THE EMPIRICAL FORMULA OF AN OXIDE

Introduction

The empirical formula of a compound gives the simplest ratio of the relative number of atoms in the compound. This ratio is usually a ratio of small whole numbers.

The molecular formula of a compound gives the actual number of atoms of each element in the compound. The molecular formula provides more information than the empirical formula. The empirical formula gives only the relative number of atoms in the compound. Glucose, a sugar, has the molecular formula $C_6H_{12}O_6$. The empirical formula of glucose is CH_2O , which shows that the relative numbers of atoms of carbon and oxygen in glucose are the same. However, there are twice as many hydrogen atoms as carbon and oxygen atoms.

The molecular formula may be found from the empirical formula if the molecular mass of the compound is known. The molecular mass of glucose is 180.16 amu. Thus the empirical formula, CH₂O, which gives the simplest ratio of the atoms in the compound, must be multiplied by an integer to give the correct molecular mass. In this case the integer is 6, which gives a molecular formula of $C_6H_{12}O_6$ and a molecular mass of 180.16 amu.

The empirical formula of a compound may be determined by finding the mass of each element in the compound. The masses are converted to the number of moles of the elements. These data can then be used to determine ratios of the number of atoms. From these ratios, the empirical formula is obtained.

For example, a compound containing carbon (C), hydrogen (H), and oxygen (O), consists of 40.92 g of carbon, 4.58 g of hydrogen, and 54.50 g of oxygen. What would be the empirical formula of the compound? First, find the number of moles of each element in the compound.

Number of moles of carbon = $\frac{\text{mass of carbon}}{\text{molar mass of carbon}}$ = $\frac{40.92 \text{ g}}{12.01 \text{ g mol}^{-1}}$ = 3.407 mol C Number of moles of hydrogen = $\frac{\text{mass of hydrogen}}{\text{molar mass of hydrogen}}$ = $\frac{4.58 \text{ g}}{1.01 \text{ g mol}^{-1}}$ = 4.53 mol H

$$=\frac{54.50 \text{ g}}{16.00 \text{ g mol}^{-1}}$$
$$= 3.406 \text{ mol O}$$

Thus, we arrive at the formula $C_{3.407}H_{4.53}O_{3.406}$. However, since chemical formulas are written with whole numbers, we cannot have 3.407 carbon atoms, 4.53 hydrogen atoms, and 3.406 oxygen atoms. We convert to whole numbers by dividing by the smallest number of moles:

Carbon ratio	Hydrogen ratio	Oxygen ratio
3.407 mol C = 1.000	4.53 mol H _ 1.33	3.406 mol O _ 1.000
3.406 mol O	3.406 mol O	3.406 mol O

This gives us $CH_{1.33}O$; we need to convert 1.33 to a whole number. This can be done by trial and error: 1.33 x 2 = 2.66

 $1.33 \ge 3.99 \simeq 4$

Because 1.33 x 3 gives us an integer (4) we can multiply all the subscripts by 3 and obtain $C_3H_4O_3$ as the empirical formula of the compound.

In this experiment, the empirical formula of the oxide of magnesium will be determined. The reaction will be carried out by burning a piece of magnesium in air. Some of the magnesium will react with oxygen in the air to form the desired oxide:

magnesium + oxygen \rightarrow an oxide of magnesium

However, nitrogen in the air will interfere, producing a nitride of magnesium:

magnesium + nitrogen \rightarrow magnesium nitride

The nitride is converted readily to the desired oxide by treatment with water, followed by heating. The reaction with water produces magnesium hydroxide and ammonia. Upon heating, water is driven off as the hydroxide is converted to the desired product:

magnesium nitride + water \rightarrow magnesium hydroxide + ammonia

magnesium hydroxide <u>heat</u> magnesium oxide + water

Since all of the magnesium used will be converted ultimately to magnesium oxide, the moles of magnesium in the final product will be determined from the initial mass of magnesium used. The mass of oxygen incorporated will be obtained by weighing the final product and subtracting the mass of magnesium.

OPERATING A BUNSEN BURNER

Many types of laboratory burners are used, but most are modifications of the basic Bunsen burner. Three of the types that we use in the lab are shown below.



Figure 1 Laboratory burners.

Note that each burner has adjustable air vents; with the first type of burner gas flow is controlled directly from the laboratory bench valve, the second type has a gas control valve (needle valve) at its base, and the third type has a needle valve located above the base.

The fuel used is propane gas. Propane burns in air, producing carbon dioxide and water vapour.

$$CH_3CH_2CH_3 + 5O_2 \longrightarrow 3CO_2 + 4H_2O$$

Gas enters the inlet in the base of the burner through a rubber tube that leads from the gas supply valve on the laboratory bench. The gas then passes into the barrel, where gas and oxygen are mixed. Attached to the bottom of the barrel are air vents that may be adjusted to increase or decrease the amount of oxygen mixed with the gas.



Figure 2 Parts of a laboratory burner.

Lighting the Burner

- 1. Connect the hose to the gas outlet on your laboratory bench.
- 2. Open the needle valve if your burner has one. Be careful not to open the needle valve too far, it may come off. This could be dangerous (flame would come from the base).
- 3. Partially open the air vents.
- 4. Turn on the gas and light the burner. Hold the flint lighter over the top of the barrel, and close enough so that the sparks produced won't fail to light the burner.
- 5. Control the gas flow at the gas valve on the laboratory bench (or the needle valve if your burner has one). By regulating the gas flow you can adjust the height of the flame.
- 6. Adjust the gas control valve and the air vents to get a flame of the desired height and

intensity. You will learn how to control the burner flame by trial and error. For most laboratory work you should adjust the burner so that the flame is free of yellow color and free of the roaring noise caused by admitting too much air.

7. If your burner won't light, TURN OFF THE GAS VALVE, and consult the lab instructor.

Factors influencing Flame Properties

- 1. **Too little air** results in a **yellow sooty flame**. The yellow is due to glowing hot carbon particles produced by incomplete fuel consumption. This flame is called a luminous or cool flame. It is seldom used for laboratory work and it deposits soot on your equipment.
- 2. A high ratio of air to gas produces a very hot flame with a blue inner cone. The hottest part of the flame is just above the blue cone. The cone itself consists of gases that are still cool (unburned gas). This flame is called a non-luminous flame and is used for bringing porcelain crucibles to red heat and for softening glass.
- 3. A moderate flame is obtained by reducing the air supply somewhat. A pale uniform blue flame with no traces of yellow is obtained. It is best for boiling water and for other gentle heating.



Figure 3 Burner flame characteristics.

CRUCIBLES AND DESICCATORS

Clean the crucible and cover; support them on a clay triangle on an iron ring, above the burner.

Heating crucibles

Any object that is heated using a burner should be heated slowly for the first few seconds and cooled slowly for the last few seconds. Suddenly plunging a crucible into a very hot flame, or suddenly removing a very hot flame completely from under a crucible can cause the crucible to crack. To begin heating, first wave the flame rapidly under the object, then slowly under the object, then heat strongly. The crucible should be heated to a dull redness. When you want to stop heating, do not merely shut off or remove the burner but reverse the process (wave the flame first slowly, then rapidly, then remove). In this way you will get no sudden cooling or heating; thereby preventing cracked or broken crucibles, lost yields, and ruined experiments.

After heating, the crucible should be cooled in a desiccator and, when cool (room temperature), weighed to the proper number of significant figures on an analytical balance. The crucible and cover should be handled with crucible tongs. Even the cleanest hands will leave an oily deposit, which changes the mass of the crucible. Figure 4 illustrates the recommended procedure for handling a crucible with crucible tongs.



Figure 4 Use of crucible tongs.

Desiccators

A desiccator is a container used for storing samples and other materials that must be kept very dry. It contains a moisture absorbing material called desiccant and has a cover that closes over the desiccator so that it is air-tight.

Whenever a sample or a crucible or another object has to be brought to constant weight, it must be protected from moisture at all times when not actually being weighed or heated. Using a desiccator helps prevent objects from absorbing moisture from the air.

PROCEDURE

Caution: Wear safety glasses while doing this experiment.

Note: Throughout the procedure, use crucible tongs to handle the crucible and cover.

- 1. Obtain a clean dry crucible and cover and examine for cracks. The crucible and cover are fragile. Use caution.
- 2. Weigh the crucible and cover on an analytical balance. Record the mass to the nearest 0.0001 g on the Data Sheet (2).
- 3. Place an iron ring on a support. Place a clay triangle on the ring. Allow sufficient height to place a burner beneath the ring as shown in Figure 5.



Figure 5 Apparatus for determination of empirical formula.

4. Obtain a fresh piece of magnesium ribbon weighing 0.6-0.8 g. The magnesium used should be shiny and bright. If the magnesium has an oxide coating on it, remove the coating with steel wool.

Roll the magnesium ribbon into a ball and place it in the crucible. Place the crucible cover on the crucible. Weigh the covered crucible and the ribbon to the nearest 0.0001 g on the balance previously used. Record this mass on the Data Sheet (1)

5. Place the covered crucible on the triangle as shown in Figure 5. Remove the crucible cover with crucible tongs and place the cover on a piece of wire gauze on the laboratory bench.

Note: White smoke will appear while heating the crucible. This smoke is the oxide of magnesium. You will need to cover the crucible as soon as you see the white smoke. This action will prevent you from losing any of your product. The magnesium should be reacted completely when there are no signs of smoke and the contents of the crucible do not glow brightly.

6. Heat the crucible gently by moving the flame from the burner back and forth under the crucible. As soon as the white smoke begins to appear from the crucible, immediately remove the flame. Use your tongs to replace the cover on the crucible.

Alternate the process of heating and covering the crucible at the first sign of smoke. Continue this process until no further smoking is observed.



Figure 6 Position of crucible cover for 10 minute heating.

- 7. Replace the cover on the crucible. Allow about one quarter of the crucible to be open to the atmosphere as shown in Figure 6. Heat the covered crucible strongly for 10 minutes.
- Allow the crucible to cool for 5 minutes. Remove the crucible cover and place the cover on the wire gauze. Using a dropping pipet, add 10 drops of distilled water to the crucible. Be careful not to lose any of the white fluffy solid. Replace the cover on the crucible as shown in Figure 6.

Note: The crucible will next be heated to evaporate the excess water. Do not overheat the crucible. Excessive heating will cause splattering and loss of the oxide of magnesium from the crucible.

9. Gently heat the crucible by moving the flame back and forth under the crucible for about 3 minutes. Heat the crucible to red heat in a strong flame for 3 minutes. Remove the burner flame. Use tongs to transfer the covered crucible and contents to the wire gauze. Allow them to cool to room temperature, and then weigh the covered crucible and contents to the nearest 0.0001 g. Record the mass on the Data Sheet (4).

- 10. Use a spatula to scrape the oxide ash from the crucible to the waste container in the fume hood.
- 11. Wash the crucible and cover. Dry them thoroughly.

CALCULATIONS

1. Find the mass of magnesium used.

Subtract the mass of the crucible and cover (2) from the mass of the covered crucible and magnesium (1).

Record the mass of magnesium on the Data Sheet (3).

2. Find the mass of the oxide of magnesium.

Subtract the mass of the crucible and cover (2) from the mass of the covered crucible and magnesium oxide (4).

Record the mass of the magnesium oxide on the data sheet (5).

3. Calculate the mass of oxygen that combined with the magnesium.

Subtract the mass of magnesium (3) from the mass of the oxide of magnesium (5).

Record the mass of oxygen on the Data Sheet (6).

4. Find the number of moles of magnesium used.

Divide the mass of magnesium (3) by the molar mass of magnesium, 24.31 g mol⁻¹.

Record the number of moles of magnesium on the Data Sheet (7).

5. Find the number of moles of oxygen that combined with the magnesium.

Divide the mass of oxygen (6) by the molar mass of oxygen, 16.00 g mol^{-1} .

Record the number of moles of oxygen on the Data Sheet (8).

- 6. Find the ratio of the number of moles of magnesium to the smallest number of moles of each.Divide the number of moles of magnesium by the smallest number of moles of each.Record the ratio on the Data Sheet (9).
- 7. Find the ratio of the number of moles of oxygen to the smallest number of moles of each.Divide the number of moles of oxygen by the smallest number of moles of each.Record the ratio on the Data Sheet (10).
- Find the empirical formula for the oxide of magnesium.
 Record the formula on the Data Sheet (11)