Experiment 8- Analysis of $KClO_3$ Mixture and Determination of Gas Constant "R"

Purpose of the Experiment
(1) You will determine the value of the gas constant R, and
(2) The percentage of potassium chlorate in a mixture

Background Information

You will be given a sample containing an unknown amount of $KClO_3$, KCl, and MnO_2 Heating the $KClO_3$ decomposes the compound to form KCl and oxygen gas:

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

Without a catalyst, the temperature must be about 400 °C, but with manganese dioxide as a catalyst the reaction goes at about 250 °C. MnO_2 is included in the unknown so that it is not necessary to add more. The loss in weight of the sample mixture is the weight of oxygen driven off, from which the number of moles can be calculated.

The oxygen is collected by displacement of water, so that the volume of water displaced is the volume of oxygen. The oxygen contains water vapor, so the total pressure of gas is the sum of the partial pressure of oxygen and the vapor pressure of water. It is therefore necessary to subtract the vapor pressure of water form the total pressure to obtain the pressure of the oxygen gas.

Knowing the temperature, pressure, volume, and number of moles, you can use the ideal gas law equation to calculate the gas constant R.

$$R = \frac{PV}{nT} \qquad \text{(Eq.2)}$$

From the mass of oxygen and the stoichiometry of the balanced equation you can calculate the mass of $KClO_3$ in the sample.

Procedure

Caution: Wear your safety goggles while you or anyone in class is doing this experiment.

Chemical Alert
Potassium chlorate – Strong oxidizer

(1) Weigh a dry empty test tube on the analytical balance to the nearest 0.0001 g.

(2) From your instructor, obtain an unknown mixture containing $KClO_3$, KCl, and MnO_2. Place 1.0 to 1.5 grams of the sample into the test tube and weigh. Record the weight of test tube and contents on line 2 to the nearest 0.0001 g. By subtracting the weight of the empty test tube, you obtain the weight of sample, which should not be more than 1.5 grams. CAUTION: Be sure that NONE of the sample is in contact with the rubber stopper; the sample should be placed in the bottom of the test tube, not on the upper walls.

Assemble the apparatus as shown in the illustration (Fig. 1). One glass tube should extend as close to the bottom of the flask as possible; it is connected to rubber tubing leading to the beaker. The other glass tube in the two-hole stopper in the flask should not extend much below the stopper

The flask should be nearly filled with water, and the tubing connecting the flask to the beaker should be completely filled with water. To get air out of the tubing, disconnect the two-hole stopper from the flask and siphon water back and forth between the flask and the beaker by raising and lowering the beaker (or the flask). Or, with the 2-hole stopper in place in the flask, disconnect the tubing to the test tube and blow through it.

With everything connected, and with the water level nearly to the top of the flask, and with some water in the beaker, raise the beaker until the water level in the beaker is even with the water level in the flask. Place a clamp on the rubber tubing between the flask and the beaker. This adjusts the pressure in the flask to be the same as the measured barometric pressure.

(If the water levels an not equalized, the pressure in the flask differs from atmospheric pressure because of pressure exerted by the weight of water of depth x; see figure 1. When the water levels in the beaker and flask are the same them is no difference in pressure. We will repeat the leveling procedure at the end of the experiment to assure atmospheric pressure in the system at the beginning and at the end of the experiment.)

With the clamp in place, empty the beaker completely, but do not dry it. Place the tubing back into the beaker and remove the clamp. A small amount of water will flow into the beaker; do not dump it out. (However, if water continues to flow into the beaker, there is a leak in the system, which must be found and corrected before proceeding.)

Caution: Wear approved eye protection while doing this experiment.

Do not heat $KClO_3$ in contact with rubber stopper.

Gently begin heating the mixture in the test tube. If white vapors no longer appear, the heating can be increased. Continue heating until no further change in volume is observed in the flask and beaker, then heat strongly for an additional 5 minutes to be sure that no more oxygen is evolved. It is important to decompose ALL the $KClO_3$; if there is any evidence of further evolution of oxygen, continue heating. Another indication of completeness of the reaction is that $KClO_3$ will melt, but the final product, KCl, will be solid and no purple color will remain; if any molten material or purple color is still present, continue heating until the sample completely solidifies and no purple color remains.

When you are satisfied that the reaction is completed, allow the system to cool. Keep the tubing in the beaker and do not disconnect anything while it is coiling; as the system cools, the volume of the gas will decrease and water will siphon back into the flask. When the system reaches room temperature, equalize the pressure by raising or lowering the beaker or the flask, until the water levels are the same; again place the clamp on the rubber tubing between the flask and the beaker.

With the clamp in place, you are ready to measure the final weight, volume and temperature.

(3) Weigh the test tube containing the decomposed sample to the nearest 0.0001 gram.

(4) Remove the stopper from the flask and place a thermometer into the gas in the flask. Measure the temperature to the nearest 0.1 °C by estimating between the marks on the thermometer. This is the temperature of the gas; you can calculate the absolute temperature (Kelvin) by adding 273.2 to the Celsius temperature.

(5) The volume of the gas is the volume of water displaced into the beaker. Using a 100 or 500 mL graduated cylinder, measure the volume of water in the beaker. (Using the 500 mL cylinder you can measure to the nearest mL by estimating between the marks. A 100 mL cylinder can be read to the nearest 0.2 mL, but filling it several times to get the total volume may still give an uncertainty close to a mL.)

(6) Read and record the pressure on the barometer to the nearest 0.1 mm Hg. If you do not **know how** to read the barometer, your instructor will assist you.

(7) Read the temperature of the barometer, this temperature will be used to apply a correction to the barometric pressure reading.

At least two trials should be performed; that is, at least two separately weighed portions of the sample should be run through the entire procedure. If the calculations for percent $KClO_3$ does not agree for the two trials, you should perform a third trial on another separately weighed portion of the sample. However, if reasonable results on the determination of the gas constant R are obtained for the two trials, but the percents do not agree, you may reheat the sample (or samples) without collecting oxygen, just to get a better value for the percent $KClO_3$ (assuming that the problem is incomplete decomposition of the sample). You may then count the reheating as a third (or fourth) trial, for calculating the percent but ignoring the parts that have to do with R and the molar volume.

Calculations

(8) Obtain the aqueous vapor pressure at the temperature of the oxygen gas (NOT the temperature of the barometer) from a table in your textbook or from the CRC Handbook. The temperature of the gas should have been recorded to the nearest 0.1 °C; interpolate to get the correct vapor pressure to the nearest 0.1 mmHg.

(9) The loss in weight (test tube and sample before heating (2) minus the test tube and sample after heating (3)) is the weight of oxygen obtained.

(10) The number of moles of oxygen is obtained by dividing the weight of oxygen (9) by 32.00 g/mol.

(11) The absolute temperature (Kelvin) Is the Celsius temperature of the gas (4) + 273.2.

(12) The barometric pressure is corrected for scale expansion by subtracting 1/8 of the Celsius temperature of the barometer (7) from the barometer reading (6). For example, if the barometer reads 755.5 mm Hg at 24.0 °C, subtract 3.0 mm Hg to get a corrected reading

of 752.5 mm Hg. This correction is necessary because mercury expands with temperature so the barometer reads higher than it should.

(13) The pressure of oxygen is obtained by subtracting the vapor pressure of water (8) from the total corrected pressure (12).

(14) The ideal gas constant R = PV/nT. We want the value in liter atm/mol K. To get liters, divide the volume of oxygen (5) by 1000 mL/L. To get atmospheres, divide the pressure of oxygen (13) by 760 mmHg. Multiply this pressure by volume, divide by the number of moles (10) and divide by the Kelvin temperature (11).

(15) The molar volume at STP is the volume 1 mole of the gas would occupy at 0 °C (273.15 K) and 1 atmosphere. From V = nRT/P, if n = 1 and P = 1, then the volume (at STP) = R x 273.15. Use YOUR value for R (14) and multiply by 273.15.

(16) % Error = (experimental value - theoretical value)/ theoretical value. The theoretical value of R for an ideal gas is 0.082056 Latm)/Kmol The theoretical value for the molar volume of an ideal gas at STP is 22.4136 L/mol. Using either your value for R or your value for the molar volume, calculate the percent error. (You should get the same answer either way.)

(17) Moles of $KClO_3$ decomposed: From the balanced equation for the reaction, 2 moles of $KClO_3$decompose to form 3 moles of oxygen. Therefore, moles of potassium chlorate = two-thirds the number of moles of oxygen.

(18) Grams of $KClO_3$ = number of moles times the molecular weight 122.55.

(19) The total weight of the original sample is the weight of tube and contents before heating (2) minus the weight of the empty test tube (1).

(20) Percent $KClO_3$ =[grams $KClO_3$ (18) ÷ total weight of sample (19)] x 100

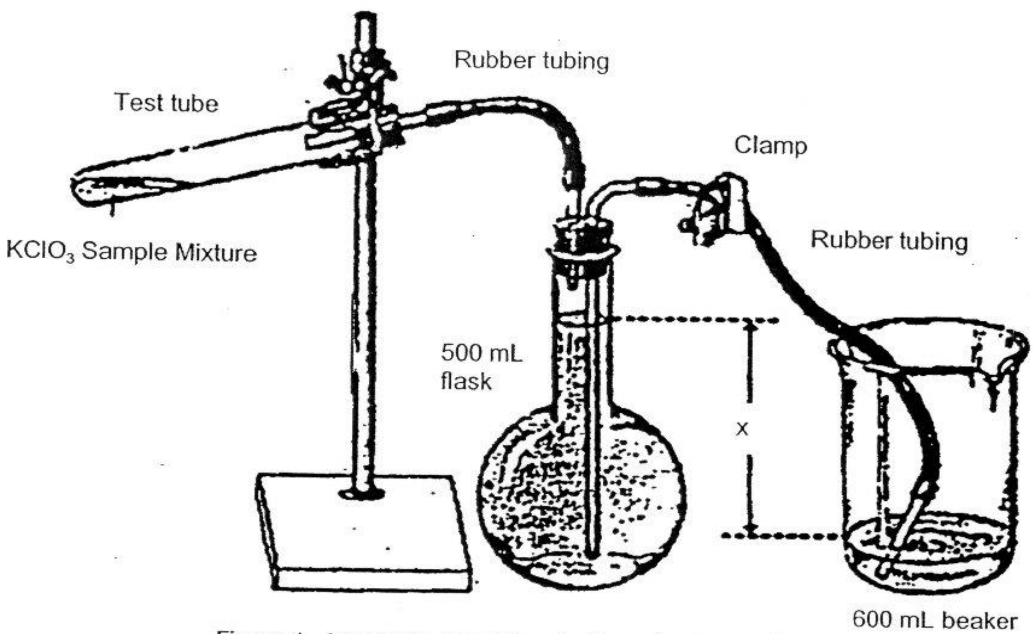

Figure 1. Apparatus for determination of volume of oxygen gas generated by decomposition of $KClO_3$

Name ______________________ ______________ Section ______________ Date

DATA SHEET

DATA

		DETERMINATIONS 1	2	3
Will this trial be used to determine R?				
Mass of test tube (grams)	(1)			
Mass of tube and contents before heating (g)	(2)			
Mass of tube and residue after heating (g)	(3)			
Temperature of oxygen (°C)	(4)			
Volume of oxygen collected (mL)	(5)			
Barometer reading, uncorrected (mm Hg)	(6)			
Temperature of barometer (°C)	(7)			

CALCULATIONS

Aqueous vapor pressure at temperature of gas	(8)			
Mass of oxygen (grams)	(9)			
Moles of your oxygen	(10)			
Temperature, absolute (Kelvin)	(11)			
Barometric pressure, corrected for scale exp.	(12)			
Pressure of oxygen alone (mm Hg)	(13)			
Gas constant R	(14)			
Molar volume of oxygen from your expt, L/mol	(15)			
Percent error	(16)			
Moles of $KClO_3$ decomposed	(17)			
Mass of $KClO_3$ in sample (grams)	(18)			
Total mass of original sample (grams)	(19)			
Percent $KClO_3$ in sample	(20)			

Name___________________

Post-Lab Assignment –Analysis of $KClO_3$ mixture & Determination of "R"

1. Based on your experimental data (i.e., volume and mass of gas produced, etc.), determine if the collected gas product obeys the ideal gas law - Compare your experimental value of R with the theoretical value by calculating the **% difference**. Does the gas behave ideally? **Explain.**

2. Other than human error, what errors could affect your %$KClO_3$ and R values? **Explain.**

3. What mass of $KClO_3$ (in kg) must be decomposed to supply 8 people with enough oxygen for 24 hours on a small submarine? Although this depends on the size of the person and their respiration rate (activity), according to NASA, an average person needs about 0.84 kg of O_2 per day. **All work must be shown clearly.**

$2KClO_3 \rightarrow 2KCl + 3O_2$

Analysis of $KClO_3$ Prelab Report.

Name______________________________

Lab Section ________________________

1. Write a balanced equation for the thermal decomposition of $KClO_3$

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 number of moles of oxygen produced? (Be careful, don't pick the wrong things.)

4. The water level in the Florence flask was higher than the water level in the collection beaker at the end of his experiment, and the student failed to raise the beaker up so the levels were equal before sealing the rubber tube between the flask and the beaker. What effect will this have on the student's calculation of?

    a. Mass of oxygen released ____________________
    b. Moles of oxygen produced ____________________
    c. Volume of oxygen collected ____________________
    d. Percent $KClO_3$ in the sample____________________
    e. Molar volume of oxygen ____________________

5. A student obtained the following data:

a. Mass of test tube	12.1456g
b. Mass of tube & sample before heating	13.3278g
c. Mass of tube and residue	12.9987g
d. Volume of oxygen collected	248 mL
e. Temperature of gas	21.0 °C
f. Corrected barometer reading	742.3 torr

From the data find: (show your work on the back of this page)

a. Grams of sample __________
b. Grams of oxygen released __________
c. Grams of $KClO_3$ decomposed __________
d. Percent $KClO_3$ in sample __________
e. Molar volume of oxygen __________