

Microscale Gas Chemistry

In-Lab Workshop

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MICROSCALE GAS CHEMISTRY. IN-LAB WORKSHOP

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Abstract: Learn to safely and conveniently generate a variety of gases for the classroom and laboratory using simple, inexpensive equipment including 60-mL disposable syringes. Participants will make syringes filled with carbon dioxide, hydrogen, and oxygen, and will perform a series of reactions with these gases. These microscale laboratory activities are suitable for use with high school and college-level chemistry students. Detailed instructions allow for the safe generation of these gases. Participants will also prepare nitric oxide (nitrogen monoxide) and nitrogen dioxide. Such gases are not suitable for most teaching lab situations, however they can be used in numerous impressive classroom demonstrations. All gases are easily prepared without the use of a hood and with negligible risk of releasing smells into the classroom. Participants will practice and master the techniques of gas-generation using syringes and will perform a dozen different experiments using gas-filled syringes. Several spectacular demonstrations will also be presented. The workshop fee covers a gas generation kit, chemicals and print materials. Microscale Gas Chemistry is supported by a free, dedicated web site that provides complete experimental details, ordering information and more. A lab manual is available for purchase.

Here are the reasons why we are “gas enthusiasts”!

Gas Chemistry:

- ❖ ***is fun and easy!*** Students find it easy to learn how to prepare gases and do the reactions. Gas samples are ready in 5 minutes.
- ❖ ***is a source of great labs and great demos!*** Students enjoy making gases. Some of the demonstrations are nothing short of spectacular.
- ❖ ***is visual!*** The best way to *see* a gas is to watch it being produced.
- ❖ ***is microscale!*** It's microscale in terms of quantities, but large enough to see (60 mL).
- ❖ ***is inexpensive.*** It costs less than $\frac{1}{2}$ cent to prepare a syringe filled with carbon dioxide. Other gases are a bit more expensive, but never more than a few cents per syringe full of gas.
- ❖ ***is green chemistry.*** There is little or no chemical waste. Well over 90% of the experiments have *no* chemical waste that require professional waste handlers.
- ❖ ***is a valuable resource for teaching a wide variety of chemistry concepts.*** Important concepts of the high school and college chemistry curriculum can be taught with gases. Our emphasis mostly on the chemical reactions of gases, however, the list of concepts covered includes gas laws, environmental issues (acid rain, air pollution), reaction stoichiometry (limiting reagents, law of combining volume, theoretical yield), intermolecular forces, catalysis, combustion, molar mass as well as more advanced topics such as kinetics and equilibrium. Experiments involving microexplosions and rocketry are favorites among the students.

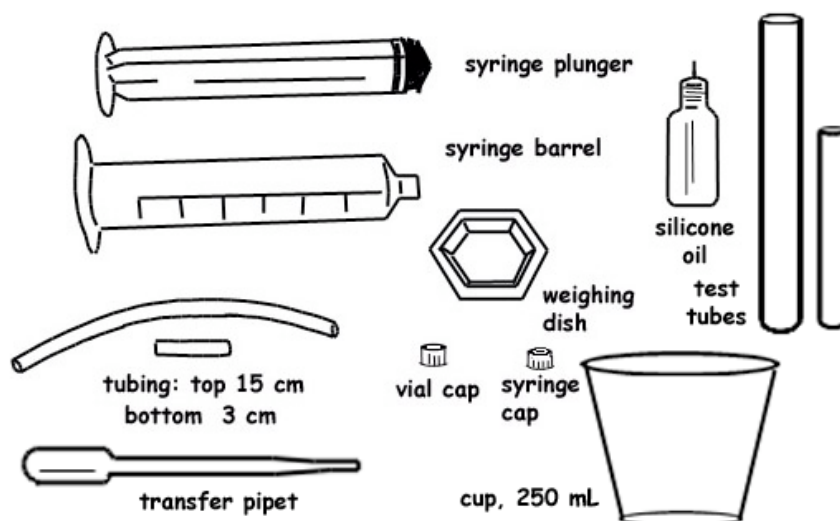
Method for Preparing Gas Samples.

The most general method for gas generation, the “In-Syringe Method” is used to prepare eleven different gases, including the six that we will use in today’s presentation: carbon dioxide, hydrogen, oxygen, nitric oxide, ethyne (acetylene) and chlorine. Originally described by Alyea, the In-Syringe Method features the generation of gases by reacting two chemicals, typically one solid and one aqueous liquid, inside a plastic syringe.

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 small bottle of silicone oil
- ❖ one plastic pipet
- ❖ one clear plastic beverage cup (250 mL/9 oz)
- ❖ two small plastic weighing dishes
- ❖ one small test tube (12 x 100 mm)
- ❖ one medium test tube (18 x 150 mm)
- ❖ one birthday candle



All of this stuff 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.

Sources, Ordering Information

- ❖ 60 mL plastic syringes with a LuerLOK fitting; Educational Innovations; GAS-140 \$2.25 for 1 - 5; \$1.95 > 5; Flinn AP8754; \$3.20; Fisher 14-823-2D; \$30.66 for 30; \$110.40 for 4 pks x 30
- ❖ Latex LuerLOK syringe caps; Educational Innovations GAS-160, ten-pack, \$1.95; Flinn AP8958; ten-pack, \$2.03
- ❖ plastic vial caps that fit within the barrel of the syringe; Educational Innovations GAS-180, ten-pack, \$2.50
- ❖ Latex tubing, 1/8-inch (3.175 mm) ID, 15 cm and 3 cm; Educational Innovations GAS-220, 5-ft, \$5.95; Flinn AP2076; 10-ft, \$5.40
- ❖ bottle of silicone oil; Educational Innovations GAS-150
- ❖ clear beverage cup, 9 oz (260 mL) grocery store

A word about syringe lubrication.

We recommend lubricating the black rubber seal of the plunger. New syringes have been treated with a thin film of silicone oil but this washes off after the first use. Educational Innovations sells small bottles of silicone oil ideal for student use and Fisher Scientific sells large bottles (500 mL) that can be used to refill small bottles. Students use only a few drops at a time. New syringes generally do not need lubrication for quite some time. If you note the plunger moves with difficulty, first try switching plungers. If that does not work, lubricating the plunger will definitely do the job.

Internet reference.

Our gas book, numerous color photographs of procedures, experiments and demonstrations, a few QuickTime movies of techniques and experiments are available on the web at our microscale gas chemistry website. Use of the site is free.

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

The In-Syringe Method for Preparing Gas Samples

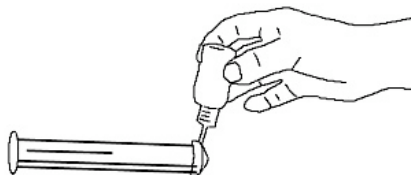
The general strategy of the method is to react two substances in a 60 mL syringe. The limiting reagent is always used in solid form and is placed in a small vial cap. The second reagent is prepared as an aqueous solution. For example, one could generate $\text{CO}_2(\text{g})$ from excess aqueous acetic acid and solid NaHCO_3 , as the limiting reagent.

Getting started: Organize equipment.

Locate and organize all of the equipment and chemicals you will need to perform the reaction. Start by “breaking-in” the syringe by moving the plunger up and down a few times.

Start by lubricating the seal.

Lubricate the black rubber seal of the plunger with silicone oil. This step is only necessary if the syringes are getting old.



The solid reagent.

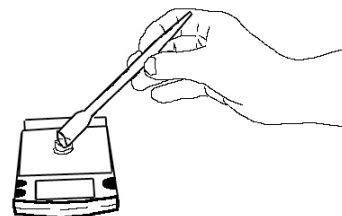
The solid reagent is placed in the vial cap that is then lowered into the syringe barrel by water flotation. In the preparation of carbon dioxide, you will use 0.21 g baking soda, NaHCO_3 , to make carbon dioxide. In subsequent chapters you will use the following quantities of solid reagents:

To make:	Use:
Carbon dioxide	0.21 g sodium bicarbonate (baking soda), $\text{NaHCO}_3(\text{s})$ (try vial cap $\frac{1}{2}$ full)
Hydrogen	0.05 g magnesium, $\text{Mg}(\text{s})$ turnings (try various numbers of turnings; about 4 turnings may be just right, for example)
Oxygen	0.10 g potassium iodide, $\text{KI}(\text{s})$ (try vial cap $\frac{1}{3}$ full)
Nitric oxide	0.25 g sodium nitrite, $\text{NaNO}_2(\text{s})$ (try vial cap $\frac{1}{3} - \frac{1}{2}$ full)

Measure out the solid reagent.

Refer to the chart above. Place the solid reagent into the vial cap. We recommend that the solid be measured directly into the vial cap to prevent losses from transferring small amounts of solids.

Hint: Cut the top off of a plastic pipet at an angle to make an inexpensive spatula.

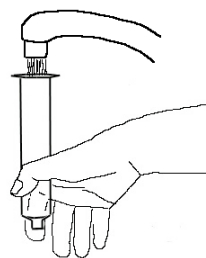


spatula made from pipet:



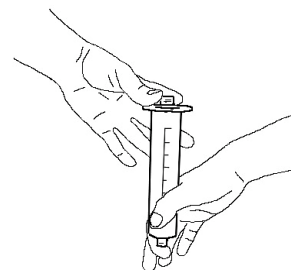
Fill the syringe barrel with water.

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



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.



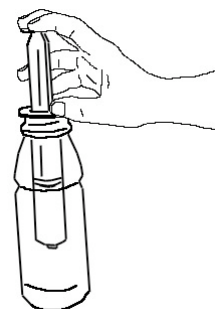
Lower the cap by flotation.

Release the seal made by finger to lower the cap into the syringe barrel without spilling its contents. Allow the syringe to drain into a wide mouth beverage container. When successfully completed, the cap should rest upright on the bottom of a syringe with all reagent still in the cap. The syringe should always be held in a vertical position during these first steps.



Install the plunger.

Install the plunger while maintaining the syringe in a vertical position. The plunger should fit snugly against the rim of the vial cap.



The aqueous reagent.

The liquid reagent is drawn into the syringe as described below. In the preparation of carbon dioxide, you will use 5 mL vinegar, $\text{HC}_2\text{H}_3\text{O}_2$, to make carbon dioxide. Later today you will use the following quantities of aqueous reagents:

To make:

Carbon dioxide
Hydrogen
Oxygen
Nitric oxide

Use: (Hint: Small weighing dish holds about 7 mL)

5 mL acetic acid (vinegar), $\text{HC}_2\text{H}_3\text{O}_2(\text{aq})$
5 mL 2 M $\text{HCl}(\text{aq})$
5 mL 6% $\text{H}_2\text{O}_2(\text{aq})$
5 mL acidic Fe^{+2} solution

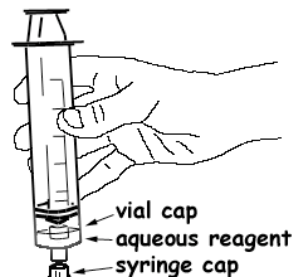
Draw aqueous reagent into syringe.

The aqueous reagent, measured into a small weighing boat, 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.



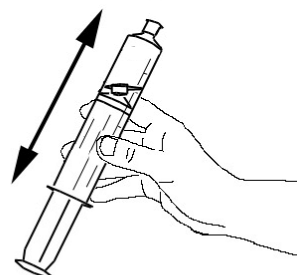
Install syringe cap.

Push the syringe cap over the syringe fitting. It simply pushes on!



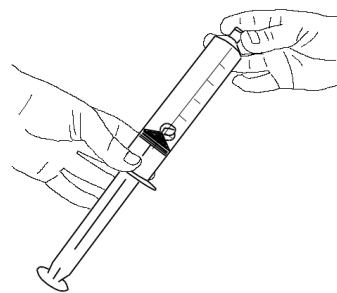
Generating the Gas.

The “fun” part is generating the gas and you are ready to do that now. 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.



Remove cap to stop the reaction.

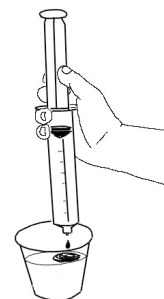
After the plunger has reached the desired mark (usually 50 mL), tip the syringe so that it is positioned with plunger downward and syringe cap upward. Carefully remove the syringe cap assuming that the syringe may be under positive pressure.



Discharge reagents

Turn the syringe 180° and discharge the liquid reagent into the plastic cup over half-filled with water to dilute the reactants.

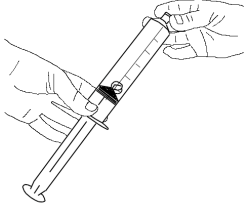
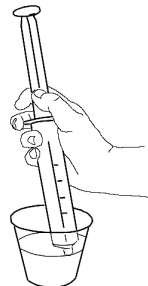
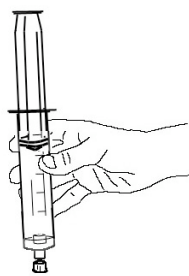
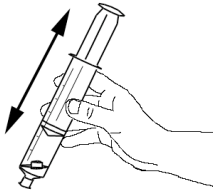
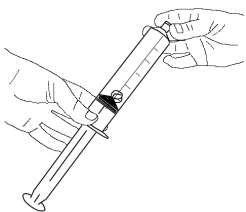
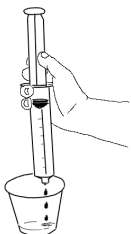
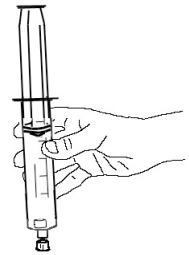
Caution: Never remove the syringe cap with the cap end of the syringe directed downward — reagents will spray out of the syringe. Immediately cap the syringe with the cap to prevent loss of gas by effusion.



Washing away contaminants.

The gas-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. (This can only be done for gases that do not dissolve readily in water.)

To wash a gas:

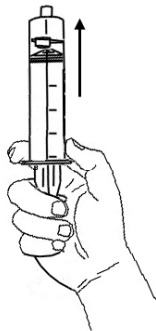
<p>1. remove the syringe cap,</p> 	<p>2. draw 5 mL water into the syringe,</p> 	<p>3. cap the syringe,</p> 	<p>4. shake syringe to wash inside surfaces,</p> 
<p>5. remove cap,</p> 	<p>6. discharge water only, and finally</p> 	<p>7. recap the syringe.</p> 	<p>8. Repeat? Repeat these Washing Steps if necessary. (All traces of the reactants should be washed away.)</p>

OTHER USEFUL GAS SYRINGE TECHNIQUES.

There are a several other techniques that come in handy when working with gases in syringes. Try to use these techniques as much as possible.

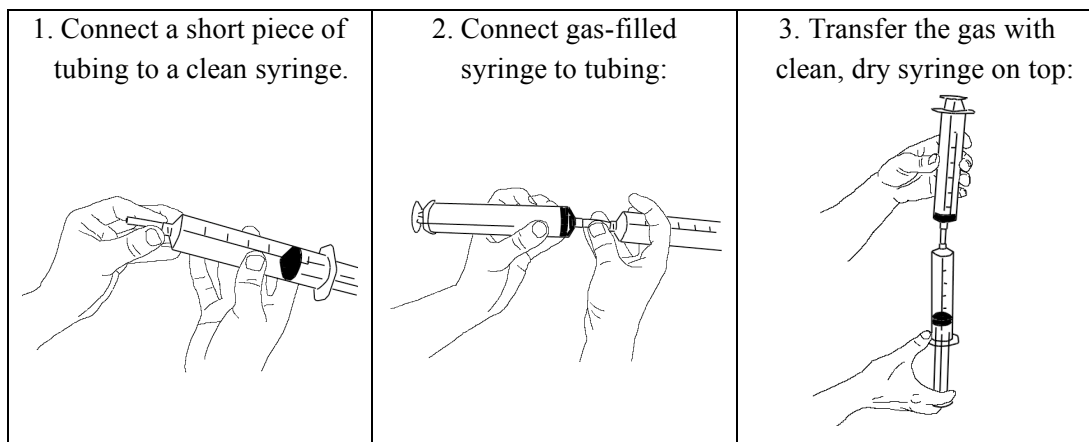
A. MINIMIZING THE QUANTITY OF AIR PRIOR TO REACTION.

This procedure is used when there is excess air in the syringe before the reaction begins. This is a common problem with beginners and is caused by accidentally drawing in air along with the aqueous reagent. Nevertheless, the problem is easy to correct. With the syringe cap directed upward, remove the cap and carefully push the plunger inward until the air is mostly gone. Install the syringe cap.



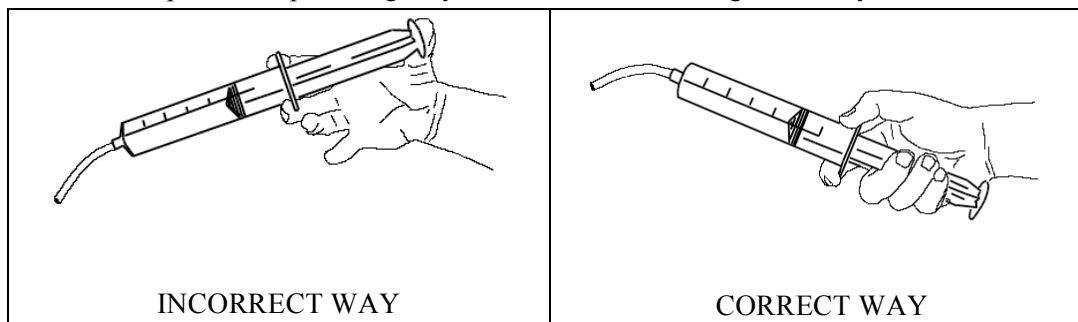
B. SYRINGE-TO-SYRINGE TRANSFER PROCEDURE.

When a gas sample cannot be washed (because the gas is water-soluble) simply transfer the gas sample to a clean, dry syringe by a short connecting tube between the two syringes.



C. CONTROLLED DISCHARGE OF GAS FROM A SYRINGE.

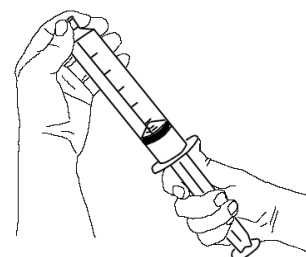
Plungers do not always move smoothly in their syringe barrels. As a result, gases may be discharged in large unintended portions (such as 40 mL all at once) if the method shown in the left diagram below is used. Instead, grasp the syringe by its plunger (right figure) and pull the barrel towards your hand. This simple technique will give you excellent control of gas delivery.



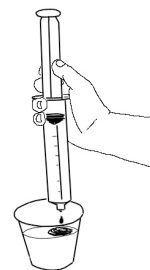
D. PREVENTING UNWANTED DISCHARGES OF NOXIOUS GAS.

Some of the gases that can be generated by the in-syringe method are noxious and must not be discharged into breathable air. These gases are: nitric oxide, NO, nitrogen dioxide, NO₂, ammonia, NH₃, sulfur dioxide, SO₂, and hydrogen sulfide, H₂S. The use of syringes to generate such gas samples works exceptionally well and far better than any other method in preventing undesired discharges. There are two simple considerations to keep in mind whenever handling noxious gases:

(1) Whenever opening the syringe (by removing the syringe cap), do so with the plunger slightly withdrawn (by 5 mL) so the contents are under a slight reduced pressure. Use your thumb to maintain the plunger in this position as shown in the drawing. This will allow a small amount of air to enter the syringe but no noxious gas will escape.



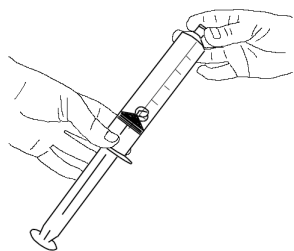
(2) After the gas sample has been generated, discharge the used reagents into a large cup of water to dilute them and prevent further reaction.



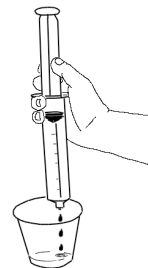
E. SAFETY FIRST. CAP UP AND OFF!

If more than 60 mL gas is going to be generated, follow these steps.

1. Position the syringe so the cap is directed upward.
2. Remove the cap by twisting.



3. Discharge the solution to prevent further gas collection. Recap syringe.



F. CLEAN-UP AND STORAGE

At the end of the experiments, clean the syringe parts, caps and tubing with soap and water. Use plenty of soap to remove oil from the rubber seal. This extends the life of the plunger. It may be necessary to use a 3 cm diameter brush to clean the inside of the barrel. Rinse all parts with distilled water. 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.

Contents of our book

Microscale Gas Chemistry, 4th Edition

Prepare 17 different gases! Over 140 experiments!

450+ pages with hundreds of helpful figures

Teacher-friendly! Student-friendly

Free web-based support (photos, QuickTime movies, down-load labs)

PART 1. GETTING STARTED — 3 SIMPLE GASES

Chapter 1 Getting Started — Generating Carbon Dioxide in a Large Syringe (download for free at gas website; see page 3)

Chapter 2 Experiments with Carbon Dioxide

Chapter 3 Hydrogen

Chapter 4 Oxygen

Chapter 5 Gas Bags

PART 2. LABORATORY EXPERIMENTS

Chapter 6. Mystery Gas

Chapter 7. Percent Composition

Chapter 8. Carbonated Beverages — Priestley's Soda-water

Chapter 9. Molar Mass

Chapter 10. Limiting Reagent

Chapter 11. Barometric Pressure without a Barometer

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Chapter 13. Ammonia

Chapter 14. Ethyne

Chapter 15. Sulfur Dioxide

Chapter 16. Chlorine

Chapter 17. Nitrogen

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Chapter 18. Catalyst Tube Reactions

PART 5. OTHER METHODS

Chapter 19. The Thermal Method: Making Hydrogen Chloride

Chapter 20. Carbon Monoxide

Chapter 21. Ethene (Ethylene)

Chapter 22. Methane

Chapter 23. Nitrous oxide

Chapter 24. Making Gases in a Microwave Oven

PART 6. ADVANCED GASES

Chapter 25. Silane

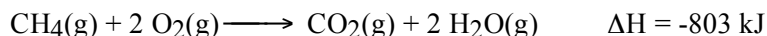
Chapter 26. Hydrogen sulfide

MICROSCALE GAS EXPERIMENTS. PART I. CARBON DIOXIDE



Physical Properties of CO₂: sublimation point -78.5 °C, colorless, odorless, sharp sensation to the nose when inhaled.

Industrial Production: Carbon dioxide is manufactured by the combustion of hydrocarbons:

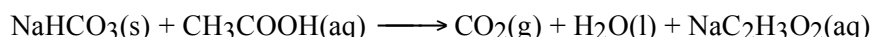


Industrial Uses. Refrigerant (accounting for over 50%), fire extinguishers, the soft drink industry, chemical reagent to make other compounds.

Solubility of CO₂ The solubility of CO₂(g) in water is 3.48 g per L at 0 °C and 1.45 g/L at 25 °C.

EXPERIMENT 1. PREPARATION OF CARBON DIOXIDE.

Chemicals: 0.22 g solid NaHCO₃, 5 mL vinegar (or 1 M HCl). The reaction is:

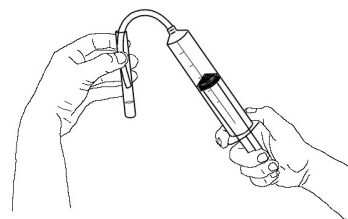
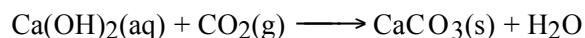


EXPERIMENT 2. TRADITIONAL TEST FOR CARBON DIOXIDE.

Equipment: Small test tube, tubing, 15 cm length

Chemicals: 20 mL CO₂(g), 3 mL limewater

Instructions: Remove the syringe cap and attach a 15 cm length of tubing to the syringe. Transfer limewater to a test tube to a depth of 1 cm. Discharge 10 mL CO₂ above the surface of the limewater as shown in the figure. Stopper the test tube with your thumb and shake the gas and liquid. Notice the production of precipitated CaCO₃. The reaction is:



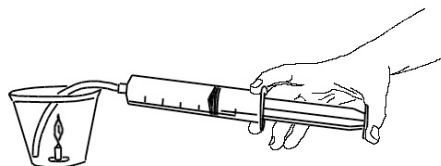
Notes:

EXPERIMENT 3. FLAME-OUT!

Equipment: birthday candle, plastic beverage cup, 15 cm length tubing, matches or lighter

Chemicals: 40 mL CO₂(g)

Instructions: Affix a short candle (1 cm) to a coin with a drop of hot candle wax. Place the candle/coin into the cup as shown. Ignite the candle. Equip the syringe with the 15 cm length tubing. *Rapidly* discharge the CO₂(g) to the bottom of the cup. The flame will go out.



Notes:

EXPERIMENT 4. ACIDITY OF CARBON DIOXIDE.

Equipment: 18 x 150 mm test tube, 15 cm tubing, plastic pipette

Chemicals: 20 mL CO₂(g), universal indicator/pH 8 solution, (*This solution has been prepared for you by transferring some ammonia vapors to a solution of universal indicator. The pH is about 8.*)

Instructions: Add universal indicator/pH 8 solution to a test tube to a depth of 1 – 2 cm. Next, transfer 10 mL CO₂ above the surface of the solution using the long tubing as in Experiment 3. Shake the contents of the test tube to assure mixing.

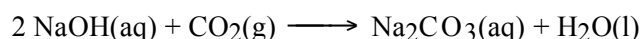
Notes:

EXPERIMENT 5. CARBON DIOXIDE REACTS WITH SODIUM HYDROXIDE.

Equipment: small weighing dish

Chemicals: 50 mL CO₂(g), 10 mL 6 M NaOH(aq)

Instructions: Pour 10 mL 6 M NaOH(aq) into a small weighing dish. Draw 5 mL of this solution into a CO₂-filled syringe. Push the syringe fitting into the cap. Shake the syringe. The plunger will move inward as the CO₂ reacts with the aqueous NaOH forming NaHCO₃ and/or Na₂CO₃. The reaction is:



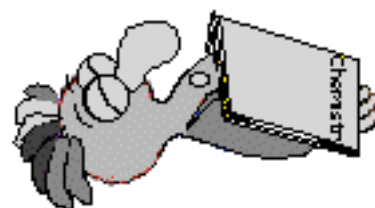
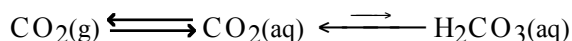
Notes:

EXPERIMENT 6. THE CO₂(g) / CO₂(aq) EQUILIBRIUM

Equipment: 9-ounce (250 mL) plastic cup, special syringe with nail hole

Chemicals: 40 mL CO₂(g), universal indicator solution, limewater

Instructions: Draw 30 mL water into a syringe containing 30 mL carbon dioxide. Record the combined volume — which should be 60 mL. Gently rock the syringe back and forth while it is held in a horizontal position (maximizing the surface area between the water and the gas. Within less than a minute the volume of carbon dioxide will decrease. If the plunger does not move freely, push it inward until there is resistance due to positive pressure inside the syringe. Let go of the plunger and it will return to a volume less than its previous value. Carbon dioxide is dissolving in the water. Continue to gently rock the solution for about a minute, but never shake it. Continue to note the volume of carbon dioxide as a function of time. 1. How much carbon dioxide dissolves? The reaction that is taking place is:



2. Perform the limewater test on some of the *solution* using 1 mL limewater. Perform the pH test on some of the solution. 3. Withdraw the plunger out to the 60 mL mark to create a reduced pressure, hold the plunger in that position and tap the syringe barrel to initiate bubble formation.

Notes:

PART II. HYDROGEN & OXYGEN EXPERIMENTS

Physical Properties

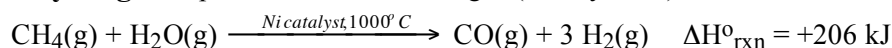
H₂: mp: -259.14 °C bp: -252.5 °C, colorless, odorless. The solubility of H₂(g) in water is extremely low.

O₂: mp: -218.4 °C; bp: -183.0 °C, colorless, odorless. At 20 °C oxygen dissolves in water to the extent of 30.8 cm³ per liter.



Industrial Production:

Hydrogen is produced from natural gas (mostly CH₄) at 1000 °C:



Oxygen is obtained by the fractional distillation of air.

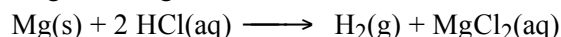
Industrial Uses.

Hydrogen: The major industrial use: synthesis of ammonia by the Haber process ($3 \text{H}_2(\text{g}) + \text{N}_2(\text{g}) \longrightarrow 2 \text{NH}_3(\text{g})$). Other uses include: (a) *hydrogenation of* oils in the food industry, (b) manufacture of methanol used in many industrial applications including the plastics and adhesives; (c) preparation of metals from their oxides at elevated temperatures.

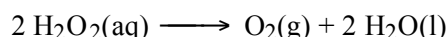
Oxygen. Largest use: manufacture of steel. Oxygen is blasted under high pressure onto molten, impure iron to burn out impurities. Other uses include medical/respiratory oxygen, welding, sewage treatment, production of TiO₂ (makes paints opaque and white), and ethylene oxide.

EXPERIMENTS 7 AND 8. PREPARATION OF HYDROGEN AND OXYGEN.

Hydrogen Chemicals: 0.10 g solid Mg and 3 - 5 mL 2 M HCl. The reaction is:



Oxygen Chemicals: 5 mL 6% H₂O₂(aq) and 0.10 g solid KI (catalyst). The reaction is:



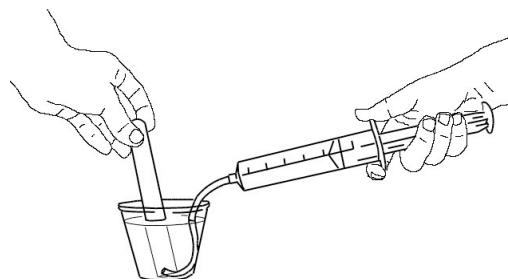
THE EXPERIMENTS. You and your partner should *each* prepare one syringe of O₂(g) and one syringe of H₂(g). Label each syringe (O₂ and H₂ look alike!). You will notice that hydrogen production is *FAST* and oxygen production is *s-l-o-w*. **SAVE** one syringe of O₂(g) for use with NO(g).

EXPERIMENT 9. TRADITIONAL TEST FOR HYDROGEN.

Equipment: Test tube, tubing, plastic cup, candle, matches

Chemicals: 30 mL H₂(g)

Instructions: Fill the test tube with water and place it upside down in the cup of water. Connect the tubing to the fitting on the syringe. Displace the water with H₂ as shown in at right. Light the candle on the coin (from Experiment 3.) Remove a test tube from the water, keeping it open-end downward, and move the open end near the lit candle. A small **BARK** sound will confirm the presence of hydrogen. Repeat the experiment starting with a test tube half-filled with water and half-filled with air.



Notes:

EXPERIMENT 10. TRADITIONAL TEST FOR OXYGEN.

Equipment: Wooden splint, matches

Chemicals: 30 mL O₂(g)

Instructions: Fill a test tube with oxygen by water displacement as you did with hydrogen in Experiment 9. Ignite and blow out the wooden splint and immediately place it into the test tube of oxygen. The splint will re-ignite confirming the presence of oxygen. Less traditional test: Discharge some of the O₂(g) from the syringe directly onto the glowing red splint.

Notes:

EXPERIMENT 11. REVERSIBLE CONVERSION OF COPPER METAL AND CuO(s)

Equipment: Copper-filled pipette, 2 cm length of tubing, burner, matches **Chemicals:** 40 mL H₂(g)

Instructions: The pipette device has been prepared for you. Work the short length of tubing into the pipet. Connect the other end to a syringe filled with air. **Part 1.** Heat the Cu/pipet for 30 s and then slowly pass 60 mL *air* through the pipet while continuing to hold the pipet in the flame. The copper will quickly blacken to copper(II) oxide: $2 \text{Cu(s)} + \text{O}_2\text{(g)} \longrightarrow 2 \text{CuO(s)}$

Part 2. Connect the H₂-filled syringe to the pipet. Heat the CuO/pipet and then slowly pass the hydrogen gas through the pipet while continuing to hold the pipet in the flame. Water droplets should appear inside the stem of the pipet. The reaction is: $2 \text{CuO(s)} + \text{H}_2\text{(g)} \longrightarrow 2 \text{Cu(s)} + \text{H}_2\text{O(l)}$

Notes:

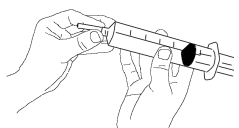
EXPERIMENT 12. HYDROGEN-OXYGEN MIXTURES.

Equipment: tubing, 2 cm, plastic cup, matches, piezoelectric sparker, weighing dish, dispo pipette

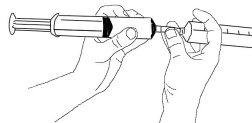
Chemicals: 30 mL H₂(g), 15 mL O₂(g), 3% dish soap solution

Instructions: Transfer O₂ to the H₂ syringe as shown. Form a 1:2 mixture. Re-label mixture!

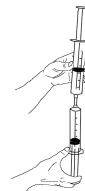
1. Connect a short piece of tubing to the O₂ syringe.



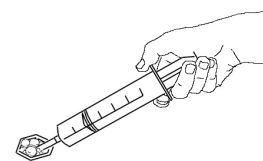
2. Connect the other end of the tubing to the hydrogen syringe:



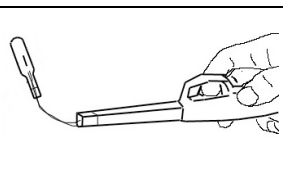
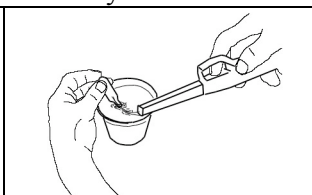
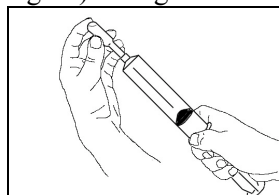
3. Transfer O₂ to the H₂



Try two experiments: 12-A. DYNAMITE SOAP! Use the short tube to form a mound of bubbles in a small weighing dish as shown at right. Remove the syringe and ignite the bubbles with a match. **Caution!** LOUD! (Unused gas mixture can be saved for the next experiment.).



12-B. H₂/O₂ ROCKET Displace water from a plastic pipette rocket (left figure). Slip the rocket over the wires on the piezo-lighter and *draw some water into the stem* (middle figure) and ignite the rocket! It'll fly 10 m!



Notes:

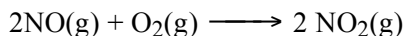
PART III. EXPERIMENTS WITH NITROGEN OXIDES



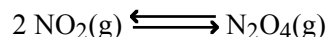
Physical Properties of Nitric oxide, NO and Nitrogen dioxide, NO₂:

	NO	NO ₂
melting point	-163.6 °C	-11.2 °C
boiling point	-151.8 °C	+21.2 °C
color	colorless	orange-brown
odor	acrid	acrid
solubility	0.00983 g/L at 0 °C	reacts

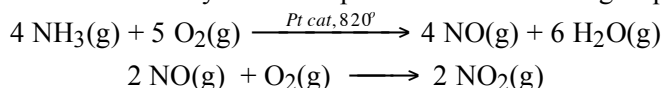
Nitric oxide is a colorless, paramagnetic gas. It has an acrid odor and has very low solubility in water. NO is the simplest thermally stable odd-electron molecule. It is extremely reactive and readily oxidizes in air to produce NO₂:



Nitrogen dioxide is a red-brown gas that is highly reactive with water. The dynamic interconversion between NO₂ and N₂O₄ makes it impossible to study either of these species alone:

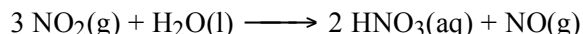


Industrial Production. Nitric oxide is formed from the combustion of ammonia. Wilhelm Ostwald studied the production of nitric acid and by 1901 had perfected the following sequence of reactions:

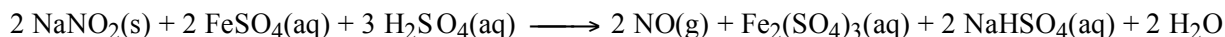


Industrial Uses. The primary use of these gases is the production of nitric acid for the production of fertilizers and a wide range of chemicals. Nitric acid is formed by the reaction of NO₂ with water.

NO₂ reacts with water. Nitrogen dioxide reacts with water to produce nitric acid and NO:



EXPERIMENT 13. PREPARATION OF NITRIC OXIDE. Chemicals: 0.25 g solid NaNO₂, 3 - 5 mL acidic ferrous solution (1.2 M FeSO₄ in 1.8 M H₂SO₄) prepared by dissolving 13.5 g FeSO₄·7 H₂O in 28 mL distilled water and adding 12 mL 6 M H₂SO₄. (This solution has been prepared for you and is in the "Box of Solutions.") The reaction is:



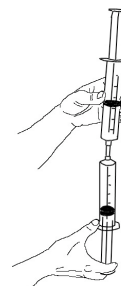
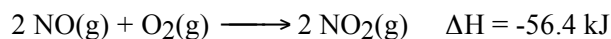
Prepare NO(g) by the general In-Syringe Method. Samples of NO(g) should be "washed".

EXPERIMENT 14. CONVERTING NO(g) TO NO₂(g): LAW OF COMBINING VOLUMES

Equipment: Clean dry syringe, short tubing

Chemicals: 40 mL NO(g), 20 mL O₂(g),

Instructions: Transfer the NO(g) to a clean, dry syringe via a short length of tubing as shown in the figure. Simultaneously push and pull on the respective plungers. (This step is necessary because water readily dissolves NO₂.) Connect the dry NO(g)-filled syringe with the O₂(g)-filled syringe via a short length of clean, dry tubing. Slowly transfer a volume of O₂(g) equal to half of the volume on the NO(g). The reaction is:



Notes: syringe becomes warmer

EXPERIMENT 15. LECHATELIER'S PRINCIPLE AND THE NO₂/N₂O₄ EQUILIBRIUM.

Chemicals: 30 mL NO₂(g)

Nitrogen dioxide is a brown-red gas that exists in equilibrium with its dimer, N₂O₄(g):



The LeChâtelier principle states that an increase in volume will favor a shift to the left in the above equilibrium. Quickly pull the syringe barrel outward to the 60 mL mark and hold it in that position. The NO₂(g) will immediately fade due to the decrease in concentration but within a fraction of a second the red color will intensify due to formation of more NO₂ as predicted by the LeChâtelier principle. What will happen if you push the plunger down rapidly and hold it in that position. Try it!

Notes:

EXPERIMENT 16. HIGH TEMPERATURE FAVORS THE ENDOTHERMIC SUBSTANCE.

Equipment: cup of very hot water and cup of ice cold water

Chemicals: two syringes of NO₂(g), 30 mL each

Refer to the NO₂/N₂O₄ equilibrium provided above in Expt. 15. A syringe of NO₂/N₂O₄ placed in hot water should shift towards the endothermic substance, NO₂. Cold water favors N₂O₄

Notes:

OPTION: DO EXPERIMENT 17 OR 18:

EXPERIMENT 17. ACID RAIN MICROCHEMISTRY

Equipment: 24-well plate, 1 gallon (4 L) sealable plastic food storage bag, overhead projector

Chemicals: NO₂(g), 5 mL of Universal Indicator, 0.1 g of sodium bicarbonate, NaHCO₃

Instructions. Automobiles produce nitrogen oxides that act to produce acid rain. In this experiment, a 24-well plate is enclosed in a sealable bag to create an ecosystem. Mix 100 mL distilled water with 5 mL of universal indicator. Fill 18 of the wells with this solution. (Slightly overfill them as shown at right if using an overhead projector.) To the remaining universal indicator solution, dissolve 0.1 g of sodium bicarbonate, NaHCO₃. Fill the remaining six wells with this solution. Place a 6 cm length of a plastic pipet stem between the four middle wells in order to prop up the bag. Slip the filled well plate and the syringe into the plastic bag. Seal the bag shut. Place the assembly on the overhead projector. Discharge the gas into the bag. As the gas drifts across the "landscape", the unbuffered lakes will become acidic. The buffered lakes will eventually become acidified as well. At the end of the experiment, shake the contents of the bag in order to dissolve all the NO₂ in the water. Open the bag and discard the contents down the drain.

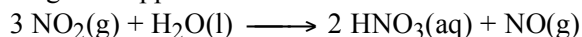
Notes: unbuffered lakes take two minutes to become acidified and it takes 10 minutes for the buffered ones

EXPERIMENT 18. FROM NITRIC OXIDE TO NITRIC ACID

Equipment: Plastic cup

Chemicals: NO₂(g)

In Expt. 1 nitric oxide and oxygen formed nitrogen dioxide. Here, this gas will be converted into nitric acid. Draw 20 mL water into a syringe containing NO₂. The gas appears to be soluble in water, however it is actually reacting with water producing nitric acid. Additional water is drawn into the syringe as the red colored gas disappears. The redox-reaction is:



Now draw some air into the syringe. The NO(g) generated will make more red NO₂(g) which will quickly react in the water present. More air, more red NO₂(g), more nitric acid.

Notes:

Microscale Gas Workshop, University of North Texas, July 2007

Microscale Gas Chemistry Kits

Each kit contains:

- ❖ two 60-mL plastic syringes with a LuerLOK fitting
- ❖ two Latex LuerLOK syringe caps
- ❖ two plastic vial caps
- ❖ one 15 cm length of amber, thick-wall rubber tubing
- ❖ one 3 cm length of the same tubing
- ❖ one dropper bottle of silicone oil
- ❖ transfer disposable polyethylene pipet
- ❖ transfer disposable polyethylene pipet mini-rocket
- ❖ one clear plastic beverage cup (250 mL/9 oz)
- ❖ one small plastic weighing dish
- ❖ one birthday candle and penny
- ❖ test tube (12 x 100 mm)
- ❖ small white cups for Flame-out!
- ❖ one wooden splint
- ❖ paper towel
- ❖ set of gas cards
- ❖ matches
- ❖ glass pipet with Cu(s)

piezo sparkers

well plates/gallon bags

hot water electric heater

cow magnet

wide-mouth plastic bottles

catalyst tubes (for sale)

books (for sale)

videos (for sale)

marker pens

DEMOS:

Cow magnet and iron

Ammonia fountain

Underwater fireworks

Chemiluminescence

Catalyst tube

2-L bottle filled with CO₂

Chemicals for In-Lab Workshop

From the grocery store:

500 mL vinegar
10 mL liquid dish soap
ice, about a half gallon cubed
1 small box of baking soda (OR NaHCO_3 as listed below under solid chemicals)

Solutions of chemicals:

250 mL limewater, not cloudy
250 mL universal indicator solution
500 mL of 2 M $\text{HCl}(\text{aq})$
40-mL 6 M HCl
200 mL of 6 M $\text{NaOH}(\text{aq})$
500 mL 6% hydrogen peroxide
40 mL 30% hydrogen peroxide
400 mL acidic ferrous solution (13.5 g $\text{FeSO}_4 \cdot 7 \text{H}_2\text{O}$ in 28 mL distilled water and adding 12 mL 6 M H_2SO_4)
40-mL FRESH household bleach (or 1 g calcium hypochlorite, $\text{Ca}(\text{OCl})_2$)
50 mL ammonium hydroxide solution, concentrated
5 mL phenolphthalein

Solid chemicals:

1 g CaC_2 (solid black chunks, not tan powder!)
100 g sodium bicarbonate (baking soda), $\text{NaHCO}_3(\text{s})$ (OR small box baking soda as listed above)
25 g magnesium, $\text{Mg}(\text{s})$ turnings
25 g potassium iodide, $\text{KI}(\text{s})$
25 g sodium nitrite, $\text{NaNO}_2(\text{s})$
1 g iron(III) oxide, $\text{Fe}_2\text{O}_3(\text{s})$

SHARED EQUIPMENT FOR IN-LAB WORKSHOP

Bunsen burners with tubing
Gas supply ON