EXPERIMENT 5:
PERCENTAGE OF OXYGEN IN $KClO_3$

Introduction: In this experiment you will determine the percentage of oxygen in potassium chlorate. You will calculate the theoretical value from the chemical formula and compare your experimental value to the theoretical value.

Background: When potassium chlorate ($KClO_3$) is heated, it undergoes chemical decomposition. Oxygen gas (O_2) is given off and potassium chloride (KCl) remains as the residue. The equation for the decomposition of potassium chlorate is:

$$\mathbf{2\ KClO_3\ (s) \longrightarrow 2\ KCl\ (s) + 3O_2\ (g)}$$

Other metal chlorates behave similarly when heated; forming oxygen gas and the corresponding metal chloride. The reaction may be speeded by adding some MnO_2 catalyst.

$$\text{Metal chlorate} \longrightarrow \text{Metal chloride + oxygen}$$

In this experiment a weighed quantity of potassium chlorate is heated. The oxygen formed in the reaction is driven off and the mass of the residue (KCl) is measured. These two quantities, the mass of the original sample of potassium chlorate and the mass of the KCl, allow us to determine the amount of oxygen in the original sample. If a catalyst is used, it will remain unaffected and its weight can be subtracted. The experimental percentage of oxygen in the sample of $KClO_3$ is calculated by using this equation.

$$\text{Experimental \% oxygen} = \frac{\text{Mass of oxygen lost}}{\text{Mass of } KClO_3} \times 100$$

The theoretical value of the % oxygen in potassium chlorate is calculated from the formula $KClO_3$ with a molar mass = 122.6 g/mol.

Frequently it is interesting – and instructive - to determine the percent error of an experimental determination. The theoretical percentage of oxygen is used as the accepted value when we calculate the percent error according to this formulation.

$$\text{Percentage Error} = \frac{|\text{experimental value} - \text{accepted value}|}{\text{accepted value}} \times 100$$

The numerator of this equation can be positive or negative – meaning the experimental value is high or low.

In this procedure, you will be asked to heat the crucible a second time to ensure that the decomposition is complete. That means that after the first heating and cooling and weighing, the process is repeated. If the sample lost more than 0.02 g between 1^{st} and 2^{nd} weighings that may mean that the sample lost some more oxygen. The process of heating and cooling and weighing must be repeated until there is negligible change in mass (±0.02 g) between consecutive heatings. This is called "heating to constant weight".

Materials Needed

Equipment	Chemicals
Iron ring, clay triangle, Crucible tongs crucible & cover or Pyrex test tube Cooling pad	Potassium Chlorate, $KClO_3$ Manganese dioxide (optional catalyst)

Procedure

In this experiment you may use either a crucible with cover or a Pyrex test tube. Before starting, ask your instructor as to which method you will be doing.

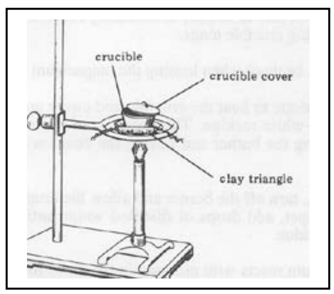
Crucible Method:

Weigh a clean dry crucible and cover. Optional, add 0.50 g MnO_2 catalyst. Next, obtain a sample of potassium chlorate, $KClO_3$, as directed by your instructor. Place about 1.5 g of $KClO_3$ in the weighed crucible, and reweigh. Remember to use the same balance for all weighings of the same sample. Mix the catalyst well if using the MnO_2.

Place the crucible with $KClO_3$ on a clay triangle and set the cover slightly ajar, see figure 1. Very gently heat the crucible containing the powders for about 8 minutes, then heat strongly for 10 minutes. Be sure the inner-blue cone of the flame is just below the crucible bottom while you are heating strongly, and that the crucible bottom and/or clay triangle are heated to redness. Allow the crucible to cool to room temperature, which takes at least 10 minutes, and then weigh the crucible and residue. Record your data in your lab notebook.

Heat strongly for a second time for about 5 minutes, then cool and weigh. If the weighings after the first and the second heatings are not the same, i.e. they differ by more than 0.02 g; repeat the process of heating and cooling and weighing as needed, until 2 successive weights agree (within +/- 0.02 g). Calculate the experimental percentage of oxygen in $KClO_3$ based on the final weight of container's contents.

Figure 1: Crucible set-up.

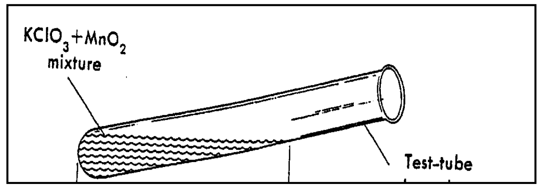


Pyrex Test tube Method:

Weigh a clean dry Pyrex test tube and beaker. Place 0.5 to 0.8 g of manganese dioxide, MnO_2, into a large-size (Pyrex) test tube. (Do not use a *small* test tube since they are more inclined to "shoot" their contents). Measure the mass of the entire container on an analytical or centigram balance. Record the mass in the space provided on the report sheet.

Add about 1.0 to *1.5 g* of potassium chlorate, $KClO_3$, to the test tube. Record the mass of the container and its contents. Mix the contents of the test tube until they have a somewhat uniform gray appearance. (Be careful not to lose any of the contents.) Carefully heat the test tube and its contents, see fig 2 below. Heat gently at first, increasing the intensity after the mixture seems to "boil," as it sometimes appears to do when bubbles of oxygen are being released. Continue heating for about five minutes, and then cool and weigh. Repeat the process in 5-minute heating cycles until constant mass is reached. Set the container and its contents aside while you complete your calculations. Do not discard the residue until your calculations are finished and satisfactory; if they are not satisfactory, it is possible that you may be able to salvage your work if the material is still on hand.

Figure 2: The proper angle for handling of the test tube while heating. Heat only in the indicated range.



Safety and Waste Disposal

Safety: Potassium chlorate is a strong oxidizing agent. It may cause fires or explosions if mixed or heated with combustible materials such as paper. **This experiment is Potentially hazardous, and if performed carelessly could lead to a serious accident!** The formation of a gas at the bottom of a test tube may result in a sudden expansion, blowing hot chemicals out of the test tube. This will not occur if the test tube is handled properly during heating. When heating a solid in a test tube, tip the tube until it is almost horizontal and tap it carefully until the contents are distributed over the lower half of the length of the tube, as shown in Figure 2. Holding it at about this angle, move the test tube back and forth in the flame of the burner, distributing the heat over the entire length of the mixture. **Do not concentrate the heat in any one area, particularly near the bottom of the test tube**.

Be very sure your test tube is not pointing toward anyone, including yourself, while it is being heated. Be aware of your surrounding while doing this experiment. Do not place yourself in front of somebody else's test tube. **Wearing goggles is absolutely.**

Waste Disposal: Excess or spilled potassium chlorate and the residue (KCl) should be placed in labeled waste containers in the hood.

Name__________________________

EXPERIMENT 5: REPORT
OXYGEN IN POTASSIUM CHLORATE Section _______

Mass of container ______________________ g

Mass of container and MnO_2 ______________________ g

Mass of the catalyst, MnO_2 ______________________ g

Mass of container + MnO_2 + $KClO_3$ ______________________ g

Mass of $KClO_3$ ______________________ g

Mass after 1st heating…….……______________________ g

Mass after 2nd heating…………______________________ g

Mass after 3rd heating………….______________________ g

Mass of container + KCl (same as mass of final heating) ______________________ g

Mass of KCl produced ______________________ g

Mass of oxygen released ______________________ g

Experimental Percent of oxygen ______________________%
(Show calculations below)

Theoretical Percent of oxygen ______________________%
(Show calculations below)

Experimental error ______________________%
(Show calculations below)

Questions and Problems

1. Write equations similar **to the decomposition of $KClO_3$**, for the thermal decompositions of two chlorate compounds to form oxygen and chloride salts

   a. $LiClO_3 \longrightarrow$

   b. $Al(ClO_3)_3 \longrightarrow$

2. Calculate the percentage of oxygen in $Al(ClO_3)_3$ from the chemical formula

3. A sample of an unknown metal chlorate, weighing 1.725 g, is heated until all of the oxygen is driven off. The residue remaining in the container weighs 0.859 g. Calculate the percentage of oxygen in this metal chlorate.

4. A student records the following data in a laboratory experiment to determine the percentage of oxygen in $Ca(ClO_3)_2$.

Mass of container		58.957 g
Mass of container and $Ca(ClO_3)_2$		60.734 g
Mass of container and contents after:		
	First heating	60.221 g
	Second heating	59.910 g
	Third heating	59.899 g

Calculate: Show setups in detail on a separate sheet of paper or notebook copy

a. The experimental percentage of oxygen in $Ca(ClO_3)_2$.

b. The theoretical percentage of oxygen in $Ca(ClO_3)_2$.

c. The percentage error.