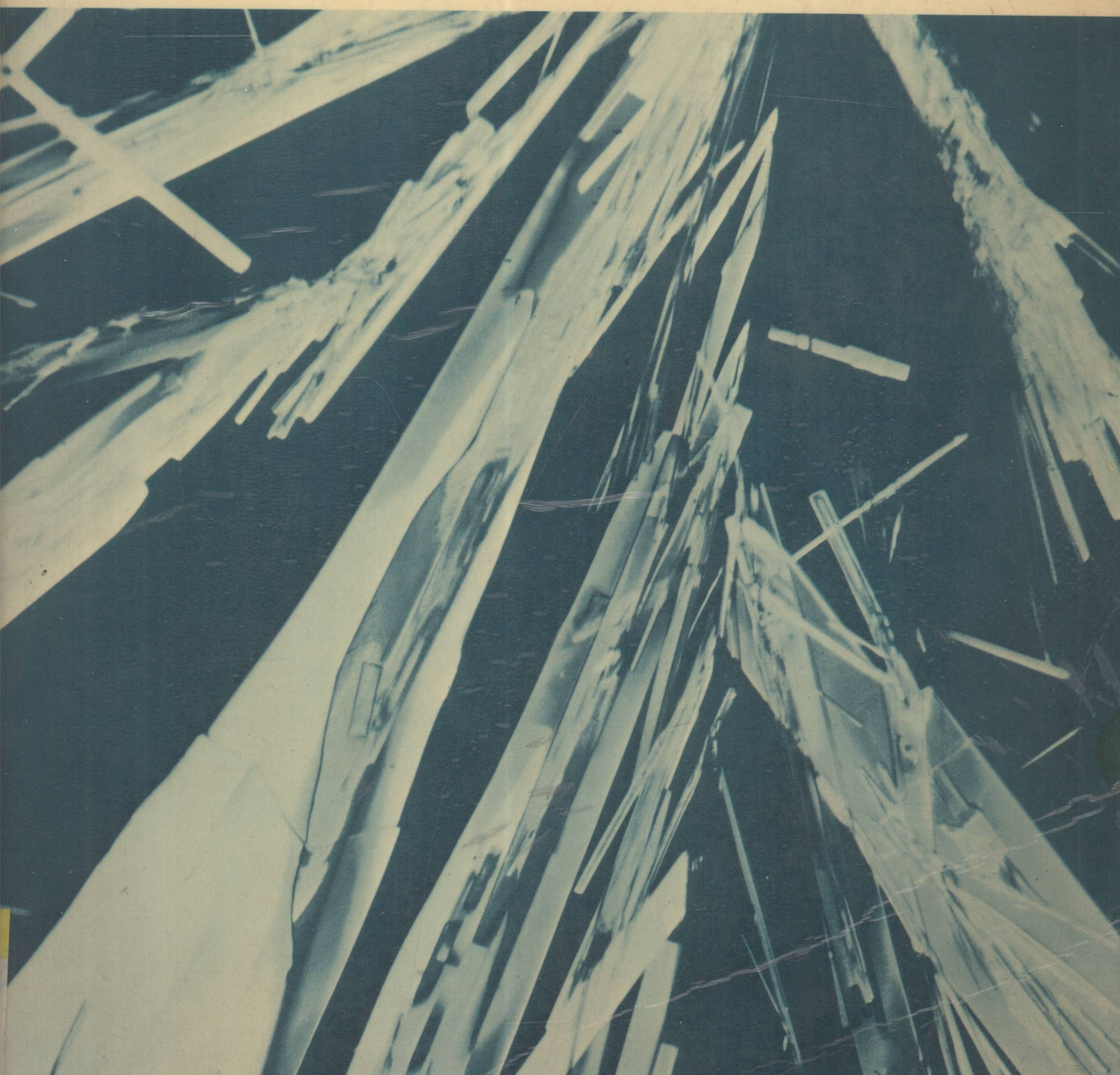
Laboratory Experiments in College Chemistry

THIRD EDITION

KING / CALDWELL / WILLIAMS



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Introduction

Although the experiments in this manual are arranged to accompany the authors' text, *College Chemistry, Sixth Edition,* the flexibility of the manual gives the instructor a choice in the order and number of experiments to be performed. The aim of this book is to present experiments to cover a full year's course in general chemistry, in which the student will spend from four to six hours a week for about thirty-three weeks in the laboratory. It is recognized that some students work faster or are more advanced than others; accordingly, more experiments are included than will be used by the average class. In courses with fewer student hours in the laboratory, some experiments may be deleted. Where facilities and equipment are limited, certain experiments or parts of experiments may be demonstrated by the instructor. Although most of the experiments may be completed in from two to three hours of laboratory time, a few may require from four to six hours.

The experiments have been selected to illustrate the principles of chemistry and to teach fundamental laboratory operations. Most of the experiments are quantitative in nature, but there are experiments which make use of semimicro qualitative methods. A number of *unknowns,* which the student must identify by measurement of certain physical properties or by chemical behavior, are included.

To expedite correcting and grading, separate report sheets are included for each experiment. If desired, these sheets may be detached and given to the instructor. The set of exercises which follows each experiment may be assigned either as laboratory work or as an extra assignment.

The authors are grateful to their colleagues at Washington State University and Oregon State University and to the graduate students of these institutions who have made helpful suggestions in the preparation and testing of the experiments.

LABORATORY RULES AND REQUIREMENTS

Apparatus. Keep your equipment clean and in order. Have your apparatus approved by the instructor in those experiments which call for generators or complicated set-ups. The laboratory assistant will initial your book in the proper space when your apparatus is assembled correctly.

Attendance. Students are expected to be at their laboratory desks at the beginning of the laboratory period, and to remain there until the end of the period. (Students who have finished the week's experiments may be excused by the instructor.)

Cleanliness. Keep your desk top clean and free from apparatus and materials not in use. At the end of the laboratory period, wash and dry the desk top thoroughly. If you have used a hood, be sure that you have left it clean and dry.

Independent work. Experiments are to be performed independently unless the instructor directs otherwise.

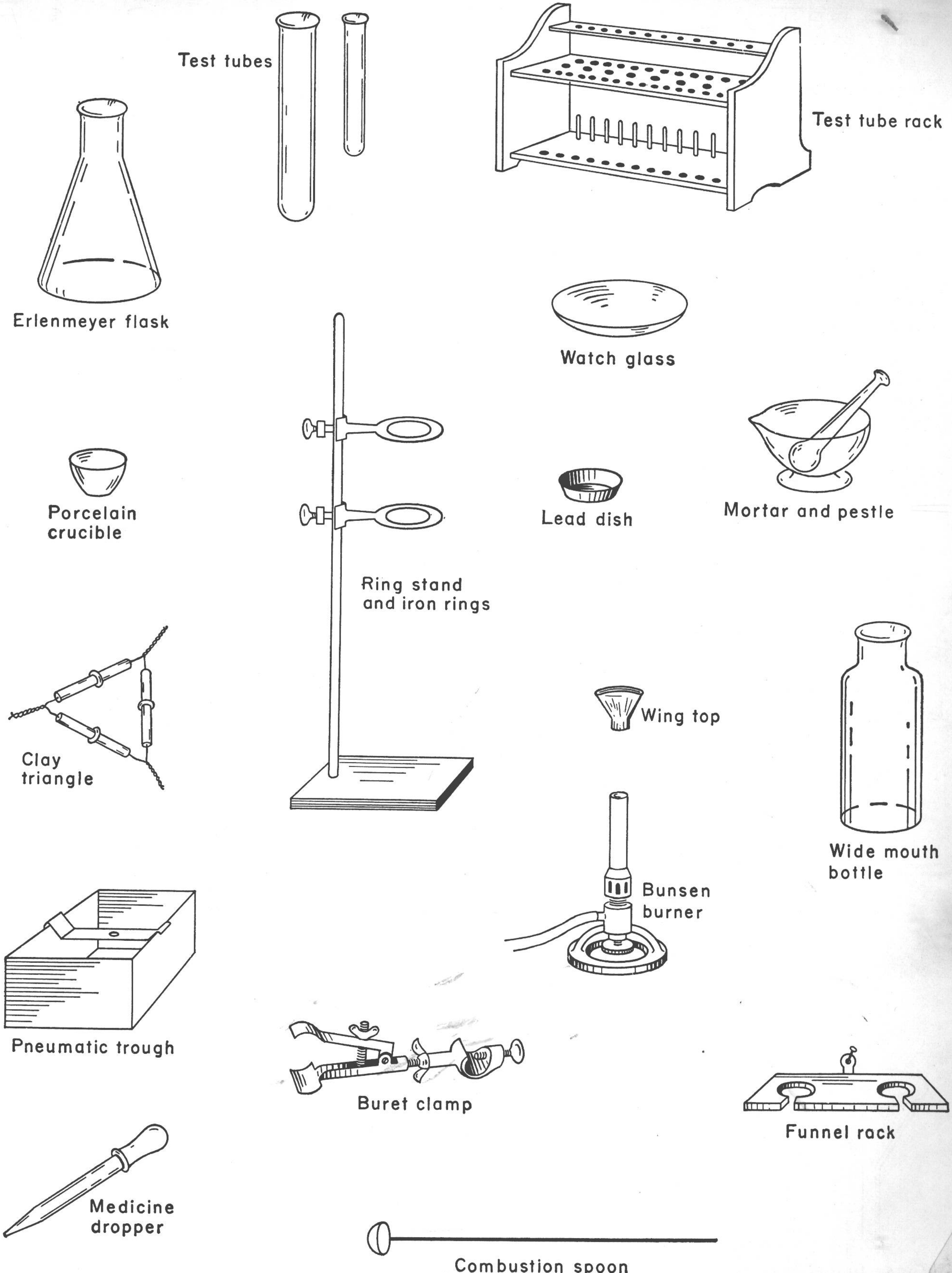
Injuries in the laboratory. If you spill acids or bases on your clothing wash it *immediately* with a *large* amount of water. Report to your instructor as soon as possible. Cuts and bruises should be reported to the instructor *immediately*.

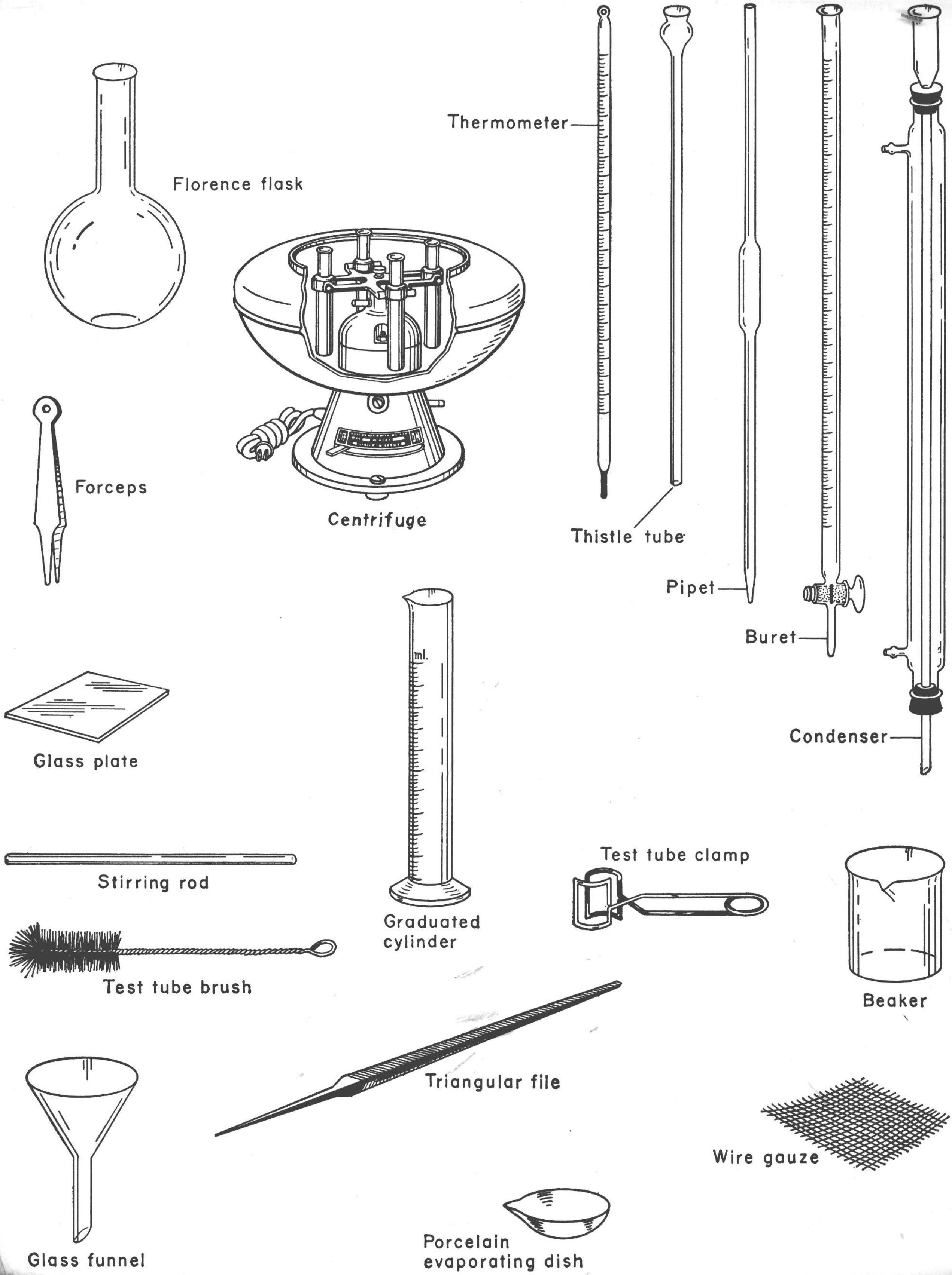
Reagents. Obtain reagents from the sideshelves. Take only what you need for the experiment. Avoid wasting chemicals. Remove solids from reagent bottles on sheets of paper, on watchglasses, or in evaporating dishes. Use test tubes for removing liquids. Do not try to pour solids into test tubes. Replace all reagent bottles in their proper places on the sideshelves. Do not carry reagent bottles to your desk.

Unauthorized experiments. Performing of unauthorized experiments is positively forbidden. If you should desire to perform an experiment which is not in this manual, consult the instructor for his advice and approval.

Waste materials. Place all unused solids, paper, match sticks, and so on in the waste jars at the end of the laboratory sink—*not* in the sink. Only liquids are to be poured in the sink.

Safety. Learn to be *safety minded* in the laboratory. Before doing any part of an experiment, ask yourself if there are any dangers involved. See your instructor if in doubt. Safety glasses are to be worn for any experiment where explosions or spattering of strong chemicals is a possibility. Never heat a liquid contained in a small test tube over an open flame. Place the test tube in a beaker of boiling water.


Test tubes
Test tube rack
Erlenmeyer flask
Watch glass
Porcelain crucible
Lead dish
Mortar and pestle
Ring stand and iron rings
Clay triangle
Wing top
Wide mouth bottle
Bunsen burner
Pneumatic trough
Buret clamp
Funnel rack
Medicine dropper
Combustion spoon


Thermometer
Florence flask
Centrifuge
Forceps
Thistle tube
Pipet
Buret
Condenser
Glass plate
Stirring rod
Graduated cylinder
Test tube clamp
Beaker
Test tube brush
Triangular file
Wire gauze
Glass funnel
Porcelain evaporating dish

Contents

EXPERIMENT 1

Apparatus and Manipulations

Purpose. To become familiar with the laboratory techniques of using a Bunsen burner, bending and working glass, and handling liquid and solid chemicals.

DISCUSSION

Familiarize yourself with the rules and requirements of the laboratory (page v). Proper laboratory manipulations and handling of apparatus will reduce breakage and the accompanying accidents and expense to you. Care and neatness in all laboratory work is essential; time will be saved and results will be more conclusive.

Reference. King, Caldwell, and Williams, *College Chemistry,* Chapters 1 and 2.

PROCEDURE

A. The Bunsen burner. Examine the Bunsen burner, particularly the adjustment of the gas and air supply. Note that there are holes at the base of the stack for introduction of air, and that the size of the air inlets is adjusted by screwing down the stack.

Attach the burner to the gas jet by a piece of rubber tubing. Close the air holes by screwing down the stack, turn on the gas, and light the burner by bringing the burning match to the opening of the burner from the side and below. (Do not light the burner by lowering the burning match from above the opening.) Next, turn the stack slowly to open the air holes, and note the changing appearance of the flame. When using the flame for heating purposes, always, unless specifically directed otherwise, use a noiseless, nearly nonluminous flame. Draw a diagram of the flame properly adjusted on top of Figure 1.

Determine the relative temperatures in different parts of the flame by holding a matchstick for a moment horizontally in the flame

(*a*) over the opening of the burner.
(*b*) about ½ inch higher.
(*c*) at the tip of the inner blue cone of the flame.
(*d*) about ½ inch higher.

Where is the hottest spot in the flame?

Hold a piece of white cardboard in the flame, allowing the edge of the card to rest on top of the burner until the cardboard is visibly charred. Remove the card, and from the effects noted, indicate the cool portion of the flame on the diagram you drew on Figure 1.

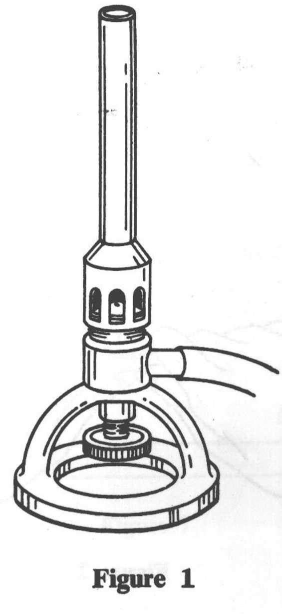


Figure 1

Insert one end of a short (6-inch) piece of glass tubing into the inner portion of the flame, and try to light the gas issuing from the other end.

With the air holes partly open, slowly turn down the gas supply with the thumb-screw at the base of the burner, until the flame "strikes back"—that is, travels from the top of the stack to the bottom. Note that the lower part of the burner gets hot. Continued burning results in the production of carbon monoxide, a poisonous gas. When the burner "strikes back," it should be turned off immediately.

B. Cutting glass rods and tubing. To cut glass tubing or a glass rod, make a slight transverse scratch on the glass with a triangular file. Hold the glass tube or rod with both hands (Figure 2), thumbs together and opposite the file scratch. Press forward with the thumbs to bend the tube away from you, and at the same time pull slightly with each hand.

Using the above technique, prepare one 6-inch and one 8-inch stirring rod. The ends of these must be "fire polished," as explained in the next section. ► **Note:** It is frequently desirable to hold the glass to be cut in a towel to protect the hands.

C. Fire polishing glass rods and glass tubing. Before glass rods or glass tubing may be used, the ends must be rounded by "fire polishing" to protect you against injuries from the sharp edges. (Sharp edges will also scratch beakers and laboratory glassware, and cut rubber stoppers if glass tubing is inserted.) Hold the end of the piece of glass to be polished in the hottest part of the flame, and slowly rotate it until the flame becomes bright yellow and the glass "flows," leaving a rounded edge to the rod or tube. Do not heat to the extent that a glass tube becomes constricted. Remove the tube from the flame and place it on the base of a ring stand so that the hot portion extends over the edge of the iron base, but does not come in contact with the laboratory desk top. When the glass is cool, fire polish the other end in the same manner.

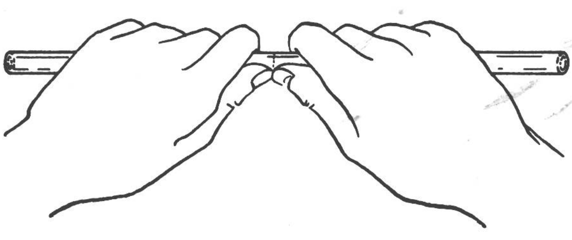


Figure 2

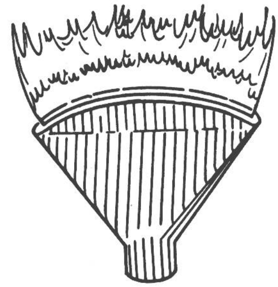


Figure 3

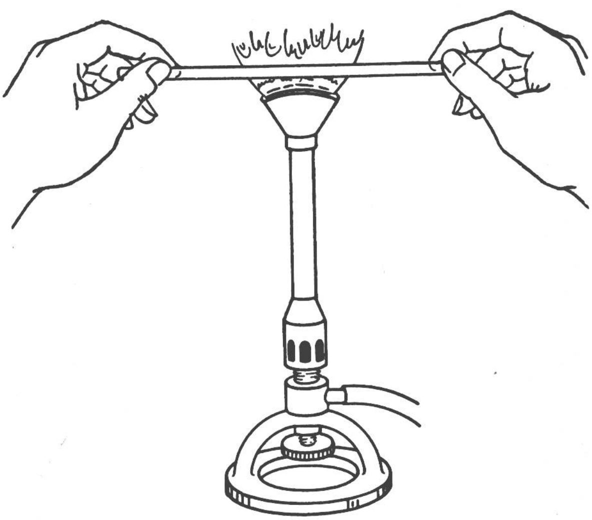


Figure 4

D. Bending glass tubing. Use the wing top on the burner for bending glass tubing (Figure 3).

The wing top should be adjusted to a wide even opening, and the flame should be as shown in Figure 3. Hold the tube lengthwise in the flame and rotate it slowly (Figure 4), moving it back and forth slowly so that the tube is uniformly softened.

Keep the tube in the flame until it begins to sag of its own weight. Then remove it from the flame and slowly bend it to the desired shape. The bend must be smooth and not too sharp, and the walls and bore must be uniform. Do not permit the heated tube to come in contact with the table top. Place it on the base of the ring stand or on a piece of wire gauze to cool. Practice several bends and be sure that you can make them like those in Figure 5—not like those in Figure 6.

Do not make a bend too close to the end of a piece of tubing; the tubing should be easily held. The excess may be removed after the tube is properly bent.

When you have demonstrated your ability to make satisfactory bends, prepare tubes like those shown in Figure 7. These bends must be carefully and accurately prepared; they will be used in later experiments.

Also prepare two straight pieces of tubing, one about 7 inches long, the other about 12 inches. Fire polish all pieces as directed above, but do not seal them. All these bends are to be made with glass tubing, not with glass rods. When they are complete, show them to the instructor for his approval.

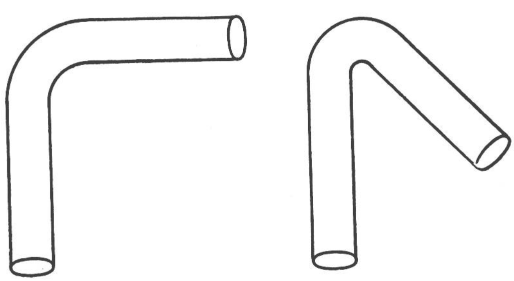


Figure 5

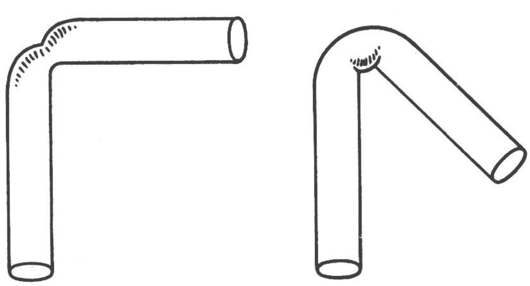


Figure 6

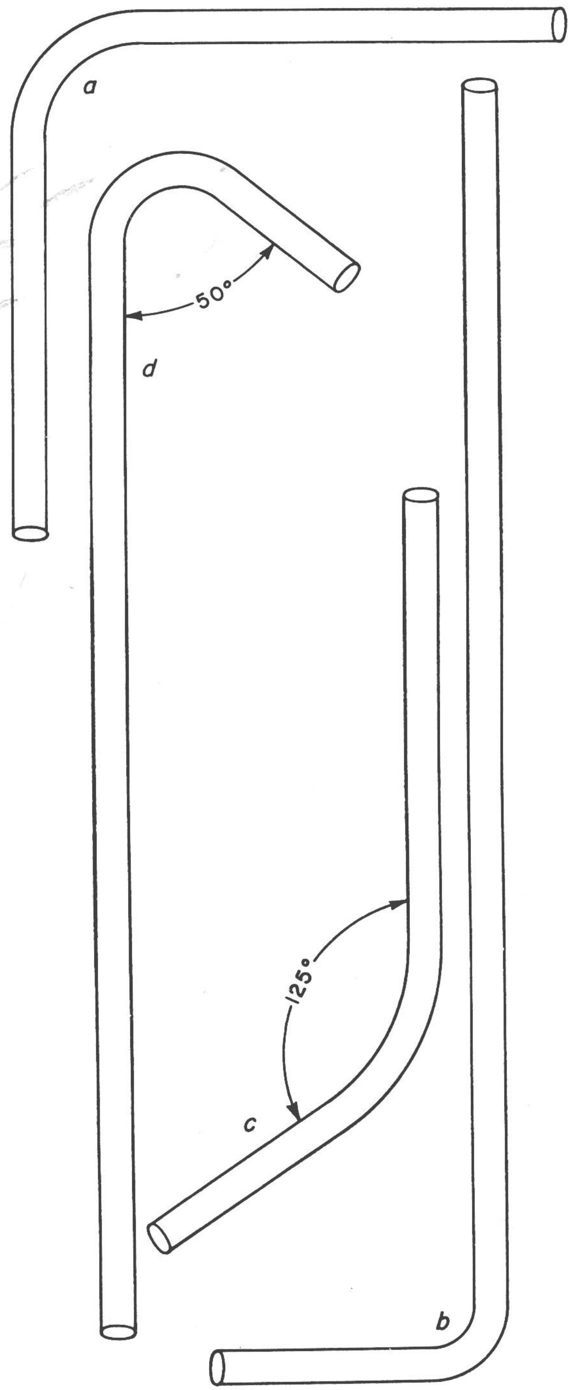


Figure 7

E. Wash bottle. Procure a plastic wash bottle. These are convenient and inexpensive and may be obtained from most chemical supply houses. Wash and fill with distilled water; no other material is ever to be used in it.

F. The metric system. The metric system of units is used in most scientific work and we have listed the commonly used units. You should familiarize yourself thoroughly with these units, and with the relationship of these units to common units of the English system.

The fundamental unit of *linear* measurement is the *meter*.

The fundamental unit of *weight* measurement is the *gram*.

The fundamental unit of *volume* measurement is the *liter*.

Commonly used prefixes of the above fundamental units are

deci	1/10 or 0.1
centi	1/100 or 0.01
milli	1/1000 or 0.001
kilo	1000

Thus 1000 meters is a kilometer, 0.1 gram is a decigram, 0.001 liter is a milliliter, and so on.

Some of the common English units and their approximate metric equivalents are

1 inch = 2.54 centimeters
1 meter = 39.37 inches
1 pound = 453.6 grams
1 liter = 1.06 quarts
1 kilogram = 2.2 pounds

For practice in evaluating units of the metric system, fill in the blanks in Exercise 1 at the end of this experiment.

G. Volumetric measurements. Numerous types of volumetric apparatus are used by chemists, but the simplest and most frequently used item is the graduated cylinder. Pour water into your cylinder to fill it partially. You will notice that there is a concave surface, with the water rising on the sides of the cylinder (Figure 8).

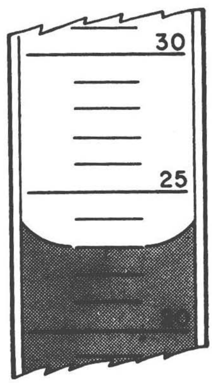


Figure 8

The question will arise: In Figure 8 does one read the volume as 23 or 24 ml? The answer is that the lower edge of the meniscus is read (except in the case of such a liquid as mercury, which exhibits a convex surface in a glass container)—thus in Figure 8 the volume is 23 ml.

Measure the volume of a test tube by filling it with water and pouring the water into the graduated cylinder.

Volume of test tube ______________ ml

H. Handling solid and liquid reagents. The laboratory instructor will demonstrate the technique of obtaining solids from bottles on the sideshelf and the pouring of liquids from reagent bottles. The instructor will also explain the arrangement of chemicals on the stock shelves. These instructions should be followed carefully: their application saves time and wasted motion, prevents contamination of contents of bottles, and makes working conditions in the laboratory more satisfactory.

I. Filtration. In the laboratory it is frequently necessary to separate a solid from a liquid. The usual method of separation is the simple filtration process shown in Figure 9.

The laboratory instructor will demonstrate the technique of preparing a filter paper and placing it in the funnel. The filtration process may be demonstrated by using a suspension of chalk in water. The proper procedure for washing the precipitate will also be demonstrated.

Using a graduated cylinder, measure 5 ml

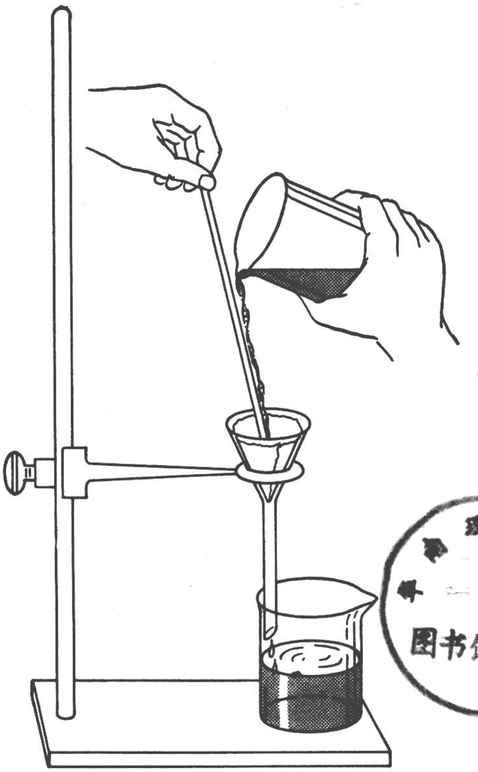


Figure 9

of copper (II) chloride solution (0.5 *M*) into a 100-ml beaker. Add exactly 10 ml of silver nitrate solution (0.5 *M*). Warm and stir for a few minutes to help coagulate the precipitate.

Silver Nitrate + Copper (II) Chloride →
Silver Chloride + Copper (II) Nitrate

Filter, wash the residue on the filter paper with about 5 ml of water, and catch the wash-

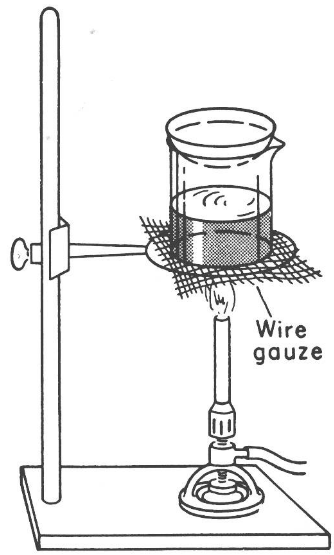


Figure 10

ings in a test tube. Add 1 ml of ammonium hydroxide to the washings. A blue color indicates the presence of a copper salt. Wash the residue until the ammonium-hydroxide test shows that all copper salts have been removed.

Open out the filter paper and expose the residue to sunlight for a short time. What are the results? What is an application of the observed results?

Place the filtrate in an evaporating dish and evaporate to dryness by placing the dish over a beaker of boiling water (Figure 10). What is the residue that remains?

J. Temperature scales. Refer to text, Chapter 2.

NAME ____________________

LABORATORY INSTRUCTOR ____________________

EXERCISES

1. Make calculations and fill in blanks.

(*a*) 1 centimeter (cm) = ____________ inch (in)

(*b*) 1 pound (lb) = ____________ kilogram (kg)

(*c*) 1 quart (qt) = ____________ liter (l)

(*d*) 1 ounce (oz) = ____________ grams (g)

(*e*) 100 meters (m) = ____________ yards (yd)

(*f*) 1 mile = ____________ kilometers (km)

(*g*) 1 gallon (gal) = ____________ milliliters (ml)

(*h*) 1 square inch (in^2) = ____________ square centimeters (cm^2)

(*i*) 1 cubic decimeter (dm^3) = ____________ cubic centimeters (cc)

2. Give the approximate metric equivalents of

(*a*) 100 yards.

(*b*) 5 gallons.

(*c*) 1 ton (weight).

3. Add: 5.25 g, 0.07 kg, 0.25 g, 550 mg, 3 dg, 50 cg.

4. Add: 2.5 l, 3500 ml, 0.25 l, 50 ml.

5. Add: 0.30 m, 250 cm, 2.4 m, 800 mm, 2.5 cm, 5 dm, 50 mm.

6. How many liters are contained in a cubic meter?

7. Make the following temperature conversions.
   (*a*) 98° F to C

   (*b*) 37° C to K

   (*c*) 104° F to C

   (*d*) −150° C to F

   (*e*) −150° C to K

   (*f*) 293° K to F

8. Calculate
   (*a*) the temperature at which a centigrade thermometer and a Fahrenheit thermometer give the same reading.

   (*b*) the temperature at which both are numerically the same but opposite in sign.

   (*c*) the Fahrenheit equivalent of absolute zero.

9. What are some of the advantages of the metric system over the English system of measurement?

EXPERIMENT 2

Practice in Weighing

Purpose. To attain skill in the use of several types of balances.

Special materials. Platform, triple-beam, and analytical balances.

DISCUSSION

One of the most common and important operations in a laboratory is weighing objects. Chemistry, being one of the exact sciences, calls for the careful determination of the amounts (weights) of substances which enter into and result from chemical change. This experiment will acquaint you with various types of balances used for weighing objects.

Several types of balances for weighing things may be employed in a chemical laboratory. Balances are among the finest and most accurate pieces of laboratory equipment and, to be maintained in good condition, they must always be treated with great care. Certain general rules must be observed.

1. Keep the balance clean.
2. Balance adjustments should be made only by the instructor or his assistant.
3. Never place any chemical directly on the balance pan; always use a piece of paper or a weighing dish.
4. The object or substance being weighed is always placed on the left pan; the weights are always placed on the right pan.
5. Never (except when using rough *weights*, such as those of the platform balance) pick up weights with the fingers; always use forceps.

The type of balance used in an experiment will depend on the accuracy desired or required for that particular experiment.

PROCEDURE

A. Rough weighings. For *rough* weighings, where accuracy of no greater than 0.1 g is required, the platform balance may be used (Figure 11). Weigh an evaporating dish on the platform balance, and record the weight in the table below. Place several chunks of roll sulfur in the evaporating dish and weigh it. Record the data in the table and put the sulfur back into the supply bottle.

Weight of evaporating dish and sulfur ________ g

Weight of evaporating dish ________ g

Weight of sulfur ________ g

B. Triple-beam balance. The triple-beam balance (Figure 12) is conveniently used when an accuracy no greater than 0.01 g is required. For most experiments in the beginning course, this degree of accuracy is sufficient.

Weigh a penny and a nickel separately on the triple-beam balance.

Weight of penny ________ g

Weight of nickel ________ g

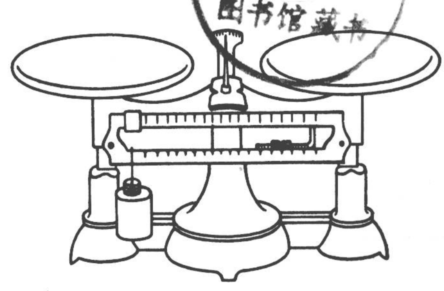


Figure 11