Skills Practice

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, gloves, 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. If there are no precautions stated on the label, ask your teacher what precautions to follow. 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.

When you insert glass tubing into stoppers, lubricate the glass with water or glycerin and protect your hands and fingers. Wear leather gloves or place folded cloth pads between both your hands and the glass tubing. Then gently push the tubing into the stopper hole. In the same way, protect your hands and fingers when removing glass tubing from stoppers and from rubber or plastic tubing.

PART 1—THE BURNER

MATERIALS

- Bunsen burner and related equipment
- copper wire, 18 gauge
- evaporating dish
- forceps
- heat-resistant gloves
- heat-resistant mat
- 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.
3. When lighting the burner, partially close the ports at the base of the barrel, turn the gas full on, hold the sparker 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 forceps, hold an evaporating dish in the tip of the flame for about 3 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

MATERIALS
• cloth pads or leather gloves
• glass funnel
• lab apron
• rubber hose
• rubber stopper, 1-hole
• safety goggles
• 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.

   ![Figure 2](image)

2. 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**

**MATERIALS**
- glazed paper
- gloves
- lab apron
- safety goggles
- sodium chloride
- spatula
- test tube

**Procedure**

1. Solids are usually kept in wide-mouthed bottles. A spatula should be used to dip out the solid as shown in 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.

   Using a spatula, remove a quantity of sodium chloride from its reagent bottle. In order to transfer the sodium chloride to a test tube, first place it on a piece of glazed paper about 10 cm square. Roll the paper into a cylinder and slide it into a test tube that is lying flat on the table. When you lift the tube to a vertical position and tap the paper gently, the solid will slide down into the test tube, as shown in Figure 4.

   **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.

2. Dispose of the solid sodium chloride and the glazed paper in the waste jars or containers provided by your teacher.

   **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.
3. Remember to clean up the lab station and to wash your hands at the end of this part of the experiment.

**Figure 3**

**Figure 4**

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**PART 4—THE BALANCE**

**MATERIALS**

- balance, centigram
- glazed paper
- gloves
- lab apron
- safety goggles
- sodium chloride
- spatula
- weighing paper

**Procedure**

1. When a balance is required for determining mass, you will use a centigram balance like the one shown in **Figure 5**. The centigram balance has a readability of 0.01 g. This means that your mass readings should all be recorded to the nearest 0.01 g.

2. 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.
3. 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 sodium chloride.

a. Make sure that the pointer on the balance is set at zero. Obtain a piece of weighing paper and place it on the balance pan. Determine the mass of the paper by adjusting the riders on the various scales. Record the mass of the weighing paper to the nearest 0.01 g.

**Mass of paper:**

b. Add 13 grams to the balance by sliding the rider on the 100 g scale to 10 and the rider on the 10 g scale to 3.

c. Using a spatula, obtain a quantity of sodium chloride from the reagent bottle and place it on a separate piece of glazed paper.

d. Now slowly pour the sodium chloride from the glazed paper onto the weighing paper on the balance pan until the pointer once again comes to zero. In most cases, you will only have to be close to the specified mass. Do not waste time trying to obtain exactly 13.00 g. Instead, read the exact mass when the pointer rests close to zero and you have around 13 g of sodium chloride in the balance pan. The mass might be 13.18 g. Record your exact mass of sodium chloride and the weighing paper to the nearest 0.01 g. (Hint: Remember to subtract the mass of the weighing paper to find the mass of sodium chloride.)

**Mass of NaCl and paper:**

4. Wash your hands thoroughly with soap and water at the end of each lab period.
PART 5—MEASURING LIQUIDS

MATERIALS

- beaker, 50 mL
- beaker, 250 mL
- buret
- buret clamp
- gloves
- graduated cylinder, 100 mL
- lab apron
- pipet
- ring stand
- 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 and describe the scale of your cylinder in the space below.

   Observations: ________________

2. A pipet or a buret is used for more accurate volume measurements. Pipets, which are made in many sizes, are used to deliver measured volumes of liquids. A pipet is fitted with a suction bulb, as shown in Figure 7. 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.
3. 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:

a. Clamp the buret in position on a ring stand, as shown in Figure 8.

b. Place a 250-mL beaker under the tip of the buret. The beaker serves to catch any liquid that is released.

c. 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.

d. 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.

4. 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. Such a curved surface is called a meniscus. If a liquid wets the glass, read the bottom of the meniscus, 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 buret.

**Buret reading:**
5. 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.

6. 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.

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.

7. Remember to wash your hands thoroughly with soap at the end of this part of the experiment.

PART 6—FILTRATION

MATERIALS
- beaker, 250 mL (2)
- Bunsen burner and related equipment
- evaporating dish
- filter paper
- fine sand
- funnel
- glass stirring rod
- gloves
- iron ring
- lab apron
- ring stand
- safety goggles
- sodium chloride
- sparker
- wash bottle
- water
- wire gauze, ceramic-centered
Procedure

1. Sometimes liquids contain particles of insoluble solids that are present either as impurities or as precipitates formed by the interaction of the chemicals used in the experiment. If the particles are denser than water, they soon sink to the bottom. Most of the clear, supernatant liquid above the solid may be poured off without disturbing the precipitate. This method of separation is known as decantation.

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. Use a beaker to collect the filtrate. Adjust the funnel so that the stem of the funnel just touches the inside wall of the beaker.

3. Fold a circular piece of filter paper along its diameter, then fold it again to form a quadrant, as shown in Figure 11. Separate the folds of the filter paper, with three thickness on one side and one on the other; then place the resulting filter paper cone in the funnel.
The funnel should be wet before you insert the filter paper. Use your plastic wash bottle to wet the funnel and the filter paper. Press the edges of the filter paper firmly against the sides of the funnel 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?*

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4. Dissolve 2 or 3 g of sodium chloride in a beaker containing about 50 mL of water, and then stir into the solution an equal volume of fine sand. Filter out the sand by pouring the mixture 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. Do not completely fill the funnel. It must never overflow.

   c. Try to establish a water column in the stem of the funnel to eliminate air bubbles, and then add the liquid quickly enough to keep the mixture level about 1 cm from 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 is retained on the filter paper. What property of the sand enables it to be separated from the water by filtration?

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What does the filtrate contain?

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5. 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.*
6. Remove the flame as soon as the liquid begins to spatter. Shut off the burner. What property of salt prevents it from being separated from the water by filtration?

7. All equipment should be clean, dry, and put away in an orderly fashion for the next lab experiment. 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 laboratory period.

**Analysis**

Answer the following questions in complete sentences.

1. **Organizing Ideas** As soon as you enter the lab, what safety equipment should you put on immediately?

2. **Organizing Ideas** Before doing an experiment, what should you read and discuss with your teacher?

3. **Organizing Ideas** Before you light a burner, what safety precautions should always be followed?

4. **Organizing Ideas** What immediate action should you take when the flame of your burner is burning inside the base of the barrel?
5. Organizing Ideas  What type of flame is preferred for laboratory work, and why?

6. Analyzing Ideas  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?

7. Analyzing Ideas  What do you think might be a common cause of fires in lab drawers or lockers?

8. Analyzing Ideas  Why are broken glassware, chemicals, matches, and other laboratory debris never discarded in a wastepaper basket?

9. Organizing Ideas  List the safety precautions that should be observed when inserting or removing glass tubing from a rubber stopper or rubber hose.
10. Analyzing Ideas  Why should you never touch chemicals with your hands?

11. Organizing Ideas  What precaution can help prevent chemical contamination in reagent bottles?

12. Analyzing Ideas  Why are chemicals and hot objects never placed directly on the balance pan?

13. Organizing Ideas  List three pieces of equipment used in the laboratory for measuring small quantities of liquids. What is the correct procedure for filling a buret with liquid?

14. Organizing Ideas  What is the rule about the size of filter paper to be used with a funnel?

15. Organizing Ideas  How can a liquid be transferred from a beaker to a funnel without spattering and without running down the outside wall of the beaker?
16. Organizing Ideas  Describe the condition of all lab equipment at the end of an experiment. What should be checked before you leave the lab?

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

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

19. Analyzing Ideas  Why is it important to use low flame when evaporating water from a recovered filtrate?
General Conclusions

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.)
TRUE OR FALSE

Read the following statements and indicate whether they are true or false. Place your answer in the space next to the statement.

_____ 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 and facewash 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.