Experiment 4

FV 8/18/03

STOICHIOMETRY OF THE DECOMPOSITION OF POTASSIUM CHLORATE

MATERIALS: Two test tubes, clamp, ring stand, weighing boat, glass wool, MnO_2, pure $KClO_3$, unknown mixture containing $KClO_3$ and an inert substance.

PURPOSE: The purpose of this experiment is two-fold: 1) to determine the stoichiometry of the decomposition of potassium chlorate; 2) to determine the percent by mass of potassium chlorate in an unknown mixture.

LEARNING OBJECTIVES: By the end of this experiment, the student should be able to demonstrate the following proficiencies:

1. Explain the relationship between the mass of a substance and the number of moles of a substance.
2. Apply stoichiometric ratios between the moles of reactant(s) and product(s) in balanced chemical reactions.
3. Calculate the percent by mass of a compound in a mixture.
4. Explain the purpose of a catalyst.

DISCUSSION:

Stoichiometry is the study of the quantities of substances involved in chemical reactions. The stoichiometric ratio refers to the ratio of the number of moles of reactants and the number of moles of products in a balanced chemical equation.[1] Stoichiometry is very important in the study of chemical reactions and their applications.

On submarines, a decomposition reaction is responsible for generating oxygen in an emergency. The "chlorate candle" consists of a mixture of sodium chlorate, iron, a small amount of barium peroxide, and a fibrous binding material.[2] The basic process in burning the "candle" involves the thermal decomposition of sodium chlorate:

$$NaClO_3\ (s) + Fe\ (s) \rightarrow NaCl\ (s) + O_2\ (g) + (Fe_xO_y)\ (s)$$

The iron combines with some of the oxygen and produces enough heat to sustain the reaction. The (Fe_xO_y) represents several different iron oxide products. Barium peroxide (BaO_2) is added to remove undesirable chlorine products including free chlorine and hypochlorous acid.

$$BaO_2\ (s) + Cl_2\ (g) \rightarrow BaCl_2\ (s) + O_2\ (g)$$

$$2\ BaO_2\ (s) + 4\ HOCl\ (aq) \rightarrow 2\ BaCl_2\ (s) + 3\ O_2\ (g) + 2\ H_2O\ (l)$$

Each candle burns near 400°F for 45-60 minutes, and produces approximately 115 SCF (standard cubic feet) of O_2 at 0.5 psig (pounds per square inch, gauge pressure) which is enough oxygen for about 100 people. The smoke and salt also produced must be separated by filtration. The stored candles represent a significant fire hazard since they are self-sustaining in oxygen.[3]

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[1]See "Chemistry," Brown, LeMay, and Bursten, section 3.1, pp 68-70.

[2]See "Applications of Chemistry," at http://www.chemistry.usna.edu/navapps/.

[3]For more information on the debate concerning the safety of oxygen candles, see http://www.cnn.com/US/9605/15/oxygen.generators/.

In Part A of this experiment, the stoichiometry of the decomposition of potassium chlorate will be determined. In Part B, this stoichiometry will be used to analyze a mixture containing potassium chlorate and an inert material. This allows the percent by mass of potassium chlorate in the mixture to be determined.

Potassium chlorate ($KClO_3$) decomposes when heated to at least 400°C, releasing oxygen gas:

$$\text{solid potassium chlorate} \longrightarrow \text{oxygen gas} + \text{solid residue}$$

Since it is not always possible to achieve and maintain the necessary temperature for rapid decomposition of potassium chlorate with a Bunsen burner, a catalyst will be added to this reaction. A catalyst is a substance that increases the rate of a chemical reaction without being consumed by the reaction. In this experiment, manganese(IV) oxide, MnO_2, will be used as a catalyst. Manganese(IV) oxide causes the decomposition rate of potassium chlorate to increase but does not itself generate any oxygen gas. Therefore, the mass lost from the test tube during the reaction is due only to the loss of oxygen gas from the decomposition of potassium chlorate.[4]

[4]Experiments using MnO_2 enriched with ^{18}O show that MnO_2 does indeed enter into the reaction because some ^{16}O-^{18}O oxygen is produced. However, since the amount of MnO_2 at the beginning of the reaction and at the end remain the same, it is a catalyst and not a reactant. See *Journal of Chemical Education*, **1962**, *39*, 573.

PROCEDURE:

Note: Transferring chemicals from a reagent bottle to a container should never be done in the balance room. The analytical balance is a sensitive instrument designed for determining the mass of small amounts of material with high precision. These sophisticated balances operate electronically with built-in microprocessors and care must be taken to avoid chemical spills. Be sure to transfer chemicals at your station in the lab.

Part A. Determination of the Stoichiometry of the Decomposition Reaction of Potassium Chlorate

1. Add a small amount of manganese(IV) oxide (about half as large as a pea) to a clean, dry test tube and determine the mass of the tube and manganese(IV) oxide on an *analytical balance*. Record this mass, using the proper number of significant figures and units, in the Data Section.

2. Use the *top-loading balance* to pre-weigh about 3 g of pure potassium chlorate into the weighing boat provided. If any clumps of potassium chlorate are present, break them up with a spatula, and then add this potassium chlorate to the test tube. **Be careful not to introduce any foreign material into the bottles of potassium chlorate since explosive mixtures could be produced.**

3. Determine the mass of the test tube and its contents on the same *analytical balance* used initially (in step A.1) and record the mass in the Data Section.

4. Carefully tap the test tube against your hand to mix the contents to obtain a uniform mixture. Tapping the test tube should also knock down to the bottom any materials which may be adhering to the upper inside walls.

5. Place a <u>*loose*</u> plug of glass wool in the mouth of the test tube. This will allow oxygen gas to escape but prevent any solid from spilling out of the tube. Determine the mass of the tube and its contents on the same analytical balance used previously and record the mass in the Data Section.

6. Clamp the test tube to a ring stand as shown in Figure 1. Be sure that the clamp does not have plastic sleeves as these will burn during the experiment. Place the clamp near the open end of the test tube so that the clamp will not melt while the test tube is being heated. Be sure the open end of the test tube is not pointed toward anyone or toward the lab aisles.

7. Heat the tube gently at first since oxygen is driven off quickly as the decomposition of the potassium chlorate begins. Move the burner around to achieve uniform heating. Increase the rate of heating as the rate of gas evolution decreases, finally heating as strongly as possible for an additional three or four minutes.

Answer Question #1 on page E4-9.

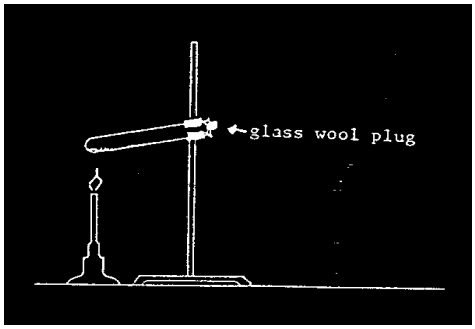

Figure 1 - Apparatus for Decomposition of Potassium Chlorate

8. Allow the tube to cool to room temperature and determine the mass of the tube and its total contents, including the residue and the glass wool, on the same analytical balance used previously. Record the mass in the Data Section.

✍ Answer Question #2 on page E4-9.

✍ Answer Question #3 on page E4-9.

9. If time permits, heat the same sample a second time and record its mass when cooled.

Part B. Determination of the Percent by Mass of Potassium Chlorate in an Unknown Mixture

1. From your instructor, obtain an unknown sample containing a mixture of potassium chlorate and an inert substance. **Record the unknown number in the space provided in the Data Section**.

2. Repeat the procedure outlined in Part A, using approximately 3 g of your unknown in place of the pure potassium chlorate. Record all masses, using the proper number of significant figures and units, in the Data Section.

Clean Up:

1. Place all used test tubes, including their contents, in the designated solid waste container in the laboratory.

2. Return the unknown tube and cork, without washing, to the instructor.

3. As directed by your instructor, complete through page E4-6 before leaving lab.

Name _____ Section_____

Partner _____ Date_____

DATA SECTION
Experiment 4

Part A. Determination of the Stoichiometry of the Decomposition Reaction of Potassium Chlorate

All masses should be from the analytical balance.

Mass of tube + MnO_2 _____

Mass of tube + MnO_2 + $KClO_3$ _____

Mass of $KClO_3$ _____

Mass of tube and total contents (including glass wool)

Before heating _____

After heating _____ _____*

Mass of oxygen gas evolved _____ _____*

Part B. Determination of the Percent by Mass of Potassium Chlorate in an Unknown Mixture

Unknown number _____

Mass of tube + MnO_2 _____

Mass of tube + MnO_2 + unknown _____

Mass of unknown _____

Mass of tube and total contents (including glass wool)

Before heating _____

After heating _____ _____*

Mass of oxygen gas evolved _____ _____*

* If time permits, heat the same sample a second time.

DATA TREATMENT
Experiment 4

Part A. Determination of the Stoichiometry of the Decomposition Reaction of Potassium Chlorate

In this section the balanced chemical equation for the decomposition of potassium chlorate will be determined. From the mass information recorded in the Data Section, calculate the number of moles of potassium chlorate present in the test tube before heating and the moles of oxygen gas generated. After these two calculations, determine the stoichiometric ratio between potassium chlorate and oxygen gas and generate a balanced chemical equation. This equation should also include the chemical formula for the solid material left in the test tube after heating. Show all work.

(A.1) From the mass of potassium chlorate used, calculate the number of moles of potassium chlorate consumed.

_____

(A.2) From your data, calculate the number of moles of oxygen gas (O_2) evolved.

_____

(A.3) Compare the moles of oxygen gas produced to the moles of potassium chlorate consumed and write a balanced chemical equation for the decomposition of potassium chlorate. Use whole number coefficients in the equation. Show how you determined the coefficients.

coefficient ↓ What is the residue that remains after heating? ↓

_____ $KClO_3$ (s) → _____ O_2 (g) + _____ _____ (s)

Have your instructor check this equation before proceeding to the calculations in Part B. _____
Instructor's initials

Part B. Determination of the Percent by Mass of Potassium Chlorate in an Unknown Mixture

Having determined the balanced chemical equation for the decomposition of potassium chlorate (from page E4-6), it is now possible to determine the percent by mass of potassium chlorate in an unknown mixture containing $KClO_3$ and an inert ingredient. As before, heating the sample will cause the decomposition to occur and oxygen gas will be produced. By calculating the number of moles of oxygen produced, the moles and mass of potassium chlorate originally present in the unknown sample can be determined.

(B.1) From your data, calculate the percent by mass of potassium chlorate in the unknown sample. Show your work.

Unknown number: _____ Percent by Mass of $KClO_3$ in unknown: _____

(B.2) Optional: To check your results, enter them into the "Data Input and Feedback" section of MOLS at http://mols.mathsci.usna.edu/login.html.

QUESTIONS
Experiment 4

1. A midshipman unknowingly spills a small amount of the manganese(IV) oxide and potassium chlorate mixture while inserting the glass wool in Part A of the experiment. Because of this error, will the number of moles of oxygen determined be too high or too low? Explain your answer.

2. If experimental errors cause the number of moles of oxygen determined in calculation (A.2) to be about 3% low, will an incorrect chemical equation for the decomposition reaction be obtained? Explain your answer.

3. If you burned a 100.0 g potassium chlorate "candle", how many grams of O_2 gas would have been released? Assume the "candle" is 100% $KClO_3$ and that the reaction reaches completion.

Name _____ Section _____

Date _____

IN-LAB QUESTIONS
Experiment 4

(Turn in this page <u>before</u> you leave lab.)

1. a. Record your observations as the mixture is heated.

   b. How do you know when the decomposition of potassium chlorate is near completion?

2. a. What is the purpose of MnO_2 in this experiment?

   b. Why doesn't MnO_2 affect the stoichiometry results in this experiment?

3. Why do you think it would be advisable to heat the sample a second time?

CHEMISTRY LABORATORY SAFETY AGREEMENT

Before working in the chemistry laboratory, read carefully the safety precautions and techniques for handling chemicals described in http://chem.mathsci.usna.edu/manual/safety.pdf. Give this agreement, signed and dated, to your laboratory instructor on the first day of lab.

When you are in the laboratory, THINK about what you are doing at all times.

1. Wear approved chemical splash goggles in the laboratory.
2. Do not attempt any unauthorized experiments.
3. Know the location and operation of safety equipment.
4. Bring only necessary materials into lab. Book bags, jackets, etc., are to be left in the hall.
5. Never work alone in the laboratory.
6. Never eat or drink in the laboratory
7. Use the fume hood when necessary.
8. Keep your work area uncluttered.
9. Use only equipment that is in good condition.
10. Dispose of waste and excess materials according to your laboratory instructor's directions.
11. No horseplay in the laboratory
12. Don’t sit or lean on laboratory work surfaces.
13. Handle chemicals with caution.
    (a) Read labels carefully.
    (b) Use only the amount required.
    (c) Leave reagent containers in their proper places.
    (d) Clean up all spills immediately.
    (e) Label all chemical containers.
14. Thoroughly wash your hands any time you leave the laboratory.
15. Immediately report all accidents and physical/chemical injuries, no matter how minor, to your laboratory instructor. Be ready to take immediate action as needed to assist any injured classmate.
16. Don’t leave the laboratory without your instructor’s approval.

I have carefully read all the safety precautions on the pages at the website above and recognize that it is my responsibility to observe them throughout my chemistry course.

Name _____ Signature _____

Course _____ Section _____

Instructor _____ Date _____