

Microscale Gas Chemistry

20 Gas Demos!

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Website:
mattson.creighton.edu/Microscale_Gas_Chemistry.html

Twenty Gas Chemistry Demos

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Gas chemistry makes great classroom demonstrations! Students enjoy spectacular demos and teachers appreciate demos that are inexpensive and create little or no chemical wastes. Gas chemistry is visual! I will perform about twenty demonstrations with a total of ten gases, each briefly described below. These experiments come from our gas chemistry website (also available as a book) in which we cover the preparation of 17 different gases, and describe over 150 chemical experiments and demonstrations with these gases. Unlike other gas chemistry experiments conducted in syringes, our experiments focus on *chemical*, not physical properties of the gases. The gas website and book are designed for easy and enjoyable use by teachers and their students. The website includes QuickTime movies, color and time-lapsed photography, and an amusing mascot gas bird named “Bill.” The book and website includes hundreds of high-quality drawings. All materials may be copied (and in some case down-loaded) for classroom use. The website and book are:

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

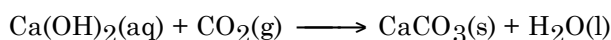
“Microscale Gas Chemistry, 4th Edition” available from Educational Innovations as Item Number BK-595. Visit their website (<http://www.teachersource.com/>)

The contents of our book are provided at the end of this program description.

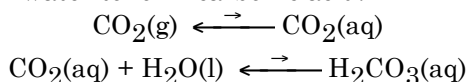
Description of the experiments that you will see today:

A. Experiments with carbon dioxide

Experiment 1. Traditional test for carbon dioxide. The simple limewater test is frequently used to detect carbon dioxide. About 5 mL carbon dioxide is delivered to a test tube containing a few mL limewater, Ca(OH)_2 (sat'd), resulting in a milky-white precipitate of calcium carbonate:

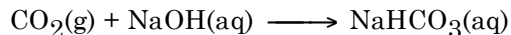


Experiment 2. The acid test. Carbon dioxide is acidic. About 5 mL carbon dioxide is delivered to a test tube half-filled with a solution of red cabbage juice or universal indicator or any acid-base indicator. Carbon dioxide dissolves slightly in water (carbonated beverages) and then to a much smaller extent, some reacts with water to form carbonic acid:



Experiment 3. Fighting Fires! Carbon dioxide extinguishes flames and has a greater density than air. Carbon dioxide is used in fire extinguishers. In this experiment, 50 mL of carbon dioxide will be poured over a burning candle. The flame will be immediately extinguished. Because carbon dioxide is about 50% heavier than air, it pours downward. Helium and hydrogen, both lighter than air would pour upward.

Experiment 4. Disappearing Gas! Carbon dioxide reacts with sodium hydroxide to produce sodium carbonate. In this experiment, aqueous sodium hydroxide is drawn into a syringe filled with carbon dioxide. A reaction ensues in which the gas is consumed in the reaction and as a result, the plunger “magically” is drawn into the syringe barrel as the gas reacts. This is a nice visual experiment that allows one to “see” a gaseous reagent being consumed.

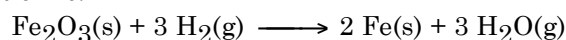


Experiment 5. Carbon dioxide reacts with sodium hydroxide in a 1-L plastic bottle. A great classroom demo that repeats Experiment 4, but accomplishes a different goal: the bottle collapses, visually showing that atmospheric pressure is “real.”

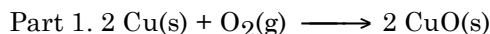
B. Experiments with hydrogen

Experiment 6. The “ON/OFF” Candle. A hydrogen-filled syringe (without a plunger) is lowered over a burning candle. The flame goes out. The syringe is raised and the candle re-ignites as long as the wick is still hot enough. The process can be repeated several times.

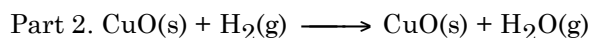
Experiment 7. Making iron! Evidence of a chemical reaction. A syringe filled with hydrogen is connected to a glass pipet containing iron(III) oxide, $\text{Fe}_2\text{O}_3(\text{s})$. A magnet shows that $\text{Fe}_2\text{O}_3(\text{s})$ is not attracted to a magnet. The pipet is heated and then the hydrogen is slowly passed through the pipet. There is not much to see because the iron that is produced looks just like the original $\text{Fe}_2\text{O}_3(\text{s})$, however, the product is attracted to a magnet, suggesting that we have produced iron or $\text{Fe}_3\text{O}_4(\text{s})$, which is also ferromagnetic. Measuring masses before and after confirm that the product is elemental iron. The reaction is:



Experiment 8. Copper again? Copper oxidation and copper oxide reduction. This two-part experiment is similar to the previous experiment, but is certainly more visual. In Part 1, shiny copper wool is heated and exposed to air. The oxygen in air reacts with the copper to produce black copper(II) oxide:



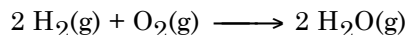
In the second step, hydrogen reacts with $\text{CuO}(\text{s})$ to produce shiny copper like we had in the beginning.



Experiment 9. Howlin’ Hydrogen. Here we will see a hydrogen flame burning in an atmosphere of oxygen. Listen for the howl and look for the water.

C. Experiments with oxygen

Experiment 10. Dynamite Soap. The name says it all! This reaction has been performed since the 18th century and is always a favorite among the BIG-BANG audience. The reaction is:



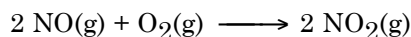
Experiment 11. Rockets. Same reaction as above, however this time the explosion propels a plastic rocket 10 m or more.

Experiment 12. Liquid oxygen is paramagnetic. This is a nice small-scale experiment in which as little as 60 mL gaseous oxygen can be liquefied and used to demonstrate the paramagnetism of oxygen.

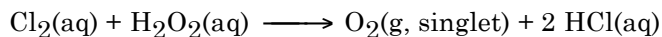
Experiment 13. Liquid oxygen from gas bag. This is a larger-scale version of the previous experiment. This time one can even see the blue color of oxygen!

D. Experiments with nitrogen oxides

Experiment 14. Colorless + Colorless = Red. Nitric oxide reacts with oxygen. This is a delightfully visual experiment in which two colorless gases are mixed to produce red nitrogen dioxide:



In this case, the first reaction produced singlet oxygen that is higher in energy than “normal” triplet oxygen that we breathe. The singlet oxygen has the same formula, $O_2(g)$, but has its electrons paired in the antibonding π -orbital.



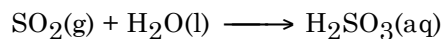
Experiment 24. Underwater Fireworks. My personal favorite. Acetylene and chlorine react underwater with flames and pops!



We can test the resulting solution with indicator solution.

H. Experiment with sulfur dioxide

Experiment 25. Acid Rain. Sulfur dioxide is extremely soluble in water and forms small amounts of sulfurous acid. In the environment, sulfur dioxide is often oxidized in the atmosphere to sulfur trioxide which reacts with water to form sulfuric acid. Either way, the solution is acidic. In this demo, we will use a 24-well plate inside a sealed plastic food storage bag to simulate lakes. All lakes will have acid-base indicator in them and some of the lakes will be buffered. Sulfur dioxide will be added via a small hole in the bag. This demo works well on the overhead projector. This is another example of how noxious gases can be handled safely in a classroom situation.



MICROSCALE GAS CHEMISTRY: PREPARING GASES IN SYRINGES

Bruce Mattson and Michael Anderson, Department of Chemistry, Creighton University, Omaha, NE 68178

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 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.

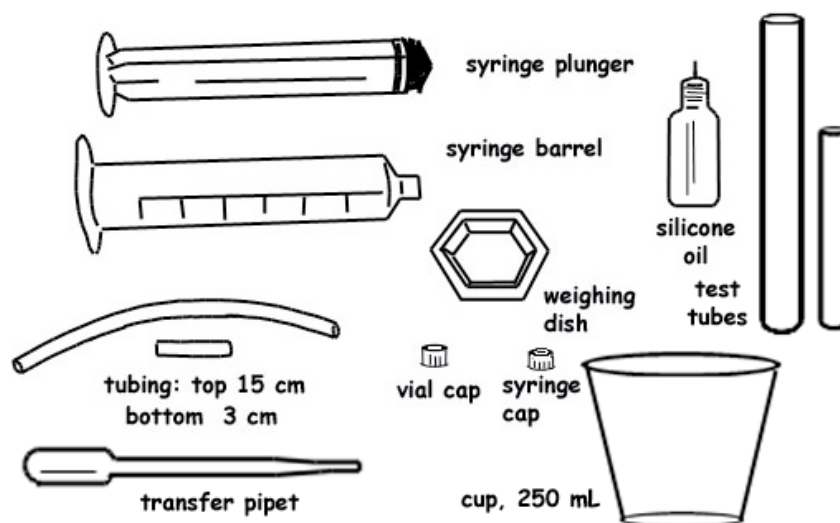
Method for Preparing Gas Samples.

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

**Educational Innovations sells worldwide!*

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 with time. 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

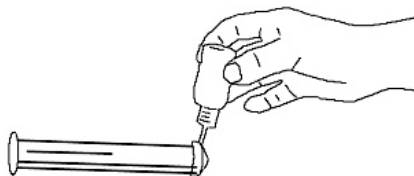
The general strategy of the method is to react two substances in a 60 mL syringe. The limiting reagent is always used in solid form and is placed in a small vial cap. The second reagent is prepared as an aqueous solution. For example, one could generate $\text{CO}_2(\text{g})$ from excess aqueous acetic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

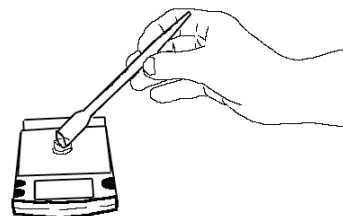
The solid reagent is placed in the vial cap that is then lowered into the syringe barrel by water flotation. In the preparation of carbon dioxide, one would use 0.21 g baking soda, NaHCO_3 . Here are the chemicals needed for four common gases:

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.

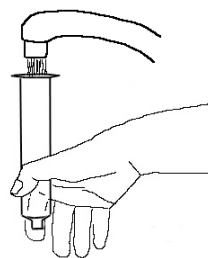


spatula made from pipet:



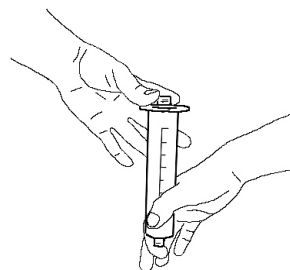
Fill the syringe barrel with water.

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.



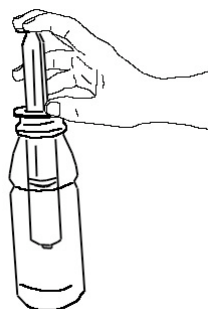
Lower the cap by flotation.

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, one would use 5 mL vinegar, $\text{HC}_2\text{H}_3\text{O}_2$. Here are the chemicals needed for four common gases:

To make:

Carbon dioxide

Hydrogen

Oxygen

Nitric oxide

Use: (Note: 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

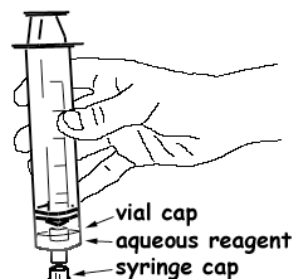
Draw aqueous reagent into syringe.

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.



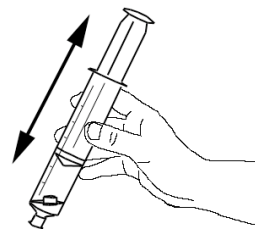
Install syringe cap.

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



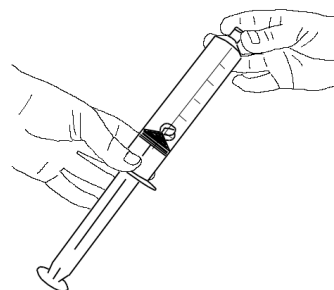
Generating the Gas.

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.



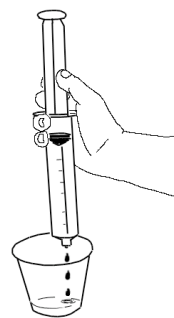
Remove cap to stop the reaction.

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

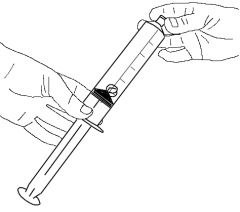
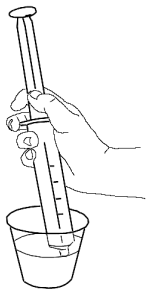
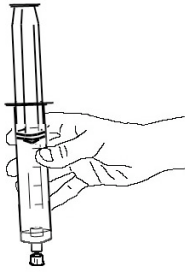
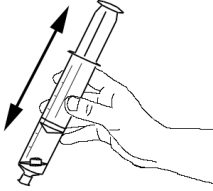
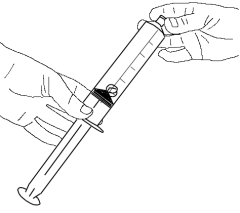
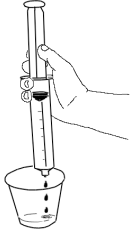
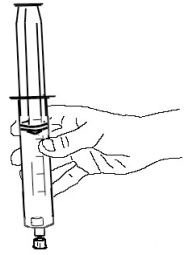
Turn the syringe 180° and discharge the liquid reagent into the plastic cup. Caution: Never remove the syringe cap with the cap end of the syringe directed downward — reagents will spray out of the syringe. Immediately cap the syringe with the cap to prevent loss of gas by effusion.



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:

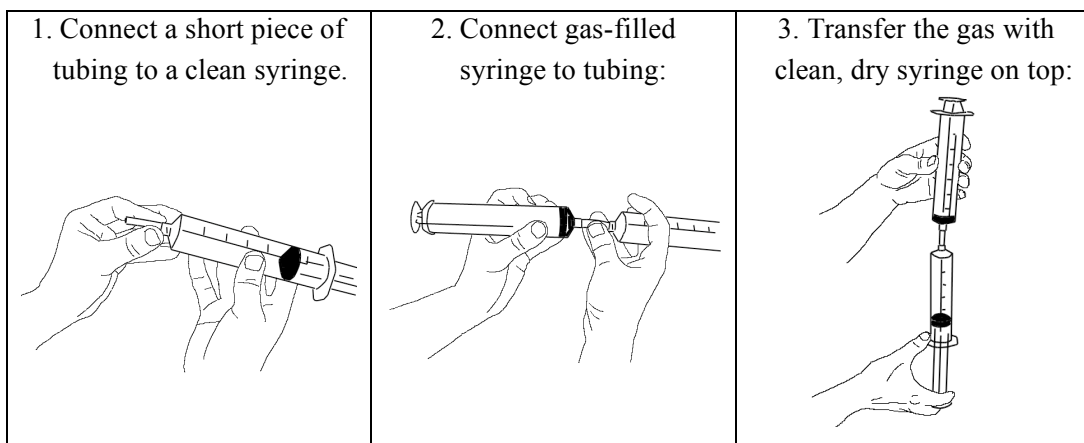
<p>1. remove the syringe cap,</p> 	<p>2. draw 5 mL water into the syringe,</p> 	<p>3. cap the syringe,</p> 	<p>4. shake syringe to wash inside surfaces,</p> 
<p>5. remove cap,</p> 	<p>6. discharge water only, and finally</p> 	<p>7. recap the syringe.</p> 	<p>8. Repeat? Repeat these Washing Steps if necessary.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

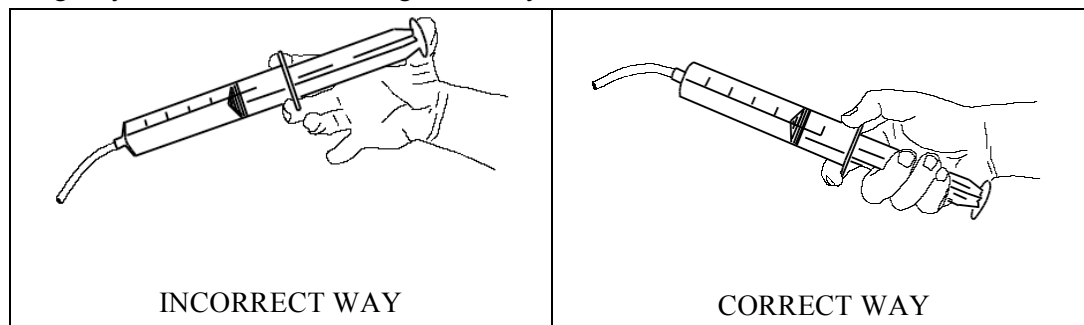
A. SYRINGE-TO-SYRINGE TRANSFER PROCEDURE.

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.



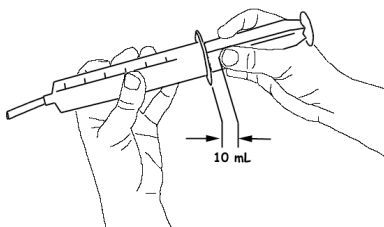
B. CONTROLLED DISCHARGE OF GAS FROM A SYRINGE.

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.



C. DISCHARGING A SPECIFIC VOLUME OF GAS.

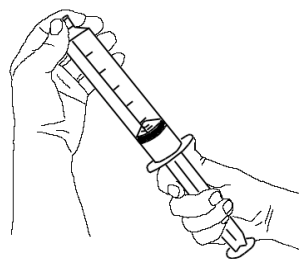
Position thumb as a “stop” to discharge desired volume of gas and then push inward.



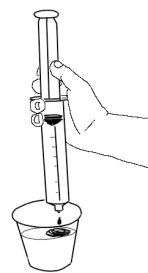
D. PREVENTING UNWANTED DISCHARGES OF NOXIOUS GAS.

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.



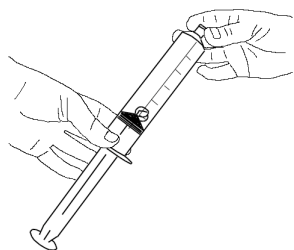
(2) After the gas sample has been generated, discharge the used reagents into a large cup of water to dilute them and prevent further reaction.



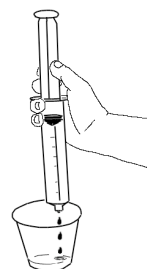
E. SAFETY FIRST. CAP UP AND OFF!

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)

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Chapter 2 Experiments with Carbon Dioxide

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Chapter 14. Ethyne

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Chapter 18. Catalyst Tube Reactions

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