Reading assignment: Chang, Chemistry 10th edition, pp. 135-141.

Goals

We will observe the thermal decomposition of several oxygen-containing compounds called oxides. We will collect molecular oxygen and demonstrate its reactivity with several chemical elements.

Safety Note: Safety glasses are required when performing this experiment

Equipment and Materials

Solid samples of sodium nitrate, sand (silicon dioxide), lead(IV) oxide, magnesium oxide, mercury(II) oxide, and potassium chlorate in test tubes. Potassium chlorate mixed with manganese dioxide in a large test tube, oxygen collection apparatus with three collection jars, solid phosphorus, solid sulfur, magnesium ribbon, universal indicator, and phenolphthalein.

Discussion

Oxygen is a plentiful and highly reactive element on the earth. Oxygen comprises about 21% of the earth's atmosphere. In its elemental form oxygen is a gas and diatomic.

Major Components in Earth's Atmosphere

Gas		Percent Mass	Gas		Percent Mass
nitrogen	N_2	78.084	neon	Ne	0.00182
oxygen	O_2	20.947	helium	He	0.00052
argon	Ar	0.934	krypton	Kr	0.00010
carbon dioxide	CO_2	0.033			

The earth's crust contains a large amount of oxygen as well, primarily in the form of silicates.

Elemental Abundances in Earth's Crust

Element	Percent Mass	Element	Percent Mass
oxygen	46.6	sodium	2.8
silicon	27.7	potassium	2.6
aluminum	8.1	magnesium	2.1
iron	5.0	others	1.5
calcium	3.6		

Many metals react with oxygen to form oxides. For example, copper reacts with oxygen to form copper (II) oxide. Iron reacts with oxygen to form iron(III) oxide (rust). Magnesium also reacts with oxygen, forming magnesium oxide:

$$\mathrm{Cu(s)} + \frac{1}{2}\mathrm{O_2(g)} \longrightarrow \mathrm{CuO(s)}$$

$$\mathrm{2Fe(s)} + \frac{3}{2}\mathrm{O_2(g)} \longrightarrow \mathrm{Fe_2O_3(s)}$$

$$\mathrm{Mg(s)} + \frac{1}{2}\mathrm{O_2(g)} \longrightarrow \mathrm{MgO(s)}$$

Metalloids will also react with oxygen. Upon reaction with oxygen silicon forms silicon dioxide, the main component of sand.

$$\mathrm{Si(s)} + \mathrm{O_2(g)} \longrightarrow \mathrm{SiO_2(s)}$$

Just as with metals and metalloids, the product of the reaction of a nonmetal with oxygen is an oxide.

Elemental sulfur (S_8) reacts with oxygen to form sulfur dioxide, a poisonous gas that can be used as a food additive to sterilize dried fruit and wine. Carbon burns in oxygen to form carbon monoxide and carbon dioxide gases. Elemental phosphorus (P_4) combines with oxygen to form solid tetraphosphorus decaoxide:

$$S_8\,(s) + O_2\,(g) \longrightarrow SO_2\,(g)$$

$$C\,(s) + O_2\,(g) \longrightarrow CO\,(g) \qquad C\,(s) + O_2\,(g) \longrightarrow CO_2\,(g)$$

$$P_4\,(s) + O_2\,(g) \longrightarrow P_4O_{10}\,(s)$$

Preparation of Molecular Oxygen

One way to obtain highly pure oxygen is to compress and cool air until it liquefies. Since air is composed primarily of nitrogen and oxygen, liquefying air is an efficient method for obtaining oxygen. Oxygen liquefies at 88 K (-185°C). Nitrogen liquefies at 77 K (-196°C). By cooling to a temperature below 77 K both oxygen and nitrogen liquefy. Oxygen can then be separated from nitrogen by heating the liquefied air to a temperature between 77 K and 88 K. This method is used for large-scale production of oxygen.

Thermal decomposition is a less sophisticated method that can be used to generate small amount of molecular oxygen. In this process, molecular oxygen is generated by heating a metal oxide until it chemically decomposes. For example, mercury(II) oxide decomposes into mercury and oxygen at 400°C:

$$2HgO\,(s) \xrightarrow[\text{heat}]{} 2Hg\,(l) + O_2\,(g)$$

Some oxides, like magnesium oxide, are very stable and do not decompose under the heat from a Bunsen burner:

$$MgO \xrightarrow[\text{heat}]{} \text{No Reaction}$$

Whether a metal oxide compound decomposes easily, like mercury(II) oxide, depends to a large extent on the strength of the metal-oxygen bonds.* If the bonds are strong, as in the case of magnesium oxide, then a large amount of energy (high temperature) is required to break them. If they are relatively weak, as in the case of mercury(II) oxide, then the amount of energy required to break the bonds is smaller, and the temperature at which the compound decomposes is relatively low.

Oxides formed from reaction with oxygen can be studied by considering their acid-base properties. Oxides that contain nonmetals react with water to form acids. Oxides that contain metals react with water to form bases. For example:

$$\underset{\text{sulfur dioxide}}{SO_2\,(g)} + H_2O\,(l) \longrightarrow \underset{\text{sulfurous acid}}{H_2SO_3\,(aq)} \qquad \text{nonmetal}$$

$$P_4O_{10}\,(s) + H_2O\,(l) \longrightarrow \underset{\text{phosphoric acid}}{H_3PO_4\,(aq)} \qquad \text{nonmetal}$$

$$MgO\,(s) + H_2O\,(l) \longrightarrow \underset{\text{magnesium hydroxide (base)}}{Mg(OH)_2\,(aq)} \qquad \text{metal}$$

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*The actual term for bond strength when considering ionic compounds, as in HgO and MgO, is lattice energy. Lattice energy is the amount of energy released when separated gaseous ions are packed together to form an ionic solid:

$$M^+\,(g) + X^-\,(g) \rightarrow MX\,(s) + \text{Energy released}$$

where M^+ is a metal cation and X^- is a nonmetal anion.

Decomposition of Oxides

The heating of many oxides with a Bunsen burner results in their decomposition. This results in the formation of molecular oxygen (O_2). For example, potassium chlorate decomposes under heat to form potassium chloride and oxygen gas. Many oxides decompose to form oxygen gas:

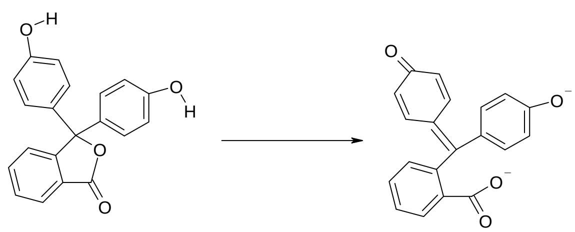
Potassium chlorate:	$KClO_3$ (s) $\rightarrow$ KCl (s) + O_2 (g)
Lead (IV) oxide:	PbO_2 (s) $\rightarrow$ PbO (s) + O_2 (g)
Potassium nitrate:	KNO_3 (s) $\rightarrow$ KNO_2 (s) + O_2 (g)
Mercury (II) oxide:	HgO (s) $\rightarrow$ Hg (l) + O_2 (g)

On the other hand, some oxides are particularly stable and do not decompose, even under the intense heat of a Bunsen burner. These include:

Magnesium oxide:	MgO (s) $\rightarrow$ No decomposition
Silicon dioxide:	SiO_2 (s) $\rightarrow$ No decomposition

Testing Acid-Base Properties of Oxides

Acid-base indicators are compounds that can measure the level of acidity for solutions. For example, phenolphthalein is a compound that is colorless in acidic solutions and pink in basic solutions. The structure of phenolphthalein is shown below:



in acidic solution (colorless) in basic solution (pink)

By adding a few drops of acid-base indicator solution to an aqueous solution of an oxide compound we can determine whether the oxide forms an acidic solution or a basic solution. Another acid-base indicator is called universal indicator. It's actually a mixture of several indicators that give a better idea of how acidic a solution is. The color change for several acid-base indicators is given below. Universal indicator is actually a mixture of several indicators that are sensitive to different ranges of acidity. A protocol for making universal indicator is to mix thymol blue, methyl red, bromothymol blue, and phenolphthalein. This mixture is then sensitive to how acidic or basic the solution is. When the solution is very acidic universal indicator is red. If the solution is somewhat acidic the color changes to orange-yellow. If the solution is somewhat basic the color changes to blue. And if the solution is very basic then the solution turns violet.

Acid-base indicator	**in acidic solutions**	**in basic solutions**
Phenolphthalein	Colorless	Pink
Litmus	Red	Blue
Bromothymol blue	Yellow	Blue
Universal indicator	Red, orange, yellow	Blue, indigo, violet

Procedure

SAFETY PRECAUTIONS

Safety glasses or goggles are required for this experiment. Potassium chlorate reacts violently with organic materials like rubber, wood, clothing, and skin. The heating of potassium chlorate should be carried out with caution. The burning of sulfur and phosphorus must take place in the fume hood. The products of the combustion of sulfur and phosphorus are irritants and should not be inhaled. Burning magnesium metal produces a large amount of ultraviolet light and should not be looked at directly.

Students will work in pairs throughout this experiment.

Part 1: Decomposition of oxygen-containing compounds

Place the following solids in separate dry test tubes to a depth of about one-half of a centimeter:

1. sodium nitrate
2. silicon dioxide
3. lead(IV) oxide
4. magnesium oxide
5. potassium chlorate

Write the chemical formulas for each compound on the data sheet. Describe the physical properties of these oxides. Physical properties include color, texture, and state of matter. Ignite the Bunsen burner. Place the first oxide in a test tube holder and begin heating it, slowly at first. Be sure that the opening of the test tube is pointed away from you and any other person. Keep the test tube away from your body. Slowly expose the test tube to more heat. Observe any changes that occur. Any changes in color, texture, gas formation, melting, etc., should be written in the data sheet. Eventually, the bottom of the test tube should be exposed to the hottest part of the flame for at least two minutes.

Optional

Gas formation is an indication that the oxide is decomposing and producing oxygen gas. If there is a visible formation of gas you can try to observe whether the gas is oxygen. To do this heat a small wooden splint with the Bunsen burner until it is glowing. Insert this splint half-way into the test tube and observe what happens. Do not drop the wood splint into the decomposing oxide.

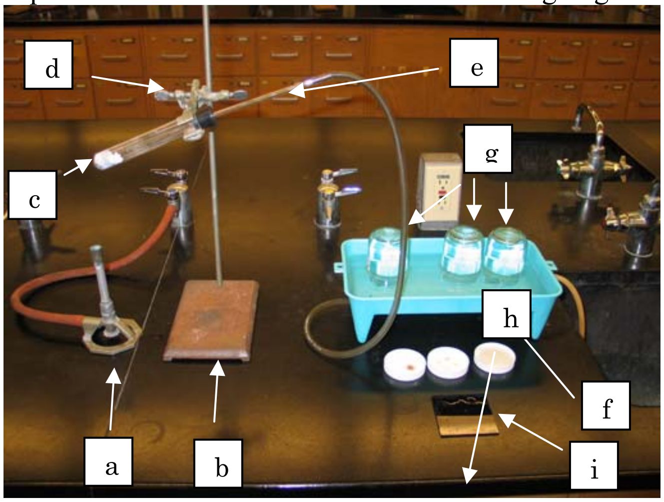
Allow the hot test tube to cool for one minute and then place it into a 250 mL beaker, not in the wooden test tube holder. Heat each of the remaining oxides using the same procedure as with the sodium nitrate.

Part 2: Collection of Oxygen Over Water

Procedure

1. Measure about seven grams of potassium chlorate ($KClO_3$) using a weighing boat and the digital analytical balance. Record this mass in the data sheet.

2. Add about one gram of manganese dioxide* to the potassium chlorate and mix them in the weighing boat. Pour this mixture into a clean, dry test tube provided for you.



Consider the oxygen collection apparatus to the right. The major components are:
a. Bunsen burner.
b. Ring stand
c. Test tube containing potassium chlorate and manganese dioxide.*
d. Clamp and clamp holder.
e. Oxygen transfer hose with rubber stopper.
f. Collection trough.
g. Collection bottles
h. Latex overflow hose.
i. Glass plate.

Assemble your apparatus like the one shown to the right.

3. Clamp the test tube near its opening.

4. Rubber is an organic material and the decomposing potassium chlorate will react violently with it or other organic materials. You must ensure that the decomposing potassium chlorate does not make contact with the rubber stopper. Tilt the clamp so that the test tube's opening is above the rest of the test tube and this will reduce the ability of the potassium chlorate to make contact with the stopper.

5. Insert the rubber stopper of the transfer hose into your test tube. The hose should be securely fastened to the collection trough. Position the trough so that the latex overflow hose is hanging over the sink.

6. Rinse out the three collection bottles with tap water so that they are clean. Measure and record the volume of one collection bottle using a 100 mL graduated cylinder and tap water.

7. Fill the collection trough with tap water.

8. Fill the other two collection bottles with tap water and place all three in the collection trough upside down. The glass plate can be used to cover the opening of the bottles as you place them under the surface of the water in the trough. Each bottle should be filled with water and positioned upside down in the trough.

*The systematic name of this compound is manganese(IV) oxide. However, it is commonly called manganese dioxide.

9. Once your apparatus has been checked by your instructor, begin heating the potassium chlorate/manganese dioxide mixture. Start by slightly heating the top of the mixture. Do not expose the mixture to a lot of heat at first. Bubbles should start to form in the water in the collection trough. This indicates that oxygen gas is being generated from the decomposition of potassium chlorate. The air in the rubber tubing must be pushed through before collecting any of the oxygen being generated. Keep heating the sample for about thirty seconds after you first see bubbles forming. Be sure that the hot mixture does not climb up the tube to near the rubber stopper. If it appears to be climbing up the tube, remove the heat from the tube and allow it to cool a little before exposing it to more heat.

10. Slide one of the collection bottles over the hole that the bubbles are passing through and hold the bottle down with one hand. As oxygen enters the bottle, it will displace the water, and the bottle will become buoyant. This could cause it to flip over. Increase the heating of the sample until a large number of bubbles are being formed.

11. Once almost all of the water in the bottle has been completely displaced, use one of the bottle lids to trap the oxygen in the bottle. You want to have a small amount of water (2-3 mL) in the bottle before you cap it. Slide a second bottle over the hole and collect more oxygen. Fill it with oxygen as you did with the first bottle. Continue with the third bottle.

12. Once you have collected three bottles with oxygen turn off the Bunsen burner and allow the mixture in the test tube to cool to room temperature. The bottles will be used in part 3 of this procedure.

13. Once the mixture has cooled to room temperature disconnect the rubber stopper from the test tube. Fill the test tube with tap water and clamp the test tube to the ring stand.

Part 3: The Chemistry of Oxygen and Oxides

As stated earlier, oxygen is a reactive element. To demonstrate this idea we will use the oxygen collected in Part 2 to react with elemental sulfur, phosphorus, and magnesium. In each case, the reaction will produce an oxide.

Procedure

1. Label one of your collection bottles "sulfur." Label the second bottle "phosphorus." Label the third bottle "magnesium."

2. Bring the bottle labeled "sulfur" to a fume hood. Place a small amount of sulfur in a deflagrating spoon. The spoon should be about one-third full.

3. Using the Bunsen burner, heat the sulfur until it starts to burn. Quickly open the bottle containing oxygen, and quickly place the spoon into the bottle. Allow the sulfur to burn in the oxygen-enriched atmosphere. Replace the lid onto the bottle. Shake the solution in the bottle. Take the bottle back to your work station.

4. Repeat the same procedure using phosphorus and the bottle labeled "phosphorus."

5. Obtain a 4 centimeter length of magnesium ribbon.

6. Using crucible tongs, hold the ribbon at one end, and place the third oxygen bottle labeled "magnesium" next to a Bunsen burner.

7. Carefully, but quickly, ignite the magnesium ribbon with the flame of the burner. Place it in the bottle. Magnesium metal emits ultraviolet light when it burns which can damage eyes. Do not look at the burning magnesium directly. Do not drop the magnesium in the bottle until it completely stops burning since its temperature is great enough to break the bottle if it makes contact with the glass. Once the magnesium does stop burning, drop the ashes of the reaction into the bottle. Replace the lid of the bottle and place it next to the other two bottles.

Part 4: The Acidity of Oxides Reacting with Water

1. Add two drops of universal indicator solution to the jar labeled "sulfur." Record the color of the solution.

2. Add two drops of universal indicator to the jar labeled "phosphorus." Record the colors of the solution.

3. Pour some of the solution in the jar labeled magnesium into a clean test tube. Add two drops of phenolphthalein indicator to the test tube and two drops of universal indicator to the jar. Record the colors of the two solutions.

4. Rinse the contents of the collection bottles into the sink.

5. Accurately measure the volume of one of your collection bottles (to the very top) using a 100 mL graduated cylinder. This will require greater than two volumes of the cylinder.

Acid-base properties of air and exhaled breath

6. Fill a round bottom flask with tap water. Place three drops of universal indicator solution into the flask.

7. Using a pasteur pipet or a 2 mL serological pipet, exhale into the first test for one to two minutes. Use the air from deep in your lungs. Record any color changes to the solution.

Date ______________

Data and Calculations Sheet
The Preparation and Properties of Oxygen
Part 1

Name ________________________________

Partner's Name________________________________

Name of oxide	Chemical formula	Physical properties and observations upon heating
sodium nitrate	_____________	____
silicon dioxide	_____________	____
lead(IV) oxide	_____________	____
magnesium oxide	_____________	____
potassium chlorate	_____________	____

Reaction with oxygen	**color of universal indicator**	**color of phenolphthalein indicator**
Sulfur $S_8(s) + O_2(g) \longrightarrow SO_2(g)$	____________	
Phosphorus $P_4(s) + O_2(g) \longrightarrow P_4O_{10}(s)$	____________	
Magnesium $Mg(s) + O_2(g) \longrightarrow MgO(s)$	____________	____________

Air and Breath

	color of universal indicator	**acidic or basic?**
in water	__________________	____________
in exhaled breath/water	__________________	____________

Measurement of Volume of Oxygen Gas

Volume of 1 collection bottle ____________ mL

Total volume of oxygen collected ____________ mL