

Laboratory Procedures

The best way to become familiar with chemical apparatus is to handle the pieces yourself in the laboratory. This experiment is divided into several parts in which you will learn how to adjust the gas burner, insert glass tubing into a rubber stopper, use a balance, handle solids, measure liquids, and filter a mixture.

Great emphasis is placed on safety precautions that should be observed whenever you perform an experiment and use the apparatus. In many of the later experiments, references will be made to these "Laboratory Techniques."

In later experiments you will also be referred to the safety precautions and procedures explained in all parts of this experiment. It is important that you develop a positive approach to a safe and healthful environment in the lab.

OBJECTIVES

Observe proper safety techniques with all laboratory equipment.

Use laboratory apparatus skillfully and efficiently.

Recognize the names and functions of all apparatus in the laboratory.

Develop a positive approach toward laboratory safety.



Always wear safety goggles and a lab apron to protect your eyes and clothing. If you get a chemical in your eyes, immediately flush the chemical out at the eyewash station while calling to your teacher. Know the location of the emergency lab shower and eyewash station and the procedures for using them.



Do not touch any chemicals. If you get a chemical on your skin or clothing, wash the chemical off at the sink while calling to your teacher. Make sure you carefully read the labels and follow the precautions on all containers of chemicals that you use. Do not taste any chemicals or items used in the laboratory. Never return leftover chemicals to their original containers; take only small amounts to avoid wasting supplies.



Do not heat glassware that is broken, chipped, or cracked. Use tongs or a hot mitt to handle heated glassware and other equipment because hot glassware does not always look hot.

When using a Bunsen burner, confine long hair and loose clothing. If your clothing catches on fire, WALK to the emergency lab shower and use it to put out the fire.

When heating a substance in a test tube, the mouth of the test tube should point away from where you and others are standing. Watch the test tube at all times to prevent the contents from boiling over.



Never put broken glass in a regular waste container. Broken glass should be disposed of separately according to your teacher's instructions.

PART 1—THE BURNER

MATERIALS

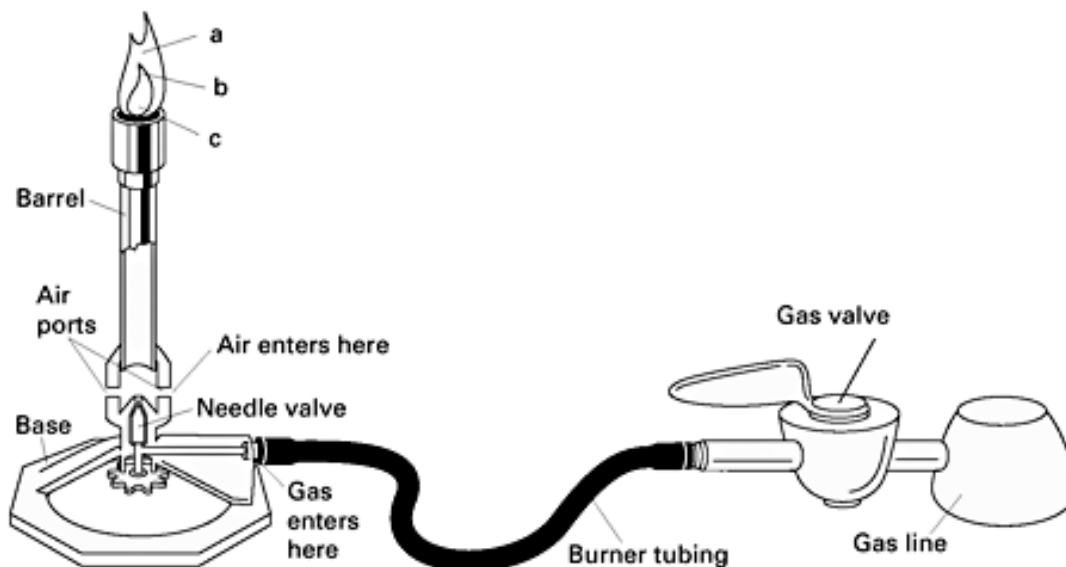
- Bunsen burner and related equipment
- copper wire, 18 gauge
- evaporating dish
- crucible tongs (not beaker tongs)
- heat-resistant mat
- cloth towel
- lab apron
- safety goggles
- sparker

Procedure

1. Put on safety goggles, and a lab apron. Use heat-resistant gloves when handling hot items.
2. The Bunsen burner is commonly used as a source of heat in the laboratory. Look at **Figure 1** as you examine your Bunsen burner and identify the parts. Although the details of construction vary among burners, each has a gas inlet located in the base, a vertical tube or barrel in which the gas is mixed with air, and adjustable openings or ports in the base of the barrel. These ports admit air to the gas stream. The burner may have an adjustable needle valve to regulate the flow of gas. In some models the gas flow is regulated simply by adjusting the gas valve on the supply line. The burner is always turned off at the gas valve, never at the needle valve.

CAUTION: Before you light the burner, check to see that you and your partner have taken the following safety precautions against fires: Wear safety goggles, aprons, and heat-resistant gloves. Confine long hair and loose clothing: tie long hair at the back of the head and away from the front of the face, and roll up long sleeves on shirts, blouses, and sweaters away from the wrists. You should also know the locations of fire extinguishers, fire blankets, safety showers, and sand buckets and the procedure for using them in case of a fire.

Figure 1 (LEARN THE PARTS OF A BUNSEN BURNER!!)



3. When lighting the burner, partially close the ports at the base of the barrel, turn the gas full on, hold the sparkler about 5 cm above the top of the burner, and proceed to light.

The gas flow may then be regulated by adjusting the gas valve until the flame has the desired height. If a very low flame is needed, remember that the ports should be partially closed when the gas pressure is reduced. Otherwise the flame may burn inside the base of the barrel. When the flame is improperly burning in this way, the barrel will get very hot, and the flame will produce a poisonous gas, carbon monoxide.

CAUTION: If the flame is burning inside the base of the barrel, immediately turn off the gas at the gas valve. Do not touch the barrel, because it is extremely hot. Allow the barrel of the burner to cool, and then proceed as follows:

Begin again, but first decrease the amount of air admitted to the burner by partially closing the ports. Turn the gas full on, and then relight the burner. Control the height of the flame by adjusting the gas valve. By taking these steps, you should acquire a flame that is burning safely and is easily regulated.

4. Once you have a flame that is burning safely and steadily, you can experiment by completely closing the ports at the base of the burner. What effect does this have on the flame?

Using the tongs (not beaker tongs!!), hold an evaporating dish in the tip of the flame for about 2 min. Place the dish on a heat-resistant mat and allow the dish to cool. Then examine the bottom of the dish. Describe the results and suggest a possible explanation.

Such a flame is seldom used in the lab. For laboratory work, you should adjust the burner so that the flame is free of yellow color, nonluminous, and also free of the roaring sound caused by admitting too much air.

5. Regulate the flow of gas so that the flame extends roughly 8 cm above the barrel. Now adjust the supply of air until you have a noisy, steady flame with a sharply defined, light-blue inner cone. This adjustment gives the highest temperature possible with your burner. Using the forceps, insert a 10-cm piece of copper wire into the flame just above the barrel. Lift the wire slowly up through parts of the flame. Where is the hottest portion of the flame located?

Hold the wire in this part of the flame for a few seconds. What happens?

6. Shut off the gas burner. Now think about what you have just observed in steps 4 and 5. Why is the nonluminous flame preferred over the yellow luminous flame in the laboratory?

7. Clean the evaporating dish and put away the burner. All the equipment you store in the lab locker or drawer should be completely cool, clean, and dry. Be sure that the valve on the gas jet is completely shut off. Remember to wash your hands thoroughly with soap at the end of each laboratory period.

PART 2—GLASS MANIPULATION (*Read only*)

MATERIALS

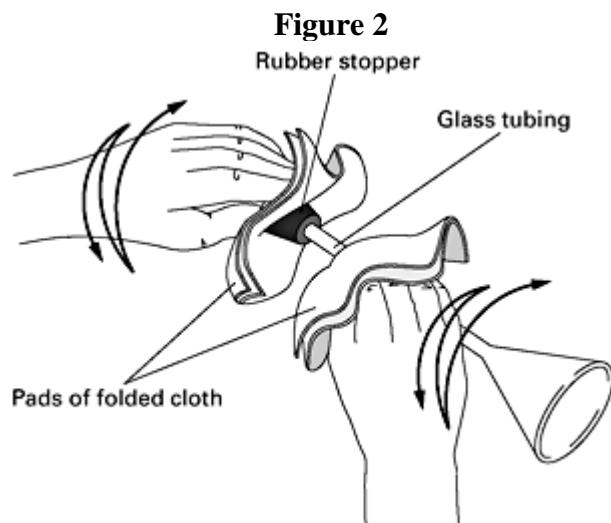
- cloth pads or leather gloves
- glass funnel
- rubber hose
- rubber stopper, 1-hole
- safety goggles & lab apron
- water or glycerin

Procedure

1. Inserting glass tubing into rubber stoppers can be very dangerous. The following precautions should be observed to prevent injuries:
 - a. Never attempt to insert glass tubing that has a jagged end. Glass tubing should be fire polished before it is inserted into a rubber stopper. To fire polish glass tubing, heat the end in a flame until the end is smooth. **Never fire polish anything without your teacher's permission, and proper supervision. Use tongs or a hot mitt to handle heated glassware and other equipment, because heated glassware does not look hot.**
 - b. Use water or glycerin as a lubricant on the end of the glass tubing before inserting it into a rubber stopper. Ask your teacher for the proper lubricant.

CAUTION: Protect your hands and fingers when inserting glass tubing into a rubber stopper.

- c. Wear leather gloves or place folded cloth pads between your hands and the glass tubing. Hold the glass tubing as close as possible to the part where it is to enter the rubber stopper. Always point the glass tubing away from the palm of your hand that holds the stopper, as shown in **Figure 2** below. Using a twisting motion, gently push the tubing into the stopper hole.
- d. At the end of the experiment, put on leather gloves, or place folded cloth pads between your hands and the glass tubing, and remove the rubber stoppers from the tubing to keep them from sticking or “freezing” to the glass. Use a lubricant, as directed in **step 1b**, if the stopper or tubing won't budge.



- When inserting glass tubing into a rubber or plastic hose, observe the same precautions discussed in **steps 1a–1c**. The glass tubing should be lubricated before inserting it into the rubber or plastic hose. The rubber hose should be cut at an angle before the insertion of the glass tubing. The angled cut in the hose allows the rubber to stretch more readily.

CAUTION: Protect your hands when inserting or removing glass tubing.

At the end of an experiment, immediately remove the glass tubing from the hose. When disassembling, follow the precautions that were given in **step 1d**.

Carefully follow these precautions and techniques whenever an experiment requires that you insert glass tubing into either a rubber stopper or a rubber or plastic hose. You will be referred to these safety precautions, wherever appropriate, throughout the lab course.

PART 3—HANDLING SOLIDS (Read only)

MATERIALS

- Weigh boat
- lab apron
- safety goggles
- salt and sand mixture
- spatula

Procedure

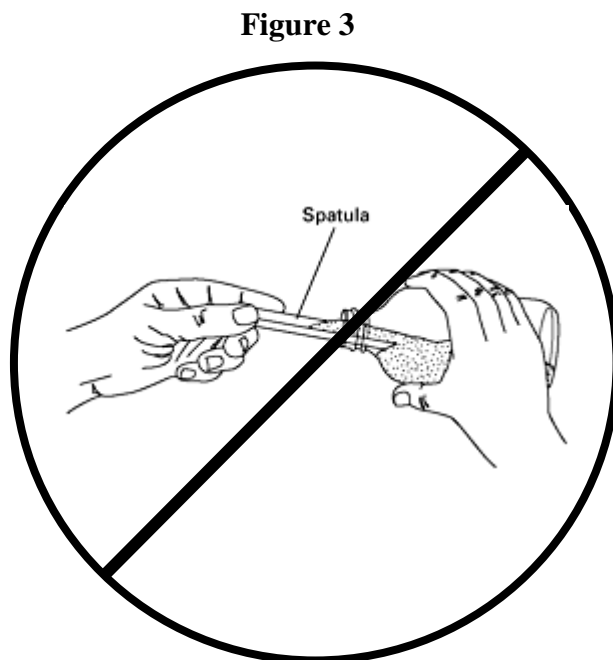
- Solids are usually kept in wide-mouthed bottles. Read the label at least three times to make sure you have the correct chemical.

Using a spatula, remove a quantity of sodium chloride from the container the teacher has provided to your weigh boat. NEVER insert a spatula, pipet, or any other apparatus into the main reagent bottle – see **Figure 3**.

CAUTION: Do not touch chemicals with your hands. Some chemical reagents readily pass through the skin into the bloodstream and can cause serious health problems. Some chemicals are corrosive. Always wear an apron, gloves, and safety goggles when handling chemicals. Carefully check the label on the reagent bottle or container before removing any of the contents. Never use more of a chemical than directed. You should also know the locations of the emergency lab shower and eyewash station and the procedures for using them in case of an accident.

CAUTION: Never try to pour a solid from a bottle into a test tube. As a precaution against contamination, never pour unused chemicals back into their reagent bottles.

CAUTION: Never discard chemicals or broken glassware in the waste paper basket. This is an important safety precaution against fires, and it prevents personal injuries (such as hand cuts) to anyone who empties the wastepaper basket.



PART 4—THE BALANCE

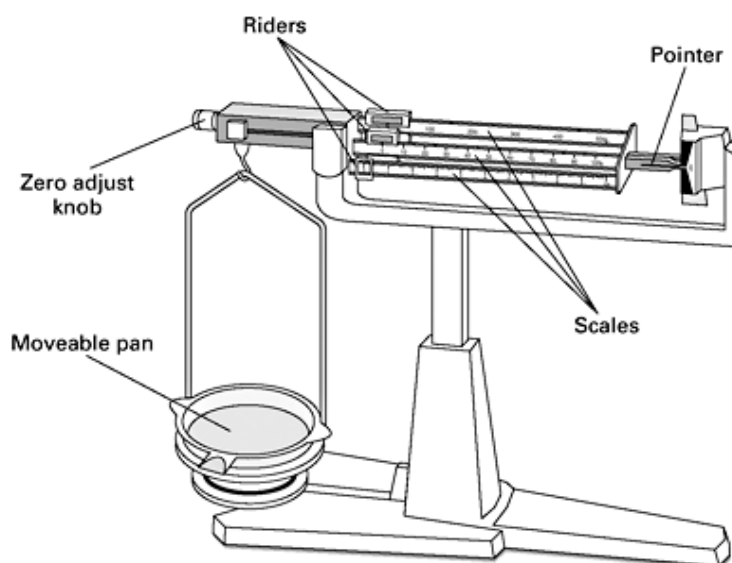
MATERIALS

- balance, centigram
- weigh boat
- lab apron
- safety goggles
- salt and sand mixture
- spatula

Procedure

1. Make sure you have carefully read Part 3 before you begin this part.
2. When a balance is required for determining mass, you will use a centigram balance like the one shown in **Figure 5**. The centigram balance can estimate to 0.001 g. This means that your mass readings should all be recorded to the nearest 0.001 g.
3. Before using the balance, always check to see if the pointer is resting at zero. If the pointer is not at zero, check the riders on the scales. If all the scale riders are at zero, turn the zero-adjust knob until the pointer rests at zero. The zero-adjust knob is usually located at the far left end of the balance beam, as shown in **Figure 5**. Note: The balance will not adjust to zero if the movable pan has been removed. **Never place chemicals or hot objects directly on the balance pan.** Always use weighing paper or a glass container. Chemicals can permanently damage the surface of the balance pan and affect the accuracy of measurements.

Figure 5 (LEARN THE PARTS OF A BALANCE!!)



4. In many experiments, you will be asked to determine the mass of a specified amount of a chemical solid. Use the following procedure to obtain approximately 13 g of mixture.
 - a. Using a spatula, obtain a quantity of salt/sand mixture from the container the teacher has provided and place it in a separate weigh boat. About a heaping teaspoon is a good amount to start with. You can always come back for more if you need it.
 - b. Make sure that the pointer on the balance is set at zero. Obtain a weigh boat and place it on the balance pan. Determine the mass of the weigh boat by adjusting the riders on the various scales. On the next page, record the mass of the weigh boat to the nearest 0.001 g.

- c. Move the riders on the balance to read 13 grams by sliding the rider on the 100 g scale to 10 and the rider on the 10 g scale to 3. The riders must fit into the slot with the number showing through the window.
- d. Now slowly pour the salt and sand mixture from the weigh boat onto the second weigh boat on the balance pan until the pointer once again comes to zero. You can bend the weigh boat to make a spout or you can use your spatula.

This time you will only have to be close to the specified mass. Do not waste time trying to obtain exactly 13.000 g. Instead, when you think you have almost 13 g, move the other riders until the pointer rests close to zero and then read the exact mass. For example, the mass might be 13.183 g. Record your exact mass of sodium chloride and the weighing paper to the nearest 0.001 g. (Hint: Remember to subtract the mass of the weighing paper to find the mass of sodium chloride.)

Mass of weigh boat: _____

Mass of salt/sand mixture and weigh boat: _____

Mass of salt/sand mixture alone: _____

- e. Save your sample for Part 6. If you will be doing Part 6 at a later time, be sure and put your sample in a labeled zip-top bag and place it in your lab drawer. The label should have both your name and your partner's name and your class period.
 - f. Return any excess chemicals to the excess chemical container at the front lab station.
5. Wash your hands thoroughly with soap and water at the end of each lab period.

PART 5—MEASURING LIQUIDS

MATERIALS

- beaker, 250 mL
- graduated cylinder, 100 mL
- lab apron
- thin stem pipet
- safety goggles
- water

Procedure

1. For approximate measurements of liquids, a graduated cylinder, such as the one shown in **Figure 6**, is generally used. These cylinders are usually graduated in milliliters (mL), reading from the bottom up. They may also have a second column of graduations reading from top to bottom. Examine your cylinder for these markings. Record the capacity of your cylinder and describe the smallest graduation it can read accurately in the space below.

Observations:

Figure 6

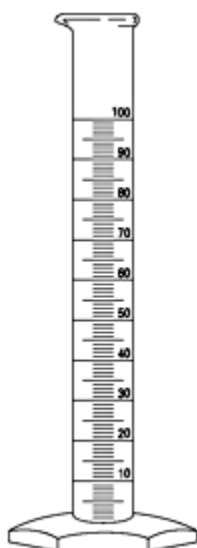


Figure 7



2. A pipet may be used for more accurate volume measurements. Pipets, which are made in many sizes, are used to deliver measured volumes of liquids. A pipet may be fitted with a suction bulb, as shown in **Figure 7**, or be made with a bulb – such as the thin stem pipet in your lab station. The bulb is used to withdraw air from the pipet while drawing up the liquid to be measured.

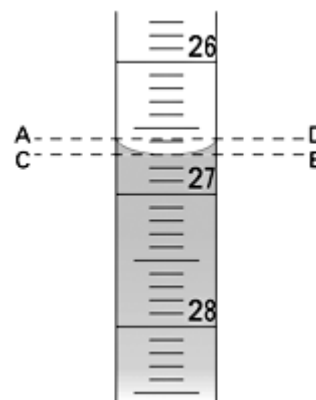
CAUTION: Always use the suction bulb. NEVER pipet by mouth.

- a. Accurately measure a volume of water in your graduated cylinder by following these steps:

CAUTION: When obtaining liquid chemical, first read the label three times to make sure you have the right chemical. Never put a pipet into the reagent bottle. Never pour any unused liquid back into the reagent bottle.

- a. Pour a quantity of liquid that you want to measure from the liquid's reagent bottle into a beaker. In this case, use more than 50 mL but less than 100 mL. (NOTE: The beaker will only ESTIMATE the volume of water.) You can use a pipet to help add or take away the liquid to get a more accurate reading.
- b. Notice that the surface of a liquid in the graduated may be slightly curved. It is concave if it wets the glass and convex if it does not wet the glass. Such a curved surface is called a meniscus.
- c. Get eye level with the meniscus and read the bottom of the meniscus that is curved downward, as shown in **Figure 9**. This is the line *CB*. If you read the markings at the top of the meniscus, *AD*, you will get an incorrect reading. Locate the bottom of the meniscus and read the water level in your graduated cylinder. (*You read the top of a meniscus that is curved upward – but ordinarily, liquids in our lab will curve downward.*)

Figure 9



Graduated cylinder reading: _____

CAUTION: In many experiments, you will have to dispose of a liquid chemical at the end of a lab. Always ask your teacher about the correct method of disposal. In many instances, liquid chemicals can be washed down the sink's drain by diluting them with plenty of tap water. Toxic chemicals should be handled only by your teacher. All apparatus should be washed, rinsed, and dried.

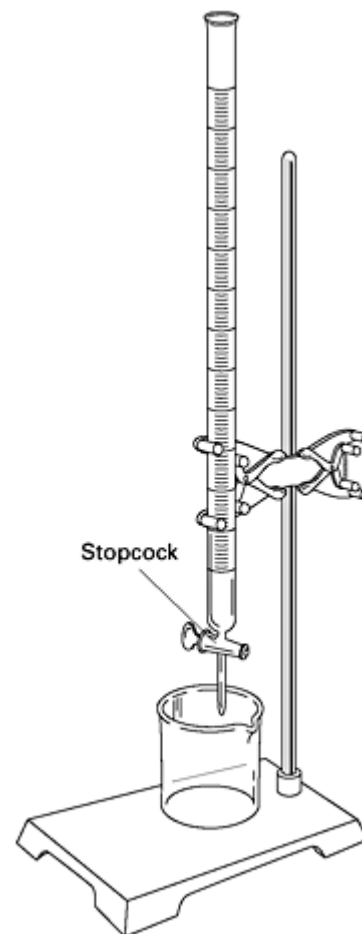
4. **(Read only)** A buret is also used for more accurate volume measurements. Burets are used for delivering any desired quantity of liquid up to the capacity of the buret. Many burets are graduated in tenths of milliliters. When using a buret, follow these steps:

- Clamp the buret in position on a ring stand, as shown in **Figure 8**.
- Place a 250-mL beaker under the tip of the buret. The beaker serves to catch any liquid that is released.
- Pour a quantity of liquid that you want to measure from the liquid's reagent bottle into a 50-mL beaker. (NOTE: In this first trial, you will use water.) Pour the liquid from the beaker into the top of the buret, being careful to avoid spills. (NOTE: Always carefully check the label of any reagent bottle before removing any liquid).

CAUTION: Never pour a liquid directly from its reagent bottle into the buret. You should first pour the liquid into a small, clean, dry beaker (50 mL) that is easy to handle. Then pour the liquid from the small beaker into the buret. This simple method will prevent unnecessary spillage. Never pour any unused liquid back into the reagent bottle.

- Fill the buret with the liquid and then open the stopcock to release enough liquid to fill the tip below the stopcock and bring the level of the liquid within the scale. The height at which the liquid stands is then read accurately. Practice this procedure several times by pouring water into the buret and emptying it through the stopcock.
5. Notice that the surface of a liquid in the buret is slightly curved. It is concave if it wets the glass and convex if it does not wet the glass. Locate the bottom of the meniscus that is curved downward and read the water level in your buret.
6. After you have taken your first buret reading, open the stopcock to release some of the liquid. Then read the buret again. The exact amount released is equal to the difference between your first and final buret reading. Practice measuring liquids by measuring 10 mL of water, using a graduated cylinder, a pipet, and a buret.
7. At the end of this part of the experiment, the equipment you store in the lab locker or drawer should be clean, dry, and arranged in an orderly fashion for the next lab experiment.
8. Remember to wash your hands thoroughly with soap at the end of this part of the experiment.

Figure 8



PART 6—FILTRATION & EVAPORATION

MATERIALS

- salt/sand mixture from Part 4
- beaker, 250 mL (2)
- filter paper
- funnel
- glass stirring rod
- pipe stem triangle
- iron ring
- ring stand
- lab apron
- safety goggles

Procedure

1. Sometimes liquids contain solids that are present either as impurities or as precipitates formed from chemical reactions in the experiment. If the solid particles are denser than water, they soon sink to the bottom. Let the solid particles settle to the bottom of the liquid and then gently pour off the liquid as carefully as you can without disturbing the solid. **This method of separation is known as decanting.**
2. Fine particles, or particles that settle slowly, are often separated from a liquid by filtration. Support a funnel on a small ring on the ring stand, as shown in **Figure 10**. **If your iron ring is too large, place a pipe stem triangle over the iron ring to hold up the funnel.** Use a beaker to collect the clear liquid that will come through the filter. **This liquid is called the filtrate.** Adjust the funnel so that the stem of the funnel just touches the inside wall of the beaker.
3. To prepare your filter paper, first ONLY hold it by the very edges. The oil from your fingers will clog the filter paper and water will not be able to go through it. Use the edge of a pencil or pen to make creases in your filter paper, instead of using your fingers.
4. Fold a circular piece of filter paper along its diameter, and then fold it again to form a quadrant, as shown in **Figure 11**. Separate the folds of the filter paper, with three thicknesses on one side and one on the other; then place the resulting filter paper cone in the funnel.

Figure 10

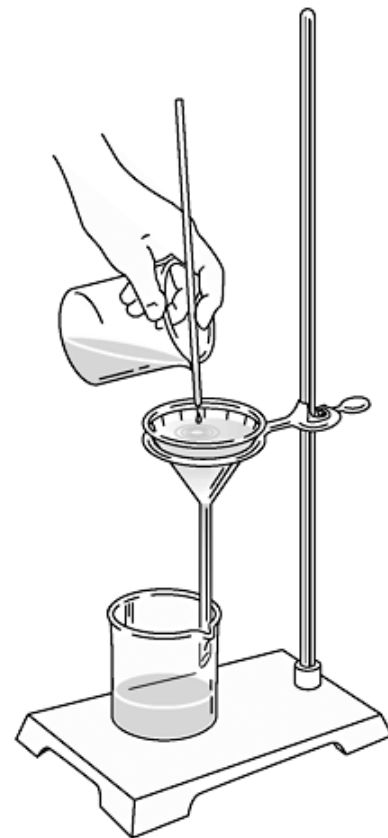
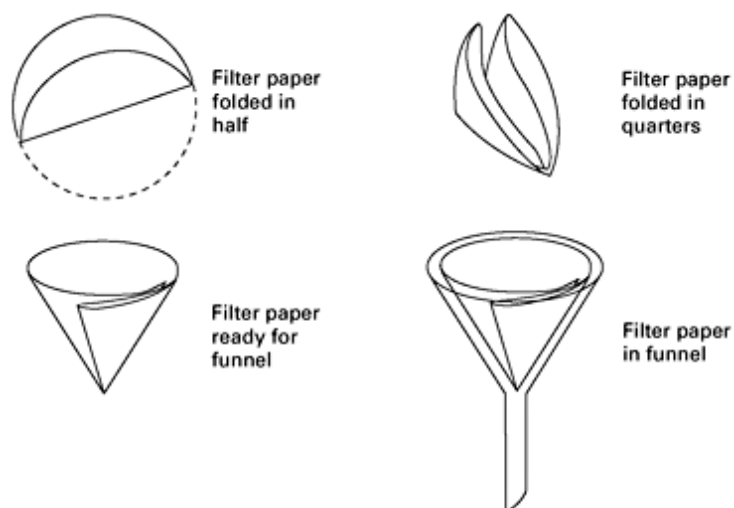


Figure 11



5. The funnel should be wet before you insert the filter paper. Gently run the funnel under the faucet of your sink to wet the funnel and the filter paper. Press the edges of the filter paper firmly against the sides of the funnel with the edges of a pencil or pen so no air can get between the funnel and the filter paper while the liquid is being filtered. *EXCEPTION: A filter should not be wetted with water when the liquid to be filtered does not mix with water. Why?*

6. Dissolve your salt/sand mixture from Part 4 in a beaker containing about 30 mL of water. Let the sand settle to the bottom of the beaker and gently pour the salt water into the filter observing the following suggestions:
- a. The filter paper should not extend above the edge of the funnel. Use filter paper that leaves about 1 cm of the funnel exposed.
 - b. Keep as much sand out of the filter paper as possible. Sand will clog your filter paper.
 - c. Do not completely fill the funnel. The liquid must NEVER go above the top of the filter paper.
 - d. When a liquid is poured from a beaker, it may adhere to the glass and run down the outside wall. This may be avoided by holding a stirring rod against the lip of the beaker, as shown in **Figure 10** on the previous page. The liquid will run down the rod and drop off into the funnel without running down the outside of the beaker. The sand that was in the water is retained on the filter paper. What TWO properties of the sand enabled it to be separated from the water in this procedure?

What does the filtrate contain?

PART 7—EVAPORATION

MATERIALS

- Filtrate from Part 6
- Bunsen burner and related equipment
- evaporating dish
- watch glass
- wire gauze, ceramic-centered
- heat resistant mat
- cloth towel
- beaker tongs
- iron ring
- ring stand
- lab apron
- safety goggles
- sparker

Procedure

1. The salt can be recovered from the filtrate by pouring the filtrate into an evaporating dish and evaporating it over a low flame nearly to dryness. **Figure 12** shows a correct setup for evaporation.

CAUTION: When using a Bunsen burner, confine loose clothing and long hair. Wear your safety goggles, lab apron, and heat-resistant gloves.

2. Remove the flame as soon if the liquid begins to boil over. Continue heating until all the liquid has evaporated. Shut off the burner.
3. Use beaker tongs to remove the evaporating dish and place it on a heat resistant mat. Use the beaker tongs to gently remove the watch glass. Describe what you see in the evaporating dish.

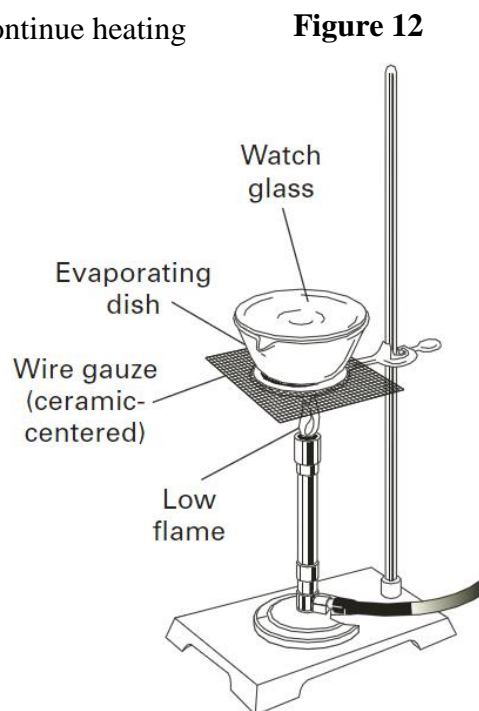
4. What property of salt prevents it from being separated from the water by filtration?

5. All equipment should be clean, dry, and put away in an orderly fashion for the next lab.

CAUTION: Be careful of the iron ring, it may still be hot! DO NOT put up any equipment that is still hot. It could start a fire in your lab station. Leave hot equipment out on the lab station. Be sure that the valve on the gas jet is completely shut off.

Make certain that the filter papers and sand are disposed of in the waste jars or containers and not down the sink.

Remember to wash your hands thoroughly with soap at the end of each lab.



Analysis Questions

Answer the following questions in complete sentences.

1. As soon as you enter the lab, what safety equipment should you put on immediately?
2. Before you light a burner, what safety precautions should always be followed?
3. What immediate action should you take when the flame of your burner is burning inside the base of the barrel?
4. What type of flame is preferred for laboratory work, and why?
5. When inserting glass tubing, why is it important that you wear safety goggles and gloves and that you cover the tubing and stopper with protective pads of cloth?
6. What do you think might be a common cause of fires in lab drawers or lockers?
7. Why are broken glassware, chemicals, matches, and other laboratory debris never discarded in a wastepaper basket?

Name _____ Class _____ Date _____

8. List three safety precautions that should be observed when inserting or removing glass tubing from a rubber stopper or rubber hose.

9. Why should you never touch chemicals with your hands?

10. What precaution can help prevent chemical contamination in reagent bottles?

11. Why are chemicals and hot objects never placed directly on the balance pan?

12. List three pieces of equipment used in the laboratory for measuring small quantities of liquids. Label them as “accurate” and “not accurate.”

13. What important guideline should you follow when folding filter paper?

Name _____ Class _____ Date _____

14. Describe the condition of all lab equipment at the end of an experiment. What should be checked before you leave the lab?

15. What is the correct procedure for removing a solid reagent from its container in preparation for its use in an experiment?










16. What is the correct procedure for removing a liquid reagent from its container in preparation for its use in an experiment?

17. Why is it important to use low flame when evaporating water from a recovered filtrate?

General Conclusion Questions

SAFETY CHECK

Identify the following safety symbols:

- a.  _____
- b.  _____
- c.  _____
- d.  _____
- e.  _____
- f.  _____
- g.  _____
- h.  _____
- i.  _____
- j.  _____

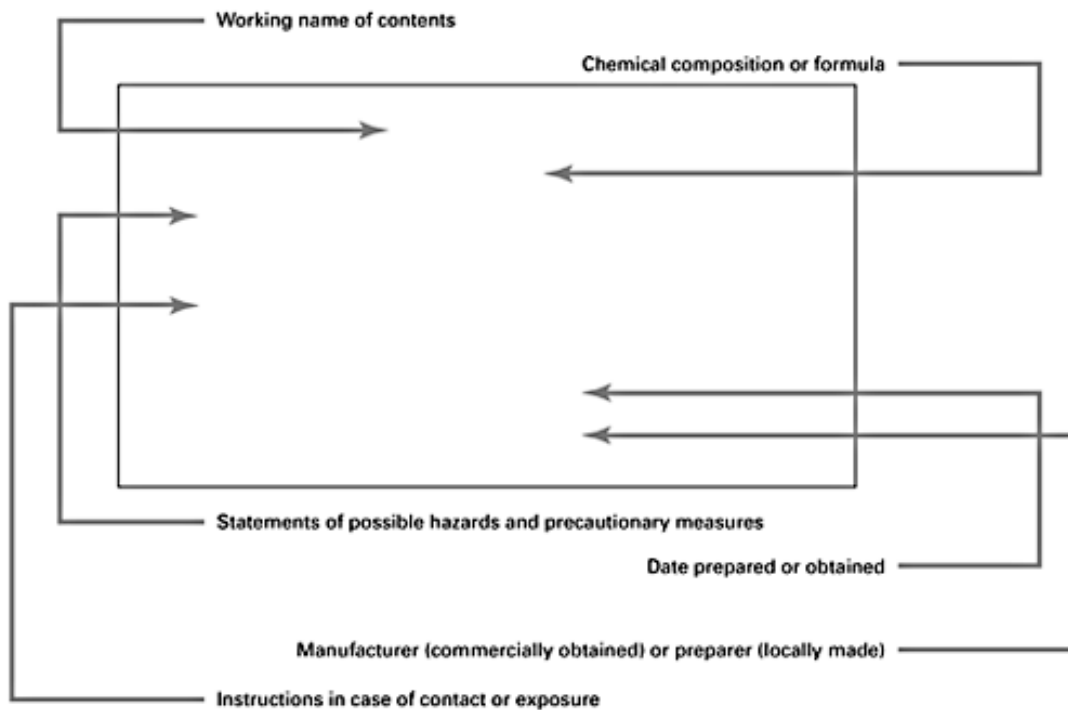
LABELING

Practice labeling a chemical container or bottle by filling in the appropriate information that is missing from the label pictured on the following page.

Use 6 M sodium hydroxide (NaOH) as the solution to be labeled. (Hint: 6 M sodium hydroxide is a caustic and corrosive solution, and it can be considered potentially as hazardous as 6 M HCl.)

Go back and check out the label information in the Safety Packet if you have a question.

SAMPLE LABEL



TRUE OR FALSE

Read the following statements and indicate T or F in the blank if they are true or false.

- _____ 1. Never work alone in the laboratory.
- _____ 2. Never lay the stopper of a reagent bottle on the lab table.
- _____ 3. At the end of an experiment, in order to save the school's money, save all excess chemicals and pour them back into their stock bottles.
- _____ 4. The quickest and safest way to heat a material in a test tube is by concentrating the flame on the bottom of the test tube.
- _____ 5. Use care in selecting glassware for high-temperature heating. Glassware should be Pyrex or a similar heat-treated type.
- _____ 6. A mortar and pestle should be used for grinding only one substance at a time.
- _____ 7. Safety goggles protect your eyes from particles and chemical injuries. It is completely safe to wear contact lenses under them while performing experiments.
- _____ 8. Never use the wastepaper basket for disposal of chemicals.
- _____ 9. First aid kits may be used by anyone to give emergency treatment after an accident.
- _____ 10. Eyewash fountains and safety showers should be checked daily for proper operation.

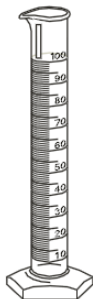
CHEMICAL APPARATUS

Identify each piece of apparatus. Place your answers in the spaces provided.

a.



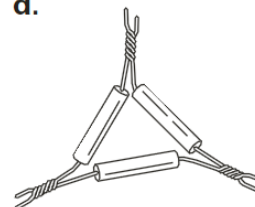
b.



c.



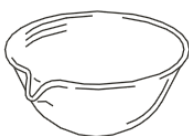
d.



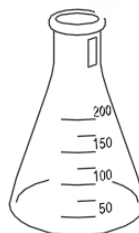
e.



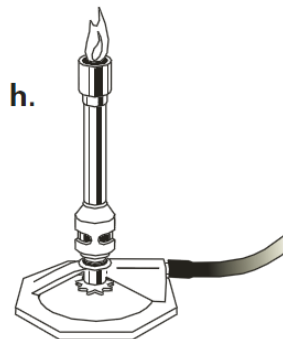
f.



g.



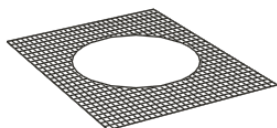
h.



i.



j.



k.



l.



a. _____

g. _____

b. _____

h. _____

c. _____

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k. _____

f. _____

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