1. Preparation and Properties of Some Common Gases

Objective

Two of the most common gaseous elemental substances (hydrogen and oxygen gases) will be prepared by chemical reaction. A gaseous compound substance (carbon dioxide) will also be prepared. In Choice I, hydrogen gas will be prepared by the action of acid on metallic zinc. The hydrogen will be collected by displacement of water. The flammability of pure hydrogen and of hydrogen/oxygen mixtures will be investigated. In Choice II, oxygen gas will be generated by the catalytic decomposition of hydrogen peroxide. The oxygen will be collected by displacement of water. The chemical properties of the oxygen collected will be examined. In Choice III, carbon dioxide will be generated by treatment of calcium carbonate with acid. The gas will be collected by water displacement. The properties of the carbon dioxide collected will be investigated.

General Technique for the Generation of the Gases

In each of the choices of this experiment, the gas generated will be collected by the technique of **displacement of water**. The gases to be generated—hydrogen, oxygen, and carbon dioxide—are not very soluble in water, so if they are bubbled from a closed reaction system into an inverted bottle filled with water, the water will be pushed from the bottle. The gas thus collected in the bottle will be saturated with water vapor, but is otherwise free of contaminating gases from the atmosphere.

Before beginning the specific choices assigned for your lab work, construct the gas generating apparatus shown in Figure 1-1 (it will be used for each of the choices). The tall vertical tube shown inserted into the Erlenmeyer flask is called a **thistle tube**. It is a convenient means of delivering a liquid to the flask. If a thistle tube is not available, a long-stemmed gravity funnel may be used.

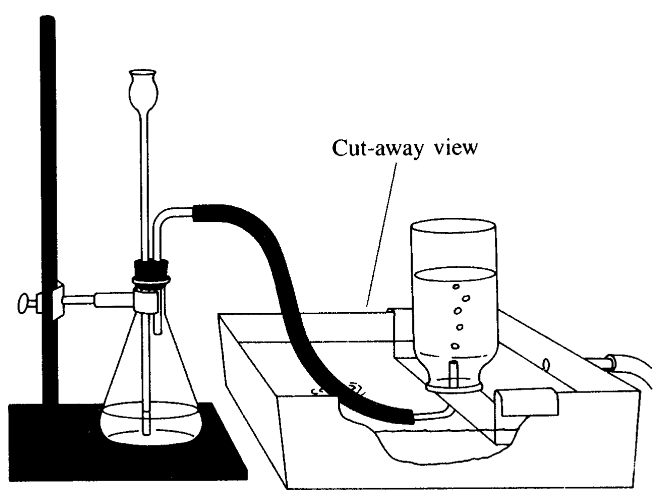

FIGURE 1-1
Apparatus for the generation and collection of a water-insoluble gas. A long-stemmed funnel may be substituted for the thistle tube shown. Be certain that the thistle tube or funnel extends to almost the bottom of the flask.

When inserting glass tubing or the stem of your thistle tube/funnel through the two-hole stopper, use plenty of glycerine to lubricate the stopper, and protect your hands with a towel in case the glass breaks. Make certain that the two-hole stopper fits snugly in the mouth of the Erlenmeyer flask. Rinse all glycerine from the glassware before proceeding.

Set up a trough filled with water. Fill three or four gas bottles or flasks to the rim with water. Cover the mouth of the bottles/flasks with the palm of your hand and invert into the water trough. Set aside several stoppers or glass plates; use them to cover the mouths of the bottles/flasks once they have been filled with gas. In the following procedure, be sure to keep the gas collection bottles or flasks under the surface of the water in the trough until a stopper or glass plate has been placed over the mouth of the bottle/flask to contain the gas.

When the trough and gas collection equipment are ready, begin the evolution of gas by the specific method discussed for each choice. Liquids required for the reaction are added to the gas generating flask through the thistle tube or funnel. The liquid level in the Erlenmeyer flask must *cover the bottom* of the thistle tube/funnel during the generation of the gas, or gas will escape up the stem of the funnel rather than passing through the rubber tubing. Add additional liquid as needed to ensure this.

Gas should begin to bubble from the mouth of the rubber tubing as the chemical reaction begins. Allow the gas to bubble out of the rubber tubing for 2–3 minutes to sweep air out of the system. Add additional portions of liquid through the thistle tube/funnel as needed to continue the production of gas, and do not allow gas evolution to cease (or water may be sucked from the trough into the mixture in the Erlenmeyer flask).

After air has been swept from the system, collect three or four bottles full of gas by inserting the end of the rubber tubing into the bottles of water in the trough. Gas will displace the water from the bottles. Remember to keep the mouth of the bottles under the surface of the water in the trough.

When the bottles of gas have been collected, stopper them while they are still under the surface of the water; then remove them. If glass plates are used to contain the gas rather than stoppers, slide the plates under the mouths of the gas bottles while under water and remove the bottles from the water. For hydrogen only, keep the bottles in the inverted position after removing them from the water trough (hydrogen is less dense than air).

Choice I. Preparation and Properties of Elemental Hydrogen

Introduction

Elemental hydrogen is a colorless, odorless, tasteless gas. Hydrogen can be generated by many methods, most notably during the electrolysis of water or by replacement from acids by active metals:

$$2H_2O(l) \rightarrow 2H_2(g) + O_2(g)$$

$$M(s) + nH^+(aq) \rightarrow M^+(aq) + (n/2)\ H_2(g) \quad \text{(M is an active metal)}$$

Pure elemental hydrogen burns quietly itself, but forms *explosive mixtures* when mixed with air prior to ignition. Hydrogen gas is considerably less dense than air, and for this reason was used as the buoyant medium for airships in the earlier part of this century. Because hydrogen forms explosive mixtures with air, however, it was replaced by helium for this purpose after the explosion of the German airship *Hindenberg*: helium is considerably more expensive than hydrogen, but is not at all flammable.

Elemental hydrogen is used extensively in the commercial production of the substance ammonia, which is a vital component in the production of fertilizers, explosives, and other important chemical substances.

$$N_2 + 3H_2 \rightarrow 2NH_3$$

Typically, ammonia manufacturing plants are located near sources of inexpensive electricity (such as hydroelectric stations) so that elemental hydrogen can be produced for the synthesis of ammonia by the electrolysis of water.

Elemental hydrogen has been used as the reducing agent in the production of pure metallic substances from their oxide ores, but generally cheaper and easier to handle reducing agents are preferred.

$$MO(s) + H_2(g) \rightarrow M(s) + H_2O(g)$$

Elemental hydrogen will be produced in this experiment by treating metallic zinc with an excess quantity of hydrochloric acid. Since hydrogen gas is relatively insoluble in water, the hydrogen gas will be collected by displacement of water from an inverted bottle.

SAFETY PRECAUTIONS

- **Wear safety glasses at all times while in the laboratory.**
- **Elemental hydrogen is flammable and forms highly explosive mixtures with air. Although the quantity of hydrogen generated in this experiment is quite small, exercise caution when dealing with the gas.**
- **Hydrochloric acid is damaging to skin. Wash immediately if it is spilled and inform the instructor.**
- **Examine the bottles/flasks to be used to collect the hydrogen gas. Replace any bottles/flasks which are cracked, chipped, or otherwise imperfect.**

Apparatus/Reagents Required

250-mL Erlenmeyer flask with tightly fitting two-hole stopper, glass tubing, thistle tube or long-stemmed funnel, rubber tubing, water trough, gas collection bottles (or flasks), stoppers to fit the gas collection bottles (or glass plates to cover the mouth of the bottles), wood splints, mossy zinc, 3 *M* hydrochloric acid, glycerine

Procedure

Record all data and observations directly in your notebook in ink.

A. Generation and Collection of Hydrogen

Construct the gas generator and water trough as indicated in Figure 1-1 for use in collecting hydrogen.

Add approximately a half-inch layer of mossy zinc chunks to the Erlenmeyer flask. Replace the two-hole stopper (with thistle tube/funnel and glass delivery tube) in the Erlenmeyer flask, and make certain that the stopper is set tightly in the mouth of the flask. Make certain that the stem of the funnel/thistle tube extends almost to the bottom of the flask.

When the trough and gas collection equipment are ready, begin the evolution of hydrogen by adding approximately 30 mL of 3 *M* hydrochloric acid to the zinc through the thistle tube or funnel. The liquid level must cover the bottom of the thistle tube/funnel or hydrogen will escape up the stem of the funnel rather than passing through the rubber tubing. Add additional HCl as needed to ensure this.

After sweeping air from the system for several minutes, collect three or four bottles of hydrogen. When the hydrogen has been collected, stopper the gas bottles under the surface of the water; then remove the bottles. Since hydrogen is lighter than air, keep the bottles inverted on your lab bench to prevent loss of the gas.

B. Tests on the Hydrogen

Bring two bottles of hydrogen to your instructor, who will peform two tests on the hydrogen for you. Be sure to wear your safety glasses at all times.

To demonstrate that hydrogen forms explosive mixtures with air, the instructor will hold one bottle of hydrogen, mouth downward, and will remove the stopper or glass plate. The bottle of hydrogen will be tilted *momentarily* at a 45-degree angle to allow some hydrogen to escape from the bottle and a quantity of air to enter the bottle. The instructor will then hold the bottle of hydrogen/air mixture at arm's length, and will bring the mouth of the bottle near a burner flame. A loud "pop" will result as the hydrogen explodes.

The instructor will light a wooden splint. The instructor will then hold a second bottle of hydrogen in the inverted position, will remove the stopper, and will quickly insert the burning splint deeply into the bottle of hydrogen. Observe whether or not the splint continues to burn when deep in the hydrogen. The instructor will then slowly withdraw the splint to ignite the hydrogen as it escapes from the mouth of the bottle. If this is done carefully, the hydrogen should burn *quietly* with a pale blue flame (rather than exploding as in the first test).

Open the third bottle of hydrogen and turn the bottle mouth upward for 30 seconds. After the 30-second period, invert the bottle and attempt to ignite the gas in the bottle. There will be no "pop." Because hydrogen is considerably lighter than air, the hydrogen will have already escaped from the bottle.

Choice II. Preparation and Properties of Elemental Oxygen

Introduction

Elemental oxygen is essential to virtually all known forms of living creatures. Oxygen is used on the cellular level in the oxidation of carbohydrates. For example, the sugar glucose reacts with oxygen according to

$$C_6H_{12}O_6 + 6O_2 \rightarrow 6CO_2 + 6H_2O$$

This reaction is the major source of energy in the cell (the reaction is exergonic). The chief source of oxygen gas in the earth's atmosphere is the activity of green plants. Plants contain the substance chlorophyll, which permits the reverse of the preceding reaction to take place. Plants convert carbon dioxide and water vapor into glucose and oxygen gas. This reverse reaction is endergonic and requires the input of energy from sunlight (photosynthesis).

Elemental oxygen is a colorless, odorless, and practically tasteless gas. While elemental oxygen itself does not burn, it supports the combustion of other substances. Generally a substance will burn much more vigorously in pure oxygen than in air (which is only 20% oxygen by volume). Hospitals, for example, ban smoking in patient rooms in which oxygen is in use.

Elemental oxygen is the classic oxidizing agent after which the process of oxidation was named. For example, metallic substances are oxidized by oxygen, resulting in the production of the metal oxide. Iron rusts in the presence of oxygen, resulting in the production of the rust-colored iron oxides.

$$2Fe + O_2 \rightarrow 2FeO$$

$$4Fe + 3O_2 \rightarrow 2Fe_2O_3$$

SAFETY PRECAUTIONS

- **Wear safety glasses at all times while in the laboratory.**
- **Hydrogen peroxide and manganese(IV) oxide may be irritating to the skin. Wash after use.**
- **Oxygen gas supports vigorous combustion.**
- **Sulfur dioxide gas is toxic and irritating to the respiratory tract. Generate and use this gas only in the exhaust hood.**

Apparatus/Reagents Required

250-mL Erlenmeyer flask with tightly fitting two-hole stopper, glass tubing, thistle tube or long-stemmed funnel, rubber tubing, water trough, gas collection bottles (or flasks), stoppers to fit the gas collection bottles (or glass plates to cover the mouth of the bottles), wood splints, deflagrating spoon, pH paper, 6% hydrogen peroxide solution, manganese(IV) oxide, sulfur, clean iron/steel nails, 1 *M* hydrochloric acid

Procedure

Record all data and observations directly in your notebook in ink.

A. Generation and Collection of Oxygen Gas

Construct the gas generator and set up the water trough as indicated in Figure 1-1 for use in collecting oxygen.

Add approximately 1 g of manganese(IV) oxide to the Erlenmeyer flask. Add also about 15 mL of water, and shake to wet the manganese(IV) oxide (it will *not* dissolve). Replace the two-hole stopper (with thistle tube/funnel and glass delivery tube) in the Erlenmeyer flask, and make certain that the stopper is set tightly in the mouth of the flask. Make certain that the stem of the funnel/thistle tube extends almost to the bottom of the flask.

When the trough and gas collection equipment are ready, begin the evolution of oxygen by adding approximately 30 mL of 6% hydrogen peroxide to the manganese(IV) oxide (through the thistle tube or funnel). Make certain that the liquid level in the flask covers the bottom of the stem of the thistle tube/funnel or the oxygen gas will escape from the system. Add more hydrogen peroxide as needed to ensure this.

The hydrogen peroxide should immediately begin bubbling in the flask, and oxygen gas should begin to bubble from the mouth of the rubber tubing. After sweeping air from the system for several minutes, collect three or four bottles of oxygen. Add 6% hydrogen peroxide as needed to maintain the flow of gas. When the oxygen has been collected, stopper the gas bottles under the surface of the water; then remove the bottles.

B. Tests on the Oxygen Gas

Ignite a wooden splint. Open the first bottle of oxygen, and slowly bring the splint near the mouth of the oxygen bottle in an attempt to ignite the oxygen gas as it diffuses from the bottle. Is oxygen itself flammable?

Ignite a wooden splint in the burner flame. Blow out the flame, but make sure that the splint still shows some glowing embers. Insert the glowing splint deeply into a second bottle of oxygen gas. Explain what happens to the splint.

Take the third stoppered bottle of oxygen to the exhaust hood. Place a small amount of sulfur in the bowl of a deflagrating spoon, and ignite the sulfur in a burner flame in the hood. Remove the stopper from the bottle of oxygen, and insert the spoon of burning sulfur.

Allow the sulfur to burn in the oxygen bottle for at least 30 seconds. Then add 15–20 mL of distilled water to the bottle, and stopper. Shake the bottle to dissolve the gases produced by the oxidation of the sulfur. Test the water in the bottle with pH paper. Write the equation for the reaction of sulfur with oxygen, and explain the pH of the solution measured.

Clean an iron (not steel) nail free of rust by dipping it briefly in 1 *M* hydrochloric acid; rinse with distilled water. If the surface of the nail still shows a coating of rust, repeat the cleaning process until the surface of the nail is clean.

Using tongs, heat the nail in the burner flame until it glows red. Open the fourth bottle of oxygen and drop the nail into the bottle. After the nail has cooled to room temperature, examine the nail. What is the substance produced on the surface of the nail?

Choice III. Preparation and Properties of Carbon Dioxide

Introduction

The amount of carbon dioxide in the earth's atmosphere is still relatively small but is increasing at an alarming rate. Carbon dioxide is a gaseous product of the combustion of petroleum-based fuels, and with the continued dependency of civilization on these fuels, the amount of carbon dioxide discharged into the atmosphere each year continues to increase. Although carbon dioxide is odorless, tasteless, colorless, and nontoxic, when present in the atmosphere, it enables radiant energy from the sun to be trapped on the earth as heat. In the past few decades, the mean temperature of the earth has increased by several degrees. If this increase in temperature continues, such consequences as partial melting of the polar ice caps could follow, which in turn would lead to serious flooding of low-lying coastal areas. The increase in temperature of the earth due to increasing levels of CO_2 is called the **greenhouse effect**.

Carbon dioxide is also produced by living creatures as an end product of the metabolism of carbohydrates. For example, when the sugar glucose is metabolized in cells, the reaction is

$$C_6H_{12}O_6 + 6O_2 \rightarrow 6H_2O + 6CO_2$$

Nature provided for a balance of carbon dioxide in the atmosphere. Whereas animals use oxygen from the atmosphere and produce carbon dioxide as a waste product, green plants are able to use carbon dioxide (through photosynthesis) and release oxygen gas. However, the quantity of carbon dioxide released from combustion of hydrocarbon fuels is too large for green plants to use fully. Also, the fraction of the earth's surface covered by green plants decreases each year as civilization spreads, as does the quantity of plankton in the earth's oceans.

In the laboratory, carbon dioxide can be generated chemically by treatment of a carbonate or hydrogen carbonate (bicarbonate) salt with acid. For example, if sodium hydrogen carbonate or sodium carbonate is treated with hydrochloric acid, the reactions are

$$NaHCO_3 + HCl \rightarrow NaCl + H_2O + CO_2$$

$$Na_2CO_3 + 2HCl \rightarrow 2NaCl + H_2O + CO_2$$

Carbon dioxide finds use in many situations. The most common type of fire extinguisher contains pressurized carbon dioxide (CO_2 does not burn and does not support combustion). The solid form of carbon dioxide (called dry ice) is used in the food industry to keep perishables frozen during shipment. The beverage industry uses carbon dioxide to carbonate sodas.

SAFETY PRECAUTIONS

- **Wear safety glasses at all times while in the laboratory.**
- **Hydrochloric acid is damaging to skin. If it is spilled, wash immediately and inform the instructor.**
- **Calcium hydroxide (limewater) is caustic and damaging to skin. If the solution is spilled, wash immediately and inform the instructor.**
- **Excess levels of carbon dioxide in the atmosphere of the lab may cause lightheadedness and cardiac distress. Make certain that the room is well ventilated, or perform the generation of CO_2 in the exhaust hood.**
- **Dry ice will cause frostbite if handled with the fingers. Use tongs or forceps to pick up the dry ice.**

Apparatus/Reagents Required

250-mL Erlenmeyer flask with tightly fitting two-hole stopper, glass tubing, thistle tube or long-stemmed funnel, rubber tubing, water trough, gas collection bottles (or flasks), stoppers to fit the gas collection bottles (or glass plates to cover the mouth of the bottles), wood splints, pH paper, calcium carbonate, 3 *M* hydrochloric acid, glycerine, saturated calcium hydroxide solution (limewater), drinking straw

Procedure

Record all data and observations directly in your notebook in ink.

A. Generation and Collection of Carbon Dioxide

Construct the gas generator and set up the water trough as indicated in Figure 1-1 for use in collecting carbon dioxide.

Add approximately 5 g of calcium carbonate (chalk) to the Erlenmeyer flask. Add about 10 mL of distilled water and shake to make a slurry (the salt will *not* dissolve). Replace the two-hole stopper (with thistle tube/funnel and glass delivery tube) in the Erlenmeyer flask, and make certain that the stopper is set tightly in the mouth of the flask.

When the trough and gas collection equipment are ready, begin the evolution of carbon dioxide by adding approximately 30 mL of 3 *M* hydrochloric acid to the calcium carbonate through the thistle tube or funnel. Make certain that the liquid level covers the bottom of the stem of the thistle/tube funnel, or carbon dioxide will escape from the system. Add additional HCl to ensure this.

The calcium carbonate should start bubbling immediately in the flask, and carbon dioxide gas should begin to bubble from the mouth of the rubber tubing. After sweeping air from the system for several minutes, collect three or four bottles of carbon dioxide. Add 3 *M* hydrochloric acid as needed to maintain the flow of gas. When the carbon dioxide has been collected, stopper the gas bottles under the surface of the water; then remove the bottles.

B. Tests on the Carbon Dioxide

Ignite a wooden splint. Open a bottle of carbon dioxide and slowly bring the burning splint near the mouth of the bottle to try to ignite the carbon dioxide as it mixes with air. Does carbon dioxide itself burn?

Ignite a wooden splint. Open a second bottle of carbon dioxide and insert the burning splint deeply into the bottle. Does carbon dioxide support combustion?

Open a third bottle of carbon dioxide; *quickly* add 25 mL of distilled water to the bottle and restopper. Shake the bottle for 5 minutes to allow carbon dioxide to dissolve in the water. Open the bottle and test the carbon dioxide solution with pH paper. Why is the solution acidic?

Open the fourth bottle of carbon dioxide and add 25 mL of clear limewater solution (saturated calcium hydroxide). Stopper the bottle *quickly* and shake for several minutes. What is the identity of the solid that forms in the solution?

Place about 25 mL of limewater in a beaker. Obtain a clean drinking straw, take a deep breath, and exhale through the straw into the limewater solution. Why does a precipitate form in the limewater?

Place about 25 mL of limewater in a beaker. Pick up a small piece of dry ice with tongs or forceps; add it to the limewater. Let the beaker stand until the dry ice has completely disappeared. Why does a precipitate form in the limewater?

Preparation and Properties of Some Common Gases

Date: ....
Course: ....
Section: ....
Instructor: ....
Student name: ....
Team members: ....
....
....

Prelaboratory Questions

1. In this experiment, various laboratory techniques are used to generate small quantities of some common gases. Use your textbook or a chemical encyclopedia to look up the major industrial methods of preparing the three gases (hydrogen, oxygen, and carbon dioxide) when very large quantities of the gases are needed. Summarize your findings here.

2. When collecting hydrogen gas in this experiment, you are cautioned to keep the bottles or flasks in the *inverted* position because hydrogen is lighter than air. Use a handbook of chemical data or the ideal gas law to determine the densities of hydrogen, oxygen, nitrogen, and carbon dioxide gases at 25°C and 1 atm pressure. Determine also the density of air (approximately 80% nitrogen, 20% oxygen by volume) under these conditions, and compare the density of hydrogen to the densities of each of the other gases.

Preparation and Properties of Some Common Gases

Date: ....
Course: ....
Section: ....
Instructor: ....

Student name: ....
Team members: ....
....
....

Results/Observations

Choice I. Hydrogen

Observation on adding acid to zinc

Test on igniting hydrogen/air mixture

Test on igniting pure hydrogen

Test on igniting hydrogen after inverting bottle

Choice II. Oxygen

Observation on adding hydrogen peroxide to MnO_2

Test on attempting to ignite oxygen

Test on inserting glowing splint into oxygen

Test on adding burning sulfur to oxygen

pH of sulfur/oxygen product solution

Equation for sulfur/oxygen reaction

Test on adding glowing iron nail to oxygen

Equation for iron/oxygen reaction

Choice III. Carbon Dioxide

Observation on adding acid to calcium carbonate

Test on trying to ignite carbon dioxide

Test on inserting burning splint into carbon dioxide

pH of carbon dioxide/water solution

Why is the CO_2/water solution acidic?

Test on shaking limewater with CO_2

Test on exhaling into limewater

Test on adding dry ice to limewater

What is the precipitate that forms with CO_2/limewater mixtures?

Write the balanced equation for the reaction.

Student name: .... Course/Section: .... Date: ....

Questions

1. Hydrochloric acid with metallic zinc was used to generate hydrogen gas. Was this reaction *specific* with HCl, or could *any* acid have been used?

2. What was the purpose of the manganese(IV) oxide added to the hydrogen peroxide in the generation of oxygen? Could any other substances have been used for this purpose?

3. Why do gases become saturated with water vapor when collected by the method used in this experiment? Suggest a method by which such water vapor could be removed from the gases.