PREPARATION OF OXYGEN

Source: from ***Microscale Gas Chemistry***, Educational Innovations, copyright Bruce Mattson, 2003
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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

Oxygen is non-toxic in normal quantities. Pure oxygen can be toxic if inhaled in large quantities as the pure gas, but this is not a concern with these experiments. Do not intentionally inhale oxygen samples produced in these experiments.

Syringe lubrication

We recommend lubricating the black rubber seal of the plunger with silicone oil.

Equipment

Gas Generator Kit

Chemicals (needed for each syringe full of oxygen generated)

0.05 g solid KI powder
5 mL 6% $H_2O_2(aq)$

The production of O_2 is ***slow*** and it typically takes a minute or more to fill a syringe. Assist the plunger in its outward movement. To speed up the reaction, hold the plunger so that the contents inside the syringe are under reduced pressure, and while doing so, tap or shake the syringe. This process drives oxygen bubbles out of the solution. Potassium iodide is the catalyst in the reaction:

$$2\ H_2O_2(aq) \longrightarrow 2\ H_2O(l) + O_2(g)$$

The actual mechanism has two steps:

$$\text{Step 1.}\ \ H_2O_2(aq) + I^-(aq) \longrightarrow H_2O(l) + IO^-(aq)$$

$$\text{Step 2.}\ IO^-(aq) + H_2O_2(aq) \longrightarrow I^-(aq) + H_2O(l) + O_2(g)$$

Oftentimes the solution takes on a yellow color due to $I_3^-(aq)$ that results form a competing side reaction:

$$2\ H^+(aq) + IO^-(aq) + 2\ I^-(aq) \longrightarrow I_3^-(aq) + H_2O(l)$$

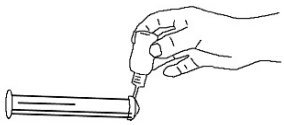
Generating oxygen gas samples

Samples of oxygen are generated by the In-Syringe Method. A summary of these steps is provided here:

1. Wear safety glasses!

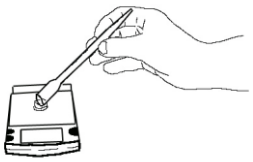
2. Lubricate the seal

Lubricate the black rubber seal of the plunger with silicone oil.



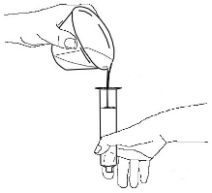
3. Measure out 0.05 g KI

Place the potassium iodide directly into the vial cap to prevent loss.



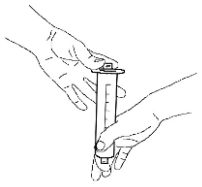
4. Fill the syringe barrel with water

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



5 Float the vial cap

Float the vial cap containing the solid reagent on the water surface.

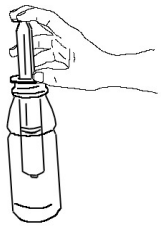


6. Lower the cap by flotation

Release the seal made by finger to lower the cap into the syringe barrel without spilling its contents.

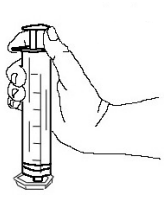
7. Install the plunger

Install the plunger while maintaining the syringe in a vertical position.



8. Draw 5 mL 6% H_2O_2(aq) into syringe

Pour the 6% H_2O_2(aq) into a small weighing dish. Draw 3 – 5 mL of the solution into the syringe.

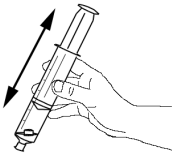


9. Install syringe cap

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

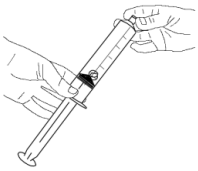
10. Generate the gas

Shake the device up and down in order to mix the reagents. Gently help the plunger move up the barrel.



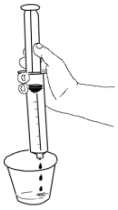
11. Remove cap to stop gas collection

Remove the syringe cap with the syringe held "cap-up" as shown. Assume contents are under positive pressure.



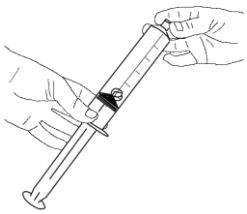
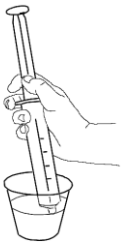
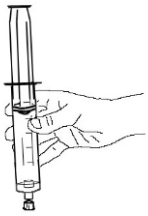
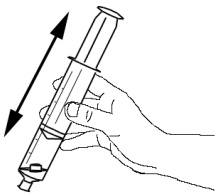
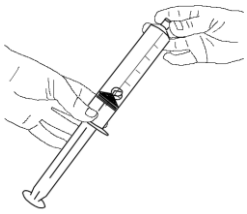
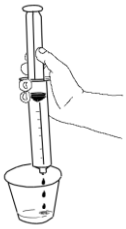
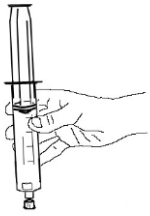
12. Discharge reagents

Discharge the liquid reagent into the plastic cup. Immediately cap the syringe to prevent loss of gas.



Wash away contaminants

Oxygen-filled syringes must be washed in order to remove traces of unwanted chemicals from the inside surfaces of the syringe before the gases can be used in experiments. Follow the procedure summarized here.

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

Disposal of oxygen samples

Unwanted oxygen samples can be safely discharged into the room.