Instructor's Guide - Investigation 1

PREPARATION AND PROPERTIES OF GASES IN AIR

BRIEF DESCRIPTION AND OBJECTIVES

This investigation is designed for the first few days of the course. Students generate samples of carbon dioxide and oxygen and carry out a few tests on each. The investigation ties to the opening discussion at the beginning of Chapter 1. It is relatively simple and fast and teaches some basic skills about manipulating chemicals in a laboratory. Students encounter several chemical reactions and are asked to make observations about the reactions. No numerical data or calculations are involved.

The approach used is somewhat novel in that the reactions are carried out in disposable plastic "zipper" bags of the type used for food storage. This has several advantages: (1) The novelty grabs student interest, (2) everything is contained and disposal is easy, (3) it is easy to see what is happening and students can quite literally get a feeling for the volume of gas produced and temperature changes, and (4) it is relatively easy to collect small samples of gas for tests.

Some instructors have used a modified version of this investigation in a lecture room. To do this, the bags must be assembled in advance. It may not be feasible for students to do all of the tests, but the reactions can be observed. The instructor can demonstrate some of the tests.

ESTIMATED TIME REQUIRED: 1 hour

MATERIALS NEEDED FOR 10 PAIRS OF STUDENTS

(*All investigations in this lab manual assume that students will work in pairs.)*

- 30 pint-size **heavy duty** freezer-type plastic "zipper" bags.
  - Any of the several brands of air-tight reclosable plastic bags can be used. They do **not** need to be the new style with a pull-type zipper handle (and in fact the new type bags are less satisfactory for sampling the gases). They are found in most supermarkets. Whatever bags are used, they must be the heavy-duty type (often labeled as freezer storage bags). The regular bags are too thin and can melt from the exothermic reaction when oxygen is generated.
  - If pint-size heavy duty bags are not available, quart-size bags can be used, but some adjustment in reaction quantities may be needed. See Note 3 below.
- 100 plastic transfer pipets, ca. 5 mL. Thin-stem style is preferred, but you can use whatever kind is on hand.
- 10 24-well wellplates OR 50 10x125mm test tubes and 10 test tube racks (See Note 2)
- Wood splints or a box of wood toothpicks to use for wood splints
- Roll of ¾ inch cellophane tape to protect the limewater from carbon dioxide in the air
- 20 plastic teaspoons
- 10 pieces of black paper that are cut to ~ 10 cm x 10 cm square
- Matches

Solid Chemicals Needed

- 40 grams of sodium bicarbonate ($NaHCO_3$)
- 5 grams of potassium iodide (KI)
- 100 mg bromthymol blue (to prepare the indicator solution)
- 3-4 grams calcium oxide, CaO (to prepare limewater)

Solutions Needed

- 150 ml fresh 10% H_2O_2.
- 300 ml household vinegar (4-8% acetic acid)
- Saturated $Ca(OH)_2$ solution
- Bromthymol blue reagent

PREPARATION OF SOLUTIONS

10% Hydrogen peroxide

Add 50 mL of 30% hydrogen peroxide to 100 mL of distilled or deionized water. An exact concentration is not necessary, but it should be *at least* 8%. It is important to test the solution before use. See Notes 6 and 8. As a last resort, drugstore-variety 3% hydrogen peroxide can be used, but it is generally not very satisfactory. If 3% solution is used, it will be necessary for students to use at least 3 filled pipets to generate enough oxygen gas.

Vinegar

Vinegar can be used as received. The exact concentration of acetic acid is not critical.

Saturated Calcium Hydroxide (limewater)

This can be prepared from either reagent grade calcium oxide (CaO) or calcium hydroxide ($Ca(OH)_2$). (Horticultural lime can also be used if it is fresh.) See Note 2 in the Trouble Shooting section about old bottles of calcium oxide or calcium hydroxide. Shake 3-4 grams of solid with 100-200mL of distilled water, then filter to give a clear solution. Check to be sure that the filtered solution gives a good CO_2 test by placing a few mL in a small test tube and blowing exhaled air through it. A definite milkiness should be observed.

Bromthymol Blue Reagent

> *(Caution: This is a common indicator and may be available already made up. However, it will be much too concentrated and may not be adjusted to the optimal pH for this investigation. If you wish to use existing indicator solution, you will have to have to adjust the concentration by trial and error and then adjust the pH.)*

First, prepare a stock solution of 0.1% bromthymol blue in 50% ethanol. Dilute the stock solution approximately 30-fold with distilled water (e.g.. 3 mL in 90 mL of water). The resulting solution should be a transparent bright blue after *carefully* adjusting the pH. If the solution is yellow or green, add 1 drop of 0.10 M sodium hydroxide and mix the solution well. Continue adding the sodium hydroxide one drop at a time with mixing until the solution just turns blue and the color does not change to green on standing. Because it is easy to add too much sodium hydroxide, the solution should be tested to see that it works in the investigation. To do this, place about 1 ml of the reagent in a pint "zipper" bag. Breathe in and out 2 or 3 times and then seal the bag when it is inflated with your breath. Shake the bag. The indicator should turn yellow. If it remains blue, adjustments can be made to the indicator with very dilute acid. Finally, seal the bottle. The solution is stable, but the color may gradually fade to green or yellow as carbon dioxide from the air slowly dissolves in the solution. The indicator should be checked before and during each laboratory class period to see if it still works. Adjustments can be made with very dilute sodium hydroxide. Dispense in small plastic bottles.

NOTES

1. This investigation introduces students to the use of small disposable plastic transfer pipets. These pipets have become very popular in "microscale" lab work and are used extensively throughout this Laboratory Manual. They are sometimes called Beral pipets. Several sizes are available and instructors have some latitude in choice of size. The size to be used will depend, in part, on what you have available and/or how many sizes you wish to stock. The suggested size for this investigation is the 4 mL thin stem type, which is one of the most common types. Many chemistry departments now stock only the 4-mL graduated-stem variety, which is advantageous for some later investigations. Larger 15 mL ("jumbo jumbo") pipets have the obvious advantage of holding more of the reagent and being able to sample larger volumes of gas for testing. In that case, solution concentrations must be adjusted downward.
2. This investigation also introduces the 24-well wellplates that are used for a number of investigations in this Lab Manual. Some instructors prefer to do the tests in small test tubes. Either approach works well.
3. **Size of plastic bags.** The preparation of gases is best done in pint-size zippered bags, although these have been harder to find in recent years. Inquiry to the Ziploc company revealed that pint-size bags are still being manufactured, although many stores choose not to stock them. Best sources are WalMart, K-Mart and Target. If quart-size zip-lock bags are used, it may be desirable to generate larger quantities of gases. Alternatively, instructions can be given on how to roll up the bag from the bottom so that most of the gas is near the zipper area. This facilitates collecting samples of gas in pipets.
4. The success of this investigation depends on being able to rapidly and reproducibly generate sufficient volumes of two gases so that several sub-samples can be removed for testing. It is strongly recommended that the

reactions be checked in advance with the particular reagents, bags, and pipets that students will use. Adjustments in volumes or concentrations can be made as needed.

5. There is a special technique to filling plastic transfer pipets with liquids so that they are nearly full. *This should be demonstrated to students.* Squeeze the pipet as completely as possible, insert the tip into the desired liquid and release. If the pipet is the thin-stem variety, the stem can be bent into the liquid while holding the bulb end down and the bulb squeezed a second time to expel more air.

6. **Volumes of generated gas.** For carbon dioxide, the amount of vinegar used should generate enough gas to slightly *under-inflate* a pint-size bag and avoid the possibility of bag rupture. If a bag is over inflated, the zip seal usually opens with a loud pop. NOTE: If the amount of generated carbon dioxide is not enough, students may need to use larger amounts of vinegar and sodium bicarbonate. For oxygen, the challenge is to produce enough gas without increasing the safety problems of more-concentrated hydrogen peroxide and excessive heat build-up. Stoichiometric calculations give the following theoretical volumes of gas at room temperature, based on the liquid reagents. For CO_2: 8 mL (2 pipets-full) of vinegar should produce 150-200 mL of CO_2, which should be satisfactory in a 500 mL (1-pint) bag. For O_2: 8 mL (2 pipets-full) of 10% H_2O_2 should produce about 280 mL of O_2, which is satisfactory in a 1-pint (500 mL) bag.

7. **Catalyst for O_2 generation.** Oxygen is generated by catalytic decomposition of hydrogen peroxide. Several catalysts can be used for this purpose. The first edition of this Lab Manual used $FeCl_3$, but the reaction is extremely exothermic with a consequent risk of melting the plastic bag and causing burns. Yeast was the recommended catalyst for the 2nd and 3rd editions -- it works well and presents no disposal problem, but creates quite a mess in the bags. (About 1/2 tsp of dry yeast will work, *providing that it is not past the expiration date*.) We are now recommending KI – only a small pinch of solid is needed. Other catalysts are possible, and some instructors may prefer to use their own favorite type.

8. **Hydrogen peroxide.** Solutions of hydrogen peroxide are unstable and will slowly decompose unless kept cold and in the dark. This investigation calls for 10% H_2O_2, which is normally prepared by dilution of 30% stock solution. If there is any uncertainty about the age or concentration of the hydrogen peroxide, the diluted solution should be tested to be sure that one pipet will produce a good volume of gas under the conditions of this investigation. The dilution can be adjusted as needed. Drugstore-variety 3% hydrogen peroxide can also be used, but it will be necessary to use *several* pipets of 3% solution to produce enough oxygen. CAUTIONS: (a) Concentrations of H_2O_2 greater than 10% should not be used because the reaction may generate enough heat to soften or melt the plastic bag, and (b) concentrated hydrogen peroxide solutions can cause skin burns. Tests indicate that 10% H_2O_2 should not cause a problem, but, if in doubt, the concentration can be reduced somewhat.

9. **Acid-base Indicator.** Bromthymol blue is used in the current edition rather than the phenol red that was used in earlier editions. There are several modest advantages: it is more readily available, it is less susceptible to atmospheric CO_2, and it gives 3 colors rather than 2. The pH transition region for bromthymol blue is 6.0 to 7.6. It is blue at 7.6, yellow at pH 6.0, and green at intermediate pH. In dilute solution it can be used to detect CO_2, based on the formation of carbonic acid when CO_2 is mixed with water.

10. Many students do not understand the difference between supporting combustion and flammability. They think oxygen is flammable.

11 For filling a bag with exhaled air, some instructors prefer to give students a clean straw.

12. Because of the problem of generating enough oxygen without using excessively concentrated hydrogen peroxide, some instructors have students generate oxygen as specified but then give them baggies filled with oxygen from a compressed gas cylinder for the subsequent tests.

13. Students need to be alerted to use care in sampling gases from the baggies to be sure that the pipet does not touch the liquids or solids inside the bag.

TROUBLESHOOTING

1. If not enough O_2 gas is generated to nearly fill the bags:

   Possible causes: old hydrogen peroxide that has partially decomposed or incorrect concentration of hydrogen peroxide.

2. If not enough CO_2 gas is generated to nearly fill the bags:

   If this occurs, the quantities of vinegar (and possibly sodium bicarbonate) should be increased.

2. If the $CaCO_3$ precipitate is not visible with the limewater test:

   The amount of precipitate is small, but it should be seen easily when the solution is viewed against a black background. The most probable source of difficulty is a bad limewater solution, either because it is not saturated or because it was made with an old bottle of calcium hydroxide that had converted to calcium carbonate. (An alternate procedure that works well is to use small test tubes for the limewater. A full pipet of CO_2 bubbled slowly into the test tube should produce a quite milky mixture.) NOTE: 0.1 M $Ba(OH)_2$ is reported to work better than lime water.

3. If only 3 % hydrogen peroxide is available:

   Use at least 3 full pipets of hydrogen peroxide in each bag.

4. If wellplates are not available:

   Use small test tubes (10x125 mm).

5. If bromthymol blue is not available:

   Slightly pink phenolphthalein solution can be substituted, but the color is less stable due to absorption of carbon dioxide from air.

6. If the glowing splint (toothpick) does not burst into flame with oxygen:

   A quick puff of oxygen is necessary with the end of the pipet held very close to the ember (1-2 mm away). One student should hold the splint and one student puff the oxygen at the ember. A number of users have suggested that the splint be thrust into a bag of oxygen. This is not recommended because it often sets the bag on fire. An alternative to the glowing splint or toothpick is simply a match after it has been blown out. Remind students that they can't resqueeze the pipet since it will fill with room air. If the first pipet of gas gives an inconclusive result, they must refill the pipet from the baggie.

WASTE DISPOSAL:

The contents of all of the bags can be safely flushed down the drain and the bags placed in trash containers. The plastic transfer pipets can be rinsed with water and discarded or saved. The wellplates should be washed and saved for future investigations. *Do not rinse the wellplates with acetone.*

SAMPLE STUDENT DATA

1. Observations regarding the generation of carbon dioxide.

   gas is evolved, bubbles form, foam forms, the bag gets cold and inflates

2. Observations regarding the generation of oxygen.

   bubbles form, foamforms, gas is evolved, the bag gets hot and inflates, rapid bubbling occurs

3. Reactions with **limewater** ($Ca(OH)_2$ solution).

Gas	Observations
Carbon dioxide	*Solution turns cloudy [milky] white*
Oxygen	*No change*
Exhaled air	*Solution turns slightly cloudy white*
Air	*No change*

4. Reactions with **bromthymol blue acid-base indicator**.

Gas	Color of BTB	Is the solution acidic?
Carbon dioxide	*yellow*	*yes*
Oxygen	*blue*	*no*
Exhaled air	*yellow*	*yes*
Air	*blue*	*no*

5. Observations with a glowing splint.

Gas	Observations
Carbon dioxide	*Glow goes out*
Oxygen	*Splint bursts into flame*
Exhaled air	*No change; splint glows then gradually goes out*
Normal air	*No change; splint glows then gradually goes out*

SUGGESTED ANSWERS TO QUESTIONS

Reflecting on the Investigation

1. According to Chapter 6 of *Chemistry in Context*, the oceans absorb 25-40% of all anthropogenic CO_2 emissions.

   a. Based on your observations in this investigation, what effect will this have on the pH of the oceans?

   It should lower the pH, making the oceans less basic (or more acidic).

   b. List two consequences of ocean acidification. (Hint: See Chapter 6 of your textbook.)

   Thinning of shells of sea creatures, damage to coral reefs.

2. Based on what you observed about the interaction of carbon dioxide with the glowing splint, explain how CO_2 fire extinguishers work.

   Carbon dioxide doesn't support combustion so that when a burning object is surrounded by carbon dioxide the flame goes out.

3. Based on what you observed about the interaction of oxygen with the glowing splint, explain why liquid oxygen is an extremely hazardous material.

   Flammable objects burst in o flame in pure oxygen. Liquid oxygen, a concentrated source of oxygen, would cause flammable objects to burn furiously.

4. If your blood becomes too acidic, you may begin to hyperventilate, which causes your blood O_2 levels to increase, and your CO_2 levels to decrease. Write the equation for reaction of CO_2 with water, and explain why reducing CO_2 in the blood will reduce the acidity of the blood.

   $CO_2 + H_2O \rightarrow H_2CO_3$

   CO_2 reacts with water to form carbonic acid. If CO_2 levels decrease, the amount of carbonic acid in the blood decreases, thereby lowering the acidity in the blood.

5. Are carbonated beverages acidic or basic? What do you think happens to the acidity of a carbonated soft drink as it "flattens"?

   Carbonated beverages are acidic because they contain dissolved carbon dioxide. The acidity of a carbonated soft drink is reduced as it flattens because it contains less carbon dioxide.

6. What is the purpose of including ordinary air in these investigations?

   Ordinary air contains all of the gases we were testing but in lower concentrations than the specific gases we tested. Using the tests we could confirm that these gases are in air. Also, it's important to demonstrate that *what is being observed in the samples is different than what is observed with normal air – that the samples are not just air.*

7. You may have indicated that nitrogen is the most abundant gas in air. Why do you think you did not perform these investigations with pure nitrogen?

   Nitrogen is inert and would not have reacted in our tests. Also, the air sample is mostly nitrogen so testing air is similar to testing nitrogen.

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