Laboratory Experiments in General Chemistry

Chandler · Barnes

LABORATORY EXPERIMENTS IN GENERAL CHEMISTRY

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GLENCOE PUBLISHING CO., INC.
Encino, California
Collier Macmillan Publishers
London

Printed in the United States of America

Glencoe Publishing Co., Inc.
17337 Ventura Boulevard
Encino, California 91316
Collier Macmillan Canada, Ltd.

1 2 3 4 5 6 7 8 9 10 84 83 82 81 80 81

ISBN 0-02-471170-5

PREFACE

Chemistry is a laboratory science, and its study is meaningless without a laboratory experience. This manual provides the basis for the broad but comprehensive laboratory experience the general chemistry student needs. It acquaints students with a variety of chemical reaction types and a large number of laboratory techniques and includes many questions probing students' understanding of the experiments they have done. It offers a full spectrum of experiments and, for many concepts, experiments of different levels of difficulty or complexity and expense of equipment. Brief introductions are given for experiments whose principles are covered in any introductory text, and more extensive introductions are given for more complex experiments. Although each experiment is independent of any other, new concepts are frequently related to principles and facts learned in previous experiments, thereby reinforcing them.

To insure that students understand laboratory procedures thoroughly, care has been taken to discuss when and why, as well as how, procedures are performed. In particular, this manual contains an extensive presentation on error that emphasizes the student understanding of the significance of measurements performed and results obtained, rather than focusing on the treatment of error for its own sake.

We have provided an opportunity for students to gain a knowledge of the chemistry of some typical metal ions and to learn some modern separation techniques so they can design their own separation schemes. Traditional qualitative analysis, with its emphasis on repetitive precipitation is not stressed.

Laboratory Experiments in General Chemistry was prepared as a companion to *Chemistry* by Henry I. Abrash and Kenneth I. Hardcastle (Glencoe, 1981), but it is compatible with most other general chemistry texts as well. The concepts and techniques presented in this manual will provide a student with the background necessary for entry into any field of scientific investigation.

We wish to thank many colleagues for their help and suggestions, with special thanks to Everett Reed, John George, and Everett Turner. The encouragement, constant help, and patience of Ramon Barnes is remembered with gratitude.

University of Massachusetts, Amherst
1981

John Chandler
Dorothy Barnes

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INTRODUCTION

Chemistry is a laboratory science, and its study is meaningless without a laboratory experience. In order to make this experience a positive and profitable one, it is useful to establish a few common-sense rules for laboratory safety, general laboratory operations, and proper use of equipment.

I. SAFETY RULES

1. *PREVENT ACCIDENTS* by being sensible and well informed. Read each experiment thoroughly before coming to the laboratory, do only authorized experiments, and follow directions carefully.

2. *KNOW THE LOCATION OF SAFETY AND EMERGENCY EQUIPMENT, AND LEARN HOW TO USE THE EQUIPMENT PROPERLY.* Be familiar with procedures to be followed in the specific emergencies described below. If an emergency occurs, remain calm, think a moment to plan the best action, then act with common sense.

3. *KNOW WHERE TO GET HELP FAST*, generally from an instructor. Report all accidents and unusual occurrences to your instructor immediately.

4. *WEAR EYE PROTECTION AT ALL TIMES.* Use other protective equipment or fume hoods as directed.

5. *DRESS APPROPRIATELY.* Bare feet are not allowed in a chemistry laboratory. Laboratory aprons and old clothing are well advised.

6. *TASTE OR SMELL CHEMICALS ONLY WHEN DIRECTED TO DO SO.* If requested to smell an odor, use the technique illustrated in Figure I-1.

NEVER put your face directly over the container. Instead, fan a little of the vapor toward you by sweeping your hand over the top of the container.

Figure I-1. Noting chemical odors

7. *IMMEDIATELY CLEAN UP ANY CHEMICALS THAT YOU SPILL.* If necessary, obtain advice on the cleanup procedure from your instructor.

SPECIFIC LABORATORY EMERGENCIES

1. *CHEMICAL SPILLS* anywhere on the body: Flush the area immediately with large volumes of water from the nearest faucet. Remove contaminated clothing if necessary. Use no medication on the injury, but bandage and obtain medical attention if necessary.

a. *EYES:* Hold the eyes open while flushing for at least 5 minutes. Obtain medical help immediately.

b. *CONCENTRATED ACIDS OR BASES* spilled over a large body area. Use a safety shower, then obtain medical help.

2. *FIRE: Extinguish all burners,* remove flammable solvents from the immediate vicinity, and have all people not involved in fighting the fire leave the laboratory by the nearest exit. *Do not* attempt to fight a major fire. Pull the building alarm, shut the windows and fire doors, and leave by the nearest exit.

a. *CLOTHING ON FIRE:* Prevent the victim from running. Have the victim lie down, and smother the flames with available clothing, a fire blanket, or by rolling the victim around on the ground. Towels, books, or other items aflame can be put or pushed in the sink with tongs and doused with water.

b. *BURNS:* Apply no medication for major burns. Cover the area with a sterile dressing and obtain medical help immediately. For minor burns, immerse in ice water or in cold water and bandage when comfortable.

3. *CUTS AND PUNCTURES:* Clean your hands and the victim's wound with soap and water, and remove any glass slivers from the wound. Apply a sterile dressing, and control any severe bleeding by applying pressure directly over the wound. Apply no medication. Always obtain medical attention for punctures or severe cuts because of the possibility of tetanus.

4. If the victim of an accident is sent to a medical facility, *BE SURE THE VICTIM IS ACCOMPANIED BY ANOTHER PERSON.*

II. LABORATORY OPERATIONS

1. *Before* coming to the laboratory, read the entire experiment you expect to perform, including the introduction as well as the procedure. Become familiar with the safety rules in Section I of the Introduction, and with the specific techniques described in Section III below.

2. When seeking reagents, know both the name and the quantity of material needed, and go to the supply area equipped with a container for transporting the material back to your work bench. As a courtesy to your fellow students, never remove a reagent container from the supply area. Remove the necessary reagent from containers in a way that avoids contaminating the stock supply. See Figures I-2 and I-3 for proper methods of removing reagents from containers.

3. Avoid taking excess reagent. If an excess is accidentally acquired and is still in the lid of the original container, it may be returned to the stock bottle. Excess reagent which has been touched with a spatula or transferred to another container

should not be returned to stock since this may contaminate the entire supply. Such excess may, however, be shared with another student.

4. Ask your instructor about the disposal of waste materials: some wastes may be harmful to the environment or the plumbing! Unless otherwise advised, solid wastes are discarded into waste crocks, and liquids are flushed into the sink with copious amounts of water.

5. Record all data in the Table of Data provided for each experiment as it is acquired, and show the method of calculation whenever data workup is required. Scraps of paper with data and calculations scribbled on them have no place in a science laboratory.

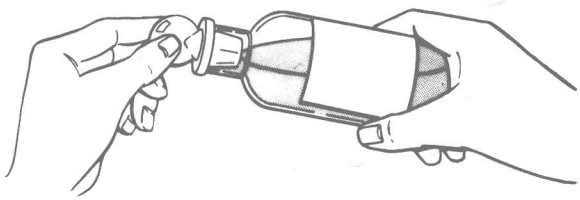
6. Practice good housekeeping at your lab bench, especially in cleaning it after you finish your experiment so the next student to work at the area will find it usable and free of hazard.

7. Before leaving the lab, check to see that the gas and water are turned off, and that all special equipment has been returned to your instructor.

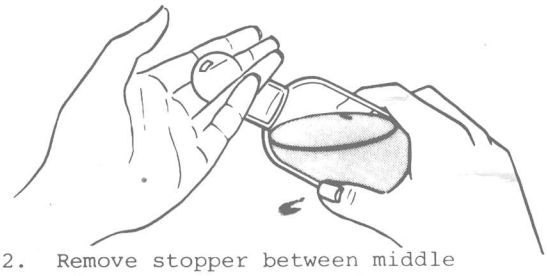
a. Hold reagent bottle with label up, tilt and roll bottle so that the desired amount of reagent falls into the container.

b. Hold reagent bottle as in (a) until some reagent is transferred to the reagent bottle cap. Tap the cap to transfer the desired amount of reagent to the container.

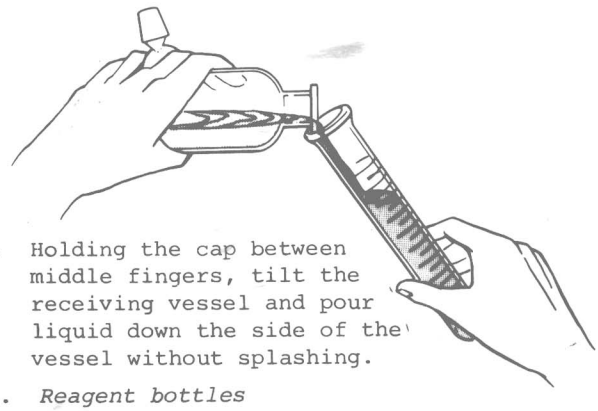
Figure I-2. Dispensing a solid from a reagent bottle



1. With label up, tilt bottle to moisten stopper.

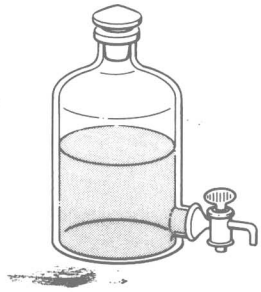


2. Remove stopper between middle fingers. DO NOT set the stopper on the bench top.



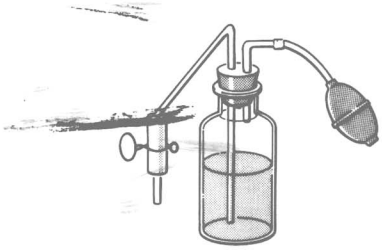
3. Holding the cap between middle fingers, tilt the receiving vessel and pour liquid down the side of the vessel without splashing.

a. Reagent bottles



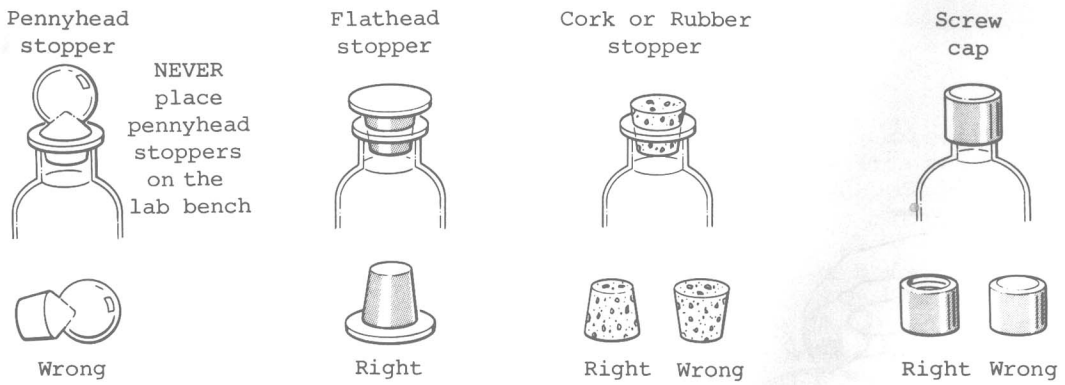
Liquid will flow by gravity feed, but air must be allowed to replace liquid removed.

b. Aspirator bottle



c. Siphon bottle

1. Squeeze bulb to pump air into the bottle.
2. Open clamp on delivery tubing to allow liquid flow.
3. Close clamp to stop liquid flow and check to be sure no dripping occurs.

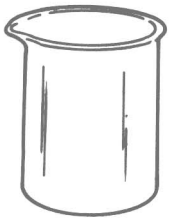


d. Placement of stoppers on the lab bench

Figure I-3. Dispensing liquids from reagent bottles

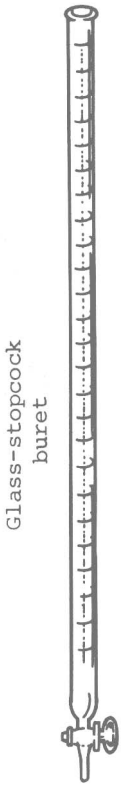
A. Common Equipment (Alphabetical Order)

Asbestos mat



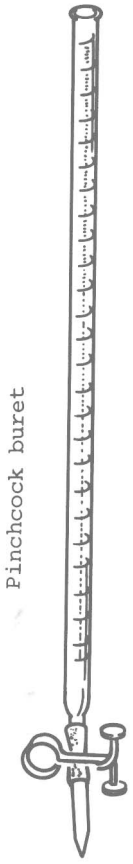
Beaker

Brush

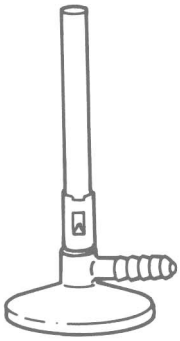


Glass-stopcock buret

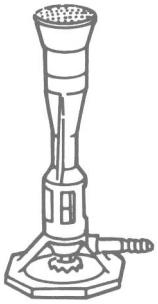
Burets



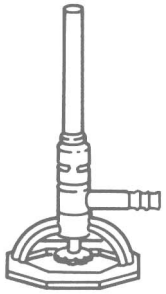
Pinchcock buret



Bunsen



Meker



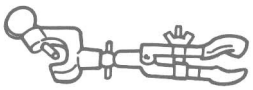
Tirrill

Burners

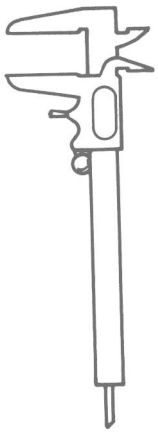
Burner tip

Flame spreader

Clamp holder



Clamp holder

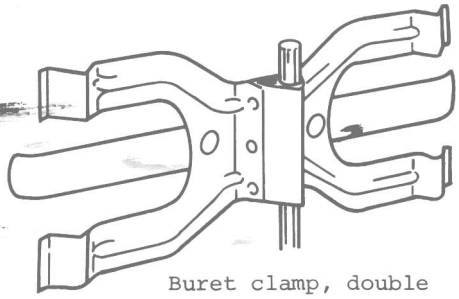


Caliper

Extension clamp

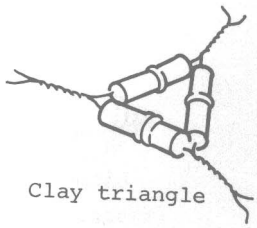
Pinchcock clamp

Screw clamp

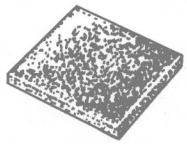


Buret clamp, double

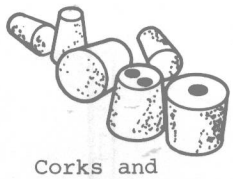
Clamps



Clay triangle

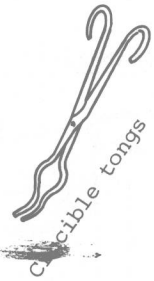


Cobalt glass



Corks and Rubber stoppers

Crucible with lid, porcelain



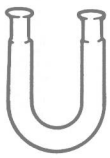
Crucible tongs

Cylinger, graduated

Deflagration spoon

Dessicator

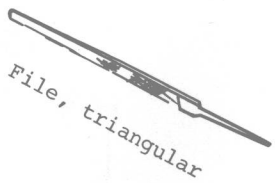
Straight drying tube



U-shaped drying tube

Drying Tubes

Evaporating dish



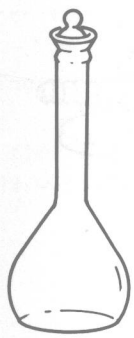
File, triangular

Filter paper

Erlenmeyer flask

Filter flask

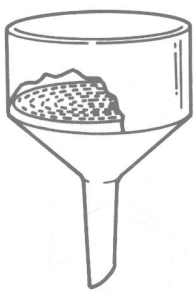
Florence flask



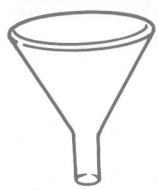
Volumetric flask

Flasks

Funnel



Büchner funnel



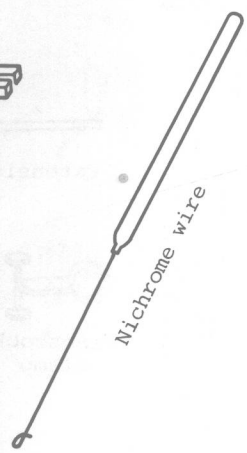
Powder funnel

Funnel support

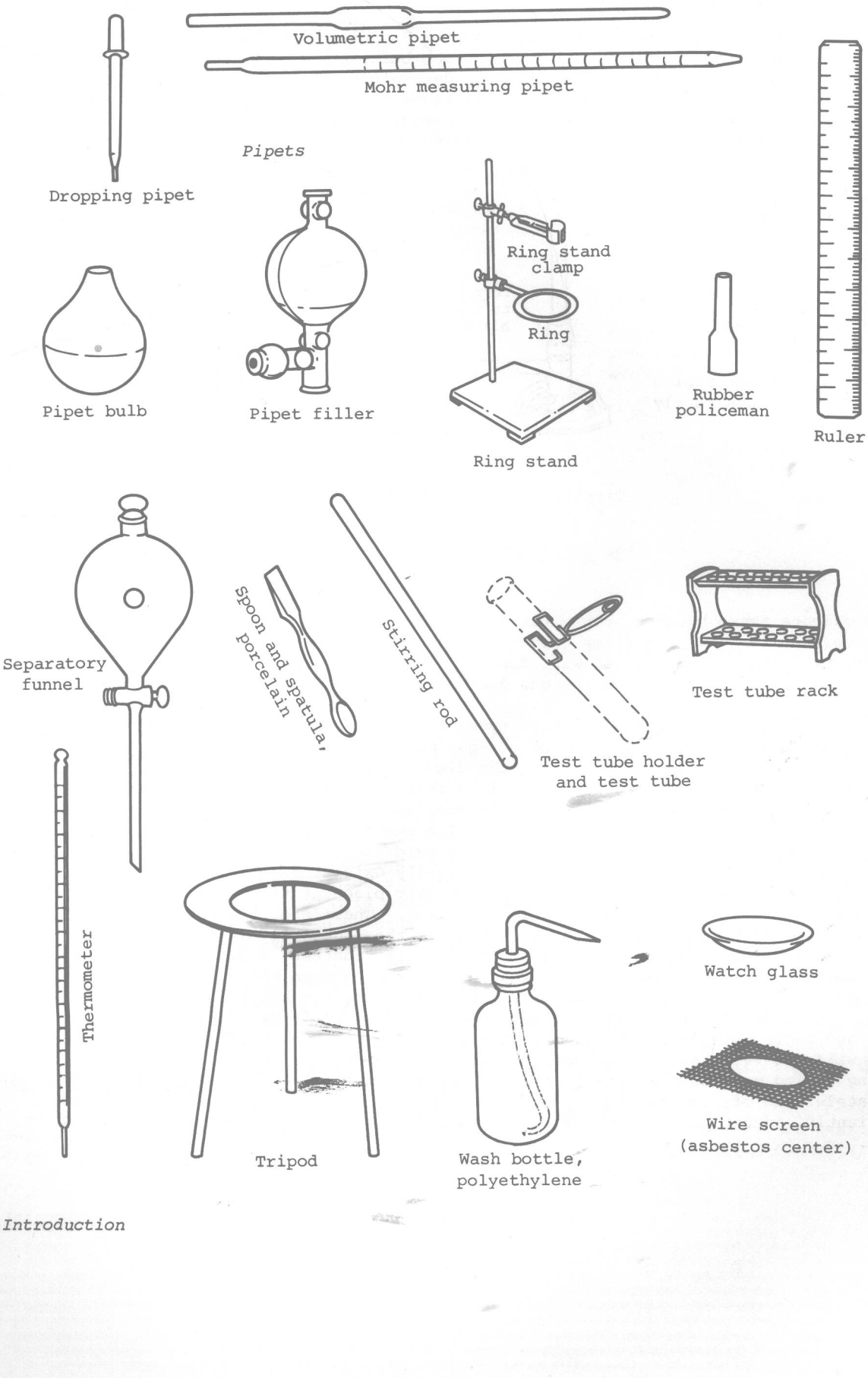
Funnels

Litmus paper

Mortar and pestle



Nichrome wire


Volumetric pipet
Mohr measuring pipet
Pipets
Dropping pipet
Pipet bulb
Pipet filler
Ring stand clamp
Ring
Ring stand
Rubber policeman
Ruler
Separatory funnel
Spoon and spatula, porcelain
Stirring rod
Test tube holder and test tube
Test tube rack
Thermometer
Tripod
Wash bottle, polyethylene
Watch glass
Wire screen (asbestos center)

B. *Use of the Laboratory Burner*

Laboratory heating is most typically done by Bunsen or Bunsen-type burners, some of which are illustrated in Figure I-4. The Bunsen burner has a gas inlet and a collar to regulate air flow, since the gas-air mixture determines the quality and intensity of the burner flame. Improved burners have modifications such as a fine-adjustment control for gas flow (Tirrill burner), or are designed to produce a hotter flame over a much larger area (Fisher and Meker burners).

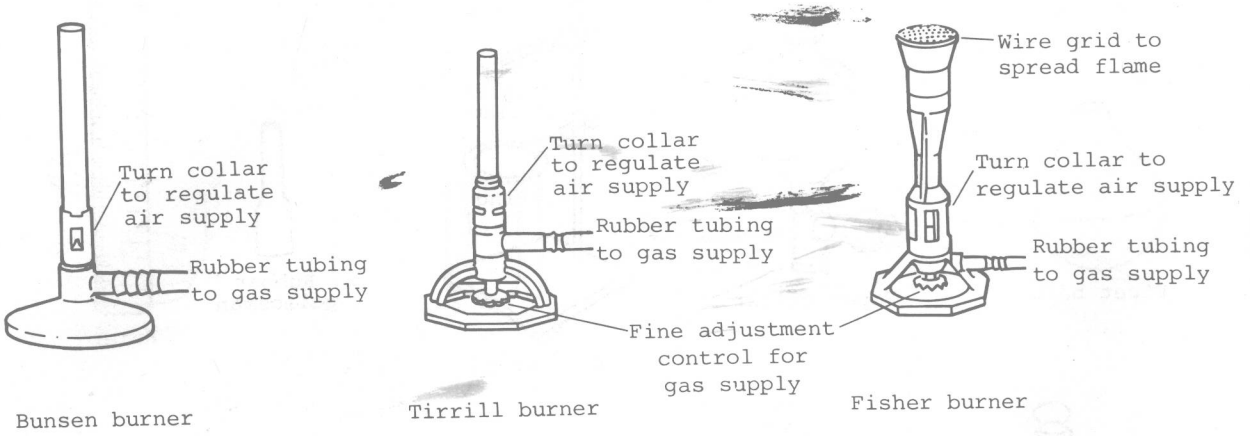
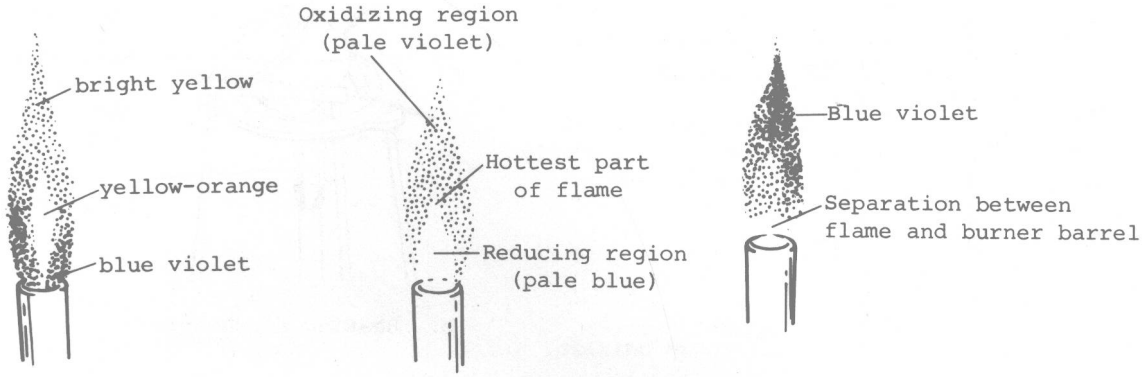


Figure 1-4. Laboratory burners

To operate these burners, close the air intake, turn on the gas, and hold a lighted match over the burner. The flame produced will be yellow, *luminous*, and probably smoky (see Figure I-5a). Such a flame will deposit soot on the object heated, and is a result of the incomplete combustion of the gas due to an insufficient supply of oxygen to the flame.

Slowly open the air intake so that oxygen is admitted to the gas stream. A blue-violet flame will be produced when the air-gas mixture is correct for complete combustion (see Figure I-5b). Characteristically, the flame will have an inner pale blue region containing mostly hot unburned fuel and air. This is a relatively cool region of the flame, and is called the *reducing region* because materials inserted here may be chemically reduced by the hot fuel. The outer violet region of the cone contains mainly hot combustion products and air, and is called the *oxidizing region* of the flame because the hot combustion products plus air can chemically oxidize materials inserted at this point. The most complete full combustion is taking place at the interface between the two cones; this is the hottest region of the flame. Therefore, the best place to hold objects to be heated is in this region just above the light blue inner cone.

Too large a supply of air may cause the flame to "chatter" audibly, to blow out, or to separate from the burner so that there is a gap between the burner and the flame (see Figure I-5c). In any of these cases, turn down the air supply and relight the burner. Occasionally, this type of flame may "flash back" or be sucked into the burner barrel and begin burning at the base of the barrel in the fuel intake area. The burner barrel will get very hot, a chattering sound will probably be audible, and the odor of charring tubing may occur as the fuel line starts to melt. *THIS IS A DANGEROUS SITUATION*. *Immediately* turn off the gas supply at the bench source. When the burner barrel cools sufficiently, adjust the air intake collar to reduce the air supply and relight the burner.



a. Luminous sooty flame (Increase air supply to remove yellow soot-depositing region, even if it appears only at the flame tip.)

b. Well-adjusted clean flame

c. Potentially dangerous flame (Decrease air supply to avoid "flash back.")

Figure 1-5. Flame characteristics

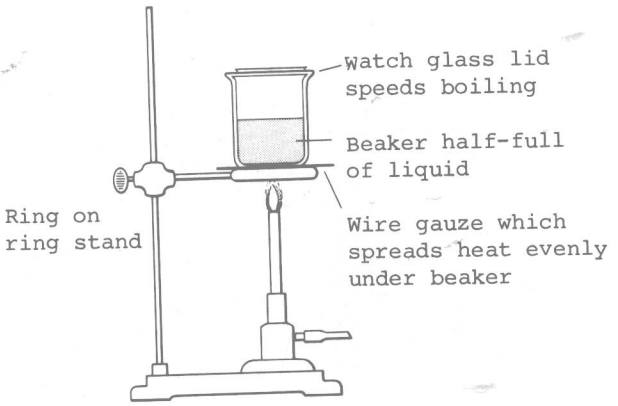
Figure I-6, on the following page, illustrates correct heating procedures for several laboratory operations. For an even boiling, it is useful to add a boiling chip (a piece of porous solid material) to the liquid. The air trapped in the pores of the boiling chip provides a gas phase into which the hot liquid may readily vaporize. Without such a gas-phase "steam valve," superheating and violently rapid vaporization may take place at the bottom of the container of liquid, causing "bumping" of the liquid.

C. Glassworking

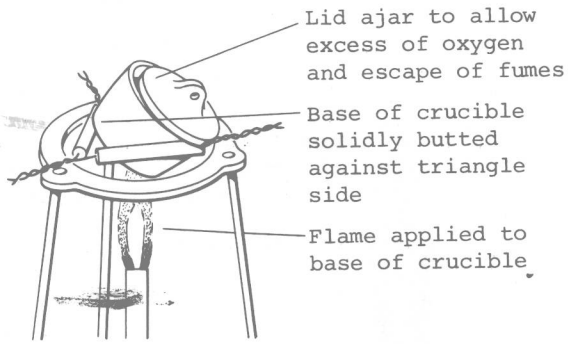
Most laboratory apparatus is made of hard, borosilicate glass such as Pyrex or Kimax. This glass has many properties which are desirable in a chemical laboratory, but its working temperature of 800-1100°C is beyond the temperatures of normal burner flames. Because soft soda lime glass can be bent and worked in the range from 400-800°C, it can be worked on the burners used in general chemistry and where exacting standards are not required.

To cut a piece of glass rod or tubing, first make a single scratch with a triangular file on the glass where you wish to cut it. Do not "saw" the glass; a single scratch will suffice (see Figure I-7). Using a towel to protect both hands, hold the glass with the scratch away from you and with your two thumbs behind and on opposite sides of the scratch. Pull toward you as if attempting to bend the glass, and it will snap at the scratch.

Fire polish the sharp edges of the glass by holding the end in the hottest part of the burner flame and rotating it to allow continuous even heating (see Figure I-8). The glass will melt and smooth out the sharp areas. Do not seal off the tube opening.

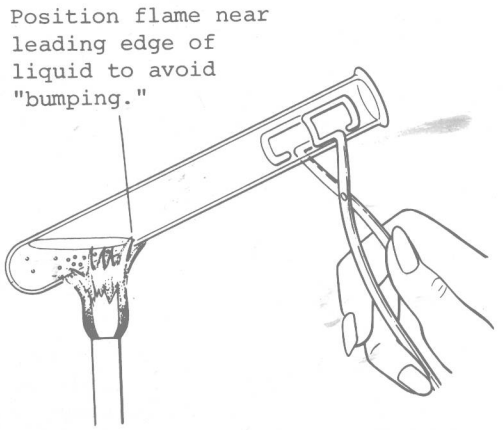


a. Heating a beaker of liquid



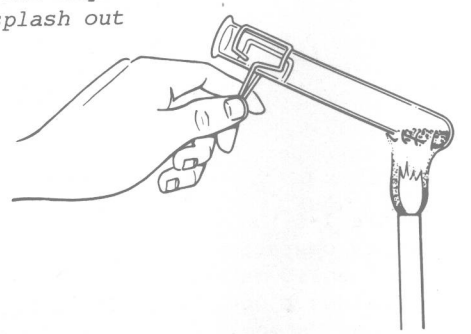
b. Heating a crucible

[NOTE: Tripods and ring stands are interchangeable good supports, but a ring stand adds flexibility in adjusting heights.]

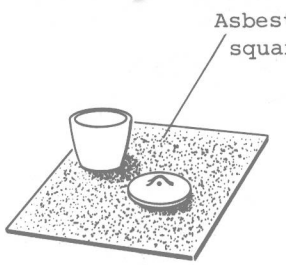


c. Heating a liquid in a test tube

NEVER point a test tube of boiling liquid at yourself or a neighbor. The liquid may "bump" and splash out of the tube.



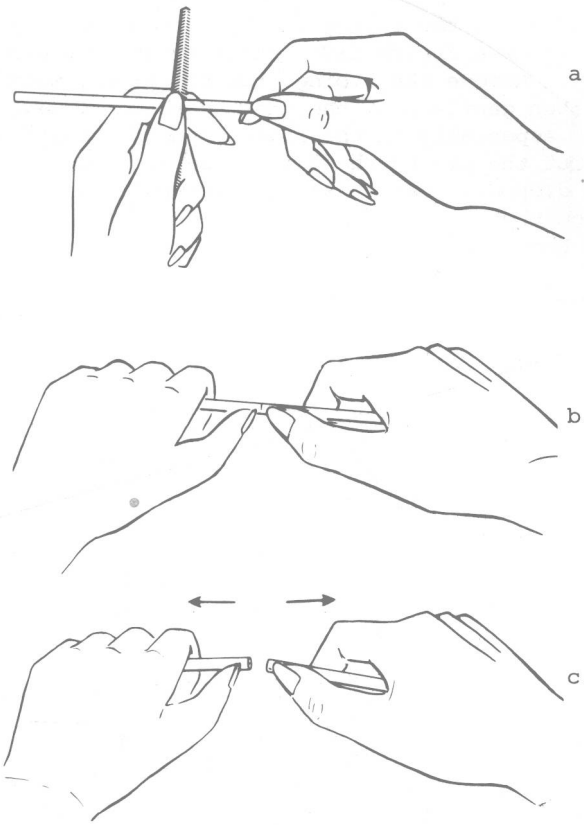
d. Heating a solid in a test tube



e. Cooling hot objects

CAUTION:
1. HOT GLASSWARE LOOKS LIKE COLD GLASSWARE. Always cool heated objects on an asbestos square to avoid cracks from the thermal shock of a cold benchtop, and to signal that the ojbect may be DANGEROUSLY HOT.
2. NEVER use a burner to directly heat volumetric glassware, bottles, or non-heat-resistant apparatus. Such apparatus will probably crack or shatter.
3. NEVER use an open flame near flammables or combustibles.
4. Use HOODS as directed to remove funes produced upon heating.

Figure I-6. Heating laboratory apparatus



a. Using a triangular file, make a single scratch on the glass.

b. Position hands for the breaking motion.

c. Pull the hands in opposite directions and bend slightly away from yourself.

Figure I-7. Cutting a piece of glass rod or tubing

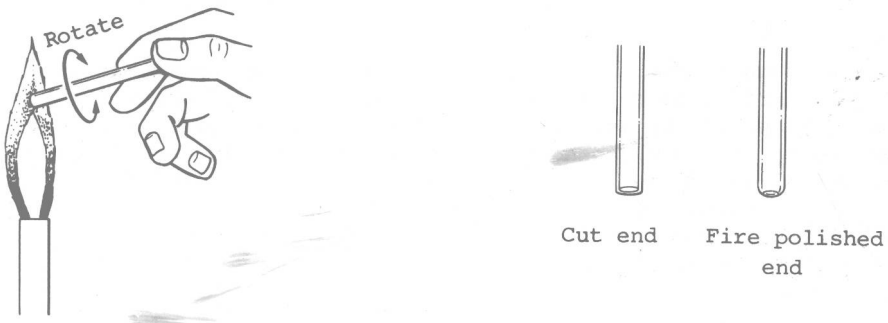


Figure I-8. Fire polishing

CAUTION: Hot glass looks like cold glass!

To make a dropper nozzle or constricted tip, heat the tubing in the hottest part of the flame and rotate it to allow uniform hearing (see Figure I-9). As the tubing begins to soften, allow the walls to thicken slightly. Remove the tubing from the flame, wait a few seconds while still rotating the tubing, then gently pull the ends apart to achieve the desired taper. Pulling the ends too soon, especially in the flame, causes a thinning of the tubing, producing a fragile dropper. Cut the tip to the desired length and fire polish the tip. To flare the bulb end of the dropper, heat it as for fire polishing, but allow it to get softer. Remove the tubing from the flame and use the warmed end of a triangular file to flare the end, as shown in Figure I-9d.

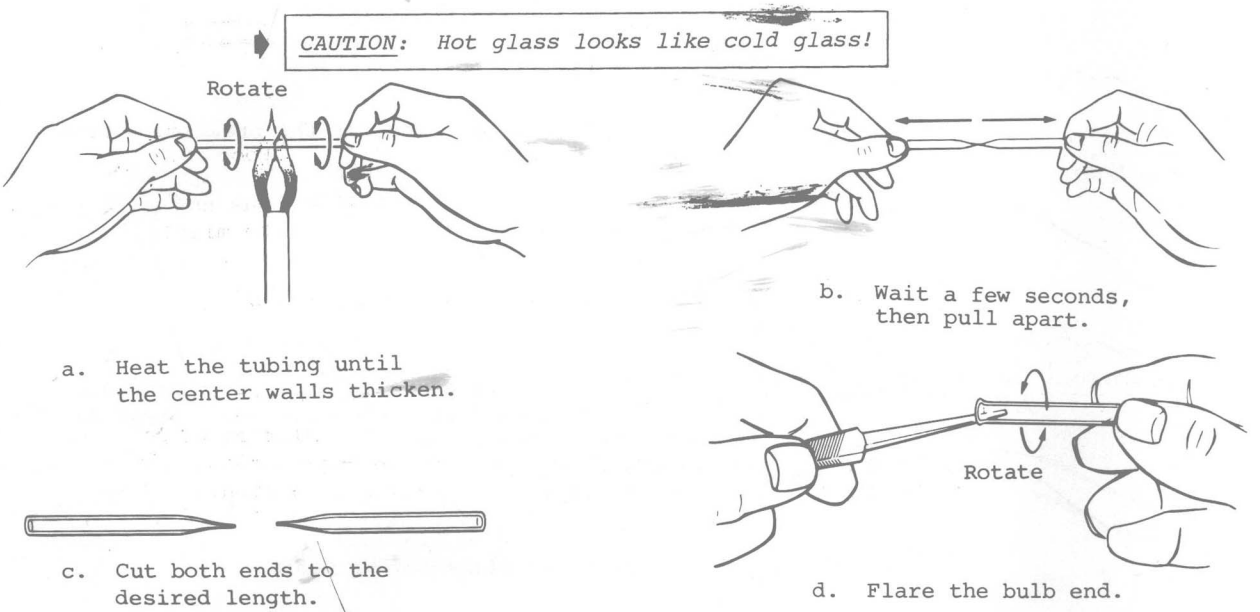


Figure I-9. Making a dropper or constricted tip

To make a glass bend, place a flame spreader on your unlit burner to allow a relatively long area of glass to be heated (see Figure I-10). Check the flame produced with the spreader; it should be uniform and flat-topped to provide even heating. Adjustments may be made by carefully widening the spreader with a thin coin: consult with your instructor before attempting any adjustment. Position the tubing so that the region to be bent is in the hottest portion of the flame. Rotate the tubing slowly until it softens (but does not sag), remove it from the flame, bend it to the angle desired, and hold the tubing until it hardens. A good bend has a uniform curve and is free of constrictions (see Figure I-11).

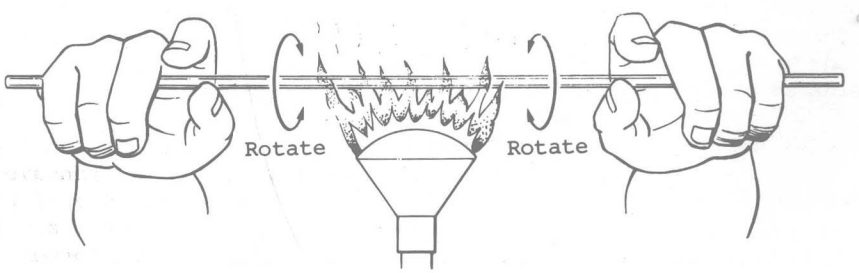


Figure I-10. Making a glass bend