Experiment 10

Analysis of a $KClO_3$ Mixture and Determination of "R"

Pre-Lab Assignment

Before coming to lab:

- Read the lab thoroughly.
- Answer the pre-lab questions that appear at the end of this lab exercise.

Purpose

The mass percent of potassium chlorate in a mixture will be determined by its thermal decomposition and production of oxygen gas, which will be measured via displacement of water. The Ideal Gas Constant (R) and the molar volume of an ideal gas at standard temperature and pressure (STP) will also be determined by measuring the conditions of the oxygen gas produced.

Background

When potassium chlorate ($KClO_3$) is heated, it thermally decomposes to produce solid potassium chloride and oxygen gas by the following balanced reaction in Eqn. 1.

$$2\ KClO_3(s) \rightarrow 2\ KCl(s) + 3\ O_2(g) \qquad \text{Eqn. 1}$$

Normally this reaction requires temperatures of 400°C, but a catalyst such as manganese dioxide (MnO_2) can be added to lower the temperature to a more achievable 250°C. Since the KCl and MnO_2 are solids, they will be left behind in the reaction container while $O_2(g)$ can be driven off to be collected and measured separately.

Since gases are hard to measure in a laboratory, the $O_2(g)$ will instead be measured by the displacement of water so that the volume of water collected is equal to the volume of $O_2(g)$ produced. However, since water is a liquid, the gas trapped inside the container will be a mixture of $O_2(g)$ and $H_2O(g)$, or water vapor, so that the total pressure will be the sum of both gases.

The Ideal Gas Law relates the pressure (P), volume (V), temperature (T), and moles (n) for any gas in terms of the Ideal Gas Constant, R, as seen in Eqn. 2:

$$R = \frac{PV}{nT} \qquad \text{Eqn. 2}$$

R has a standardized value of 0.08206 L*atm/mol*K. By measuring the P, V, T, and n of any one gas, R can be determined and should be close to the accepted value.

The molar volume for an ideal gas is defined as the L/mol that it occupies at 273.15 K and 1.00 atm, or standard temperature and pressure (STP). With rearranging, it can be expressed via the Ideal Gas Law as well as in Eqn. 3:

$$\frac{V}{n} = \frac{RT}{P} \qquad \text{Eqn. 3}$$

This also has a standardized value of 22.4 L/mol at STP.

For this experiment, an unknown mixture of $KClO_3$, KCl, and MnO_2 solids are heated to allow just $KClO_3$ to decompose and the O_2(g) made allowed to escape into a second vessel. The change in

Example Problem: Determination of the Mass Percent of $KClO_3$

A 1.565 g mixture of $KClO_3$, KCl, and MnO_2 is heated and the O_2(g) produced allowed to escape to displace water. At the end of the reaction, the remaining mixture weighed 1.323 g. Calculate the mass percent of $KClO_3$ present in the original mixture.

Step 1: Find the mass of O_2(g) produced

$$1.565 \text{ g before reaction} - 1.323 \text{ g after reaction} = 0.242 \text{ g } O_2 \text{ made}$$

Step 2: Find the mass $KClO_3$ in the original sample

$$0.242 \text{ g } O_2 \times \frac{1 \text{ mol } O_2}{32.00 \text{ g } O_2} \times \frac{2 \text{ mols } KClO_3}{3 \text{ mols } O_2} \times \frac{122.55 \text{ g } KClO_3}{1 \text{ mol } KClO_3} = 0.617 \text{ g } KClO_3$$

Step 3: Find the mass percent $KClO_3$ in the original sample

$$\frac{0.617 \text{ g } KClO_3}{1.565 \text{ g mixture}} \times 100 = 39.4 \text{ \% } KClO_3$$

mass before and after the reaction will correspond to the mass of O_2(g) produced since all other components are solids. The amount of O_2(g) produced will also be used to determine the mass percent of $KClO_3$ in the original sample.

The pressure, temperature, and volume of O_2(g) produced will also be measured. The pressure inside the container the sum of *both* gases, O_2(g) and H_2O(g) as seen in Eqn. 4. To find the pressure of O_2(g) alone, the tabulated vapor pressure of water at the gases' temperature must be subtracted from the total.

$$P_{total} = P_{O2} + P_{H2O} \qquad \text{Eqn. 4}$$

The total pressure of the container is assumed to be equal to the outer atmospheric pressure for the day once corrected for the expansion of mercury inside the barometer by using Eqn. 5.

$$P(\text{atmosphere}) - (1/8)(T \text{ of barometer, in °C}) = P(\text{corrected}) \qquad \text{Eqn. 5}$$

The temperature of O_2(g) can be measured directly with a thermometer and the volume of O_2(g) will be measured via the water it displaces. The value for R and the molar volume at STP can then be calculated and compared to standardized values.

Example Problem: Determination of R

The mixture from the example problem above ended the reaction with a temperature inside the gases' container of 31.0°C and a volume of water displaced of 145.0 mL. The barometer for the day read 713.5 mmHg and the temperature of the barometer was 21.6°C. Calculate R.

Step 1: Find the corrected atmospheric pressure

713.5 mmHg – (1/8)(21.6) = 710.8 mmHg

Step 2: Find the vapor pressure of water, P_{H2O}

From a table: at 31.0°C, the vapor pressure of water is 33.7 mmHg

Step 3: Find the pressure of O_2(g) alone, P_{O2}

710.8 mmHg – 33.7 mmHg = 677.1 mmHg

Step 4: Convert P_{O2} to atm

$$677.1\ \text{mmHg} \times \frac{1\ \text{atm}}{760\ \text{mmHg}} = 0.8909\ \text{atm}$$

Step 5: Convert volume to L

$$145.0\ \text{mL} \times \frac{1\ \text{L}}{1000\ \text{mL}} = 0.1450\ \text{L}$$

Step 6: Convert temperature to K

31.0 + 273.15 = 304.15 K

Step 7: Find mols O_2(g)

$$0.242\ \text{g}\ O_2 \times \frac{1\ \text{mol}\ O_2}{32.00\ \text{g}\ O_2} = 0.00756\ \text{mols}\ O_2$$

Step 8: Find R

$$R = \frac{(0.8909\ \text{atm})(0.1450\ \text{L})}{} = 0\ 0\ 0562\ \text{L*atm/mol K}$$

Example Problem: Determination of Molar Volume at STP

Using the data from the example problems above, calculate the molar volume of O_2(g) at STP.

Step 1: Find molar volume

$$\frac{V}{n} = \frac{RT}{P} = \frac{(0.0562\ \text{L*atm/mol K})(273.15\ \text{K})}{1.00\ \text{atm}} = 15.4\ \text{L/mol}$$

Procedure

1. Weigh a dry, empty ignition tube inside a beaker to prevent it from tipping. Record this weight in your data sheet.

2. Add approximately 1.0-1.5 g of unknown sample to the ignition tube in the beaker and weigh again. Record this weight in your data sheet.

Caution: do NOT let the $KClO_3$ contact the rubber stopper. $KClO_3$ is an oxidizer that will react violently with rubber!

Note: the sample should be at the ***bottom*** *of the ignition tube, not along the sides.*

3. Collect rubber tubing with attached stoppers, a 500 mL Florence flask, a 600 mL beaker, a tubing clamp, and a bar clamp. Assemble the apparatus as shown in Fig. 1.

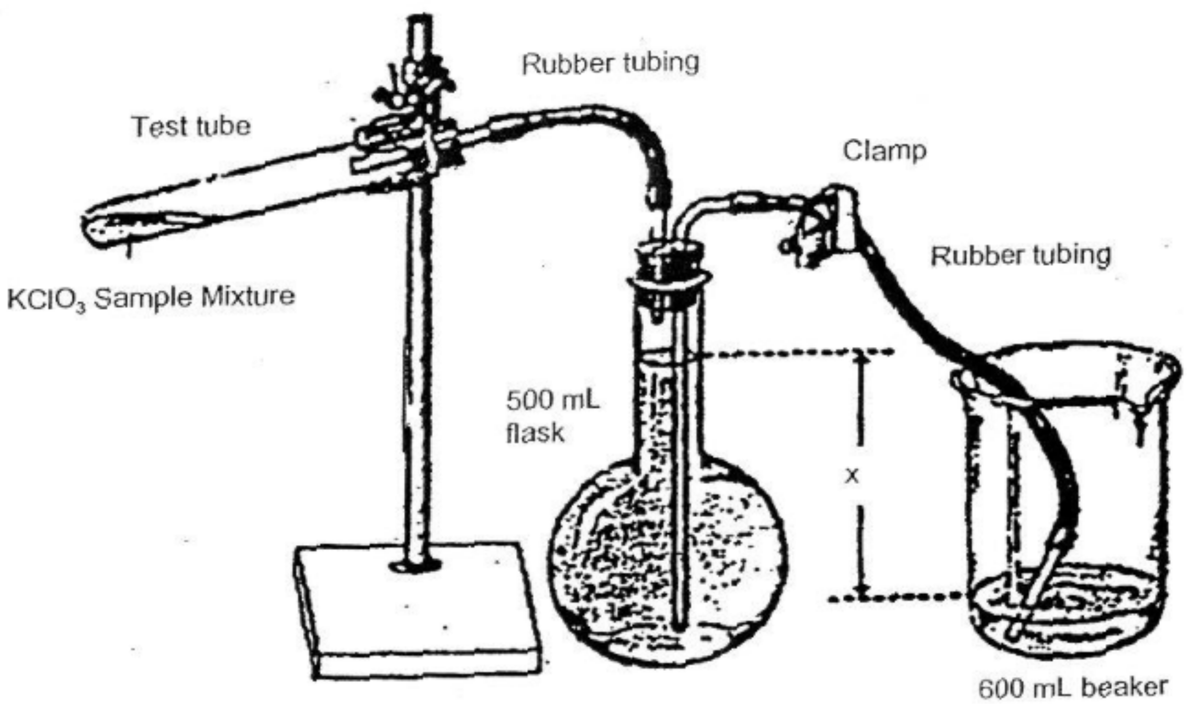

Figure 1: Gas Apparatus

4. Fill the Florence flask with tap water just underneath the shortest glass tubing. Add approximately 100 mL of tap water to the 600 mL beaker.

Note: the long glass tubing should be submerged while the short glass tubing should ***not*** *be touching the surface of the water.*

5. Unstopper the ignition tube. Blow through this end to force water out of the Florence flask, into the other tubing, and then to the beaker. Continue blowing until **all** air bubbles have been removed from the second tubing and water can be easily transferred between the beaker and the Florence flask by raising one or the other.

Note: do ***not*** *touch your lips to the equipment.*

6. Raise the beaker until the Florence flask is full again as in Step 4. Clamp the tubing. Stopper the ignition tube and dump out the water from the 600 mL beaker. Do **not** dry it.

7. Unclamp the tubing. A small amount of water should flow into the beaker and then stop.

Note: if the flow of water to the beaker does not stop, your apparatus has a leak. Tighten all stoppers and check all tubing and repeat Steps 3-7 as necessary.

8. Using a Bunsen burner directly under the solid sample in the ignition tube, **gently** begin heating. Some of the solids will begin to liquefy and produce white vapors. As the latter subsides, begin increasing the heat to ensure all the $KClO_3$ has fully reacted. The volume of water in the 600 mL should steadily increase so long as $O_2(g)$ is still being produced.

9. Continue heating until no further vapors or change in volume of water in the beaker is observed and then heat for five additional minutes. The mixture in the ignition tube should be entirely white solid; no purple color or molten appearance should remain. Turn off the Bunsen burner.

Note: do not melt the rubber clamp holding the ignition tube.

10. Allow the apparatus to fully cool to room temperature while **still stoppered**.

11. Clamp the tubing between the Florence flask and 600 mL beaker. Now both stoppers can be removed and the apparatus disassembled.

12. Use a thermometer to measure the temperature (T_{O2}) of the gases inside the Florence flask.

13. Use a graduated cylinder to measure the volume of water (V_{O2}) inside the 600 mL beaker.

Note: you may have to use the cylinder repeatedly by emptying it between portions.

14. Weigh the ignition tube and same beaker used in Step 1. Record this weight in your data sheet.

15. Record the atmospheric pressure (P_{atm}) and the temperature of the barometer ($T_{barometer}$) for the day from the classroom barometer.

16. The residue inside the ignition tube can be loosened with water and disposed of in the labeled waste container.

17. Repeat Steps 1-16 for a second trial.

18. Calculate the mass percent $KClO_3$, R, and the molar volume at STP for each trial. These values should agree with each other as well as the standardized values for R and molar volume with reasonable percent error.

Experiment 10—Data Sheet

Name: ____

	Trial One	**Trial Two**
1. Mass of ignition tube and beaker (g)	____________	____________
2. Mass of ignition tube, beaker, and sample *before* reaction (g)	____________	____________
3. Temperature of O_2(g) (T_{O2}, °C)	____________	____________
4. Volume of O_2(g) (V_{O2}, mL)	____________	____________
5. Mass of ignition tube, beaker, and sample *after* reaction (g)	____________	____________
6. Atmospheric Pressure (P_{atm}, mmHg)	____________	____________
7. Temperature of Barometer ($T_{barometer}$, °C)	____________	____________
8. Mass of sample (g)	____________	____________

show calculation:

9. Mass of O_2(g) produced (g) ____________ ____________

show calculation:

10. Mass of $KClO_3$ in sample (g) ____________ ____________

show calculation:

11. Mass Percent of $KClO_3$ in sample (5) ____________ ____________

show calculation:

	Trial One	Trial Two
12. Moles of O_2(g) produced (mols)	____________	____________

show calculation:

13. Temperature of O_2(g) (T_{O2}, K)	____________	____________

show calculation:

14. Volume of O_2(g) (V_{O2}, L)	____________	____________

show calculation:

15. Corrected Atmospheric Pressure ($P_{corrected}$, mmHg)	____________	____________

show calculation:

16. Vapor Pressure of H_2O (P_{H2O}, mmHg)	____________	____________
17. Pressure of O_2(g) (P_{O2}, mmHg)	____________	____________

show calculation:

	Trial One	Trial Two
18. Pressure of O_2(g) (P_{O2}, atm) *show calculation:*	____________	____________

19. Gas Constant, R (L atm/mol K) *show calculation:*	____________	____________

20. Molar Volume at STP (L/mol) *show calculation:*	____________	____________

21. Percent Error for R *show calculation:*	____________	____________

Experiment 10—Post-Lab Assignment

1. Based on your calculated values for R and percent error, are your results accurate, precise, both, or neither? Explain.

2. Give three possible sources of experimental error.

3. A student failed to completely decompose all of the $KClO_3(s)$ present in their sample, leaving some unreacted. What effect (too high, too low, no effect) will this error have in the following calculated values? For each, explain your reasoning clearly.

a. Mass Percent $KClO_3$

b. Gas Constant, R

c. Molar Volume at STP

4. A student failed to correct the atmospheric pressure for the expansion of mercury, reporting the uncorrected pressure as the total pressure for their calculations. What effect (too high, too low, no effect) will this error have in the following calculated values? For each, explain your reasoning clearly.

a. Mass Percent $KClO_3$

b. Gas Constant, R

c. Molar Volume at STP

5. What mass of $KClO_3$ (in kg) must be decomposed to supply 8 people with enough oxygen for 24 hours on a small submarine? According to NASA, the average person needs about 0.84 kg of $O_2(g)$ per day.

$$2\ KClO_3(s) \rightarrow 2\ KCl(s) + 3\ O_2(g)$$

Experiment 10—Pre-Lab Assignment

Name: ______________________________________

For all calculations, show all work and draw a box around the final answers.

1. Write the balanced equation for the thermal decomposition of potassium chlorate.

2. Why is it important that you NOT heat the potassium chlorate in contact with the rubber stopper?

3. What data items are used in this experiment to determine the following calculated values:

   a. Mass Percent of $KClO_3$ ______________________________
   b. Gas Constant, R ______________________________
   c. Molar Volume ______________________________

4. A student performing the experiment obtained the following data:

Mass Test Tube (g)	12.1456 g
Mass Tube + Sample before heating (g)	13.3278 g
Mass Tube + Sample after heating (g)	12.9987 g
Volume of H_2O(l) collected (mL)	248 mL
Temperature of Gas (°C)	21.0°C
Atmospheric Pressure (mmHg)	742.3 mmHg
Temperature of Barometer (°C)	19.5°C

From this data, find:

   a. Mass Percent $KClO_3$ in sample

b. Gas Constant, R

Molar Volume at STP