# Experiment 4A

FV 8/4/04

# THE DECOMPOSITION OF POTASSIUM CHLORATE

**MATERIALS:** test tubes, one-hole rubber stopper fitted with glass tube, glass wool, rubber tubing, gas collection bottle, wooden splints, potassium chlorate, manganese (IV) oxide

**PURPOSE:** The purpose of this experiment is to study the decomposition of potassium chlorate, both by verifying the identify of one of the products and by quantitatively determining the correct stoichiometry.

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

1. Find the MSDS and/or Safety Card for a chemical species, and locate important information related to physical properties, reactivity, and appropriate handling protocols.
2. Propose and perform simple tests to verify gas production in general, and oxygen gas in particular.
3. Propose and perform a simple procedure for assuring that a reaction is complete.
4. After carrying out the decomposition reaction for potassium chlorate, quantitatively verify its stoichiometry.
5. Perform appropriate error analysis on the stoichiometric measurements obtained, including accounting for any systematic error observed.

**DISCUSSION:**

*Material Safety Data Sheets and International Chemical Safety Cards.* Any institution where chemicals are used is required to have copies of the material safety data sheets (MSDS) available for use. These sheets provide key information relating to health hazards, appropriate storage, handling and disposal arrangements, fire and explosive hazards, required control measures, physical/chemical properties, and reactivity data. In this experiment, the MSDS for potassium chlorate will be used to help guide the experimental study of its decomposition reactions. In general, prior to any chemical procedure, the relevant MSDS should be consulted to assure safe and proper procedures are followed. Another system which provides similar information is the International Chemical Safety Card system. Both MSDS and Safety Cards are available on-line through links found on the Plebe Chemistry homepage.

*Relevant Naval Application.* On submarines, oxygen for breathing is normally produced through electrolysis of water. Details relating to this process will be studied later in the course. In an emergency, a chemical process is used to produce oxygen gas for breathing, i.e., the decomposition of sodium chlorate at high temperature (i.e., above 300$^{o}$C), producing oxygen gas and sodium chloride. Unfortunately, there are several complications associated with this reaction which must be remedied if the production of oxygen gas for breathing is to be performed safely and efficiently.

First, though the decomposition reaction occurs at temperatures above 300$^{o}$C, it is extremely slow and therefore impractical for oxygen production. This is remedied by adding a *catalyst*, in this case manganese (IV) oxide, which significantly increases the rate of the reaction, without itself being consumed.

Second, the intense flame used to raise the temperature of the sodium chlorate above 300$^{o}$C is produced by a combustion reaction, which *consumes* large quantities of oxygen gas, whereas the

purpose of the overall process is to *produce* oxygen gas. While this issue cannot be completely remedied, small amounts of iron metal are mixed in, reacting with some of the oxygen to produce iron oxide and releasing large quantities of energy which helps maintain the mixture above the 300$^{o}$C decomposition temperature. The oxygen-consuming flame used to initialize the decomposition reaction is replaced by this iron combustion process.

Third, while the desired decomposition reaction predominates, there is another decomposition reaction which produces toxic chlorine gas, oxygen gas and sodium oxide. This is remedied by including small amounts of barium peroxide in the mixture, which reacts with the toxic chlorine gas to produce barium chloride and oxygen gas.

In summary, the "chlorate candle" used for emergency production of oxygen gas for breathing on submarines consists of a mixture of sodium chlorate, iron, a small amount of barium peroxide, and a fibrous binding material. In practice, each candle burns near 400$^{o}$C for 45-60 minutes, and produces approximately 115 SCF (standard cubic feet) of oxygen gas at 0.5 psig (pounds per square inch, gauge pressure), which is enough oxygen for about 100 people. The stored candles represent a significant fire hazard since they are self-sustaining in oxygen.

*Use of potassium chlorate.* In this experiment, potassium chlorate will be used instead of sodium chlorate. Analogous reactions occur with all of the same complications. The only remedy that will be applied here will be the inclusion of the manganese (IV) oxide catalyst. Since all of the procedures will be carried out in the fume hood, any toxic chlorine gas produced will be safely carried away in the ventilation system.

**PROCEDURE:**

1. Observe the correct usage of the Bunsen burner, as demonstrated by your instructor. Make any special notes for future reference.

2. Find the MSDS for potassium chlorate. Determine if there are any health hazards and what conditions produce these hazards. Determine what to do in case of a spill and how to dispose of the chemical. Under what conditions does it produce fire or explosion hazards? What are the measures that should be taken to assure safe handling? Since this experiment involves high temperatures, what are the melting point and decomposition temperature? What are the known decomposition products (this is particularly relevant to this experiment)?

3. Using available equipment, devise and implement a physical test to verify that gases are not produced at any noticeable rate when about 1 g of pure potassium chlorate is heated. Repeat this test when a small amount of the catalyst manganese (IV) oxide is added to about 1 g potassium chlorate. Make sure the two solids are as uniformly mixed as possible before heating. There is no exact mass of catalyst required, but the uniform mixture should appear *light grey* after mixing. Is there any evidence that the secondary decomposition reaction is occurring?

4. Using available materials, with similar amounts of chemicals as used in #3 above, devise and implement a chemical test to verify that the main gas produced is oxygen gas. Your instructor will provide important safety information regarding this chemical test.

5. Devise and implement a procedure for quantitatively studying the decomposition reaction, using only a Bunsen burner, the test tube, mass balances (i.e., no gas collection), and a spreadsheet. How will your procedure assure that the reaction is complete? How will your procedure assure that the gases produced escape to the surrounding atmosphere, but that all

solid residues remain completely inside the test tube? Use no more than 3 g of potassium chlorate, along with an amount of catalyst yielding a light grey mixture.

6. From your data, and assuming that only the primary decomposition reaction producing potassium chloride and oxygen gas occurs, determine the mole ratio of oxygen gas to potassium chlorate. Is this ratio consistent with the stoichiometry of this decomposition pathway? Report your ratio to the instructor.

7. Using the class data, determine the average and standard deviation from the mean of these mole ratios. Do these results indicate that there is a systematic error inherent to the study of this decomposition reaction? See question 2 below for further analysis of this issue.

**QUESTIONS FOR CONSIDERATION:**

1. Assuming all of the analogous reactions occur, why would lithium chlorate be a more practical choice for the "chlorate candle" than either sodium or potassium chlorate?

2. Starting with 100.0 g of potassium chlorate, and assuming that 95% decomposes into potassium chloride/oxygen and 5% decomposes into potassium oxide/chlorine/oxygen, how many grams of residue would remain after complete reaction? If the investigator had assumed that only the potassium chloride/oxygen reaction was taking place, what would he find for a mole ratio of oxygen gas: potassium chorate? Does this match with what was found by most students in the lab? How does this calculation provide information for the error analysis of the class data?

3. In doing this laboratory, the energy needed to maintain the temperature above the decomposition temperature was provided from the Bunsen burner. Remember that the purpose of the "candle" reaction is to *produce* oxygen. Explain the factors that must be considered in determining the net change in the amount of oxygen in the context of how you actually performed the experiment.

4. In practice, rather than using a Bunsen burner to maintain the heat needed for this reaction, a small amount of iron metal is introduced into the mixture. Write the balanced reaction with oxygen, if iron(II) oxide is the product. Explain why this reaction is only useful if it produces large quantities of energy.

5. If the test tube had not been fully dried off upon weighing after the reaction was apparently complete, what would be the effect on the calculated mole ratio of oxygen : potassium chlorate?

6. When used for producing oxygen, the small percentage of decomposition which follows the second pathway produces hazardous chemicals. In practice, small amounts of barium peroxide are introduced to react with these products, as described in the Discussion section above. Balance this reaction.

7. What would have been the expected mass of residue if the potassium chlorate reaction produced potassium hypochlorite instead of potassium chloride, assuming no alternate decomposition pathways? Determine the oxygen to potassium chlorate mole ratio for this scenario. Does this possible reaction pathway appear to contribute at all, based on the experimental data?