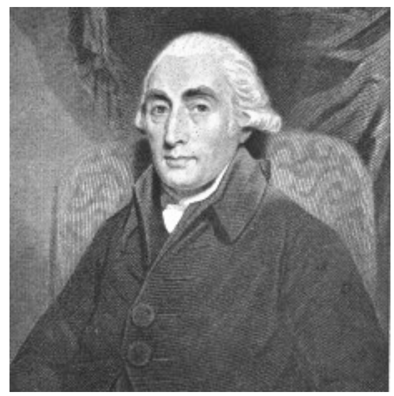
CHAPTER 2

EXPERIMENTS WITH CARBON DIOXIDE

CARBON DIOXIDE WAS DISCOVERED over 250 years ago by the 24-year old Englishman Joseph Black.[1] He prepared and characterized samples of $CO_2(g)$ which he called ***fixed air***. He found that the gas could be produced by heating chalk which lost mass during the heating process. We now know this reaction is:

$$CaCO_3(s) \rightarrow CaO(s) + CO_2(g)$$



Joseph Black

Carbon dioxide is a colorless gas present in our atmosphere at very low levels. The level of CO_2 have been rising throughout the 20th century which is believed to cause the *greenhouse effect* by which the earth's atmosphere is slowly warming up. Carbon dioxide is essentially odorless, however it causes a sharp sensation in one's nose when inhaled — such as from the bubbles of a carbonated beverage.

Carbon dioxide has many important uses. It is used in fire extinguishers, the soft drink industry, and as a chemical reagent to make other compounds. The major use of carbon dioxide is as a refrigerant (accounting for over 50%). ***Dry ice***, $CO_2(s)$ was first commercially introduced as a refrigerant in 1924. Dry ice sublimes to a gas at -78.5 ^oC at standard pressure. By the 1960s dry ice was replaced by liquid CO_2 (commonly called ***liquid carbonic***) as the most common CO_2-refrigerant. Carbon dioxide has a melting point of -56.6 ^oC at 5.2 atmospheres. Liquid CO_2 is used to freeze materials such hamburger meat and metals and this improves the grindability of the material. It is also used to rapidly cool loaded trucks and rail cars. Another 25% of all CO_2 produced

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[1] To learn more about Joseph Black, visit our gas website, http:// mattson.creighton.edu/Microscale_Gas_Chemistry.html and click on “History of Gas Chemistry”

is used in the soft drink industry. Carbon dioxide is now widely used as the propellant in aerosol cans.

Carbon dioxide is manufactured by the combustion of hydrocarbons such as natural gas:

$$CH_4(g) + 2\ O_2(g) \rightarrow CO_2(g) + 2\ H_2O(g) \qquad \Delta H = -803\ kJ$$

The solubility of $CO_2(g)$ in water is 3.48 g per L at 0 °C and 1.45 g/L at 25 °C. This is equivalent to 1.77 mL CO_2 per 1 mL water at 0 °C and 0.74 mL CO_2 per 1 mL water at 25 °C. When $CO_2(g)$ dissolves in water it produces $CO_2(aq)$ for the most part. Solutions of $CO_2(aq)$ last longer if they are kept cool. As the solution of $CO_2(aq)$ is warmed, $CO_2(g)$ is released as bubbles.

$$CO_2(aq) \rightleftarrows CO_2(g)$$

The density of $CO_2(g)$ is over 50% greater than that of air. At 25 °C and standard pressure, the density of carbon dioxide is 1.799 g/L, compared with 1.18 g/L for air.

Suitability

The following experiments are included in this chapter.
Experiment 1. Traditional limewater test for carbon dioxide
Experiment 2. Acidity of carbon dioxide
Experiment 3. Carbon dioxide extinguishes fires
Experiment 4. Carbon dioxide and aqueous sodium hydroxide react
Experiment 5. Equilibrium between carbon dioxide and carbonic acid

The first three experiments are suitable as laboratory experiments for a wide variety of grade levels from middle school up through university-level. Experiment 4 requires the use of a 6 M NaOH(aq) and should be used with prudent caution (or as a demo). It is also an example of a useful technique: Conducting a reaction with a gas inside a syringe. Experiment 5 makes an excellent classroom demonstration or laboratory experiment.

For use in high school settings, these experiments can be conducted at about the time that chemical formulas and reactions are being introduced. As a laboratory activity, these experiments are appropriate when discussing chemical compounds, chemical formulas, chemical reactions, the mole, as well as a variety of topics including physical and chemical changes. Experiment 5 can be discussed on several levels ranging from a simple discussion of how carbonated beverages contain aqueous carbon dioxide to the details of the carbon dioxide/carbonic equilibrium that can be a challenge even for university students.

Background skills required

Students should be able to:

- ❖ generate a gas as learned in Chapter 1
- ❖ measure quantities of liquid reagents
- ❖ use a balance
- ❖ identify a precipitate

Time required

Students should be able to perform all of these experiments in a single 45 minute laboratory period.

Website

This chapter is available on the web at website:

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

Instructions for your students

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

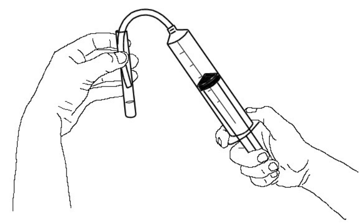
All of this will fit into the food storage container. In addition, each pair of students will need a wide-mouth beverage bottle for draining and supporting their syringes. Ordering information for kit materials is given at the end of this chapter and in Appendix E.

EXPERIMENTS WITH CARBON DIOXIDE[1]

EXPERIMENT 1. TRADITIONAL LIMEWATER TEST FOR CARBON DIOXIDE

Equipment

Microscale Gas Chemistry Kit



Chemicals

CO_2(g), 20 mL

limewater, 3 – 5 mL

Suitability

middle school lab, high school lab, university lab, and classroom demonstration

Applications, Topics, Purpose

chemical formulas, chemical properties of gases, types of chemical reactions, precipitation reactions, characterization of gases

Instructions

Prepare a syringe full of CO_2. Pour 1 – 2 mL of limewater, $Ca(OH)_2$(aq), into a test tube. Remove the syringe cap and attach a 15 cm length of tubing to the syringe. Discharge 10 – 20 mL CO_2 over the limewater surface as shown in the figure. Stopper the test tube with your thumb or finger. These chemicals are not dangerous if contacted to skin. Shake the gas and liquid. Notice the production of precipitated $CaCO_3$, which makes the solution cloudy. The reaction is:

$$Ca(OH)_2(aq) + CO_2(g) \rightarrow CaCO_3(s) + H_2O(l)$$

Save the syringe of unused carbon dioxide for the next experiment.

Teaching tips

1. Limewater is saturated $Ca(OH)_2$. Limewater should be clear — not cloudy. See appendix for construction of a limewater dispenser from a plastic beverage bottle.

Introductory Questions

1. What is a precipitate?
2. What is limewater?
3. What is the formula of carbon dioxide?
4. What do the symbols (aq), (g), (s) and (l) stand for in the equation for the reaction given above?

[1] Content for this chapter first appeared as “Microscale Gas Chemistry, Part 2. Experiments with Carbon Dioxide” Mattson, B. M., ***Chem13 News***, **252**, November, 1996.

Questions

5. Would the precipitate settle if allowed to stand for a period of time?
6. What does calcium carbonate look like?
7. What is the formula of the carbonate ion?
8. What makes this a good test for carbon dioxide?
9. What is the traditional test for the carbonate ion?

Advanced Questions

10. Carbon dioxide is a covalent molecular compound. What class of compound is calcium carbonate?
11. Write the chemical reaction that took place in the form of a sentence: *"Aqueous calcium hydroxide and …."*

EXPERIMENT 2. ACIDITY OF CARBON DIOXIDE

Equipment

Microscale Gas Chemistry Kit
100 mL graduate cylinder
plastic square, 5 cm x 5 cm (for Part 2; cut from a sandwich bag or food wrap, etc.)
rubber band (Part 2)

Chemicals

$CO_2(g)$, 20 - 40 mL
universal indicator solution, 10 mL (or red cabbage juice solution)
concentrated ammonium hydroxide solution, (only the NH_3 fumes will be used)

Suitability

middle school lab, high school lab, university lab, and classroom demonstration

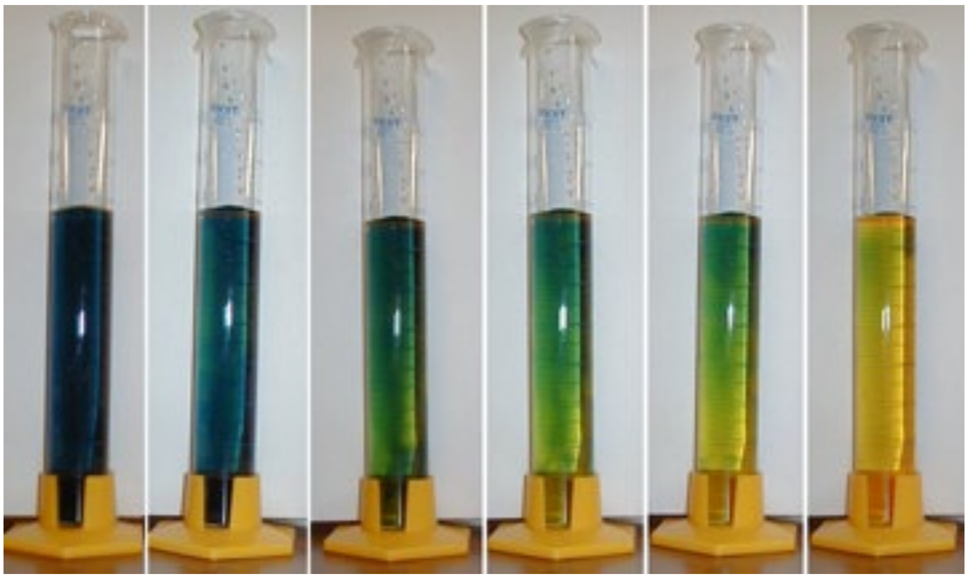
Applications, Topics, Purpose

acid-base properties of gases, chemical properties of gases, solubility of gases, acid-base reactions, indicators, acidic nature of non-metal oxides

Instructions

Part 1. Prepare a syringe full of carbon dioxide or use the leftover carbon dioxide from Experiment 1. Add 2 mL water to the test tube. Add 10 drops of Universal Indicator solution. Use a plastic pipet to transfer some ammonia vapors to the indicator solution. Stopper the test tube with your thumb or finger. Shake the contents of the test tube to assure mixing. Next, transfer 20 mL CO_2 above the surface of the solution using the long tubing. Recall the correct way to dispense gas: Grasp the plunger and pull the barrel towards you. By holding the syringe opening upward, no liquid is accidentally discharged. Shake the contents of the test tube to assure mixing.

Part 2. Prepare 75 mL of the NH_3-vapors/indicator solution and transfer it to a graduated cylinder. Discharge the CO_2 above the surface of the solution and cover the graduated cylinder with the plastic square and rubber band. Swirl gently to agitate the surface a small amount. Layers of color will develop.



Teaching tips

1. Red cabbage juice solution works as well as universal indicator solution. See Appendix D for instructions.
2. You may wish to prepare a large quantity of the NH_3-vapors/indicator solution for all to use.
3. Chart of indicator color vs. the corresponding pH.

Indicator Colors

pH	Universal	Red Cabbage
4.0	Red	Red
5.0	Orange Red	Purple
6.0	Yellow Orange	Purple
7.0	Dark Green	Purple
8.0	Light Green	Blue
9.0	Blue	Blue-Green
10.0	Reddish Violet	Green
11.0	Violet	Green
12.0	Violet	Green
13.0	Violet	Green-Yellow
14.0	Violet	Yellow

Introductory Questions

1. Explain why carbonated beverages are slightly acidic.
2. Would vinegar, which contains acetic acid, cause universal indicator to be violet?
3. Suppose your friend tested the pH of carbonated water as per this experiment. Suppose also that your friend did not remember whether he/she used universal indicator or red cabbage indicator, however, the solution is purple. Which indicator did he/she use and why do you know?

Questions

4. Does ammonia seem more soluble than carbon dioxide?
5. Why does the indicator solution eventually turn the color associated with acid?

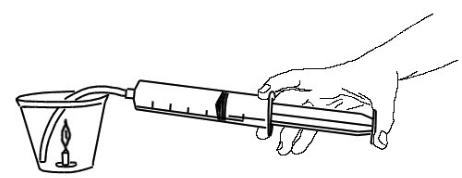
Advanced Questions

6. What is the pH of the distilled water in your laboratory? Explain why.
7. What does carbon dioxide form when it dissolves in water?

EXPERIMENT 3. CARBON DIOXIDE EXTINGUISHES FIRES

Equipment

Microscale Gas Chemistry Kit
Matches or lighter
Small beaker (~100 mL), plastic cup or jar



Chemicals

CO_2(g), 50 mL

Suitability

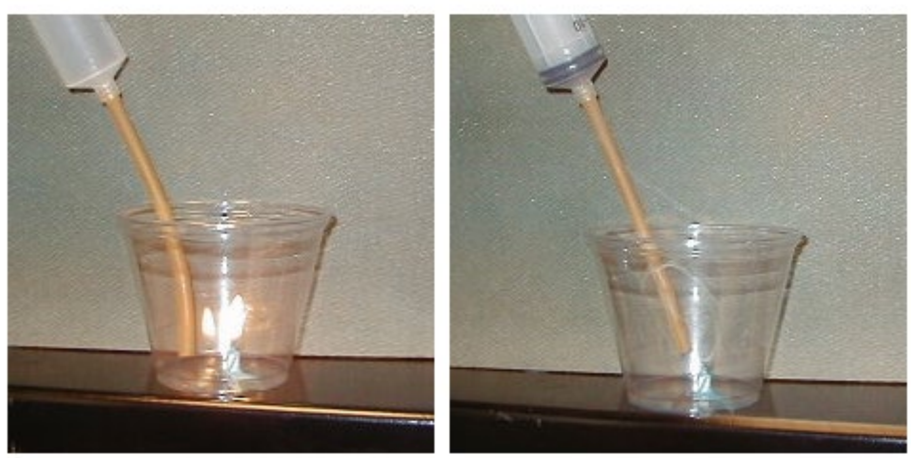
middle school lab, high school lab, university lab, and classroom demonstration

Applications, Topics, Purpose

Gas density, physical properties of gases, chemical properties of gases, how fire extinguishers work, combustion

Instructions

If necessary, prepare a syringe full of carbon dioxide. Affix a short candle to a coin with a drop of molten wax. Place the candle/coin into the cup as shown. Ignite the candle. Equip the syringe with the tubing and transfer the CO_2 to the bottom of the cup.



Variation 1. Discharge the entire contents of the syringe in a quick manner — within a second. The flame will go out. (The picture shows the gas being discharged in the "incorrect way" according to the discussion in the previous experiment. We usually do not discharge gas in this manner; however in this experiment rapid discharge is crucial.)

Variation 2. This variation works because CO_2 is more dense than air. Your syringe must be nearly full of carbon dioxide (60 mL). Remove the plunger from the syringe and immediately invert the syringe above the beaker so that the carbon dioxide pours into the beaker with the burning candle (capped end up). This demonstrates that $CO_2(g)$ is denser than air. The candle should immediately go out.

Teaching tips

1. Prepare the coin/candle devices for your students: Using a scissors, cut a candle to a length of 1.5 cm including the wick. By partially bearing down on the scissors, the wax portion of the candle will be cut, but not the wick. Light the candle and allow a drop of hot candle wax to fall on the coin. Push the base of the candle into the molten wax.

2. Explain that combustion is the reaction of oxygen with another substance, often organic. Combustion is simply a rapid oxidation of the organic compound and reduction of oxygen. Combustion is stopped because the oxygen is replaced by the carbon dioxide and carbon dioxide is not a combustible gas.

Introductory Questions

1. Can a candle burn in carbon dioxide? Does carbon dioxide burn?

2. Can a candle burn in oxygen?

3. What happened to the burning candle? Could carbon dioxide be used as a fire extinguisher?

4. Why should you release the carbon dioxide in the bottom of the cup (Variation 1)?

Questions

5. Why was it important to use a short candle?

6. Which gas has a greater density, carbon dioxide or air? How could you tell? Hint: Compare the molar masses of oxygen and nitrogen with carbon dioxide.

Advanced Question

7. If the carbon dioxide is discharged slowly rather than quickly, this experiment will not work. Explain why. Sketch the flow of gases around a heat source.

8. Design an experiment to determine whether carbon dioxide or air has the greater density.

9. Look up Lake Nyos in Africa on the web. Periodically, this lake releases vast quantities of carbon dioxide that has killed people and animals nearby.

EXPERIMENT 4. CARBON DIOXIDE AND AQUEOUS SODIUM HYDROXIDE REACT

Equipment

Microscale Gas Chemistry Kit

Chemicals

CO_2(g), 50 mL

sodium hydroxide, NaOH(aq), 6 M, 10 mL; See Precautions

Suitability

high school lab, university lab, and classroom demonstration

Precautions

Aqueous sodium hydroxide, NaOH(aq) (6 M) is a caustic substance that can cause serious damage to skin and eyes. Use care when handling this chemical. Always wear safety glasses!

Applications, Topics, Purpose

chemical formulas, chemical properties of gases, types of chemical reactions, balancing chemical reactions, household chemicals, to illustrate that the carbon dioxide is no longer in the gas phase after it reacts with the sodium hydroxide

Instructions

Prepare a syringe full of carbon dioxide. Fill a small weighing dish with 6 M NaOH(aq). Draw 5 mL NaOH(aq) into a CO_2-filled syringe. Fit the syringe with a syringe cap. Shake the syringe. The plunger will move inward as the CO_2(g) reacts with aqueous NaOH(aq) forming $NaHCO_3$(aq) and/or Na_2CO_3(aq). The reaction is:

$$2\ NaOH(aq) + CO_2(g) \rightarrow Na_2CO_3(aq) + H_2O(l)$$

Teaching tips

As CO_2(g) reacts with NaOH(aq), the pressure in the syringe decreases forcing the plunger inward.

Introductory Questions

1. What are the formulas for sodium hydroxide and sodium carbonate?
2. Did the carbon dioxide dissolve in the solution or react with the solution?
3. Why does shaking the syringe speed up the reaction?

Questions

4. Suggest an explanation for what you observed in this experiment.

5. Solutions of bases such as sodium hydroxide or calcium hydroxide are not "stable" if they sit in the air for an extended period of time. Based on your experiments with CO_2(g) suggest a reason for this.

Advanced Questions

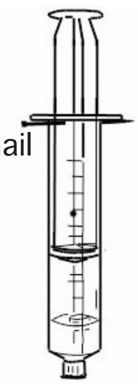
6. Carbon dioxide is a covalent molecular compound. What class of compound is sodium carbonate?

7. Write the chemical reaction that took place in sentence form: *"Aqueous sodium hydroxide and …"*

EXPERIMENT 5. CARBON DIOXIDE/CARBONIC ACID EQUILIBRIUM

Equipment

Plastic cup, 9-ounce (250 mL)
special purpose syringe for Variation 1 (with nail through plunger)



Chemicals

CO_2(g), 40 mL for Variation 1; 30 mL for Variations 2 and 3
ice water for Variation 1 only

Suitability

high school lab, university lab, and classroom demonstration

Applications, Topics, Purpose

carbonation and soft drinks, chemical formulas, chemical properties of gases, gas solubility, household chemicals

Instructions

This experiment is described in three variations. Variation 1 takes about an hour to perform (mostly waiting) but gives the most impressive results. Variation 2 takes just a few minutes and gives guaranteed results. Variation 3 is similar to Variation 2, but requires a syringe in which the plunger can move rather easily in the barrel (such as a new syringe). In all variations, the equilibrium involves CO_2 as the primary aqueous species. Approximately one CO_2(aq) in 600 exists as H_2CO_3(aq):

$$CO_2(g) \rightleftharpoons CO_2(aq) \rightleftharpoons H_2CO_3(aq)$$

Variation 1. This experiment requires the special nail-syringe and takes about an hour. Transfer 40 mL CO_2(g) into the special purpose syringe. Pull 10 mL water into the syringe and install the syringe cap. Push the plunger inward until the nail can be inserted into the middle hole in the plunger as shown in the figure. The volume of the gas should be compressed to 20 mL or less. Place the syringe into a large container of crushed ice and water. Allow the system to come to equilibrium over the next 30 minutes. Remove from the ice and allow to warm to room temperature for 15 minutes. Next pull the plunger up to the 50 mL mark and insert the nail in the hole near the rubber seal. Tap the syringe on the countertop. You should see bubbles of CO_2 swirling out of solution. The process can be repeated.

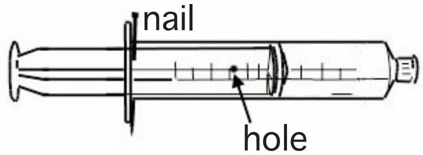
Variation 2. Draw 30 mL water into a syringe containing 30 mL carbon dioxide and fasten the syringe cap. Hold the plunger and barrel together so that the plunger cannot move. Gently rock the syringe back and forth while it is held in a horizontal position, maximizing the surface area between the water and the gas. After about a minute or two, hold the syringe vertically with the syringe cap directed downward. Remove the syringe cap – bubbles of air should stream up through the aqueous solution in the syringe.

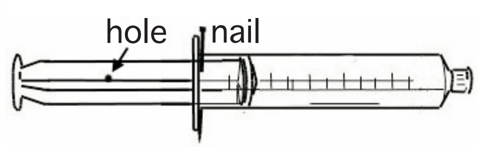
Variation 3. Draw 30 mL water into a syringe containing 30 mL carbon dioxide. Record the combined volume — which should be 60 mL. Gently rock the syringe back and forth while it is held in a horizontal position, maximizing the surface area between the water and the gas. Within less than a minute the 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. How much carbon dioxide dissolves?

All variations. 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.

Teaching tips

1. The special purpose syringe is constructed by drilling two holes through the plunger, one is drilled in such a position that the syringe will hold about 30 mL when the nail is flush with the rim of the barrel (left figure). Use a drill bit that is somewhat larger in diameter than the nail that is to be used. Drill the second hole in such a position as shown in the right figure below: the syringe will hold about 55 mL when the nail is flush with the rim of the barrel (left figure).





2. One can tap the syringe with knuckles as well. Avoid hitting the syringe cap.

Questions for Variation 1.

1. Why did we use ice in this experiment? Is carbon dioxide more soluble in cold or warm water?
2. Why was pressure used?
3. What familiar product is based on the solubility of carbon dioxide at low temperature and high pressure?
4. What happens to an opened bottle or bottle of soda pop if it is allowed to warm up?
5. When the syringe was tapped, carbon dioxide bubbles were formed. What analogy exists with the properties of soda pop?

Questions for Variations 2 and 3.

1. Why did you gently rock the syringe in order to dissolve the carbon dioxide? Why wouldn't it work to shake the syringe?
2. Variation 2: Why did you observe bubbles perculating through the solution when the syringe cap was removed? Variation 3: Why did the plunger move inward?
3. What familiar product contains aqueous carbon dioxide?
4. What happens to an opened bottle or bottle of soda pop if it is allowed to warm up?

Advanced Question

It is known that if the syringe cap is left off, carbon dioxide will diffuse at a rate of about 0.3 mL per minute. Reviewing the experiments performed in this chapter, suggest an experiment that could be used to verify this statement.

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.

SUMMARY OF MATERIALS AND CHEMICALS NEEDED FOR CHAPTER 2. EXPERIMENTS WITH CARBON DIOXIDE

Equipment required

This list summarizes all of the equipment necessary for performing the reactions with carbon dioxide described in this chapter.

Item	For Demo	For 5 pairs	For 10 pairs
Microscale Gas Kit	1	5	10
wooden splint	1	5	10
special purpose syringe plus finishing nail, 5 cm (Option for Experiment 5)	1	5	10
matches or lighter	1	5	10
100 mL graduate cylinder	1	5	10

Materials required

Item	For Demo	For 5 pairs	For 10 pairs
short candle on coin	1	5	10
white 6-oz cup	1	5	10
plastic square, 5 cm x 5 cm	1	5	10
rubber band	1	5	10
ice water	1 cup	-	-

Chemicals required

Item	For Demo	For 5 pairs	For 10 pairs
sodium bicarbonate, $NaHCO_3$	2 g	10 g	20 g
vinegar	25 mL	125 mL	250 mL
limewater (Appendix D)	2 mL	10 mL	20 mL
universal indicator solution*	10 mL	50 mL	100 mL
concentrated ammonium hydroxide solution	a	a	a
sodium hydroxide, 6 M NaOH	10 mL	50 mL	100 mL

* or cabbage juice solution

a. only the NH_3 fumes from the concentrated ammonium hydroxide solution will be used