that is, NaCl ion pairs.

In some cases, it is experimentally straightforward to find the mass of each element in a sample of compound. Dividing the mass of each element by the mass of one mole of the element (its atomic mass) gives the moles of each element present. The resulting molar ratio, when simplified to small whole numbers, gives the empirical formula of the compound. The molecular formula, which gives the number of each kind of atom in a molecule of compound, may be identical to or a multiple of the empirical formula. It is necessary to know the molecular weight of a compound to obtain the molecular formula from the empirical formula. For instance, hydrogen peroxide has the empirical formula HO. (This formula only means the hydrogen peroxide molecule contains equal numbers of hydrogen and oxygen atoms.) Its molecular weight, however, is 34, not 17 Daltons. The 34-Dalton formula mass corresponds to the molecular formula H_2O_2.

Example 1: To find the empirical formula of a sulfide of copper, 1.956 g of copper wire is heated with a large excess of elemental sulfur. An excess of sulfur ensures that all the copper reacts. Unreacted sulfur burns off to leave behind copper sulfide weighing 2.477 g. The calculations are as follows:

Mass of Cu wire:	1.956 g
Mass of copper sulfide formed:	2.447 g
Mass of combined sulfur:	0.491 g

The molar amount of each element is found by multiplying its mass by the conversion factor derived from the equality: one mol of any element = the atomic mass of the element expressed in grams.

$$1.956\,\text{g Cu} \times \frac{1\,\text{mol Cu}}{63.546\,\text{g Cu}} = 3.078 \times 10^{-2}\ \text{mol Cu}$$

$$0.491\,\text{g S} \times \frac{1\,\text{mol S}}{32.066\,\text{g S}} = 1.53 \times 10^{-2}\ \text{mol S}$$

The molar amounts of each element depend on the amount of copper starting material. More important is the molar *ratio*, which remains the same no matter how much starting material you have. Find the smallest whole number molar ratio by dividing both molar amounts by the smaller of the two values.

$$\frac{3.078 \times 10^{-2}\ \text{mol Cu}}{1.53 \times 10^{-2}} = 2.01\,\text{mol Cu} \qquad \frac{1.53 \times 10^{-2}\ \text{mol S}}{1.53 \times 10^{-2}} = 1.00\,\text{mol S}$$

The molar ratio of the two elements is close to 2:1, so the empirical formula of the compound must be Cu_2S. This is an ionic compound; there are no molecules present so the question of a molecular formula does not arise.

Example 2: Commercial analytical laboratories often provide the percent composition of a compound sent in for analysis. (Example 2 does not apply directly to this experiment report.) Percent composition values require an extra computational step to find the empirical formula. For instance, the composition of an organic compound is reported to be 53.38% carbon, 11.18% hydrogen and 35.53% oxygen by mass. For simplicity, assume that you have exactly 100 g of the compound so you can work with the mass quantities 53.38 g C, 11.18 g H and 35.53 g O. Next, find the number of moles of each element in the hypothetical 100-gram sample.

$$53.38\,\text{g C} \times \frac{1\,\text{mol C}}{12.011\,\text{g C}} = 4.444\,\text{mol C}$$

$$11.18\,\text{g H} \times \frac{1\,\text{mol H}}{1.00794\,\text{g H}} = 11.09\,\text{mol H}$$

$$35.53\,\text{g O} \times \frac{1\,\text{mol O}}{15.9994\,\text{g O}} = 2.221\,\text{mol O}$$

As before, convert the molar amounts to ratios by dividing each molar amount by the smallest number of moles present, in this case 2.221 (mol of oxygen).

$$\frac{4.444\,\text{mol C}}{2.221} = 2.001\text{ mol C} \cong 2\text{ mol C}$$

$$\frac{11.09\,\text{mol H}}{2.221} = 4.993\text{ mol H} \cong 5\text{ mol H}$$

$$\frac{2.221\,\text{mol O}}{2.221} = 1.000\text{ mol O} \cong 1\text{ mol O}$$

The empirical formula of this molecular compound is C_2H_5O, which has a 45.012 g/mol formula mass. A separate experiment gives a compound molecular mass of 90.12 g/mol. Therefore, the molecular formula must be twice the empirical formula or $C_4H_{10}O_2$. Verify this for yourself as an exercise.

At times, computations like the previous ones give decimal fraction molar ratios that need to be converted to whole numbers by multiplying all the ratios by the same small whole number such as 2, 3 etc. Thus an atomic or molar ratio of 1:1.33:1 becomes 3:4:3. Similarly, $NO_{1.5}$ becomes N_2O_3.

Percent Composition

Since percent composition is a common way of reporting analytical results, it is important to know how to calculate percent compositions from analytical data. The mass fraction of each element present is the mass of an element in the sample divided by the total sample mass. Each fraction multiplied by 100% gives the percent of that element in the compound.

For the 2.447 g sample of Cu_2S in Example 1,

$$\frac{1.956\text{ g Cu}}{2.447\text{ g of compound}} \times 100\% = 79.93\%\text{ Cu by mass}$$

$$\frac{0.491\text{ g S}}{2.447\text{ g of compound}} \times 100\% = 20.1\%\text{ S by mass}$$

Laboratory Experiment No. 3

The Empirical Formula of a Compound

EXPERIMENTAL PROCEDURE

NOTE:

- The magnesium oxide preparation described below can be safely done on an open laboratory bench.
- Molten magnesium metal corrodes porcelain so it is preferable to use old crucibles rather than clean new ones.
- It is necessary to cover burning magnesium during most of the procedure to avoid losing magnesium oxide which may escape as dense, white smoke. Covers limit the reaction rate and capture most of the airborne magnesium oxide that would otherwise escape the system.

Obtain an old crucible and a cover of a size that will span the entire crucible opening so you can easily adjust its position to control the amount of air entering the crucible. Place a layer of dry sand (to be dried the night before for the students) about 0.5 cm deep in the bottom of the crucible to provide some protection from the corrosive hot magnesium. The presence of sand in the crucible will not affect your weighing results. Sand is extremely resistant to heat and chemical changes. Once the sand is dried, its mass will not change and it will not react with the burning magnesium. Consider the sand to be part of the container in which you weigh your magnesium and magnesium oxide. As long as the container mass does not change, the mass of anything in it will always equal the total system mass minus the container mass.

1. Record the original mass of the crucible, dried sand, and cover to 0.001g (at least 3 times, on the same balance until you get three close readings). If your balance does not read consistently, tell your instructor. It is not necessary to zero in your balance. Do not move your balance, adjust the zero knob or switch to another balance once you perform your initial weighing.

2. Place 0.8 to 0.9 g of magnesium turnings on the sand in the bottom of the crucible and measure the total mass of crucible, sand, magnesium and cover. Put the covered crucible and its contents on a clay triangle mounted on a ring stand and heat the assembly strongly for ten minutes with the cover slightly ajar. The magnesium will glow as it reacts with air and forms a mixture of magnesium oxide and magnesium nitride. Do not remove the cover completely because this will admit too much air and accelerate the rate of the reaction. That may allow combustion products to escape as visible white smoke, reducing the amount of oxide in the crucible.

3. After ten minutes, stop heating and remove the cover to admit air. The magnesium may flare up slightly in response to incoming oxygen. A slight flare-up is acceptable. If the magnesium flares up

strongly and white smoke escapes, place the cover on again ajar and heat for another five minutes. When it appears the magnesium will no longer react strongly, heat the crucible with the cover off until the magnesium stops glowing. Then, leaving the cover off, heat the crucible to redness for 4 min more to complete the reaction.

4. From now on, handle the crucible carefully in case it has cracked from the corrosive hot magnesium. A crack will not affect your results as long as the crucible does not break apart and spill its contents.

5. Let the crucible cool to room temperature. Add enough distilled water to wet the magnesium oxide/nitride mixture and sand completely. This will convert the nitride ion in the magnesium nitride side product to hydroxide ion and ammonia (Equation 1). Waft some of the escaping vapors toward you to smell the ammonia.

6. Carefully heat the crucible with the cover in place to evaporate all the water without spattering the product. Then heat the crucible to redness with the cover on for 4 minutes to drive off residual water vapor and convert any hydroxide ion present to oxide (Equation 2). Let the crucible cool to room temperature and determine the total mass of crucible, cover, sand, and magnesium oxide.

$$N^{3-} + 3\ H_2O(l) \rightarrow 3\ OH^- + NH_3(g) \qquad \text{(Equation 1)}$$

$$2\ OH^- \rightarrow O^{2-} + H_2O(g) \qquad \text{(Equation 2)}$$

7. After you weigh the cooled crucible, scrape out the sand and magnesium oxide and dispose of them as heavy metal waste. Neither component in the crucible meets the criteria of hazardous waste, but is preferable to dispose of them this way, rather than as ordinary trash. Do a second trial either concurrent or after the first trial. Your instructor will examine your data to see if you did the experiment correctly. Do a third trial if one the instructor feels one of your determinations was not done satisfactorily. Report your two best trials separately. Do not average your results.

Safety

Chemical splash goggles and a waterproof apron must be worn at all times during this and all chemistry experiments, from the *very* beginning to the *very* end of the time you spend in the laboratory.

Disposal

Dispose of the sand and magnesium oxide product in the heavy metal hazardous waste container as explained above.

Cleanup

Use a test tube brush and soapy water to wash the crucibles and rinse them with tap water. Keep one crucible in your laboratory locker and return all others to the laboratory supply area. At the conclusion of the lab period, wipe down all work surfaces with a damp sponge.

Laboratory Experiment No. 3

<u>The Empirical Formula of a Compound</u>

Data Page for Magnesium Oxide Determination

Trial Number	1	2	3	4
Mass of Crucible, Cover and Sand (At least three times)				
Average Mass of Crucible, Cover and Sand				
Mass of Crucible, Cover, Sand and Magnesium Metal (At least three times)				
Average Mass of Crucible, Cover, Sand and Magnesium				
Mass of Magnesium Metal (By Subtraction)				
Mass of Crucible, Cover, Sand and Magnesium Oxide (At least three times)				
Average Mass of Crucible, Cover, Sand and Magnesium Oxide				
Mass of Magnesium Oxide (By Subtraction)				
Mass of Oxygen (By Subtraction)				

Laboratory Experiment No. 3

<u>The Empirical Formula of a Compound</u>

Report

For full credit, show clear, concise setups for all your calculations. Express all answers with proper units, the proper number of significant figures and, where appropriate, in proper scientific notation.

1. Calculate the empirical formula of magnesium oxide using the data from your two best trials. (4 points)

2. Calculate the percent composition of the tin oxide from the masses of the elements in your two best trials. (4 points)

3. If some magnesium oxide were lost by spattering, would it *appear* that your oxide product contained more or less oxygen? Explain the reasoning for your answer. (2 points)

4. For one of your trials, use the experimental masses of tin and oxygen to calculate the number of tin atoms and the number of oxygen atoms in your tin oxide sample. (2 points)

Laboratory Experiment No. 3

<u>The Empirical Formula of a Compound</u>

Prestudy

1) What is the difference between an empirical and a molecular formula? (1 point)

2) What is the proper empirical formula for the compound $ClO_{3.5}$? (1 point)

3) A compound has the empirical formula CH_2O and a molecular weight of 150.0 Daltons. What is the molecular formula? (1 point)

4) Calculate the mass of 1.00 mole of F_2. (1 point)

5) What are two hazards associated with the use of concentrated nitric acid? (1 point)

6) What is the empirical formula of a compound that consists of 69.9 % Fe and 30.1 % O? Show your calculation steps. (3 points)

7) What is the percent composition of a compound if a 3.25 mg sample contains 3.00 mg of carbon and 0.25 mg of hydrogen? Show your setup. (2 points)