EXERCISES IN CHEMISTRY

McPHERSON AND HENDERSON

REVISED EDITION

EXERCISES IN CHEMISTRY

SYSTEMATICALLY ARRANGED TO ACCOMPANY THE SECOND REVISED EDITION OF "AN ELEMENTARY STUDY OF CHEMISTRY"

BY

WILLIAM McPHERSON
"
AND

WILLIAM EDWARDS HENDERSON

PROFESSORS OF CHEMISTRY, OHIO STATE UNIVERSITY

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PREFACE

The exercises contained in this book are systematically arranged to accompany the revised edition of McPherson and Henderson's "An Elementary Study of Chemistry." It has been thought desirable to include in the list a somewhat larger number of exercises than will ordinarily be performed, thus giving the instructor some choice in his selections. A number of simple exercises bearing on subjects of special interest to students of home economics are included under Appendix A. These have to do with simple tests for food constituents, detection of different kinds of textile fibers, and similar topics. If time is not available for the students interested in such subjects to perform these experiments in addition to the regular ones, the instructor may find it possible under the circumstances to make certain substitutions that will appeal to his judgment.

While the experiments selected cover a wide field and are believed to illustrate the general topics included in the presentation of an introductory course in chemistry, nevertheless they may be performed with simple apparatus and with chemicals that are readily available. Detailed information in regard to the apparatus and materials required for performing the experiments will be found in Appendix B.

It is no longer necessary to emphasize the importance of laboratory work as a part of the course in elementary chemistry, since it is universally admitted that laboratory experience is essential for a thorough comprehension of the subject. It is none the less true, however, that laboratory work is of very doubtful value unless carefully directed by an experienced teacher toward some definite end. It is in the hope of aiding such a teacher that this exercise book has been prepared.

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APPENDIX A

APPENDIX B

DIRECTIONS

A list of the apparatus and chemicals necessary for performing the experiments, together with suggestions as to their purchase, is given in Appendix B. It is expected that each desk will be provided with five of the most common reagents (see Appendix, p. 141). When reference is made to any of these five reagents under "materials" required in performing each exercise, it will be understood that the reagents on the desk are the ones to be used. Other reagents used less frequently should be placed on a reagent shelf available to all. The abbreviation (R. S.) used in listing the materials necessary for performing each individual experiment refers to the reagents on the reagent shelf. The abbreviation (R) implies that the equation for the reaction involved in the experiment is to be written. The character (?) implies that the results obtained in that part of the experiment are to be recorded in the notes. The word "hood" signifies that the experiment is to be performed in a well-ventilated compartment so that any poisonous gases evolved will be removed at once. All thermometer readings refer to the centigrade scale.

Exercises or parts of exercises marked with a star (*) may be omitted if time is not available for performing all the experiments.

EXERCISES IN CHEMISTRY

EXERCISE 1

THE METRIC SYSTEM

(See Appendix B.)

Apparatus. Graduated test tube; balance sensitive to 1 cg. and set of weights from 50 g. to 1 cg.; watch glass; 100 cc. beaker.

Materials. Teaspoonful of common salt; test tube full of distilled water; nickel five-cent piece.

a. *Length.* By means of the scale (see Appendix), 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? Finally, 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 off the amount of the liquid in a graduated tube, always read from the lower part of the meniscus; that is, the curved surface of the liquid.)

c. *Weight.* Ask the assistant for instruction in regard to the use of the balance; then weigh various small objects, as a porcelain crucible, a watch glass. What is the approximate weight of a nickel five-cent piece?

Accurately balance a watch glass on the scalepan (using either the weights or a small pill box and fine shot) and weigh out on this exactly 5.2 g. of common salt.

Balance a small (clean and dry) beaker on the scalepan; then remove it from the pan and pour into it, as nearly as possible, 10 cc. of distilled water (measured in the graduated tube included in your laboratory outfit). Reweigh, and note the weight of the water. How do you account for the fact that it does not weigh exactly 10 g.?

EXERCISE 2

THE BUNSEN BURNER; MANIPULATION OF GLASS TUBING

Apparatus. Bunsen burner and wing-top attachment, as shown in Fig. 4; hard-glass test tube with good cork to fit; set of cork-borers; triangular file; round file.

Materials. Wooden splint (1 cm. × 12 cm. is a convenient size); white cardboard (10 cm. square); soft glass tubing 15 cm. in length; glass rod, 25 cm. in length.

a. *The Bunsen burner.* The Bunsen burner is a form of apparatus used for producing heat and is commonly employed in the laboratory. It consists of the tube *A* (Fig. 1), screwed into the base *C*. The tube has two small openings near its lower part. A small band *B*, provided with similar openings, fits around the lower part of the tube in such a way that the openings of the tube may be closed or kept open by turning the band. Gas is admitted through *D* by means of rubber tubing.

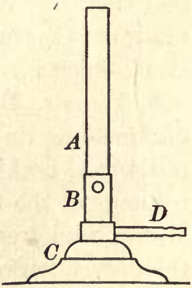


FIG. 1

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 supply should be adjusted so as to give a flame about 10 cm. high. The gas flowing through the tube mixes with the air drawn in through the openings 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 directed otherwise.

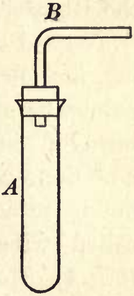


FIG. 2

Hold a small wooden splint horizontally in the base of the Bunsen flame for two or three seconds and note the results. In the same way determine the relative temperatures of various parts of the flame. Turn the gas down until the flame is 7 or 8 cm. in height, then quickly thrust a piece of white cardboard, about 10 cm. square, *vertically* through the center of the flame, the lower end of the cardboard resting against the top of the burner. Remove the cardboard before it is ignited and from the scorched portions note the relative temperatures of different parts of the flame. Draw a diagram to illustrate your results.

b. *To fit a tube with stopper and glass tube as shown in Fig. 2.* In all operations requiring the application of a strong heat to glass, the heat must be applied gently at first. Highly heated glass must be cooled slowly; otherwise it is easily broken.

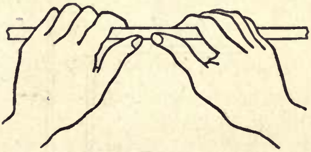


FIG. 3

From one of the lengths of soft-glass tubing cut a piece about 15 cm. in length. To do this, 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. After the glass has been scratched, take the tube in the hands with the thumbs placed near together just back of the scratch (Fig. 3), and gently pull the glass apart, at the same time exerting a slight pressure with the thumbs. If the tube does not yield readily to a gentle pressure, a deeper scratch must be made. In the case of large tubing it may be found necessary to file a groove entirely around the glass. The edges of the cut tube will be sharp, and should be rounded by being rotated in the tip of the Bunsen flame.

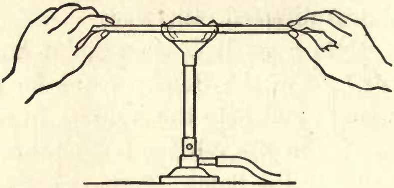


FIG. 4

To bend the glass tubing, first heat it at the point where you wish to bend it, in the luminous Bunsen flame spread out by means of the so-called "wing-top" burner (Fig. 4). Hold the tube lengthwise in the flame, gently rotating it so that all sides may be equally heated. Continue the heating until the glass bends easily, then remove it from the flame and quickly bend it to a right angle *B* (Fig. 2). Great care must be taken to heat the tube uniformly, otherwise the bore of the tube will be contracted (*A*, *B*, Fig. 5), forming a bend which is not only unsightly but is easily broken.

Next select a cork of such a size that the smaller end will just enter the hard-glass test tube *A* (Fig. 2). Soften the cork by rolling it between the desk and a block of wood. Now insert into the cork the glass tube prepared as directed above. To do this, select a borer slightly smaller than the tube. Place the cork on the desk and cut half through it with the borer, not by punching but by rotating the borer

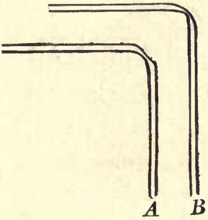


FIG. 5

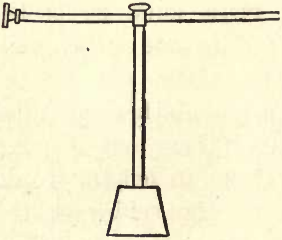


FIG. 6

under gentle pressure (Fig. 6); then reverse the cork and bore through from the other end. Care must be taken to keep the borer at a right angle to the top and base of the cork. The hole should be straight and smooth.

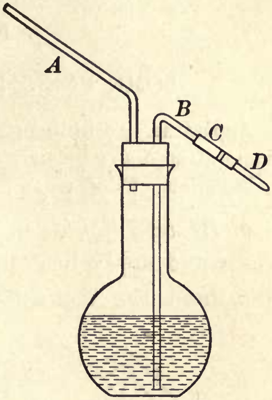


FIG. 7

The glass tube, rounded at the edges, is now inserted in the cork 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. Now put the cork in the test tube and set the apparatus aside for use in preparing oxygen (Exercise 7).

c. To make glass stirring-rods. Make two of them, one about 10 cm. and one about 15 cm. in length. Cut the rods to the proper length and round the ends by heating in the Bunsen flame. Place the finished rods in the desk for future use.

**d. To make a wash bottle.* The student often will find a wash bottle useful in laboratory work. It may be readily made as represented in Fig. 7. A 500 cc. flask is used. *A* and *B* represent soft glass tubes bent as shown in the figure. *B* is connected with the glass jet *D* by a piece of rubber tubing *C*. The glass jet *D* is made as follows: Heat a piece of soft glass tubing 12 or 15 cm. in length in the Bunsen flame until the walls of the heated portion thicken and the size of the bore diminishes (Fig. 8, *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

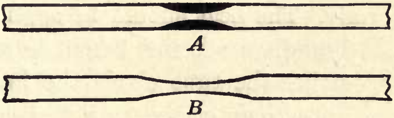


FIG. 8

position, gently pull the ends apart until the bore is of the desired size (Fig. 8, *B*). The glass jet is then formed by cutting the tube at *B* and rounding the edges in a flame.

EXERCISE 3

SOME PRELIMINARY MANIPULATIONS

Apparatus. Test tubes; glass rod; 2 beakers; funnel; ring stand; evaporating dish; burner.

Materials. Filter paper; piece of chalk; 2 or 3 g. of common salt.

a. *Heating liquids in test tubes.* Half fill a test tube with water and apply heat until the water boils rapidly. To do this, hold the test tube between the thumb and fingers (Fig. 9), constantly rotating it 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 the tube becomes heated, it may be supported by a test-tube holder (Fig. 10) or by a band of paper wrapped about the upper part of the tube (Fig. 11). The sudden formation of vapor sometimes causes the contents of the tube to be thrown out; hence care must be taken not to point the tube toward anyone.

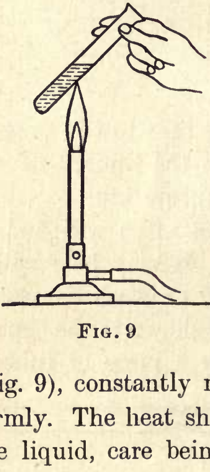


FIG. 9

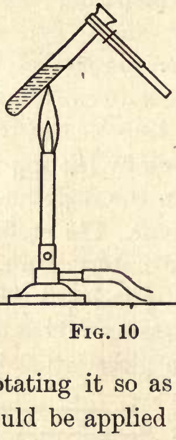


FIG. 10

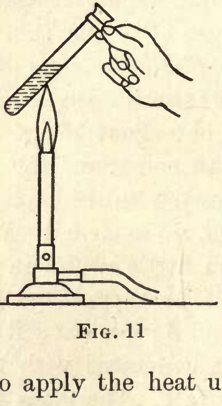


FIG. 11

b. Pouring liquids from one vessel to another. In doing this 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. 14. 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.

In pouring liquids from bottles 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. 12

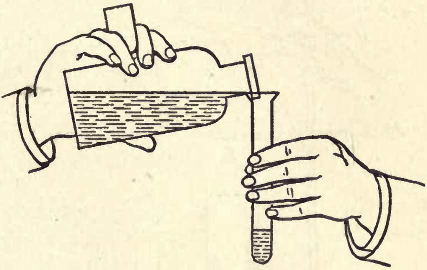


FIG. 13

(Fig. 13). This will prevent the liquid from running down the side of the bottle. The stopper must never be laid down on the desk (?). Catch it between the fingers, as shown in Fig. 12. This leaves the hand free to grasp the bottle, as shown in Fig. 13.

c. Decantation; filtration; solution; evaporation. It is often necessary to separate a liquid from a finely divided solid which is suspended in it. This may be done by one of the two following methods.

1. *Decantation.* When the solid is heavy and readily settles to the bottom of the vessel the liquid may be carefully poured off or *decanted.* Thus sand suspended in water may be separated from the liquid in this way.

2. *Filtration.* Usually, however, 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. Now 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. The filter is now ready for use (Fig. 14). The process of filtration not only enables us to separate liquids from solids but also certain solids from each other. To illustrate this, grind a piece of chalk to a powder in a mortar, and mix the product intimately with about an equal bulk of common salt. Place the mixture in a small beaker, add about 50 cc. of distilled water, and stir with a glass rod. (The ends of the

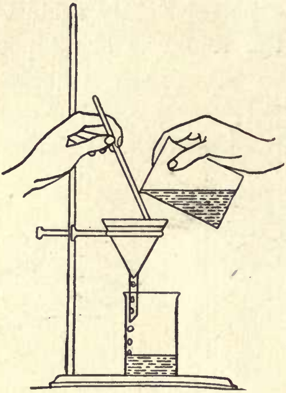


Fig. 14

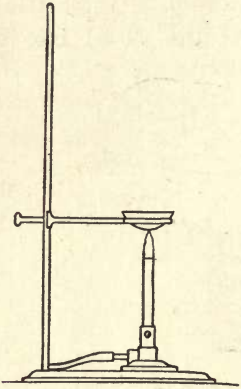


Fig. 15

rod should 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,* that is, the clear liquid which passes through the filter paper, in a beaker (Fig. 14). The salt may be recovered from the filtrate by the process of *evaporation.* To perform this operation, pour the filtrate into an evaporating dish, support the dish on a ring stand (Fig. 15), and heat gently. The liquid may be made to simmer, but should not actually boil (?). Withdraw the heat as soon as the water is evaporated. Note the residue left. Convince yourself that it is common salt.

EXERCISE 4

A STUDY OF SOME OF THE CHANGES TAKING PLACE WHEN A SUBSTANCE BURNS

Apparatus. Porcelain crucible; burner; ring stand; pipe-stem triangle to support the crucible while being heated; pneumatic trough (or dish); wide-mouthed bottle or large beaker; candle.

Materials. 2 or 3 g. powdered iron; 2 or 3 g. granulated tin; pellet of phosphorus, size of a small pea (to be obtained from the instructor when needed); 5 cc. limewater (R.S.).

a. *The burning of metals.* Place 2 or 3 g. of powdered iron in a procelain crucible and accurately weigh. Now heat the crucible (Fig. 16) until the iron begins to glow (burn); then withdraw the heat. Does the iron continue to burn? After the crucible is cool, reweigh. Compare the weight of the product with that of the unburned iron (?). The experiment may be repeated, using tin.

b. *The burning of phosphorus.* (PRECAUTION. *Phosphorus must be kept and handled only under water; otherwise it may ignite, and serious results follow.*) Cover the bottom of a pneumatic trough with water to a depth of 2 or 3 cm. On the water float a porcelain crucible containing a piece of phosphorus the size of a small pea.

Ignite the phosphorus by touching it with a hot wire or the end of a hot file, and quickly invert over the crucible a large beaker (or a wide-mouthed bottle), being careful to keep the rim of the beaker below the surface of the water. The white powder formed by the burning phosphorus floats in the air in the beaker but is gradually dissolved by the water.

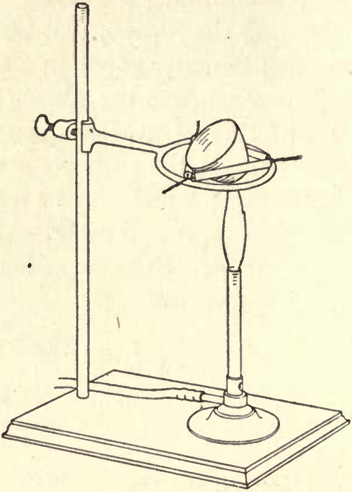


FIG. 16

Leave the beaker in position until the powder has entirely disappeared. Note that the water has risen in the beaker. How do you account for this fact? Is your conclusion in accord with the results obtained by burning iron in air? Suppose it were possible for you to collect and weigh the white powder formed by the burning phosphorus, how would you expect its weight to compare with that of the unburned phosphorus?

c. The burning of a candle. Hold a cold, dry, wide-mouthed bottle over a candle flame, as shown in Fig. 17. Note the film of moisture collecting on the bottle. After one or two minutes remove the bottle quickly and pour into it 5 cc. of clear limewater. Place the palm of the hand tightly over the mouth of the bottle and shake the contents. Note any change in the appearance of the limewater.

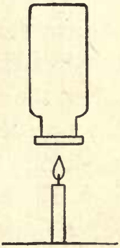


FIG. 17

Clean and dry the bottle and repeat the experiment, but omit holding the bottle over the candle flame. What conclusions do you draw from the experiment? How do you account for the fact that a burning candle gradually loses weight, while iron on burning increases in weight?

EXERCISE 5

CHEMICAL ACTION; ELEMENTS, COMPOUNDS, MIXTURES

Apparatus. Burner; evaporating dish; ring stand; magnet; magnifying glass; test tubes.

Materials. Iron wire, 10 cm. in length; wooden splint; 2 or 3 g. of sugar; 1 g. common salt; piece of granulated zinc; sulfuric acid; 2 g. sulfur; 2 g. powdered iron.

a. *Chemical action.* 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 (Fig. 15). 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***?

f. Elements; compounds; mixtures. 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 powdered iron 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?

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, remove the substance (it may be necessary to break the tube to do so), 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?

EXERCISE 6

COLLECTION OF GASES; PREPARATION OF OXYGEN FROM (*A*) MERCURIC OXIDE, (*B*) SODIUM PEROXIDE

Apparatus. 250-cc. wide-mouthed bottles; glass plate; pneumatic trough or granite-ware pan; hard-glass test tube; * apparatus Fig. 19.

Materials. 0.5 g. mercuric oxide; wooden splint*; 5 g. sodium peroxide.

a. 1. *Collection of gases.* Fill a wide-mouthed bottle (250-cc.) with water. Cover its mouth with a glass plate, being careful to exclude all air bubbles. Hold the plate firmly in place, invert the bottle, and dip its mouth into the water in a pneumatic trough. Remove the glass plate. Why does the water remain in the bottle? Now fill the bottle with exhaled air by placing one end of a piece of glass or rubber tubing under the mouth of the bottle and blowing gently through the other end. Before the bottle, so filled, is removed from the trough, cover its mouth tightly with a glass plate. The bottle so covered may then be placed on the desk in either an upright or an inverted position. (When should it be placed in an inverted position?)

2. Fill a bottle with exhaled air and then transfer the air to another bottle. Draw a diagram to show the method of doing this.

FIG. 18

b. Preparation of oxygen from mercuric oxide. In the bottom of a clean, dry test tube place about $\frac{1}{2}$ g. of mercuric oxide. This is done by placing the oxide near the end of a narrow strip of folded paper and introducing it carefully into the tube, as shown in Fig. 18. On inclining the tube and gently tapping the paper the oxide will be deposited in the bottom of the tube. The paper is now withdrawn, leaving the sides of the tube perfectly clean. Now hold the tube between the thumb and fingers (Fig. 9), and apply a gentle heat to the oxide. The tube must be rotated constantly, to distribute the heat; otherwise it may be melted. During the heating insert a glowing splint from time to time into the mouth of the tube. Note the result. Continue to heat as long as any gas is evolved. What remains in the tube? How has the heat affected the mercuric oxide? What kind of change has the mercuric oxide undergone (p. 14 of text)?

***c.** *Preparation of oxygen from sodium peroxide.* Sodium peroxide is a white solid containing 41 per cent of oxygen, and when treated with water, a part of this is set free.

Arrange an apparatus according to Fig. 19. By means of a short piece of rubber tubing *A* connect the funnel *B* with a glass tube *C*, pinching the rubber tube shut with a screw clamp. Place about 5 g. of sodium peroxide in the bottom of *D* and partly fill the funnel with warm water. Very cautiously open the screw clamp so that the water will run down and fall, drop by drop, upon the peroxide. (The funnel must be kept partially filled with water (?)). A steady current of oxygen is given off. Collect a bottle of the gas and test it by thrusting a glowing splint into the bottle. What is the source of the oxygen? What substances remain in the generator bottle (p. 27 of text)?

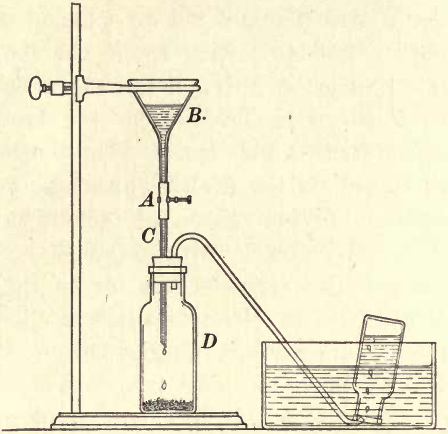


Fig. 19

EXERCISE 7

USUAL LABORATORY PREPARATION OF OXYGEN; PROPERTIES OF OXYGEN

Apparatus. Test tubes; test tube prepared in Exercise 2 (Fig. 2), connected with rubber tube as shown in Fig. 20; trough and wide-mouthed bottles shown in Fig. 20; burner; deflagrating-spoon; 4 pieces of window glass 10 cm. square; funnel; evaporating-dish.

Materials. 10 g. potassium chlorate; 4 g. manganese dioxide; wooden splint; 1 g. of sulfur; picture-frame wire 20 cm. long; bit of cotton; filter paper.

a. *Preparation of oxygen from potassium chlorate (preliminary experiment).* Select two test tubes of the same size, and clean and dry them thoroughly. Into the one introduce 2 g. of potassium chlorate; into the other introduce 2 g. of potassium chlorate mixed intimately with 1 g. of manganese dioxide.

Now heat the contents of the two tubes, applying the flame so that both tubes are equally heated. Repeatedly thrust a glowing splint into each tube in order to detect any oxygen that may be evolved. Note the results. What effect has the manganese dioxide? Does the addition of the manganese dioxide enable one to obtain an increased amount of oxygen (p. 26 of text)? What term is applied to substances which act like the manganese dioxide?

b. *Preparation of oxygen; usual laboratory method.* Mix intimately on paper 6 g. of potassium chlorate and 3 g. of manganese dioxide. The presence of impurities in the materials may lead to a serious explosion when heat is applied; hence test a small portion of the mixture, say 0.5 g., by heating it in a test tube. In the absence of impurities the oxygen is evolved quietly, unaccompanied by any very marked sparking in the materials.

Place the apparatus prepared in Exercise 2 (Fig. 2) on a ring stand as shown in Fig. 20, and connect with it a piece of rubber tubing *C*; also fill 4 wide-mouthed bottles (250-cc.) with water and invert them in a pneumatic trough, as shown in the figure. Now transfer the mixture of potassium chlorate and manganese dioxide to the glass tube *A*, and insert the cork (Fig. 20); then, holding the burner in the hand, heat the mixture gently with a small flame, applying the heat at first to the upper part of the mixture. The flame must not strike the upper part of the test tube, as the cork may be ignited. At first the heat expands the air and a few bubbles of air escape; then the oxygen is evolved. Regulate the heat so as to secure a uniform and not too rapid evolution of the gas. Will the gas which passes over at first be the pure oxygen? Collect four 250-cc. wide-mouthed bottles of the gas. Before the heat is withdrawn, remove the cork from the tube (?). What is the source of the oxygen? Place the tube and contents aside for use as described in *d*.

c. *Properties of oxygen.* 1. Note the physical properties (color, odor, taste, solubility in water) of the gas. (The slight cloud that is often present when oxygen is prepared from potassium chlorate is due to an impurity and will disappear if the gas is allowed to stand over water.)

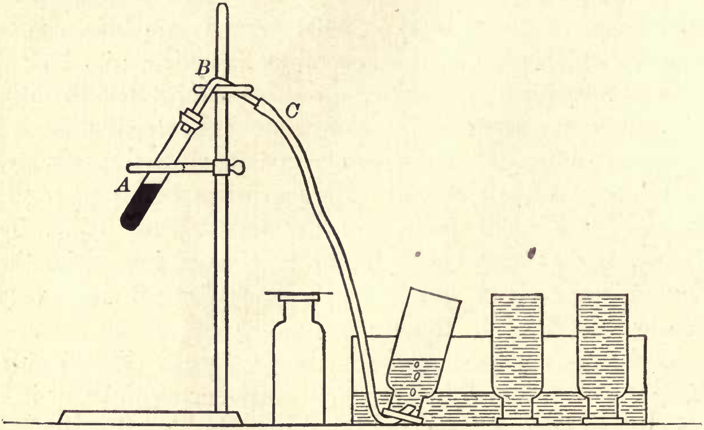


FIG. 20

2. Repeatedly thrust a glowing splint into a bottle of the gas.

3. Heat some sulfur in a deflagrating-spoon until it begins to burn. Note the color and size of the sulfur flame. Now lower the burning sulfur into a bottle of oxygen and note the change.

4. Tip the piece of picture-frame wire with sulfur by wrapping a very small bit of cotton about the end of the wire and dipping this into melted sulfur (for this purpose melt a little sulfur in a deflagrating-spoon). Ignite the sulfur by holding it in a Bunsen flame for an instant, and then thrust the wire into a bottle of oxygen.

Describe the results obtained in 2, 3, and 4. What becomes of the oxygen?

d. Separation of the compounds present in the residue left in the preparation of oxygen. Heat the tube containing the residue until no more oxygen is evolved. After the tube is cool, nearly fill it with water and shake the contents thoroughly. After a few minutes, filter off the solid matter (what is it?), repeating the filtration, if necessary, in order to obtain a perfectly clear filtrate. Evaporate about one third of the liquid, and set it aside until crystals are deposited. Convince yourself that the substance is different from the potassium chlorate with which you started. What is the compound (p. 26 of text)?

EXERCISE 8

THE PREPARATION AND PROPERTIES OF HYDROGEN

Apparatus. Test tube; apparatus, bottles, and trough as shown in Fig. 21 (the bottles are 250-cc., wide-mouthed); beaker; stirring-rod; 60-cc. bottle; burner; evaporating-dish; * funnel.

Materials. Bit of sodium (size of a small pea); filter paper; wooden splints; 10 g. granulated zinc; copper sulfate solution (R. S.); sulfuric acid.

a. Preparation from water. Fill a test tube with water and invert it in a beaker of water. Wrap a piece of sodium in a bit of filter paper previously moistened with coal oil. Raise the inverted test tube until its mouth dips just below the surface of the water in the beaker, and quickly insert the sodium. Stand at arm's length, as a slight explosion sometimes occurs. Notice that the sodium decomposes the water, liberating a gas which is caught in the tube. Place your thumb tightly over the mouth of the tube to prevent the gas from escaping, and bring the tube to an upright position. Light a splint, remove the thumb from the tube, and quickly bring the flame to the mouth of the tube. Does the gas act like oxygen? What is the source of the gas? What other methods may be employed for obtaining it from the same source?

b. *Preparation from acids* (*usual laboratory method*). Prepare a hydrogen generator according to Fig. 21. *D* represents a wide-mouthed bottle of about 250-cc. capacity. The gas-delivery tube *B*, *C* is the same as that used in the preparation of oxygen (Fig. 20). A rubber stopper for the bottle

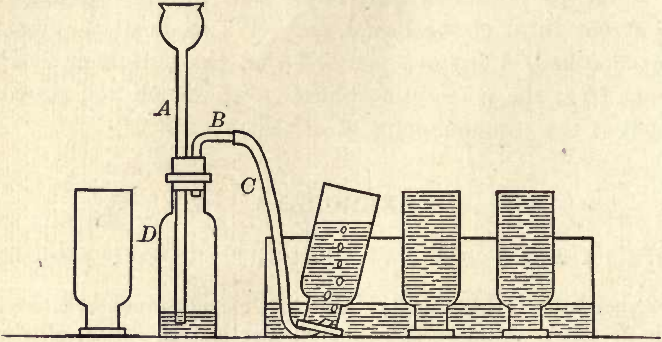


Fig. 21

D is preferable, although a good cork stopper will do. The funnel tube *A* must extend nearly to the bottom of the bottle (?). Put 10 g. of granulated zinc (why granulated?) into *D*. Pure sulfuric acid will not react with pure zinc; hence it is advisable to add to the zinc 8 or 10 drops of a solution of copper sulfate, which will start the reaction. Now pour just enough water through the funnel tube to cover the zinc.

Prove that the apparatus is air-tight by blowing into the delivery tube *C* until the water is forced nearly to the top of the funnel tube; then quickly close the rubber tube either by tightly pinching it or by placing the tongue firmly against its end. If the apparatus is air-tight, the water in the funnel tube will not fall.

Prepare some dilute sulfuric acid by pouring *slowly*, a few drops at a time, 15 cc. of concentrated acid into a beaker containing 50 cc. of water. Stir the water with a glass rod while the acid is being added. *Notice that the acid is poured into the water — never the reverse* (?).

Cool the mixture and pour a few drops of it through the funnel tube. Hydrogen is at once evolved. Enough of the acid must be added from time to time to cause a gentle and continuous evolution of the gas. An excess of the acid should be avoided, however, or the action will become too violent and a large quantity of zinc will have to be added at the close of the exercise.

It is evident that the gas which passes over first is a mixture of hydrogen and air. *The student must remember that such a confined mixture of hydrogen and air explodes with great violence if ignited.* Hence see that the end of the delivery tube is not brought near any flame. Determine when the hydrogen is free from air by repeatedly collecting a test tube full of gas and igniting it, holding the tube mouth downward (?). If pure, the gas burns quietly; otherwise there is a slight explosion. After all the air has been expelled from the generator, collect four bottles (250-cc., wide-mouthed) of the gas.

What is the source of the hydrogen? What is the use of the zinc?

Remove the cork from the generator, add a few more pieces of zinc, and set aside. Sufficient zinc should be used so that at least a small portion of it remains undissolved.

c. *Properties of hydrogen.* 1. Thrust a lighted splint into a bottle of the gas held mouth downward. Slowly withdraw the splint and again thrust it into the gas. Describe the results. What do they prove?

2. Fill a small (60-cc.) wide-mouthed bottle or test tube one-third full of water and invert it in a pneumatic trough. Displace the remaining water with hydrogen from one of the

bottles. What does the bottle now contain? Withdraw it from the water and, holding it at arm's length, quickly bring it, mouth downward, over a flame. What do the results prove?

3. Uncover a bottle (mouth upward) of the gas. After a minute, test for the presence of hydrogen with a lighted splint. Repeat, holding the bottle mouth downward. Describe the results. Is the gas heavier or lighter than air?

*4. Without removing the fragments of undissolved zinc, pour the liquid set aside in Experiment ***b*** into an evaporating-dish and boil gently on a ring-stand support. As soon as white crusts begin to form on the side of the dish, just above the liquid, filter the hot liquid into a beaker and set it aside to cool. How does the product which separates from the filtrate compare in properties with the original zinc? What is the product (p. 41 of text)? How does it differ in composition from sulfuric acid?

EXERCISE 9

THE COMBUSTION OF HYDROGEN; THE OXY-HYDROGEN BLOWPIPE

Apparatus. Hydrogen generator *A* (Fig. 22) attached by rubber tubing *C* to a drying-tube *B*. This tube is filled with granulated calcium chloride, held in place by loose plugs of cotton placed at each end of the tube. *D* is a glass tube drawn to a jet.

Materials. Granulated calcium chloride; cotton; 8 g. zinc; copper sulfate solution (R. S.); dilute sulfuric acid; picture-frame wire 10 cm. long; bit of charcoal.

a. Charge the generator *A* (Fig. 22) with 6 or 8 g. of zinc, add the solution of copper sulfate, cover with water, and add dilute sulfuric acid as in ***b***, Exercise 8. Slip a piece of rubber tubing over the tube *D*, collect samples of the gas in test tubes over water, and test with a flame to see whether the hydrogen is free from air. *After all the air has* *been expelled, wrap a towel carefully about the generator* and cautiously ignite the hydrogen. The flame is nearly invisible and is very hot. Test the heat of the flame by holding in it different objects, such as a splint, a piece of picture-frame wire, a bit of charcoal.

Finally, hold over the flame a cold, dry beaker or bottle and note the liquid deposited on the sides of the vessel. Explain.

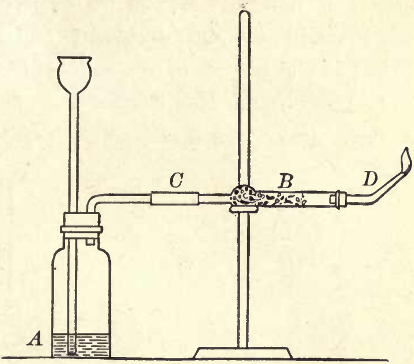


FIG. 22

b. Examine the structure of the oxyhydrogen blowpipe. Draw a diagram representing a cross section of it. Compare it with the blast-lamp used in the laboratory. Why not have a short inner tube?

EXERCISE 10

OXIDATION AND REDUCTION

Apparatus. Hydrogen generator and tubes as shown in Fig 23 (*A* is the hydrogen generator, *B* is a drying-tube filled with calcium chloride, *C* is a straight glass tube, and *D* is a hard-glass test tube); burner; apparatus used in preparing oxygen (Fig 20).

Materials. 2 g. copper oxide; calcium chloride sufficient to fill the drying-tube *B*; 8 g. zinc; dilute sulfuric acid for preparing hydrogen (see Exercise 8); 4 g. potassium chlorate; 2 g. manganese dioxide.

a. Remove the tube *D* (Fig. 23), introduce into it 1 or 2 g. of copper oxide, and return it to the position shown in the figure.

Now generate hydrogen in *A* as in Exercise 8. After all the air has been expelled from the apparatus and the generator wrapped in a towel (see Exercise 9), cautiously heat the copper oxide to redness, being careful to keep the flame away from the mouth of the tube *D* (?). Note the condensation of moisture in the cold portions of the tube. Is there any invisible evidence of change in the copper oxide? Explain. What is the object of the calcium chloride in tube *B*?

b. Disconnect the bottle *A* at *E* from the rest of the apparatus and force a little air through the tube *B* to remove the hydrogen present. Now connect the apparatus used in preparation of oxygen (Fig. 20) at *E* to the tubes *B*, *C*, and *D*. Generate oxygen and conduct a slow current of the gas through *B*, *C*, and *D*, at the same time heating the residue in *D* (?).

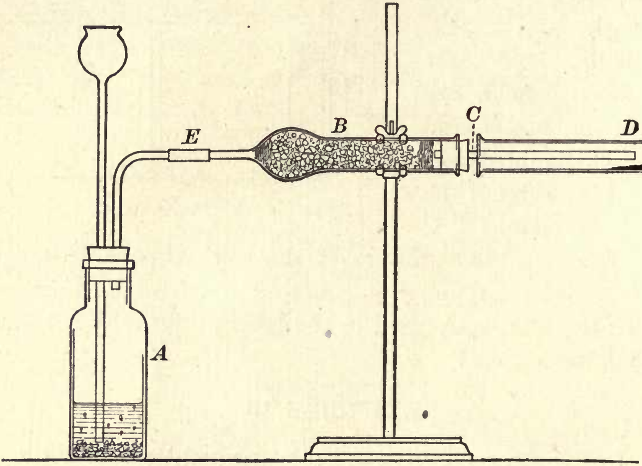


FIG. 23

c. Explain the terms *reduction* and *oxidation* and give an example of each process from the above experiment.

EXERCISE 11

MEASUREMENT OF GAS VOLUMES

Apparatus. Graduated tube and cylinder as shown in Fig. 24.

a. (It is suggested that the instructor arrange one or more pieces of apparatus as shown in Fig. 24. The students will then take the readings and solve the problems.)

Partially fill a graduated tube with water and invert it in a cylinder (or other vessel) of water as shown in Fig. 24. Adjust the tube until the level of the liquid inside and outside of the tube is the same; then take the reading of the volume of the air in the tube. Note the temperature of the air (place a thermometer by the side of the tube) and likewise the pressure of the atmosphere as indicated by the barometer. Insert these values in their appropriate places in the following table:

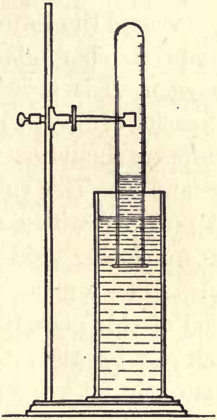


Fig. 24

Volume of air in tube . . . . cc.
Temperature of air . . . . °
Vapor pressure at temperature of air (see Appendix) . . . . mm.
Barometric pressure . . . . mm.
Effective pressure on gas in tube (barometric pressure less vapor pressure) . . . . mm.

From the above values calculate what volume the air inclosed in the tube would have under standard conditions.

EXERCISE 12

THE DETERMINATION OF THE WEIGHT OF ONE LITER OF OXYGEN; ALSO, OF THE PERCENTAGE OF OXYGEN IN POTASSIUM CHLORATE (QUANTITATIVE)

Apparatus. Apparatus shown in Fig. 25; burner; 500-cc. beaker.
Materials. 1 g. potassium chlorate.

Prepare the form of apparatus shown in Fig. 25. *A* represents the hard-glass test tube used in the preparation of oxygen, *B* is a common (narrow-mouthed) bottle having a capacity of about 1 liter; rubber stoppers should be used in both *A* and *B*. The rubber tube *C* is provided with a screw clamp *D*, for closing the tube, and has in its end a glass tube *E*. The end of this glass tube is drawn out to a jet, the internal diameter of the jet being about 2 mm. *F* is a 500-cc. beaker.

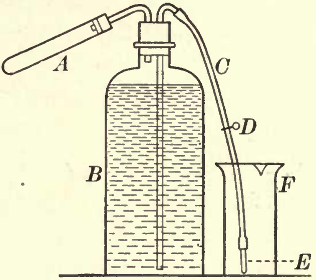


FIG. 25

The bottle is nearly filled with water, as shown in the figure, and allowed to stand until it acquires the room temperature. The tube *A* is now removed and a gentle suction is applied to the glass jet *E*. The water siphons over, through the tube *C*, into the beaker and is allowed to run for a moment so as to fill completely both the rubber and the glass tubes (*C* and *E*). The rubber tube is then quickly closed with the screw clamp *D*.

Now thoroughly clean and dry the tube *A* and carefully weigh it; then introduce about 1 g. of potassium chlorate into the bottom of the tube by means of a folded paper (Fig. 18), and reweigh. Attach the tube, as shown in Fig. 25, *care being taken to have the apparatus air-tight.*

The pressure of the air within the bottle is now adjusted to that of the air outside as follows: Water is added to the beaker *F*, if necessary, until the end of the glass tube *E* is covered. The screw clamp is then opened and the beaker at once raised vertically until the water in the beaker is at the same level as the water in the bottle and is retained in this position until the screw clamp *D* is closed. The beaker is then emptied and returned to the position shown in Fig. 25.

Now open the clamp *D* and gently heat the potassium chlorate in *A*. The oxygen is evolved and forces the water from the bottle into the beaker. Gradually increase the heat, and *continue the heating until all the oxygen has been expelled* (shown by the fact that no more water passes over into *F*). Let the apparatus stand until it has acquired the room temperature, care being taken that the glass jet *E* is kept below the surface of the water in the beaker (?).

Now bring the level of the water in the beaker to that of the water left in the bottle, and while holding it in this position close the screw clamp (?). Carefully measure the water in the beaker; also take the reading of the thermometer and the barometer. Disconnect the tube *A* and carefully reweigh. Insert the values in the following table:

Weight of tube *A* . . . . g.
Weight of tube *A* + potassium chlorate . . . . g.
Weight of tube *A* + potassium chloride . . . . g.
Weight of oxygen evolved (loss in weight of contents of tube *A* on heating) . . . . g.
Volume of water in beaker = volume of oxygen evolved . cc.
Temperature of water . . . . °
Barometric reading . . . . mm.
Value of vapor pressure (see Appendix) . . . . mm.

From your results calculate the weight of 1 liter of oxygen under standard conditions; also calculate the percentage of oxygen in potassium chlorate. Compare your results with the actual values (pp. 25 and 29 of text).

EXERCISE 13

A STUDY OF THE PROCESS OF DISTILLATION

Apparatus. Flask (250-cc.); condenser and connections as shown in Fig. 26, or apparatus shown in Fig. 27; ring stands; wire gauze 12 cm. square; watch glass.

Materials. 10 cc. alcohol.

a. (Two or more students may work together if the apparatus is not available for each.) Connect a Liebig condenser *B* with a 250-cc. flask *A*, as represented in Fig. 26. The flask is set on a piece of wire gauze supported by the iron ring attached to the ring stand. The tube *C* is connected with the water pipe by means of rubber tubing, and a current of cold water is conducted through the outer tube of the condenser, escaping through the tube *D*. (Why is cold water forced in at *C* rather than at *D*?)

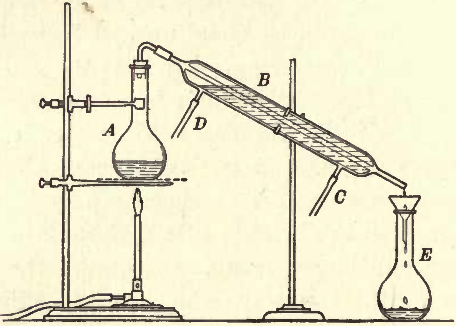


FIG. 26

Fill the flask one-fourth full of hydrant or well water and boil until 50 cc. or more of liquid has collected in the *receiver E*. (In case condensers are not available, the apparatus shown in Fig. 27 may be used. When this is used the steam from the boiling water in flask *A* is condensed by conducting it through *B* into the test tube *C*, which is kept cold by the ice water in the beaker *D*.)

Compare the *distillate* (distilled water) with the hydrant water in appearance and in taste (?).

Place 4 or 5 drops of the distilled water on a watch glass and evaporate, holding the watch glass 10 or 15 cm. above the tip of the flame. Is there any residue? Repeat, using hydrant water. Why is distilled water used in the laboratory?

b. Repeat the distillation, using a sample of muddy water in *A* (?).

c. Pour 1 or 2 cc. of alcohol into a porcelain dish and bring a flame in contact with it. Is the alcohol inflammable? Now distill a mixture of 10 cc. of alcohol (boiling point 78.3°) and 30 cc. of water. Collect the first 1 or 2 cc. of the distillate in an evaporating dish and test with a flame. In the same way test successive portions of the distillate. Does there seem to be a partial separation of the two liquids? In this way a mixture of liquids boiling at different temperatures may generally be separated more or less perfectly. The process is termed *fractional distillation.*

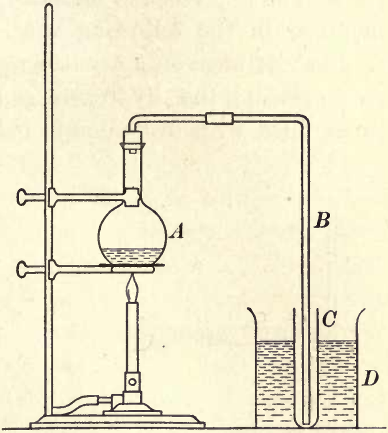


FIG. 27

EXERCISE 14

THE COMPOSITION OF WATER

Apparatus. Hydrogen generator and tubes as shown in Fig. 28 and described below; balance; apparatus shown in Fig. 25, but without test tube and stopper.

Materials. 10 g. granulated zinc; 2 g. black powdered copper oxide; sulfuric acid; granular calcium chloride, sufficient to fill tubes *B* and *D*.

a. Recall the experiments included under Exercises 9 and 10. What do the results show in reference to the composition of water? Is the process employed in Exercise 10 one of synthesis or analysis? Are the results qualitative or quantitative in character?

****b.*** The experiments included under Exercise 10, may be modified in the following way so as to give quantitative results. Arrange an apparatus as shown in Fig. 28, in which *A* represents the hydrogen generator, and *B* and *D* are tubes filled with dry calcium chloride. The hard-glass tube *C* and the porcelain boat *E* are obtained from the storeroom. The tube is about 35 cm. in length. Introduce about 2 g. of black oxide of copper into the boat and weigh accurately to milligrams. Introduce the boat into the glass tube so that the end of the boat is about 8 cm. from the end of the tube connected with *D*. Close the ends of tube *D* with short pieces of rubber tubing, one end of each being closed with a small glass rod; then weigh the tube accurately. Remove the rubber tubes and glass rods, carefully preserving them for use when the tube is again weighed. Now connect the apparatus as shown in the figure, taking care to render it air-tight. How is this determined? Generate hydrogen slowly, and when the apparatus is free from air, heat the boat very gently, using the wing-top burner. Gradually increase the heat, all the time maintaining the slow current of hydrogen. When the copper oxide is reduced,

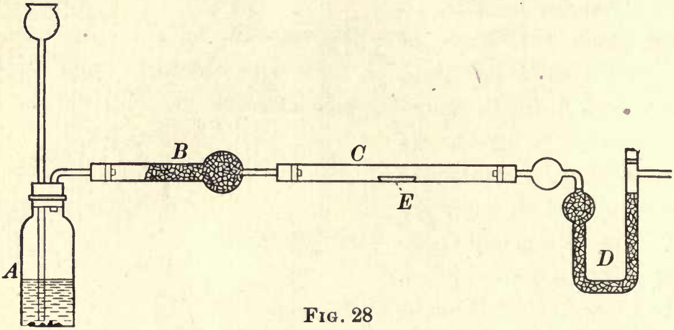


Fig. 28

or nearly so, withdraw the heat *but maintain the current of hydrogen until the apparatus is cool.* If any of the water formed remains condensed in the end of tube *C*, a very gentle heat is cautiously applied (the flame must not strike the tube) until it is driven into the tube *D*.

When the apparatus has acquired the room temperature, disconnect *A* from the remainder of the apparatus and attach *D* (Fig. 28) by a short piece of rubber tubing to the short bent glass tube in bottle *B* of Fig. 25. The bottle is filled with water, and a portion of it is slowly siphoned over through *C*, *D* (Fig. 25). In this way a current of air is drawn through the apparatus, displacing the hydrogen. Finally, disconnect the apparatus, and at once close the ends of the tube *D* (Fig. 28) with the rubber tubes provided with glass rods. Weigh the boat and contents; also the tube *D*. From your results calculate the composition of water. Compare your results with those obtained by other members of the class. Calculate the general average of all the results. How does this result compare with the actual values (p. 77 of text). What sources of error are included in the experiment?

EXERCISE 15

THE PREPARATION AND PROPERTIES OF HYDROGEN PEROXIDE

Apparatus. Test tube; 200-cc. beaker; glass rod; funnel and filter paper.

Materials. 10 cc. of ordinary hydrogen peroxide solution; wooden splint; 1 g. manganese dioxide (powder); starch paste (R.S.); crystal potassium iodide; 6 cc. ether; potassium dichromate (R.S.); 1 g. sodium peroxide; ice water (100 cc.); piece of blue litmus paper; sulfuric acid.

a. What is the strength of the hydrogen peroxide solutions sold by the druggist (p. 83 of text)? Pour 3 cc. of the solution into a large test tube and add 1 g. of finely powdered manganese dioxide. Test the gas evolved with a glowing splint (?). What kind of an agent is hydrogen peroxide?

Filter the mixture remaining in the tube. The solid is the unchanged manganese dioxide. In what other experiment has manganese dioxide been used to assist in bringing about a chemical action, the dioxide apparently at least undergoing no change?

b. To 1 cc. of starch paste, add 4 cc. of water. Dissolve in this a small crystal of potassium iodide and then add a few drops of a solution of hydrogen peroxide. The peroxide liberates the iodine from the potassium iodide and the resulting free iodine colors the starch paste. Note the results.

c. Pour 3 cc. of the solution of hydrogen peroxide into a test tube and add an equal volume of ether (CAUTION: ether is very inflammable). Shake the solution vigorously and notice that the ether quickly rises to the top of the tube when the tube is set aside. Now add 1 drop of a solution of potassium dichromate and again shake the mixture. Note the color of the layer of ether now. A blue color in the ether constitutes a delicate test for hydrogen peroxide.

d. When sodium peroxide is added to water, hydrogen peroxide is formed, but this decomposes even at ordinary temperatures into water and oxygen (p. 27 of text). If the temperature is kept low however, the hydrogen peroxide remains unchanged and it is possible in this way to prepare the compound. To prepare the peroxide proceed as follows: Add 1 g. of sodium peroxide, a little at a time, to 100 cc. of ice water. Now add dilute sulfuric acid, a few drops at a time, to the liquid (stir thoroughly with a glass rod) until a drop of the resulting liquid placed on a piece of blue litmus paper changes the blue to a red color. Then test the liquid for the presence of hydrogen peroxide in accordance with the methods given above under ***b*** and ***c***.

EXERCISE 16

STATES OF MATTER

Apparatus. Watch crystal; 500-cc. beaker; thermometer graduated to at least 110°; ring stand and burner.

Materials. 1 cc. ether or chloroform; 10 cc. benzene; pieces of ice.

a. Place about 1 cc. of ether or of chloroform on a watch crystal and blow upon the surface of the liquid. Account for the rapid evaporation. Do you notice any change in the temperature of the watch glass? Explain.

b. Pour about 250 cc. of distilled water into a suitable flask or beaker, and place a thermometer in the liquid so that the temperature may be noted. Now heat the water at such a rate that the temperature rises slowly but steadily. At what temperature do you observe the formation of bubbles? Where do they appear to form? Of what are they composed? At about what temperature do larger bubbles begin to form at the bottom of the vessel? What becomes of them? Explain. At what temperature do they move freely up through the liquid to the surface? Do they get larger or smaller as they rise? Why? When the water is gently boiling, try increasing the heat. Is the boiling any more energetic? Does the temperature rise any? How do you define the boiling point?

c. Pour about 10 cc. of benzene into a clean, dry hard-glass test tube. *This liquid is inflammable and must be kept away from all flames.*. Pour into a beaker some ice water in which some pieces of ice are floating. Now immerse in this ice water that part of the test tube which is filled with benzene. Stir the benzene constantly with a thermometer, noting the temperature from time to time. Carefully note the temperature when the benzene begins to freeze and continue the reading until the entire liquid, or nearly all of it, is frozen. Now remove the tube from the ice water and hold the lower part of it in the hand until the benzene is melted, noting the temperature during this change. Note the results and discuss.

d. Experiment ***c*** may be repeated, using water in the test tube and immersing it in a freezing-mixture made by mixing 3 parts of powdered ice with 1 part of common salt.

EXERCISE 17

THE FORMATION OF CHARCOAL AND COKE

Apparatus. Hard-glass test tube *A* connected as shown in Fig. 29; *B* is a test tube fitted with a glass tube *C* drawn to a jet; large beaker; porcelain dish; burner.

Materials. Small pieces of hard wood (sawdust will do) sufficient to half fill the hard-glass test tube; pieces of soft coal sufficient to half fill the test tube; blue and red litmus paper.

a. Half fill the tube *A* (Fig. 29) with hard-wood splints or sawdust and connect it as shown in the figure. The tube *B* is kept cool by ice water in the beaker. Heat the wood in *A*, gradually increasing the heat until no further change takes place. During the heating, test the gas escaping from the jet *C* to determine whether it will burn (?).

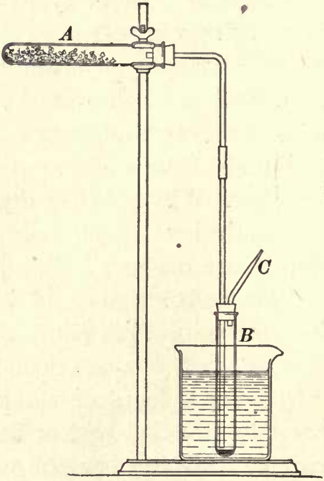


Fig. 29

When the tube *A* is cool, remove the residue (?). Note its properties. Is it combustible?

Note the odor and appearance of the liquid condensed in *B*. Name two important compounds prepared commercially from the liquid obtained by heating hard wood in the absence of air (p. 121 of text). What name is applied to the process undergone by the wood?

b. Repeat experiment ***a***, substituting small pieces of *soft* coal for the wood. Describe the results. What is left in the tube *A*? The liquid condensed in *B* is known as *coal tar* (p. 306 of text). Note its appearance and odor.

EXERCISE 18

A FURTHER STUDY OF CARBON

Apparatus. Hard-glass test tube; porcelain dish; burner; funnel.

Materials. 3 g. sugar; test tube one-fourth full of bone black; 1 cc. litmus solution; filter paper; common acids; 3 g. copper oxide and an equal bulk of powdered charcoal; 5 cc. limewater (R.S.).

a. Heat 2 or 3 g. of sugar in a test tube until no further change takes place. Note the results. What is the residue?

b. Bring a cold porcelain dish into a small luminous Bunsen flame. Note the deposit. What is this form of carbon called? In what other forms does carbon exist?

c. Put into a small flask enough bone black to fill a test tube one-fourth full and pour over it about 50 cc. of water, to which has been added a few drops of a solution of litmus or indigo. Thoroughly mix the contents of the flask; then heat gently for a few minutes, and filter. If the filtrate is not decolorized, repeat the process, using more bone black. What is the composition of bone black? By what other name is it known? What use does this experiment suggest for it?

d. Is carbon an active element at ordinary temperatures? Test it with the common acids. How does the charring of wood preserve it?

e. Mix together in a mortar 2 or 3 g. of black copper oxide and an equal bulk of powdered charcoal. Transfer to a hard-glass test tube and heat gently. The copper oxide is reduced to copper, the oxygen combining with the hot carbon to form carbon dioxide.

EXERCISE 19

A STUDY OF CARBON DIOXIDE

Apparatus. Hydrogen generator with connections, as shown in Fig. 30; three 250-cc. bottles; small beaker or test tube; hard-glass test tube.

Materials. 5 pieces of marble (size of walnuts); hydrochloric acid; splints; limewater (R. S.); 3 g. of copper oxide and an equal bulk of powdered charcoal.

a. *Usual laboratory method for preparing carbon dioxide.* Place some pieces of marble in your hydrogen generator and connect as shown in Fig. 30. Add water through the funnel tube until the marble is covered with the liquid; then add hydrochloric acid, a few drops at a time. Collect three bottles of the gas by displacement of air. To test when filled, hold a burning splint at the mouth of the bottle; the gas will extinguish the flame. Why not collect the gas over water as in the case of oxygen and hydrogen?

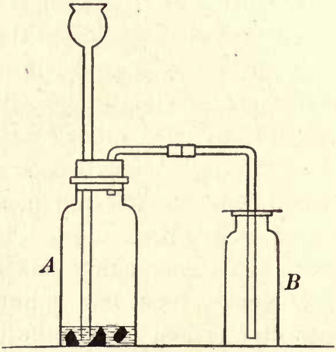


FIG. 30

b. Thrust a burning splint into one of the bottles of the gas (?). The results suggest what use for the gas?

c. Devise an experiment to show whether the gas is heavier or lighter than air. Attempt to pour it from one bottle to another, as you would a liquid, and test with a burning splint for its presence in the second bottle (?).

d. Pass a few bubbles of the gas through 10 cc. of limewater (?). This serves as a good test for carbon dioxide.

e. Prove that the air exhaled from the lungs contains carbon dioxide; also prove that it is formed when ordinary fuels burn.

**f.* Repeat experiment *e*, Exercise 18, and prove that carbon dioxide is evolved.

EXERCISE 20

PREPARATION AND PROPERTIES OF NITROGEN

Apparatus. 250-cc. flask, with cork and glass delivery tube, like that used in preparing oxygen (*B*, *C*, Fig. 20); ring stand; burner; pneumatic trough; 3 wide-mouthed bottles.

Materials. 3 g. ammonium chloride; 6 g. sodium nitrite.

a. Recall experiment *b*, Exercise 4. What is the gas left in the beaker after the combustion of the phosphorus?

b. In a 250-cc. flask introduce a mixture of 3 g. of ammonium chloride and 6 g. of sodium nitrite, and add 20 cc. of water. Provide the flask with a one-hole cork and delivery tube so that the evolved gas may be collected over water, as in the case of oxygen and hydrogen. Have at hand a vessel of cold water so that the flask may be cooled by lowering it into the water in case the action becomes too violent.

Clamp the flask and apply a *very gentle* heat, moving the burner about with the hand. As soon as the action begins, withdraw the heat. After the air has been expelled from the apparatus, fill three bottles (250-cc.) with the gas. If the action becomes too violent, immerse the flask in cold water.

Write the equations for the reactions involved (p. 130 of text).

Note the physical properties of the gas. Can it be poured from one bottle to another as in the case of carbon dioxide?

Is the gas combustible (test with burning splint)? Is it a supporter of combustion?

EXERCISE 21

THE COMPOSITION OF AIR

Apparatus. Test tube; beaker; apparatus shown in Fig. 31.

Materials. 10 cc. ice water; 5 g. potassium hydroxide dissolved in 5 cc. water and cooled to room temperature; 4 g. pyrogallic acid dissolved in 10 cc. water; bit of calcium chloride; 10 cc. limewater (R. S.).

a. Experiments already performed have proved the presence of certain constituents of the air. Enumerate the experiments and their bearing on the composition of air.

b. Thoroughly dry the outside of a test tube or beaker and partially fill it with ice water. What is the liquid which condenses on the outside of the container? What is the source of this liquid? Place a small piece of calcium chloride on a watch glass and leave it exposed to the air for two or more hours. Note the results. (A substance which, like calcium chloride, takes up moisture from the air is said to be *deliquescent* (p. 411 of text).)

c. Pour 5 cc. of limewater into a small beaker and expose it to the air for one half hour. What do the results indicate?

****d.*** *Determination of the relative amounts of nitrogen and oxygen in the air* (*quantitative*). The relative volumes of oxygen and nitrogen in the air may be determined by bringing in contact with a definite volume of air a liquid which absorbs the oxygen and in so doing flows into the tube which contains the air, and fills a space equal to that previously occupied by the oxygen. The volume of this liquid can be easily measured and the volume of the absorbed oxygen ascertained.

The solution used to absorb the oxygen soon loses its strength on exposure to air; the experiment must therefore be performed rapidly. Before preparing the solution the student should practice the manipulations involved in the experiment, as skill is required for accurate results.

Arrange an apparatus like that shown in Fig. 31. *A* represents a test tube about 15 cm. in length (use the hard-glass tube employed in the preparation of oxygen). The tube is fitted with a two-hole rubber stopper. One hole is closed with a glass rod *B*, while the other is fitted with a small glass tube, the upper end of which extends 4 or 5 cm. above the stopper. A piece of soft-rubber tubing *C*, 12 or 15 cm. in length, connects the glass tube with a small funnel, as shown in the figure.

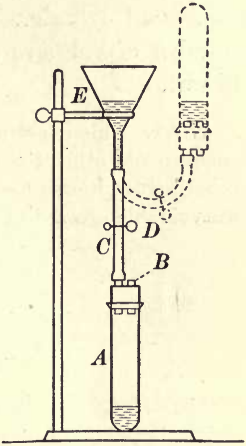


FIG. 31

Close the rubber tube tightly with the screw clamp *D*. Disconnect the test tube and remove the glass rod *B* from the stopper preparatory to performing the experiment.

Add the solution of potassium hydroxide to the solution of pyrogallic acid, mix, and *at once* pour the resulting liquid into the funnel *E*. Quickly open the screw clamp *D* until the rubber tube and the glass tube are both filled with the liquid, and then close the clamp tightly.

Connect the test tube, holding it by the rim to avoid heating the contained air, and insert the glass rod in the cork. The air inclosed in the tube is now at the same temperature and pressure as the surrounding air.

Now open the screw clamp. The liquid flows in, absorbing the oxygen. When the liquid ceases to enter, grasp the tube by the rim and invert it, as shown by the dotted lines of the figure, adjusting it so that the level of the liquid is the same in both tube and funnel (?); then clamp the rubber tube tightly and return the test tube to its original position.

Mark the volume of the air originally inclosed in the tube by placing a narrow strip of gummed paper about the tube at the lower end of the stopper; mark also, by a strip of paper placed at the level of the liquid in the tube, the volume of the oxygen absorbed.

Disconnect the tube and rinse it. Measure the volume of the tube to each strip of paper by pouring in water from a graduated cylinder. From these measurements calculate the number of volumes of oxygen and nitrogen in 100 volumes of air.

NOTE. This experiment disregards the presence in the air of all constituents other than oxygen and nitrogen. The volume of such constituents, however, in the amount of air taken is smaller than the unavoidable errors in the experiment.

EXERCISE 22

A STUDY OF SOLUTIONS

Apparatus. Test tubes; funnel and filter paper; 100-cc. beaker; watch glass; ring stand and burner; apparatus shown in Fig. 32 (*A* is a 250-cc. flask, *B* is a thermometer, and *C* an open glass tube).

Materials. 2 crystals of potassium permanganate; 0.2 g. powdered calcium sulfate; 3 g. common salt; 3 g. potassium nitrate; 1 g. sugar; 10 cc. carbon tetrachloride (R. S.); 1 cc. oil (cotton-seed or olive); 10 g. sodium thiosulfate.

a. Nearly fill two test tubes with water and set them in a rack. Drop into each a small crystal of potassium permanganate. Shake the contents of one tube and repeat the shaking after a few minutes but do not move the other tube. At the close of the laboratory period note the appearance of the liquid in each tube (?).

b. Introduce about 0.2 g. of finely powdered calcium sulfate into a test tube and add 5 cc. of distilled water. Shake the mixture thoroughly and set it aside for 10 minutes. Is there any evidence that the solid is soluble in water? Now filter a few drops of the liquid (?), collecting the filtrate on a watch glass. Set the glass on a small beaker half full of boiling water (Fig. 33) until the liquid is evaporated (?).

c. Place exactly 3 g. of common salt in one test tube and an equal weight of potassium nitrate in another. Add to each exactly 1 cc. of water and heat each tube until the water boils. If the solid does not dissolve, add an additional cubic centimeter of water and again heat. Repeat until the solid in each tube is dissolved. Compare the solubilities of the two solids in the boiling water.

Cool the solutions in each tube and note the approximate amounts of solids separating (?) (compare the results with the table of solubility of solids given on page 561 of text).

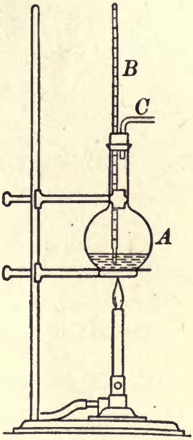


FIG. 32

d. Introduce about 0.5 g. of sugar into each of two test tubes. To the one tube add 5 cc. of water and to the other 5 cc. of carbon tetrachloride (a low-boiling gasoline may be used in place of the carbon tetrachloride, but if used, great care must be taken to avoid any flame, as the gasoline is very inflammable). Shake the tubes gently and note the results. Repeat the experiment, substituting a small bit of lard or an oil for the sugar. How could you remove stains on cloth, due to (1) a sugar sirup (molasses); to (2) oils or grease; to (3) a mixture of the two?

e. Introduce into a test tube 10 g. of sodium thiosulfate (ordinary "hypo" of the photographer) and add 2 cc. of water. Heat the tube gently until a uniform solution is obtained, care being taken that no particles of the solid remain on the side of the tube; stopper the tube loosely with a plug of cotton and set it aside until the solution is cold. If sufficient care has been taken, no solid will have separated.

Now remove the cotton plug and drop into the solution a bit of the solid "hypo" as large as a pin point. Note the results (hold the tube in a good light) and explain.

f. Prepare an apparatus as shown in Fig. 32. *A* is a small flask, *B* is a thermometer, and *C* an open glass tube. Pour some water into *A* and heat it to boiling, noting the temperature of the liquid as the boiling continues (?). Now withdraw the flame and add common salt to the water until it is saturated· then determine the boiling point of the solution (?).

EXERCISE 23

DETERMINATION OF THE SOLUBILITY OF COMMON SALT (QUANTITATIVE)

Apparatus. 60-cc. bottle; evaporating-dish with watch-glass cover; beaker, tripod, and burner as shown in Fig. 33; funnel and filter paper; thermometer.

Materials. 15 g. common salt.

a. Introduce 15 g. of common salt into a 60-cc. bottle and add 40 cc. of water. Shake the mixture vigorously and set aside, repeating the shaking several times at intervals of from one to two minutes, so as to form a saturated solution. Note the temperature of the solution.

Accurately weigh a small evaporating-dish and watch-glass cover; then filter into the dish about 20 cc. of the saturated solution of salt and reweigh. Remove the watch-glass and place the dish in a beaker partially filled with water as shown in Fig. 33. Keep the water in the beaker boiling. The resulting

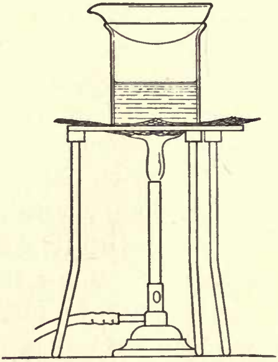


FIG. 33

steam heats the dish and causes the water in the dish to evaporate. After the solution has evaporated to dryness, cover the dish with the watch-glass and heat it directly with the burner, regulating the flame so that the tip barely touches the dish. Continue the heating until all the moisture has been expelled and the under part of the watch-glass is free from moisture. (Why use the watch-glass cover?)

Now withdraw the burner, and after the dish is cool (room temperature) weigh once more. From your results calculate the approximate number of grams of salt that will dissolve in 1 liter of water at the temperature of the original solution.

b. If time permits, the solubility of other substances such as calcium sulfate and potassium dichromate may be determined in like manner; or different students may select different substances and compare their results.

EXERCISE 24

*A METHOD FOR DETERMINING WHETHER OR NOT A GIVEN LIQUID IS A CONDUCTOR OF ELECTRICITY

Apparatus. Apparatus as shown in Fig. 34 (p. 153 of text); current from electric-lighting system.

Materials. 5 g. common salt; 5 g. sugar; 5 cc. sodium hydroxide solution added to 20 cc. water; 3 cc. sulfuric acid added to 20 cc. water; tap or well water.

a. Obtain from your instructor the apparatus shown in Fig. 34. Polish the copper-wire electrodes with emery paper until they are bright and free from oxide. *At the beginning of each experiment see that the electrodes are bright and dry, and that the cell is also perfectly clean.* Unscrew a lamp *C* from a convenient socket above your desk, screw it *loosely* into the socket on your apparatus, and attach the apparatus to the empty socket on the lighting system by means of the extension cord and plug *B.* Every time a change is to be made in the cell, loosen the lamp in the socket, and do not screw it down to make contact until all of the connections of the cell have been arranged.

b. Partly fill the cell *A* with dry powdered salt, dip the electrodes into the powder, arrange the connections at the binding-posts, and screw down the lamp *C*. Does the salt conduct the electric current?

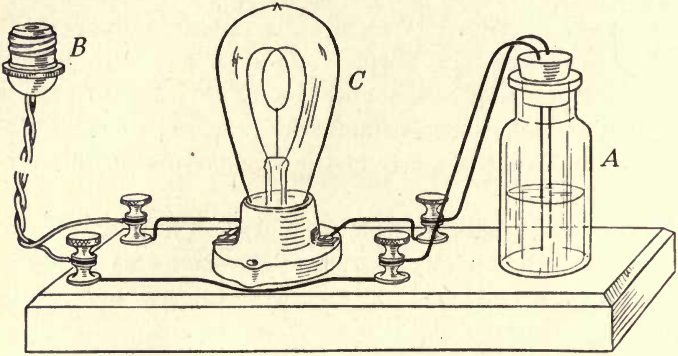


Fig. 34

c. Repeat ***b***, using distilled water (?).

d. Repeat ***b***, using a solution of the salt in distilled water (?).

e. Test the conductivity of the following substances and interpret the results: (1) dry powdered sugar; (2) a solution of sugar in water; (3) tap or well water; (4) distilled water containing a few drops of sulfuric acid; (5) a solution of sodium hydroxide.

EXERCISE 25

THE PREPARATION AND PROPERTIES OF CHLORINE

Apparatus. Apparatus as shown in Fig. 35 (*A* is a 250-cc. flask and *B* and *C* 250-cc. bottles; *B* contains some sulfuric acid); 4 additional 250-cc. bottles (dry); glass plates; 200-cc. beaker with glass plate cover.

Materials. 25 g. manganese dioxide; hydrochloric acid; bit of powdered antimony; strip of copper foil; strips of colored calico; piece of printed paper (printer's ink); paper written over with ordinary ink; 1 g. potassium permanganate.

PRECAUTION. *All of the following experiments must be performed in the hood, and great care must be taken not to inhale the chlorine.*

a. (Two students may work together.) Arrange an apparatus according to Fig. 35. Put into the flask from 20 to 25 g. of manganese dioxide. Insert the cork and pour 150 cc. of hydrochloric acid through the funnel tube. Shake the flask so as to mix the contents thoroughly. Warm gently, applying just enough heat to cause a gentle evolution of the gas, but not sufficient to boil the liquid. The chlorine generated bubbles through the sulfuric acid in *B* (which removes the moisture) and is collected in *C*. Fill four bottles with the gas (note the color), cover them with glass plates, and set them aside.

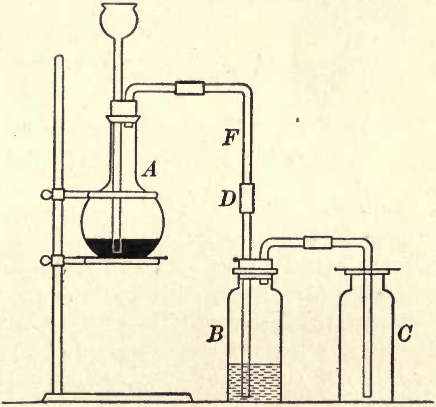


FIG. 35

b. Grind a fragment of antimony to a fine powder and sprinkle a pinch of the powder into one of the bottles of the gas. $SbCl_3$ is formed (R).

c. Support by forceps a small piece of copper foil, heat it to redness, and *immediately* thrust it into a bottle of the gas. Describe the result. What is formed?

d. Suspend strips of colored calico in a bottle of the dry gas; also two strips of paper, the one with writing in ink on it, the other with printing (printer's ink) on it.

e. Repeat ***d***, using similar strips, but moistened with water. Describe the results in ***d*** and ***e***. What part does the water play in the bleaching?

f. Place a few crystals of potassium permanganate on the bottom of a clean beaker. Pour over these 3 cc. of hydrochloric acid and cover the beaker with a glass plate. Examine after a few minutes and test (?) the gas in the beaker for the presence of chlorine (?).

EXERCISE 26

THE PREPARATION AND PROPERTIES OF HYDROGEN CHLORIDE AND OF HYDROCHLORIC ACID

Apparatus. Flask and bottle connected as shown in Fig. 36 (this is same as shown in Fig. 35, except that the glass tube extending into the bottle *B* does not touch the liquid (water) in *B*); two 250-cc. bottles (dry); burner; large beaker.

Materials. Dilute sulfuric acid prepared by slowly pouring (3 or 4 drops at a time with constant stirring) 30 cc. of the concentrated acid into 10 cc. water; 50 g. sodium chloride; splint; blue litmus paper.

a. *Usual laboratory method of preparation.* Put about 50 g. of common salt into the generator flask (Fig. 36), insert the cork, pour the cold dilute sulfuric acid through the funnel tube, mix the contents by a gentle motion of the flask, and after two or three minutes warm gently with a *small* flame. Notice the currents in the water in *B*. What causes them? As soon as the gas is evolved regularly, disconnect the generator flask at *D* long enough to collect two bottles of the gas by displacement of air (as in Fig. 30). Cover these tightly with glass plates and set them aside; then connect the generator with *B* again and continue to apply a gentle heat as long as any gas is evolved (R).

b. What is the color of the gas (examine that in the generator)?

c. Test the gas in one of the bottles with a lighted splint. Is it combustible? Is it a supporter of combustion?

d. Fill a large beaker with water. Now uncover the remaining bottle, invert it, and at once bring its mouth under the surface of the water in the beaker. Describe the results. What does the experiment prove? Why not extend the tube in bottle *B* (Fig. 36) to the bottom of the bottle?

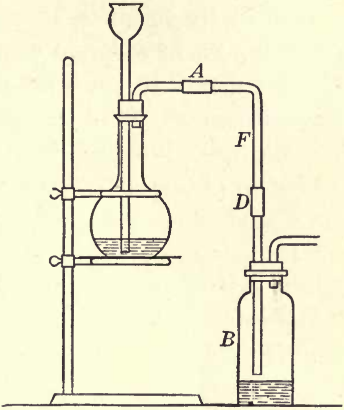


FIG. 36

e. Put a drop of the aqueous solution of the acid from bottle *B* on a bit of blue litmus paper. Note the result. Pour 2 drops of the solution into 3 or 4 cc. of water and taste a drop. Perform a test-tube experiment to prove the presence of chlorine in the acid (R); also one to prove the presence of hydrogen (R). How does the solution compare with the hydrochloric acid on your desk?

f. Distinguish clearly between hydrogen chloride and hydrochloric acid.

EXERCISE 27

SODIUM; SODIUM HYDROXIDE

Apparatus. Evaporating-dish with glass-plate cover; burner.

Materials. Bit of sodium, half as large as a pea; red litmus paper.

Recall experiment *a*, Exercise 8. Obtain from your instructor a bit of sodium. Cut it and note the rapidity with which the freshly cut surface is tarnished. Half fill your evaporating-dish with water; then drop the sodium into this and quickly cover the dish with a glass plate (R). Is the sodium heavier or lighter than water? After the action has ceased, place a drop of the resulting liquid on a piece of red litmus paper. Contrast its action with that of hydrochloric acid on litmus paper (Exercise 26). Mix 1 or 2 drops of the solution with 5 cc. of water and taste a drop of the resulting solution. Contrast with the taste of hydrochloric acid (Exercise 26). Evaporate the solution to dryness (?).

EXERCISE 28

THE PROPERTIES OF ACIDS, BASES, AND SALTS

Apparatus. 3 small beakers; stirring-rod; evaporating-dish: ring stand and burner; apparatus shown in Fig. 34.

Materials. A few drops of each of the following acids: hydrochloric, sulfuric, nitric, acetic (R. S.); solutions of the following bases: sodium hydroxide, potassium hydroxide (R. S.), calcium hydroxide (R. S.); strips of blue and of red litmus paper; * 10 cc. solution of hydrogen chloride in benzene.

a. Recall the properties of hydrochloric acid (Exercise 26). Prepare a dilute solution of each of the following acids by adding 1 or 2 drops of the acid to 10 cc. of water: hydrochloric, sulfuric, nitric, acetic.

By means of a clean glass rod transfer a drop of each to a piece of blue litmus paper. Note the result. In a similar way try their effect on red litmus paper. Taste *one* drop of the dilute solutions (rinse the mouth with water after tasting). Determine whether or not the solutions conduct the electric current, using the method employed in Exercise 24.

Compare the formulas of the acids. In what respect are the acids similar in composition?

b. In a similar way try the effect on red litmus paper of a solution of each of the following bases: sodium hydroxide (recall Exercise 27), potassium hydroxide, calcium hydroxide. Do they affect the blue litmus paper? Taste a drop of the calcium hydroxide solution. Determine whether or not the solutions conduct the electric current, using the method employed in Exercise 24.

Compare the formulas of the bases. In what respect are the bases similar in composition?

c. Dilute 5 cc. of the ordinary laboratory solution of sodium hydroxide (1 part of the hydroxide to 10 parts of water) with an equal volume of water. To this solution add 4 or 5 drops of hydrochloric acid (R). Stir the resulting solution with a glass rod and test its action on blue and on red litmus paper. Has it acid or basic properties?

Now continue to add the acid drop by drop until the resulting solution is neutral (that is, has no effect on either blue or red litmus paper) or is, at most, slightly acid. Pour the solution into an evaporating-dish and evaporate to dryness. What compound remains? Taste it.

What is the name given to the compounds formed by the interaction of acids and bases?

****d.*** Obtain from your instructor 10 cc. of a solution of hydrogen chloride in benzene. Does it conduct the electric current (?)? Try its action on litmus paper (?); on zinc (?). Account for the results.

e. Characterize acids and bases (1) as to composition, (2) as to their action on litmus, (3) as to taste, (4) as to their conductivity, (5) as to their interaction with each other.

EXERCISE 29

THE RATIO OF ACID TO BASE IN NEUTRALIZATION (QUANTITATIVE)

Apparatus. 2 burettes and supports, as shown in Fig. 37; small beaker and stirring-rod.

Materials. Sodium hydroxide solution prepared by adding 20 cc. of the laboratory reagent to 100 cc. water; 1 cc. sulfuric acid added to 100 cc. water; a few drops of a phenolphthalein solution (R. S.).

(Two students may work together.)

Rinse out a burette, first with distilled water and then with a little of the solution of sodium hydroxide. Support the burette (Fig. 37), and pour into it the hydroxide solution until the level of the liquid is 1 or 2 cm. above the zero mark. Turn the stopcock and let the solution flow out until the bottom of the curved surface (meniscus) of the liquid in the burette is on a level with the zero mark. In a similar way fill a second burette with the acid solution.

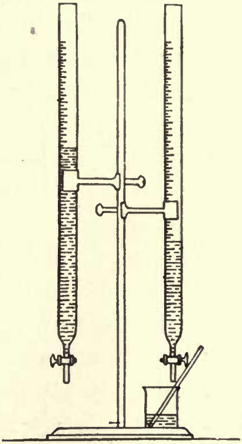


FIG. 37

Now let exactly 15 cc. of the acid solution flow into a small beaker, add two drops of phenolphthalein solution, and run in 2 or 3 cc. of the hydroxide solution. Notice that where the liquids come in contact a reddish color is produced, which disappears quickly on stirring. Run in more of the solution, a little at a time, until the color fades slowly, and then a drop at a time until the entire liquid, on stirring, remains colored *faintly* pink. This marks approximately the point of neutralization. Note the number of cubic centimeters of the hydroxide solution used.

Repeat the experiment, using different volumes of acid, say 10 cc. and 20 cc. Calculate in each case the number of cubic centimeters of the hydroxide solution required to neutralize 1 cc. of the acid solution. What do the results prove?

EXERCISE 30

THE DISPLACEMENT OF METALS FROM THEIR COMPOUNDS

Apparatus. 4 test tubes; test-tube rack.

Materials. 4 strips each of zinc and copper (1 cm. × 10 cm.); 0.5 g. lead nitrate dissolved in 10 cc. water; 0.5 g. copper nitrate dissolved in 10 cc. water; 0.5 g. mercuric nitrate dissolved in 10 cc. water; 3 cc. sulfuric acid dissolved in 10 cc. water.

a. Pour into separate test tubes to a depth of 4 or 5 cm. solutions of the following compounds: (1) lead nitrate, (2) dilute sulfuric acid, (3) copper nitrate, (4) mercuric nitrate. Set the tubes in a rack in the order given above and label them "A," "B," "C," and "D" respectively.

Now place in each tube a strip of zinc. (It is convenient to have a strong thread attached to the upper part of each strip so that the strip may easily be withdrawn from the tube.) Note any change taking place in the appearance of the zinc.

After from twenty to thirty minutes withdraw the strips and wipe them carefully on a piece of white paper. Note any evidence tending to show that the zinc has displaced the lead, hydrogen (p. 191 of text), copper, and mercury from their salts. Has the solution of copper nitrate faded in color? (Metals in a very finely divided form are black, as a rule.)

b. Repeat experiment ***a***, substituting for the zinc a strip of copper. Contrast the results obtained with those obtained in ***a***. How do you account for the change in the color of the solution of mercuric nitrate after the addition of the copper strip? Are your results in accord with the displacement-series table given on page 191 of text?

EXERCISE 31

*THE DETERMINATION OF THE AMOUNT OF HYDROGEN DISPLACED BY A DEFINITE WEIGHT OF DIFFERENT METALS (QUANTITATIVE)

Apparatus. Apparatus shown in Fig. 38 (the bottle *B* and fittings are the same as used in Exercise 12 (Fig. 25)).

Materials. About 1 g. granulated zinc; dilute sulfuric acid prepared by adding 10 cc. concentrated acid to 30 cc. water; dilute hydrochloric acid prepared by adding 20 cc. of the concentrated acid to 20 cc. water.

(Two students work together. Some should use sulfuric acid as the solvent and others hydrochloric acid, so that the results may be compared. The apparatus is not difficult to prepare and gives excellent results.)

Arrange an apparatus in accordance with Fig. 38. *A* is a 60-cc. wide-mouthed bottle. The bottle *B* and accompanying tubes are the same as shown in Fig. 25 except that the hard-glass test tube has been removed. The glass tube *G* is drawn out to a jet at the lower end, and the other end is connected to the funnel *H* by means of a rubber tube which can be closed by the screw clamp *I*. The bottles *A* and *B* are joined by a rubber tube *K*.

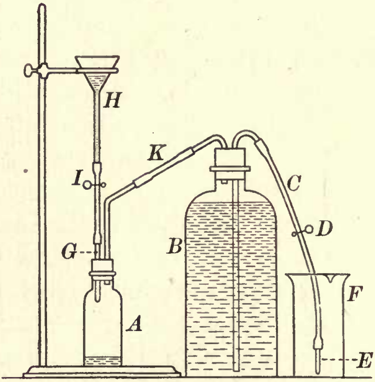


FIG. 38

Disconnect the bottles *A* and *B* at *K* and fill the bottle *B* and the exit tube *C E* with water, as in Exercise 12, and tightly close the screw clamp *D*.

Weigh out accurately about 1 g. of zinc. Wind a piece of platinum wire (used in place of copper sulfate solution) about the zinc, place it in the bottle *A*, and set the bottle aside.

Close the screw clamp *I* and nearly fill the funnel with the dilute sulfuric acid. Place a beaker under the tube *G* and open the screw clamp *I* until the rubber and the glass tubes are both completely filled with the dilute acid; then quickly close the clamp. Next, connect the apparatus just as shown in the figure, *taking care to make the apparatus air-tight.*

Now adjust the pressure of the air inside the bottles to atmospheric pressure, just as directed in Exercise 12.

Next, open the clamp *D*, then partially open the clamp *I* and allow 8 or 10 drops of the acid to flow into the bottle *A*. The hydrogen evolved forces the water from bottle *B* into the beaker. If necessary, more acid is added from time to time until the zinc is dissolved. After the zinc has all dissolved and the apparatus has acquired the room temperature, again adjust the pressure of the gas within the bottles to the atmospheric pressure and close the clamps *D* and *I*.

Insert the values in the table below:

Weight of metal taken	g.
Volume of water forced into the beaker *F* . . .	cc.
Volume of liquid left in bottle *A*	cc.
Volume of hydrogen liberated = volume of water in *F* less volume of liquid in *A*	cc.
Temperature	°
Barometric reading	mm.
Value of vapor pressure (Appendix)	mm.
Volume that the hydrogen would occupy under standard conditions	cc.
Weight of this volume of hydrogen (1 liter H = .089879)	g.
Weight of hydrogen liberated by 1 g. of zinc . .	g.

Compare the results obtained by the students using sulfuric acid as the solvent with the results obtained by using hydrochloric acid as the solvent (?).

EXERCISE 32

THE PREPARATION AND PROPERTIES OF AMMONIA

Apparatus. Test tube; hard-glass test tube fitted with cork and tubing as shown in Fig. 39; ring stand and burner; three 250-cc. wide-mouthed bottles; piece of window glass; piece of cardboard.

Materials. Solution of sodium hydroxide; 9 g. ammonium chloride; 15 g. powdered slaked lime (slaked or hydrated lime, $Ca(OH)_2$); red litmus paper; 5 cc. red litmus solution prepared by heating a bit of litmus in water and filtering.

a. Dissolve 0.5 g. of ammonium chloride in 3 or 4 cc. of water in a test tube and heat to boiling. Note the odor (?).

Now add 5 cc. of a solution of sodium hydroxide to the hot solution of ammonium chloride and continue the heating. Again note the odor (?). Moisten a strip of red litmus paper and hold it at the mouth of the tube but not in contact with it (?).

Dip the end of a glass rod in a concentrated solution of hydrochloric acid and hold it in the mouth of the test tube (?).

Complete the following equations:

$$NH_4Cl + NaOH \longrightarrow$$
$$NH_3 + H_2O \longrightarrow$$
$$NH_4OH + HCl \longrightarrow$$

b. *Usual laboratory method for preparing ammonia.* This differs from the method used in ***a*** only in the fact that the less expensive calcium hydroxide (slaked lime) is substituted for the sodium hydroxide. The form of apparatus used is shown in Fig. 39. The bottle *B* (250-cc.) contains 25 cc. of water. The glass tube *C* extends through a hole in a cardboard resting on the mouth of the bottle. The end of the tube must *just touch* the water in the bottle.

Place in the tube *A* a mixture of 15 g. of powdered slaked lime and 8 g. of ammonium chloride. Connect the tube as shown in Fig. 39 and heat the mixture gently, beginning with that portion near the mouth of the tube and gradually extending the heat to the other portions. As soon as the gas is evolved freely (as shown by the bubbles at the end of the tube *C*), bring the tube *C* to an upright position, as shown in the dotted lines, and collect two bottles of the gas by upward displacement. To do this, bring each bottle successively down over the exit tube and hold in this position until a drop of hydrochloric acid on the end of a glass rod fumes strongly when held at the mouth of the bottle (?); then withdraw the bottle, cover its mouth with a glass plate, and set it aside, mouth downward (?).

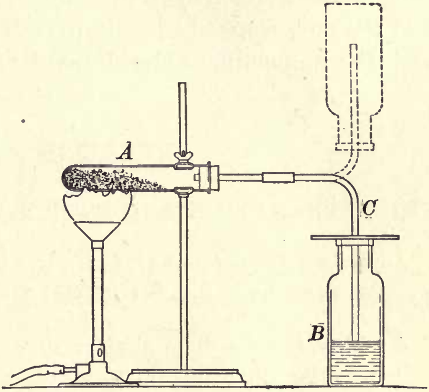


Fig. 39

When both bottles are filled, bring the tube *C* into the bottle *B* again and continue to heat the mixture gently as long as any gas is generated. Write the equations for all the reactions involved.

c. Note the color and odor (*caution*) of the gas. Is it heavier or lighter than air?

d. Test a bottle of the gas with a burning splint. Describe the results.

e. Fill a large beaker with water and color it with a few drops of red litmus solution. Uncover the remaining bottle of the gas and at once bring its mouth under the surface of the water in the beaker. Leave it in this position for five minutes, taking care to keep the mouth of the bottle below the surface of the water. What do the results prove?

f. Note the odor of the liquid in the bottle *B*. Try its effect on blue and on red litmus paper. How does it compare with the aqua ammonia of the druggist in its odor and its action on litmus? Does the gas combine with the water, or is it simply dissolved in the water? Give reasons for your answer. Now neutralize the liquid with hydrochloric acid (R) and evaporate just to dryness. Compare the residue with the ammonium chloride used in experiment ***a***.

EXERCISE 33

THE PREPARATION AND PROPERTIES OF NITRIC ACID

Apparatus. Glass retort (150-cc.), test tube, and beaker (500-cc.), arranged as shown in Fig. 40; funnel; evaporating-dish; ring stand and burner.

Materials. 12 g. sodium nitrate; 10 cc. sulfuric acid; small piece of tin; small strip of copper; pieces of ice, size of walnut.

a. Arrange an apparatus like that shown in Fig. 40. Put in the retort *A* about 12 g. of sodium nitrate and 10 cc. of sulfuric acid, pouring the latter through a funnel placed in the tubulus *B* of the retort. Heat the mixture gently with a small flame. Nitric acid is set free (R), distills over, and is condensed in the test tube *C*, which is kept cold by being partly immersed in ice water in the beaker *D*. Convince yourself that it is an acid (?).

b. When nitric acid is heated a part of it is decomposed into water, nitrogen dioxide, and oxygen (R). On this account it is a good oxidizing agent. To test its oxidizing properties, put a small piece of tin in a test tube, cover it with a little nitric acid, and gently heat (hood). The white residue formed is composed mainly of tin and oxygen, the latter being supplied by the nitric acid.

Pure nitric acid is colorless. How do you account for the color of the acid which you have prepared?

c. Place a small strip of copper in an evaporating-dish (hood) and add some of the acid you have prepared, a few drops at a time, until the copper is just dissolved. Evaporate the solution to dryness (Fig. 33). Note the appearance of the residue. Since copper is below hydrogen in the displacement series, how do you account for the fact that nitric acid dissolves the metal (study the reaction on page 212 of text)? Save the residue in the dish (what is it?) for use in the following experiment.

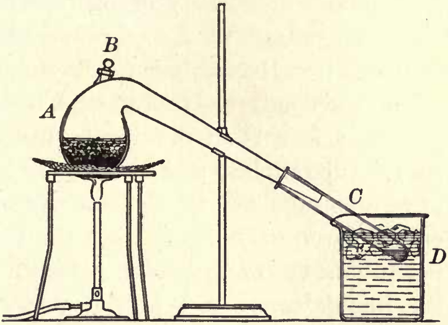


Fig. 40

EXERCISE 34

THE PROPERTIES OF THE SALTS OF NITRIC ACID (NITRATES)

Apparatus. Evaporating-dish containing the copper nitrate prepared in Exercise 33; 5 test tubes in test-tube rack.

Materials. Strip of copper; crystal of lead nitrate; crystals of such nitrates as are available; sulfuric acid; 2 g. ferrous sulfate dissolved in 10 cc. water.

a. Heat the dish (hood) containing the copper nitrate, prepared in Exercise 33, with a small flame. Note the color of the gas evolved, also the color of the residue (?). Compare the residue with a sample of copper oxide.

b. Place a crystal of lead nitrate in the evaporating-dish and heat gently. Compare with the results obtained in ***a.***

c. Place a small crystal of such nitrates as are available in your laboratory in separate test tubes and test their solubility in water. What nitrates are insoluble in water (p. 394 of text)?

d. *How to detect the presence of nitrates.* Dissolve a crystal of sodium nitrate in 2 or 3 cc. of water in a test tube, carefully add (recall experiment ***b***, Exercise 8) an equal volume of sulfuric acid, mix, and cool to room temperature or below. The sulfuric acid acts on the nitrate, liberating nitric acid. Now tip the tube slightly and gently pour 2 or 3 cc. of the solution of ferrous sulfate down the side of the tube, so that it floats on the heavier liquid, and set the tube aside, being careful not to mix the two liquids. A brown ring soon forms where the liquids meet. Repeat the experiment, using potassium nitrate. This is a good test for nitrates. The brown ring is due to the presence of a complex compound formed by the action of ferrous sulfate on nitric acid.

EXERCISE 35

THE PREPARATION AND PROPERTIES OF SOME OF THE OXIDES OF NITROGEN

Apparatus. Hard-glass test tube, with delivery tube, as used in preparing oxygen (Fig. 20); 3 wide-mouthed bottles (250-cc.); pneumatic trough; hydrogen generator (Fig. 21).

Materials. 8 g. ammonium nitrate; wooden splints; 5 small strips of copper; 10 cc. nitric acid.

a. *Nitrous oxide.* Put 6 or 8 g. of ammonium nitrate in the hard-glass test tube used in the preparation of oxygen. Attach a delivery tube and heat *gently*, applying no more heat than is necessary to cause a slow evolution of the gas.

As soon as the gas is regularly evolved, collect two or three bottles of it over water. Notice the water deposited on the sides of the test tube. What is the source of it? Note the color, odor, and taste of the gas. Test it with a glowing splint. Account for the result. How can you distinguish between nitrous oxide and oxygen?

b. *Nitric oxide and nitrogen dioxide.* Put a few pieces of copper in your hydrogen generator (hood), just cover them with water, and add 2 or 3 cc. of nitric acid. Collect over water two bottles of the evolved gas, adding more nitric acid to the liquid in the generator if necessary.

Compare the color of the gas in the generator with that collected in the bottles and account for any difference. Write the equations for all the reactions involved.

Uncover one of the bottles of the gas and account for the result (R). Test the gas in the second bottle with a burning splint. Which is the more stable, nitrous oxide or nitric oxide? Give reasons for your answer.

EXERCISE 36

SPEED OF REACTIONS; EQUILIBRIUM; HYDROLYSIS

Apparatus. 6 test tubes; test-tube rack.

Materials. Hydrochloric acid; ammonium molybdate solution (R. S.). Silver nitrate solution (R. S.); 0.1 g. sodium phosphate; crystal of sodium chloride dissolved in 5 cc. water; 0.3 g. of each of the following salts dissolved in separate portions (3 cc.) of water: potassium nitrate, common salt, borax, sodium carbonate, alum.

a. *Speed of a reaction.* Add 2 drops of hydrochloric acid to 3 cc. of water and mix the two liquids; then add 1 or 2 drops of a solution of silver nitrate. Is a precipitate formed at once? Does the precipitate appear to increase on standing?

Dissolve a crystal of sodium phosphate the size of a grain of wheat in 10 cc. of water and add 5 cc. of a solution of ammonium molybdate. Is a precipitate formed at once? Set the tube aside until the end of the laboratory period, noting its appearance from time to time. Contrast the results with those obtained by the addition of silver nitrate to hydrochloric acid.

b. Factors affecting speed of a reaction. Recall, from experiments already performed, the effect of temperature; also of concentration upon the speed of a reaction.

c. Reversible reactions. What is meant by a *reversible reaction*? The reaction used in the preparation of nitric acid (Exercise 33) is reversible. How can it be made to complete itself?

Repeat the first part of experiment *a*, substituting a solution of potassium nitrate for that of silver nitrate (?). The potassium nitrate acts upon the hydrochloric acid just as the silver nitrate does. In the former case, however, the reaction soon comes to an equilibrium while in the latter it goes to practical completion. Explain.

Recall the process of neutralization. To what extent are such reactions completed reactions?

d. Hydrolysis. Pour into separate test tubes small amounts of solutions of (1) potassium nitrate, (2) sodium chloride, (3) borax (p. 380 of text), (4) sodium carbonate (p. 403 of text), (5) alum (p. 459 of text). Test the action of each on blue and on red litmus paper, note the results, and explain.

EXERCISE 37

THE PROPERTIES OF SULFUR

Apparatus. 3 test tubes; smallest-sized beaker; magnifying glass; porcelain crucible; ring stand and burner; large beaker.

Materials. 5 cc. carbon disulfide; 20 g. powdered sulfur; strip of copper; 5 g. iron powder.

a. Examine the physical properties of a piece of brimstone. Pour 2 or 3 cc. of carbon disulfide (hood) (*keep carbon disulfide away from flame and do not inhale the vapor*) over 3 g. of powdered brimstone in a test tube. Cover the mouth of the tube with the thumb and shake the contents gently until the sulfur is dissolved, adding more carbon disulfide if necessary. Pour the clear solution into a small beaker, cover it loosely with a filter paper, and set it aside in the hood. The carbon disulfide soon evaporates, the sulfur being deposited in crystals. Examine these with a magnifying glass (?).

b. Half fill a test tube with powdered brimstone and heat it gently until the sulfur is just melted. Note the properties of the liquid.

Now apply a stronger heat and observe that the liquid becomes darker and at a certain temperature (200°–250°) is so thick that the tube may be inverted without spilling it.

Finally, increase the heat until the sulfur boils (444°), and then pour the boiling liquid into a beaker of cold water. Examine the product. What name is given to this form of sulfur? Expose it to the air for an hour. Are its properties the same as those of the original substance?

c. Fill a porcelain crucible with powdered brimstone and apply a very gentle heat until the sulfur is just melted. Withdraw the flame and examine the liquid carefully as it cools. Crystals soon begin to form on the surface of the melted sulfur, rapidly extending from the circumference toward the center. Before they reach the center, quickly pour off the remaining liquid and examine the crystals. Contrast them with those formed in ***a***. In how many forms have you obtained sulfur?

d. Burn a small piece of sulfur. Note the appearance of the burning sulfur and the odor of the gas formed (?).

e. Boil a little sulfur in the test tube used in ***b***, and drop a small strip of hot copper foil into the boiling liquid. Is there any visible evidence of a chemical change? What is formed?

Grind together some iron powder with an equal weight of sulfur and heat the mixture in a test tube (?).

EXERCISE 38

THE PREPARATION AND PROPERTIES OF HYDROGEN SULFIDE

Apparatus. Hydrogen generator and tubes, as shown in Fig. 41; 2 wide-mouthed bottles (250-cc. and 60-cc.); funnel; evaporating-dish.

Materials. 10 g. ferrous sulfide; 20 cc. hydrochloric acid added to 20 cc. water; 3 cc. nitric acid; blue and red litmus paper; silver coin; filter paper.

a. (Hood.) Attach a delivery tube to the hydrogen generator, as shown in Fig. 41. Put into the generator *A* a few pieces of ferrous sulfide (FeS) and insert the stopper. Now pour a little water through the funnel tube of the generator until the end of the tube just dips below the surface of the water; then pour in a few cc. of the hydrochloric acid, adding more from time to time, if necessary, to maintain a gentle evolution of the gas (R). The gas escapes into the bottle *B*, which gradually becomes filled. Note the odor (Caution: *the gas is poisonous if inhaled in concentrated form*) and color of the evolved gas. Continue the evolution of the gas until it is ignited by a flame held at the mouth of the bottle *B*. Account for the deposit on the sides of the bottle (?).

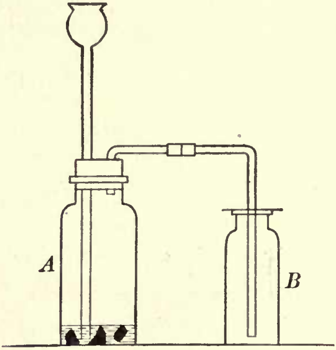


Fig. 41

b. Replace the bottle *B* with a 60-cc. bottle half filled with water, and allow the gas from the generator (add more acid if necessary) to bubble through the water for one or two minutes. Test the resulting solution with blue and with red litmus paper. What is the solution called? How does it compare with the so-called "sulfur water" of many springs?

Drop a silver coin into the solution and account for the results. Why do certain foods, as eggs, blacken silver spoons?

c. Pass a few bubbles of hydrogen sulfide through 3 cc. of nitric acid. Sulfur separates as a white solid (?). Account for the fact that sulfur waters deposit sulfur on exposure to air.

****d.*** Filter off the liquid left in the generator and evaporate (hood) to dryness in an evaporating-dish. Note the results.

EXERCISE 39

THE PREPARATION AND PROPERTIES OF THE SALTS OF HYDROSULFURIC ACID (SULFIDES)

Apparatus. Hydrogen generator and connections, as shown in Fig. 42; 6 test tubes; funnel; watch glass.

Materials. Ferrous sulfide and dilute hydrochloric acid, as used in Exercise 38; separate solutions of silver nitrate, copper sulfate, cadmium chloride, lead nitrate, and sodium chloride, made by dissolving about 0.3 g. of the solid in 5 cc. water (solutions on reagent shelf may be used); 5 filter papers; sulfuric acid; lead acetate (R. S.).

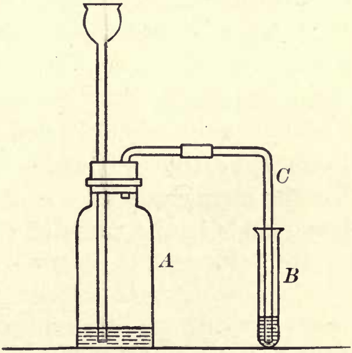


FIG. 42

a. Charge the hydrogen sulfide generator as in Exercise 38 and pass a few bubbles of the gas (Fig. 42) through each of the following solutions: (1) silver nitrate, (2) copper sulfate, (3) cadmium chloride, (4) sodium chloride, (5) lead nitrate. The exit tube *C* through which the gas bubbles into the solutions must be thoroughly cleaned each time (?). Note the color of the precipitates obtained. Write the equations for the reactions involved. Do any of the solutions fail to give a precipitate when the gas is passed through them? How do you account for this?

b. Intimately mix 5 g. of sulfur with 3 g. of powdered lime. Transfer to a beaker and add 150 cc. of water. Stir the mixture and heat just to boiling for ten minutes. Now fill a test tube with the resulting mixture, cork the tube loosely, and set it aside until the end of the laboratory period; then examine. Describe the results. For what is the solution used? What is its composition (p. 232 of text)?

c. *Test for hydrogen sulfide.* Dip a strip of filter paper into a solution of lead acetate. Remove the cork from the hydrogen sulfide generator and insert the paper for a moment. Note the results (R). This serves as a convenient test for the gas. What property would also serve to detect it if present in any marked quantity?

d. *Test for sulfides.* Filter off the precipitated sulfides obtained in ***a***, and wash them with water until the odor of hydrogen sulfide is no longer noticeable. Transfer each in succession to a watch glass and add to the solid 1 or 2 drops of sulfuric acid. Carefully note the odor. Most of the sulfides when treated in this way evolve hydrogen sulfide, which can be detected by the odor and by its action on paper moistened with lead acetate.

All sulfides when heated in air evolve sulfur dioxide (formed by the combustion of the sulfur present), which has the characteristic odor of burning sulfur. Heat a little iron sulfide in the flame of the burner and note the odor.

EXERCISE 40

SULFUR DIOXIDE AND SULFUROUS ACID

Apparatus. 250-cc. flask fitted with funnel tube and glass exit tube, as shown in Fig. 43; ring stand and burner; 3 bottles (250-cc.); 2 test tubes; evaporating-dish.

Materials. 10 g. copper; 25 cc. sulfuric acid; blue litmus paper; sodium hydroxide solution; strips of colored calico or a red flower.

a. *Preparation of sulfur dioxide and sulfurous acid.* (Hood.) Place about 10 g. of copper turnings or small pieces of sheet copper in a generator arranged as in Fig. 43. Add 25 cc. of concentrated sulfuric acid and apply a gentle heat. As soon as the action begins (R), lower the flame, regulating it so as to obtain a uniform evolution of the gas. Collect two bottles of the gas by displacement of air; then cause it to bubble through 50 cc. of water as long as any is dissolved.

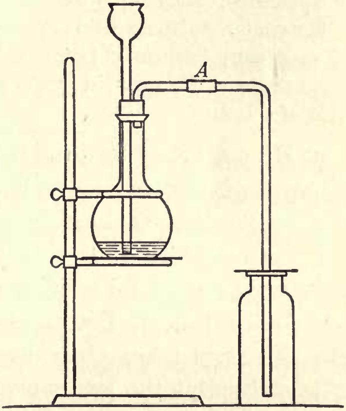


FIG. 43

b. *Properties.* Note the odor of the gas. Is the gas combustible?

Invert one of the bottles of the gas so that its mouth is under water, and examine after several minutes (?). Account for the results.

Test the saturated aqueous solution of the gas with blue litmus paper. Is the gas combined with the water or simply dissolved in it?

Set aside 10 cc. of the solution for a future experiment (Exercise 41) and divide the remainder into two equal parts.

Immerse in the one part some small strips of colored calico or some petals of a red flower and note any results.

c. *Salts of sulfurous acid — the sulfites.* To the remainder of the liquid add a solution of sodium hydroxide, drop by drop, until neutral (R), and evaporate just to dryness. What is the residue? Moisten it with 2 or 3 drops of sulfuric acid and note the odor of the evolved gas (?). *All sulfites evolve sulfur dioxide when treated with sulfuric acid.* This reaction serves as a good test for sulfites.

EXERCISE 41

A STUDY OF SULFURIC ACID

Apparatus. Burner; 5 test tubes.

Materials. Sulfuric acid; 2 pieces of granulated zinc; splint; 0.5 g. sugar; barium chloride solution (R.S.); hydrochloric acid; nitric acid; sulfurous acid prepared in Exercise 40; bit of charcoal (size of a bean).

a. Heat a bit of charcoal with 1 or 2 cc. of sulfuric acid. What gas is evolved (odor)? Account for the formation of this gas, recalling that carbon has a strong affinity for oxygen.

b. Into one test tube pour 3 cc. of water and add 5 drops of sulfuric acid; into another test tube pour 3 cc. of concentrated sulfuric acid. Drop a small piece of zinc into each tube. If no reaction takes place, heat the acid gently. Test with a lighted splint the gas evolved by the action of the dilute acid on zinc. Note the odor of the gas evolved by the action of the concentrated acid on the metal. Account for the difference in the action of the dilute and the concentrated acid.

c. Put a drop of concentrated sulfuric acid on a splint. Pour a few drops on 0.5 g. of sugar in a test tube. Examine after a few minutes. Account for the results.

d. Recall the action of sulfuric acid on sodium nitrate (Exercise 33). What property of sulfuric acid enables it to be used in the preparation of nitric acid?

e. Add 3 drops of sulfuric acid to 5 cc. of water in a test tube. To this add a few drops of a solution of barium chloride. Note that a precipitate forms (R). Now add 3 or 4 drops of hydrochloric acid. Does the precipitate dissolve? *The formation with barium chloride of a precipitate which is insoluble in hydrochloric acid* constitutes a good test for sulfuric acid.

f. Divide the solution of sulfur dioxide obtained in Exercise 40 into two portions. To the one, apply the test for sulfuric acid (?). To the other, add 1 cc. of concentrated nitric acid and heat gently nearly to boiling; then cool and apply the test for sulfuric acid (?).

EXERCISE 42

SALTS OF SULFURIC ACID (SULFATES)

Apparatus. 6 test tubes.

Materials. Crystals or small amounts (0.1 g.) of the sulfates available in the laboratory; 2 cc. barium chloride solution (R.S.); hydrochloric acid.

a. Examine the physical properties of (1) sodium sulfate, (2) calcium sulfate, (3) barium sulfate, (4) copper sulfate, (5) magnesium sulfate, (6) ferrous sulfate, (7) such other sulfates as are available.

Test the solubility of each of the above sulfates in water. What sulfates are insoluble (p. 395 of text)?

b. Prepare a dilute solution of different sulfates by dissolving a crystal of each in 2 or 3 cc. of water. Add to each 1 drop of barium chloride solution (?). Add 1 or 2 drops of hydrochloric acid to the mixture in each tube. Does the precipitate dissolve? *All soluble sulfates give in solution a white precipitate ($BaSO_4$) with barium chloride solution, which precipitate is insoluble in hydrochloric acid.* This reaction serves as a good test for sulfates.

It will be noted that both sulfuric acid and its salts give with barium chloride the same product; namely, a white precipitate of barium sulfate. This is evident from the following facts. It will be recalled (Chapter XVI of the text) that both acids and salts are ionized in solution. In the case of sulfuric acid and sulfates, ions are formed as follows:

$$H_2SO_4 \longrightarrow H^+, H^+ + SO_4^{--}$$
$$Na_2SO_4 \longrightarrow Na^+, Na^+ + SO_4^{--}$$
$$CuSO_4 \longrightarrow Cu^{++} + SO_4^{--}$$

Likewise, barium chloride solution contains the ions Ba^{++} and Cl^-, Cl^-. Now when a solution of barium chloride is mixed with any solution containing the SO_4^{--} ion, the two ions Ba^{++} and SO_4^{--} unite to form the insoluble $BaSO_4$, which precipitates; hence the reaction proceeds to completion (p. 225 of text). *The barium chloride test is therefore really a test for the presence of the* SO_4^{--} *ion.* Since only sulfuric acid and its salts give this ion, however, it is customary to say that it is a test for sulfuric acid and the sulfates.

EXERCISE 43

HYDRATES; EFFLORESCENCE

Apparatus. Burner; test tubes; porcelain crucible; ring stand; pipe-stem triangle; balance; evaporating-dish.

Materials. 2 g. zinc sulfate crystals; 6 g. copper sulfate crystals; clear crystal of sodium sulfate.

a. *Hydrates.* Heat some small crystals of zinc sulfate in a dry test tube. What evidence have you of the presence of water in the crystals? Examine the residue. How does it differ from the original crystals in form and composition?

Select some small crystals of copper sulfate. Do they appear to be dry? Fill a test tube one-fourth full of these crystals, and heat until no further apparent change takes place. Compare the residue in form, color, and composition with the original crystals. Dissolve the residue in as little hot water as possible, pour the solution (note its color) into an evaporating-dish, and set aside until crystals are deposited.

Do these appear to be identical with the original crystals of copper sulfate in shape and color?

The water evolved when hydrates are heated is sometimes called "water of crystallization." Is the term appropriate? Distinguish between the terms "hydrate," "anhydrous," "anhydride."

b. *Efflorescence.* Expose a clear crystal of sodium sulfate to the air for one or two hours. Note the change in its appearance. To what is the change due? What are such bodies called?

c. *Quantitative determination of the amount of water expelled on heating the hydrate of copper sulfate.* Accurately weigh (or counterpoise) a porcelain crucible and cover. Then add 2 or 3 g. of crystals (no larger than a pea) of the hydrate of copper sulfate and again accurately weigh. Place the covered crucible on a pipe-stem triangle and heat it with a gentle flame until the crystals lose their color. This will require from twenty to thirty minutes. The tip of the flame should not quite touch the crucible. The product is anhydrous copper sulfate. When the crucible is cool, reweigh. From your results calculate the percentage of water of crystallization present in the crystals. Compare your results with those obtained by other members of the class.

EXERCISE 44

THE PREPARATION AND PROPERTIES OF HYDROGEN FLUORIDE

Apparatus. Piece of window glass; small lead dish (laboratory outfit).

Materials. 2 or 3 small pieces of paraffin (size of a pea); 3 g. fluorite; sulfuric acid.

PRECAUTION. *Hydrogen fluoride is very corrosive and must not be inhaled; neither must its solution be brought in contact with the skin.*

Place some pieces of paraffin on a glass plate and gently warm over a small flame. When the paraffin is melted, tilt the plate about so as to form a uniform layer of the wax. When the wax is cold, scratch your name *through* the wax with a pin. Place 3 g. of fluorite in a lead dish and add sufficient sulfuric acid to make a paste of it. Cover the dish tightly with the waxed side of the glass plate and set it in the hood for an hour; then scrape off the paraffin and examine the glass. Write the equations for all the reactions involved.

EXERCISE 45

THE TEST FOR HYDROCHLORIC ACID AND ITS SALTS (CHLORIDES)

Apparatus. 6 test tubes.

Materials. 2 cc. silver nitrate solution (R. S.); hydrochloric acid; nitric acid; ammonium hydroxide; crystals of different chlorides, such as those of sodium, calcium, and iron.

NOTE. Experiments on chlorine and hydrochloric acid were included under Exercises 25 and 26. The student should carefully review the results obtained, since they have an important bearing upon the experiments now to be performed on the remaining members of the Chlorine Family.

a. Add 4 or 5 drops of hydrochloric acid to 4 cc. of water, mix well, and add 2 or 3 drops of silver nitrate solution (?).

Divide the resulting mixture into two equal parts: to the one add ammonium hydroxide until the liquid is alkaline (?); to the other add 2 or 3 drops of nitric acid (?).

b. Examine the physical properties of such chlorides as are available. Test their solubility in water. What ones are insoluble (p. 394 of text)? Dissolve a small crystal of different chlorides each in 5 cc. of water and add silver nitrate solution as in ***a*** (?). *The formation of a white precipitate ($AgCl$) with silver nitrate, which precipitate is soluble in ammonium hydroxide and insoluble in nitric acid, serves as a good test for hydrochloric acid and its salts.*

How do you account for the fact that both hydrochloric acid and its salts react toward silver nitrate in the same way?

EXERCISE 46

THE PREPARATION AND PROPERTIES OF BROMINE AND OF HYDROGEN BROMIDE

Apparatus. Retort, test tube and beaker, as shown in Fig. 40; burner; funnel; 2 test tubes.

Materials. 3 g. potassium bromide or sodium bromide; 4 g. manganese dioxide; 10 cc. sulfuric acid dissolved in 40 cc. water; strips of colored calico; 1 cc. silver nitrate solution (R.S.).

PRECAUTION. *The vapor of bromine must not be inhaled. Perform the experiments in a hood.*

a. Put into the retort a mixture of 2 g. of potassium bromide or of sodium bromide and 4 g. of manganese dioxide, and add to this through a funnel a cold dilute solution of sulfuric acid. Shake the retort so as to mix the contents thoroughly. The test-tube receiver should contain sufficient water to allow the end of the retort to dip just below its surface.

Now heat the retort gently. The bromine is liberated and distills over (R). Continue the heating until all the bromine has distilled over. Remove the stopper from the retort before the heat is withdrawn.

b. Note the properties of the bromine collected in the bottom of the receiver. Has any dissolved in the water? What property is implied in the name of the element?

Test the bleaching property of the aqueous solution. How does it compare with chlorine as a bleaching agent?

c. Add 3 or 4 drops of sulfuric acid to about 1 g. of potassium bromide in a test tube. Some hydrogen bromide is evolved, which attracts moisture as it escapes from the tube, forming a light cloud (test the vapor with a moist piece of blue litmus paper). At the same time there appears in the tube a reddish vapor. Explain (p. 270 of text). Distinguish between hydrogen bromide and hydrobromic acid.

d. Hydrobromic acid is unstable and is but little used. Its salts (bromides) are stable. Study the properties of potassium bromide as well as of any other available bromides.

Dissolve a crystal of potassium bromide in 5 cc. of water and apply the silver nitrate test as outlined for testing for chlorides in Exercise 45 (?). Would this test alone serve to distinguish between chlorides and bromides?

EXERCISE 47

THE PREPARATION AND PROPERTIES OF IODINE AND OF HYDROGEN IODIDE

Apparatus. Retort and connections, as shown in Fig. 40; burner.

Materials. 4 g. potassium iodide (or sodium iodide); 4 g. manganese dioxide; sulfuric acid; chlorine water (R.S.); silver nitrate solution (R.S.); starch solution (R.S.); alcohol (R.S.).

a. Introduce into the retort a mixture of 2 g. of potassium iodide and 4 g. of manganese dioxide. Pour over this mixture 5 cc. of sulfuric acid. Insert the stopper and apply a gentle heat (R). Note the vapor of the iodine in the bulb of the retort; also note the grayish-black crystals, which are soon deposited in the neck of the retort. What property does the name of the element suggest?

b. Half fill two test tubes with starch solution. To the first add a few drops of a solution of iodine prepared by shaking a small crystal (obtained in ***a***) in water (?). To the second add a few drops of an aqueous solution of potassium iodide (?). Now add to the second tube 2 or 3 drops of chlorine water and mix the contents (?). Determine whether the chlorine water alone changes the color of the starch. What is the function of the chlorine water added to the second tube? What other substance studied might be substituted for the chlorine in this experiment? (See Exercise 15.)

Dissolve a crystal of iodine in alcohol. What is the solution called?

c. Hydrogen iodide is still less stable than hydrogen bromide. What reaction would you expect to take place when concentrated sulfuric acid is added to a crystal of potassium iodide? (Compare **c**, Exercise 46.) Try the reaction and note any evidences in favor of your opinion.

d. *The salts of hydriodic acid; the iodides.* While hydriodic acid is unstable, its salts are stable. Study the properties of potassium iodide. Prepare a solution of the salt and study the action upon it of silver nitrate, as in the case of chlorides and bromides.

EXERCISE 48

HOW TO DISTINGUISH BETWEEN CHLORIDES, BROMIDES, AND IODIDES

Apparatus. 3 test tubes.

Materials. 0.2 g. each of the chloride, the bromide, and the iodide of either potassium or sodium, dissolved separately in 5 cc. water; 5 cc. carbon tetrachloride; chlorine water (R. S.).

a. Recall the action of silver nitrate solution upon solutions of chlorides, bromides, and iodides (?).

b. Pour the solutions of the chloride, of the bromide, and of the iodide of potassium (or sodium) into separate test tubes and set the tubes in a rack in the order given.

Add to each solution 1 cc. of carbon tetrachloride (carbon disulfide will do as well but is inflammable). Shake the contents of the tubes and set them aside for two or three minutes (?). Add to each tube 2 or 3 cc. of chlorine water and shake again the contents of the tubes vigorously and set aside for a few minutes (?). Explain the action of the chlorine water.

The silver nitrate test will enable you to tell whether or not a given compound belongs to the group of chlorides, bromides, and iodides. The test with chlorine water and carbon tetrachloride will enable you to distinguish the three members of the group from one another.

EXERCISE 49

THE PREPARATION AND PROPERTIES OF CARBON MONOXIDE

Apparatus. The apparatus shown in Fig. 44: *A* is a funnel, connected by a rubber tube with a piece of glass tubing which passes through a stopper into the 250-cc. flask *B*; *C* is a small clamp; pneumatic trough; 3 wide-mouthed bottles; 3 glass plates 10 cm. square; burner; ring stand. Apparatus shown in Fig. 45: *A* and *B* are glass tubes connected by rubber tubing; *C* is a wide-mouthed bottle.

Materials. Concentrated sulfuric acid; 25 cc. formic acid (50%); limewater (R.S.); 1 g. black copper oxide powder.

PRECAUTIONS. *Carbon monoxide is a nearly odorless and very poisonous gas. All of the experiments must be performed in the hood. After the gas is generated, pour the contents of the generator flask into a sink or jar in the hood.*

a. Remove the stopper from the flask *B* (Fig. 44), pour in 15 cc. of sulfuric acid and connect the apparatus as shown in the figure. Close the clamp *C* and partially fill the funnel *A* with the formic acid. Now open the clamp carefully so that the formic acid will enter the flask, a drop at a time. Allow 8 or 10 drops to flow in; then close the clamp. If the reaction does not begin (as indicated by absence of effervescence of the liquid in the flask and escape of gas through the exit tube), heat the flask very gently until the reaction starts; then open the clamp again and admit the formic

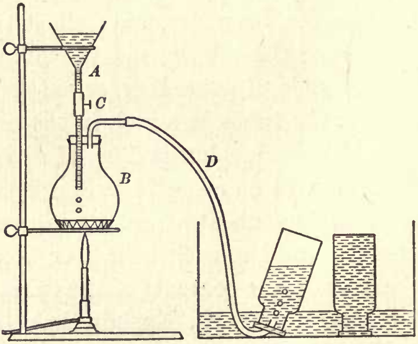


FIG. 44

acid, a drop at a time, so as to secure a regular flow of gas from the flask. If necessary, add more formic acid to the funnel so as to keep it partially filled (?). Collect three bottles of the gas as shown in the figure. Close the clamp so as to stop further generation of gas. Slip the glass plates over the mouths of the bottles and remove the bottles from the trough. In the first bottle filled, is the gas pure carbon monoxide? Remove the glass cover and test it with a flame (?). Repeat with the second bottle filled (?). Slip the glass plate from the third bottle just far enough to pour into the bottle 5 cc. of clear limewater; then quickly replace the glass plate and, holding it firmly against the mouth of the bottle, shake the contents of the bottle. Note any change in the appearance of the limewater. Now tip the bottle as far as possible without spilling the limewater; remove the glass plate and quickly ignite the gas, holding the bottle in this position so that at least a portion of the combustion product may be retained in the bottle. When the flame dies out, at once cover the mouth of the bottle with the glass plate and shake the contents. Note the results.

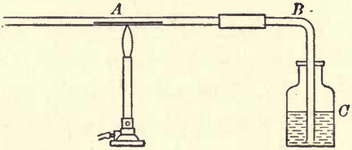


FIG. 45

b. Introduce into the tube *A*, Fig. 45, a small amount of copper oxide and arrange the apparatus as shown in the figure. Now connect the exit tube *D* (Fig. 44) with the tube *A* (Fig. 45). Heat the copper oxide gently; at the same time pass a slow current of carbon monoxide through the tube, generating the gas as under ***a***. Continue until the limewater and copper oxide both have visibly changed. Describe the results and write the equations for all the reactions involved.

EXERCISE 50

CARBONIC ACID AND ITS SALTS (CARBONATES)

Apparatus. Hydrogen generator, as used for preparing carbon dioxide in Exercise 19; small beaker; 5 test tubes.

Materials. Pieces of marble for generating carbon dioxide; hydrochloric acid; blue litmus paper; 5 cc. sodium hydroxide solution diluted with 10 cc. water; 1 g. of the common carbonates, such as sodium carbonate, magnesium carbonate, calcium carbonate; 25 cc. limewater (R. S.).

a. Generate carbon dioxide and pass the gas through 25 cc. of water. The gas combines with the water to form carbonic acid (R). Taste the liquid. Is the acid formed strong enough to affect blue litmus paper?

b. Pass carbon dioxide through a solution of sodium hydroxide until the gas is no longer absorbed; then evaporate the solution to dryness. Explain. Could a solution of sodium hydroxide be used in place of a solution of calcium hydroxide (limewater) in testing for carbon dioxide?

c. Examine the physical properties of such carbonates as are available. What ones are soluble in water (p. 395 of text)? Test the action of hydrochloric acid or sulfuric acid on each by adding 1 or 2 drops of the acid to 0.1 g. of the carbonate on a watch glass. What evidences have you that a gas is evolved? Arrange a simple apparatus to determine whether or not the gas evolved is carbon dioxide, then test one or more of the carbonates.

All carbonates when treated with hydrochloric acid or sulfuric acid evolve carbon dioxide. This reaction serves as a good test for carbonates.

Can you suggest any reason why carbon dioxide is so readily liberated from carbonates?

EXERCISE 51

A STUDY OF SOME OF THE HYDROCARBONS

Apparatus. Evaporating-dish; burner; hard-glass tube fitted with a cork and a delivery tube, as shown in Fig. 20; two 250-cc. bottles; pneumatic trough; watch glass; beaker; stirring-rod; test tubes.

Materials. 15 g. soda lime (mixture of sodium hydroxide and calcium hydroxide); 10 g. fused sodium acetate; iron wire; bit of calcium carbide (size of a bean); sulfuric acid; sodium hydroxide; wooden splints; 10 cc. each of low-boiling gasoline and of kerosene; 1 g. paraffin; 1 cc. of cottonseed oil.

* ***a.*** *Methane.* Intimately mix, by grinding together in a mortar, 15 g. of soda lime and 10 g. of fused sodium acetate. Transfer the mixture to the hard-glass tube used in preparing oxygen and proceed just as in the preparation of oxygen, except that the tube should be clamped in a horizontal position while being heated. Collect over water one or two bottles of the evolved gas. Note the color and odor of the gas. Is it inflammable?

Sodium acetate has the formula $NaC_2H_3O_2$. When heated with the sodium hydroxide, methane is generated according to the following reaction:

$$NaC_2H_3O_2 + NaOH \longrightarrow Na_2CO_3 + CH_4$$

b. *Acetylene.* Nearly fill a test tube with water and drop into it a small piece of calcium carbide. Note the gas evolved (R). Ascertain by holding a lighted splint at the mouth of the tube whether the gas is inflammable.

c. Obtain from the instructor a few drops each of gasoline and of kerosene. Test the inflammability of each by dipping the end of a glass rod in the liquid and then touching it to the tip of a flame. Place ten drops of gasoline (PRECAUTION: *keep away from all flames*) on a watch glass (hood) and set the glass on a beaker half filled with boiling water.

Note the time required for its complete evaporation. Repeat the experiment, using 10 drops of kerosene. Which of the two substances is the more volatile?

d. Pour 2 or 3 drops of gasoline into a warm 250-cc. wide-mouthed bottle. Cork the bottle and shake it vigorously. Now remove the cork and, standing at arm's length, bring a lighted splint to the mouth of the bottle (?). What use is suggested by the property noted in this experiment?

e. Compare benzene with low-boiling gasoline (benzine) in odor, solubility in water (test by adding 3 drops to 5 cc. of water and shaking), and inflammability. Are both good solvents for fats (test with 3 or 4 drops of cottonseed oil)? What advantage has carbon tetrachloride over benzene and benzine as a fat solvent?

f. Study the properties of paraffin. Is it soluble in water? in kerosene? Will it melt at the temperature of boiling water? Will it burn? Test the action of acids and alkalies on paraffin by adding a few drops of each to a bit of the paraffin on a watch glass. What properties are suggested by the term "paraffin" (see derivation of word)?

EXERCISE 52

A STUDY OF THE FLAME

Apparatus. Burner; wire gauze; porcelain dish; glass tube 15 cm. long.

Materials. Charcoal (size of a bean); 5 cc. limewater (R. S.); candle; wooden splint.

a. Note and account for the difference between the combustion of a wooden splint and that of a piece of charcoal. What are the conditions necessary for the production of a flame? Light a candle and place it so that the flame is against a black background and is not disturbed by air drafts; then note the different cones in the flame. Test the relative temperatures of different parts of the flame by means of narrow strips of splints. Draw a diagram showing the different parts of the flame. Extinguish the candle flame and hold a lighted splint 2 or 3 cm. from the wick in the little column of smoke (?).

b. What two elements constitute the main parts of ordinary fuels? What products form when these elements burn in air or oxygen? Devise simple experiments to show the presence of these products in the gases evolved by the burning candle.

c. What is meant by the kindling temperature of gases? When a lamp is first lighted a film of liquid often spreads over the chimney for an instant. Explain. Press a piece of wire gauze halfway down on a Bunsen flame. Notice that the flame does not extend above the gauze. Is this due to the absence there of combustible gases? Test for their presence by means of a lighted splint.

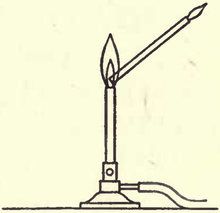


FIG. 46

Turn off the gas, then turn it on and ignite it over a piece of wire gauze held horizontally 4 or 5 cm. above the top of the burner. Note the results and explain. How does the miner's safety lamp prevent explosions?

Hold a porcelain dish in a small luminous Bunsen flame. Account for the deposition of carbon (lampblack). Does the nonluminous flame deposit carbon? To what is the luminosity of the flame due?

d. Recall the experiment on the Bunsen flame in Exercise 2.

That the center of the base of the Bunsen flame contains the unburned gas may be shown by holding in it the end of an inclined glass tube (Fig. 46) and igniting the gas at the upper end of the tube.

EXERCISE 53

THE SUGARS

Apparatus. 3 test tubes; 2 small beakers; stirring-rod; burner.

Materials. 3.5 g. copper sulfate crystals dissolved in 50 cc. water (label this solution "A"); 17.5 g. sodium potassium tartrate (Rochelle salts) dissolved in 50 cc. sodium hydroxide solution (label this solution "B"; solutions A and B should be poured into bottles and reserved for future exercises); 5 cc. commercial glucose or Karo corn sirup; 2 g. sucrose; 1 g. each of sweets such as candy, honey, molasses; 2 or 3 g. sodium carbonate dissolved in as little water as possible; red litmus paper; hydrochloric acid.

a. *The test for dextrose.* The most common test for dextrose is the reaction with Fehling's solution. This is prepared as needed by mixing equal volumes of solutions A and B, prepared as directed above.

Pour into a test tube about 3 cc. each of solutions A and B. When thoroughly mixed, the resulting solution should be deep blue, but perfectly clear. Heat the blue solution nearly to boiling, add 1 or 2 drops of commercial glucose (Karo corn sirup will do as well), and continue the heating for a few moments. The copper sulfate in the solution is reduced to cuprous oxide by the dextrose, and this separates in the form of a red or yellow solid. Levulose will act in the same way. Dissolve samples of candy, honey, and molasses in a little water and test for the presence of dextrose and levulose in these sweets.

b. *The action of cane sugar on Fehling's solution.* In a similar way try the action of pure cane sugar on Fehling's solution (?).

Now dissolve about 1 g. of the sugar in 10 cc. of water. Add 4 or 5 drops of concentrated hydrochloric acid and slowly heat nearly to boiling. Set the solution aside for about five minutes, then cool and neutralize the acid present by adding a concentrated solution of sodium carbonate until the resulting mixture is just alkaline to litmus paper. Now test this with Fehling's solution as in ***a***. Account for the result.

EXERCISE 54

A STUDY OF STARCH

(Students interested in the subject of foods should perform the additional exercises included under Appendix A.)

Apparatus. Microscope; 200-cc. beaker; stirring-rod; ring stand and burner; 3 test tubes.

Materials. Iodine solution prepared by dissolving 0.5 g. iodine and 2.5 g. potassium iodide in 25 cc. water (label this "Iodine Solution" and preserve for use in a number of exercises); 0.1 g. flour; 10 g. starch; piece of bread; hydrochloric acid; 3 g. sodium carbonate dissolved in a little water; red litmus paper; starch solution (R. S.); Fehling's solution.

a. *Microscopic appearance.* Examine under the microscope the appearance of starch from different sources (corn, wheat) when magnified. Draw diagrams of the starch granules.

b. *Actions of acids on starch.* Try the action of starch solution on Fehling's solution (as in Exercise 53) (?).

Add 2 cc. of hydrochloric acid to 50 cc. of starch solution in a beaker and boil the contents gently for thirty minutes, allowing the solution to concentrate to about 25 cc. Cool the liquid, neutralize with sodium carbonate, and again test the solution with Fehling's solution (?).

c. *Test for starch.* Recall the action of iodine on starch (Exercise 47). This constitutes a good test for starch.

Test different foods (such as bread, potatoes, and corn meal) for starch. To do this, boil from 5 to 10 g. of the food with 100 cc. of water, stirring the mass thoroughly so as to break it into small pieces; then filter it and cool the filtrate. Now stir the filtrate with a glass rod, the end of which is first dipped into a solution of iodine (?).

***d.** *The action of heat on starch.* Place 3 or 4 g. of starch in a test tube and heat *slightly* for ten or fifteen minutes, regulating the heat so as not to burn the starch (the same results may be obtained by heating a piece of bread in an oven until it is dry and crisp). How does the product differ in taste from the original starch? The heat changes a part of the starch into an isomeric compound known as *dextrin*. This is sweet and dissolves in water, forming a mucilage-like solution which is used on the back of postage stamps and for other similar purposes.

EXERCISE 55

THE PREPARATION AND PROPERTIES OF COMMON ALCOHOL (ETHYL ALCOHOL)

Apparatus. One 2000-cc. flask (or bottle) connected with tube and bottle, as shown in Fig. 47; test tube; stirring-rod; apparatus shown in Fig. 26; evaporating-dish.

Materials. 200 g. molasses or Karo corn sirup; cake of yeast; 50 cc. limewater (R. S.); 25 cc. alcohol; 15 cc. methyl alcohol; small amounts (size of a pea) of sugar, starch, salt; 1 cc. cottonseed oil; iodine solution prepared in Exercise 54; sodium hydroxide solution; soda lime sufficient to fill tube *C* (Fig. 47).

a. *Preparation of alcohol.* (It is suggested that this experiment be performed by the instructor or by students selected by the instructor; after the alcohol is generated the liquid may be divided among the different members of the class who will then test for the alcohol as directed in ***d.***)

Dissolve about 200 g. of ordinary molasses in 2000 cc. of water in the flask *A* (Fig. 47). Grind a cake of yeast with a little water and add it to the solution in *A*. Connect the flask as shown in the figure (the bottle *B* contains limewater and the tube *C* contains soda lime). Set the apparatus aside in a warm place (30° is best) for one or two days. Note that a gas is evolved in *A* and bubbles through the limewater in *B* (?). Examine a drop of the mixture in *A* under the microscopes for yeast cells (see Fig. 135 of text).

b. *Properties of alcohol.* Pour a few drops of alcohol into an evaporating-dish, ignite, and note the characteristics of the flame.

Determine whether alcohol is a good solvent for (1) sugar; (2) salt; (3) starch; (4) oils, such as cottonseed oil (?). Does alcohol mix with water in all proportions? Repeat these experiments, using methyl alcohol (?).

c. *Test for alcohol.* Pour 2 cc. of alcohol into a test tube and add to this 5 cc. of iodine solution. Now add a solution of sodium hydroxide, *one* drop at a time (mix after the addition of each drop), until the iodine color vanishes; then warm gently and set aside for a few minutes. A yellow precipitate of *iodoform* (p. 302 of text), of characteristic odor, forms. (If the amount of alcohol present is small, the iodoform may not separate, but its presence will be revealed by its odor.)

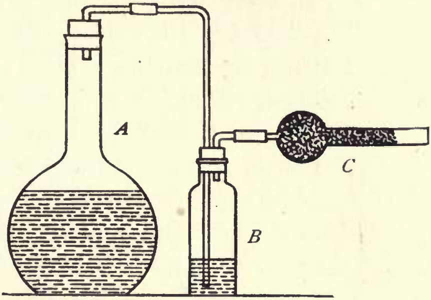


FIG. 47

d. After the molasses in experiment ***a*** has fermented divide the liquid in *A* (Fig. 47) so that each student or group of students will have from 150 to 200 cc. Pour the liquid into a flask and distill over 3 or 4 cc. (Exercise 13). Dip the end of a glass rod in the distillate and touch it to the edge of a flame (?). Test the remainder of the distillate for alcohol as in ***c***, above.

EXERCISE 56

THE PREPARATION OF A SIMPLE ESTER

Apparatus. 250-cc. flask; ring stand and burner; test tube.
Materials. Acetic acid (R.S.); alcohol (R.S.); sulfuric acid.

Preparation of ethyl acetate. What is an ester? Ethyl acetate is an ester derived from acetic acid by replacing an atom of hydrogen in the acid by the univalent radical *ethyl* (C_2H_5) and has the formula $C_2H_5 \cdot C_2H_3O_2$. It is a colorless liquid boiling at 78° and having a characteristic fragrant odor.

Pour into a small flask 10 cc. of acetic acid (or of a saturated solution of sodium acetate), 5 cc. of sulfuric acid, and 3 cc. of alcohol. Mix well and heat slightly. Ethyl acetate is formed and may be recognized by its odor. Do not mistake the odor of the vapor of alcohol for that of ethyl acetate (warm a little alcohol in a test tube and note the difference between its odor and that of ethyl acetate). The equation for the reaction is

$$H \cdot C_2H_3O_2 + C_2H_5OH \longrightarrow C_2H_5 \cdot C_2H_3O_2 + H_2O$$

The sulfuric acid is added to absorb the water formed.

This reaction serves as a test for both acetic acid and acetates.

EXERCISE 57

PHOSPHORUS AND ITS COMPOUNDS

Apparatus. 250-cc. wide-mouthed bottle; deflagrating-spoon; glass plate; small beaker; porcelain crucible; burner.

Materials. 0.5 cc. phosphorus trichloride; phosphorus (size of a pea); litmus paper (red and blue); 10 cc. ammonium molybdate solution (R.S.); ammonium hydroxide; nitric acid; silver nitrate solution (R.S.); 2 g. disodium phosphate.

a. Pour into a test tube about 0.5 cc. of phosphorus trichloride (hood) and add a little water, a drop at a time. Mix the liquids by shaking the tube. What gas is evolved (R)? Finally add about 5 cc. of water, pour the liquid into an evaporating-dish, and evaporate to a sirupy mass. Dilute this with a little water; transfer the solution to a test tube and add a few drops of a solution of silver nitrate. Boil the resulting mixture; finely divided metallic silver (black) precipitates. Explain.

b. Cover the bottom of a wide-mouthed bottle (250-cc.) with water to a depth of about 1 cm. Place a piece of phosphorus on a deflagrating-spoon and ignite it by touching it with a hot wire. Quickly lower the phosphorus into the bottle and cover the mouth of the bottle with a glass plate. When the phosphorus ceases to burn, withdraw the spoon and allow the fumes in the bottle to dissolve in the water. Test the solution with litmus paper. What is present in the water?

Pour the solution into a small beaker, add 2 or 3 cc. of nitric acid, and boil the solution until about half of it evaporates. The nitric acid oxidizes to phosphoric acid all the phosphorus compounds present. Add a few drops of the solution to 10 cc. of a solution of ammonium molybdate and warm gently. Note the result (the compound formed has a very complex composition). Add ammonium hydroxide to the mixture until the liquid is alkaline. Note the result. Again acidify the liquid with nitric acid. Note the result. *The formation of a yellow precipitate upon the addition of ammonium molybdate, which precipitate is insoluble in nitric acid and soluble in ammonium hydroxide, serves as a good test for phosphoric acid and its salts.*

c. Apply a gentle heat to 1 or 2 g. of disodium phosphate placed in a porcelain crucible. Gradually increase the heat to the full extent and continue the heating for about 10 minutes. When cool, dissolve the product in water and test the solution with silver nitrate solution. Compare with the product obtained by adding silver nitrate to disodium phosphate which has not been heated. Note the results. (The silver salts of the acids present are formed (pp. 353, 354 of text).)

EXERCISE 58

ARSENIC AND SOME OF ITS COMPOUNDS

Apparatus. Blowpipe; piece of charcoal (2 cm. × 8 cm.); hard-glass tube (10 cm. long and 6 or 7 mm. wide); file; burner.

Materials. Arsenic (size of a grain of wheat); 0.1 g. arsenious oxide.

a. Note the physical properties of arsenic. Place a bit of the arsenic in a cavity on a piece of charcoal and gently heat it (hood), using a blowpipe. Note the peculiar, garlic-like odor (poisonous).

b. Introduce into a hard-glass tube an amount of arsenious oxide equal in bulk to a grain of wheat. Cover this to a depth of 2 or 3 cm. with somewhat finely powdered charcoal which has been previously heated to a high temperature in a porcelain crucible. See that the inner surface of the tube above the charcoal is perfectly clean.

Incline the tube and heat the *upper* portion of the charcoal to a high temperature, then, while maintaining the charcoal at this temperature, gradually bring the lower part of the tube also into the flame. The upper part of the tube must be kept as cool as possible. The arsenious oxide is changed into a vapor, which passes over the hot charcoal. Account for the result (R).

Cut the tube as near the bottom as possible and remove the charcoal; then, inclining the tube, apply a very *gentle* heat to that portion of it which contains the coating. Note that small white crystals are slowly deposited in the colder portions of the tube (R). Examine these with a magnifying glass. Note their form.

* ***c.*** *Marsh's test — perform in hood, as the arsine formed is poisonous.* Arrange an apparatus according to Fig. 22, substituting for the tube *D* a clean, hard-glass tube about 30 cm. long and 8 mm. in diameter, drawn out to a jet at the end. (Use the blast lamp in making the jet.) Generate hydrogen by the usual method, and, *after taking the general precautions,* ignite it as it escapes from the glass jet. Sufficient acid is added from time to time to cause a gentle evolution of the gas. Now apply a strong heat to the hard-glass tube at a place near its center, using the "wing-top" burner. After a few minutes note whether any deposit forms just beyond the heated portion of the tube. If none forms, the materials are free from arsenic. Now add two drops of a dilute hydrochloric acid solution of arsenious oxide (made by dissolving a bit of the oxide no larger than a pinhead in 1 cc. of the dilute acid) to the generator, rinsing it down the funnel tube with a little water. Continue the heating of the hard-glass tube at the same place. Note the deposit formed on the sides of the tube. Withdraw the heat and hold the lid of a porcelain crucible in the flame. A black deposit of arsenic forms. Cut the tube containing the deposit so as just to remove the jet and, inclining it, apply a *gentle* heat, as in ***b.*** Account for the results and write the equations for all the reactions involved in the experiment.

EXERCISE 59

A STUDY OF SOME OF THE PROPERTIES OF ANTIMONY

Apparatus. Blowpipe; beaker; stirring-rod; hydrogen sulfide generator (Fig. 42).

Materials. Piece of charcoal (2 cm. × 8 cm.); 2 pieces of antimony (size of a grain of wheat); hydrochloric acid; nitric acid; strip of zinc; 10 g. ferrous sulfide.

a. Heat a bit of antimony on charcoal as in ***a,*** Exercise 58. The product is Sb_2O_3.

b. Introduce into a test tube a bit of antimony no larger than a grain of wheat and add about 3 cc. of hydrochloric acid and then 2 or 3 drops of nitric acid (?). After the metal has dissolved, pour the solution into 50 cc. of water. If a precipitate forms, add hydrochloric acid, drop by drop, with constant stirring, until the precipitate dissolves. Half fill a test tube with the resulting solution and insert a strip of zinc. Note the results (compare Exercise 30). Through the remainder of the solution pass a few bubbles of hydrogen sulfide. The product is Sb_2S_3. Note its properties.

EXERCISE 60

A STUDY OF SOME OF THE PROPERTIES OF BISMUTH

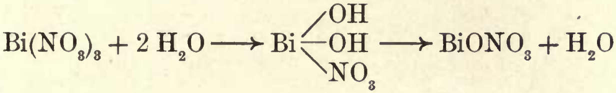
Apparatus. Blowpipe; beaker; stirring-rod; hydrogen sulfide generator (Fig. 42).

Materials. 2 pieces of bismuth (size of a grain of wheat); piece of charcoal (2 cm. × 8 cm.); nitric acid; 10 g. ferrous sulfide; hydrochloric acid.

a. Heat a piece of bismuth on charcoal, as in the case of antimony (Exercise 59). Bi_2O_3 is formed and is deposited on the charcoal. Contrast the effect of heating arsenic, antimony, and bismuth in air.

b. Repeat *b*, Exercise 59, substituting bismuth for antimony and using nitric acid alone as the solvent. The precipitate formed by hydrogen sulfide is Bi_2S_3. Note its properties.

$Bi(NO_3)_3$ is formed when bismuth is dissolved in nitric acid. Upon pouring this into water a reaction takes place in accordance with the following equation:



$$Bi(NO_3)_3 + 2\,H_2O \longrightarrow Bi\begin{array}{l} \diagup OH \\ -OH \\ \diagdown NO_3 \end{array} \longrightarrow BiONO_3 + H_2O$$

The bismuth subnitrate ($BiONO_3$) is insoluble and separates as a white solid. Upon the addition of nitric acid, a drop at a time, the reaction is reversed, $Bi(NO_3)_3$ being formed, which dissolves in the liquid.

c. Define the terms *hydrolysis, basic salt*, and *reversible reaction*, and illustrate with examples from this exercise.

EXERCISE 61

COMPOUNDS OF SILICON

Apparatus. Evaporating-dish; ring stand; burner.

Materials. 2 cc. water glass (solution of Na_2SiO_3); hydrochloric acid.

a. Recall the formulas and names of the important acids of silicon.

b. Place 2 cc. of a solution of water glass (Na_2SiO_3) in an evaporating-dish, dilute with 10 cc. of water, and add 2 or 3 cc. of hydrochloric acid. Note the gelatinous precipitate (R). Evaporate to dryness and heat the dish gently with the bare flame (?). When cool, add water, filter, and examine the residue. What is it?

c. Recall the action of hydrofluoric acid on silica (R).

EXERCISE 62

COMPOUNDS OF BORON

Apparatus. Platinum wire (piece 10 cm. long, fused in glass tube for handle (Fig. 165, p. 414 of text)); burner; beaker; porcelain crucible; stirring-rod.

Materials. 8 g. borax; litmus paper (red and blue); sulfuric acid; alcohol (R.S.); 1 or 2 drops cobalt nitrate solution (R.S.).

a. Write the names and formulas for three important compounds of boron.

b. Make a little loop on the end of a platinum wire and heat it to redness in a Bunsen flame, then quickly bring the loop in contact with some borax and reheat. The borax adhering to the loop will swell up (owing to the expulsion of water from the hydrate) and finally form a clear, glassy bead. Note the color imparted to the flame.

Moisten the bead with a drop of sulfuric acid and again touch it to the edge of the flame. Note the result (see ***c***, below, for explanation). This serves as a simple test for borax.

Moisten the bead with a drop of a solution of a cobalt compound and reheat until the bead is transparent when cold. Note the color of the bead now. This property serves as a simple test for cobalt. (Some of the other metals likewise impart characteristic colors to the bead.)

c. Dissolve 5 g. of borax in 15 cc. of boiling water. Test the solution with litmus paper. Explain. Carefully add to the hot solution 2 or 3 cc. of sulfuric acid and stir. Cool the solution and filter off the precipitate. Compare the precipitate with borax, first, as to solubility in alcohol, and, second, as to the color imparted to a flame when a small portion on a platinum wire is held in the flame.

d. Dry a little of the boric acid prepared in ***c***, transfer to a porcelain crucible, and heat until a clear liquid is formed (?).

EXERCISE 63

COLLOIDS AND EMULSIONS

Apparatus. 6 test tubes; 2 beakers; hydrogen sulfide generator; stirring-rod; tripod and burner.

Materials. 0.5 g. flowers of sulfur; alcohol (R.S.); 1% solution of tartar-emetic (R.S.); ferrous sulfide and hydrochloric acid for preparing hydrogen sulfide; ferric-chloride solution (R.S.); sulfuric acid; 0.5 g. of dried gelatin; ice water; white of an egg; nitric acid; ammonium hydroxide; 0.5 cc. kerosene or cottonseed oil; 1% soap solution (R.S.); 10 cc. water glass, density 1.08 (R.S.).

a. Place in a test tube a small amount of flowers of sulfur (volume of a pea). Pour over this 5 cc. of alcohol and heat to the boiling point for 2 minutes, so that a saturated solution may be formed. Set the tube aside until the undissolved sulfur settles to the bottom of the tube; then pour the clear supernatant solution into 100 cc. of water and stir the liquid. The sulfur separates (?) in the form of a colloid.

b. To 18 cc. of water add 2 cc. of the solution of tartar-emetic. (So far as the experiment is concerned, the tartar-emetic may be regarded as the antimony salt of tartaric acid; its real formula is $KSbOC_4H_4O_6$.) Slowly bubble hydrogen sulfide through the solution for 2 or 3 minutes; antimony sulfide (Sb_2S_3) forms. Does it precipitate (explain)? Now add 3 drops of hydrochloric acid to the liquid; mix and set the tube aside for a few minutes (?). (See last paragraph, p. 383 of text.) Would you expect antimony chloride to give the same results as the tartar-emetic? (Recall that tartaric acid is a weak acid and hydrochloric acid a strong one.)

c. Heat 100 cc. of water to boiling and add 3 drops of the solution of ferric chloride. Ferric hydroxide ($Fe(OH)_3$) forms but remains in colloidal suspension. Now add 2 or 3 drops of sulfuric acid, stir, and set the beaker aside for a few minutes. The ferric hydroxide precipitates (?).

d. Place in a test tube a sufficient amount of small pieces of dried gelatin to make a layer 1 or 2 cm. in depth. Add about 10 cc. of water and heat the water until the gelatin forms a colloidal solution (gelatin sol). Now place the tube in ice water and note the formation of a gel. Again heat and cool the substance to see if the coagulation of the gelatin is a reversible process (?). Save the substance for use under ***f.***

e. Dissolve a small portion of the white of an egg in 10 cc. of cold water. Divide the liquid into two equal parts. Heat the one portion to boiling (?). To the other portion add 2 or 3 drops of nitric acid (?). Make suitable tests to determine whether the coagulation of the white of the egg (albumin) is a reversible process (?).

f. To 10 cc. of water glass, density 1.08, add 10 drops of sulfuric acid. Mix the liquids and set aside until the next laboratory period (?).

g. Often a colloid will prevent the formation of a precipitate or will delay crystallization; it is then called a *protective colloid.* This principle may be illustrated as follows: To 20 cc. of water add 4 drops of a solution of ferric chloride. Mix thoroughly and half fill each of two test tubes with the solution. To the solution in one of the tubes, add 2 or 3 cc. of the gelatin sol prepared in ***d*** above, and shake the mixture; then to each of the tubes add 3 or 4 drops of ammonium hydroxide and heat the resulting liquids to boiling. Set the tubes aside for a few minutes (?).

h. Pour 0.5 cc. of kerosene or cottonseed oil into each of two test tubes. Add water to one of the tubes until it is three fourths filled; to the other add an equal volume of a 1 % solution of soap. Close the tubes tightly with the thumbs and shake the mixtures violently for 1 minute; then set the tubes aside and note from time to time the changes in the mixtures in each tube (?).

EXERCISE 64

GENERAL METHODS FOR THE PREPARATION OF THE COMPOUNDS OF THE METALS

Apparatus. 6 test tubes.

Materials. 0.1 g. of each of the following salts, dissolved in 5 cc. water: (1) calcium chloride, (2) lead nitrate, (3) barium chloride, (4) ferric chloride, (5) silver nitrate (solutions of any of these on the reagent shelf may be used); ammonium carbonate solution (R. S.); 0.1 g. potassium iodide dissolved in 5 cc. water; hydrochloric acid.

a. *By the direct union of the elements.* Recall the formation of sulfides of copper and of iron (Exercise 37); of chlorides of copper and antimony (Exercise 25). Write the equations for the reactions involved.

b. *By dissolving a metal or its hydroxide in appropriate acids.* Recall the formation of zinc sulfate (Exercise 8); of sodium chloride (Exercise 28); of copper nitrate (Exercise 33). Write the equations for the reactions. When a metal or its hydroxide is acted upon by an acid, what becomes of the metal?

c. *By acting upon a salt of an acid with an acid having a higher boiling point.* Recall the action of sulfuric acid upon sodium nitrate (Exercise 33); of hydrochloric acid upon iron sulfide (Exercise 38); of sulfuric acid upon fluorides (Exercise 44); of sulfuric acid on chlorides (Exercise 26); of hydrochloric acid on carbonates (Exercise 50). Write the equations for each reaction and show in what respects they are all similar.

d. *By the decomposition of a compound.* Recall the action of heat upon potassium chlorate (Exercise 7); upon copper nitrate and lead nitrate (Exercise 34). Write the equations for the reactions involved.

e. The following compounds are insoluble (see Appendix, Table of Solubility of Various Solids): calcium carbonate ($CaCO_3$), lead sulfate ($PbSO_4$), barium carbonate ($BaCO_3$), ferric hydroxide ($Fe(OH)_3$), silver chloride ($AgCl$), lead iodide (PbI_2). Prepare a small amount of each in a test tube (R) and state the general principle involved in each reaction.

EXERCISE 65

THE COMPOUNDS OF SODIUM

Apparatus. Beaker; hydrogen generator for generating carbon dioxide (Fig. 30); 250-cc. bottle; hard-glass test tube; platinum wire; burner; piece of cobalt glass (10 cm. square).

Materials. Litmus paper (red and blue); hydrochloric acid; 5 g. sodium chloride; 5 g. sodium carbonate; sulfuric acid; 2 or 3 crystals of Glauber's salt; 10 g. ammonium carbonate; marble for generating carbon dioxide; 3 g. sodium bicarbonate; limewater (R. S.).

a. Recall experiments with sodium, Exercise 27.

b. Dissolve 5 g. of sodium chloride in as little water as possible. Set the solution aside overnight until some of the salt crystallizes out. Examine the shape of the crystals (use magnifying glass) (?).

c. Dissolve 5 g. of sodium carbonate in 20 cc. of water. Test the solution with red and with blue litmus paper (?).

Now convert the sodium carbonate present into common salt (R). How can you be sure that the product contains no unchanged sodium carbonate? Describe the method (R). Treat some of the salt so prepared with sulfuric acid. What gas is evolved (R)?

d. What changes does Glauber's salt undergo when exposed to air (refer to Exercise 43)?

e. Dissolve 10 g. of finely powdered ammonium carbonate in 100 cc. of cold ammonium hydroxide, shaking or stirring the mixture to secure solution. Saturate this solution with sodium chloride by shaking it with the finely pulverized salt. Pour off the clear solution into a 250-cc. bottle and pass a slow current of carbon dioxide through it until a precipitate (sodium bicarbonate) forms. Filter off the precipitate, dry between two pieces of filter paper, and examine (R). If a precipitate does not form by the end of the laboratory period, cork the bottle, and set it aside until the next laboratory period.

f. Fill a hard-glass test tube about one-fourth full of sodium bicarbonate and heat gently. Prove that carbon dioxide is evolved. What liquid condenses in the colder part of the tube? Write equations for the reactions by which sodium carbonate is converted into the bicarbonate, and vice versa.

g. Bend the end of a platinum wire into the form of a small loop and hold it in the Bunsen flame until it ceases to give any color to the flame; then dip it into a solution of a compound of sodium so that a drop of the solution is suspended in the loop; then hold it in the outer film of the base of a Bunsen flame. Note the color. Note the appearance of the sodium flame when viewed through a piece of cobalt glass.

EXERCISE 66

* THE DETERMINATION OF THE WEIGHT OF COMMON SALT OBTAINED BY ADDING HYDROCHLORIC ACID TO A DEFINITE WEIGHT OF SODIUM BICARBONATE (QUANTITATIVE)

Apparatus. Evaporating-dish and watch-glass cover; ring stand; burner; balance.

Materials. 1 g. sodium bicarbonate; hydrochloric acid.

Carefully weigh the evaporating-dish and watch glass. Transfer to the dish about 1 g. of sodium bicarbonate and reweigh. Pour 4 or 5 cc. of water on the bicarbonate, and place the watch glass on the dish so that only the lip of the dish remains uncovered. Now pour down the lip of the dish

2 or 3 drops of hydrochloric acid. Wait until the effervescence caused by the escape of the carbon dioxide ceases, then add a few drops more of the acid. Repeat until the addition of the acid no longer causes any effervescence. Now hold the watch glass in the hand just above the dish and with a little water carefully rinse back into the dish the liquid which has collected on the undersurface of it. Remove the watch glass and slowly evaporate the solution (Fig. 33).

When the solution has evaporated nearly to dryness, cover the dish with the watch glass and heat the dish with the tip of the flame. Continue the heating until there is no more liquid left in the dish or clinging to the undersurface of the glass. Then withdraw the heat and, after the dish is cool, reweigh.

From your results calculate the amount of salt formed from 1 g. of the bicarbonate. Compare your result with those obtained by other members of the class. How does the average of the results obtained compare with the theoretical results?

EXERCISE 67

THE TEST FOR POTASSIUM; THE PREPARATION OF POTASSIUM NITRATE

Apparatus. Platinum wire for flame test; cobalt glass; burner; small beaker; stirring-rod; funnel.

Materials. 0.1 g. potassium nitrate and 0.1 g. sodium chloride, each dissolved in a little water; 17 g. sodium nitrate; 15 g. potassium chloride; filter paper.

a. *Test for potassium.* Repeat ***g,*** Exercise 65, using a solution of a compound of potassium (?). Note the appearance of the flame through a piece of cobalt glass (?). Repeat, using a solution containing a compound of sodium as well as one of potassium. How could you detect both sodium and potassium if they were present in the same solution?

****b*. *Preparation of potassium nitrate.*** Dissolve 17 g. of sodium nitrate in 15 cc. of boiling water; also 15 g. of potassium chloride in 30 cc. of boiling water. Mix the two solutions in a small beaker and evaporate (stirring the mixture) to about 20 cc.; then quickly filter the hot solution and set the filtrate (filtrate A) aside until cold.

The reaction between potassium chloride and sodium nitrate is reversible, and the number of grams of each of the four compounds involved which dissolve in 100 g. of water at 15° and 100° is as follows:

	15°	*100°*		*15°*	*100°*
Sodium nitrate . .	84	180	Potassium nitrate .	26	247
Potassium chloride .	33	57	Sodium chloride . .	36	40

From a study of these solubilities, what compound should you expect would separate when the hot solutions of sodium nitrate and potassium chloride are mixed together? Test the solid on the filter paper. (Examine the crystals with a magnifying glass and compare with ***b***, Exercise 65, to see if your conclusion is correct. Taste the crystals.)

What solid should you expect would separate from filtrate A when it is cooled? Should you expect it to be pure? Examine it with a magnifying glass. Can you detect crystals of sodium chloride in this solid?

Dissolve the solid in as little hot water as possible, cool the solution, and again filter off the solid. Repeat until no crystals of sodium chloride can be detected. Prove the identity of this compound (?).

EXERCISE 68

THE PROPERTIES OF AMMONIUM COMPOUNDS

Apparatus. Evaporating-dish and watch-glass cover; 5 test tubes; burner; hydrogen sulfide generator (Fig. 42).

Materials. Ammonium hydroxide; hydrochloric acid; sodium hydroxide solution; litmus paper (red and blue); 10 g. ferrous sulfide; 0.2 g. ferrous sulfate dissolved in 10 cc. water; ammonium carbonate (R.S.); barium chloride (R.S.); calcium chloride (R.S.); zinc acetate (R.S.).

a. Pour 10 cc. of ammonium hydroxide into evaporating-dish, neutralize with hydrochloric acid, and evaporate to dryness on a water bath (Fig. 33) (?). Note the odor of the residue.

Introduce about one half of the residue (?) into a test tube, add a few drops of sodium hydroxide solution, and heat gently. Note the odor of the evolved gas and its action on a moist strip of red litmus paper (?). *This reaction serves as a good test for all ammonium compounds.*

Cover the evaporating-dish containing the remainder of the residue with a watch glass and heat *gently* with a *small* flame. Note that the solid *sublimes*; that is, passes directly from the solid form into a vapor which condenses (partly) on the cold surface of the watch glass.

b. Saturate 5 cc. of water (Fig. 42) with hydrogen sulfide and set aside; also saturate 5 cc. of ammonium hydroxide with the gas (?). Add a few drops of each to separate portions of ferrous sulfate solution (?) (ferrous sulfide is soluble in strong acid). In general, what metals will be precipitated by ammonium sulfide and not by hydrogen sulfide?

c. Add a few drops of ammonium carbonate solution to solutions of compounds of each of the following elements: barium, calcium, zinc (?).

EXERCISE 69

DETECTION OF COMPOUNDS OF THE ALKALI METALS

Recall such reactions of sodium, potassium, and ammonium compounds; also of carbonates, sulfates, nitrates, sulfites, sulfides, chlorides, bromides, iodides, and phosphates as will serve to identify them, and outline a method of procedure in the identification of them. Then ask the instructor for unknown compounds falling within this list and identify them.

EXERCISE 70

THE PREPARATION AND PROPERTIES OF SOAP

Apparatus. Evaporating-dish; ring stand and burner; large beaker; stirring-rod; funnel; small beaker; 4 test tubes.

Materials. Alcohol (R.S.); 5 g. cottonseed oil (or lard); 1 g. sodium hydroxide dissolved in 2 cc. water; hydrochloric acid; magnesium sulfate (R.S.); calcium chloride solution (R.S.); 1 g. sodium chloride dissolved in 5 cc. water; litmus paper (red and blue).

a. Add 10 cc. of alcohol to 5 g. of cottonseed oil in an evaporating-dish. To the resulting mixture add 1 g. of sodium hydroxide dissolved in 2 cc. of water. Evaporate carefully (use small flame and do not let the tip touch the dish), stirring the mixture constantly until the odor of alcohol can no longer be detected. What is formed? What remains in the dish?

b. Add 50 cc. of cold water to the residue in the dish, stir well for a few minutes, and filter. Pour 5 cc. of the filtrate into each of three test tubes. To the first add 2 or 3 drops of hydrochloric acid; to the second add a few drops of a solution of magnesium sulfate. In like manner add a few drops of a solution of calcium chloride to the third.

Note what takes place in each test tube. Why do waters containing calcium and magnesium compounds (*hard waters*) not lather freely with soap?

c. Add a few drops of sodium chloride solution to 10 cc. of the filtrate obtained in ***b*** (?) (p. 417 of text). The soap in the filtrate prepared in ***b*** is not in solution but in colloidal suspension (p. 383 of text). The salt causes the precipitation of the colloid (soap).

d. Recall the effect of soap in the formation of emulsions (Exercise 63). What influence has this property upon the cleansing action of soap?

EXERCISE 71

A STUDY OF SOME OF THE COMPOUNDS OF CALCIUM

Apparatus. Evaporating-dish; ring stand and burner; watch glass; small beaker; 2 test tubes; wire gauze; glass plate; platinum wire for flame test.

Materials. Piece of lime (size of a walnut); bits of marble; hydrochloric acid; ammonium carbonate (R.S.); ammonium hydroxide; disodium phosphate (R.S.); 10 g. plaster of Paris; 0.1 g. each of the chloride or the nitrate of calcium, strontium, and barium.

a. Immerse a piece of calcium oxide (lime) as large as a walnut in a beaker of water for three or four seconds. Repeat (if necessary) until the surface of the lime remains moist for ten seconds after the piece is withdrawn from the water; then place the piece on a watch glass and set the watch glass aside until the end of the period (?). *This is a convenient way of preparing calcium hydroxide.*

b. Dissolve 1 or 2 g. of marble in hydrochloric acid (R). What does the effervescence indicate? Evaporate the solution to dryness (use the bare flame and evaporate to complete dryness (Fig 33)). What is the composition of the residue? Expose a small piece of it to the air for an hour and account for the results.

Dissolve the remainder of the residue in a little water and divide the liquid into two portions. To the one add a few drops of ammonium carbonate (R); to the other add a few drops of ammonium hydroxide and then of a solution of disodium phosphate. The precipitate is $Ca_3(PO_4)_2$.

c. Place a piece of marble on a wire gauze and apply a strong heat for about fifteen minutes. When cool, drop the residue into 25 cc. of water and stir. Filter, and test the filtrate with litmus paper. Then blow exhaled air through the filtrate. Explain.

****d.*** Place on a glass plate a penny which has been rubbed with a drop of oil. Pour over the coin a thick paste prepared by adding water to plaster of Paris. Set the glass plate aside until the paste hardens, then remove the coin and note the result.

e. Try the flame tests for compounds of calcium, of strontium, and of barium (use the chloride or nitrate), as in ***g***, Exercise 65, and note the results.

EXERCISE 72

HARD WATERS AND METHODS FOR SOFTENING THEM

Apparatus. 60-cc. bottle; carbon dioxide generator (Fig. 30); ring stand and burner; funnel; 2 test tubes; small beaker.

Materials. 30 cc. limewater (R. S.); bit of soap dissolved in water; 10 to 15 g. of marble; magnesium sulfate (R. S.); filter paper; 1 g. sodium carbonate dissolved in as little water as possible; hydrochloric acid.

a. Bubble carbon dioxide into 25 cc. of limewater diluted with an equal volume of water. Note that a precipitate forms (R), which gradually dissolves as more of the gas is passed through (?). Add a few drops of a soap solution to 5 cc. of the resulting solution and note the result.

Divide the remainder of the solution into two parts. Gradually boil the one part and note the result. To the other part add a few drops of clear limewater, mix intimately, and note the results. Explain.

b. Shake 1 g. of calcium sulfate with 10 cc. of water in a test tube for two or three minutes; filter, and add to the filtrate 2 or 3 drops of a saturated solution of sodium carbonate (R).

All hard waters contain more or less calcium acid carbonate, calcium sulfate, calcium chloride; also the corresponding compounds of magnesium. (The methods used for removing the calcium compounds likewise serve for removing the magnesium compounds.) How could such waters be softened on a large scale? Waters softened in this way would contain what compounds in solution?

c. Test some hard waters from wells by the above methods.

EXERCISE 73

* SOME ADDITIONAL EXPERIMENTS WITH SOAP

Apparatus. Three 250-cc. bottles; burette or graduated cylinder; 2 test tubes.

Materials. 1 g. soap dissolved in 100 cc. distilled water; ordinary reagents; 0.5 g. sodium sulfate.

a. *The determination of the amount of soap lost by using hard water for washing.* Place three 250-cc. bottles on the desk. Into the first pour 100 cc. of *hard water* (preferably an average sample of the water used in your town or city); into the second pour 100 cc. of distilled (or rain) water; and into the third pour 100 cc. of distilled water containing 0.5 g. of sodium sulfate in solution. Now add to each the soap solution, 1 cc. at a time, and shake the bottle *vigorously.* Continue adding the soap solution until the lather formed persists for five minutes. Compare the amounts of the soap solution required in each case to produce a permanent lather (?). Does the presence of sodium sulfate prevent the formation of the lather (sodium sulfate is left in waters softened by ordinary methods) (p. 432 of text)?

It will be interesting to make at least a rough approximation of the amount of hard water used yearly in your city for washing, and then to determine the cost of the soap lost in one year owing to the use of hard water.

b. *The analysis of washing-powders.* Devise methods for detecting the presence of the following substances if present in washing-powders: (1) sodium carbonate; (2) borax (*b*, Exercise 62); (3) mineral matter, such as sand. Test one or more washing-powders for these substances.

EXERCISE 74

THE PREPARATION AND PROPERTIES OF BLEACHING-POWDER

Apparatus. Flask (250-cc.) and two bottles (250-cc.) connected with glass tubing as shown in Fig. 48; 200-cc. beaker; stirring-rod.

Materials. 15 g. manganese dioxide; hydrochloric acid; 25 cc. of water in bottle *B*; 10 cc. of sodium hydroxide solution added to 50 cc. of water in *D*; sufficient calcium hydroxide (slaked lime) to half fill the tube *C*; strips of colored calico; sulfuric acid.

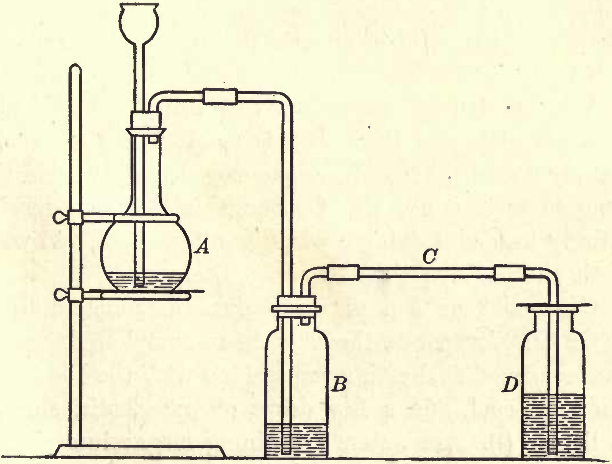


FIG. 48

(Hood.) Generate chlorine in *A* (Fig. 48). The gas must be evolved *slowly*, for which reason only a *very gentle* heat is applied to the flask. The gas bubbles through the water in *B*, then passes over the calcium hydroxide in *C*, any unabsorbed gas being caught in *D* (?).

When all of the chlorine has passed over from *A*, disconnect the apparatus and transfer the contents of the tube *C* to a beaker and pour over it 75 cc. of water containing 1 cc. of sulfuric acid. Stir well and immerse in the mixture some strips of colored calico for a few minutes (?). Complete the following equations:

$$Ca(OH)_2 + Cl_2 \longrightarrow$$
$$CaOCl_2 + H_2SO_4 \longrightarrow$$

EXERCISE 75

MAGNESIUM AND ITS COMPOUNDS

Apparatus. Porcelain crucible and cover; ring stand and burner; pipe-stem triangle; evaporating-dish; 2 test tubes.

Materials. Strips of magnesium 5 cm. long; small beaker; red litmus paper; 2 to 3 g. magnesium carbonate; magnesium sulfate solution (R. S.); ammonium chloride (R. S.); disodium phosphate (R. S.).

a. Wind a strip of magnesium wire into a coil and place it in a porcelain crucible. Put the cover on the crucible and apply a gentle heat. Raise the cover slightly from time to time so as to admit air. Continue until the magnesium is entirely burned, leaving a white powder. Cool, add water, and stir thoroughly; then test with litmus (?).

b. Convert 2 or 3 g. of magnesium carbonate into the chloride (R). Evaporate the solution to complete dryness in an evaporating-dish, heating the residue with the bare flame. When it is cool, add a few drops of water, stir, and test with litmus (?). Are waters containing magnesium chloride objectionable for use in steam boilers?

c. Pour 3 cc. of a solution of magnesium sulfate into a test tube and add an equal volume of a solution of ammonium chloride. Now add to this a few drops of a solution of disodium phosphate (Na_2HPO_4). The precipitate has the composition $MgNH_4PO_4$. What is the name of this compound? To what class of compounds does it belong? The above reaction serves as a good test for magnesium compound.

EXERCISE 76

ZINC AND ITS COMPOUNDS

Apparatus. Blowpipe; burner; 3 test tubes; beaker.

Materials. 0.5 g. zinc; sulfuric acid; sodium hydroxide solution; ammonium sulfide (R.S.); 0.2 g. sodium carbonate dissolved in 3 cc. of water; 2 g. zinc sulfate; hydrochloric acid; pieces of charcoal.

a. Place a bit of zinc on charcoal and heat it in the tip of the flame of a blowpipe (R). The resulting compound is deposited as a film on the charcoal. Note the color of it. Is its color the same when hot as when cold?

b. Dissolve 0.5 g. of zinc sulfate in 20 cc. of water. Divide this solution into 3 parts and test with the following reagents: (1) sodium hydroxide solution (1 drop, or just sufficient to cause a precipitate); zinc hydroxide precipitates (R), but the precipitate dissolves again if an excess of sodium hydroxide is added; (2) ammonium sulfide (R) (note the color of the precipitate); *zinc is the only metal which forms an insoluble white sulfide*; (3) sodium carbonate solution; a basic carbonate is formed.

c. Devise a process for converting zinc sulfate into zinc chloride. Submit the process to your instructor for approval. When approved, prepare some of the chloride according to your process. For what is the compound used?

EXERCISE 77

ALUMINIUM AND ITS COMPOUNDS

Apparatus. 5 test tubes; blowpipe; burner; 2 beakers; piece of charcoal.

Materials. 1 g. aluminium; hydrochloric acid; ammonium hydroxide; sodium hydroxide; aluminium sulfate solution (R.S.); 2 or 3 drops of cobalt nitrate solution (R.S.); 1 g. sodium carbonate in 5 cc. of water; aluminium sulfate and potassium sulfate sufficient to make 20 g. of crystals of potassium alum.

a. Note the physical properties of aluminium. Add 5 cc. of water to a bit of the metal in a test tube and add hydrochloric acid, a drop at a time, sufficient to dissolve the metal. Filter (if necessary) and dilute the solution to about 10 cc. and divide it into two equal parts. To the first add ammonium hydroxide until the solution reacts alkaline (R); to the second add sodium hydroxide, a drop at a time, until a precipitate forms (R). Shake the latter tube and divide the mixture into two equal portions. To the one add hydrochloric acid, noting the result (R); to the other add sodium hydroxide solution until the precipitate dissolves (R). Is aluminium hydroxide an acid or a base?

b. Prepare some aluminium hydroxide by adding ammonium hydroxide to a solution of aluminium sulfate, and heat it on charcoal in the blowpipe flame (R). Moisten the residue with a drop or two of a solution of cobalt nitrate and reheat. Note the result. Advantage is taken of this property in detecting the presence of aluminium.

c. Add a solution of sodium carbonate to a solution of any salt of aluminium. Note that a gas is evolved (?). Devise a method for determining whether or not this gas is carbon dioxide, and make the test.

d. Calculate the weights of aluminium sulfate (the crystals of aluminium sulfate have the formula $Al_2(SO_4)_3 \cdot 18\,H_2O$)

and of potassium sulfate required to prepare 20 g. of crystals of potassium alum; then dissolve these amounts of the two compounds separately in as little water as possible, mix the two solutions thoroughly, and set aside for a few days to crystallize. If a string is suspended in the liquid, the crystals will deposit on it. These may then be withdrawn and their properties studied (?).

EXERCISE 78

THE USE OF ALUMINIUM SULFATE IN THE PURIFICATION OF WATER

Apparatus. Three 250-cc. wide-mouthed bottles; graduated tube.
Materials. Aluminium sulfate solution (R.S.); limewater (R.S.).

Label the three bottles *A*, *B*, and *C* respectively. Nearly fill *A* and *B* with muddy water, pouring a like volume of distilled water into *C*. Add 5 drops of aluminium sulfate solution to *A* and *C* respectively. Mix the contents of each bottle thoroughly. Now to bottles *A* and *C* (each) add 10 cc. of limewater. Set the bottles aside and examine at the beginning of the next laboratory period (?).

For experiments on the use of aluminium compounds as mordants in dyeing, see Exercise 99, Appendix A.

EXERCISE 79

REACTIONS OF BAKING-POWDERS

Apparatus. Large test tube, and cork to fit; stirring-rod.
Materials. 4 g. sodium bicarbonate; limewater (R.S.); alum; cream of tartar.

a. Grind together 2 g. of sodium bicarbonate and sufficient (?) alum to react with the bicarbonate. Put the mixture into a large test tube and cover the mixture with water. Cork the tube tightly and shake the mixture. Dip a glass rod into limewater and remove it gently so that a drop of the clear liquid clings to the end of the rod; then quickly remove the cork from the test tube and lower the end of the rod into the tube (?). Write all reactions involved.

b. Repeat ***a***, substituting cream of tartar for the alum (?).

c. What compounds remain in food as a result of the use of an alum baking-powder? of a cream of tartar baking-powder? For methods of analysis of baking-powders, see Exercise 98, Appendix A.

EXERCISE 80

A STUDY OF IRON, COBALT, AND NICKEL

Apparatus. 2 beakers; watch glass; funnel; flask (250-cc.); 8 test tubes; ring stand and burner.

Materials. Piece of watch spring, 10 to 15 cm. in length; 5 g. of small tacks or fine iron wire; 0.5 g. powdered iron; filter paper; hydrochloric acid; sulfuric acid; nitric acid; ammonium hydroxide; potassium ferrocyanide (R. S.); potassium ferricyanide (R. S.); potassium sulfocyanide (R. S.); 0.5 g. cobalt nitrate dissolved in 10 cc. of water: 0.5 g. nickel nitrate dissolved in 10 cc. of water.

a. Heat a piece of watch spring (from 10 to 15 cm. in length) to a white heat in a Bunsen flame. Let it cool slowly, and when cold bend it to determine if it is brittle (?). Again heat and *at once* plunge into a beaker of cold water. Bend the piece as before (?). Reheat the piece, allow it to cool slowly, and again examine it (?).

b. Place 5 g. of fine iron wire or small tacks in a beaker and pour over it 15 cc. of water. Now add 4 cc. of concentrated sulfuric acid and heat very gently (hood) until a vigorous evolution of gas takes place (R), then cover the beaker with a watch glass and set it aside in the hood until near the end of the laboratory period. Then add 10 cc. of water and heat slowly until the liquid boils, stirring the mixture constantly. Filter off any undissolved solids, collecting the filtrate in a beaker. Set the filtrate in your desk until the next laboratory period; then examine the crystals (?).

c. Place about 0.5 g. of iron powder in a small flask, pour over it 5 cc. of water, and then from 1 to 2 cc. of hydrochloric acid. Mix the contents of the flask, heat the flask slightly, and set aside in the hood for five minutes (?). Add 50 cc. of water, mix well, and filter. Divide the filtrate into two equal parts. Mark one of these A and set it aside. Add to the other portion about 1 cc. of hydrochloric acid and heat it nearly to boiling; then withdraw the flame and add nitric acid (about 2 cc. of the concentrated acid will be required), a drop at a time, with constant stirring, until the solution, which is at first dark brown, becomes light yellow in color (?). Cool the resulting solution. Call this solution B. How do solutions A and B differ in composition? Now compare the action of the following reagents upon solutions A and B (add 2 or 3 drops of the reagents to 5 cc. of the solutions in test tubes): ammonium hydroxide, potassium ferrocyanide, potassium ferricyanide, potassium sulfocyanide (KCNS). Tabulate your results as follows:

	FERROUS CHLORIDE (Solution A)	FERRIC CHLORIDE (Solution B)
Ammonium hydroxide . .		
Potassium ferrocyanide . .		
Potassium ferricyanide . .		
Potassium sulfocyanide . .		

d. Test separate solutions of a salt of cobalt and nickel with a borax bead (*b*, Exercise 62); with a solution of sodium hydroxide; with ammonium sulfide. Note the results.

e. Write on a piece of paper, using the cobalt nitrate solution as ink. Now heat the paper gently over a flame (?) (invisible ink); moisten the paper with a damp cloth (?).

EXERCISE 81

A STUDY OF COPPER AND ITS COMPOUNDS

Apparatus. 3 test tubes; beaker; ring stand and burner.

Materials. Nail; copper sulfate solution (R. S.); sodium hydroxide; ammonium sulfide (R. S.); ammonium hydroxide; 10 cm. copper wire; hydrochloric acid.

a. Recall the action of nitric acid and of sulfuric acid on copper (Exercises 33, 40); also the action of chlorine and sulfur on copper (Exercises 25, 37). Place a nail in a solution of copper sulfate. Account for the result.

b. To a *cold* solution of copper sulfate add one half its volume of sodium hydroxide solution. Copper hydroxide ($Cu(OH)_2$) is precipitated. Now heat to boiling. The hydroxide is decomposed into water and cupric oxide (black).

c. Try the action of ammonium sulfide on copper sulfate (R).

d. Add 1 drop of ammonium hydroxide to a dilute solution of copper sulfate; now continue to add the ammonium hydroxide, drop by drop, until the precipitate which is at first formed is dissolved. How does the color of this solution compare with that of the original solution? This reaction is characteristic of copper compounds.

e. Recall the formation of cuprous oxide (Exercise 53).

f. Moisten the end of a copper wire with hydrochloric acid and hold it in the edge of a Bunsen flame (?).

EXERCISE 82

A STUDY OF MERCURY AND ITS COMPOUNDS

Apparatus. 100-cc. beaker; 2 test tubes.

Materials. Globule of mercury (size of a grain of wheat); nitric acid; copper penny; 0.5 g. mercuric oxide; 3 cc. solution of mercurous nitrate (R.S.); hydrochloric acid.

a. Note the physical properties of mercury. Place a globule of it in a small beaker and add (hood) just enough nitric acid to dissolve it. Dilute the product with 10 cc. of water and place a copper penny in the solution. After a few minutes remove the coin and polish it with a piece of cloth. Account for the result.

b. For what purpose have we used mercuric oxide? Place 0.2 g. of it in a test tube and dissolve it in as little nitric acid as possible (R). Then add water until the test tube is one-fourth full. Into a second test tube pour a similar volume of a solution of mercurous nitrate. Now add 2 or 3 drops of hydrochloric acid to each test tube (R). What conclusions do you draw in reference to the solubility of the two chlorides of mercury?

c. Prepare some mercuric sulfide (?).

EXERCISE 83

A STUDY OF SILVER AND ITS COMPOUNDS

Apparatus. 200-cc. beaker; funnel; blowpipe; 4 test tubes; burner; ring stand.

Materials. Piece of cotton cloth 2 or 3 cm. square; silver dime; nitric acid; hydrochloric acid; ammonium hydroxide; filter paper; hot water; 2 or 3 g. sodium carbonate; piece of charcoal; 10 cc. silver nitrate solution (R.S.); solutions of potassium bromide and of potassium iodide (R.S.); 1 cc. formaldehyde solution.

a. Place a drop of silver nitrate solution on a piece of cotton cloth and warm gently. Can you wash the stain away?

What is it? Try ammonia water. Owing to the permanence of this stain, silver nitrate is sometimes used in making indelible ink.

b. Place a silver dime in a small beaker and add (hood) sufficient nitric acid to dissolve it. The solution may be hastened by applying a gentle heat. When the solution is complete, dilute the product with about 25 cc. of water. Account for the color of the liquid. Now add a solution of hydrochloric acid until a precipitate ceases to form. On being stirred, the precipitate (?) settles to the bottom of the beaker. Carefully decant the clear supernatant liquid and add ammonium hydroxide until the solution becomes alkaline (?) (***d***, Exercise 81). Wash the precipitate two or three times by pouring hot water over it and decanting. Finally, remove any remaining water by filtration. Mix the product with an equal bulk of sodium carbonate, transfer to a small cavity in a piece of charcoal, and heat it with a blowpipe. The silver salt is gradually reduced to metallic silver, which will fuse into a globule if sufficient heat is applied. How does this differ in composition from the metal constituting the silver dime?

c. Prepare small amounts of the chloride, the bromide, and the iodide of silver (R). Expose to the sunlight the test tubes containing the precipitates and note any changes. For what are these compounds used?

d. Recall the formation of silver sulfide (Exercise 39).

e. Thoroughly clean a test tube by rinsing it with a few drops of nitric acid and then with distilled water. Pour into the tube 2 cc. of silver nitrate solution and dilute with an equal volume of water. Now add ammonium hydroxide, *a drop at a time* (shake the tube after addition of each drop), until the precipitate which forms at first redissolves, leaving a clear liquid. Next add 2 drops of a solution of formaldehyde, mix thoroughly, and place the tube in a beaker of cold water. Gradually heat the water to boiling (?).

EXERCISE 84

PROPERTIES OF TIN AND ITS COMPOUNDS

Apparatus. 100-cc. beaker; piece of charcoal; blowpipe; ring stand and burner.

Materials. 2 pieces of tin (size of a pea); hydrochloric acid; mercuric chloride solution (R.S.).

a. Note the physical properties of tin (?). Heat a bit of it on charcoal (?).

b. Dissolve a small piece of the metal in hydrochloric acid (R). Cool, dilute with a little water, and add 1 or 2 drops of the solution to 3 cc. of mercuric chloride solution. A white precipitate of mercurous chloride forms (R).

Now add a few drops more of the stannous chloride solution and heat the mixture gently. The mercurous chloride is reduced to metallic mercury, which forms a dark-gray precipitate (R).

EXERCISE 85

A STUDY OF LEAD AND SOME OF ITS COMPOUNDS

Apparatus. Ring stand and burner; blowpipe; 200-cc. beaker; 5 test tubes; 100-cc. beaker; funnel and filter paper.

Materials. 2 g. lead (obtain some scrap lead from a plumber); piece of charcoal; nitric acid; ammonium sulfide (R.S.); sulfuric acid; potassium chromate (R.S.); hydrochloric acid; strip of zinc; 2 g. red lead.

a. Note the physical properties of the metal. Heat a small bit on charcoal. Is it easily melted? Note the coating formed on the charcoal (?).

b. Place about 1 g. of the metal in a beaker (hood) and add 20 cc. of water and 5 cc. of nitric acid. Heat *gently* until the metal is dissolved (?). Dilute to 100 cc. and filter, if necessary, to obtain a clear solution. Call this solution A.

Now test small portions of this solution with ammonium sulfide, sulfuric acid, and potassium chromate (K_2CrO_4) respectively (R). Note the color of the precipitates. Add a few drops of hydrochloric acid to a test tube one-fourth full of solution A. Lead chloride is precipitated. Heat the mixture to boiling, and if the liquid does not become clear, add just enough boiling water to dissolve the precipitate; then set it aside until cool and note the result. How can you distinguish between lead chloride and silver chloride?

In the remainder of solution A suspend a piece of zinc (?).

c. Introduce 1 g. of red lead into a 100-cc. beaker and add 5 cc. of water. Mix well and add 1 cc. of nitric acid. Stir the mixture and heat it gently. Notice the change in color (?). Now add 25 cc. of water, stir the mixture, and filter. Test the filtrate for the presence of lead (?). Place the filter paper and contents in a small beaker, add 2 or 3 cc. of hydrochloric acid, and stir with a glass rod so that the acid will come in contact with the solid on the paper. Heat the beaker gently. What gas is evolved (color and odor)?

EXERCISE 86

* DETECTION OF SILVER, LEAD, AND MERCURY, WHEN PRESENT IN THE SAME SOLUTION

Apparatus. 300-cc. beaker; ring stand and burner; stirring-rod; funnel.

Materials. Solutions of silver nitrate, lead nitrate, and mercurous nitrate (R. S.); filter paper; potassium chromate (R. S.); sulfuric acid; ammonium hydroxide; nitric acid; hot water.

The detection of any one metal becomes more complicated when other metals are present in the same solution. As a rule it is necessary so to treat the mixture as to separate the metals from each other. The principle involved is illustrated in the following procedure, the solution containing the nitrates of silver, lead, and mercury (*ous*).

Prepare a solution containing 0.2 g. of each of the following compounds: silver nitrate, lead nitrate, and mercurous nitrate. Dilute with water to about 200 cc. Precipitate with hydrochloric acid (R). Filter and fill the paper with boiling water three or four times, collecting the liquid as it flows from the funnel. This liquid contains the lead chloride which has been dissolved by the hot water. Its presence may be proved by testing separate portions of the filtrate with solutions of potassium chromate and sulfuric acid respectively.

To the residue on the filter paper (of what is it composed ?) add 2 or 3 cc. of ammonium hydroxide and collect the liquid as it drops from the funnel. This liquid contains the silver chloride dissolved from the residue by the ammonium hydroxide. To prove its presence add nitric acid to the liquid until just acid to litmus paper. The silver chloride is precipitated.

What effect did the ammonium hydroxide have upon the color of the residue on the filter paper ? This change in color is due to the action of ammonium hydroxide on the mercurous chloride, and serves as a test for the presence of the latter.

Supposing that the original solution contained only one or two of the metals of the group, how would the absence of the remaining ones be indicated ?

EXERCISE 87

A STUDY OF SOME OF THE COMPOUNDS OF MANGANESE

Apparatus. 6 test tubes.

Materials. 0.1 to 0.2 g. potassium permanganate ($KMnO_4$); crystal of ferrous sulfate; sulfuric acid; ammonium sulfide (R. S.); ammonium carbonate (R. S.); sodium hydroxide; manganese chloride solution (R. S.).

a. Examine the physical properties of potassium permanganate (?). Dissolve about 0.1 g. of it in 5 cc. of water (?). Add a drop of the solution to a solution containing a small crystal of ferrous sulfate and 2 or 3 drops of sulfuric acid. The ferrous sulfate is changed to ferric sulfate, the oxygen in the reaction (see equation below) coming from the potassium permanganate, which is a good oxidizing agent.

$$2\,FeSO_4 + H_2SO_4 + O \longrightarrow Fe_2(SO_4)_3 + H_2O$$

b. In potassium permanganate the manganese acts as an acid-forming element. It also acts as a base-forming element in certain compounds. Try the action of ammonium sulfide, ammonium carbonate, and sodium hydroxide, respectively, on a solution of manganese chloride (?).

EXERCISE 88

A STUDY OF SOME OF THE COMPOUNDS OF CHROMIUM

Apparatus. 6 test tubes.

Materials. Solution of potassium chromate (R. S.); lead acetate solution (R. S.); barium chloride solution (R. S.); ammonium sulfide (R. S.); sodium carbonate solution (R. S.); sodium hydroxide; 0.5 g. chromium chloride or chromium sulfate dissolved in 25 cc. of water.

a. *Chromates and dichromates.* Write the formula for potassium chromate; for potassium dichromate. Is the chromium an acid-forming or a base-forming element in these compounds? Add 2 or 3 drops of sulfuric acid to a little potassium chromate solution (?).

b. Try the effect of a solution of potassium chromate on a solution of a compound of lead (R); also on a compound of barium (R).

c. Add a few drops of hydrochloric acid to a little solid potassium chromate and explain the results (R). Repeat, using potassium dichromate (R).

d. *Salts of chromium.* Try the effect of the following reagents on a solution of a salt of chromium: ammonium sulfide, sodium carbonate, sodium hydroxide. Write all the equations.

EXERCISE 89

*BORAX-BEAD TESTS

Apparatus. Platinum wire; burner.

Materials. Borax (R. S.); 1 cc. of a solution of a compound of each of the following metals: nickel, iron, manganese, copper.

Recall the effect of adding a trace of cobalt nitrate to a borax bead (*b*, Exercise 62). Repeat the experiment, substituting for the cobalt nitrate, salts of the following metals: nickel, iron, manganese, copper (?).

APPENDIX A

SOME OPTIONAL EXERCISES OF SPECIAL INTEREST TO STUDENTS OF HOME ECONOMICS

EXERCISE 90

A STUDY OF TEXTILE FIBERS

Apparatus. 4 small beakers or test tubes; stirring-rod; burner; evaporating-dish; 2 large beakers; microscope.

Materials. 3 strips each of uncolored cotton, wool, silk, and linen cloth (3 cm. × 15 cm.); 50 cc. of sodium hydroxide solution; strip of filter paper; 3 strips of filter paper (2 cm. × 10 cm.); ammonium hydroxide; hydrochloric acid; sulfuric acid.

a. *Effect of heat upon textile fibers.* Ignite the end of a strip of cotton cloth in a Bunsen flame; then withdraw from the flame. Note the odor of the burning cloth. Does the cloth when ignited continue to burn?

Repeat, using strips of wool, silk, and linen. Can you distinguish in this way between vegetable fibers (cotton, linen) and animal fibers (wool, silk)?

b. *How to distinguish between vegetable fibers (cotton, linen) and animal fibers (wool, silk).* Place a strip of each kind of cloth in small beakers, cover the cloth with sodium hydroxide solution, and boil the liquid for ten minutes, replacing the water as it evaporates (the cloth must always be completely covered with the liquid); then set the beakers aside until cool (?).

c. *How to distinguish between the animal fibers.* Immerse strips of silk and wool in concentrated hydrochloric acid and note the change after they have stood a few minutes (?).

d. *How to distinguish between the vegetable fibers.* Immerse strips of cotton and linen in concentrated sulfuric acid for two minutes (?).

e. *Microscopic appearance of textile fibers.* Examine the appearance of each kind of fiber under the microscope. Compare with the diagrams on page 330 of text.

f. *Parchment paper.* Pour 20 cc. of sulfuric acid slowly (CAUTION), and with constant stirring, into a beaker containing 10 cc. of water. Pour the solution into an evaporating-dish and allow to cool. Draw strips of filter paper slowly through the acid and then immerse them in a large beaker of water. Finally, wash the strips in a large beaker of water containing 2 or 3 drops of ammonium hydroxide.

When the strips are dry, compare their properties with those of the untreated paper.

EXERCISE 91

THE DETERMINATION OF THE AMOUNT OF ALCOHOL PRESENT IN AN ALCOHOLIC LIQUID (QUANTITATIVE)

Apparatus. Flask and condenser connected as is shown in Fig. 49 (*A* is a 250-cc. flask, and *C* a flask which holds 100 cc. when filled to a point marked on its neck (a graduated cylinder may be used in its place)); ring stand and burner; hydrometer reading from 0.900 to 1.000; cylinder.

Materials. 100 cc. of any alcoholic liquid, such as an alcoholic medicine.

The method commonly used is based on the fact that the specific gravity of an aqueous solution of alcohol varies according to the percentage of alcohol present. Tables have been worked out with great care, giving the specific gravities of solutions of alcohol of various strengths. If we know the specific gravity of an aqueous solution of alcohol, therefore, it is only necessary to refer to the table and read the percentage of alcohol. These tables are calculated for certain temperatures, so that in determining the specific gravity of the alcohol care must be taken that the temperature of the solution is the same as that indicated in the tables used. These tables are given in Allyn's "Elementary Applied Chemistry" (pp. 66–75); also in *Bulletin 107*, United States Department of Agriculture; also in the "United States Pharmacopœia" used by the druggist.

Pour exactly 100 cc. of any alcoholic liquid into a flask *A* (Fig. 49) and distill over about 50 cc., collecting the distillate in the 100-cc. flask, as shown in the figure. Since alcohol is quite volatile (boiling point 78.3°), all of the alcohol present in the 100 cc. of liquid will be contained in the 50 cc. of the distillate. Now dilute the distillate to exactly 100 cc., mix thoroughly, pour into a cylinder, and having brought it to the proper temperature, determine its specific gravity by means of a hydrometer. Refer to the tables for the percentage of alcohol present in the distillate. Why is it necessary to distill the liquid? Why dilute the distillate to 100 cc. before taking the specific gravity?

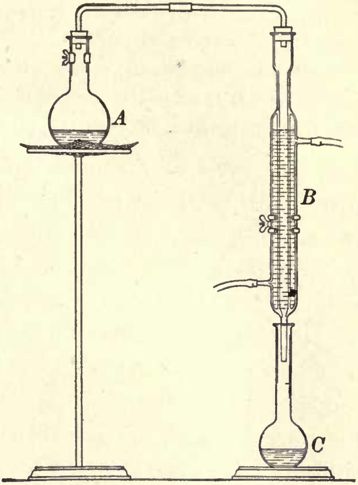


Fig. 49

EXERCISE 92

THE EFFECT OF PRESERVATIVES

Apparatus. Two 250-cc. bottles or beakers; 500-cc. beaker; 2 test tubes; ring stand and burner.

Materials. 300 cc. sweet milk; drop of formalin; 10 cc. hydrochloric acid to which is added 1 drop of a solution of ferric chloride (R. S.); 1 g. sodium benzoate.

a. *The effect of formaldehyde on milk.* Thoroughly clean two small bottles with hot water and half fill each with sweet milk. Add to the milk in one of the bottles a drop of formalin and mix thoroughly. Now pour about 5 cc. of milk from each of the two bottles into separate test tubes; add to each an equal volume of concentrated hydrochloric acid to which has been added just a trace of ferric chloride solution (the ordinary commercial hydrochloric acid serves the purpose well, since it usually contains a trace of ferric chloride as an impurity).

Mix the contents of each of the tubes thoroughly and set them in a beaker of boiling water. Note any change in color. How can you detect the presence of formaldehyde in milk?

Set the two bottles containing the remainder of the milk aside and examine from time to time, noting when the milk in each becomes sour.

b. *The effect of sodium benzoate on sweet cider or grape juice.* The student may likewise study the action of sodium benzoate in preventing the fermentation of sweet cider or grape juice. In such cases the weight of the preservative added to the beverage should be from 0.1 to 0.2 per cent of the weight of the beverage.

The use of formaldehyde in foods is no longer permitted by the Federal laws, since it is a poison. The use of sodium benzoate is permitted in certain foods. Do you see any reason why the Federal law should prohibit entirely the use of preservatives in milk but permit the use of sodium benzoate in certain foods, such as jams and catchup?

EXERCISE 93

A STUDY OF VINEGAR (QUANTITATIVE)

Apparatus. Evaporating-dish; beaker; ring stand and burner; 2 burettes.

Materials. 30 cc. each of different kinds of vinegars; 1 cc. phenolphthalein solution (R. S.); 10 cc. sodium hydroxide solution (on desk) diluted to 100 cc. with water; standard oxalic acid solution (R. S.).

a. *Determination of the solids in vinegar* (*quantitative*). Weigh a small evaporating-dish and pour into it a definite volume (say 25 cc.) of vinegar. Evaporate the vinegar to complete dryness (Fig. 33) and carefully wipe the bottom of the dish with a dry towel. Weigh it once more.

From your results calculate the amount of solid matter in 100 cc. of the vinegar. Pure cider vinegar should contain not less than 1.6 g. of solids in 100 cc. of vinegar (this is the limit fixed by the Federal government as well as by the statutes of many of the individual states).

Note the odor and taste of the solid matter obtained from the vinegar. The solids from pure cider vinegar should have an odor and taste suggestive of baked apples. Other kinds of vinegar may be tested in the same way. The character of the solids varies according to the source of the vinegar.

b. *Determination of the acidity of vinegar.* Dilute with water 10 cc. of sodium hydroxide solution to 100 cc. and determine the exact strength of this solution as follows: Measure out from a burette exactly 10 cc. of the standard oxalic acid ($C_2H_2O_4 \cdot 2\,H_2O$) solution, and add to it 2 or 3 drops of phenolphthalein solution. Fill another burette with the dilute sodium hydroxide solution and allow this to run slowly into the oxalic acid solution (see Exercise 29) until the last drop, when stirred through the acid solution, turns it a permanent pink color. Note, from the burette reading, the exact volume of the hydroxide solution used in neutralizing the acid. Now calculate the strength of the hydroxide solution as follows: The equation for the reaction is

$$C_2H_2O_4 \cdot 2\,H_2O + 2\,NaOH \longrightarrow Na_2C_2O_4 + 4\,H_2O$$

The strength of the oxalic acid solution being known, — namely, 15 g. to 1000 cc., — the strength of the sodium hydroxide solution can easily be calculated. Insert the following values:

Milligrams of oxalic acid in 10 cc. of solution . . . .
Number of cubic centimeters of sodium hydroxide solution required to neutralize the oxalic acid in 10 cc. of the acid solution . . . .
Milligrams of sodium hydroxide in this volume . . .
Milligrams of sodium hydroxide per cubic centimeter of solution . . . .

Having determined the strength of the sodium hydroxide solution, proceed as follows to determine the acidity of a sample of vinegar:

Introduce exactly 5 cc. of vinegar into a small beaker and dilute with about 50 cc. of water. Add 2 drops of phenolphthalein and run in the solution of sodium hydroxide from a burette until the liquid in the beaker is neutral, as in Exercise 29. The equation for the reaction is as follows:

$$H \cdot C_2H_3O_2 + NaOH \longrightarrow Na \cdot C_2H_3O_2 + H_2O$$

Knowing the strength of the sodium hydroxide solution and the volume of this solution required to neutralize the acetic acid in 5 cc. of vinegar, it is easy to calculate the amount of acetic acid present in the 5 cc. of vinegar. The Federal law requires that vinegar must contain not less than 4 g. of acetic acid in 100 cc. of vinegar. Give results of different vinegars tested.

EXERCISE 94

TESTS FOR FATS AND PROTEINS

Apparatus. Stirring-rod; evaporating-dish; 2 beakers; ring stand and burner.

Materials. 1 or 2 drops of cottonseed oil; 1 or 2 g. of lard or butter; bit of cheese; small piece of bread; small portion of white of egg; 25 cc. milk; nitric acid; ammonium hydroxide; 1 g. flour.

a. *Fats.* In different places on a piece of white paper rub 1 drop of various fats, such as cottonseed oil, cream, melted lard, and butter. Note the appearance of the spot when held in front of a light (?). Heat the paper slightly to see if the spot will disappear (?). Test different samples of cheese (melt a small piece and rub on paper) and milk for fats by this method.

b. *Proteins.* Place a small portion of the white of an egg (protein) in a test tube and heat it by dipping the tube into boiling water (?).

Pour 20 cc. of milk into an evaporating-dish and heat gently. Note the scum that collects on the surface. Remove the scum with a glass rod, collect other portions of the scum in the same way, and save for further tests.

Transfer a portion of the coagulated white of egg to a small beaker and moisten it with 2 or 3 drops of nitric acid (?). Wash the egg free from acid with repeated portions of water, then moisten it with ammonium hydroxide (?).

In the same way test for the presence of protein in the scum which separated when the milk was heated. Test other substances for protein, such as flour, woolen cloth, a clipping of a finger nail (?). Nitric acid stains the skin yellow (?).

Burn a small bit of different kinds of protein, such as egg, hair, the scum of milk (?).

EXERCISE 95

THE COMPOSITION OF FLOUR

Apparatus. Beaker (500-cc.); ring stand and burner; porcelain crucible; pipe-stem triangle.

Materials. 50 g. flour; iodine solution prepared in Exercise 54 for testing for starch; cheesecloth (from 12 to 15 cm. square); carbon tetrachloride (R.S.).

a. Weigh out about 25 g. of flour and mix with just enough water to form a stiff dough, working it in the hands until it becomes smooth and elastic. Place the dough on a piece of cheesecloth, then fold the cloth about the dough and tie it with a string so as to form a little bag. Nearly fill your largest beaker with water, immerse the bag and contents in the water, and repeatedly squeeze the bag between the fingers. Note that the water becomes cloudy. Retain a portion of this cloudy liquid for further tests (see ***b***, below).

Continue the washing in fresh portions of water until the resulting wash water remains clear. The action may be hastened by working the dough in a small stream of running water. Test for protein the residue remaining in the cloth (?). The substance is known as *gluten*; burn a portion of the gluten (?).

b. Test separate portions of the cloudy liquid reserved in ***a*** for starch (Exercise 54) and for sugar (Exercise 53) (?).

c. Fill a test tube about one-fourth full of flour, add carbon tetrachloride until the tube is half filled, and shake the contents vigorously. Set the tube aside until the flour settles, then pour off the clear liquid, evaporate it (Fig. 33), and test the residue, if any, for fat (?).

d. Place about 0.1 g. of flour in a porcelain crucible; heat it with the Bunsen flame (Fig. 16); gradually increase the heat until only a white residue remains (?).

EXERCISE 96

SOME EXPERIMENTS WITH MILK

Apparatus. Evaporating-dish; stirring-rods; ring stand and burner; 2 beakers.

Materials. 150 cc. milk; acetic acid (R. S.); Fehling's solution; one-half junket tablet (these tablets may ordinarily be obtained from any druggist; they may always be had at very little cost by addressing Chr. Hansen's Laboratory, Little Falls, New York).

a. *Determination of the solids and the water in milk.* Place a short stirring-rod in a small evaporating-dish and accurately weigh the two together. Introduce about 20 cc. of milk and again weigh. Now evaporate the milk to dryness (Fig. 33), occasionally stirring it with the rod to break the scum (?), which would otherwise retard evaporation. (While the evaporation is taking place, proceed with ***b***.)

When the residue is perfectly dry, carefully wipe the outside of the dish, cool, and reweigh. From your results calculate the percentages of solids and water in the milk. (Pure milk contains not less than 12 per cent of solid matter. Consult your state laws as to the minimum amount of solid matter allowed in milk sold in the state.)

b. *The separation of the protein (casein) in milk by rennin.* Dissolve about one half of a so-called "junket" tablet in a little cold water and add the solution to about 100 cc. of sweet milk heated until just lukewarm. Stir the solution, then cool it and set it aside until the rennin contained in the tablet causes the separation of the curd (casein). Break up the curd by stirring, and filter it. Save the filtrate for further tests.

c. *The separation of the protein in milk by acid.* Add 1 or 2 drops of acetic acid (vinegar will serve) to 5 cc. of milk, mix thoroughly, and set aside (?). The presence of an acid causes the casein to separate (hence the appearance of sour milk).

d. The separation of the lactose in milk. Evaporate the filtrate reserved in *b*, refiltering it if more solid matter separates. Taste the residue (?). Test it with Fehling's solution (Exercise 53) (?).

EXERCISE 97

METHODS FOR DISTINGUISHING BETWEEN BUTTER AND OLEOMARGARINE

Apparatus. Iron spoon; burner; wooden splint (match stick will do); 100-cc. beaker.

Materials. 10 g. each of fresh butter and oleomargarine (also process butter, if available); 50 cc. sweet milk.

a. Foam test. Heat gently over a small flame from 2 to 3 g. of the sample (butter or oleomargarine) in a spoon. Under these conditions butter will melt without sputtering and with the formation of much foam on the surface, while oleomargarine will sputter and give but little foam. Process, or renovated, butter (rancid butter which has been purified by melting the fat, skimming it off the surface, and churning it with milk under certain conditions) acts like oleomargarine.

b. Sweet-milk test. Pour 50 cc. of sweet milk into a small beaker and heat nearly to boiling. Add to the hot milk 4 or 5 g. of the sample and stir it with a wooden splint until the fat is melted, then place the beaker in ice water and continue the stirring until the fat solidifies. Under these conditions butter will solidify in the form of granules which mix with the milk. Oleomargarine, on the other hand, will collect in a single mass so that it can be removed from the milk in one lump with the wood stirrer.

EXERCISE 98

ANALYSIS OF BAKING-POWDERS

Apparatus. 100-cc. beaker; stirring-rod; evaporating-dish; ring stand and burner; 5 test tubes.

Materials. 10 g. each of various kinds of baking-powders; barium chloride solution (R.S.); sulfuric acid; nitric acid; ammonium molybdate solution (R.S.); sodium hydroxide solution.

a. Introduce 10 g. of a baking-powder into a beaker and pour over it 50 cc. of water. Stir the mixture thoroughly until no more gas is evolved, then filter it, and test the residue and the filtrate for the various ingredients, as explained below.

b. *Starch.* Will any starch present be in the residue or in the filtrate? Make appropriate test (?).

c. *Sulfates.* If the baking-powder contains alum, the filtrate will contain sulfates (?). Make appropriate test (?).

d. *Tartrates.* Pour 5 cc. of the filtrate into an evaporating-dish, add 5 drops of sulfuric acid, and evaporate to dryness. Finally, heat the dish gently with a bare flame. The presence of a tartrate is indicated by an odor similar to that of burning sugar.

e. *Phosphates.* If calcium phosphate is present in the baking-powder, the filtrate will contain calcium acid phosphate. To detect phosphates, treat 5 cc. of the filtrate with a few drops of nitric acid, heat nearly to boiling, add a few drops of the mixture to 5 cc. of ammonium molybdate solution (?) (compare Exercise 57).

f. *Ammonium salts.* Ammonium alum is sometimes used in baking-powders. To detect this, pour 5 cc. of the filtrate into a test tube, add an equal volume of sodium hydroxide solution, and heat gently. If ammonium salts are present, ammonia will be evolved (?).

g. Aluminium and calcium. A baking-powder containing sulfates always contains aluminium, while one containing phosphates always contains calcium; hence the presence or absence of these metals may be inferred from the tests made for sulfates and phosphates respectively.

EXERCISE 99

THE USE OF MORDANTS IN DYEING

Apparatus. 200-cc. beaker; ring stand and burner; stirring-rod; large beaker.

Materials. 2 strips (2 cm. × 6 cm.) of white woolen cloth (nun's veiling serves well); 6 strips of white cotton cloth (cheesecloth will do); 1 g. sodium carbonate in 50 cc. water; 0.5 g. tannic acid in 50 cc. water; 0.2 g. tartar-emetic (*b*, Exercise 63) in 50 cc. water; 0.1 g. of any of the following dyes in 100 cc. water (different students should select different dyes and compare results): fuchsine, methyl violet, Bismarck brown, malachite green; a solution containing 1 g. sodium carbonate, 5 g. Glauber's salt, and 0.1 g. Congo red in 50 cc. water.

a. Most dyes will dye animal fibers (wool, silk) directly, but will dye vegetable fibers (cotton, linen) fast only when mordants are used (p. 458 of text).

Place the strips of cotton cloth in a beaker and cover them with the sodium carbonate solution (1 g. in 50 cc. of water) and boil the liquid for five minutes. Remove the strips and thoroughly rinse them with water. This treatment serves to remove all foreign matter from the cloth.

Now *completely immerse* two strips of the cotton cloth in the tannic-acid solution, heat until it is fairly warm to the hand (50° or 60°), and maintain the temperature for ten minutes. Now remove the cloth from the solution and squeeze out the liquid, but do not rinse the cloth; then immerse the strips for one minute in the slightly warmed solution of tartar-emetic. The tannic acid in the cloth reacts with the tartar-emetic, forming a salt (known as antimonyl tannate) which becomes incorporated in the meshes of the fiber and serves as a mordant. Remove the cloth from the solution and rinse it.

Divide the solution of the dye chosen into three equal portions. Heat one portion to boiling, immerse a strip of *woolen* cloth, and continue the heating for from one to two minutes; then remove the cloth and rinse it thoroughly. To test whether the cloth is dyed fast, rinse it thoroughly and then wash it in a beaker of water and note whether the water becomes colored (?). In a similar way dye a strip of *unmordanted* cotton cloth in the second portion of the dye and a strip of *mordanted* cloth in the third portion, and determine in each case whether the cloth is dyed fast (?). Finally, dry the three strips of cloth and insert them in your notebook.

b. Some dyes (known as *substantive dyes*) have the property of dyeing cotton fast without the use of mordants. The so-called "Diamond Dyes for Cotton," which may be purchased at any drug store, belong to this class. Congo red is a typical dye of this class.

Heat the solution of Congo red to boiling, immerse in it a strip of *wet* unmordanted cotton, and continue the boiling for one or two minutes. Remove the cloth, rinse, and test to see whether the cloth is dyed fast. Dry the strip and insert it in your notebook. (The sodium carbonate and the sodium sulfate assist in the process, but do not act as mordants.)

(Save the remaining strips of cotton for use in Exercise 100.)

EXERCISE 100

A STUDY OF LAKES; ALSO THE EFFECT OF USING DIFFERENT MORDANTS WITH THE SAME DYE

Apparatus. Two 250-cc. wide-mouthed bottles; evaporating-dish; 2 small beakers; ring stand; burner; stirring-rods.

Materials. 5 cc. of a 20% solution of alizarin paste; solutions of aluminium sulfate and ferric sulfate (R. S.); ammonium hydroxide; 2 strips of cotton cloth.

a. *Formation of lakes.* Label two wide-mouthed bottles *A* and *B* respectively. Into each introduce about 10 drops of the alizarin paste. Next add to each of the bottles 2 cc. of ammonium hydroxide and then 200 cc. of water, and mix the contents thoroughly. Now add 10 cc. of aluminium sulfate solution to bottle *A*, and 10 cc. of ferric sulfate to bottle *B*, and set the bottles aside; note the appearance of the contents at the end of the laboratory period, also at the beginning of the next laboratory period (?). What is the function of each of the materials used?

b. *Mordanting strips of cloth with aluminium hydroxide and ferric hydroxide.* Pour about 20 cc. of aluminium sulfate solution into a small beaker and heat to boiling. Completely immerse in this solution one of the strips of cotton cloth prepared in Exercise 99, and continue the heating for from two to three minutes. Remove the cloth, squeeze it between the fingers to remove the excess of the solution, and immerse it in 20 cc. of water containing from 1 to 2 cc. of ammonium hydroxide. Warm the liquid slightly for two minutes, then remove the cloth and rinse it twice in water. What compound is now incorporated in the cloth?

Repeat the operation, using a solution of ferric sulfate in place of the aluminium sulfate. The resulting strip of cloth is mordanted with ferric hydroxide.

c. Dyeing the strips of mordanted cloth with alizarin. Pour 1 cc. of alizarin paste into a small beaker, add 20 cc. of water, stir, and heat to boiling. Completely immerse one of the strips of mordanted cloth prepared in ***b*** and continue the heating and stirring for three minutes; then remove the cloth and rinse thoroughly. Repeat the operation, using the second strip of mordanted cloth prepared in ***b***. Dry the dyed strips and insert them in your notebook.

EXERCISE 101

THE DETECTION OF DYES IN FOODS

Apparatus. Small beaker; stirring-rod; ring stand and burner.

Materials. Samples (colored) of pop and candy; strips of woolen cloth; hydrochloric acid.

Select different samples of colored pop. Pour 50 cc. of each into a beaker, add 2 or 3 drops of hydrochloric acid, and heat to boiling; then introduce a strip of woolen cloth and continue the heating for five minutes. Remove the cloth, rinse, and note the color (?).

Samples of colored candies may be tested by first dissolving the candy in water and then testing the solution for dyes by using strips of woolen cloth, as in the above case.

Tomato catchup is sometimes colored, although the practice is forbidden by Federal law. To test a catchup for dyes, heat a portion of the catchup diluted with water and immerse a strip of woolen cloth in the hot mixture for five minutes. Remove the cloth and rinse thoroughly. If artificial dyes are present, the cloth will be deeply colored; otherwise it will have only a slight brownish tinge produced by the natural coloring-matter of the tomato.

EXERCISE 102

THE REMOVAL OF STAINS

Apparatus. 4 test tubes; 2 beakers; evaporating-dish.

Materials. 0.2 g. tannic acid dissolved in 10 cc. water; ferric sulfate (R.S.); 2 pieces of white cloth 10 cm. square; 1 g. oxalic acid dissolved in 50 cc. water; hydrochloric acid; 2 strips of black cloth 5 cm. square; nitric acid; ammonium hydroxide; a few drops each of cottonseed oil and sirup (molasses); 25 cc. carbon tetrachloride; blotting-paper; strips of cloth stained with coffee and fruit juice; acetic acid (R.S.); 20 g. bleaching-powder; hot water.

a. Stain two strips of white cloth by dipping them into a solution of ferric sulfate until thoroughly saturated and then into a solution of tannic acid (or they may be stained directly with black ink). Wash one of the strips repeatedly with boiling water (?). Leave the other exposed to the air until dry; then try the effect of hot water upon the stain. If the stain is not removed, wash with a dilute solution of oxalic acid and finally with hot water (?).

b. Place 2 or 3 drops of dilute hydrochloric acid upon a strip of black cloth (?). Wash the spots with 10 cc. of water containing 4 or 5 drops of ammonium hydroxide (?). Repeat, using nitric acid in place of hydrochloric acid (?).

c. Place in separate test tubes 4 or 5 drops of a sirup and a like amount of fat, such as cottonseed oil. Test the solubilities of each in water and in carbon tetrachloride. Suggest a method for removing stains made by sirups and one for those made by fats. Stain some strips of cloth with sirup and with an oil and test your methods for removing these stains. (In applying a solvent it is convenient to place the stained portion of the cloth over a piece of blotting-paper (?). A small bit of sponge or cloth, saturated with the solvent, is then rubbed about the stained portion, gradually nearing the stain itself, which is finally thoroughly rubbed.)

Benzine (low-boiling gasoline) may be used in place of the carbon tetrachloride. *If benzine is used, however, it must be remembered that it is very inflammable. Never use it in the vicinity of a flame.*

d. Stain some strips of cloth with coffee and with fruit juices. Are the stains removed by washing with hot water? If the stain cannot be removed in this way, wash the stained portion of the cloth in bleaching-powder to which have been added some water and 3 or 4 drops of acetic acid.

APPENDIX B

TABLE OF CONSTANTS

LIST OF THE COMMON ELEMENTS, THEIR SYMBOLS, AND THEIR ATOMIC WEIGHTS

O = 16

Aluminium	Al	27.1	Iodine	I	126.92
Antimony	Sb	120.2	Iron	Fe	55.84
Arsenic	As	74.96	Lead	Pb	207.2
Barium	Ba	137.37	Magnesium	Mg	24.32
Bismuth	Bi	208.0	Manganese	Mn	54.93
Boron	B	11.0	Mercury	Hg	200.6
Bromine	Br	79.92	Nickel	Ni	58.68
Cadmium	Cd	112.4	Nitrogen	N	14.01
Calcium	Ca	40.07	Oxygen	O	16.00
Carbon	C	12.00	Phosphorus	P	31.04
Chlorine	Cl	35.46	Potassium	K	39.1
Chromium	Cr	52.0	Silicon	Si	28.3
Cobalt	Co	58.97	Silver	Ag	107.88
Copper	Cu	63.57	Sodium	Na	23.0
Fluorine	F	19.0	Sulfur	S	32.06
Gold	Au	197.2	Tin	Sn	118.7
Hydrogen	H	1.008	Zinc	Zn	65.37

TENSION OF AQUEOUS VAPOR AT VARIOUS TEMPERATURES, EXPRESSED IN MILLIMETERS OF MERCURY

Temperature	Pressure	Temperature	Pressure
16°	13.62	21°	18.62
17°	14.4	22°	19.79
18°	15.46	23°	21.02
19°	16.45	24°	22.32
20°	17.51	25°	23.69

WEIGHT IN GRAMS OF 1 LITER OF VARIOUS GASES MEASURED UNDER STANDARD CONDITIONS

Acetylene	1.1621	Hydrogen sulfide	1.5392
Air	1.2928	Methane	0.7168
Ammonia	0.7708	Nitric oxide	1.3402
Carbon dioxide	1.9768	Nitrogen	1.2507
Carbon monoxide	1.2504	Nitrous oxide	1.9777
Chlorine	3.1674	Oxygen	1.4290
Hydrogen	0.08987	Sulfur dioxide	2.9266
Hydrogen chloride	1.6398		

THE METRIC SYSTEM

This system is now used in all civilized countries with four or five exceptions. The United States and Great Britain are among the few countries that have not formally adopted it, but even in these countries the system is universally used by scientists and is coming into use more and more by manufacturers.

In the metric system each unit is 10 times as large as the next lower unit; hence the system is often termed the "decimal system."

1. Length. The unit is the *meter.* It is equal to 39.37 inches.

10 millimeters (mm.) = 1 centimeter (cm.)
10 centimeters = 1 decimeter (dm.)
10 decimeters = 1 meter (m.)
1000 meters = 1 kilometer (km.)

The only measures of length ordinarily used by the chemist are the millimeter and the centimeter; thus, the height of the barometer at the sea level is recorded as 76 cm. (or more commonly as 760 mm.), and not 7 dm. and 6 cm.

2. Volume. The unit generally used is the *cubic centimeter.*

1000 cubic millimeters = 1 cubic centimeter (cc.)
1000 cubic centimeters = 1 cubic decimeter = 1 liter
1000 cubic decimeters = 1 cubic meter

The chemist uses only the cubic centimeter and the liter as measures of volume. Thus, the volume of a test tube is given as (say) 25 cc.; that of a flask as (say) 500 cc., or ½ liter.

3. Weight. The unit is the *gram.* This is approximately the weight of 1 cc. of pure water at its temperature of greatest density (4°). It is equal to 15.43 grains.

10 milligrams (mg.) = 1 centigram (cg.)
10 centigrams = 1 decigram (dg.)
10 decigrams = 1 gram (g.)
1000 grams = 1 kilogram (kg.)

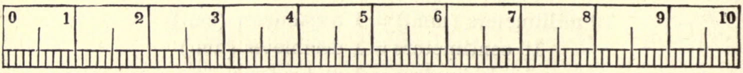
The gram and kilogram are the units of weight most generally used by the chemist. Thus, the weight of a crucible is given as (say) 10.532 g. and not 10,532 mg. or 10 g. 5 dg. 3 cg. 2 mg.

Note that

1 pound troy = 5760 grains = 373.24 grams.
1 pound avoirdupois = 7000 grains = 453.59 grams.
1 ounce avoirdupois = 28.35 grams.
1 U.S. liquid quart = 946.36 cubic centimeters.

Also note that

1 centimeter = nearly $\frac{2}{5}$ inch.
1 meter = nearly 1.1 yards.
1 kilogram = nearly $2\frac{1}{5}$ pounds avoirdupois.



TEN-CENTIMETER SCALE

TABLE OF SOLUBILITIES OF SOME OF THE COMPOUNDS OF THE METALS

	ACETATE	BROMIDE	CARBONATE	CHLORATE	CHLORIDE	CHROMATE	HYDROXIDE	IODIDE	NITRATE	OXIDE	PHOSPHATE	SILICATE (ORTHO)	SULFATE	SULFIDE
Aluminium . . .	W	W		W	W		A	W	W	A	A	A	W	A
Ammonium . . .	W	W	W	W	W	W	W	W	W		W		W	W
Barium	W	W	A	W	W	A	W	W	W	A	A	A	I	W
Calcium	W	W	A	W	W	X	W	W	W	X	A	A	X	X
Cobalt	W	W	A	W	W	A	A	W	W	A	A	A	W	A
Copper	W	W	A	W	W	W	A	W	W	A	A	A	W	A
Ferric	W	W		W	W	W	A	W	W	A	A	A	W	A
Ferrous	W	W	A	W	W		A	W	W	A	A	A	W	A
Lead	W	X	A	W	X	A	A	X	W	A	A	A	I	A
Magnesium . . .	W	W	A	W	W	W	A	W	W	A	A	A	W	A
Manganese . . .	W	W	A	W	W	W	A	W	W	A	A	A	W	A
Mercuric	W	W	A	W	W	X		A	W	A	A		X	I
Mercurous	W	A		W	A	A		A	W	A	A		X	
Nickel	W	W	A	W	W	A	A	W	W	A	A	A	W	A
Potassium	W	W	W	W	W	W	W	W	W	W	W	W	W	W
Silver	W	I	A	W	I	A		I	W	A	A		X	A
Sodium	W	W	W	W	W	W	W	W	W	W	W	W	W	W
Stannic	W	W			W		A	W		A	A			A
Stannous	W	W			W	A	A	W		A	A		W	A
Zinc	W	W	A	W	W	W	A	W	W	A	A	A	W	A

W, soluble in water.

A, insoluble in water, soluble in either HCl or HNO_3 or in both.

I, insoluble in water and in acids.

X, slightly soluble in water and slightly or readily soluble in acids.

Every laboratory should be supplied with the materials necessary for the treatment of cuts and burns. Such wounds when immediately and intelligently treated give little or no trouble, but if left to take care of themselves infection may occur and serious results follow. In case of severe wounds, the student, after the preliminary treatment in the laboratory, should be sent to a physician.

The following materials required for the treatment of wounds should be kept in the laboratory in a tight cabinet or cupboard:

1. Plain, sterile gauze bandage: 6 rolls, 1 inch by 10 yards; 6 rolls, 2 inches by 10 yards.
2. Adhesive plaster for holding gauze in place: 2 rolls, 0.5 inch by 10 yards.
3. 1000 cc. of benzine (low-boiling gasoline).
4. 2000 cc. of Seiler's solution (this may be made from tablets purchased at any drug store).
5. Boric acid solution prepared by dissolving 20 g. of boric acid in 1000 cc. of water.
6. Iodine solution — ordinary tincture of iodine; may be purchased at any drug store.
7. 100 cc. of Agnew's solution prepared after the following prescription:

Tannic acid	10 grains
Borax	10 grains
Glycerine	1 drachm
Camphorated water	q.s. to make 1 ounce

Different wounds should be treated as follows:

a. *Cuts from glass.* Remove any dirt or grease from the wound and surrounding skin by washing with gauze saturated with benzine. A rather free bleeding at first will help to prevent infection. Finally wash the wound with a piece of gauze saturated with the iodine solution; then bandage so as to prevent contamination.

b. *Burns.* Apply gauze saturated with Seiler's solution and then bandage.

c. *Burns from acids.* Treat the same as ordinary burns, as directed under ***b.***

d. Burns from alkalies. Wash with boric acid solution on gauze and then bandage.

e. Acid in eyes. Wash the eyes with a large volume of water, then with a solution of boric acid. Finally add a few drops of Agnew's solution and apply to the eyes a small gauze pad which is kept saturated with ice water.

INFORMATION REGARDING APPARATUS AND CHEMICALS

The lists following include the apparatus and chemicals required for the experiments in this notebook. It is always best to furnish each student with as complete an outfit as possible and to hold him responsible for the same. Certain pieces may, however, be used in common by a number of students, and these have been placed in a separate list. It is always cheapest to purchase the apparatus and chemicals in as large quantities as possible. The amounts of most of the chemicals needed for a class of ten are so small that the cost of the same will be proportionately much greater than when larger quantities are ordered. *It is always best to order the definite amounts of chemicals listed in the catalogues, such as 100 g. or 1 lb.; otherwise the cost of weighing out odd quantities and preparing these for shipment may amount to more than the cost of the chemicals.* The supplies may be obtained from any of the large dealers. Catalogues will be sent on application and should be in every school. The following are the addresses of some of the largest firms:

Central Scientific Company, 412 Orleans St., Chicago, Ill.
Chicago Apparatus Co., 32 Clinton St., Chicago, Ill.
Kauffman-Lattimer Co., Columbus, Ohio.
L. E. Knott Apparatus Co., Boston, Mass.
Eimer and Amend, 205 Third Avenue, New York City.
Standard Scientific Co., 70 Fifth Avenue, New York City.
Scientific Materials Co., 711–719 Forbes St., Pittsburgh, Pa.
Arthur H. Thomas Company, Philadelphia, Pa.
E. H. Sargent & Co., 145 Lake St., Chicago, Ill.
C. H. Stoelting Co., 3037 Carroll Avenue, Chicago.
The Will Corporation, Rochester, New York.

A list of the supplies needed should be sent to a number of firms for quotations on prices. In ordering any piece of apparatus a certain form in some catalogue should be designated; otherwise it will be impossible to compare the prices. In general it is best to purchase as simple a form of apparatus as possible; for example, 20 cents will buy a Bunsen burner which for ordinary purposes is preferable to those costing $1. A much higher price will have to be paid for small orders placed for immediate delivery. A person experienced in the purchase of supplies will always find it possible to reduce materially the cost of the order.

APPARATUS REQUIRED FOR EACH STUDENT (TO BE KEPT IN STUDENT'S LOCKER)

Beakers, nest of 7 (100-cc. to 700-cc.).
Blowpipe, mouth.
Bottle, narrow-necked, 1000-cc. (Fig. 25, *B*).
Bottles, wide-mouthed: 1 (60-cc.) (Fig. 38, *A*); 5 (250-cc.) (Fig. 20).
Burner, wing-top, for bending glass tubing (Fig. 4).
Calcium chloride drying-tube, straight, 15 cm. in length (Fig. 23, *B*).
Charcoal, 1 piece 8 cm. × 3 cm.
Deflagrating-spoon.
Dish, lead, diameter about 6 cm., depth 3 cm.
Evaporating-dish, diameter 7 cm.
File, round, about 15 cm. in length.
File, triangular, about 15 cm. in length.
Filters, 25, diameter about 11 cm.
Flasks: 2 (250-cc.); 1 (500-cc.).
Funnel, diameter about 6.5 cm.
Funnel tube, external diameter of tube 6 mm.
Glass rod, diameter 3 mm., 1 piece 15 cm. in length.
Glass rod, 1 piece 10 cm. in length.
Glass tubing, 200 g., soft, external diameter 6 mm., walls 1 mm. thick.
Glass tubing, hard, internal diameter 1 cm., 1 piece 30 cm. in length.
Glass tubing, hard, internal diameter 6 mm., 1 piece 20 cm. in length.
Mortar (diameter about 8 cm.) and pestle (both of porcelain).
Pipe-stem triangle, for holding porcelain crucible (Fig. 16).
Platinum wire, small (No. 28), 8 cm. long, for flame tests.
Porcelain crucible and lid, diameter about 3.5 cm.

Retort, glass-stoppered, 150-cc. (Fig. 40).
Rubber tubing, internal diameter 6 mm., 1 piece 50 cm. in length.
Rubber tubing (soft), pure gum, internal diameter 5 mm., 1 piece 60 cm. in length, for connections, etc.
Screw clamp (Fig. 19, *A*).
Splints, wooden (ordinary cigar lighters), 125.
Sponge.
Stopper, rubber, one-hole, to fit hard-glass test tube.
Stopper, rubber, two-hole, to fit wide-mouthed 250-cc. bottle.
Stopper, rubber, two-hole, to fit wide-mouthed 60-cc. bottle.
Stopper, rubber, two-hole, to fit 1000-cc. narrow-mouthed bottle.
Test tube, graduated, 30-cc., about 20 cm. long, with 0.5-cc. graduations.
Test tube, hard glass, 15 cm. in length, diameter about 1.8 cm.
Test tubes, 12, length 12 cm., diameter about 1.7 cm.
Test-tube brush.
Test-tube rack.
Towel.
Watch glass, diameter about 8 cm.
Window glass, 4 pieces 10 cm. square.
Wire gauze, 2 pieces 12 cm. square.

APPARATUS TO BE LEFT ON EACH DESK

Bunsen burner, with 75 cm. of rubber tubing to fit.
Clamp, iron, large, for holding flasks and condensers.
Iron tripod (Fig. 33).
Pneumatic trough (Fig. 21). The trough should be about 12 to 15 cm. deep, and large enough to hold 4 or 5 wide-mouthed bottles (250-cc.). It may be round or rectangular. A pan made of granite ware or an earthen crock serves well, or any tinsmith can readily make suitable troughs of galvanized iron.
Ring stand and 3 rings.

REAGENTS ON EACH DESK

250-cc. bottles filled with the reagents named below. The bottles containing the sodium hydroxide should have ordinary corks, the others should be glass-stoppered.
Ammonium hydroxide (density 0.90).
Hydrochloric acid (density 1.2).
Nitric acid (density 1.4).

Sodium hydroxide solution (10 g. in 100 cc. of water).
Sulfuric acid (density 1.84).

GENERAL APPARATUS FOR TEN STUDENTS

2 sets apparatus for testing conductivity of solutions (Fig. 34); this may be purchased of supply houses or can easily be constructed.
1 balance, weighing from 0.5 g. to 500 g., with accompanying weights.
2 balances, sensitive to 1 cg. and made to carry a load of 100 g.
1 barometer.
1 bottle or flask, 2000-cc. (Fig. 47, *A*).
4 burettes, 50-cc., graduated in 0.1 cc. (Fig. 37).
5 calcium chloride tubes (Fig. 28, *D*).
5 pieces cobalt glass 10 cm. square (for flame tests).
4 condensers, Liebig (Fig. 26, *B*), with rubber tubing and large clamp.
2 sets cork-borers (6 in a set).
2 gross corks, best grades, sizes 7, 8, 9, 10, and 12.
1 cylinder, glass, about 30 cm. in length and 4 to 5 cm. in width.
1 cylinder, graduated, 200-cc.
1 cylinder, graduated, 500-cc.
1 distilling apparatus for preparing distilled water.
2 flasks, holding 100 cc. when filled to point marked on neck (Fig. 49, *C*).
1 flask, graduated to hold 1000 cc.
5 pieces hard-glass tubing 20 cm. long and 15 mm. internal diameter (Fig. 28, *C*).
1 hydrometer, reading from 0.900 to 1.000.
1 magnifying glass, small.
1 microscope, eyepiece 1 inch, objective $\frac{2}{3}$ and $\frac{1}{6}$.
5 thermometers, graduated from − 10° to + 150° C.
2 tubes, graduated in cubic centimeters, 50 cm. in length and about 2 cm. in width.
2 sets weights, 1 cg. to 50 g., in covered wooden box.

CHEMICALS ON REAGENT SHELF (FOR USE OF ALL STUDENTS)

The bottles containing solutions should be glass-stoppered. Gummed letters of the alphabet, of different sizes, may be obtained at little cost from any stationer, and these may be used in making the labels for the bottles. If the class is small, bottles holding 250 cc. will ordinarily serve; if the class is large, then it is better to use bottles holding at least 500 cc. A few of the reagents, such as limewater, are used so extensively that it is better to use a 1000-cc. bottle. Distilled water must be used in making all solutions. A 10% solution signifies 10 g. dissolved in 100 cc. of water.

Acetic acid (36%).
Alcohol (95%).
Aluminium sulfate (10% solution).
Ammonium carbonate (25 g. of the solid dissolved in 70 cc. of water and 10 cc. of ammonium hydroxide (density 0.90) and the solution diluted to 100 cc. with water).
Ammonium chloride (10% solution).
Ammonium molybdate solution.
Ammonium sulfide solution.
Barium chloride (10% solution).
Borax (solid).
Calcium chloride (10% solution).
Carbon tetrachloride.
Chlorine water (water saturated with chlorine. Must be freshly prepared as needed).
Cobalt nitrate (5% solution).
Copper sulfate (10% solution).
Disodium phosphate (10% solution).
Ferric chloride (10% solution).
Ferric sulfate (10% solution).
Lead acetate (10% solution).
Limewater (saturated solution of calcium hydroxide).
Magnesium sulfate (10% solution).
Manganese chloride (10% solution).
Mercuric chloride (5% solution).
Mercurous nitrate (10% solution).
Oxalic acid. Standard solution for Exercise 93. Prepare by dissolving exactly 15 g. c. p. oxalic acid ($C_2H_2O_4 \cdot 2\,H_2O$) in 1000 cc. of water.
Phenolphthalein (1 g. dissolved in 200 cc. of alcohol).
Potassium bromide (10% solution).
Potassium chromate (10% solution).
Potassium ferricyanide (10% solution).
Potassium ferrocyanide (10% solution).
Potassium hydroxide (10% solution).

Potassium iodide (10% solution).
Potassium sulfocyanide (10% solution).
Silver nitrate (4% solution).
Soap (1% solution).
Sodium carbonate (10% solution).
Sodium chloride (solid).
Sodium peroxide (solid).
Starch solution (prepared by rubbing to a paste 4 or 5 g. of starch with cold water, and then adding, 3 or 4 drops at a time and with stirring, to 1 liter of boiling water. Add also about 10 g. of zinc chloride (this acts as a preservative). Mix thoroughly, set the mixture aside, and use the clear supernatant liquid).
Tartar-emetic (1% solution).
Zinc acetate (10% solution).

CHEMICALS REQUIRED FOR A CLASS OF TEN

The terms in parentheses after the names of the chemicals refer to the grade of materials to be purchased. The abbreviation c. p. signifies "chemically pure."

	APPROXIMATE AMOUNTS
Acid, acetic (36%) (c. p.)	500 g.
Acid, formic (50%)	500 g.
Acid, hydrochloric (density 1.2) (c. p.)	2.5 kg.
Acid, nitric (density 1.4) (c. p.)	3 kg.
Acid, oxalic (c. p.)	200 g.
Acid, pyrogallic	100 g.
Acid, sulfuric (density 1.84) (c. p.)	4 kg.
Acid, tannic (commercial)	100 g.
Alcohol, ethyl (95%)	1 kg.
Alcohol, methyl	250 g.
Alum (ammonium) (pure)	500 g.
Aluminium (turnings or filings)	50 g.
Aluminium sulfate (pure, crystals)	500 g.
Ammonium carbonate (pure)	100 g.
Ammonium chloride (pure)	200 g.
Ammonium hydroxide (density 0.90) (c. p.)	1.5 kg.
Ammonium molybdate solution	500 g.
Ammonium nitrate (pure)	100 g.
Ammonium sulfate (commercial)	500 g.
Ammonium sulfide solution	500 g.

Antimony	30 g.
Arsenic	30 g.
Arsenic trioxide (arsenious oxide) (commercial)	30 g.
Barium chloride (c. p.)	100 g.
Benzene	200 g.
Bismuth	30 g.
Bleaching-powder	500 g.
Bone black	250 g.
Borax (commercial)	500 g.
Boric acid (pure)	100 g.
Cadmium	25 g.
Cadmium chloride (c. p.)	25 g.
Calcium carbide	500 g.
Calcium carbonate (precipitated)	500 g.
Calcium chloride (fused or granular)	1 kg.
Calcium fluoride (fluorspar)	200 g.
Calcium hydroxide (hydrated lime)	500 g.
Calcium sulfate (plaster of Paris)	2 kg.
Carbon disulfide (commercial)	250 g.
Carbon tetrachloride (commercial)	500 g.
Chloroform	250 g.
Cobalt nitrate (pure)	25 g.
Copper (turnings or scrap)	250 g.
Copper foil (thin)	100 g.
Copper nitrate (pure)	25 g.
Copper oxide (black, ordinary)	100 g.
Copper sulfate crystals (c. p.)	200 g.
Cottonseed oil	200 g.
Dyes (Bismarck brown, fuchsine, methyl violet, malachite green, Congo red)	10 g. of each
Alizarin paste (20%)	100 g.
Ether, sulfuric	500 g.
Formalin	100 g.
Gelatin	50 g.
Glucose (sirup) or Karo corn sirup	1 kg.
Gypsum (crystals)	200 g.
Hydrogen peroxide	200 g.
Iodine	25 g.
Iron chloride (ferric) (c. p.)	100 g.
Iron powder (iron reduced by alcohol)	150 g.
Iron sulfate (ferrous)	200 g.
Iron sulfide	1 kg.

Iron wire (picture-frame wire), No. 0	25 yds.
Junket tablets	10 tablets
Lead, red	25 g.
Lead acetate (sugar of lead) (powdered)	200 g.
Lead monoxide (commercial)	150 g.
Lead nitrate (pure)	50 g.
Litmus cubes	50 g.
Litmus paper — 100 strips red, 100 strips blue	1 tube of each
Magnesium carbonate (powdered)	100 g.
Magnesium sulfate (Epsom salts)	500 g.
Magnesium wire or ribbon	25 g.
Manganese chloride	50 g.
Manganese dioxide (commercial)	1 kg.
Marble (pieces size of a walnut)	3 kg.
Mercuric chloride (corrosive sublimate)	25 g.
Mercuric nitrate (c. p.)	25 g.
Mercuric oxide	25 g.
Mercurous nitrate (c. p.)	100 g.
Mercury	50 g.
Nickel nitrate	25 g.
Paraffin	500 g.
Phenolphthalein	25 g.
Phosphorus	50 g.
Potassium bitartrate (cream of tartar)	100 g.
Potassium bromide (granular, pure)	50 g.
Potassium carbonate (c. p.)	50 g.
Potassium chlorate (small crystals)	500 g.
Potassium chloride (c. p.)	100 g.
Potassium chromate (pure, crystals)	100 g.
Potassium chromium sulfate (chrome alum)	100 g.
Potassium dichromate (pure)	100 g.
Potassium ferricyanide (c. p.)	100 g.
Potassium ferrocyanide (c. p.)	100 g.
Potassium hydroxide (sticks, electrolytic)	200 g.
Potassium iodide (pure)	50 g.
Potassium nitrate (pure)	200 g.
Potassium permanganate (pure)	50 g.
Potassium sulfate (pure, anhydrous)	200 g.
Potassium sulfocyanide (c. p.)	25 g.
Silver nitrate	25 g.
Soda lime (granular)	500 g.
Sodium	100 g.

Sodium acetate (fused)	200 g.
Sodium benzoate (pure)	25 g.
Sodium bicarbonate (baking-soda)	500 g.
Sodium carbonate (pure, anhydrous)	250 g.
Sodium hydrogen phosphate (disodium phosphate) (c. p.)	100 g.
Sodium hydroxide (sticks, electrolytic)	1 kg.
Sodium nitrate (pure)	200 g.
Sodium peroxide	200 g.
Sodium potassium tartrate (Rochelle salts) (powdered)	500 g.
Sodium silicate solution (water glass)	1 kg.
Sodium sulfate (crystals)	200 g.
Sulfur	500 g.
Tartar-emetic (potassium antimonyl tartrate)	75 g.
Tin (granulated)	75 g.
Zinc (granulated, arsenic-free)	1 kg.
Zinc (sheet)	200 g.
Zinc acetate (c. p.)	25 g.
Zinc chloride	100 g.
Zinc sulfate (crystals)	100 g.

NOTE. This does not include substances always easily obtained, such as sugar, salt, lard, lime, clay, coal, cotton, iron wire, calico strips, lead (pieces cut from lead pipe), soap, kerosene, gasoline, candles, cardboard, starch, cloth, milk, vinegar, butter, etc.