

Working in the Chemistry Laboratory

EXPERIMENT 1A

One of the important components of your chemistry course is the laboratory experience. Perhaps you have done experiments in other science courses, so you are somewhat familiar with some of the lab equipment. The chemistry laboratory is unique, however. There is a great deal to be learned about the equipment and safety procedures in a chemistry lab.

A number of important procedures need to be mastered in order for you to be successful in the later experiments of this course. You need to know how to use a lab burner; how to filter liquids; how to manipulate glass tubing; how to handle solid chemicals and solutions; and how to heat materials safely in the chemistry laboratory. In addition, it is important that you learn to measure using graduated cylinders, thermometers, and balances.

In this introductory experiment, you will have a chance to learn about all of these lab techniques while doing an experiment involving an important chemical reaction. Limewater is a solution of calcium hydroxide, $\text{Ca}(\text{OH})_2$, dissolved in water. Limewater is used as a test for carbon dioxide, an important product of animal respiration. When carbon dioxide is bubbled through some limewater, a cloudy appearance is noted. This is called a *precipitate*.

OBJECTIVES

1. to learn the following lab techniques:
 - a. using a lab burner
 - b. using a funnel and filter paper
 - c. bending and fire-polishing glass tubing
 - d. handling solid chemicals and solutions
 - e. measuring mass with a balance
 - f. measuring temperature with a thermometer
 - g. measuring volume with a graduated cylinder
2. to correctly set up the equipment in order to perform an experiment
3. to observe the reaction between limewater and carbon dioxide
4. to test the gaseous products of two chemical reactions for the presence of carbon dioxide

MATERIALS

Apparatus

lab burner	ring stand and ring
centigram balance	wire gauze
flame spreader	thermometer
funnel	2 beakers (250-mL)
filter paper	spark lighter or matches
stirring rod	file for glass tubing
glass tubing (6-mm)	graduated cylinder (100-mL)
2 Erlenmeyer flasks (125-mL)	metric ruler
one-hole rubber stopper, to fit Erlenmeyer flask	marking pen
two-hole rubber stopper, to fit Erlenmeyer flask	lab apron
rubber or plastic tubing	safety goggles
	plastic gloves
	funnel holder
	folded cloths or towels
	face shield

Reagents

1M HCl (hydrochloric acid)
magnesium ribbon
glycerin
sodium carbonate
calcium hydroxide

PRELAB

Answer questions 1-8 on the Report Sheet.

PROCEDURE

Part I

Figure 1A-1 Two common laboratory burners: (a) the Bunsen burner, (b) the Tirrill burner.

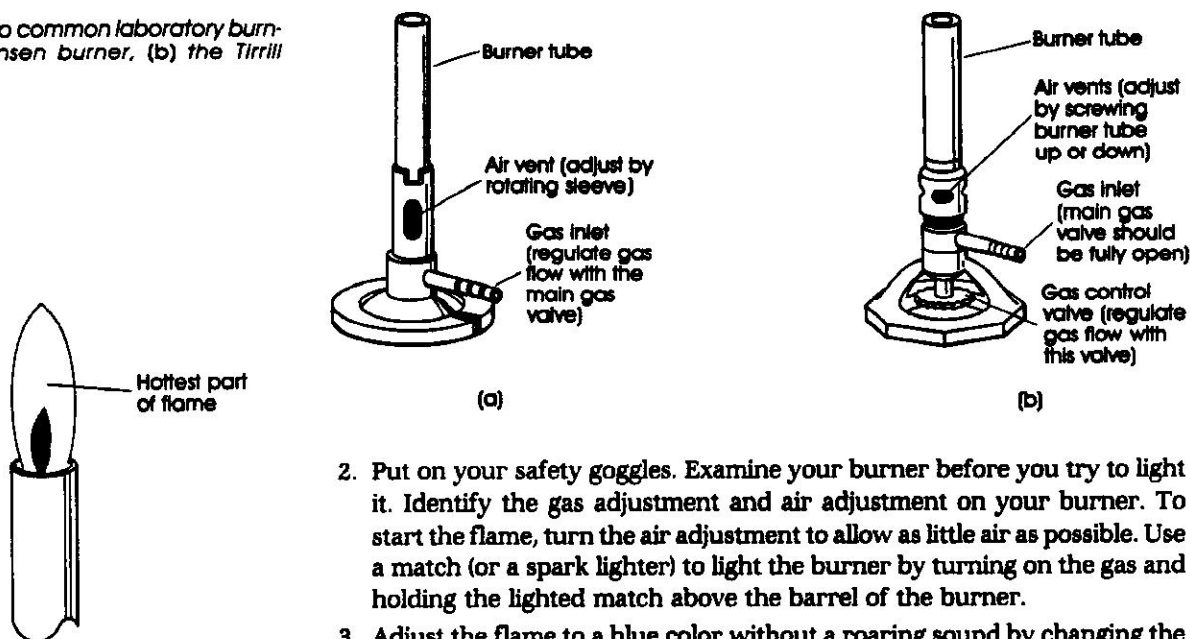


Figure 1A-2 The hottest part of the flame produced by a laboratory burner is just above the tip of the inner (blue) cone.

1. Most lab burners are constructed in a similar fashion. There is an inlet for the gas, an adjustment for the amount of gas, and an adjustment for the amount of air. (See Figure 1A-1.) A proper mix of air and gas will yield a faint blue flame for maximum heat and minimum soot.

2. Put on your safety goggles. Examine your burner before you try to light it. Identify the gas adjustment and air adjustment on your burner. To start the flame, turn the air adjustment to allow as little air as possible. Use a match (or a spark lighter) to light the burner by turning on the gas and holding the lighted match above the barrel of the burner.
3. Adjust the flame to a blue color without a roaring sound by changing the amount of air and the amount of gas. Note, for future reference, where the flame is hottest (Figure 1A-2).
4. Turn off the burner and go on to the next part of the experiment.

Part II



CAUTION: To avoid cuts, wear gloves when you break glass tubing.



CAUTION: Be careful with hot glass tubing. Place the hot pieces on a piece of wire gauze or other fireproof material and be careful to wait for each piece to cool down.

1. Obtain a piece of glass tubing that is 50 cm long. Using a metric ruler and a marking pen, mark the tubing for cutting into two 20-cm pieces and one 10-cm piece.
2. Place the tubing on a firm surface as shown in Figure 1A-3. Using a single firm stroke of a triangular file, make a deep scratch at the point where you want to cut the glass tubing. Wearing gloves, hold the tubing with both thumbs behind the scratch. (The scratch should be pointing away from you.) Push firmly with your thumbs and the tubing should break cleanly.
3. Continue at each of the marks on your tubing.
4. In order to smooth the ends of the glass tubing, a technique called fire-polishing is used. Light the burner again and place the end of one piece of tubing into the flame. Rotate the tubing so that the heating is even. You will quickly notice that the end of the tubing is becoming smooth. Do not leave the glass in the flame for too long or the end of the tube will close. Continue the fire polishing with each piece of tubing.

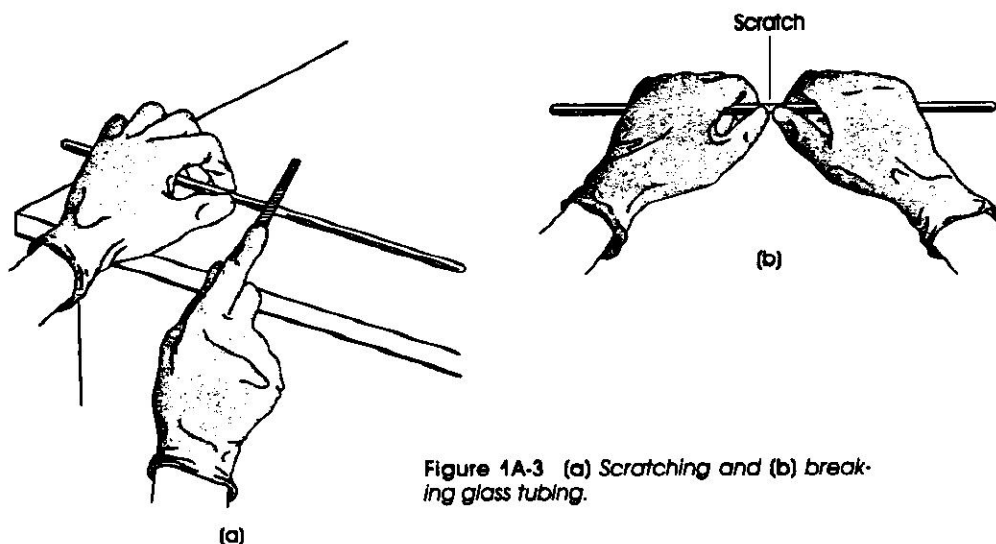


Figure 1A-3 (a) Scratching and (b) breaking glass tubing.

5. Turn off the burner and place the flame spreader on the top of the burner, as in Figure 1A-4a. This will allow you to bend the glass tubing more easily.
6. Hold one of the 20-cm pieces of the tubing in both hands and place the center of the tubing in the flame while rotating the tubing, as in Figure 1A-4b. Continue rotating until you feel the tubing getting soft. At this point, remove the tubing from the flame and bend it at a 90-degree bend in one smooth motion. Repeat with the other 20-cm piece.
7. Allow the bends to cool. Then place one or two drops of glycerin into the holes of a two-hole rubber stopper. The glycerin acts as a lubricant for the glass tubing. Protecting both hands with folded cloths, put one of the right-angle bends into one of the holes, extending the tubing as far as possible into the stopper. It is very important that care be taken to gently push the tubing into the rubber stopper. In the other hole, place the 10-cm piece so that it is just below the stopper. (See Figure 1A-5.) Save the second 20-cm bend for Part V.

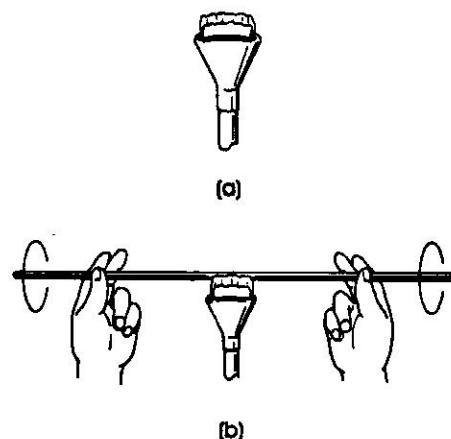
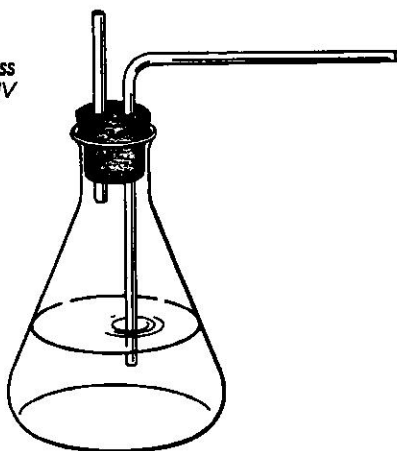


Figure 1A-4 (a) Properly adjusted flame with a flame spreader on the burner. (b) How to heat glass tubing before bending it.

Figure 1A-5 Two-hole stopper with glass tubing, inserted in a flask for use in Parts IV and V.



CAUTION: Protect your hands with cloth pads when inserting glass tubing into a stopper. Use glycerin as a lubricant. Protect your hands whenever you are adjusting or removing the tubing.

Part III

1. In order to make the limewater solution, you must be able to use the lab balance. A picture of a common balance is shown in Figure 1A-6.
2. The balance must be zeroed before it is used to find masses. Place the balance on a level table and find the zero-adjusting screw. Adjust the balance to the zero point. Probably the balance that you are using has three beams on it. In order to properly record the mass, you must add up the masses indicated by the riders on the beams.

Figure 1A-6 A centigram balance.

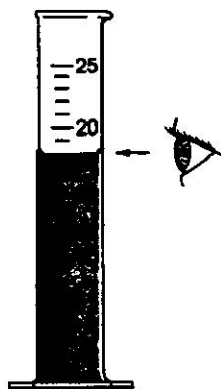
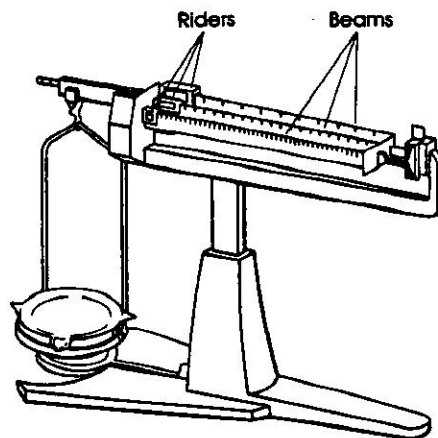


Figure 1A-7 When you read a graduated cylinder, your eyes should be at the same level as the top of the liquid, and the scale reading should correspond to the bottom of the meniscus. In this example, the correct reading is 18.0 mL.



CAUTION: Calcium hydroxide and limewater are both irritating to skin and eyes. Wear safety goggles, lab apron, and gloves. Do not touch the chemical; use a scoop or spatula for the calcium hydroxide. If you get any on your skin or in your eyes, wash it off immediately with plenty of water.

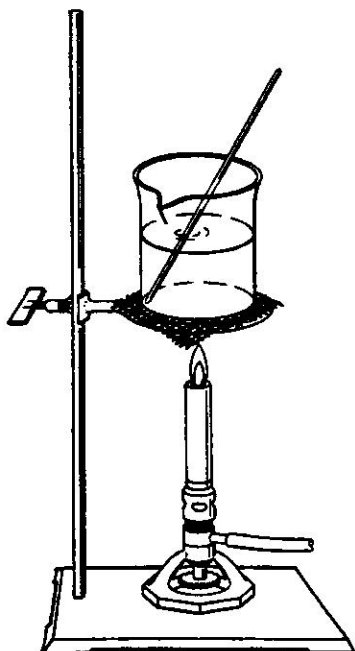


Figure 1A-8 Setup for heating the limewater solution.

3. When you find the mass of solid chemicals, it is important that you do not put the chemical directly on the pan or platform of the balance. Place a clean, dry 250-mL beaker on the pan of the balance and find the mass of the beaker. Record it on the Report Sheet.
4. Now, get approximately 3 g of calcium hydroxide from your instructor and find the mass of the beaker plus the calcium hydroxide. Record it on the Report Sheet.
5. Using the graduated cylinder, place 175 mL of distilled water into the 250-mL beaker. It is important that you know how to read a graduated cylinder. Graduated cylinders are designed to read correctly only if the bottom of the meniscus is the point where the volume is recorded. A *meniscus* is the slight curve that appears because the water is slightly higher on the surface of the glass and therefore lower in the middle of the cylinder. See Figure 1A-7 for the correct reading of the graduated cylinder.
6. Using a stirring rod, begin stirring the solution.
7. Calcium hydroxide is difficult to dissolve. Heating the solution will speed up the dissolving process. Set up the ring stand and ring with the wire gauze as shown in Figure 1A-8. Heat the solution gently, stirring constantly. Heat for approximately 5-10 min. Even after 10 min., you will probably find that some of the solid has not dissolved. Put the beaker down to cool and allow the solid to settle while you set up a funnel and filter paper to obtain a clear solution of limewater.
8. Set up the filtration apparatus shown in Figure 1A-10. Filter paper is supplied in the shape of a circle. You need to fold it two times, then open up the paper so that it forms a cone. (See Figure 1A-9a.) Before you pour the solution into the funnel, you should moisten the filter paper with a few drops of water, so that the filter paper will stick to the funnel, as in Figure 1A-9b.
9. Pour the solution into the funnel as shown in Figure 1A-10 and allow it to filter into the beaker below. You will need the clear limewater for Parts IV and V.

Figure 1A-9 (a) Folding filter paper. (b) Fitting a moistened filter paper cone to a funnel.

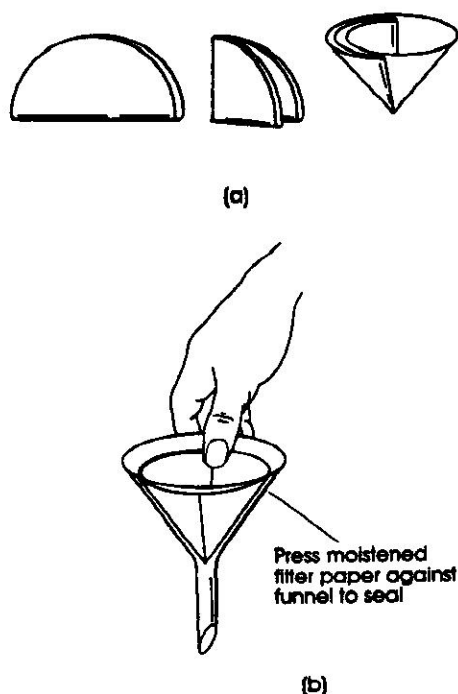
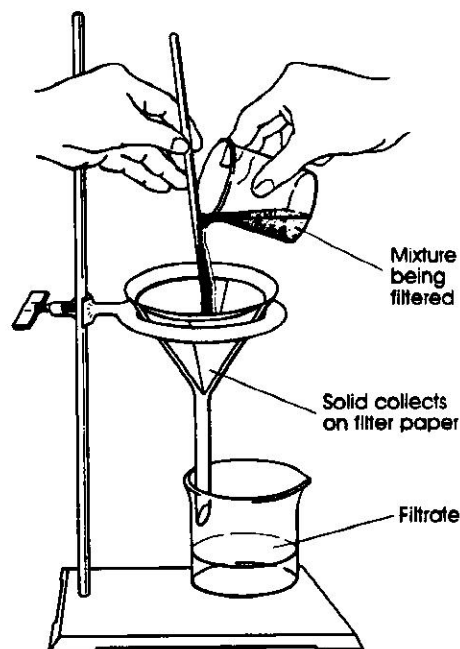


Figure 1A-10 When filtering, use a glass stirring rod to control the flow of the solution into the funnel. If the stem of the funnel touches the side of the beaker, the liquid flows more smoothly into the beaker.



Part IV

1. Place just enough of the clear limewater into the flask so that the glass tubing is just below the liquid when the stopper that contains the glass tubing is inserted in the flask. Put the stopper on the flask tightly.
2. Gently blow into the right-angle bend. You will exhale carbon dioxide (among other gases). Continue to exhale until you observe a change. Write down your observations on the Report Sheet.

Part V

1. Place the second 90-degree bend into the one-hole stopper, using the glycerin as a lubricant. Again, be very careful to *gently* push the tubing into the rubber stopper. If you encounter any resistance, put more glycerin on the glass tubing. Attach a short piece of rubber or plastic tubing to the ends of both of the 90-degree bends and set up the flasks as shown in Figure 1A-11.
2. Rinse out the flask that you used for the limewater and fill it with fresh, clear limewater to the same level.
3. In the other flask, place 35 mL of 1M hydrochloric acid.
4. With a thermometer, measure the temperature of the acid and record it on the Report Sheet. Be sure to rinse the end of the thermometer after removing it from the acid.
5. Obtain a piece of magnesium ribbon from your instructor. Carefully drop the magnesium into the acid and quickly place the stopper on the top of the flask. Record your observations on the Report Sheet.
6. As soon as the reaction stops, remove the stopper and measure the temperature of the acid. Record it on the Report Sheet.

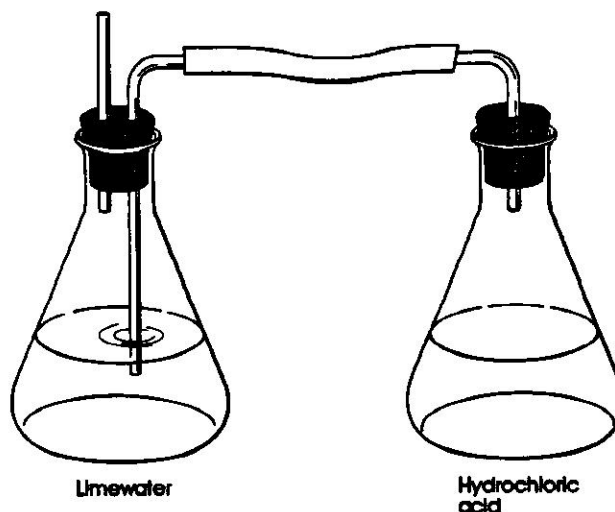


CAUTION: Protect your hands with cloth pads when inserting glass tubing into a stopper.



CAUTION: Hydrochloric acid is corrosive to skin, eyes, and clothing. When handling 1M hydrochloric acid, wear safety goggles, lab apron, gloves, and use a full face shield. Wash off spills and splashes with plenty of water. Call your teacher.

Figure 1A-11 Setup for testing gases with limewater.



CAUTION: Always protect your hands with cloth pads when removing glass tubing from a stopper.

7. Rinse out all of the glassware and repeat Steps 2, 3, and 4. Then obtain approximately 2 g of sodium carbonate. Carefully pour the sodium carbonate into the flask and quickly put the stopper on the flask. Record your observations on the Report Sheet.
8. As soon as the reaction stops, remove the stopper and measure the temperature of the acid. Record it on the Report Sheet.
9. Rinse out all of the glassware and put away all of the equipment.
10. Before you leave the laboratory, wash your hands thoroughly with soap and water, using a fingernail brush to clean under your fingernails.

POST LAB DISCUSSION

Both reactions that you observed in Part V involved the formation of a gas. You observed in Part IV that carbon dioxide causes limewater to turn cloudy. This should help you in determining if carbon dioxide is formed in either of the last two reactions.

Reactions that give off heat are called *exothermic*, while reactions that absorb heat are *endothermic*.

The precipitate that forms when carbon dioxide reacts with limewater is calcium carbonate. Calcium carbonate is insoluble in water.