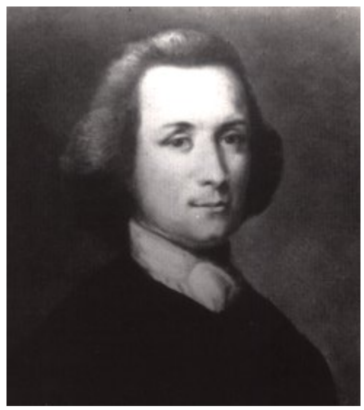
CHAPTER 4 OXYGEN

Carl Scheele, *1772*



Joseph Priestley, 1774

Antoine Lavoisier, 1789

These three men, Carl Scheele (Sweden), Joseph Priestley (England), and Antoine Lavoisier (France) all claimed credit for the discovery of the element that we now call oxygen.[1] Carl Scheele discovered ***fire air*** [oxygen] sometime before 1773. He produced the gas several ways. In one method, he reacted (using modern names) nitric acid with potash (KOH and/or K_2CO_3), which formed KNO_3. Distilling the residue with sulfuric acid produced both NO_2 and O_2. The former was absorbed by limewater (saturated $Ca(OH)_2$), leaving *fire air*. He also obtained *fire air* from strongly heating HgO and MnO_2 and by heating silver carbonate or mercuric carbonate and then absorbing the CO_2 by ***alkali*** (KOH):

$$AgCO_3(s) \rightarrow Ag(s) + CO_2(g) + O_2(g)$$

On August 1, 1774 Joseph Priestley first prepared oxygen by directing the sun's light with a 12-inch diameter burning lens onto a sample of red *mercurius calcinatus per se* (now HgO). Thus, Priestley independently had discovered oxygen, which he called ***dephlogisticated air***. His explanation of the reaction using was:

[1] To learn more about these three men, visit our gas website, mattson.creighton.edu/Microscale_Gas_Chemistry.html and click on “History of Gas Chemistry”

mercurius calcinatus per se + heat yields quicksilver + dephlogisticated air

Today, we would describe the same reaction as follows:

$$HgO(s) \rightarrow Hg(l) + O_2(g)$$

Priestley sampled his ***dephlogisticated air*** and wrote:

> *"[My] breast felt particularly light and easy for some time afterwards...... Who can tell, but that, in time, this pure air may become a fashionable article of luxury. Hitherto only two mice and myself have had the privilege of breathing it."*[2]

Priestley shared his discovery with Antoine Lavoisier later that same year and also described the discovery in published form shortly thereafter. Carl Scheele's account of his discovery did not appear in print until 1777, by which time the scientific community had given credit for the discovery to Priestley.

Antoine Lavoisier's claim to the discovery of oxygen is based on the fact that he established that oxygen was an element. He proposed the name oxygen and it became the cornerstone of his ***oxygen theory*** which debunked the prevailing ***phlogiston theory*** that had governed the minds of scientists for nearly one century. To spread his ideas and the Oxygen Theory, Lavoisier published ***Traité élémentaire de chemie*** in 1789. In his book, Lavoisier named a total of 33 elements, most of which are still in use today. It has been said that the book would be recognizable to a student of chemistry as it reads "like a rather old edition of a modern textbook."[2]

Oxygen, O_2, is a colorless, odorless, and tasteless gases that has a very low solubility in water. Oxygen can be condensed to a pale-blue liquid by cooling to -183 °C (90 K) at a pressure of one atmosphere. Oxygen's melting point is -218.4 ^{o}C (55 K).

Oxygen is the second largest component of the Earth's atmosphere (21%). It occurs as O_2 as well as the allotrope O_3, called ***ozone***, in the atmosphere. Oxygen represents 89% by mass of water molecules so that the Earth's water supplies are largely oxygen. Much of the Earth's lithosphere (rocks, solid parts of the crust) is composed of silicates and other oxides. Taken together, over 46% of the mass of the lithosphere is oxygen. Photosynthesis accounts for virtually all of the oxygen present in the Earth's atmosphere. Water and carbon dioxide are converted by chloroplasts to oxygen and plant carbohydrates, "CH_2O" respectively:

$$H_2O_{(l)} + CO_{2(g)} \rightarrow O_{2(g)} + \text{"}CH_2O\text{"}$$

The reaction is endothermic and occurs only because of the energy supplied by the sun.

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[2] ***A Short History of Chemistry***, J. R. Partington, 3rd edition, (1957)

The density of O_2 is 1.308 g/L at 25 ^oC and 1 atm — 10% greater than that of air. At 20 °C oxygen dissolves in water to the extent of 30.8 cm^3 per liter. It is more soluble in some non-aqueous solvents.

Oxygen has numerous important uses in society. Oxygen is obtained by the fractional distillation of air. The single largest use of oxygen is in the manufacture of steel. Oxygen is blasted under high pressure onto molten, impure iron to burn out impurities. Oxygen has a variety of other uses including respiratory oxygen in medical facilities, welding, sewage treatment, production of TiO_2 (used to make paints opaque and white), and ethylene oxide to name a few.

Suitability

The following experiments are included in this chapter.

Part 1. Experiments with Oxygen

Experiment 1. Traditional test for oxygen
Experiment 2. Oxygen supports combustion
Experiment 3. Dynamite soap
Experiment 4. Hydrogen-oxygen rockets

Part 2. Demonstrations and Advanced Experiments with Oxygen

Experiment 5. Steel wool burns in oxygen
Experiment 6. The Blue Bottle experiment
Experiment 7. Oxygen makes the flame hotter
Experiment 8. Mini-sponge shooter
Experiment 9. Chemiluminescence
Experiment 10. Paramagnetism of oxygen

The first four experiments are suitable as laboratory experiments for a wide variety of grade levels from middle school up through university-level. Due caution is required with hydrogen/oxygen mixtures and students must not be allowed to try different experiments without the express consent of the instructor.

In most high school settings, the first four experiments can be used very early in the year — at about the time that chemical formulas and reactions are being introduced. As a laboratory activity, these experiments are appropriate when discussing chemical compounds, chemical formulas, and chemical reactions. Experiments 1 and 2 demonstrate oxygen's ability to support and enhance combustion. The chemical reactions in Experiments 3 and 4 are very simple: hydrogen and oxygen produce water. Mole calculations may be done, but are optional. The law of combining volumes can be determined experimentally by playing with the H_2/O_2 mixture ratio in Experiments 3 and

4. Experiment 4 requires a piezoelectric sparking device that can be constructed from a piezoelectric lighter. Details are given in Appendix C.

Experiments 5 - 10 are suited for use as classroom demonstrations. Experiment 5 involves glowing hot steel wool and is quite impressive as a demonstration, but steel wool is messy and hot metal is dangerous. This experiment can be used to demonstrate that metals can burn and undergo rapid oxidation. A discussion of corrosion and rust formation can help students compare rapid oxidation with slow oxidation of metals. The experiment can be used when combustion reactions are discussed or oxidation as a type of chemical reaction is introduced.

The Blue Bottle experiment (Experiment 6) features a clear solution that turns blue upon shaking. This demonstration can be used repeatedly for over 24 hours. It can be used when discussing oxygen's solubility in water, including the depletion of dissolved oxygen. It makes a good demonstration when discussing natural waters, biological oxygen demand, water stagnation, etc. In a traditional chemistry course, we use it to as a demonstration of LeChâltelier's principle.

Experiment 7, Oxygen makes the flame hotter, is used as a demonstration because the ensuing discussion about the nature of a flame is best done as a teacher-led discussion.

Experiment 8, Mini-sponge shooter, in which a sponge projectile is shot up to 10 m requires a special syringe in which a hole has been drilled through the barrel. Use this demonstration when discussing explosions and explosive mixtures.

Experiment 9, Chemiluminescence uses a relatively toxic chemical, dimethylsulfoxide (dmso), that is inappropriate for handling by students. Use this demonstration when discussing types of energy — heat, light and electrical.

Experiment 10, Paramagnetism of Oxygen uses liquid nitrogen to generate liquid oxygen, which is then tested with a neodymium magnet. Cryoscopic liquids and neodymium magnets are inappropriate for handling by students. Use this demonstration when discussing paramagnetism and molecular orbital theory.

Background skills required

Students should be able to:

- ❖ generate a gas as learned in Chapter 1
- ❖ measure quantities of liquid reagents
- ❖ use a balance
- ❖ light matches and work with small flames in a responsible and appropriate manner for a science laboratory

Time required

Students should be able to perform these four experiments in one or two 45 minute laboratory periods.

Preparation of oxygen in a "gas bag"

Large samples of $O_2(g)$ can be prepared conveniently in 1 L food storage bag. See Chapter 5 for details.

Website

This chapter is available on the web at website:

http://mattson.creighton.edu/Microscale_Gas_Chemistry.html

Instructions for your students

Microscale Gas Chemistry Kits

Each pair of students will need certain equipment in order to prepare gases and perform experiments with the gases. We recommend organizing this equipment in 8 cup plastic food storage containers. Each kit should contain:

- ❖ two 60 mL plastic syringes with a LuerLOK fitting
- ❖ two Latex LuerLOK syringe caps
- ❖ two plastic vial caps
- ❖ one 15 cm length of Latex tubing
- ❖ one 3 cm length of Latex tubing
- ❖ one plastic pipet
- ❖ one clear plastic beverage cup (250 mL/9 oz)
- ❖ one small plastic weighing dish
- ❖ one small test tube (12 x 100 mm)
- ❖ one medium test tube (18 x 150 mm)
- ❖ one birthday candle

All of this will fit into the food storage container. In addition, each pair of students will need a wide-mouth beverage bottle for draining and supporting their syringes. Ordering information for kit materials is given at the end of this chapter and in Appendix E.

PREPARATION OF OXYGEN[1]

General Safety Precautions

Always wear safety glasses. Gases in syringes may be under pressure and could spray liquid chemicals. Follow the instructions and only use the quantities suggested.

Toxicity

Oxygen is non-toxic in normal quantities. Pure oxygen can be toxic if inhaled in large quantities as the pure gas, but this is not a concern with these experiments. Do not intentionally inhale oxygen samples produced in these experiments.

Equipment

Microscale Gas Chemistry Kit

Chemicals (needed for each syringe full of oxygen generated)

0.05 g solid KI powder (a spatula tip)
5 mL 6% $H_2O_2(aq)$

The production of O_2 is ***slow*** and it typically takes a minute or more to fill a syringe. Assist the plunger in its outward movement. To speed up the reaction, hold the plunger so that the contents inside the syringe are under reduced pressure, and while doing so, tap or shake the syringe. This process drives oxygen bubbles out of the solution. Potassium iodide is the catalyst in the reaction:

$$2\ H_2O_2(aq) \rightarrow 2\ H_2O(l) + O_2(g)$$

The actual mechanism has two steps:

$$\text{Step 1.}\quad H_2O_2(aq) + I^-(aq) \rightarrow H_2O(l) + IO^-(aq)$$

$$\text{Step 2.}\quad IO^-(aq) + H_2O_2(aq) \rightarrow I^-(aq) + H_2O(l) + O_2(g)$$

Oftentimes the solution takes on a yellow color due to $I_3^-(aq)$ that results form a competing side reaction:

$$2\ H^+(aq) + IO^-(aq) + 2\ I^-(aq) \rightarrow I_3^-(aq) + H_2O(l)$$

[1] Content for this chapter first appeared as “Microscale Gas Chemistry, Part 4. Experiments with Oxygen and Nitrogen” Mattson, B. M.; Lannan, J. ***Chem13 News***, **254**, January, 1997.

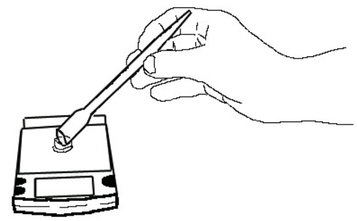
Generating oxygen gas samples

Samples of oxygen are generated by the In-Syringe Method as described in Chapter 1. A summary of these steps is provided here:

1. Wear your safety glasses

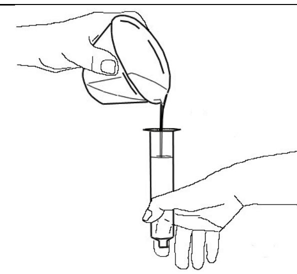
2. Measure out the solid reagent

(Use 0.05 g KI to make O_2)



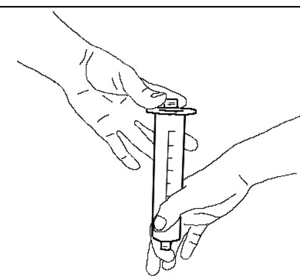
Place the solid potassium iodide into the vial cap. It may be difficulat to measure out such a small amount of potassium iodide. Your teacher may tell you how to estimate this quantity by showing you what 0.05 g KI looks like – the amount that fits on the tip of a spatula.

3. Fill the syringe barrel with water



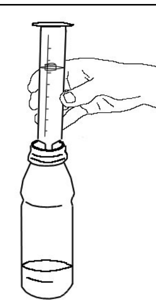
Fill the barrel with water. Place your finger over the hole to form a seal. Fill completely to the top.

4. Float the vial cap



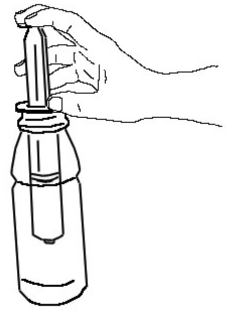
Float the vial cap containing the solid reagent on the water surface. This is easiest if the syringe barrel is filled completely to the top with water.

5. Lower the cap by flotation



Release the seal made by finger to lower the cap into the syringe barrel without spilling its contents. Option: Allow the syringe to drain into a wide mouth beverage container. When successfully completed, the cap should rest upright on the bottom of the syringe with all reagent still in the cap.

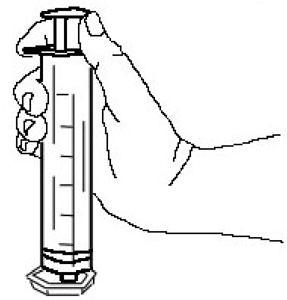
6. Install the plunger



Install the plunger while maintaining the syringe in a vertical position. The plunger has a plastic “rib” near the rubber seal that snaps past the “catch” — a small ridge just inside the mouth of the syringe. Usually it takes a firm push to move the rib past the catch. After that, the plunger should move smoothly. The plunger should fit snugly against the rim of the vial cap.

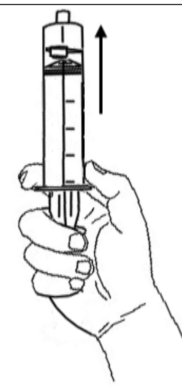
7. Draw aqueous reagent into syringe

(Use ~3 mL 6% H_2O_2 to make O_2)



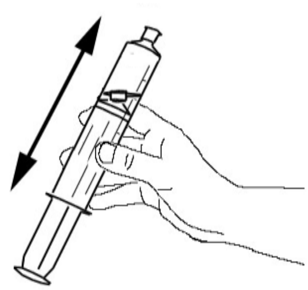
The aqueous reagent, measured into a small weighing dish, is drawn into the syringe while maintaining the vertical position of the syringe. The vial cap with the solid reagent should float on the solution. (The strength of hydrogen peroxide varies. Your teacher may have tested the hydrogen peroxide and may instruct you to use a different amount of hydrogen peroxide.)

8. Minimizing the quantity of air prior to reaction.



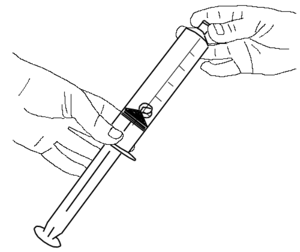
This procedure is used when there is excess air in the syringe before the reaction begins. Carefully rotate the syringe so as not to let the vial cap spill ints contents. With the syringe opening directed upward, carefully push the plunger inward until the air is mostly gone. Install the syringe cap by turning or pushing it on.

9. Generate the gas



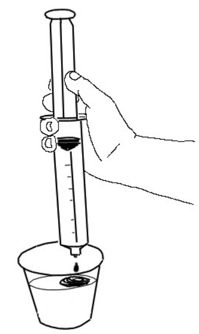
Read and understand this and the next two steps before proceeding. Hold the syringe so that the cap is up. Shake the syringe in order to mix the reagents. As the liquid reagent splashes into the vial cap, gas generation will commence and the syringe plunger should move outward. It is sometimes necessary to gently help the plunger move up the barrel. **CAUTION!** This reaction produces gas more slowly than either carbon dioxide or hydrogen, however, the solid reagent is not the limiting reagent this time and the amount of oxygen generated will often exceed 60 mL, depending on the quality and quantity of hydrogen peroxide used. One must watch the reaction continuously. Do not set it down or look away. Be prepared for the next step as soon as the plunger reaches the 60 mL mark!

10. Remove cap to stop gas collection



Keep the cap up! After the plunger has reached the desired mark (usually 60 mL), remove the syringe cap and leave it off through Step 11. The syringe is under positive pressure so there will be a tiny gas release sound.

11. Discharge reagents

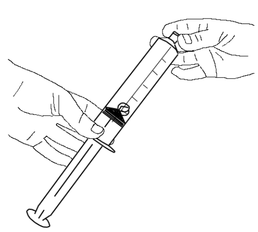
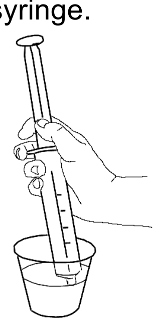
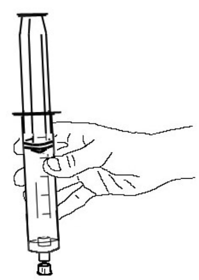
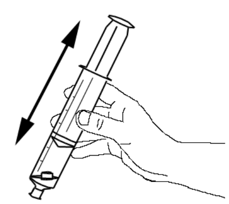
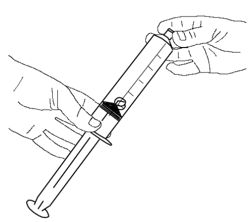
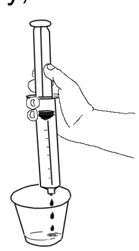
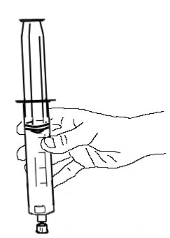


Turn the syringe 180º and discharge the liquid reagent into the plastic cup over half-filled with water in order to dilute the reactants. Immediately cap the syringe with the syringe cap to prevent loss of gas by effusion.

Caution: Always use Steps 9, 10, and 11 as described. Never remove the syringe cap with the cap end directed downward: Reagents will spray out of the syringe onto your hands and possibly clothing, leaving yellow iodine stains. **See Teaching Tip #4 to plan ahead in the event a student does get the solution on hands/clothing.**

12. Wash away contaminants

The oxygen-filled syringe may be "washed" in order to remove traces of unwanted chemicals from the inside surfaces of the syringe before the gas is used in experiments. To wash a gas:

1. Remove the syringe cap. 	2. Draw 5 mL water into the syringe. 	3. Cap the syringe. 	4. Shake syringe to wash inside surfaces. 
5. Remove cap. 	6. Discharge water only, and… 	7. …recap the syringe. 	8. Repeat? One rinse is usually enough.

Disposal of oxygen samples

Unwanted oxygen samples can be safely discharged into the room.

Teaching tips

1. The generation of oxygen is a slow reaction and students should be warned to be patient. Show them how to speed up the process by tapping the syringe while contents are under reduced pressure.
2. **CAUTION!** The amount of H_2O_2(aq) used (5 mL) is capable of generating more than 60 mL O_2(g) and syringes left unattended will eventually "pop" their plungers. This must be avoided because it sprays reagents that easily stain clothing and skin.
3. Using 10 mL 3% H_2O_2(aq) also works, although it is slower.
4. Aqueous sodium bisulfite (<1 M) can be used to remove iodine stains from clothing and skin. Rinse treated areas thoroughly with water.

Introductory Questions

1. What is the formula for oxygen gas?
2. What are the formulas for each of the following: (a) potassium iodide; (b) hydrogen peroxide; and (c) water?
3. Did the reaction speed up when you tapped the syringe while holding the plunger slightly outward? Why does this work? What happens when you tap a bottle of carbonated beverage? Is this somehow similar?

Questions

4. What would happen if some of the potassium iodide were to spill out of the cap before you were able to draw up the hydrogen peroxide? Specifically, what would happen when you tried to draw up the hydrogen peroxide?
5. Write the balanced chemical equation for the reaction occurring inside the syringe.
6. What is a catalyst?

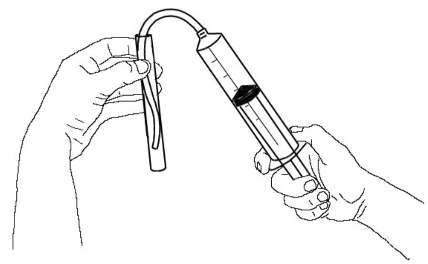
Advanced Questions

7. What mass of hydrogen peroxide is present in 5 mL of 6% H_2O_2(aq)? Assume the density of the solution is 1.00 g/mL. Convert this mass to moles.
8. Using the ideal gas law and your answer to the previous question, what volume of gas is predicted? (Assume the temperature is 25 ^oC and standard pressure)

PART 1. OXYGEN EXPERIMENTS FOR STUDENTS

EXPERIMENT 1. TRADITIONAL TEST FOR OXYGEN

Equipment



Microscale Gas Chemistry Kit
Wooden splint
Matches

Chemicals

$O_2(g)$, 30 mL
Limewater, 2 mL

Suitability

middle school lab, high school lab, university lab, and classroom demonstration

Applications, Topics, Purpose

combustion reactions, chemical properties of gases, oxygen supports combustion, characterization of gases. To demonstrate the traditional test for oxygen and to repeat a classical experiment of Joseph Priestley.

Instructions

Transfer 30 mL $O_2(g)$ to the kit's larger test tube (18 x 150 mm) using the long piece of tubing so that the gas can be discharged near the bottom in order to displace the air. Ignite and blow out the wooden splint and immediately plunge the burning splint into an oxygen-filled test tube. The splint will re-ignite. Hold the splint inside the test tube until it goes out (just a few seconds). Add about 2 - 3 mL of limewater to the test tube and shake the test tube to mix gas and limewater. Note the results.

Teaching tips

1. The larger test tube holds approximately 30 – 35 mL gas A test tube larger than the two in the kit (e.g. 25 x 200 mm) works well, too. Discharge all of the gas in the syringe into the test tube.

2. Joseph Priestley (1774) noted that a candle burns with greater brightness in oxygen.

Introductory Questions

1. Did the glowing splint re-ignite with oxygen? What would happen if you were to discharge carbon dioxide onto the glowing splint instead?

2. Why does wood burn with a flame sometimes and just glow red other times? Why does blowing gently on a glowing piece of wood often cause it to burst into flames?

3. Suppose you were given three test tubes and only one of them contained oxygen. What experiment would you use to determine which test tube contained oxygen?

Questions

4. As a continuation to Question 3, suppose that the other two test tubes contained carbon dioxide and hydrogen. What experiments would you use to determine the contents of the other two test tubes?
5. What type of chemical reaction is occurring in the experiment?
6. What gas is being produced when the wood splint burns?
7. Describe the results of the limewater test.

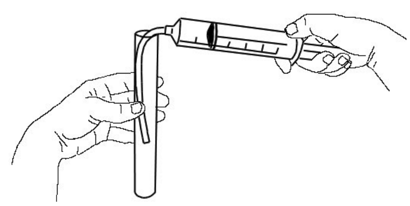
Advanced Questions

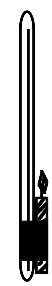
8. Write the chemical reaction that occurs during the limewater test.
9. When oxygen displaces air during the first part of the experiment, sketch how you perceive the air being displaced. Is it like pouring water through oil?

EXPERIMENT 2. OXYGEN SUPPORTS COMBUSTION

Equipment

Microscale Gas Chemistry Kit
test tube, large (25 x 200 mm)
glass rod
tape
matches





Chemicals

O_2(g), 50 mL
Limewater, 2 mL

Suitability

middle school lab, high school lab, university lab, and classroom demonstration

Applications, Topics, Purpose

combustion reactions, chemical properties of gases, oxygen supports combustion

Instructions

Tape the candle (in your kit) to the glass rod as shown. Transfer the oxygen gas to the large test tube using the long piece of tubing so that the gas can be discharged near the bottom in order to displace the air. Light the candle and lower it into the test tube. It should burn brightly for a moment before the oxygen is depleted. Test the gaseous contents of the test tube with limewater. Repeat the experiment with a test tube full of air: Either use a fresh test tube or fill the previous test tube with water to displace the gaseous contents of the test tube; drain the test tube and it is now filled with air.

Teaching tips

1. Try the experiment in the dark.
2. Joseph Priestley in the 1770s noted that a candle burns with greater brightness in oxygen.

Introductory Questions

1. Did the candle burn more brightly in the oxygen?
2. Did the candle burn longer in air or oxygen?
3. Do you think that the candle burned hotter in oxygen than in air?
4. Why does the candle go out?

Questions

5. Did anything unusual happen to the candle wax? What does this indicate?
6. What are the two products of combustion?
7. Discuss and interpret the results of the limewater test.
8. What are the three components needed to sustain a fire?

Advanced Question

9. Calculate the ratio of oxygen in the oxygen-filled test tube to the oxygen in air, given that air is 21% oxygen.

EXPERIMENT 3. DYNAMITE SOAP

Equipment

Microscale Gas Chemistry Kit

matches

Chemicals

$O_2(g)$, 15 mL

$H_2(g)$, 30 mL (Chapter 3)

3% dish soap, 10 mL

Caution!

Always wear safety glasses! When performed as instructed, this is a very rewarding experiment. An explosive mixture of hydrogen and oxygen is produced and stored in a syringe for experiments. Due care must be exercised to prevent the mixture from unintentionally exploding in the syringe. **Keep the syringe capped when not discharging gas!** Explosions are caused by (1) the syringe cap not in place, and (2) a source of ignition such as sparks, flames, or static electricity. This will result in the plunger being discharged from the barrel with considerable force, potentially causing injury. Discharge remaining gas into the room when finished with this and the next experiment.

Suitability

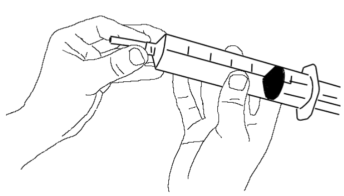
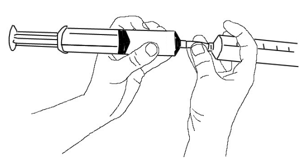
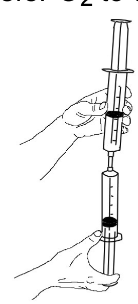
high school lab, university lab, and classroom demonstration

Applications, Topics, Purpose

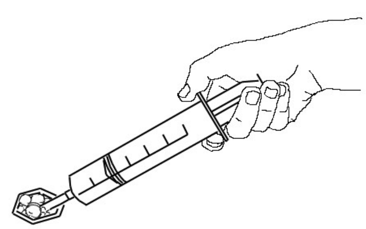
combustion reactions, kinetics, stoichiometry of reactions, activation energy, explosive mixtures

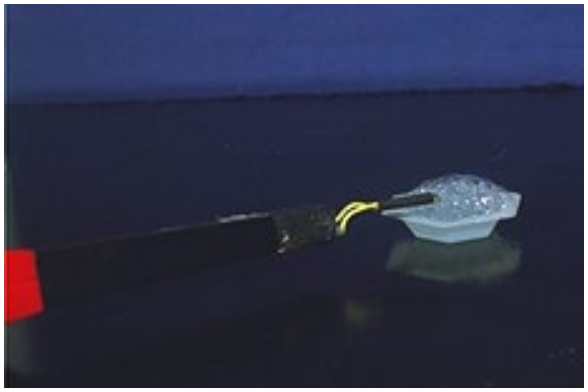
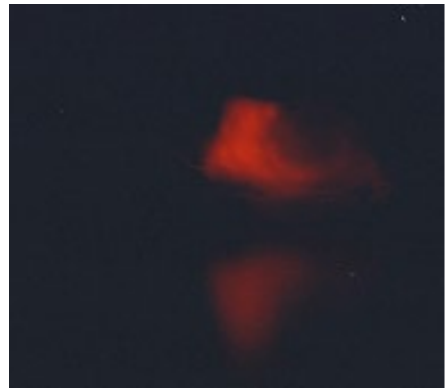
Instructions

Transfer 15 mL of the O_2 to a syringe containing 30 mL H_2 syringe as follows:

1. Connect a short piece of tubing to the oxygen syringe. 	2. Connect the other end of the tubing to the hydrogen syringe: 	3. Transfer O_2 to the H_2 

Form a 1:2 mixture. Re-label the syringe mixture. Use the short tube to form a mound of bubbles in a small weighing dish as shown at right. Cap the H_2 + O_2 syringe and remove it to a safe distance (at least 250 cm) from any flame source. Ignite the bubbles with a match. Caution! LOUD! (Unused gas mixture can be saved for the next experiment.)



Teaching tips

1. Students need to tell everyone in the room before igniting their soap bubbles.
2. The name 'dynamite soap' was coined by "Weird Science," (Western Chicago Area Chemistry Teachers' Alliance)
3. You may wish to generate a bag of hydrogen for distribution to the students in order to save time in the laboratory. See Chapter 5.

Introductory Questions

1. Describe your observations in writing for someone who did not see this reaction.
2. What is the purpose of the soap solution?
3. Why should extreme care be exercised when working with hydrogen-oxygen mixtures?
4. This experiment is an example of a mini-explosion. What are some dangers associated with scaling reactions such as this one up in size?
5. Were you aware that it is illegal to scale up any reaction to a size that can hurt others?

Questions

6. Would you expect a louder or softer BANG if you used the same volume of air instead of the oxygen you used in the experiment?
7. Why did you need a flame or spark to get the reaction to go?

Advanced Questions

8. What volume of oxygen gas is needed to react with 10 mL hydrogen?
9. Is energy absorbed or released by the reaction in the soap bubbles?

EXPERIMENT 4. HYDROGEN-OXYGEN ROCKETS

Equipment

Microscale Gas Chemistry Kit
piezoelectric sparker (Appendix C)
wide-stem disposable pipet

Chemicals

O_2(g), 30 mL
H_2(g), 30 mL (Chapter 3)

Caution!

Always wear safety glasses! When performed as instructed, this is a very rewarding experiment. An explosive mixture of hydrogen and oxygen is produced and stored in a syringe for experiments. Due care must be exercised to prevent the mixture from unintentionally exploding. **Keep the syringe capped when not discharging gas!** Explosions are caused by (1) the syringe cap not in place and (2) a source of igition such as sparks, flames, or static electricity. This will result in the plunger being discharged from the barrel with considerable force. Discharge remaining gas into the room when finished with this experiment.

Suitability

middle school lab, high school lab, university lab, and classroom demonstration

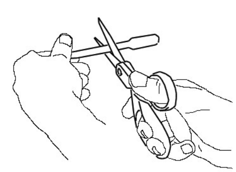
Applications, Topics, Purpose

combustion reactions, kinetics, stoichiometry of reactions, activation energy, explosive mixtures, rocketry, types of chemical reactions

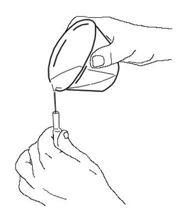
Instructions

Prepare a mixture of O_2 and H_2 via the method of syringe-to-syringe transfer as described in the previous experiment. The procedure for filling rockets with gas is:

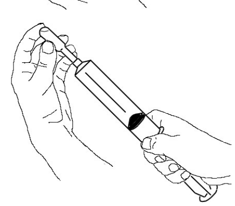
1. Cut the end off of a pipet leaving at about 2 cm of stem attached to the bulb.



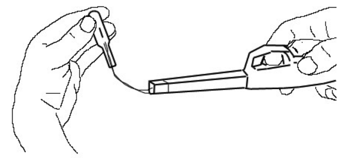
2. Completely fill the “rocket” with water.



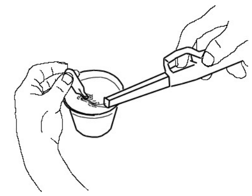
3. Slip the water-filled “pipet rocket” into the syringe fitting. Slowly displace the water with the hydrogen-oxygen mixture. The water will dribble out onto the bench top or floor. Holding the syringe and bulb at a 45° angle works best.



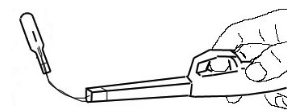
4. Slip the pipet rocket over the wire leads of the piezoelectric sparker. Remember that hydrogen is lighter-than-air! Do not tip the gas-filled rocket open-end up — the gas will escape.



5. Draw some water into the stem — without this, the rocket will not fly far when "launched". The ends of the wire leads of the piezoelectric lighter must be above the water in the gas-filled region of the rocket. If all of the water escapes, draw a little more up from a cup or beaker of water.



6. With some water in the stem, launch the rocket by triggering a spark. DO NOT aim the rocket at anyone! If the water leaks out of the stem while positioning the rocket over the wires, immediately fill the stem again by holding the wires and rocket in a cup of water and drawing a very small amount of water into the stem. The rocket should fly up to 10 m!



Teaching tips

1. Use *large* bulb plastic pipets for the rockets.
2. Construction of the piezoelectric launcher is given in Appendix C.
3. Award prizes for the rockets traveling the greatest distances.
4. One objective of this experiment is to encourage students to try various mixtures of hydrogen and oxygen. They will empirically discover that the best mixture is 2 hydrogen : 1 oxygen.
5. This reaction is used by NASA in their Saturn launch rockets.
6. The rocket idea comes from David Ehrenkrantz and John Mauch. Design for piezoelectric igniter is modified from a model developed by Bob Becker.

7. You may wish to generate a bag of hydrogen for distribution to the students in order to save time in the laboratory. See Chapter 5.

Introductory Questions

1. How far, in meters, did your rocket fly?
2. Why did you start by filling the rocket with water?
3. Which rocket would fly further: (a) a rocket filled with pure hydrogen; (b) a rocket filled with a mixture of hydrogen and air; (c) a rocket filled with a mixture of hydrogen and oxygen?

Questions

4. Why must some water be left in the stem of the rocket in order for the launch to be successful?
5. What is the reaction occurring inside the rocket?
6. Which hydrogen-oxygen rocket is expected to fly farther, a rocket that is mostly filled with oxygen and some hydrogen or one mostly filled with hydrogen and some oxygen?
7. Would rockets filled with hydrogen and air fly at all?

Advanced Questions

8. What ratio of hydrogen to oxygen is optimal? Use a balanced chemical equation to answer this question.
9. Real rockets such as the NASA's Saturn launch vehicles use liquefied gaseous hydrocarbons and liquid oxygen for the rocket's first stage and liquid hydrogen and liquid oxygen for the second stage. What sort of design feature would keep liquid hydrogen and liquid oxygen from reacting until they are supposed to?

Clean-up and storage.

At the end of the experiments, clean the syringe parts, caps and tubing with water. Rinse all parts with distilled water if available. Be careful with the small parts because they can easily be lost down the drain. **Important:** Store plunger out of barrel unless both are completely dry.

PART 2. DEMONSTRATIONS AND ADVANCED EXPERIMENTS WITH OXYGEN

EXPERIMENT 5. STEEL WOOL BURNS IN OXYGEN

Equipment

Microscale Gas Chemistry Kit
Large test tube, 25 x 200 mm
Tweezers or metal hemostat or pinch-nosed pliers
Lighter or match

Chemicals

$O_2(g)$, 50 mL
Steel wool, ‘000’ grade

Suitability

high school lab, university lab, and classroom demonstration

Applications, Topics, Purpose

Corrosion, oxidation, rust, household chemicals, combustion

Instructions

Transfer oxygen to the large test tube using the long piece of tubing so that the gas can be discharged near the bottom in order to displace the air. Grip a small ball of '000' grade steel wool with a tweezers/hemostat/pliers. Light the steel wool with a match and immediately plunge the glowing steel wool into the test tube of oxygen. The steel wool will burn with a bright light.

Long-term Variant

Generate O_2 as before. Wash twice. Place a small ball of '000'-grade steel wool (cleaned with alcohol to remove oil film) in a vial cap and float the cap in a plastic cup (250 mL) half-filled with water. Remove the plunger from the O_2-filled syringe and place it over the floating cap. Clamp the syringe in this position. The reaction is extremely slow. Within a week the water level (and the cap) will start to rise within the syringe as the iron reacts with the O_2.

Teaching tips

1. Steel wool is iron and it is a reactant with the oxygen.
2. This experiment demonstrates that metals can burn and undergo rapid oxidation.
3. The experiment can be used when discussing types of chemical reactions. Combustion reactions are all oxidation reactions.

Questions

1 What would happen if you used coarse steel wool instead of the fine steel wool?

2. Assuming that the product is Fe_2O_3, write the balanced chemical equation for the reaction.

EXPERIMENT 6. THE BLUE BOTTLE EXPERIMENT

Equipment

Microscale Gas Chemistry Kit

Chemicals

$O_2(g)$, 50 mL
KOH(s), one pellet
Dextrose, 0.9 g
methylene blue, 1 drop

Suitability

classroom demonstration

Applications, Topics, Purpose

LeChatelier's principle, oxidation/reduction, equilibrium, limiting reagents. This demonstration is often used to show how a reaction at equilibrium can be disturbed and will return to equilibrium.

Instructions

Prepare a solution of 0.01 g KOH (one pellet) and 0.9 g dextrose in 50 mL water. Add 1 drop methylene blue. Draw 25 mL of the solution into the syringe. Draw another 25 mL of the solution into another syringe — this one containing air — for use as a control. Apply a single shake to the solution whenever the color fades. Another shake will return the blue color. This process can be repeated dozens of times and will last for at least one day.

Teaching tips

1. This demonstration can be scaled up everything by 10 times and performed as a demonstration in a 500 mL flask enriched with one syringe of oxygen.
2. This experiment is based on one published by J. A. Campbell, ***Journal of Chemical Education***, **40,** 578 (1963). A full explanation is given in this reference
3. Review LeChatelier's principle. Adding $O_2(g)$ to the solution by shaking the syringe results in the position of the equilibrium to shift to the right and the blue color of the solution is seen. Shaking the syringe causes more $O_2(g)$ to dissolve.
4. This makes a good demonstration when discussing natural waters, biological oxygen demand, water stagnation, etc.
5. This reaction destroys the syringe by discoloring it. Use an old syringe.

Questions

1. Does the concentration of dissolved oxygen, $O_2(g)$, increase or decrease when you shake the syringe?
2. When the solution in the syringe becomes colorless, what must have happened to the amount of $O_2(aq)$?

EXPERIMENT 7. OXYGEN MAKES THE FLAME HOTTER

Equipment

Microscale Gas Chemistry Kit
Bunsen burner, small
Glass Pasteur pipet

Chemicals

$O_2(g)$, 60 mL
(or a bag of oxygen; See Chapter 5)

Suitability

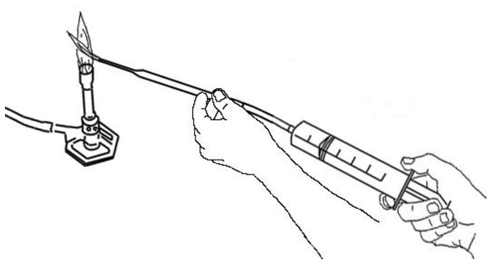
high school lab, university lab, and classroom demonstration

Applications, Topics, Purpose

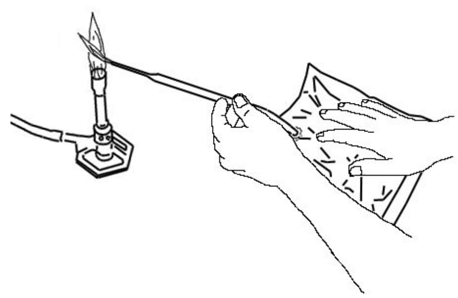
Role of oxygen in combustion, torches, temperature of flames

Instructions

Generate either (a) a syringe filled with O_2 using the general method and wash the O_2-filled syringe, or (b) a gas bag filled with O_2 using the gas bag method described Chapter 5.



Option A. Use of an O_2–filled syringe



Option B. Use of an O_2–filled gas bag

Option A. Connect the O_2-filled syringe to a glass pipet with a 15 cm piece of tubing (the tubing forms a snug fit inside the pipet). **Option B.** Connect the tubing from the gas bag (Chapter 5) to the glass pipet. Light a small Bunsen burner. Position the other end of the pipet *near* (but not in) the burner flame as shown in figures. Slowly discharge the oxygen into the flame. A very hot, intensely blue flame will be produced.

Teaching tips

1. Flames have hot and cool regions. The inner cone is a cooler endothermic region where gas molecules are broken into fragments. There is relatively little air in the inner cone. One can demonstrate the relative coolness of the inner cone of a flame by sticking a pin through a match near the head of a match and then balancing the pin over the opening of the Bunsen burner. Center the match in the barrel of the burner. Light the burner and the match will remain unlit while the flame of the Bunsen burner burns around it.
2. When oxygen is introduced discharged into the flame, the hot gas immediately reacts in a very exothermic process giving a bright blue flame.

3. Try the reaction in a darkened room.

Questions

1. What are the colors of the various parts of a flame? What color is associated with the hottest region?
2. What real-world applications take advantage of oxygen's ability to increase the temperature of a flame?
3. Balance the chemical reaction that takes place when methane burns in oxygen. How might you confirm that water is a product? How might you confirm that carbon dioxide is a product?

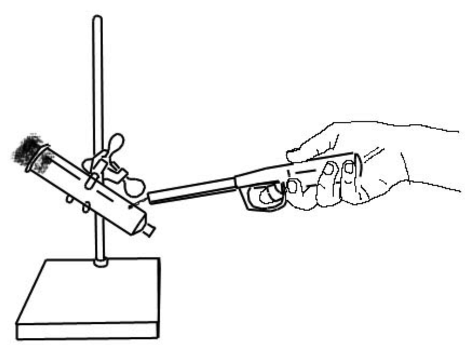
Advanced Question

4. In the inner part of the flame, fuel molecules are broken into fragments due to the abundant energy nearby. If the fuel were methane, list the two most common fragments expected assuming that in most cases, only one bond breaks per methane.

HYDROGEN BURNS WITH A "ROAR" IN OXYGEN.

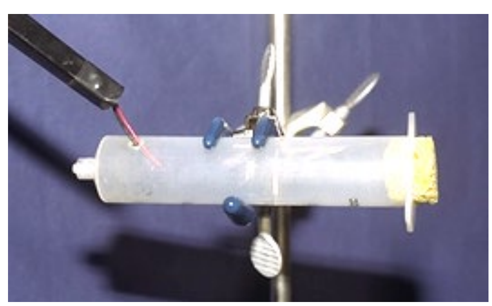
This impressive classroom demonstration, related to Experiment 7, is described in Chapter 5 (Gas Bags).

EXPERIMENT 8. MINI-SPONGE SHOOTER



Equipment

Microscale Gas Chemistry Kit
sponge
scissors
electric drill
electricians tape or equivalent
piezoelectric lighter (Appendix C)
ring stand and clamp



Chemicals

$O_2(g)$, 60 mL
Methanol or ethanol

Suitability

classroom demonstration

Applications, Topics, Purpose

Explosive mixtures, rocketry, activation energy, flash point

Instructions

Begin by cutting a "projectile" from a 1.5 cm thick sponge. The projectile should be round and disk-shaped with a diameter about 20% larger than that of the syringe. Moisten the sponge and see if it fits snugly into the syringe barrel as shown in the figure. Remove the sponge and set it aside. Use an electric drill to make a 5 mm diameter hole through the syringe barrel near the 10 mL mark (or melt a hole with a hot nail). Remove any traces of the burr. Tape over the hole by strapping electrician's tape all the way around the syringe.

Insert the plunger. Transfer O_2 and to the syringe. Next, draw up 2 – 3 mL methanol or ethanol. Shake the syringe to vaporize some of the alcohol. Note: The syringe must be at room temperature or slightly above; the vapor pressure of alcohol is much lower at low temperatures.

Drain the excess alcohol, remove the plunger and replace it with the moistened sponge. Clamp the syringe in position. Remove the electrician's tape and hold a piezoelectric lighter into the hole in the syringe and pull the trigger. The sponge will fly 5 m or more.

Teaching tips

1. Provide the balanced equation that occurs between methanol (or ethanol) and oxygen.
2. Explain the role of vapor pressure and temperature
3. Flammable liquids have flash points, the temperature at which they explode in air with a spark. In pure oxygen, the flash point is much lower and explosive mixtures are a much bigger problem.

Questions

1. Why is it necessary for the liquid to evaporate before the mixture is “explosive”?
2. Do liquids or gases react more explosively with air?
3. Would the reaction work better at higher or lower temperatures? Would it be possible that at some temperature at which the explosion does not take place?

EXPERIMENT 9. CHEMILUMINESCENCE

Equipment

Microscale Gas Chemistry Kit

Chemicals

O_2(g), 60 mL

KOH pellets, 4 g

Luminol, 0.1 g

DMSO (dimethylsulfoxide), 10 mL

Suitability

classroom demonstration

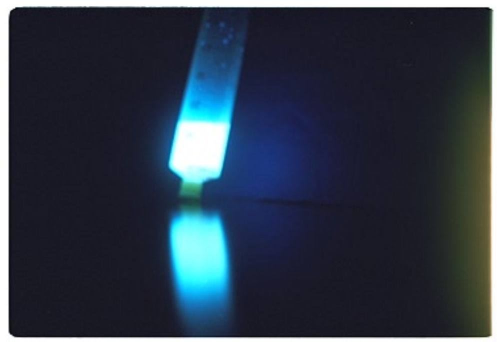
Applications, Topics, Purpose

forms of energy, chemiluminescence

Precaution

Read the safety information available for DMSO, dimethylsulfoxide! Wear gloves. Avoid dermal contact.

Instructions



Measure out 4 g KOH pellets and 0.1 g luminol and set them aside for use later. You will also need 10 mL DMSO. Generate oxygen and wash the gas twice. Remove the plunger, dump out the vial cap, add the solid KOH and luminol through the syringe mouth and reinsert the plunger to its previous mark (about 60 mL.) Because O_2 has a similar density to that of air, O_2 loss is not excessive if you work quickly and replace the plunger as soon as possible. Remove the syringe cap and draw 10 mL of DMSO into the syringe and 2 - 5 mL H_2O. Darken the room. Shake the solution and it will emit a blue chemiluminescent glow which will last for a very long time, depending on the amount of shaking. We have had the system last 48 hours.

Clean-up: Do not reuse this syringe. DMSO must be disposed of properly. One effective method that we have used is to remove the plunger and dump in 30 mL of absorbent cat litter, reinsert the plunger, remove the excess air, and recap. Place capped syringe containing dmso and cat litter in a sealed food storage bag and place in the trash. Check state regulations.

Teaching tips

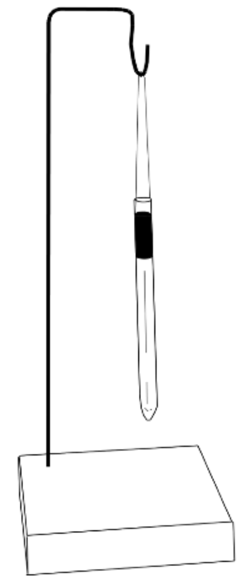
1. Relate the demonstration to light sticks.

Questions

1. What are the three ways in which a chemical reaction can give off energy?
2. What are other examples of chemiluminescence?
3. Why does shaking the syringe make the light brighter?
4. Why does the reaction eventually stop?

EXPERIMENT 10. PARAMAGNETISM OF OXYGEN

Equipment



glass Pasteur pipet
20 cm length of sewing thread
25 cm length of heavy (e.g. 12 gauge) copper wire
3 cm electricians' tape
wooden base (approximately 10 cm x 10 cm x 2 cm.)
Latex tubing, 1/8-inch (3.175 mm) ID, 20-cm length
pinch clamp such as a hemostat
Neodymium magnet
plastic beverage cup, clear PETE (9 oz, 250 mL)
freezer-quality quart (1 L) size food storage bag
Dewar flask

Chemicals

O_2(g), 1 L
Liquid nitrogen, N_2(l), 500 mL

Suitability

classroom demonstration

Applications, Topics, Purpose

paramagnetism, molecular orbital theory

Precautions

Always wear safety glasses! Liquefied gases must never be stored in a closed system. Note: None of the equipment described in this article would create such a situation. Liquefied gases cause cryoscopic burns. Avoid dermal contact. Liquid oxygen can react violently with organic matter. Do not use oxygen near open flames or where sparks are generated.

Background information

Many teachers have seen the impressive photograph of liquid oxygen suspended between the poles of a large magnet. The photograph is commonly found in general chemistry books. In conjunction with a discussion of how molecular orbital theory correctly predicts oxygen's paramagnetism, those of us who make use of classroom demonstrations are left with relatively few options to show the class an actual example.

Apparatus

The apparatus shown in the figure is constructed from a glass Pasteur pipet, a 20 cm length of sewing thread, a 25 cm length of heavy (e.g. 12 gauge) copper wire, 3 cm electricians' tape, and a wooden base (approximately 10 cm x 10 cm x 2 cm.) Drill a suitable diameter hole in the base to support the copper wire. Bend the copper wire into the shape shown in the figure. Use a Bunsen burner to seal the bottom of a glass pipet at the point there the diameter changes between the tip and the body. This will create a small test tube with a capacity of approximately 2.5 mL. Tape both ends of a 15 cm length of sewing thread at opposite sides of the pipet's open end to form a pail-like handle. The loop of thread will hang from the support hook. The vessel must be suspended above the base.

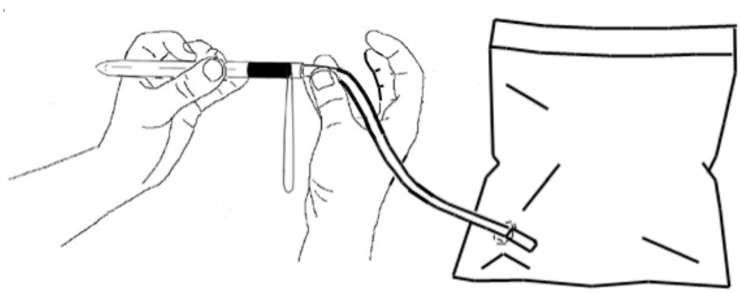
Gas bag of oxygen

A 1-L freezer-quality plastic, sealable, food storage bag is used. Instructions for construction of this gas bag are given in Chapter 5.

Fill a gas bag with oxygen from a compressed gas cylinder as follows. Run some oxygen out of the tank order to purge the regulator of air. The regulator should have a fine-control-knob. Adjust the pressure to 100 kPa (1 atm, 15 psi) using the gas regulator knob. Connect the syringe/tubing syringe (plunger fully inserted) to the regulator and ***slowly*** fill the syringe with oxygen. **Caution:** Do not over-inflate! If compressed oxygen is not available, you may make oxygen gas in a gas bag according to instructions given in Chapter 5.

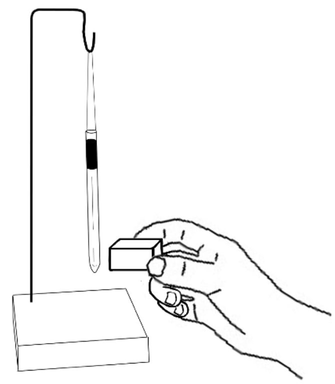
Instructions for each demonstration

Remove the pipet/thread device from the apparatus and connect the pipet to the bag of oxygen using the Latex tubing as shown below. Note: The tubing makes a snug fit *inside* the opening of the open end of the glass pipet; do not attempt to stretch it over the pipet.



Pour liquid nitrogen into the plastic cup. Hold the bottom third of the pipet in the liquid nitrogen. Within a few seconds the gas bag will start to deflate as the oxygen gas condenses in the pipet. After the oxygen has condensed to a depth of 1 – 2 cm or more, one has a unique opportunity to show the class the pale blue color of liquid oxygen: Do not remove the pipet from the liquid nitrogen as it protects the outside of the pipet from becoming covered with frost.

After the students have seen the blue color, proceed as follows. Remove the gas bag tubing from the pipet and immediately suspend the pipet from the wire hook of the apparatus. Quell any random movement that the pipet may be making by touching a finger to the pipet above the cold region. The liquid oxygen is initially about 1 – 2 cm deep inside the pipet but vaporizes rapidly. Some liquid should remain present for at least 30 s, so the demonstration must be completed quickly. Hold the neodymium magnet near the side and bottom of the pipet. Once the magnet is within a few mm of the pipet, it will swing towards the magnet and possibly hit it with a small, but audible ping.



Clean-up: Return unused liquid nitrogen to the Dewar flask. Allow liquid oxygen to evaporate.

Teaching tips

1. This is a small scale demonstration. Using it in a large classroom will require some sort of projection. We created a pipet vessel with a longer string (90 – 100 cm) that we hung from the lens head of an Elmo® document camera. This projected well while providing a "top view" of the pipet. Motion caused by the magnet was easily observed by the class. Using this approach, one does not need the wire support shown in the figure.
2. Bob Becker has a simpler version of this demo using soap bubbles and a neodymium magnet.

Questions

1. Describe the color of liquid oxygen.
2. Describe how the pipet behaved when the magnet was brought close.
3. Sketch the Lewis dot structure for oxygen, O_2. Does this drawing suggest that the compound is paramagnetic or diamagnetic? This "flaw" to the Lewis dot structure model is serious, but not fatal. The method is so simple and so powerful, that it is best to use it, always knowing that it sometimes (although exceedingly rarely) makes mistakes in prediction.
4. Sketch the molecular orbital diagram for O_2. Does this drawing suggest that the compound is paramagnetic or diamagnetic?

Summary of Materials and Chemicals Needed for Chapter 4. Experiments with Oxygen.

Equipment required for Part 1: Student Experiments (Experiments 1 – 4)

Item	For Demo	For 5 pairs	For 10 pairs
Microscale Gas Kit (See Chapter 1)	1	5	10
candles (included in kits)	1	5	10
glass rod	1	5	10
matches	1	5	10
piezoelectric sparker	1	2 - 3	3 - 5
test tube, (25 x 200 mm)	1	5	10
wide-stem disposable pipet	1	5	10
wooden splint	1	5	10

Materials required for Part 1: Student Experiments (Experiments 1 – 4)

Item	For Demo	For 5 pairs	For 10 pairs
tape	2 cm	10 cm	20 cm

Chemicals required for Part 1: Student Experiments (Experiments 1 – 4)

Item	For Demo	For 5 pairs	For 10 pairs
potassium iodide, KI, powder	1 g	5 g	10 g
6% H_2O_2(aq)*	20 mL	100 mL	200 mL
3% dish soap solution**	10 mL	50 mL	100 mL
Limewater, clear	10 mL	50 mL	100 mL
magnesium powder, turnings or ribbon	1 g	2 g	3 g
2 M HCl(aq)	10 mL	50 mL	100 mL

* 3% H_2O_2(aq) will also work, but not quite as well — the reaction will be slower.

** 3% v/v dish soap solution (3 g dish soap + 97 g distilled water)

Equipment required for Part 2: Advanced Experiments and Demonstrations (Experiments 5 – 10)

Item	For Demo
Microscale Gas Kit (See Chapter 1)	1
Bunsen burner, small	1
glass Pasteur pipet	2
large test tube, 25 x 200 mm	1
lighter or match	1
piezoelectric lighter	1
ring stand and clamp	1
Latex tubing, 1/8-inch (3.175 mm) ID	20-cm
pinch clamp such as a hemostat	1
Neodymium magnet	1
Dewar flask	1

Materials required for Part 2: Advanced Experiments and Demonstrations (Experiments 5 – 10)

Item	For Demo
electric drill	1
electricians tape or equivalent	10 cm
scissors	1
sponge	1
tweezers or hemostat or pinch-nosed pliers	1
steel wool, ‘000’ grade	1 pad
freezer-quality quart (1 L) food storage bag	1
sewing thread	20 cm
heavy (e.g. 12 gauge) copper wire	25 cm
wooden base, approx. 10 cm x 10 cm x 2 cm	1
plastic beverage cup, clear, 250 mL	1

Chemicals required for Part 2: Advanced Experiments and Demonstrations (Experiments 5 – 9)

Item	For Demo
potassium iodide, KI, powder	1 g
6% $H_2O_2(aq)$*	50 mL
dextrose	< 1 g
DMSO (dimethylsulfoxide)	10 mL
potassium hydroxide, KOH	4 g
luminol	< 1 g
methanol or ethanol	3 mL
methylene blue	1 drop
$O_2(g)$	1 L
Liquid nitrogen, $N_2(l)$	250 mL

* 3% $H_2O_2(aq)$ will also work, but not quite as well — the reaction will be slower.

