Lab #3: The Empirical Formula of a Compound

Introduction

A look at the mass relationships in chemistry reveals little order or sense. The ratio of the masses of the elements in a compound, while constant, does not tell anything about the formula of a compound. For instance, while water always contains the same amount of hydrogen (11.11% by mass) and oxygen (88.89% by mass), these figures tell us nothing about how the formula H_2O is obtained.

A chemical formula is a whole number ratio showing the relative numbers of the atoms present. It can be determined from the masses of each element present, if you take into account the differing masses of each kind of atom. The standard of chemical quantity is not mass but the number of moles (mol) of particles (ions, atoms or molecules). The mass of one mole of any substance is numerically equal to its formula weight. Thus, one mole of carbon atoms has a mass equal to 12.011 grams. One mole of glucose ($C_6H_{12}O_6$) has a mass equal to 180.15768 grams. One mole of nitrate ions (NO_3^-) has a mass equal to 62.0049 grams. You should verify these formula weights for yourself.

In many cases, it is a straightforward matter, experimentally, to find the mass of each element present in a sample of a compound. Dividing each mass by the mass of one mole (the atomic mass) of that element gives the number of moles of each element present. The resulting ratio, when simplified to small whole numbers, gives you the empirical formula of the compound, which is the smallest whole number atomic ratio. The molecular formula may be a multiple of that and requires the molecular mass be determined. For instance, hydrogen peroxide has the empirical formula HO. Its molecular mass, however, is approximately 34, which corresponds to a molecular formula of H_2O_2.

Example 1**:** One simple (but expensive) experiment is determining the empirical formula of silver chloride. A weighed sample of silver is converted to silver chloride by a chemical reaction and the mass of silver chloride, Ag_xCl_y, is determined. Typical results might be:

Mass Ag:	1.007 g
Mass AgCl:	1.338 g
By difference: Mass Cl:	0.331 g

The number of moles of each element is calculated by multiplying its mass by the conversion factor derived from the equality 1 mol = the atomic mass of the element expressed in grams

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For Ag: $$1.007\text{ g Ag}\left(\frac{1\text{ mol Ag}}{107.870\text{ g Ag}}\right) = 0.009335\text{ mol Ag}$$

For Cl: $$0.331\text{ g Cl}\left(\frac{1\text{ mol Cl}}{35.453\text{ g Cl}}\right) = 0.00934\text{ mol Cl}$$

The numbers of moles of each element present in silver chloride are essentially the same, so the empirical formula must be AgCl (meaning Ag^+ and Cl^- are present in a 1:1 ratio). Since this is an ionic compound, there are no molecules present and the question of a molecular formula does not arise.

Example 2: In some cases, you may be supplied with the percent composition of a compound from an analytical laboratory. This requires an extra computational step to find the empirical formula. For instance, an organic compound is reported as consisting of 53.38% carbon, 11.18% hydrogen and 35.53% oxygen. For simplicity, assume you have 100.00 grams of the compound. Then you can work with 53.38 g C, 11.18 g H and 35.53 g O. First find the moles of each element in the 100 g.

Carbon: $$53.38\text{ g C}\left(\frac{1\text{ mol C}}{12.011\text{ g C}}\right) = 4.4443\text{ mol C}$$

Hydrogen: $$11.18\text{ g H}\left(\frac{1\text{ mol H}}{1.00794\text{ g H}}\right) = 11.092\text{ mol H}$$

Oxygen: $$35.53\text{ g O}\left(\frac{1\text{ mol O}}{15.9994\text{ g O}}\right) = 2.2207\text{ mol O}$$

These can be converted to whole number ratios by dividing each by the smallest number present (2.2207):

Carbon: $$\frac{4.4443}{2.2207} = 2.001\text{ mol C} = 2\text{ mol C}$$

Hydrogen: $$\frac{11.092}{2.2207} = 4.995\text{ mol H} = 5\text{ mol H}$$

Oxygen: $\frac{2.2207}{2.2207} = 1.000 \text{ mol O} = 1 \text{ mol O}$

Thus the empirical formula of the compound is C_2H_5O. This compound is found to have a molecular weight of 45.061, thus its molecular formula is the same as the empirical formula. **Verify this for yourself!**

In some cases, the last step will result in simple decimal fractions instead of whole numbers. These are converted into whole numbers by multiplying the decimal numbers by the smallest whole number that will successfully convert **all** the values to whole numbers. Thus, a ratio of $N_{1.0}O_{1.5}$ would be changed to N_2O_3 by multiplying by 2 and a formula ratio of $C_{1.00}H_{1.33}O_{1.00}$ would be changed to $C_3H_4O_3$ by multiplying by 3.

Since percent composition is the preferred method of reporting analytical results, you should know how to calculate it from analytical data. The fraction of each element present is the mass of that element in the sample, divided by the total mass of the sample.
Thus for the 1.338 g of silver chloride in example 1:

$$\%Ag = \frac{1.007 \text{ g Ag}}{1.338 \text{ g silver chloride}} \text{ x } 100 = 75.26\%$$

$$\%Cl = \frac{0.331 \text{ g Cl}}{1.338 \text{ g silver chloride}} \text{ x } 100 = 24.7\%$$

In this experiment, you will make two valid determinations of the empirical formula of an oxide of tin, which is a compound composed of only tin and oxygen. You will take a known mass of tin and react it with nitric acid, the source of oxygen, to obtain tin oxide. The other products of the reaction, nitrogen dioxide and water, will be removed by heating, leaving only tin oxide as the isolated product. The difference in mass between the original tin and the tin oxide is the mass of oxygen in the compound.

The unbalanced equation for the reaction is:

$$Sn_{(s)} + HNO_{3\,(aq)} \rightarrow Sn_xO_{y\,(s)} + H_2O_{(g)} + NO_{2\,(g)}$$

Alternatively, you might make an oxide of magnesium, by reacting a known mass of magnesium with oxygen from the air. The unbalanced equation for the reaction is

$$Mg_{(s)} + O_{2\,(g)} \rightarrow Mg_xO_{y\,(s)}$$

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Procedure

To save time, work on two determinations simultaneously, using two crucibles. Weigh each crucible to 0.001 grams. Obtain pieces of tin sheet metal. (If the tin surfaces are not shiny, buff them with steel wool and wipe clean with a paper towel.) Follow the procedure below for *each* of your trials in this experiment.

Place tin in one of the crucibles and weigh. The mass of the tin should be about 1 g. Be sure that the mass is not greater than 1.1 g or less than 0.75 g. Obtain a 10 - 15 mL supply of concentrated HNO_3 in a small beaker. IN THE HOOD slowly add 3 mL of concentrated HNO_3 to the tin and gently swirl (DO NOT STIR WITH A STIRRING ROD) until all the evolution of brown NO_2 gas has stopped. Add another 3 mL of the acid and swirl until brown gas evolution has stopped. Then add the acid dropwise with an eyedropper, continuing to swirl, until no more NO_2 is evolved. There should now be white precipitate visible and no tin pieces visible. Use the minimum amount of acid to convert the metal to a white solid.

Heat the mixture on a hot plate, in the hood, at a setting of **2** with intermittent swirling to complete the reaction and drive off the water and nitrogen dioxide.

CAUTION: Do not get nitric acid on your hands. If you accidentally do, wash immediately with soap and running water and report to your instructor.

Continue heating with occasional swirling until the sample appears dry. The product should be white at this point. If there are still pieces of metal visible, allow the sample to cool to room temperature and add HNO_3 dropwise, while swirling, until no more brown gas is evolved. Then repeat the heating process. If all the tin has been converted to a white solid, transfer the crucible to a wire gauze and heat (gently at first) with a hot flame for 5 minutes.

CAUTION: NO_2 is toxic and irritating. Avoid inhaling these fumes.

Return the crucible to the flame and heat for 5 minutes over the Bunsen burner. Allow the crucible to cool and obtain the mass of crucible plus the tin oxide. Reheat the crucible and contents for another 5 minutes over the Bunsen burner, allow it to cool and weigh. The two masses should be within 0.005 grams of each other. If a large difference is found, reheat for another 5 minutes. This is called heating to constant weight and insures that all water has been driven off. The tin oxide may have a tan or yellow color at this time. This will not affect your results. **Discard the tin oxide in the container provided.**

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Alternate Procedure

Weigh two dry crucibles and covers. Put 0.5 to 0.6 g of magnesium turnings into each of the crucibles and reweigh. Put one crucible on a clay triangle-iron ring assembly and heat strongly for ten minutes with the cover slightly ajar. The magnesium will glow as it is converted to a mixture of its oxide and nitride. Do not open the cover and let any of the white smoke (the combustion products) out of the crucible. After ten minutes, stop heating and remove the cover to let in air. The magnesium may flare up slightly in response to the added air. With the cover off, reheat until the magnesium stops glowing, then heat the crucible to redness for five more minutes.

Allow the crucible to cool nearly to room temperature. (While the first crucible is cooling, begin heating the second one as described above.) To the first, cool crucible, add the minimum amount of water needed to wet the oxide/nitride mixture. This will convert magnesium nitride to magnesium hydroxide and ammonia (Equation 1). Waft some of the escaping vapors toward you to see if you can smell the ammonia. Carefully, to avoid spattering, heat the crucible with the cover slightly ajar to evaporate the water. Then remove the cover and heat to redness to five minutes to convert the magnesium hydroxide to magnesium oxide (Equation 2). Let the crucible cool to room temperature and weigh the crucible, its cover and contents. (While the first crucible is cooling, continue with the second crucible.) Heat the first crucible to redness a second time then cool and weigh again. If the first and second weights are not within 0.005 g of each other, heat the crucible for a third time, cool and weigh. You should obtain two successive heating, cooling and weighings that are within 0.005 g. (This process is referred to as heating to a constant weight.) Finish the heatings, coolings and weighings with the second crucible.

Equation 1: $N^{3-} + 3\ H_2O \rightarrow 3\ OH^- + NH_3$

Equation 2: $2\ OH^- \rightarrow O^{2-} + H_2O$

NAME ________________________________ General Chemistry I (FC, 09 - 10)

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DATA SHEET

Metal used: ________________

	Trial 1	Trial 2
Mass of crucible	____________	____________
Mass of crucible plus metal	____________	____________
Mass of metal	____________	____________
Mass of crucible plus metal oxide (first heating)	____________	____________
Mass of crucible plus metal oxide (second heating)	____________	____________
Mass of crucible plus metal oxide (third heating, if necessary)	____________	____________
Mass of crucible plus metal oxide (fourth heating, if necessary)	____________	____________
Mass of metal oxide*	____________	____________
Mass of oxygen	____________	____________

* Use the lowest mass of crucible plus metal oxide to calculate the mass of metal oxide.

Instructor's Initials ______________

Results and Calculations

1. (2) If some metal oxide splattered out of your crucible, **how** would this affect the amount of oxygen in your empirical formula? Explain your answer.

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2. (6) Calculate the percent composition by mass of the metal oxide for both trials.

3. (12) Calculate the empirical formula of metal oxide for both trials. Show all your work clearly for full credit. Use your masses of metal and oxygen for these calculations. (To avoid a potential error, do not use your percentages.)

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PRESTUDY (page 1 of 2)

1. (l) A compound has the molecular formula $C_{12}H_8F_2$. What is its empirical formula?

2. (3) Consider the compound $Fe(NO_3)_2$.
a. What is the molecular weight of $Fe(NO_3)_2$?

b. What is the mass of 1.58 x 10^{-2} moles of $Fe(NO_3)_2$?

c. How many moles are there in 350.0 grams of $Fe(NO_3)_2$?

3. (l) A compound has an empirical formula of C_3H_2O and an approximate gram-formula-weight (molecular weight) of 270.24 g/mol. What is its molecular formula?

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PRESTUDY (page 2 of 2)

4. (3) What is the empirical formula of a compound that consists of 47.20% V and 52.80% F? Show your work.

5. (2) What is the percent composition of $Cu(OH)_2$? Show your work.