LABORATORY MANUAL

ARRANGED TO ACCOMPANY

"A COURSE IN GENERAL CHEMISTRY"

McPHERSON AND HENDERSON

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GINN AND COMPANY

BOSTON · NEW YORK · CHICAGO · LONDON
ATLANTA · DALLAS · COLUMBUS · SAN FRANCISCO

516.4

The Athenæum Press
GINN AND COMPANY · PROPRIETORS · BOSTON · U.S.A.

PREFACE

From an educational point of view, chemistry is really the oldest of the experimental sciences. The problem as to what laboratory work should constitute the first year's course is therefore one to which a great deal of thought has been devoted, and many educational experiments have been made in an endeavor to solve it. Much ingenuity has been exercised in the development of suitable laboratory experiments and in the invention of simple apparatus adapted to a beginner's inexperience, and a great wealth of admirable illustrative experiments is now at the command of every teacher.

For one who sets about the task of arranging an experimental course for the beginner, there remains little opportunity for originality or invention. His problem is rather one of selection. Accordingly this laboratory manual lays no claim to originality, either in method or in content. It has been slowly developed in connection with the large beginning classes in which the authors have been interested, and has been revised and reprinted privately a number of times. In response to many requests it has again undergone a thorough revision and has been arranged to accompany the text by the authors, entitled, "A Course in General Chemistry."

In common with nearly all teachers of chemistry, the authors have had to deal with the fact that the first course in college comprises students who have had an elementary course in the high school, as well as those who have had no earlier introduction to chemistry. As far as the laboratory is concerned, the authors have found that the most practical solution of this problem is to develop a manual ample enough to meet the needs of both classes of students. The more experienced student can then omit the exercises with which he is familiar, and the student with less experience can omit some of the quantitative exercises.

Every well-ordered laboratory has its own system of supplies, stock solutions, desk reagents, and locker equipment, and the directions in a manual will not always harmonize with this system. For example, in many cases in which the student is directed to prepare solutions or mixtures for specific purposes, it may be much better to have a supply on the side shelf, properly labeled, for general use. In a number of experiments, particularly those involving apparatus that is not a part of the locker equipment, two or even more students may work together to advantage. The time at the disposal of the class will not always permit each student to complete all of the exercises, and variety is added by assigning parallel experiments to alternate students. All such adjustments are left to the instructor.

In the Appendix will be found a number of suggestions to the instructor, relating to the details of special apparatus or reagents. Suggestions are also offered as to a suitable locker equipment and as to the apparatus that the student will need from time to time. Articles may obviously be transferred from one list to another according to the funds available or the capacity of the lockers. A list of the reagents called for in the manual is provided, together with an estimate of the quantities required for a class of ten students.

A large page has been chosen for the manual, since it makes a convenient book for filing in laboratory pigeonholes and for handling during correction. The blank pages can be used for a full record of laboratory observations, or more elaborate reports may be written from the recorded notes if the instructor so desires.

Ohio State University
Columbus, Ohio

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LABORATORY MANUAL

CHAPTER I

MANIPULATION AND FUNDAMENTAL PRINCIPLES

1. The Bunsen burner. ***a.*** The Bunsen burner is the common form of apparatus used in laboratory operations where heat is applied. It consists of the tube *A* (Fig. 1), screwed into the base *C*. The tube has two small round holes near its lower part. A band *B*, provided with similar holes, fits around the lower part of the tube in such a way that the holes in the tube may be closed or opened by turning the band. Gas is admitted to the burner through *D* by means of rubber tubing. Unscrew the tube and examine the different parts of the burner; then put them together again and light the gas by holding a lighted match 4 or 5 cm. above the tube and turning on the gas. The gas should be adjusted so as to give a flame about 10 cm. high. The gas entering the burner mixes with air drawn in through the holes in the lower part of the tube and burns with an almost nonluminous flame. If the band is adjusted so as to close the openings, the flame becomes luminous. Always use the nonluminous flame unless otherwise directed.

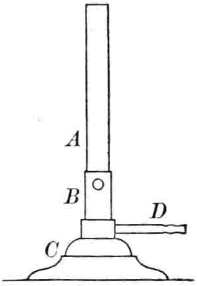

FIG. 1

b. Hold an iron wire in various parts of the flame to gain an idea as to the relative temperatures. Pass a piece of glazed paper transversely through the flame, holding it steady for an instant across the flame. What information do you get from the way in which the paper is scorched? Pass a piece of paper edgewise through the axis of the flame in the same way.

c. From these experiments draw a diagram of the flame, indicating the hot and the cooler regions.

2. Heating a liquid in a test tube. Hold the test tube between the thumb and fingers (Fig. 2), constantly rotating it backward and forward so as to apply the heat uniformly. The heat should be applied to the upper portion of the liquid, care being taken, however, that the flame does not strike the tube above the level of the liquid. In case the upper part of

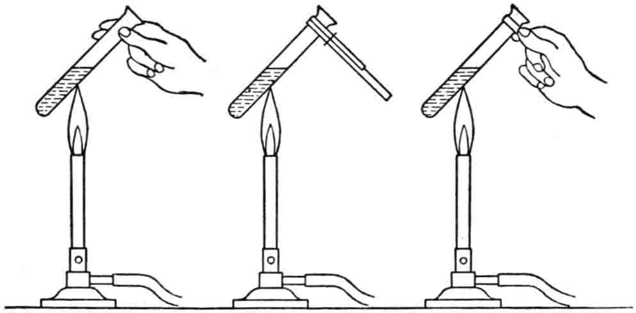

FIG. 2 FIG. 3 FIG. 4

the tube becomes heated it may be supported by a test-tube holder (Fig. 3) or by a band of paper circling the upper part of the tube (Fig. 4).

The sudden formation of vapor at the bottom of the tube sometimes causes the contents of the tube to be thrown out; hence care must be taken not to point the tube toward anyone.

Half fill a test tube with water and apply heat until the water boils rapidly.

3. Pouring a liquid from one vessel to another. In this operation care must be taken to prevent the liquid from running down the side of the vessel from which it is poured. A glass rod should be held lightly against the rim of the vessel, as shown in Fig. 7. The liquid will flow down the rod. Fill a beaker with water and transfer it slowly to another vessel without using the glass rod; repeat, using the glass rod. What difference is noted?

In pouring a liquid from a bottle a glass rod may be used; or the neck of the bottle may be placed lightly against the rim of the vessel into which the liquid is being poured (Fig. 5). This will prevent the liquid from running down the side of the bottle. Obviously the stopper should never be laid down on the desk. It should be caught between the fingers, as shown in Fig. 6. This leaves the hand free to grasp the bottle, as shown in Fig. 5.

4. Decantation. It is often necessary to separate a liquid from a finely divided solid which is suspended in it. This

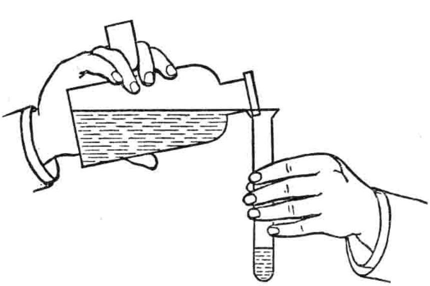

FIG. 5

FIG. 6

may be done most simply by *decantation.* The operation consists in allowing the solid to settle and then pouring off the liquid. The method can be used only when the solid is heavy and readily settles in the liquid. Shake up some fine sand in water, then allow it to settle and *decant* the water.

5. Filtration. As a rule the solid will not readily settle or will do so only after long standing. In such cases the mixture is *filtered*; that is, poured on a filter paper, which allows the liquid to run through but retains the solid. To prepare the filter paper, fold it along a diameter into halves, then, at right angles to the first fold, into quarters. The folded filter is then opened so as to form a cone, half of which is composed of three thicknesses of paper and the remainder of one thickness. Fit the cone into a funnel, of such a size that the paper does not quite reach the top. The paper must accurately fit the funnel; if it does not, make it do so by varying the fold. Place the paper in the funnel and thoroughly wet it with water. After the water has run through, press the paper firmly against the sides of the funnel with the finger so as to remove any air bubbles between the paper and the glass. The filter is now ready for use.

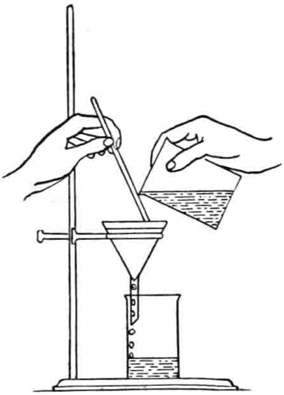

FIG. 7

The process of filtration not only enables us to separate liquids from solids but also certain solids from each other. To illustrate this fact, grind a piece of chalk to a powder in a mortar and mix the product intimately with about an equal bulk of common salt. Now separate the two solids as follows: Place the mixture in a small beaker, add about 50 cc. of distilled water, and stir with a glass rod. (The sharp ends of the rod must be rounded by rotating them in a flame, otherwise the beaker may be scratched and broken.) The salt dissolves, forming a *solution.* Filter off the insoluble chalk, collecting the *filtrate* (the clear liquid which passes through the filter paper) in a beaker (Fig. 7). The salt may be recovered from the filtrate by the process of *evaporation.* To perform this operation, pour the filtrate into an evaporating-dish, then support the dish on a ring stand (Fig. 8) and heat gently. The tip of the flame should not quite touch the dish. The liquid may be made to simmer, but should not be made to boil violently (why?). Withdraw the heat as soon as the water is evaporated. Note the residue left. Convince yourself that it is salt.

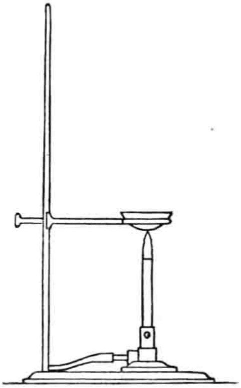

FIG. 8

6. The metric system. ***a.*** *Length.* By means of the scale on the front cover of the notebook measure the length (in centimeters) of various pieces of apparatus included in your outfit, as a test tube, file, and blowpipe. What is the diameter of your filter paper? Estimate the lengths of various objects, as a pencil, a test tube; then measure. Continue until you can approximate the lengths of small objects.

b. *Volume.* By means of a graduated test tube or cylinder measure (in cubic centimeters) the volumes of various test tubes, beakers, and flasks included in your outfit. (In reading the volume of the liquid in a graduated tube always read from the lower part of the *meniscus;* that is, the curved surface of the liquid.)

7. The balance. The weight of a body is determined on a balance, which must be adapted to the degree of precision required. For the experiments undertaken in this course the balance must weigh accurately to one one-hundredth of a gram, and, if possible, to one one-thousandth. It is convenient to remember that a gram is approximately one thirtieth of an ounce.

To be of service for even moderately accurate weighing, a balance must be used with the greatest care. The following directions must be faithfully observed:

1. At all times, save during an actual weighing, the beam must be lifted from its bearings by turning the thumbscrew at the base of the case.

2. All changing of objects to be weighed and of weights must be done after the beam has been lifted. Never take off a weight while the beam is swinging.

3. Never handle the weights in any manner save with the pliers provided with the weights. Be careful not to dent the weights by rough use with the pliers.

4. Keep the balance pans, the case, and the balance shelf scrupulously clean.

5. Never weigh chemicals directly on the pan of the balance. First weigh a suitable container,— a watch glass or a small beaker,— then place the material in this and again weigh.

8. The process of weighing. ***a.*** See that upon careful lowering of the beam the vibrations of the pointer are equal on both sides of the zero point. If there is any considerable deviation, have the instructor adjust the balance.

b. Place the object to be weighed on the left pan and the weights you judge to be sufficient on the right pan. Turn the arrest screw very slowly until you see in which direction the pointer starts to move. Arrest the beam and make a new adjustment of weights, repeating the operation until the desired equilibrium point is found.

c. Add up the weights lying on the pan, and verify the count by the empty spaces in the box. Always have the weights either on the pan or in the box, and never lay them on the floor of the balance case.

d. Record your weighings directly in your notebook — never on scraps of loose paper. Observe that the weights are in decimals and that their sum should be expressed in grams and decimal fractions thereof.

e. Select a clean watch crystal or a small beaker which you will be using for weighing. Carefully weigh it and record the weight in your notebook. With a sharp file make a slight scratch upon the beaker just below the lip. Fill the beaker with distilled water from a graduated measure and note the volume added. Weigh the beaker and water and determine the weight of the water. How does the weight in grams compare with the volume in cubic centimeters? Why is this? Make a mark on a test tube about 2 cm. from the top. Weigh the empty tube and also the tube filled with water to the mark. Determine the volume to the mark. Make use of the tube in measuring out liquids until you grow accustomed to estimating volumes in cubic centimeters.

9. Manipulation of glass tubing. ***a.*** *To cut glass tubing.* Place the tubing on the desk and draw the edge of a triangular file across the point at which you wish to cut the glass.

NOTE. In all operations requiring the application of a strong heat to glass the heat must be applied gently at first; also, the highly heated glass must be cooled slowly to anneal it properly.

After the glass is scratched, take the tube in the hands with the thumbs placed near together, just back of the scratch (Fig. 9), and gently pull the glass apart, at the same time exerting a slight pressure with the thumbs. Do not try to break the tubing as you would break a stick, for this will

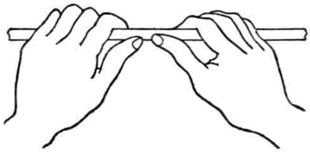

FIG. 9

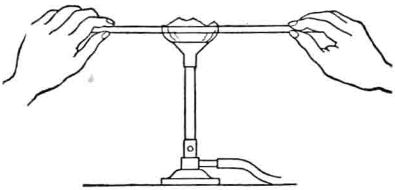

FIG. 10

splinter the glass and will never give a square end. If the tube does not yield readily to the gentle pressure, a deeper scratch must be made. In the case of large tubing it may be found necessary to file a groove around the tube. The edges of the cut tube will be sharp. They should be rounded by rotating them in the tip of the Bunsen flame. This process is called *fire-polishing*.

b. *To bend glass tubing.* Use the luminous Bunsen flame, spread out by means of the so-called "wing-top" burner (Fig. 10). Hold the tube lengthwise in the flame, gently rotating it so that all sides may be equally heated. Continue the heating until the glass *easily* bends, then quickly remove it from the flame and bend to the desired shape (Fig. 11, *A*). Great care must be taken to heat the tube uniformly, otherwise the bore of the tube will be contracted (Fig. 11, *B*), forming a bend which not only is unsightly but is easily broken.

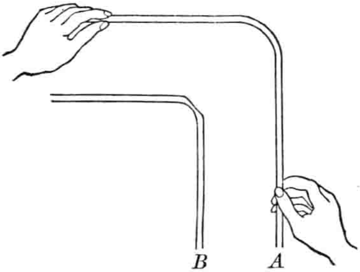

FIG. 11

c. *To insert a glass tube in a cork.* The cork should be of such a size that the smaller end will just enter the flask or bottle in which it is to be used. Soften the cork by rolling it between the desk and a piece of wood. To insert a glass tube select a borer slightly smaller than the tube. Place the cork on the desk and cut about halfway through it, not by punching, but by rotating the borer under gentle pressure (Fig. 12). Reverse the position of the cork and cut through from the other end. Care must be taken to keep the borer at right angles to the cork. The hole should be straight and smooth. The glass tube, rounded at the edges, is now inserted by a gentle screwlike motion. If the hole is too small to admit the tube when a gentle pressure is applied, it may be slightly enlarged with a round file. The tube slips in better if both cork and tube are wet.

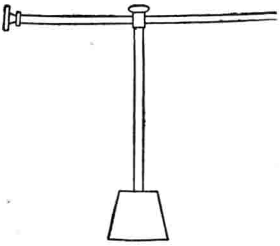

FIG. 12

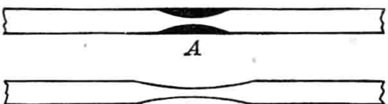

FIG. 13

d. To draw out a glass tube to a small bore. Heat a portion of the tube in the Bunsen flame until the walls of the heated portion thicken and the size of the bore diminishes (Fig. 13, *A*). The tube must be constantly rotated to prevent the softened portion from sagging. Now quickly remove the tube from the flame, and, holding it in a vertical position, gently pull the ends apart until the bore is of the desired size (Fig. 13, *B*). A glass jet may be formed by cutting the tube at *B*, and rounding the edges in a flame.

e. After practicing the above manipulations, make a wash bottle according to Fig. 14. Use a 500-cc. flask. *A* and *B* are glass tubes. *B* is connected with the

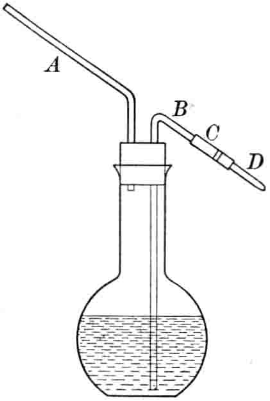

FIG. 14

glass jet *D* by a piece of rubber tubing *C*. The edges of the glass tubing must be rounded. When the wash bottle has been approved by the instructor, fill it with distilled water and set it aside for future use.

10. Chemical action. ***a.*** Hold a piece of iron wire in the Bunsen flame for a few seconds. Is the iron changed? Examine it when it has cooled. Have the properties changed? Has a chemical action occurred?

b. Repeat ***a***, using a splint of wood in place of the iron wire. How does the change produced differ from that in ***a***? Has a chemical action occurred?

c. Place enough sugar in a clean, dry test tube to cover the bottom to a depth of 1 cm. Heat it gently in the tip of the flame as long as any changes are produced. Note all the changes. Is the product sweet? Is it soluble in water? Do any properties remain unchanged? What grounds do you have for assuming that a chemical action has taken place?

d. Place about 1 g. of common salt in a test tube and dissolve it in a little water. Pour the clear solution into an evaporating-dish and evaporate to dryness. What is the solid left? How do its properties correspond to those of the original salt?

e. Cover a small piece of zinc in a test tube with about 5 cc. of water and add carefully 3 or 4 drops of sulfuric acid. Notice that the zinc dissolves with the evolution of a gas. Hold a burning splint at the mouth of the test tube and note the result. After the action has entirely ceased, filter off any undissolved zinc and evaporate the solution to dryness (hood) as in ***d***. How does the change differ from that in ***d***? Distinguish between the two examples of solution.

11. Elements; compounds; mixtures. ***a.*** What is an element? Are iron and sulfur included in the list of elements? Weigh out separately (on paper) 2 g. of sulfur and 2 g. of clean iron filings and make a careful list of their properties. Try the effect of a magnet on each. Now mix the two, and grind them together intimately in a mortar. Examine the product with a magnifying-glass. Can you distinguish the iron from the sulfur? Can you separate them with a magnet? Have they undergone any change in properties? What is such a material called?

b. Now place the product in a clean test tube and heat gently. As soon as the mass begins to glow, quickly withdraw the tube from the flame. Does the mass continue to glow? Now heat it strongly for one or two minutes; then cool the tube, break it, and examine the product with a magnifying-glass. Can you now distinguish between the iron and the sulfur? Try the effect of the magnet. Of what is the substance composed? When elements combine chemically, do they retain their original properties? What is the product of such a combination called?