

## PART 1. GETTING STARTED WITH THREE EASY GASES

Chapter 1. Getting Started. Generating Carbon Dioxide in a Large Syringe

Chapter 2. Experiments with Carbon Dioxide

Chapter 3. Hydrogen

Chapter 4. Oxygen

Chapter 5. Gas Bags

Generally, the three gases described in Part 1 and the experiments that go with them provide an easy way to get started with microscale gas chemistry. Chapter 1 introduces the equipment needed and the general approach for generating a wide variety of gases using microscale techniques. In Chapter 1 carbon dioxide is generated for study in Chapter 2. Chapters 3 and 4 include both specific instructions of gas preparation and a set of experiments for each of the gases hydrogen and oxygen, respectively.

Chapter 5 is provided as a useful shortcut for the teacher. Gases can be generated in much larger quantities using simple, homemade **gas bags** and this is useful when students need a supply of several gases. For example, in Chapter 4 students need both hydrogen and oxygen for several of the experiments. The teacher may use the gas bag technique described in Chapter 5 in order to prepare one or both of these gases for the students in the interest of saving time.

Part 1 is suited for use by a wide variety of audiences — ranging from middle school physical science students up through university-level chemistry students. Like most experiments and demonstrations, results and observations can be discussed and interpreted on a level appropriate for the students' background.



# CHAPTER

# 1

# GETTING STARTED: GENERATING CARBON DIOXIDE IN A LARGE SYRINGE

IN 1992 HUBERT ALYEA published an ingenious method for the safe generation of gases, including noxious gases, for classroom use.<sup>1</sup> The method utilizes 60 mL disposable plastic syringes. We began using this approach with our students in our descriptive inorganic laboratory course in 1992 and found the technique to be convenient, easy to use, safe, and reliable. Since 1992, when we first employed this method, we have developed numerous modifications that significantly improve the ease and safety of gas generation using syringes. We also have developed numerous experiments with a variety of gases, most of which are suitable for use as high school and introductory college level microscale laboratory experiments. All of these experiments and classroom demonstrations have been published in a series of articles that have appeared in **Chem13 News** from 1996 – 2008. This series is collected here in a refined state where all pertinent improvements have been included.

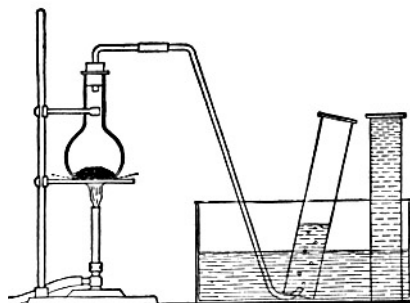


Hubert N. Alyea

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<sup>1</sup> Hubert N. Alyea is considered by many to be the “grand-daddy” of chemical demonstrations. His chemical antics in front of public audiences earned him a reputation that inspired the character in Walt Disney’s **The Absent-Minded Professor**. His proposal to use syringes to generate and collect gases is published in *J. Chem. Educ.*, **69** 65 (1992)

Laboratory experiments involving gases are almost never included in modern chemistry laboratory books although throughout most of the 19<sup>th</sup> and 20<sup>th</sup> centuries the generation and study of gases were integral parts of a college course in chemistry. The general perception is that gases are hard to see, hard to contain, and hard to use as chemical reagents. The word “gas” causes most teachers to think of expensive lecture bottles that mysteriously become empty without notice and are then impossible to dispose of — or of the drawings in textbooks that show pneumatic troughs and the exotic apparatus with its delivery funnel, round-bottom flask and tubing. If you are one of the very few individuals who has actually produced and collected a gas in a laboratory setting, it was probably using pneumatic troughs and a method of water displacement that is over 200 years old!<sup>2</sup> You may have vivid recollections of tippy inverted test tubes, water soaked lab books, and long rubber tubing that tended to tip over the round-bottom flask filled with bubbling acid. You may shudder at the memories of messy pneumatic troughs and the frustrations and dangers of trying to push long-stem glass funnels through rubber stoppers in order to construct the apparatus. With our microscale gas chemistry techniques, you and your students will learn that gases can be produced cleanly, quickly, and cleverly in completely self-contained reaction chambers (big syringes without needles) while your students watch in awe at the reaction taking place in their own hands in front of their very eyes.



*collecting oxygen with a pneumatic trough, circa 1930*

You are undoubtedly interested in the chemistry of gases or you probably would not own this book. Here are several 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 ½ 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.

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<sup>2</sup> Drawing is from **General Chemistry**, Harry N. Holmes, Macmillan Company, 1930.

- ❖ ***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. The emphasis of this book is 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.

Flipping through the pages, you will realize that there is much more here than can be done during your first year with gas chemistry. How do you get started with microscale gas chemistry? I suggest an easy, “lets-slip-into-this-slowly” approach. Gas chemistry doesn’t have to be all-or-nothing — it isn’t like changing religions or trying parenthood — it is more like deciding to explore more new places or resolving to try different things. Keep it easy and fun for yourself. The students will enjoy these techniques and experiments because they are fun and easy to learn and do.

We suggest that you plan to cover this section of the book during the first year that you try microscale gas chemistry. Chapter 1 introduces you and your students to the techniques as you learn to make carbon dioxide from vinegar and baking soda. Chapter 2 contains five easy experiments that can be performed with carbon dioxide. In Chapter 3, students prepare hydrogen and perform several reactions with the gas. Similarly, in Chapter 4, students make oxygen and study its reaction chemistry. Chapters 2, 3 and 4 each take one 40 minute laboratory period.

Let’s get started! We suggest that this chapter be given at least one complete laboratory period, perhaps two if you use the [YouTube video \(https://www.youtube.com/watch?v=UfpYQ9YwEds\)](https://www.youtube.com/watch?v=UfpYQ9YwEds) or have a set of rules that need to be covered. During the first period, you can show your students the equipment and go over the names for everything. This is a good time to emphasize that there are five small parts that are easy to lose and they must not lose them or (fill in the blank) will happen. Also during the first lab session, you should demonstrate the technique once or twice (or you could play the video and watch me show how it’s done — see Appendix E to order). During the second lab period, the students can handle the equipment for the first time and use it to generate carbon dioxide. By the end of the lab period, they will be good at it and will be able to generate a gas sample within five minutes. And what they don’t know yet is that the same general approach is used to generate quite a few different gases. Another goal of this first encounter with the syringes is to let your students work through any immature thoughts they may be harboring. After all, they are

working with huge syringes and “making gas”, so there will be the odd deviation from model student behavior. But that quickly passes. It will also occur to one or two proportional thinkers that if they use lots of reagents, they could get the plunger to go flying from the barrel. Well, they are right, however they will soon learn (providing they try this) that the plunger does not go very far at all and they have created a mess. If this is something they are going to try, it’s best that they try it with baking soda and vinegar. They will also realize that making the plunger pop out costs them a syringe full of gas and requires them to start all over after cleaning up and enduring your wrath, etc.

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## THE IN-SYRINGE METHOD FOR PREPARING GAS SAMPLES

In this chapter we describe the most general method for gas generation, the “In-Syringe Method” and use the method to prepare carbon dioxide. 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. Interestingly, Alyea did not suggest a practical way in which the solid and aqueous reagents should be brought together inside the syringe — he suggested, and I quote, “Into the cap put enough reagent to generate 50 mL of gas. Drop the cap into the syringe, and immediately fully insert the plunger.” The synthesis of hydrogen sulfide (one of the gases suggested by Alyea) by these exact instructions must have caused quite an annoyance to others working in the same wing of the building! It is in this area that we have made important contributions by designing a few simple modifications to Alyea’s procedure that assure that the reaction is safely contained within the syringe and exposure to the gases produced is prevented.<sup>3</sup>

The In-Syringe Method is used for generating the following gases:

- ❖ carbon dioxide, CO<sub>2</sub> (this chapter and Chapter 2)
- ❖ hydrogen, H<sub>2</sub>, (Chapter 3)
- ❖ oxygen, O<sub>2</sub>, (Chapter 4)
- ❖ nitric oxide, NO, and nitrogen dioxide, NO<sub>2</sub> (Chapter 12)
- ❖ ammonia, NH<sub>3</sub> (Chapter 13)
- ❖ ethyne, C<sub>2</sub>H<sub>2</sub> (Chapter 14)
- ❖ sulfur dioxide, SO<sub>2</sub> (Chapter 15)
- ❖ chlorine, Cl<sub>2</sub> (Chapter 16)
- ❖ nitrogen, N<sub>2</sub> (Chapter 17)
- ❖ silane, SiH<sub>4</sub> (Chapter 26)
- ❖ hydrogen sulfide, H<sub>2</sub>S (Chapter 27)

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<sup>3</sup> The In-Syringe Method was originally described in our first article on microscale gas chemistry that appeared in the October, 1996 issue of **Chem13 News** (Number 251.)

## 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.

## Suitability

The preparation of carbon dioxide is suited for either classroom demonstrations or as laboratory experiments conducted by students. This chapter can be used very early in the first year high school chemistry curriculum. As a laboratory activity, it is appropriate when discussing matter, chemical compounds, heterogeneous and homogeneous mixtures and physical and chemical changes. We recommend carbon dioxide as the first gas simply because the chemicals used and the products generated are not dangerous.

## Inexpensive spatulas

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



## Website

This chapter is available on the web at our microscale gas chemistry website:

[http://mattson.creighton.edu/Microscale\\_Gas\\_Chemistry.html](http://mattson.creighton.edu/Microscale_Gas_Chemistry.html)

## Instructions for your students

For classroom use by teachers. Copies of all or part of this document may be made for your students without further permission. Please attribute credit to Professors Bruce Mattson and Mike Anderson of Creighton University and this website.

# THE IN-SYRINGE METHOD FOR PREPARING GAS SAMPLES

## GENERATING CARBON DIOXIDE

### General Safety Precautions

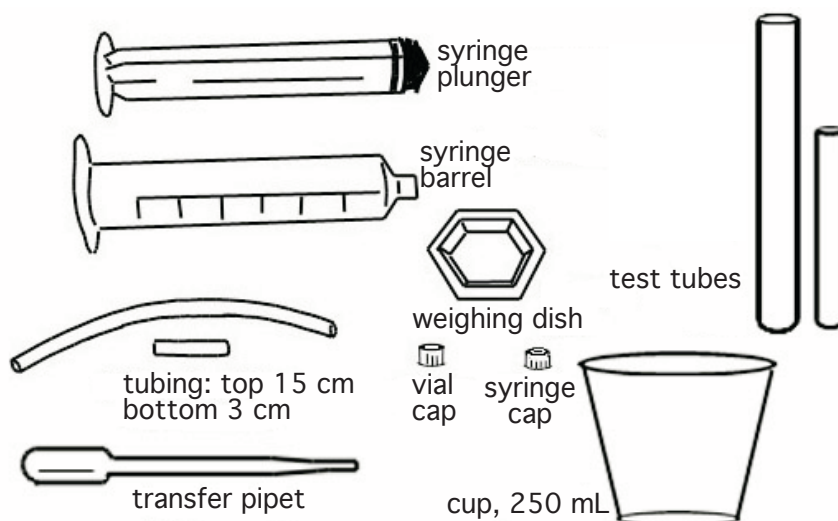
Always wear safety glasses. Gases in syringes may be under pressure and could spray liquid chemicals. Follow the instructions and only use the quantities suggested.

### Toxicity

Carbon dioxide is a relatively non-toxic gas. Like all gases other than oxygen, it is a simple asphyxiant if inhaled in very large quantities. We will not be generating very large quantities of carbon dioxide.

### GETTING STARTED

Before we start making gases, we need to know a bit more about the equipment that we will use. Many of the important pieces that we will use are pictured below. Let's start with the most dramatic, the large syringe which may invoke pangs of fear and memories of visits to the doctor's office. There are no needles, however. You will notice that after working with these syringes a few times, you will no longer think of them for their medical applications. (Incidentally, these 60 mL syringes are used by veterinarians to treat large farm animals and are not normally used by medical doctors.)





The syringe is composed for two major parts — the barrel (outside part) and the plunger (inside part). Plungers and barrels are interchangeable. On one end of the plunger you will notice an air-tight black rubber seal. Even tiny little hydrogen molecules have trouble sneaking past the seal so these are pretty impressive pieces of equipment.

The next piece of equipment to find are the two syringe caps. They are made of rubber and fit snugly onto the syringe barrel — again, an air-tight fit. They are tiny and easily lost, which would be a problem because they are used to keep the gas in the syringe. Keep an eye on them and don't lose them down the drain.

The vial cap is used to lower the solid reagent into the syringe (as discussed below) and is also small and easily lost. Again, you should have two of them. The other items in your gas kit will include a long and short piece of tubing, two weighing dishes, a plastic pipet, a plastic cup, two test tubes (two different sizes) and a birthday candle.

## MAKING CARBON DIOXIDE

You are now about to prepare your first gas sample using the syringe equipment. The general strategy of the method is to react two substances in a large 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, you will generate  $\text{CO}_2(\text{g})$  from vinegar, used in excess, and solid baking soda or sodium bicarbonate,  $\text{NaHCO}_3$ . The steps given below eventually will be used to make all sorts of gases.

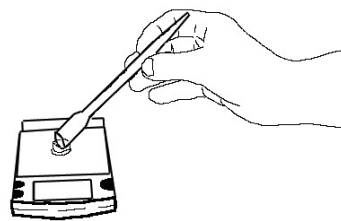
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### 1. Wear your safety glasses!

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### 2. Measure out the solid reagent (Use 0.2 g $\text{NaHCO}_3$ to make $\text{CO}_2$ )

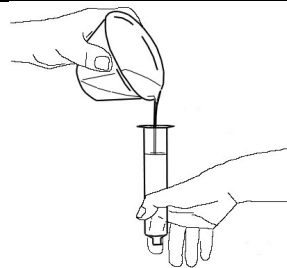
Place the solid reagent into the vial cap. We recommend that the solid be measured directly into the vial cap to prevent losses from transferring small amounts of solids. Your teacher may tell you how to estimate this quantity (for example, a half-filled vial cap), rather than having you measure the amount on a balance.



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### 3. Fill the syringe barrel with water

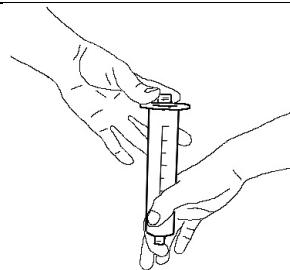
Fill the barrel with water. Place your finger over the hole to form a seal. Fill completely to the top.



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### 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.



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### 5. Lower the cap by flotation

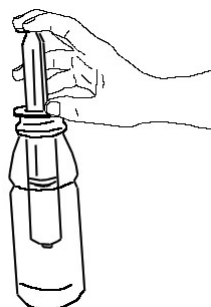
Release the seal made by finger to lower the cap into the syringe barrel without spilling its contents. Option: Allow the syringe to drain into a wide mouth beverage container. When successfully completed, the cap should rest upright on the bottom of the syringe with all reagent still in the cap.



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### 6. Install the plunger

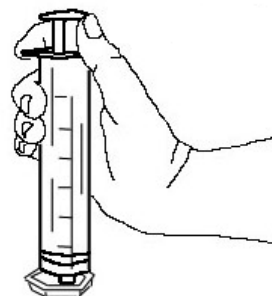
Install the plunger while maintaining the syringe in a vertical position. The plunger has a plastic “rib” near the rubber seal that snaps past the “catch” — a small ridge just inside the mouth of the syringe. Usually it takes a firm push to move the rib past the catch. After that, the plunger should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



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### 7. Draw aqueous reagent into syringe (Use 5 mL vinegar to make CO<sub>2</sub>)

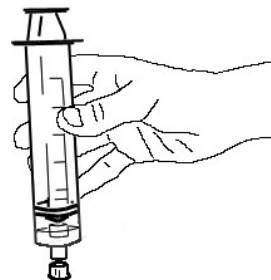
The aqueous reagent, measured into a small weighing dish, is drawn into the syringe while maintaining the vertical position of the syringe. The vial cap with the solid reagent should float on the solution.



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### 8. Install syringe cap

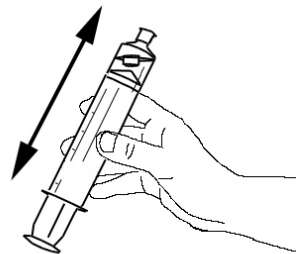
Push the syringe into the syringe cap. It simply pushes on.



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### 9. Generate the gas

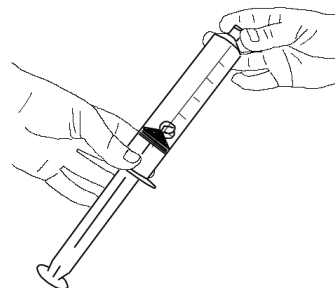
The “fun” part is generating the gas and you are ready to do that now. **Rotate the syringe so that the cap is up.** Shake the syringe in order to mix the reagents. As the liquid reagent splashes into the vial cap, gas generation will commence and the syringe plunger should move outward. It is sometimes necessary to gently help the plunger move up the barrel.



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### 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.

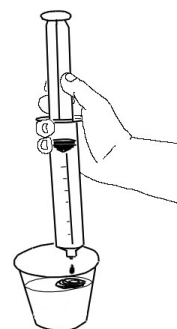


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### 11. Discharge reagents

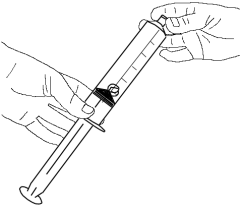
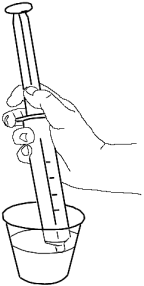
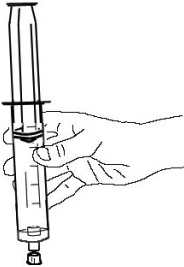
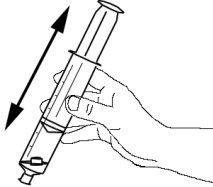
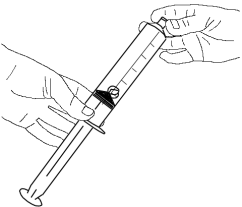
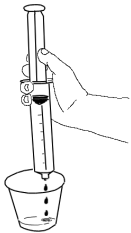
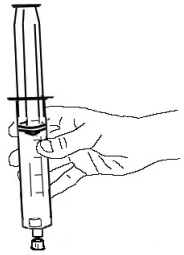
Turn the syringe 180° and discharge the liquid reagent into the plastic cup over half-filled with water in order to dilute the reactants. Immediately cap the syringe with the syringe cap to prevent loss of gas by effusion.

**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.**



## 12. Wash 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. To wash a gas:

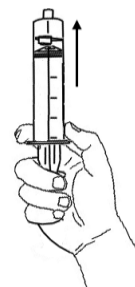
|   |  |  |  |
|---|--|--|--|
| 1. Remove the syringe cap,<br> | 2. draw 5 mL water into the syringe,<br>  | 3. cap the syringe,<br>    | 4. shake syringe to wash inside surfaces,<br> |
| 5. remove cap,<br>            | 6. discharge water only, and finally<br> | 7. recap the syringe.<br> | 8. Repeat?<br>One rinse is usually enough.   |

## OTHER USEFUL GAS SYRINGE TECHNIQUES

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

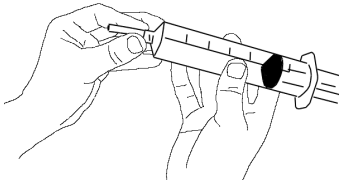
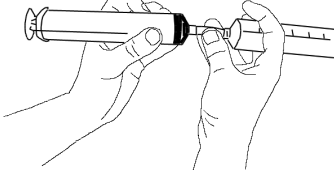
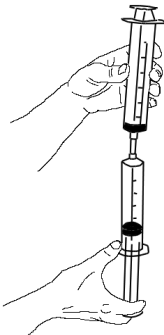
### A. Minimizing the quantity of air prior to reaction.

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.



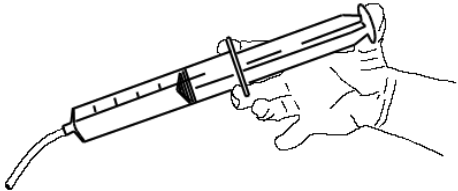
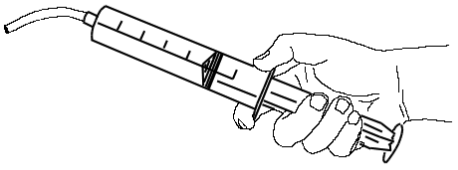
## B. Syringe-to-syringe transfer procedure

This procedure is useful under several circumstances. For example, when a gas sample needs to be in a clean, dry syringe, simply transfer the gas sample by a short connecting tube between the two syringes. This can be used instead of “washing” procedure described above.

|   |  |   |
|---|--|---|
| 1. Connect a short piece of tubing to a clean syringe.                            | 2. Connect gas-filled syringe to tubing:   | 3. Transfer the gas with clean, dry syringe on top:                                 |
|  |  |  |

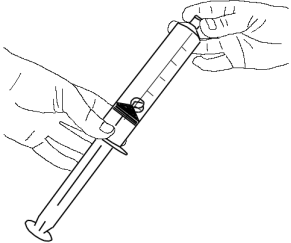
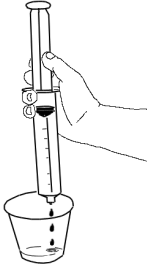
## C. Controlled discharge of gas from a syringe

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.

|   |  |
|---|--|
| <br><b>INCORRECT WAY</b> | <br><b>CORRECT WAY</b> |
|---|--|

## D. Safety First! Cap Up-and-Off! (Simply a repeat of Steps 10 and 11.)

If you used more than the recommended amount of the solid reagent, the reaction will generate more than 60 mL carbon dioxide. In anticipation of this scenario, be ready to perform Steps 10 and 11 from the procedure – and repeated here.

|   |   |
|---|---|
| <p>1. Position the syringe so the cap is directed upward.</p> <p>2. Remove the cap by twisting.</p>  | <p>3. Discharge the solution to prevent further gas collection.</p>  <p>4. Recap syringe.</p> |
|---|---|

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### 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.

### Disposal

Unwanted CO<sub>2</sub>(g) samples can be safely discharged into the room.

### Introductory Questions for high school students

1. Write the formulas for (a) baking soda; (b) vinegar; and (c) carbon dioxide.
2. Gases usually have “(g)” as the last part of their formula. For example, oxygen gas would be written as O<sub>2</sub>(g). In a similar way, solids have “(s)” and liquids have “(l)”. Aqueous solutions, substances dissolved in water, have “(aq)” as the last part of their formula. For example, salt water would be written as NaCl(aq). Add these endings to the three formulas in Question 1.
3. Why should the gas be “washed”?
4. Why is it important to use only the specified amounts of reagents?
5. Why must the syringe be upright when removing the cap?

## Questions

6. What was the purpose of vigorously shaking the syringe?
7. Why must one start over if some of the solid spills out while the vial cap of sodium bicarbonate is being lowered?
8. What is the molar mass of carbon dioxide?

## Advanced Questions

9. Write the balanced chemical equation for the reaction occurring in your syringe.
10. Using 0.22 g of  $\text{NaHCO}_3$  and 5.0 mL of 1.0 M HCl, which reactant is the limiting reactant?
11. Use the ideal gas law and your answer to the previous question to determine the volume of gas is predicted. Assume 25 °C and standard pressure.
12. Use the ideal gas law to determine the density of carbon dioxide at 25 °C and standard pressure.
13. Use the ideal gas law to determine the density of air at 25 °C and 1 atm pressure. You can use the “average molar mass of air” in your calculations; its value is 28.964 g/mol. Compare the density of carbon dioxide (Question 7) with the density of air. Calculate the ratio of densities,  $d_{\text{carbon dioxide}}/d_{\text{air}}$

## Internet reference

This chapter is available on the web at our microscale gas chemistry website:

[http://mattson.creighton.edu/Microscale\\_Gas\\_Chemistry.html](http://mattson.creighton.edu/Microscale_Gas_Chemistry.html)

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## Equipment Needed for Microscale Gas Chemistry Kits

This list summarizes all of the equipment that should be contained in each Microscale Gas Chemistry Kit. Items marked with \* are not necessary for generating carbon dioxide. Ordering information for kit materials is given below and in Appendix E.

| Item                                          | For Demo | For 5 pairs | For 10 pairs |
|-----------------------------------------------|----------|-------------|--------------|
| 8 cup plastic food storage container          | 1        | 5           | 10           |
| 60 mL plastic syringes with a LuerLOK fitting | 2        | 10          | 20           |
| Latex LuerLOK syringe caps                    | 2        | 10          | 20           |
| plastic vial caps                             | 2        | 10          | 20           |
| Latex tubing*                                 | 20 cm    | 100 cm      | 200 cm       |
| plastic pipet*                                | 1        | 5           | 10           |
| clear beverage cup                            | 1        | 5           | 10           |
| small plastic weighing dish                   | 1        | 5           | 10           |
| small test tube (12 x 100 mm)                 | 1        | 5           | 10           |
| medium test tube (18 x 150 mm)                | 1        | 5           | 10           |
| wide-mouth beverage bottles                   | 1        | 5           | 10           |

### Sources (partial list; full list in Appendix E)

- ❖ 60 mL plastic syringes with a LuerLOK fitting; Educational Innovations; GAS-140; Flinn AP8754; Fisher 14-823-2D
- ❖ Latex LuerLOK syringe caps; Educational Innovations GAS-160, ten-pack; Flinn AP8958, ten-pack
- ❖ plastic vial caps that fit within the barrel of the syringe; Educational Innovations GAS-180, ten-pack
- ❖ Latex tubing, 1/8-inch (3.175 mm) ID, 15 cm and 3 cm; Educational Innovations GAS-220; Flinn AP2076; 10-ft
- ❖ clear beverage cup, 9 oz (260 mL) grocery store

### Chemicals needed for preparation of carbon dioxide (enough to fill a syringe four times)

| Item                                 | For Demo | For 5 pairs | For 10 pairs |
|--------------------------------------|----------|-------------|--------------|
| sodium bicarbonate, $\text{NaHCO}_3$ | 1 g      | 5 g         | 10 g         |
| vinegar (or 1 M HCl)                 | 25 mL    | 125 mL      | 250 mL       |