LABORATORY MANUAL OF GENERAL CHEMISTRY

BY

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PREFACE

THIS manual was written to accompany the author's General Chemistry. In its present form it represents the third revision of mimeographed notes used by the author during the past thirteen years.

The experiments are more than sufficient in number for a year's work, so some must be omitted. It has long been the custom at Oberlin to devote the last three months or more of the Freshman year to a simple but reasonably complete system of Qualitative Analysis. The reactions of the metals are included in this part of the work. In using such a plan it is necessary to omit a considerable number of experiments given in this manual.

No apologies need be offered for the introduction of a chapter on Colloid Chemistry. The rapidly growing interest in this subject justifies the use of such material.

HARRY N. HOLMES.

OBERLIN, September, 1921.

DIRECTIONS FOR THE STUDENT.

1. At the close of each laboratory period leave your glassware and desk thoroughly clean.

2. Throw all solids in the crocks. Liquids are emptied in the sinks, and if acid, must be accompanied by a rapid flow of water. Concentrated acids should be emptied in the crocks.

3. Study the experiments **before** coming to the laboratory, and state clearly and briefly in the notebook the purpose of each experiment. Laboratory work is of little value unless you understand what you are trying to do before you do it.

4. Write up your experiments at once. A day later you will have forgotten what you have observed. Be brief and to the point. As soon as possible describe what happened by chemical equations, even if they are not perfect. To learn correct formulas refer to the index of your text. Reference books are at hand in the building. Ask your questions of them rather than of your fellow students. State your observations and conclusions.

5. You will double your speed by knowing just what you expect to do before entering the laboratory. Make a list of the extra things you need from the supply room and get them all at once. Carry on more than one experiment at a time whenever possible. Work alone unless otherwise directed.

6. Use as little of each reagent as will illustrate the point. In test tubes experiments two or three cc. are usually sufficient.

7. Remember that graduates and bottles are not to be heated; test tubes and beakers may be heated over the free flame as long as they contain liquid; evaporating dishes may be heated rather hot even after all liquid has boiled away and crucibles may be ignited red hot. It is always safer not to heat too suddenly at first.

8. Carry a text to the laboratory and refer to it constantly. In writing up the experiments give the pages in the text to which the experiments refer. Give attention to the title of the experiment.

9. Start the longer experiments first each day and at the first opportunity begin the shorter ones. It is often possible to do two things at once.

10. For "Quantitative" experiments use the finer balances. Rough balances will do for the rest of the work.

11. Do not carry side shelf bottles to your desk. Use clean test tubes for carrying liquids and small squares of glazed paper (provided at the side shelf) for carrying solids. Take less than you need, rather than more. Never return unused chemicals to the side shelf bottles. You may make a mistake. We must have perfect confidence in the labels. Read the label twice before taking anything from a bottle.

12. Never lend or borrow wash bottles. Keep distilled water in your wash bottle for reactions and for rinsing.

13. When heating liquids in test tubes do not aim at your neighbor. A sudden formation of steam may throw the contents some feet away.

14. Carefully neutralize any acid or base spilled on the desk and wash off with plenty of water. Otherwise coat sleeves suffer. Acids on clothing must be neutralized with ammonia. Any corrosive liquid on the skin should be washed off **instantly** with plenty of water. Then see an instructor.

15. In pushing a bent glass tube through a stopper it is safer to hold the tube in the folds of a towel.

16. A cut developing even a drop of blood should be shown to an instructor at once for antiseptic treatment. The danger of infection is too great to take any chances.

17. Any instructor will treat burns also.

18. Throw a towel over small fires.

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CHAPTER I.

PRELIMINARY EXERCISES.

Glass Working. — To cut glass tubing, make a deep scratch across it with a single forward motion of a triangular file. Now grasp the tube firmly in both hands, thumbs almost touching, but on the side opposite the scratch, and bend, using the thumbs as fulcrums. Smooth the sharp cut edges with a file or by holding in the flame until the edges soften.

To bend glass tubing use the broad, flat, luminous flame of an illuminating burner or a "wing tip." Hold the tube lengthwise in the flame, so that about two inches will get the full heat. Rotate the tube slowly and constantly in one direction to secure uniform heating, but do not bend it or allow it to bend, while in the flame. When the glass has become rather soft remove it and bend it to the desired angle. It is poor work to let the tube flatten on the outside or wrinkle on the inside of the bend.

To draw out tips for a wash bottle the tube must be heated until the walls are thicker than normal at the heated spot, rotating the tube slowly and heating a shorter length than for glass bending. When it is soft remove from the flame, and, with a firm, steady hand, draw it out to the extent desired. Let it cool and cut to the proper length.

Cork Boring. — Select a borer slightly smaller than the hole desired. Warm the borer very slightly and, using the inner rod as a "T" handle, bore a hole from each end of the cork so that the holes meet in the middle. Hold the cork in the hand, rotating in one direction and exerting only a slight pressure.

Do not bore against the desk. When through punch out the plug from the borer.

A glass tube is forced through corks or rubber stoppers by grasping near the entering end, pushing in gradually with a twisting motion, the hand being held against the stopper by shifting the grip as often as necessary. It is well to moisten the end of the tube.

Filter Paper. — An instructor will show you how to fold and use filter paper.

The Wash Bottle. — A 500 cc. flask should be fitted with a two-holed rubber stopper (or good cork) and tubing as shown in Fig. 1. If the directions given above are followed carefully the finished apparatus will be accepted. The short tube (mouth piece) need not project below the under surface of the stopper. The long tube should reach nearly to the bottom of the flask. The tip or nozzle is connected to the long tube by a piece of rubber tubing not more than an inch in length. You should be able to hold the flask in one hand and direct the nozzle as desired without using the other hand.

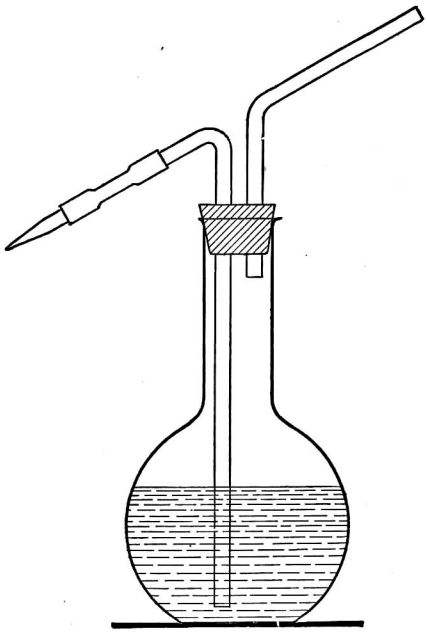

FIG. 1

Only distilled water is used in the wash bottle. On blowing in the mouthpiece a fine stream is forced out the nozzle. This is suitable for washing precipitates but when more than about 10 cc. are desired it is customary to pour water directly from the mouthpiece.

The Bunsen Burner. — In some burners the gas supply may be regulated by a screw at the base. Air is let in by turning a perforated ring in the proper direction. Experiment with your

burner, cutting off the air supply so that the flame is yellow and smoky, then adjusting the air intake so as to secure a quiet blue flame (the hottest flame). With too much air the flame sometimes "strikes back" and burns inside at the base of the burner. Turn out the gas and begin again. Quickly thrust a match in a blue flame just above the top of the burner. Remove it in a second or two and decide from the charring which part of the flame is the hotter. Do this again at various heights in the flame. For effective heating would you hold a vessel in the lower half or the upper half of a flame?

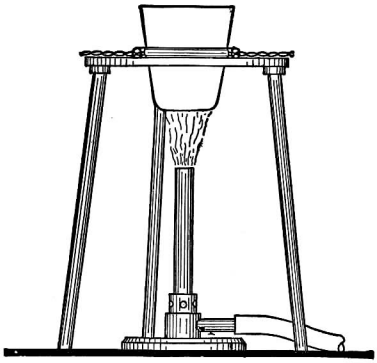

FIG. 2

Use of the Balance. — The analytical balance is a delicate instrument which is accurate only when carefully treated. The following rules indicate the important points to be observed, but they must be supplemented by a sense of personal responsibility on the part of each student:—

1. See that the balance is level as indicated by the plumb line at the side of the column.

2. The balance case should be closed during the final readings of any weighing.

3. Weights are always placed on the right pan of the balance.

4. The balance pans must rest upon their supports when not in use and every time objects or weights are added or removed.

5. Be absolutely certain that any piece of apparatus to be placed on the balance pan is clean, dry and cold.

6. No chemical is ever to be placed directly upon the balance pan. Use a suitable glass or porcelain container.

7. All movements in using a balance should be so careful and deliberate that nothing more than a barely audible click of the parts is heard.

8. If there is any apparent irregularity in the working of the balance, call the instructor to adjust it. Any damage will be charged to the last student known to have used a balance or to the entire group assigned to it. Report any accident immedi-

ately that everything may be done to prevent permanent injury to the instrument.

9. Count weights carefully and check by counting the empty places in the box in taking a reading and record results in the laboratory manual, not on a loose piece of paper that may be lost, as this would necessitate a repetition of the entire experiment.

10. All weights are to be handled with the forceps in the weight box, never with the fingers. Be very careful to avoid scratching or bending any of the weights.

11. Use only the balance to which you have been assigned. Your name should be on the card in the case.

12. After completing a weighing return all weights to their proper places in the weight box, and see that the balance case and shelf are clean and the case closed.

13. No fuming or hot chemical is ever to be taken to the balance room.

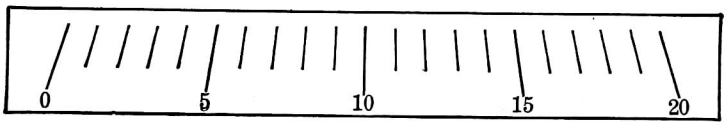

FIG. 3

The Zero Point.—Examination of the balance shows that the long vertical pointer is attached to the horizontal beam like the stem of the capital letter T. When the supports of the beam are lowered and the beam is free to "seesaw" the pointer swings from side to side. Due to friction these swings steadily decrease in extent and ultimately the pointer comes to rest at what is termed the "zero point" or the middle of its swings. The balance may be adjusted so that this zero point is "position 10" at the middle of the ivory scale (Fig. 3). Usually it is anywhere from 9 to 11. The zero point may be calculated from any five **consecutive** swings without waiting for the pointer to come to rest.

It is good preliminary practice to set the pointer swinging and call the positions on the scale reached by the tip of the pointer, left and right, estimating instantly to the tenth of a division.

To Find the Zero Point.—Consider the divisions on the scale (at the foot of the pillar) to be numbered from left to right, the middle scratch being number 10. Release the arrestment gently enough to cause the pointer to swing over not more than about half a dozen divisions. If it does not swing this far release it less gently and so on until the proper swing is secured. Observe, with the open eye placed directly in front of the pillar (error of parallax?), the exact position on the scale of each end of the swing, estimating this to tenths of a division (practice!). Record these two readings in your note book. Arrest the balance as the pointer is passing the middle of the scale. Find by arithmetic the mid-point (A) of the swing. This point is called the zero point of the balance, and must be re-determined at each weighing. Swings are never read when the balance case is open, as a current of air might introduce a large error. One extra swing is recorded on the side given the first swing. To illustrate:

	5.3	14.0
	5.6	13.6
	6.0	
Average	5.6	13.8

$$\text{Zero point} = \frac{5.6 + 13.8}{2} = 9.7$$

To Find the Sensibility of the Balance When It Is Carrying a 10 Gram Load.—Place, with forceps, a ten gram weight on each pan. Close the balance case and, not till then, gently release the arrestment. Find and record the mid-point (B) of the swing as before. If balance and weights were perfect, this (B) would be the same as the zero point (A). First making sure that you have arrested the balance, again open the case and place a ten milligram weight on the right pan beside the 10 gram weight. Close the case, release the arrestment, observe the swing, and find and record the mid-point (C) as before. Find by arithmetic the distance, in scale divisions, between (B) and (C). This is the displacement on the scale caused by an overload of one centigram (10 milligrams) on the pan, and is called the sensibility of the balance (at load 10 grams). It is used in order to estimate weights less than 0.01 grams. The sensibility of a well-treated balance remains the same for years, and need be determined only occasionally. Record thus,—"10 milligrams

(at load 10 grams) displaces zero point 2.4 scale divisions," for example.

Method of Weighing. — Determine the zero point of the empty pans. Place the object to be weighed on the left hand pan and the weights on the right. Begin with the heaviest weight that seems proper, gently release the arrestment and observe the swings. If the weight is excessive substitute the next smaller. If the weight is too small add the next smaller and so on until the zero point of the loaded balance nearly coincides with that of the empty pans.

The weight required to make the zero point of the loaded balance exactly coincide with the zero point of the empty pans is then calculated by means of the sensibility already found. Suppose the sensibility is, for example, 2 divisions of the scale for 10 milligrams and the zero point of the empty pans is 10.1 while that of the loaded pans is 10.7.

Evidently the zero point must be displaced to the left 0.6 divisions (10.7—10.1) before the weights on the two pans are equal and an exact weighing made. Since an extra weight of 10 milligrams (on the right pan) displaces the zero point 2 divisions (sensibility in this illustration), to displace it 1 division only 5 milligrams ($10 \div 2 = 5$) would be required. Therefore to displace the zero point of the loaded balance 0.6 divisions to the left requires $5 \times 0.6 = 3$ milligrams added to the weights already used.

If the displacement should be to the right the weight must be subtracted. This final correction is made with the lead pencil, not with actual weights.

EXERCISE.—Weigh accurately a section of a metal rod or a new penny secured from the stock room. Report the weight to an instructor.

CHAPTER II.

PHYSICAL AND CHEMICAL CHANGES.

1. Chemical or Physical Change. — (*a*). Examine a short piece of magnesium ribbon. With a pair of forceps hold it in the flame until a decided change is noticed. Collect the residue and compare it with the original magnesium. Has the change been chemical or physical?

(*b*). Try the same experiment with platinum wire or an iron nail or with a splinter of wood.

(*c*). Heat about 2 g. of sugar in a test tube until there is **no further change.** (Hood.) Note the steam condensing on the colder part of the tube. Will the residue in the tube dissolve in water to form a sweet, colorless solution? Has the change been temporary or permanent, chemical or physical?

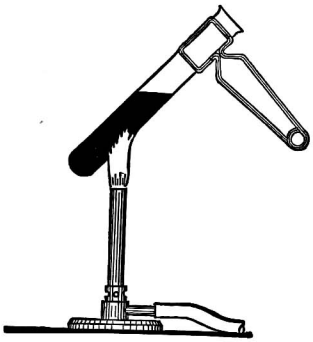

FIG. 4

(*d*). Perform this experiment in the hood. The red fumes are very poisonous. Treat a few copper filings in a test tube with a few cc. nitric acid. Warm gently. The copper disappears and a blue solution forms. Evaporate to dryness, raising the evaporating dish high enough above a very small flame to approximate the temperature of a steam bath. When the flame is properly adjusted the liquid will not boil but merely steam rapidly. Thus there is no loss of dissolved substance by spattering nor does the temperature rise much above 100°. Does the residue in its color, hardness, solubility, etc., resemble the original copper? Do not taste it—poisonous.

(*e*). Grind in a mortar some sand and salt. Heat in a test tube. Separate the salt from the sand. Did you make a compound?

2. Chemical Compound or Mechanical Mixture.— (*a*). Examine a few drops of pure carbon disulfide, a chemical compound. (Do not bring near a flame.) It was made by heating together carbon and sulfur without access of air.

Grind thoroughly in a mortar about one gram of iodine and two grams of mercury. Pour off any excess of mercury (into a crock, **never** into the sink). Does the product in any way resemble mercury or iodine? Is this product a chemical compound or mechanical mixture?

(*b*). Gunpowder is prepared from charcoal, saltpeter and sulfur. Gently heat a gram of it in about 8 cc. of distilled water for one minute. Filter (Fig. 5), collecting the filtrate (the liquid running through the paper). Boil the filtrate to dryness without spattering and compare the residue with sulfur, charcoal and saltpeter. Which is it? Dry the black residue on the filter paper on a steam pipe or in the oven. Pour a few cc. of carbon disulfide through the paper collecting on a watch glass. Let this inflammable filtrate evaporate away from any flames and note the deposit. Does it resemble sulfur, charcoal or saltpeter? Which does the final black residue on the paper resemble? Is gunpowder a chemical compound?

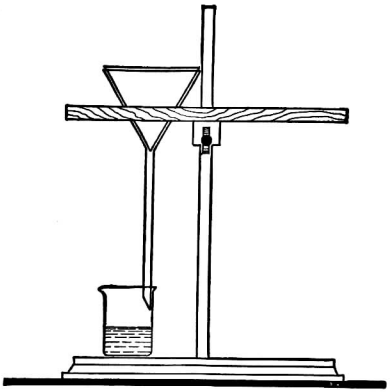

FIG. 5

3. Aids to Chemical Action. — (*a*). Mix about a gram of dry sodium bicarbonate (baking soda) with a gram of dry tartaric acid. Any action? Repeat, first dissolving each in a little water. Note the difference.

(*b*). To a solution of common salt (sodium chloride) in two tubes add a few drops of silver nitrate solution. A white precipitate forms and slowly settles. This is silver chloride. Leave one tube on the desk an hour or more and **quickly** set the other in the darkest corner of the locker. Later compare the two.