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JAMES F. NORRIS, PH.D., CONSULTING EDITOR

LABORATORY EXERCISES IN INORGANIC CHEMISTRY

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LABORATORY EXERCISES
IN
INORGANIC CHEMISTRY

BY

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PREFACE

THIS laboratory manual contains, in addition to the experiments ordinarily found in books of this class, a large number of new experiments that have been designed to illustrate the general principles which are being more and more emphasized in elementary instruction in chemistry. About one-fourth of the book is devoted to the study of gases, liquids, and solids, solutions, thermo- and electro-chemistry, and chemical equilibrium. Directions for a number of inorganic preparations are also given. In designing these experiments the authors have had in mind the student who has had a good training in chemistry in the high school, and who should have his interest stimulated in his college course and should be given the opportunity to extend his experience. Many of the experiments are quantitative; but care has been taken to have the manipulations and apparatus of such a simple character that the exercises can be performed successfully by the average student in a laboratory for beginners.

The experiments to illustrate the properties of metals and their compounds are followed by an outline of a general method for testing a simple salt for the metal and acid radical present. The short time available in the first year for qualitative analysis makes it impossible for the student to master the methods used in the separation of the metallic elements. He can, however, by the simple procedure outlined learn to identify pure salts. The method described has been found to be an excellent introduction to qualitative analysis.

Each experiment is preceded by a "discussion" which emphasizes the principles to be illustrated. The questions incorporated into the directions for the experiments have been selected in such a way that it is necessary for the student to read the discussion in order to answer them. As a result, it is impossible for him to complete the record of his work without understanding what is to be learned from the experiment.

Questions in connection with the laboratory directions serve

another purpose. It is the opinion of the authors that the student can not be left to himself in his first experimental work; he must be guided if he is to learn to observe closely and to differentiate the essential from the non-essential. A question in the right place furnishes this guidance and avoids, in most cases, the repetition of the experiment.

The notes of the student consist, in the main, of answers to these numbered questions. The plan makes it possible for the instructor to see at a glance whether the student has understood his work.

The authors are greatly indebted to Raymond E. Neal, Instructor in Chemistry, Simmons College, for valuable suggestions and for the preparation of the drawings, from which the cuts were made.

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BOSTON, MASS.
April, 1922.

CONTENTS

LABORATORY EXERCISES IN INORGANIC CHEMISTRY

GENERAL DIRECTIONS

The Object of Laboratory Work: Laboratory work is a form of study. A thorough knowledge of elementary chemistry can not be acquired from books alone. Actual first-hand observation of the properties of substances must be made by the student himself, but even these observations will be of small value unless he appreciates the object of the experiment and the principle which it illustrates. Merely "doing" the experiments, that is, mechanically following directions, is as valueless as reading a book without comprehending its meaning. No experiment should be begun until the discussion is thoroughly understood.

Notes: Observations must be recorded immediately. To facilitate clearness and brevity they are to be entered on the blank pages facing the text, as answers to the numbered questions.

The data obtained in quantitative experiments should be carefully labeled and the result of each measurement should be placed on a separate line, as illustrated by the following example in the determination of the percentage of oxygen in potassium chlorate, as carried out in Experiment 11.

Wt. crucible empty	20.30 gms.
Wt. crucible and $KClO_3$	22.03
Wt. $KClO_3$	1.73
Wt. crucible and $KClO_3$ after first heating	21.42

Wt. crucible and $KClO_3$ after second heating.....	21.35
Wt. crucible and $KClO_3$ after third heating.....	21.35
Loss of weight = weight of oxygen.....	0.68
Percentage of oxygen in $KClO_3$.....	39.3

The data must be entered at once directly in the book, never on loose pieces of paper. All calculations should be made in the book.

Individual Work: All experiments should be done entirely independently. A student can get no more out of his laboratory exercises than he puts into them. If he allows another student to perform the experiments for him or copies his notes, he is deriving no benefit from his work, he is wasting his time and, moreover, he is dishonest.

Use of the Text Book: The numbers given in the references are those of the paragraphs in "A Text Book of Inorganic Chemistry," by James F. Norris. The text book should be brought to the laboratory at each exercise, the paragraphs pertaining to the exercise of the day should be read, and reference to the book should be made whenever the student is in doubt.

Repetition of Experiments: If the results obtained are not those which you have been led to expect, search for the cause, making sure particularly that you have followed the directions precisely. Not until a possible cause of error has been detected should the experiment be repeated.

Cleanliness: Apparatus must be clean in order to obtain trustworthy results. Always have a clean towel at hand.

The desk top should be frequently wiped with a wet sponge and any material which is spilled upon it should be washed off immediately with plenty of water.

General apparatus must be cleaned and returned to its place at the close of each exercise.

Materials: All the materials required for an experiment and listed in the text should be obtained before beginning work. It is assumed that each desk is supplied with solutions of dilute hydrochloric, sulphuric, and nitric acids, of sodium and ammonium hydroxides, and with concentrated sulphuric

acid. These substances therefore are not included in the lists. It is also assumed that each student is provided with the set of apparatus given in the appendix and only additional apparatus is included in the list of materials.

Only such amounts of substances as are actually required should be obtained. Stock bottles should never be carried to the student's desk. Solids may be measured out on a watch glass or on a piece of filter paper and liquids may be carried in a test tube or beaker.

Always read the label on a stock bottle before taking material from it, and verify your reading by rereading after you have taken the substance.

Unused material must never be returned to the stock bottle but thrown away into the crock or sink.

Waste Material: Solids, particularly matches, should be disposed of in the crock, not in the sink. Liquids should be poured into the sink, and in the case of concentrated acids, the sink should be flushed immediately with a large amount of water.

Accidents: Any corrosive liquid on the skin should be washed off *immediately* with plenty of water.

Burns and cuts, even when slight, should be reported to an instructor for treatment.

Acid spilled upon the clothing should be neutralized with ammonia.

Fires should be extinguished by throwing a wet towel over the blaze.

LABORATORY PROCESSES

1. Heating: The Bunsen burner is lighted by first turning on the gas and then applying a burning match. This order of procedure allows time for the air in the tube and burner to be expelled while the match is being "struck." The burner should always be lighted before it is placed under the apparatus to be heated, never after it has been put in position.

The character of the flame is regulated by adjusting the quantity of air which enters through the holes at the bottom

of the burner. Ordinarily a clear bluish flame should be used. Sometimes the flame "strikes back," that is, begins to burn at the base where the air enters. The occurrence is usually due to the admission of too much air. Turn off the gas, and after the burner has cooled, diminish the amount of air by adjusting the movable ring and relight the burner.

Thick vessels, like a bottle or a mortar, should never be heated because the uneven expansion of the material causes them to break.

Porcelain evaporating dishes and crucibles can be heated directly in the flame, but beakers should rest on a piece of wire gauze.

Test tubes should be held in the test tube holder. When a liquid is being heated, the test tube should be inclined and held in such a position that the flame strikes the glass opposite the upper part of the liquid. The tube should be slightly shaken constantly. If the tube is held still and the bottom heated, a large amount of steam may be formed suddenly and throw the contents out of the tube.

All vessels must be dry on the outside when heated; otherwise they may crack.

2. Filtering: Fold a disc of filter paper just in halves and then again in quarters. Open one of the segments, leaving three thicknesses of paper on one side and one on the other. A paper cone will be formed which should be placed in a glass funnel. If water solutions are to be filtered, wet the paper and press it firmly to the glass with your finger. When a filtration is to be made into a beaker or an evaporating dish, the funnel should be supported by a ring or by a filter arm and the apparatus adjusted so that the stem of the funnel touches the side of the vessel which is to catch the liquid which comes through; otherwise the liquid will spatter out as it falls.

3. Drying Test Tubes: Slip the washed test tube over the end of a glass tube about 1 foot in length and, inclining it mouth downward, wave it back and forth through the flame of a burner. When the test tube is hot blow through the glass tube for an instant. Repeat the procedure if necessary.

4. Preparing Glass Tubing: (*a*) *Cutting:* To cut a piece of glass tubing, first make a slight scratch on the tube with the edge of a triangular file. Then, holding the tube in both hands, place the thumbs together against the tube on the side opposite the scratch. Press the tube as though bending it away from the scratch. A clean break should result. The broken edge of the tube will be very sharp and must *always* be smoothed by holding the end of the tube in the flame until it becomes a dull red color. This process, which is called "fire polishing," causes the sharp edges of the glass to melt and thereby become smooth.

(*b*) *Bending:* A good bend is a smooth curve rather than a sharp angle. Several inches of the tube, therefore, must be involved in the bend, and it becomes necessary to soften the glass by heating it in a wide flame. Put a "flame spreader" on the top of the Bunsen burner and close the holes at the bottom of the burner so as to give a luminous flame. Hold the tube in the yellow part of the flame, letting one end rest lightly between the fingers and thumb of the left hand and turning the tube slowly, but constantly, in one direction with the fingers and thumb of the right hand. When the tube feels pliable, remove it from the flame and make a bend of the desired angle.

The following bent tubes should be prepared as they will be used frequently in these experiments: two right-angled bends each leg of which is 10 cm. long; one right-angled bend one leg of which is 10 cm., the other 20 cm. long; one 45° bend one leg of which is 10 cm., the other 15 cm. long; and one 135° bend one leg of which is 10 cm., the other 15 cm. long.

5. Preparing Corks: Before being used corks should be softened. This can be done by means of a press, which is made for this purpose, or the cork can be rolled on the desk while it is being pressed firmly by means of a block of wood. Sharp cork borers should be used to make the holes of such a size that the tubes to pass through fit snugly. In boring corks it is advisable to push the borer with a rotary motion half way through the cork, taking care that the hole is bored through the center of the cork; the borer is then removed and a hole made from the center of the other end of the cork to meet that first made. By proceeding in this way the edges of the holes

on the two sides of the cork will be clean cut, and thus make a tight joint with the tube to be passed through the hole; and the latter will run evenly through the axis of the cork.

6. Setting Up Apparatus: Place the front of the ring stand parallel to the edge of the desk and so that the rod is away from you, not toward you. Arrange the clamps so the main weight of the apparatus is over the base of the stand. The movable jaw of the clamp should be on top. Be careful not to screw the clamp so tight as to crush thin apparatus. Keep vertical lines vertical and horizontal lines horizontal.

In putting a glass tube through a rubber stopper, wet the outside of the tube and then push it in with a twisting motion. Give three distinct pushes, pause long enough to count three and then give three more pushes. If you do not pause, you involuntarily keep pushing harder and harder, often with the result that the tube breaks and the jagged end cuts the hand. Be sure that all stoppers fit tightly and that there are no leaks around the holes of stoppers through which glass tubes pass.

7. Weighing: Objects and material to be weighed fall into two general classes, those in which only an approximate weight, to the nearest gram, is required and those in which an accurate weight, to the nearest centigram, is demanded. Cases in which a convenient amount of material for experimentation is to be weighed out belong to the first class; the quantitative determination of the composition of substances belongs to the second class. Platform scales are used in the first instance; beam balances are necessary in the second.

(*a*) The platform scales must always be tested before using them because they are exposed to the fumes of the laboratory, and easily become rusted. Be sure that the rider on the front of the scales is pushed to the extreme left, and then gently tap one of the platforms. The pointer should swing an equal number of divisions on each side of the middle of the pointer scale and should not come to rest until at least two swings on each side have been completed. If the scales are found to be out of adjustment, an assistant should be called to fix them.

In weighing out material for an experiment the empty con-