Experiment 3

DETERMINATION OF THE MOLAR MASS OF A "NEW" METAL[1]

MATERIALS:

Large test tube, gas delivery tube, metal oxide, metal slug, calorimeter, 400 mL beaker, 100 mL graduated cylinder, thermometer.

DISCUSSION:

Metal oxides are binary compounds containing a metal in a +n oxidation state (The n values which are possible are one through eight) and oxygen in the -2 oxidation state. A general formula of a metal oxide is M_xO_y, in which the x can have values of 1, 2 or 3 and y can be 1, 2, 3 or 4. Examples of metal oxides are:

silver(I) oxide	Ag_2O
palladium(II) oxide	PdO
gold(III) oxide	Au_2O_3
platinum(IV) oxide	PtO_2
osmium(VIII) oxide	OsO_4

Many metal oxides decompose upon heating to give oxygen gas and the free metal. The discovery of oxygen gas is credited to both the English chemist Joseph Priestly and the Swedish chemist Karl Scheele. In 1774, Priestly obtained the gas by heating mercury(II) oxide using the sun's rays focused with a lens as the source of the heat. In 1777, Scheele also produced oxygen gas by heating various metal oxides, including mercury(II) oxide.

In Part A of this experiment, a known mass of a metal oxide will be reduced with methane gas to release the free metal, carbon dioxide gas and gaseous water, according to the (unbalanced) equation

$$\text{metal oxide (s)} + CH_4\,(g) \xrightarrow{\text{heat}} \text{metal (s)} + CO_2\,(g) + H_2O\,(g)$$

This reaction will cause a decrease in the mass of the sample by an amount corresponding to the original mass of oxygen present. Thus, knowing the molar mass of atomic oxygen (defined as 16.00 g/mol), the number of moles of oxygen atoms originally present in the metal oxide can be determined.

In Part B of this experiment, a sample of the same metal will be heated in boiling water and then transferred to a calorimeter containing 100 mL of room temperature water. As expected, the metal will lose heat[2] to the water until an equilibrium condition is established. By the Law of Conservation of Energy, the amount of heat lost by the metal plus the amount of heat gained by the water must equal zero.

$$q(\text{metal}) + q(\text{water}) = 0 \qquad \text{(eqn. 1)}$$

The quantity of heat absorbed or evolved by a substance is symbolized by q and has units of joules. It can be determined by using equation 2 where ρ is the specific heat, m is the mass of the sample and ΔT is the change in temperature for the sample. The specific heat (ρ) is defined as the quantity of heat required to raise the temperature of one gram of that substance by one degree Celsius. Therefore, in an equation:

$$q = \rho m(\Delta T) \qquad \text{(eqn. 2)}$$

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[1] Koubek, E. and Prestia, J., *Journal of College Science Teaching*, 1979, *9*, 93.

[2] Heat is defined as the energy that flows into or out of a system because of a difference in temperature between the system and its surroundings.

Water has a specific heat (ρ) of 4.184 J/g°C. Metals such as Fe, Ag and Al have specific heats below 1.000 J/g°C. Using the changes in temperature of the metal and the water, their respective masses and the specific heat of water, one can determine the specific heat of the metal in the slug.

In 1819, the French chemists Pierre L. Dulong and Alexis T. Petit noted that the specific heat of a solid was inversely proportional to the molar mass of the solid. They quantified their observations in a law now known as the Law of Dulong and Petit.[3]

$$\text{specific heat } (\rho) \times \text{molar mass} \approx 24.9 \text{ J/mole}^\circ\text{C}$$

By applying this relationship to the system under study, one can estimate the molar mass of the metal and in turn, use this to estimate the number of moles of metal atoms present in the metal oxide sample. A ratio of the number of moles of metal atoms to the number of moles of oxygen atoms will yield the empirical formula of the metal oxide. The exact molar mass of the metal in the oxide can ultimately be determined by comparing the number of grams of metal remaining (after the metal oxide has been reduced) to the metal/oxygen molar ratio in the empirical formula. The overall purpose of this experiment is to illustrate how the molar mass of a metal can be determined experimentally.

PROCEDURE: (work individually)

A. Determination of the Moles of Oxygen Present in the Oxide

Determine the mass of a clean, dry test tube on the analytical balance. Place about 1.5 g of the metal oxide into the bottom of the tube and re-weigh it on the analytical balance. Assemble the apparatus as shown in Figure 1. Before you turn on the gas, have your instructor check out your set-up. The instructor will also ignite the gas leaving the delivery tube. (Safety note: The gas leaving the delivery tube must be flared off to prevent the dangerous build up of CH_4 and CO in the laboratory.) Now heat the metal oxide with a hot flame for at least 10 minutes. If any condensed water vapor collects on the test tube, heat the length of the tube to drive the water off. Be careful not to heat the clamp too strongly as it might melt. After 10 minutes, shut the Bunsen burner off but allow the gas from the delivery tube to flow and burn until the test tube has cooled. When the test tube has cooled to room temperature re-weigh it on the same analytical balance. The mass loss corresponds to the loss of oxygen from the oxide.

Save your test tube and metal residue. If time permits, reheat the residue to assure all the oxide was reduced. If reduction was complete, the mass of the residue should remain constant.

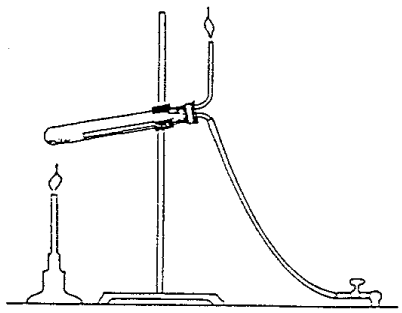

Figure 1

[3] For a discussion of how DuLong and Petit arrived at their law see: *Journal of Chemical Education*, **1947**, *3*, 143. In 1905 Einstein was able to explain why such a law existed. His explanation was based upon the quantized nature of atoms and it greatly aided in the acceptance of the quantum theory proposed by Max Planck in 1900.

B. Estimation of the Molar Mass of the Metal Using the Law of Dulong and Petit

Obtain a slug of the "unknown" metal and determine its mass on a top-loading balance. Place the weighed slug in a 400 mL beaker half-filled with water and heat to a boil.[4] Make sure the small wire handle remains available to remove the slug at a later time. While the slug and water are being heated to 100°C, place 100 mL (i.e., 100 g since the density of H_2O is very close to 1.000 g/mL) of water in the calorimeter cup provided and measure its temperature to the proper number of significant figures. When the water comes to a boil allow it to boil for 2 minutes and then quickly remove the slug from the boiling water and place it in the 100 mL of water in the calorimeter. Swirl the water gently and record the highest temperature reached.

Return the Metal slug to the slug bed and dispose of the test tube metal product in the waste jar. Dispose of the test tube properly.

[4] Do NOT attempt to measure the temperature of the boiling water with the thermometer. When the water is boiling, it may safely be assumed that the slug of the unknown metal is at 100°C.

Name _____ Section _____

Partner _____ Date _____

DATA SECTION
Experiment 3

Part A: Determination of the Moles of Oxygen Present in the Oxide

	First Heating	Second Heating
Mass of test tube + metal oxide before heating	_____	_____
Mass of test tube + metal after heating	_____	_____
Mass of test tube	_____	_____
Mass of metal oxide	_____	_____
Mass of metal	_____	_____
Mass of oxygen atoms lost	_____	_____
Moles of oxygen atoms	_____	_____

Part B: Estimation of the Molar Mass of the Metal Using the Law of Dulong and Petit

Mass of slug _____

Final temperature of 100 mL water, T_f _____

Initial temperature of 100 mL water, T_i _____

ΔT water (T_f - T_i) _____

ΔT metal (T_f - 100°C) _____

Mass of water in calorimeter _____

Specific heat (ρ) of water _____

DATA TREATMENT
Experiment 3

Be sure to include units!

1. Calculate the specific heat (ρ) of the metal.

2. Using the Law of Dulong and Petit determine the approximate molar mass of the metal.

3. Using the value of the approximate molar mass of the metal from step 2 and the mass of the metal from the data section of Part A, determine the approximate number of moles of metal atoms in the sample of metal oxide.

4. Calculate the ratio of the approximate number of moles of metal atoms to the number of moles of oxygen atoms, (determined in Part A). Knowing that the actual (or exact) ratio of moles of metal atoms to moles of oxygen atoms must be a ratio of small integers, deduce the empirical formula of the metal oxide.

5. From the actual number of moles of oxygen atoms (from part A) and the metal to oxygen ratio found above, determine the actual number of moles of metal present in the metal oxide. Then, use this number of moles of metal to calculate the molar mass of the metal. (Remember to use the mass of metal in Part A of the experiment) . Using the molar mass that was determined, identify the "unknown metal".

Questions:

1. A given midshipman weighs her or his test tube after a second heating and notices the mass differs from the mass obtained in the first weighing. What is the proper course of action? Why?

2. Estimate the amount of energy required to increase the temperature of a 10.5 g piece of calcium metal from 25.0°C to 80.0°C. Use the Law of Dulong and Petit to obtain an approximate specific heat of calcium.

3. A student ran two experiments in an effort to determine the molar mass of a new metal. The metal is hard, brittle and silvery white. It is commonly found as its oxide, known as pyrolusite. In the first experiment, the student heated a sample of the metal oxide to release the oxygen from the free metal. The data obtained were:

Mass of metal oxide before heating:	3.6077 g
Mass of metal after heating:	2.2798 g

In the second experiment, the student heated a sample of the metal in boiling water and then transferred it to a calorimeter containing 100.0 mL of water. The data obtained were:

Mass of metal sample:	45.73 g
Final temperature of water:	28.65 °C
Initial temperature of water:	25.00 °C

a. Using the data and results of the two experiments, determine the identity of the metal and its exact molar mass. (specific heat of water = 4.184 J/g°C)

b. Give the empirical formula and correct chemical name of the metal oxide.