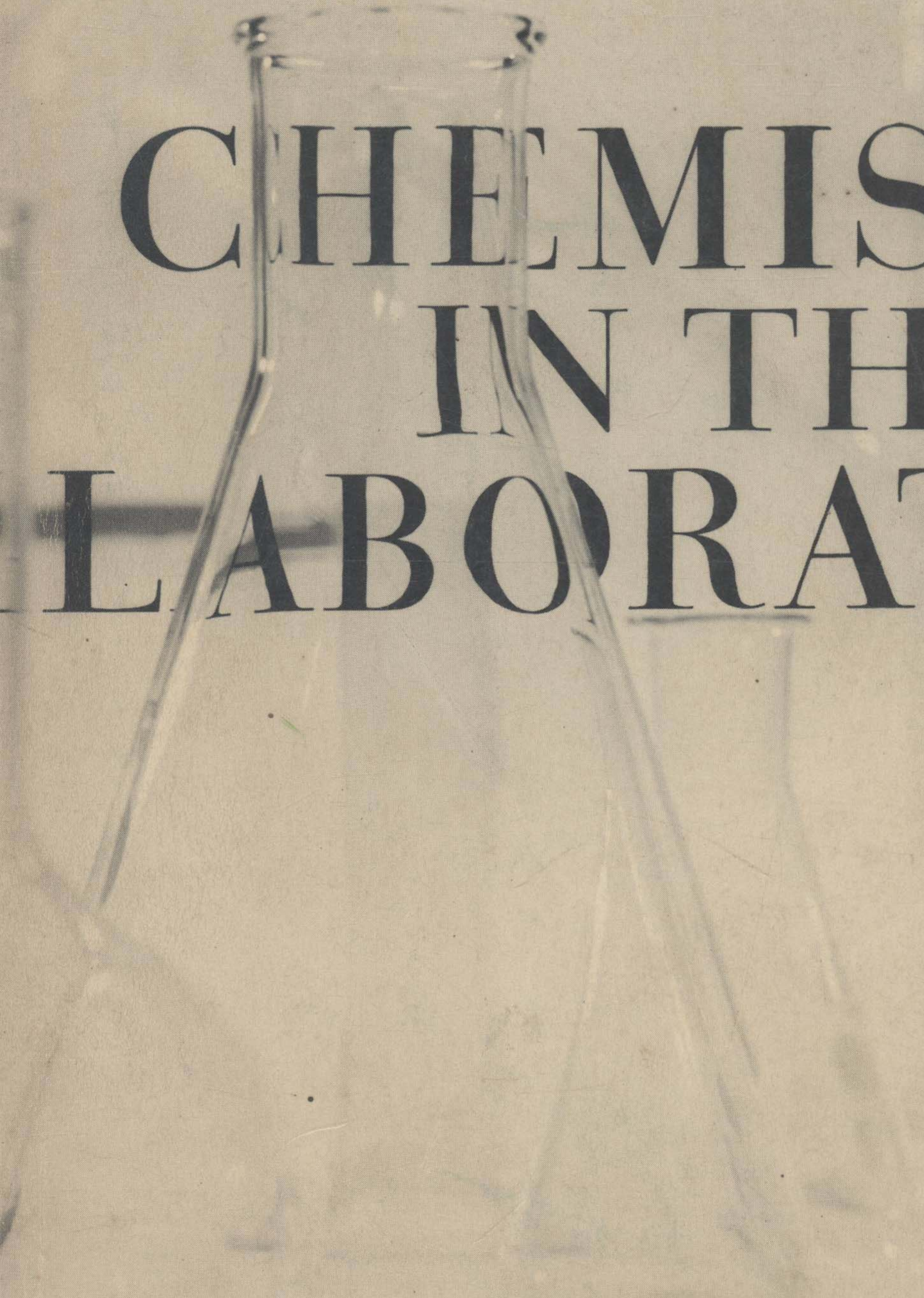

CHEMISTRY IN THE LABORATORY

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W · W · NORTON & COMPANY · INC · NEW YORK

Library of Congress Catalog Card No. 64-10891

Published simultaneously in Canada
by George J. McLeod Limited, Toronto

PRINTED IN THE UNITED STATES OF AMERICA

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Preface

In the preparation of this laboratory manual we have attempted to achieve the following objectives: (1) To provide laboratory work appropriate for use in conjunction with the textbook *Chemistry* by the present authors or any other text designed for a terminal course in chemistry. (2) To include experiments that focus attention upon the techniques and methods of the science together with others that demonstrate the bearing of chemistry upon the day-to-day experiences of the individual. (3) To minimize the number of experiments that are largely descriptive and qualitative in character and to emphasize the quantitative aspects of chemistry. (4) To provide sufficient experiments to give the teacher considerable latitude of choice in the assignment of laboratory work. (5) To include experiments that are relatively simple and may be performed with only the less expensive kinds of equipment and chemicals.

We subscribe to the belief that, even in the terminal course, efforts to develop any real appreciation of the true nature of the science of chemistry without laboratory work are wholly unrealistic. The time devoted to this phase of the course, however, need not be excessive. The course in which this manual is used at The University of Texas includes one two-hour laboratory period each week for two semesters. In a typical year, some twenty-five to thirty experiments are assigned, and the selection is varied from year to year.

The authors wish to express their gratitude particularly to many laboratory instructors whose suggestions for improvement of the discussions and procedures have been incorporated in the present manual.

George W. Watt
Lewis F. Hatch
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Austin, Texas
January, 1964

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* Experiment 32 requires several weeks for completion. If Experiment 32 is to be performed as part of the laboratory course, it is suggested that this experiment be started sufficiently far in advance of the laboratory period for which it is assigned.

NAME.. DESK NO.....................

Record of Assignments

Date	Experiment No.	Date	Experiment No.

Introduction to the Laboratory

Chemistry has been described as "the science that treats of the composition of substances, and of the transformations which they undergo," and a science is defined as "a branch of study which is concerned with observations and classification of facts, especially with the establishment and the quantitative formulation of verifiable general laws." It is apparent that if chemistry is a science, an important consideration in the study of chemistry is a familiarity with the methods and techniques employed by the chemist in making observations and obtaining the facts that "treat of the composition of substances and the transformations which they undergo." The laboratory is the source of the data on which are built the "quantitative and verifiable general laws," and the experiments presented here are designed to illustrate some of the techniques and methods commonly employed in the laboratory.

During the first laboratory period the student will be assigned equipment, and become familiar with the general procedures for operation of the laboratory, and if sufficient time remains, he should start on the assignment designated by the instructor.

Inspection of Desk Equipment. The laboratory desk locker to which each student is assigned contains numerous items of chemical laboratory apparatus. Upon being assigned to a desk, having received a desk key, and having inspected this equipment, the student is responsible for the contents of the locker. At the first laboratory period, each student must check the equipment against an inventory card provided by the instructor. This is to ensure that the items specified on the card are present and in good condition; it is expected that the equipment will be returned on the last laboratory period in the same condition as when received. As a means of assisting the student in identifying the various items, diagrams of numerous common pieces of chemical apparatus are shown in Fig. I.1.

The laboratory instructor will provide additional directions for the checking of the desk equipment, the opening of a storeroom account, and any other functions that are necessary in the conduct of the laboratory work of the course.

General Equipment. In addition to the individual sets of desk equipment contained in the lockers, certain items of apparatus are placed in the laboratory room for the common use of all students in the same or different laboratory sections. These include desk reagents, side-shelf reagents, iron rings and ring stands, gas-collection bottles, pneumatic troughs, etc. Since these items must be available at all times, they should never be locked in the desk of an individual student.

Safety and Laboratory Rules. Generally, undergraduate instructional laboratories are crowded and the student working in one must exercise care not only for his own safety, but also for the safety of the other students present. The following procedures, if adhered to explicitly, will eliminate the cause of the majority of accidents.

1. Unassigned and unauthorized experiments are not to be performed. Violation of this rule is subject to the severest disciplinary action.

2. Always protect your hand with a towel when cutting glass tubing or inserting glass tubing into a stopper. The laboratory instructor will illustrate the technique to be used.

3. Never point a test tube containing a reacting mixture or a mixture which is being heated toward your neighbor or yourself.

4. If the occasion arises to prepare a dilute acid solution, never pour water into concentrated acid. Always pour the acid slowly into the water while stirring the latter constantly.

5. If you are "burned" by a chemical, immediately rinse the burned areas under a rapidly running stream of water and have your neighbor summon the laboratory instructor.

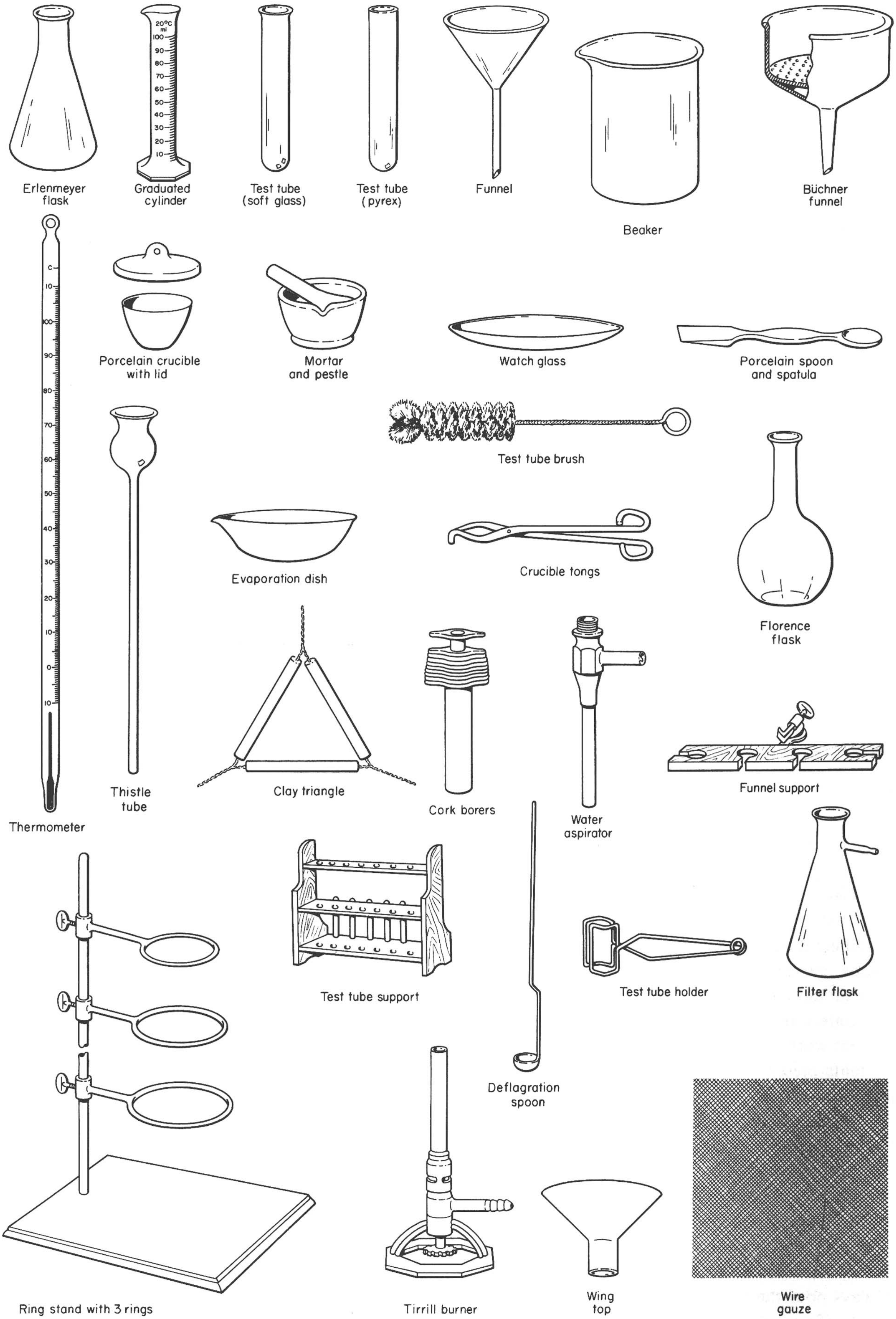
Erlenmeyer flask
Graduated cylinder
Test tube (soft glass)
Test tube (pyrex)
Funnel
Beaker
Büchner funnel
Thermometer
Porcelain crucible with lid
Mortar and pestle
Watch glass
Porcelain spoon and spatula
Test tube brush
Thistle tube
Evaporation dish
Crucible tongs
Florence flask
Clay triangle
Cork borers
Water aspirator
Funnel support
Test tube support
Deflagration spoon
Test tube holder
Filter flask
Ring stand with 3 rings
Tirrill burner
Wing top
Wire gauze

Fig. I.1

6. Read the label carefully before removing a chemical from its container. Unless specifically directed to do so, *never* return unused chemicals to their storage containers; for this reason, it is important to take no more of a chemical than is required for the purpose of the experiment.

7. Dispose of all soluble solid chemicals or solutions in the sink. All other chemicals, as well as paper and matches, are discarded in the waste containers specified by the laboratory instructor.

8. Experiments, or operations in which gases with objectionable characteristics are produced, should be performed in a hood. Consult the laboratory instructor if in doubt as to when to use a hood.

General Laboratory Procedure. Student efficiency in the laboratory is enhanced if the following procedures are observed:

1. The assignment for a given laboratory period should be studied carefully prior to coming to the laboratory. In general, the entire laboratory period is utilized in performing the experiments assigned, and little time is available for studying the assignment.

2. All data must be recorded in the manual. If time permits, the student should perform all necessary calculations and answer all questions prior to leaving the laboratory. Otherwise, the data sheet is initialed by the laboratory instructor and the experiment handed in for grading at the beginning of the next period.

3. Reagent bottles that are on the side shelves should *never* be taken to the laboratory desk. Use a clean beaker or a test tube to carry chemicals to your desk, and do not take any more than you need because many of the chemicals are costly.

4. Return reagent bottles to their proper place on the shelf immediately after you finish using them.

5. Clean up immediately any chemicals that you spill.

6. At the end of each laboratory period wipe off the top of your laboratory desk, make certain the reagent bottles over your desk are in place, check to see that the gas and water are turned off, and return all special equipment to the storeroom.

Study of a Gas Burner. Several different types of gas burners are commonly used in the laboratory of the elementary course. Some of these are shown in Fig. I.2. The burner is usually one provided with an adjustment device by means of which one may regulate the composition of the mixture of fuel gas and air.

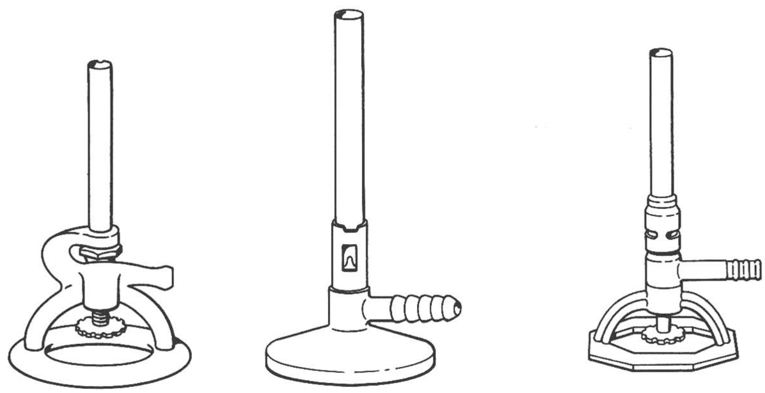

Fig. I.2

Attach the burner to the gas outlet by means of a piece of rubber tubing. Close both the gas and the air inlets at the burner and turn the gas on full at the supply outlet. Open the gas inlet at the burner and light the gas. Practice adjusting the burner by regulating the relative amounts of air and gas. Enlist the aid of the laboratory instructor if difficulty is encountered.

Close the air inlet at the burner and observe the character of the yellow luminous flame. Hold a clean porcelain dish in this flame and note the deposit of soot, which consists of particles of carbon and which indicates incomplete combustion of the fuel gas. Adjust the air supply until a non-luminous flame approximating that represented by Fig. I.3 results. If the optimum adjustment is realized, at least three distinct cones should be visible, the approximate temperatures of which are indicated in Fig. I.3. Warm a clean porcelain dish carefully (so that it will not break because of the too-sudden change in temperature when brought into contact with the non-luminous flame), insert it into the flame, and observe the result. Hold a match or splint of wood across the flame near its base and note that the wood is not burned in the inner

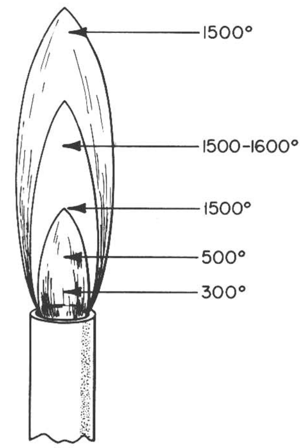

Fig. I.3

and cooler region. Attempt to insert a match head into this region without ignition. Hold a platinum wire in different regions of the flame and note the corresponding differences in the glow.

Burners of the type used in these experiments are sufficient for most common heating operations. If one wishes to provide higher temperatures, one may use a blast lamp, in which a larger quantity of fuel gas is burned per unit time in a restricted space. The operation of a blast lamp will be demonstrated by the laboratory instructor.

Glassworking. Several of the experiments that follow require that the student construct simple apparatus. In preparation for these experiments, it is necessary that each student become familiar with the more elementary aspects of the manipulation of glass tubes and rods. Ask your laboratory instructor for assistance with those techniques with which you have difficulty.

Cutting of Glass Tubes or Rods. Place a short piece of glass tube or rod flat on the desk top, and make a single scratch across the tube or rod by means of a triangular file. Protecting your hands with a towel, grasp the tube with both hands, with the thumbs opposite the scratch, which should be on the side away from the body. At the same time pull and bend the tube outward. If it does not break, make a deeper scratch with the file, and repeat.

Fire Polishing. Glass tubes or rods that have been cut as directed above should have the edges smoothed. The ends of a glass tube are best fire polished by holding them in the burner flame while rotating the tube slowly until the glass just begins to fuse (Fig. I.4). Care should be exercised to avoid reducing the inside diameter of a tube as the result of overheating. The ends of glass rods should be heated similarly until the ends are rounded.

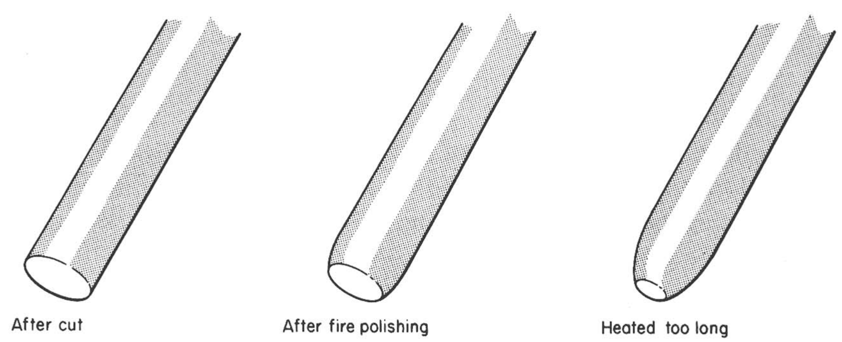

Fig. I.4

Preparation of Stirring Rods. Cut off two pieces of glass rod 12 to 15 cm. (5 to 6 in.) in length. Fire polish both ends of each piece, and preserve these for use as stirring rods. Stirring rods must always be fire polished to avoid scratching smooth glass surfaces with which the ends of these rods come into contact.

Bending of Glass Tubes. The successful bending of glass tubes depends largely upon getting the glass sufficiently and uniformly hot. Most beginners make the mistake of attempting to bend tubes which have been heated more on one side than on the other or which are at too low a temperature (Fig. I.5).

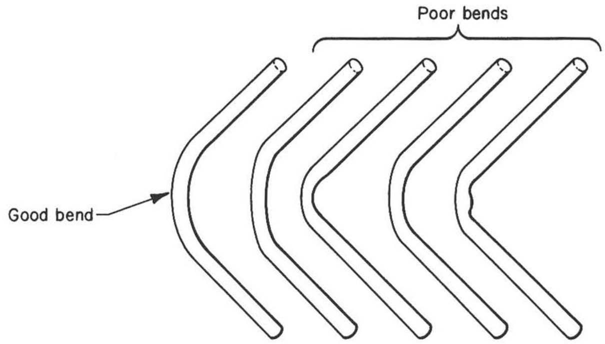

Fig. I.5

Attach a wing top to the burner and adjust the flame so that it is about 2 in. across the top. Heat a piece of 6-mm. glass tube by rotating it in the flame until the tube begins to sag. Quickly remove the tube from the flame, place it flat on a piece of asbestos board, and bend it to the desired angle (see Fig. I.5). Cut two 15-cm. (6-in.) lengths of 6-mm. glass tube and make two good right-angle bends like that shown in Fig. I.5. Fire polish both ends of each tube, and save the bends for future use.

Exercise in the Use of the Balance

The use of the balance in the determination of the weight relationships involved in chemical changes is one of the most important and useful operations performed by the chemist. It is essential that the beginning student have some practice in the measurement of weight before attempting some of the later experiments.

Instruction in the Use of the Balance. Laboratories used by students in the elementary course are commonly supplied with two types of balance. The type shown in Fig. 1.1 is used for weighings that do not require a high degree of accuracy. That shown in Fig. 1.2 is considerably more sensitive and is used for weighings in which weight must be determined to at least ± 0.01 g. Both of these balances operate on a simple lever principle like an ordinary seesaw. A straight beam is rested upon a knife-edge. An unknown weight at one end of this beam is measured by being balanced against known weights at the other. The light materials of construction and delicate adjustments necessary for accuracy make the balance readily subject to damage, but a few reasonable precautions serve to obviate such difficulties.

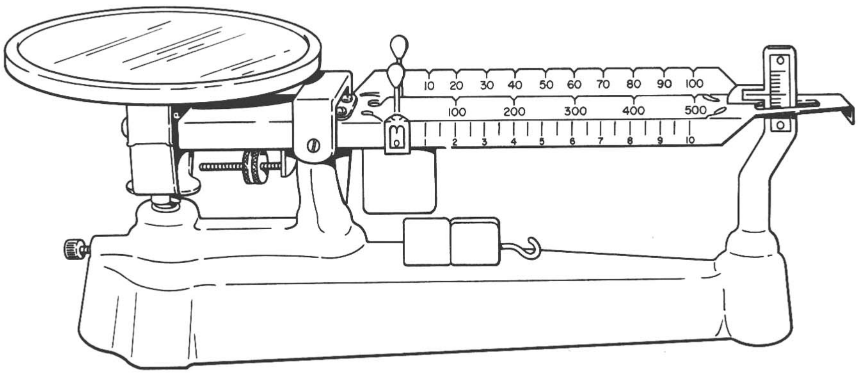

Fig. 1.1

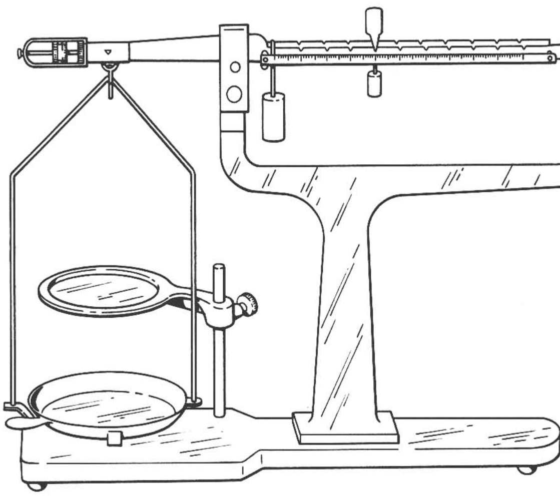

Fig. 1.2

The laboratory instructor will explain the function of the various parts of each type of balance, demonstrate the proper procedure to be used in the determination of weight, and make suggestions concerning the care and handling of the balances. In addition, the student should study the following outline carefully before attempting to use either type of balance.

Procedure for Use of the Balance. 1. Tap the beam gently and observe whether or not it swings freely and smoothly. On the more sensitive balances, a screw adjustment is provided to raise the beam off the knife-edge when not in use. The beam must always be lowered *carefully* for use and raised off the knife-edge upon completion of a weighing operation.

2. Check the zero point. With the pans or platform clean and empty and all the sliding weights in the zero notches, the beam should come to rest with the pointer at zero. If this does not occur, ask the instructor to assist in the adjustment of the balance. Hands must be clean and dry because dirt and moisture change the weights of the various parts temporarily and may also cause corrosion of the metal. Do not move the balance. Shaking or jarring changes the adjustment of the zero point and may also chip the knife-edge, bend the pointer, or otherwise damage the balance.

3. Carefully place the container holding the sample on the balance pan. Never *drop* materials onto the pan. Always use a separate container for the sample because numerous chemicals corrode the metal pans. When weighing out samples of solids, a piece of hard-surfaced paper placed upon the pan is satisfactory; for liquids use a small beaker or porcelain dish. The outside of the container must be clean and dry. Do not pour liquids into a container when it is standing on the balance pan.

4. Move the weights outward, using the largest weight first for the rough adjustment, then the smaller ones in order. When the pointer once more comes to rest at zero, the sum of the weights indicated on the scales on the beam is equal to the weight of the sample plus the container. Ordinarily the container is weighed first, and its weight is subtracted from the total to get the weight of the sample. The same balance should be used for any series of related weighings.

5. Lift the beam, return all weights to the zero notches, and remove the sample and container. Leave the balance and its surrounding area clean and ready for the next weighing.

DIRECTIONS*

I. Determination of Weight Using the Triple-beam Balance.

A. Determine the weight (to the nearest 0.01 g.) of the following objects from the equipment in the laboratory desk: (1) porcelain crucible; (2) pyrex test tube; (3) 250-ml. Erlenmeyer flask. Record the weights in the appropriate spaces on the data sheet.

B. Determine the weight of a clean, dry 100-ml. beaker. By means of a graduated cylinder, measure out exactly 25 ml. of distilled water, and transfer *all* of this water to the weighed beaker. Weigh the beaker and its contents, and from the data now available calculate the weight of 25 ml. of distilled water and the weight of 1 ml. of distilled water. In a similar manner determine the weight of 1 ml. of three of the reagents which are found on your laboratory bench; on the data sheet identify the reagents you employ.

II. Determination of Weight Using the Platform Balance.

Determine the weight of a clean, dry 500-ml. Erlenmeyer flask. By means of a graduated cylinder, place in the weighed flask exactly 350 ml. of distilled water. Weigh the flask and its contents, and from the resulting data calculate the weight of 350 ml. of distilled water and the weight of 1 ml. of distilled water.

* On this, as on all the laboratory experiments to follow, students are to work individually unless specifically directed to work in groups.