# Microscale Gas Chemistry
# Supplement: Preparing Gas Samples

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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 stackable plastic food storage containers. Each kit should contain the following equipment. Ordering information, including part numbers, is available at our website. Several vendors sell outside of the USA. The most expensive item in this list is the syringe. When bought in bulk (4 boxes of 30), one can obtain these for just over a dollar each. Veterinarians use this size syringe and perhaps one could obtain used syringes from them.

- 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 clear plastic beverage cup (250 mL/9 oz)
- two small plastic weighing dishes

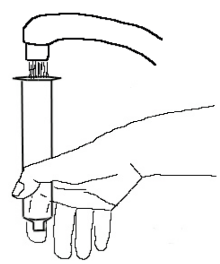
The general strategy is to place two substances in a 60 mL syringe and, when desired, cause them to mix and react. The limiting reagent is used in solid form and is placed in a small vial cap. The second reagent is prepared as an aqueous solution. For example, one could generate $CO_2(g)$ from excess aqueous acetic acid and solid $NaHCO_3$, as the limiting reagent.

**A. Getting started. Start by lubricating the seal.** Lubricate the black rubber seal of the plunger with silicone oil. (We skip this step nowadays as it is only necessary with very old syringes.)

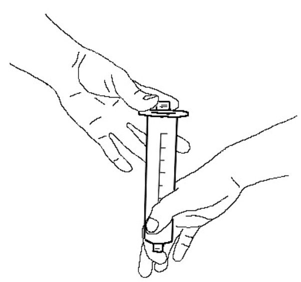
**B. The solid reagent.** The solid reagent is placed in the vial cap that is then lowered into the syringe barrel by water flotation. In the preparation of carbon dioxide, one would use a half-filled cap (approximately 0.2 g) baking soda, $NaHCO_3$. For most purposes, we seldom mass reagents anymore. If exact quantities are desired, the following quantities of solid reagents are required for $CO_2$, $H_2$ and $O_2$:

| **To make:** | **Use:** |
|---|---|
| Carbon dioxide | A half filled cap (or about 0.2 g) sodium bicarbonate (baking soda), $NaHCO_3(s)$ |
| Hydrogen | 0.05 g magnesium, Mg(s) turnings (or 4 average size turnings) |
| Oxygen | A half filled cap (or about 0.1 g) potassium iodide, KI(s) |

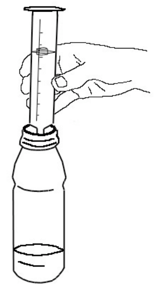
**Fill the syringe barrel with water.** Fill the barrel with water. Place a finger over the hole to form a seal. Fill completely to the top.



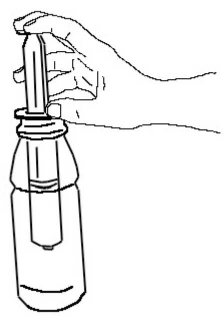
**Float the vial cap.** Float the vial cap containing the solid reagent on the water surface. This is easiest if the syringe barrel is filled completely to the top with water.



**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 of the reagent still in the cap.



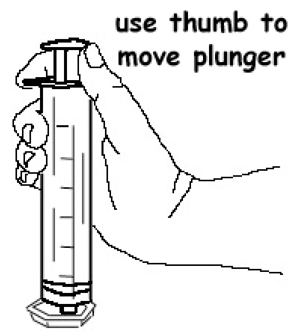
**Install the plunger.** Install the plunger while maintaining the syringe in a vertical position. The plunger should fit snugly against the rim of the vial cap.



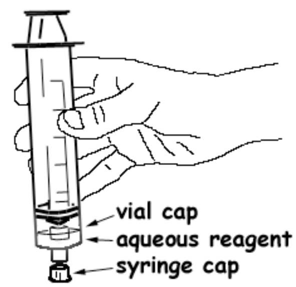
**The aqueous reagent.** The liquid reagent is drawn into the syringe as described below. In the preparation of carbon dioxide, one would use 5 mL vinegar, $HC_2H_3O_2$. The following aqueous reagents are required for $CO_2$, $H_2$ and $O_2$:

| **To make:** | **Use:** |
|---|---|
| Carbon dioxide | 5 mL acetic acid (vinegar), $HC_2H_3O_2(aq)$ |
| Hydrogen | 5 mL 2 M HCl(aq) |
| Oxygen | 5 mL 6% $H_2O_2(aq)$ |

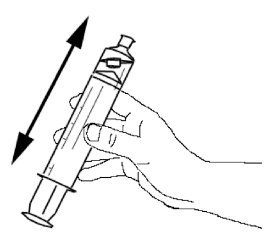
**Draw aqueous reagent into syringe.** The aqueous reagent, measured into a small weighing boat, is drawn into the syringe while maintaining the vertical position of the syringe. The vial cap with the solid reagent should float on the solution.



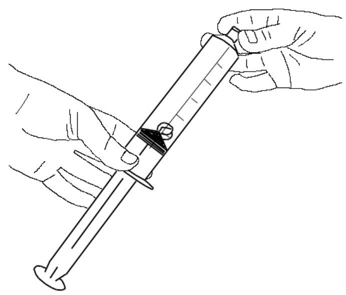

**Install syringe cap.** Push the syringe cap over the syringe fitting.  It simply pushes on!



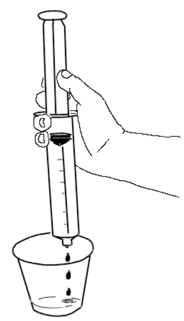

**Generating the Gas.** Turn the syringe so that it is cap up.  Is there too much air in the syringe?  This can happen during the previous step.  If so, remove the syringe cap and carefully discharge the excess air, while being careful not to tip the vial cap.  Recap the syringe.  Now you are ready to generate the gas!  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$^{o}$ 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.



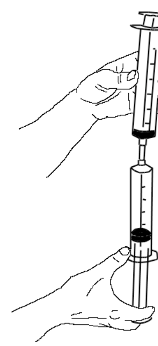
**Cleaning the gas sample.** The syringe filled with gas also contains droplets of excess reagents, and aqueous reaction products (sodium acetate in the case of $CO_2$, for example). There are two options: (1) Transfer the gas to a clean, dry syringe as described below; or (2) Wash away the drops of aqueous solutions. To do the latter, remove the syringe cap, draw 5 mL water into the syringe and recap. Shake the syringe to splash the water around, remove the cap and discharge the rinse water. For gases that are soluble in water, the first method must be used.

## Other useful gas syringe techniques.

There are a four other techniques that come in handy when working with gases in syringes.

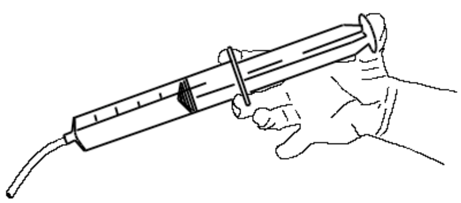
**A. Syringe-to-syringe Transfer procedure.**

This procedure is used when two gases are to be mixed or when a gas sample cannot be washed (because the gas is water-soluble). In these instances, transfer the gas sample to a clean, dry syringe by a short connecting tube between the two syringes. The clean, dry syringe is positioned on top. Usually pushing in on the lower plunger will cause the upper plunger to move outward. Sometimes assistance is needed.

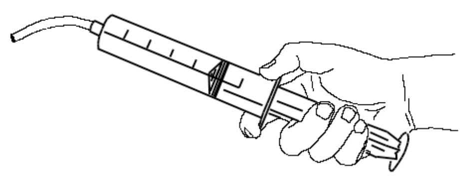


**B. Controlled discharge of gas from a syringe.**

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.



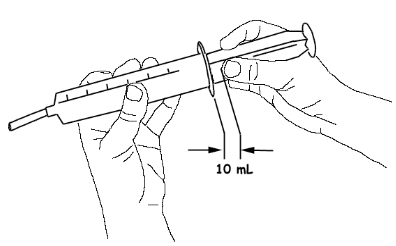
INCORRECT WAY



CORRECT WAY

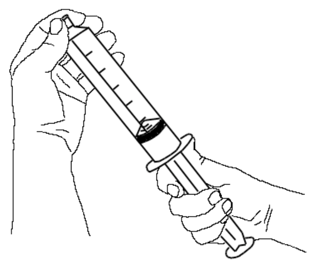
**C. Discharging a specific volume of gas.**

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

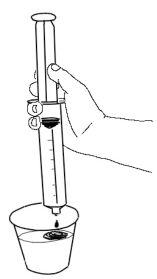


**D. PREVENTING UNWANTED DISCHARGES OF NOXIOUS GAS.**

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, $NO_2$, ammonia, $NH_3$. sulfur dioxide, $SO_2$, and hydrogen sulfide, $H_2S$. 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.



## 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. Store plunger out of barrel unless both are completely dry.

## Our Microscale Gas Chemistry Website.

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. Equipment ordering information and historical information are also available at the site. Use of the site is free.

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