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PREFACE

THIS manual is intended to accompany "Chemistry, an Elementary Textbook," by the same authors, but it may be used advantageously in connection with any other text. It will prove interesting to teachers who wish to present that kind of chemistry which appeals to students because of its intense human interest.

The work outlined is intended for classes having eight to ten periods (approximately 45 minutes each) per week to devote to the subject. It is realized that this is more than can be given in all schools, so that a choice will sometimes be necessary. Yet the better students need not be tied down to the amount of work required of the slower members. Where the allotted time is too short for each student to perform all the work, it will prove interesting if some students perform one experiment while others are doing another, both to be reported on in the classroom later. An abundance of material from which to choose is preferable to a minimum which must be amplified.

The beginning student should be given an extended experimental introduction to the subject of chemistry, inasmuch as its methods of approach are unfamiliar to him and many of its facts beyond his personal experience. Hence, more time should be devoted to experimental work at the beginning of the course than later.

Every teacher realizes that good lecture table experiments are quite as important as any feature of the course. There are included, therefore, a goodly number of experi-

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ments intended to be performed by the instructor while the class takes careful notes. These are generally too difficult for the average student, yet they cover points of such importance in the development of the science that students should see these demonstrations.

The following experiments should be required of all students: 1, 2, 5, 10, 18, 19, 21, 22, 24, 26, 28, 29, 30, 34, 36, 39, 41 or 42, 44, 46, 49, 50, 51, 52, 53, 54, 57, 58, 59, 61, 64, 65, 67, 69, 72, 73. As many others should be performed as time will permit.

Suggestions for the improvement of this manual from teachers who have used it with classes will be appreciated by the authors.

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W. C. MORGAN. J. A. LYMAN.

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SUGGESTIONS TO TEACHERS

IN the following experiments the shelf reagents are supposed to be about 10 per cent solutions; say 100 g. of the solute in a liter of water. Concentrated reagents are of the specific gravities giver. below and the dilute reagents are made as specified.

Hydrochloric acid, Sp. Gr. 1.19. Pour into 4 volumes of water.

Nitric acid, Sp. Gr. 1.42. Pour into 5 volumes of water. Sulfuric acid, Sp. Gr. 1.84. Pour into 6 volumes of water.

Ammonium hydroxid, Sp. Gr. 0.90. Pour into 4 volumes of water.

A pneumatic trough may be made of a deep baking pan and a single muffin tin with two round holes cut in it by any tinsmith, as shown in Fig. A. Painted with asphalt varnish they will last forever. FIG. A.

Capsules for sodium may be purchased, or made by cutting $\frac{3}{8}$ -inch brass tubing (wall $\frac{1}{32}$ inch) into 1-inch lengths and soldering a disk of brass air-tight on to one end. A 10-inch piece of copper wire, one end wrapped several times firmly about the capsule, serves as a holder.

Rubber stoppers are much better than corks. Only best quality rubber goods should be purchased; the poorer grades oxidize quickly and become hard. Good rubber stoppers that have become hard may be softened by boiling in a 10 per cent NaOH solution. To cut glass tubing of large diameter, make a short scratch with a file. Fold a filter paper so that its edges are parallel and moisten it. Wrap such a piece about the tube each side of the mark, leaving a space about $\frac{1}{8}$ of an inch wide exposed. Heat this space in a blowpipe with a fine flame, rotating the tube until it breaks; or touch the hot ring with a drop of water.

To avoid repeated weighings on the part of students (see Exp. 22) make small measures out of glass tubing of appropriate size sealed at one end. Weigh out 1 g. (if this is the desired amount) of the substance, insert in the tube, and jar it until the powder lies in the closed end. Cut off that part of the tube filled by the powder, affix to a wooden splint by means of glue and tough paper, and a serviceable measure has been made.

Magnesium ribbon usually runs uniform and can be measured more accurately than weighed by students (see Exp. 41).

The larger schools will find it very convenient to keep in the laboratory three Kipp generators, one fitted for hydrogen, one for hydrogen sulfid, and one for carbon dioxid. An oxone generator for oxygen is very handy.

If a gas holder is not available, a satisfactory method of handling small volumes of gas is illustrated in Exps. 47 and 59.

It is advisable to keep on hand in the laboratory a bottle containing olive oil and lime water (equal parts) to be used on burns. Shake thoroughly before applying to the burn and bandage to keep from the air. A dilute solution of baking soda may be substituted for the lime water.

SUGGESTIONS TO STUDENTS

WHEN you enter the laboratory, learn the names of the apparatus and see that you have the complete list. If not, report it to the instructor.

Wash your apparatus at the beginning and keep it scrupulously clean and your desk neat. Slovenly work causes experiments to go wrong, necessitating repetition. When work is completed for the day, put away the apparatus and clean the desk.

Vessels of *thin* glass may be heated with safety. With thick glass, uneven heating causes irregular expansion

and breaking. Always place a wire gauze between a beaker or flask and the flame. Test tubes and porcelain may be heated in the flame. Never heat glass containing a liquid at the surface of the liquid. It is sure to break. Heat as shown in Fig. *B*, using a strip of paper several times folded as a substitute for a test tube holder, if necessary. Shake the test tube continually while heating to prevent explosive boiling.



Direct the mouth of the test tube away from yourself and your neighbor as well.

Before attempting to insert glass into rubber, wet both. Don't push or pull, but *twist* a glass tube into position in a rubber stopper, thus avoiding cuts from broken glass.

Before boring, soften a cork by rolling under the hand or foot. Select a borer one size smaller than the tube to be fitted. Place the cork against a board and with a gentle pressure rotate it until it cuts neatly through. Do not attempt to push it through, as this makes ragged holes.

Be sparing in the use of reagents. Too much is often as detrimental to good work as too little. If too much of a liquid reagent has been removed, throw the excess away. Never pour it back into the bottle. The quantities of reagents specified are usually only approximate and meant to be estimated rather than weighed or measured. Learn to know how much 1 and 5 g. of ordinary substances are. An ordinary test tube $(6 \times \frac{3}{4} \text{ inches})$ holds 30 ccm. Learn what 5 and 10 ccm. are.



Never lay the stopper of a reagent bottle down on the shelf or desk, thus contaminating it with other substances. In obtaining reagents from bottles, turn the palm of the hand up and remove the stopper by grasping between the fingers and holding as shown in Fig. C.

Never taste any reagent unless definitely told to do so, for most chemicals are poisons. In smelling, waft the fumes toward the nose with the hand.

Dilute acids and solutions of reagents are always meant unless concentrated acids or solid reagents are specified.

Always place the substance to be weighed on a piece of paper on the balance, never on the balance pan itself.

To insert a powder neatly into a test tube, place it on one end of a narrow piece of paper folded lengthwise. Insert the paper into the test tube, turn upright and jar the powder from the paper.

Throw only liquids in sinks; all paper and other solids in jars or boxes. After pouring acids into sinks, flush out with water.

All temperatures are Centigrade; volumes and weights are in Metric Measures, familiarity with which is presupposed on the part of all students.

(Hood) means perform under a hood.



PRELIMINARY EXERCISES

THE BUNSEN BURNER

Apparatus. Bunsen Burner and about 60 cm. of rubber tubing. Ring stand or tripod. Wire gauze. Beaker.

Note. This burner was first made by the distinguished German scientist, Bunsen. Probably no other invention has done so much to facilitate work in the laboratory.

Procedure. (a) The parts of the burner and their uses.

Unscrew the tube of the burner, and notice the various parts. Then put the parts together again, using a little oil if the joints appear rusted, and connect the burner to the gas pipe by means of the rubber tubing. Having turned on the gas, light it by holding a burning match close to the burner tube about 1 cm. *below* the top.

Turn the ring near the base of the burner until its openings coincide with the openings in the tube. This allows air to mix with the gas before the latter reaches the flame above. Turn the ring until the air is shut off. What changes are brought about in the flame by varying the supply of air? Ascertain by means of a wire or glass rod which flame is hottest and which deposits soot. Which flame is most suitable for laboratory purposes? Hold the wire in different parts of the blue flame. Which is the hottest part of the flame?

While the flame is burning blue, hold a lighted match against one of the holes. The flame "strikes back" and

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FIG. 1.

burns at the base. Notice the odor, the character of this flame and the temperature of the burner tube. This flame is unsuitable for laboratory purposes. If a burner during use strikes back, the indication is that there is too much air mixed with the gas. To prevent the trouble, turn on more gas or shut off some of the air. If a burner strikes back, a sharp blow with the fist on the rubber tubing will usually restore the

flame to its proper condition. If not, the gas must be shut off, after which it may be lighted again.

(b) The use of wire gauze.

Place the wire gauze on the tripod, and set the lighted Bunsen burner below the gauze. Notice how the gauze moderates the heat by distributing it over a larger surface, due to the fact that metals are good conductors of heat. A beaker of water may be boiled on the hottest part of the gauze without danger of breaking (see Fig. 1).

THE MANIPULATION OF GLASS TUBING

Materials. Glass tubing, outside diameter about 5 mm. Rubber tubing, inside diameter a little less than the outside diameter of the glass tubing.

Apparatus. A sharp, three-cornered file. A Bunsen burner and wing top (Fig. 2); or a common fishtail gas burner.

FIG. 2.

Procedure. (a) To cut glass tubing.

Lay a piece of glass tubing on the table, and at the place where it is to be cut make a scratch with the file. Then grasp the tube firmly with a hand on either side of

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PRELIMINARY EXERCISES

the mark, and press with the tips of the thumbs opposite the scratch. The tube will usually break evenly across.

(b) To smooth the end of a glass tube.

Notice that the broken end of a glass tube is very sharp and would cut any soft object with which it comes in contact. Round off the sharp edges by heating in a Bunsen flame, rotating the tube until the glass begins to melt slightly. Where it is not desirable to heat the tube, the sharp ends may be smoothed by sandpaper or by the flat side of a file.

(c) To seal glass tubing.

Heat a piece of glass tubing about 30 cm. long about 5 cm. from one end in the flame of the Bunsen burner, rotating it so that it may be evenly heated. When it becomes very soft, gently draw it out as shown in Fig. 3. Fig. 3. Allow the tube to cool, then scratch with a file at the points A and B, and break as previously directed. Hold the narrow end of the longer piece in the flame, rotating it until the edges melt together and the end is closed. Repeat the drawing out, cutting, and scaling on the other

(d) To bend glass tubing.

Hold a piece of glass tubing lengthwise in the flattened

end of the tube and you have made a stirring rod.



FIG. 4.

flame of a fishtail burner or Bunsen burner with wing top, as shown in Fig. 4, so that about 5 cm. of the tubing is heated where the bend is to be. Rotate the tube so that it may be evenly heated. When it is so hot that it bends readily, remove it from the flame and immediately bend it into the desired shape, holding it in that position until the glass hardens. If the Bunsen burner has been used, turn off the supply of air, and by holding the tube in the yellow flame allow the bend to become covered with soot. This allows the tube to cool slowly, thus preventing unequal strains in the glass. This slow cooling, called "annealing," makes the glass less liable to break.

If the bend is not smooth and even like Fig. 5 A, the tubing was not heated through a sufficient length, resulting in flattening, B; or else it was not heated uniformly,



producing kinked and unsightly bends which usually break readily, C. Never attempt to make a bend by using the ordinary flame of the Bunsen burner.

If a bend is needed near the end of a piece of tubing, make the bend in a longer piece that may be handled readily and afterwards cut off such portions as are unnecessary.

Make exit and delivery tubes of the shapes and sizes indicated in A, D, and E, smoothing the ends in the flame, and preserve for future use. Three pieces like A will be needed in some experiments.

EXPERIMENTS

EXP. 1. CHEMICAL AND PHYSICAL CHANGES

Materials. Colorless rock candy. Platinum wire. Magnesium ribbon or wire. Wooden splints.

Apparatus. A mortar and pestle, small beaker and a test tube. A Bunsen burner, ring stand or tripod, and gauze. A test tube holder.

Procedure. Examine a specimen of rock candy, noticing its properties, such as color, taste, hardness, and crystalline form. Judging from its taste, of what does the rock candy consist?

1. In a clean mortar pulverize 5-10 g. of rock candy. Note any change in properties. See whether the characteristic property, *i.e.* the taste of sugar, has been changed by the grinding. Is the substance still sugar?

2. Partly fill the beaker with warm water and dissolve a little of the pulverized rock candy in it, stirring with a glass rod as the candy is added. What properties are changed? Taste the solution. Has the sugar been destroyed, or is it still in existence?

3. Heat some of the powdered rock candy in a dry test tube, applying the heat gently at first, noting carefully the progressive changes. When no further change takes place even at a more intense heat, remove the test tube and its contents from the flame. When the tube is cool, break it and examine the substance within. Taste it. Will it dissolve in water? Notice its color and softness. See whether it will burn. Does the sugar still exist? What does the new substance resemble?

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Compare the change brought about in 3 with the changes in 1 and 2, telling how they differ in their effect on the sugar. The change in 1 and 2 is physical. The change in 3 is chemical.

4. Heat successively in the flame of the burner pieces of wood, platinum wire, magnesium wire or ribbon, and glass. Compare the properties of the substances before heating with their properties after heating, and decide in each case whether the change is chemical or physical. Give reason for your answer in each case.

EXP. 2. COMPOUNDS AND MIXTURES

Materials. Powdered iron.* Sulfur. Carbon disulfid. Apparatus. Test tubes. Magnifying glass. Magnet. Watch glass or evaporating dish.

Procedure. (a) The properties of sulfur.

Note the visible properties of a piece of sulfur, examining the powdered material with a magnifying glass. Touch it with a magnet.

Burn a small bit of sulfur and cautiously notice the familiar odor.

Drop a small piece of sulfur into 3-5 ccm. of carbon disulfid in a test tube and shake for about 5 minutes. (CAUTION: Do not bring carbon disulfid near a flame, as its vapor is very inflammable!) Does the sulfur seem to dissolve? Pour off the liquid from any undissolved sulfur into a watch glass or evaporating dish and allow it to evaporate under a hood. If any sulfur is left upon the

* Iron filings do not work as satisfactorily as powdered iron, which may be purchased from any chemical dealer.

† Because of the way in which it is prepared (see text, p. 250) commercial carbon disulfid usually contains sulfur dissolved in it. For this experiment the carbon disulfid should be distilled to free it from sulfur and other impurities.

EXPERIMENTS

watch glass, it must have been dissolved in the liquid. Did any of the sulfur dissolve in the carbon disulfid? This is a general method of testing whether a substance is soluble in a liquid.

What are some of the characteristic properties of sulfur?

(b) The properties of iron.

Examine some iron powder under a magnifying glass and touch it with a magnet.

Ascertain whether iron powder will dissolve in carbon disulfid, proceeding as with sulfur.

What are some of the characteristics of iron powder?

(c) The characteristics of a mixture of iron and sulfur.

Stir together 5 g. of powdered sulfur and 7 g. of iron powder. What is the color of the new powder.

1. Bring a magnet to some of the mixture. Does the iron still exist?

2. Shake a pinch of the mixture in a test tube with 5 ccm. of carbon disulfid, carefully pour off the liquid from the undissolved material and evaporate on a watch glass. What is the residue left on the watch glass? Does the sulfur still exist in the mixture? What is left in the test tube?

From the above tests, do the iron and the sulfur appear to retain their identity when they are mixed? Is there any evidence indicating that any change has taken place?

(d) The properties of a compound of iron and sulfur.

Heat the rest of the mixture of iron and sulfur in a test tube with a small flame. When the mass begins to glow like a red-hot coal, remove it from the flame, as the change will continue without further heating. Lay the tube down on its side to cool. There is probably a little melted sulfur a part of the way up the inside of the

test tube. Do not allow this to run down and mingle with the substance at the bottom. When the tube is cool, break it open and examine with a magnifying glass the substance left where the glow had been. Can the iron or the sulfur be seen? Touch a flame to some of the substance. Does it burn? See if any sulfur can be dissolved out of it by means of carbon disulfid. Bring a magnet to a lump of the substance. Can you detect either the iron or the sulfur in the new substance?

Through what kind of a change have the iron and the sulfur passed? The product is a chemical compound. How can you distinguish a mixture from a compound?

EXP. 3. WHICH REACT MORE READILY, GASES, LIQUIDS OR SOLIDS?

Materials. Conc. ammonium hydroxid, hydrochloric acid and sulfuric acid. Alcohol. Potassium chlorate. Sulfur. Cream of tartar (hydrogen potassium tartrate). Baking soda (hydrogen sodium carbonate).

Apparatus. Graduate. Mortar and pestle. Two small evaporating dishes or watch glasses. Test tube.

Procedure. (a) Gases.

In a watch glass or evaporating dish put about 5 ccm. of conc. ammonium hydroxid solution and in another put the same quantity of conc. hydrochloric acid. Place the two liquids side by side so that the gases escaping from them may mix. If the liquids are warmed, the gases escape more readily. Catch some of the new substance on a piece of cold glass. What evidence do you observe that a chemical change is taking place?

(b) Liquids.

Measure 10 ccm. of alcohol into a test tube, and to it add in small portions an equal volume of conc. sulfuric

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acid. A change of temperature usually accompanies a chemical change. Do you notice any? Is there any visible new product to show that a chemical change has taken place?

(c) Solids.

1. Pulverize finely in separate * mortars 2 g. of potassium chlorate and 1 g. of sulfur. (CAUTION: Do not grind the two substances together !) Mix the powders thoroughly on a piece of paper, using the fingers only. Is there evidence of chemical action? Save the powder for the next exercise.

2. Thoroughly mix together 4 g. of cream of tartar and 2 g. of baking soda, both finely powdered. Is there any evidence of a chemical reaction? Save the mixture for the next exercise. State the relative readiness with which gases, liquids and solids react.

EXP. 4. HOW CHEMICAL CHANGES ARE BROUGHT ABOUT

Materials. The mixtures (potassium chlorate with sulfur, and cream of tartar with baking soda) from the last exercise. Blue print paper. Copper sulfate solution. Sulfuric acid. Zinc dust. Powdered sulfur.

Apparatus. A strip of zinc and of copper about 5 cm. wide and 10 cm. long. Two copper wires about 25 cm. long. Two platinum wires 5 cm. long. Beakers. Two dry cells. A compass.

Procedure. (a) By mechanical means.

Place the mixture of potassium chlorate and sulfur on a flat piece of iron or stone, and hit it a sharp blow with a hammer. What evidence is there of chemical action?

*Some students may pulverize the potassium chlorate, others the sulfur.

Hit the head of a parlor match a light blow, and compare the results. Do you think these reactions are endothermic or exothermic?

(b) By light.

Lay a piece of blue print paper, sensitive side up, on a book, put on the paper several coins, flat keys, or similar opaque objects, and expose the whole to bright sunlight until the parts of the paper on which the light shines have taken a bronzed appearance. Then remove the opaque objects from the paper, notice the appearance of the unexposed spots, and wash the paper in water. What evidence is there that a chemical change has taken place? In making photographs does the picture get any darker after it is removed from the sunlight? Is the reaction endothermic or exothermic?

(c) By electricity.*

"Make a hole near one end of the zinc and copper strips and connect the two by means of a copper wire. Place the metals in sulfuric acid, taking care that they do not touch each other.[†] What happens? What evidence of a chemical reaction do you see?

The chemical energy liberated by the reaction is converted into electrical energy by this apparatus and an electric current is said to flow through the wire. Bring a compass near the wire and note the indication that a current is flowing, holding the compass first above, then below the wire.

The current produced by this little cell is not sufficiently strong. For the following experiment substitute two dry cells arranged in series.

* This part of the experiment may be performed by the instructor before the class.

[†] The metals may be kept from coming in contact by tying them with a piece of string on opposite sides of a strip of shingle or thin wood. Connect the copper wires to the poles of the battery. Without allowing the wires to come into contact with each other, touch them to the tongue and "taste the electric current."

Connect platinum wires to the ends of the copper wires. Dip the platinum wires into copper sulfate solution for 1-2 minutes. What evidence is seen that the passage of an electric current through the solution has caused a chemical change? On which wire is the deposit? What is it? Is the reaction which has taken place analysis or synthesis?

Disconnect one of the copper wires from the battery. Then dip the platinum wires farther into the copper sulfate solution, and ascertain if there is any chemical change when the circuit is broken and the supply of electrical energy is stopped. Is the reaction with the copper sulfate exothermic or endothermic?

(d) By solution.

Pour a little water on the mixture of cream of tartar and baking soda, so that the ingredients can dissolve. What evidence is there of chemical action? Is the reaction exothermic or endothermic?

(e) By heat.

Mix 7 g. of zinc dust with 4 g. of powdered sulfur. Is there any chemical action? Place the mixture on a brick or piece of earthenware, and direct the flame of the Bunsen burner upon it until reaction begins. Then remove the flame. What evidence is there of chemical action? Is the reaction exothermic or endothermic? What purpose was served by the heat that was first applied?

EXP. 5. WHAT IS FLAME?

Materials. Ether. Kerosene. Paraffin or tallow candle. Sawdust.

Apparatus as shown in Fig. 6. Test tubes, burner, and ring stand.

Procedure. (a) Recall that coal gas, illuminating gas and all other gases you know of burn with a flame (if they burn at all).

(b) Put 3 ccm. of ether (CAUTION: Do not bring the bottle of ether near a flame!) in a test tube and smell of it. Can you detect it by its odor? Is sufficient ether vaporizing to burn when the mouth of the test tube is brought to a flame? Warm the test tube gently and see how it affects the size of the flame. Explain.

(c) Put 5 ccm. of kerosene in a test tube and bring it to a flame. Does it burn? Why? Heat it to boiling and. bring to a flame: What is burning, the liquid or the gaseous kerosene? Does the flame resemble the flame of an oil lamp?

(d) Put a piece of a paraffin or tallow candle in a test tube and bring to a flame. Does it burn? Why? Heat



the paraffin or tallow until it begins to bubble or smoke and then bring to a flame. Does it burn? Is it a solid, a liquid or a gas that burns at the mouth of the test tube?

(e) Set up the apparatus as shown in Fig. 6 and support on a ring stand so that the side-neck test tube dips into a beaker of cold water. Fill test

tube one quarter full of sawdust or pieces of wood and heat as long as any change takes place. Note the condensation of liquid in the side-neck test tube, and see if

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you can get anything to burn at the exit tube. What is it that burns there? Where did it come from and how was it produced? Does it resemble the flame of burning wood? Examine the material left in the test tube. What is it? How can you make charcoal? What else is produced in the same process? (The liquid in the sideneck test tube is mainly water mixed with a little acetic acid, wood alcohol and tar.)

From your experiments, what is flame? What substances burn with a flame? Would you expect charcoal to burn with a flame? Why?

EXP. 6. WHEN A SUBSTANCE BURNS, IS THERE ANY CHANGE IN WEIGHT?

Instructor's Experiment

Materials. Small candles. Metallic tin. Clear lime water. Sticks of sodium or potassium hydroxid or pieces of quicklime.

Apparatus. A fruit jar or wide-mouthed bottle covered by a glass plate. A wire about 30 cm. long. A porce-

lain crucible supported on a pipe-stem triangle. Large balance, lamp chimney filled with pieces of sodium or potassium hydroxid or lumps of quicklime as shown in Fig. 7. If a suitable large balance is not available, use Harvard trip scale, placing the chimney on a piece of wire gauze supported on



FIG. 7.

a circle of gauze as shown in Fig. 8; or improvise large balance with meter rod, etc.

gain or loss in weight?

Procedure. (a) Set a candle upright on the pan of a balance and counterpoise it exactly with weights. Light

5-10 g. of metallic tin and weigh on a balance that is sensitive to 1 cg. Support the crucible in the triangle and heat, gently at first. After half an hour allow the crucible and contents to cool

the candle. As it burns, is there an apparent

(b) In a shallow crucible without a cover place

FIG. 8.

and weigh again. Is there a gain or loss of weight? What does the change of weight indicate? From what source must the new material have come?

NOTE. The metal has been heated in air exactly as the candle was. At high temperatures such metals burn with a flame. The products formed are the ashes of these metals, similar to the products of ordinary combustion. In the case of metals the ash weighs more than the original substance. Wood and coal leave some ash. Illuminating gas, petroleum and a candle apparently burn to nothing and form no ash. Does combustion of a metal differ from combustion of a candle, or may it be that the ash of a candle is gaseous and so escapes notice? To test this point, perform the following experiment.

(c) Add 50 ccm. of clear lime water to a jar of air, put on the glass cover, and shake. Does the air produce any marked change in the lime water?

Without removing the lime water, lower a lighted candle into the jar by means of a wire wound around one end, and cover the jar with the piece of glass. In a short time what happens to the candle flame? To be sure that the candle flame has not been accidentally extinguished, take out the candle, relight it and again lower it into the jar. What effect does the burning of a candle have on the surrounding air?

Remove the candle and shake the lime water in the jar. What change do you notice? Does air cause this change? Are the products of combustion, *i.e.* the ash from a candle, solid, liquid or gaseous?

(d) Potassium hydroxid or quicklime will unite with the products of combustion of a candle, as the following experiment shows. To see whether the ash from a candle weighs more than the candle, use the apparatus shown in Fig. 7 or Fig. 8. After making sure that the apparatus is properly balanced and responds readily to a slight change of weight on either side, light the candle. As it burns, notice the change in weight on the side where the candle is burning. Is there a gain or a loss in weight? Does the candle weigh more or less than its ash?

Every substance has been found to gain in weight when burned. From what source must the extra matter come?

EXP. 7. IS THE TOTAL WEIGHT OF MATTER AFTER A CHANGE THE SAME AS THE TOTAL WEIGHT OF MAT-TER BEFORE THE CHANGE?

Instructor's Experiment

Materials. Potassium iodid. Mercuric chlorid. Phosphorus.*

Apparatus. Porcelain crucible. Balance. Magnifying glass or lens. Two clean beakers of about 150 ccm. capacity. Stirring rods. Flask or bottle whose capacity is a liter or more, fitted with a one-hole rubber stopper.

* Phosphorus is very inflammable; hence special precautions are necessary in handling it. It comes into the laboratory in the form of sticks, and is kept under water to prevent its catching fire. When a small piece is needed, remove a stick from the bottle, put it in a shallow dish containing sufficient water to cover the phosphorus, and cut it with a knife. A piece may be removed from the dish by means of the fingers, if it is thoroughly wet. If it must be dried before use, lay it on a piece of filter paper or a damp towel, turn it over a few times, handling it with forceps. If through any accident a phosphorus burn is inflicted on the skin, wash the wound well and cover it with a mixture of baking soda and oil.

Through the stopper passes a combustion spoon,* so that when the stopper is inserted the bowl of the spoon is below the center of the space inside the bottle. If the handle of the combustion spoon does not fit tightly into the stopper when it is inserted into the bottle, fasten it with sealing wax. Large balance.

Procedure. (a) Dry a piece of phosphorus the size of a small pea, pick it up with forceps and put it into a crucible. Set the phosphorus on fire and note the result.

Dry another piece of phosphorus, place it at once in the combustion spoon and introduce the latter into the flask, fitting the stopper tightly so that the whole is air-tight. Without delay counterbalance the flask and its contents accurately.

Ignite the phosphorus in the flask by focusing the rays of the sun on it by means of a lens.

When the burning is over and the flask is cool, place it upon the balance again. Explain why you notice no change in weight.

How do you account for the change in weight when a candle burns — first, ordinarily; secondly, under the lamp chimney containing potassium hydroxid? If the candle were burned inside of a sealed flask, would there be gain or loss of weight? Why?

(b) Into each of two clean beakers put 50 ccm. of water. In one dissolve 3 g. of potassium iodid and in the other 1 g. of mercuric chlorid, stirring until the crystals have disappeared. Then place the beakers with their contents side by side on one pan of the balance and counterpoise them.

Without spilling a drop, pour one half of the potassium

* If a combustion spoon is not at hand, a substitute may be made by wrapping one end of a piece of wire about a short piece of electric light carbon or chalk. Make a shallow depression in the end of the carbon or chalk. iodid solution into the other, and place the beakers again side by side on the balance pan. Do you judge that a chemical change has taken place? Why? Is it accompanied by a change in the total weight of reacting substances?

Without spilling a drop, pour the rest of the potassium iodid solution into the second beaker. Would you say that a chemical reaction had taken place? Why? Do you note any change in weight?

These reactions are typical examples. State the principle which they illustrate.

EXP. 8. THE GAS THAT MAKES A FIRE BURN

Materials. Mercuric oxid. A wooden splint. Apparatus. A test tube of hard glass.

Note. Mercury heated in a measured volume of air gains in weight and burns slowly to a red ash called mercuric oxid. When this process has been completed about one fifth of the air has been absorbed and the remaining four fifths will not support combustion, for the mercury has taken out of the air all of the gas that makes mercury or other substances burn.

Procedure. By means of a slip of paper neatly insert about 2 g. of mercuric oxid into the end of a hard glass test tube and heat, gently at first. Does the change in color indicate a chemical or a physical change? Allow the tube to cool and see if you can get an answer to this question. Heat the tube strongly and from time to time insert into the mouth of the test tube a glowing spark on the end of a match or a splint of wood. What happens? What is the gas?

When the red powder is entirely decomposed, allow the tube to cool. Then hold it in an inverted position and tap it on a sheet of paper laid on the table top. In addition to oxygen, what substance has been formed in this reaction?

This experiment was first performed by Priestley in 1774.

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EXP. 9. HOW MAY THE SPEED OF REACTIONS BE INCREASED?

Materials. Potassium chlorate. Powdered manganese dioxid.

Apparatus. Two test tubes.

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Procedure. (a) By increase of temperature.

In a test tube heat about 5 g. of potassium chlorate in the flame of the burner, cautiously at first until the potassium chlorate has melted. Notice that though minute bubbles of gas are evolved the total amount is insignificant. Then heat the contents of the tube to a much higher temperature and note the increase in the speed of the reaction with the increase in the temperature. Test the gas with a spark. What is it?

(b) By catalytic agents.

Heat a second portion of potassium chlorate in a test tube as before, until it has become thoroughly liquid. Remove the test tube from the flame, and, while its contents are still liquid, drop in a small pinch of powdered manganese dioxid, directing the mouth of the test tube away from the face. What happens? Test the gas liberated with a spark. What is it?

Is the difference in the rate at which the gas is produced due to the liberation of oxygen from manganese dioxid? To test this point the contents of the test tube may be cooled and then boiled out with water. All but the black manganese dioxid will dissolve and may be poured off from the residue. If the manipulation is carefully performed, the manganese dioxid will be found in exactly the same condition and quantity in which it was introduced. Why, then, is it well to mix manganese dioxid with the potassium chlorate in preparing oxygen?

Set up the apparatus for the next experiment.

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EXP. 10. PREPARATION AND PROPERTIES OF OXYGEN

Materials. A mixture of 30 g. of potassium chlorate and 15 g. of finely powdered manganese dioxid.* The materials need not be ground together. Small pieces of charcoal, sulfur and phosphorus. Iron picture wire.

Apparatus as shown in Fig. 9. Five or 6 pint fruit jars or wide-mouth bottles. Combustion spoon.

Procedure. (a) Preparation.

Fill the bottles *full* of water, cover each with a glass plate and invert them in the pneumatic trough, taking

care that no air is allowed to enter. Put the potassium chlorate and manganese dioxid mixture into the test tube until it is about one third full and arrange the apparatus as shown in Fig. 9. Heat gently at first, preventing moisture from con-



densing and running down upon the hot glass by occasionally waving the flame up the test tube. The first bubbles of gas driven out of the apparatus are air; they may be allowed to escape.

When gas is being liberated in a steady stream of bubbles, place one of the bottles full of water on the small pan in the pneumatic trough over the hole from which the bubbles are rising, being careful to keep the mouth of the

* Manganese dioxid is sometimes mixed with carbonaceous matter, and violent explosions have resulted from using it in this condition. To test the purity of the substance used, heat about 1 g. of the above mixture in a test tube. If chemical action goes on quietly, proceed with the work. bottle always under water so that no air enters. Fill five or six jars or bottles in a similar way. Before removing from the water, close the mouth of each bottle with glass plate or screw on the cover of the jar. Heat the test tube only sufficiently to produce a steady flow of gas, removing the flame for a moment if the gas is liberated too rapidly. When the jars are full, remove the delivery tube from the water to prevent sucking back and breaking the test tube.

It is a good plan to keep the jars of gas wrong side up after closing them, as the small amount of water they usually contain will stop any leaks around the covers.

After it has cooled, water may be added to the test tube to clean it.

(b) Physical properties.

Note whether oxygen has any color, taste or odor. Remembering that air contains oxygen, does this agree with your everyday experience?

(c) Chemical properties.

1. Action on charcoal. Place a piece of charcoal in a combustion spoon and heat it in the flame of the burner until it glows. Remove it from the flame. Does charcoal burn readily in the air?

Again heat a piece of charcoal and insert it still glowing into a bottle of oxygen. Compare the burning of charcoal in air and in oxygen.

2. Action on sulfur. (Hood.) Repeat (1) with a fresh bottle of oxygen, using sulfur instead of charcoal. Compare the burning of sulfur in air and in oxygen. Cautiously ascertain by the odors whether the two products are the same. Is the chemical action endothermic or exothermic?

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3. Action on phosphorus. (Hood.) Place a small bit of yellow phosphorus in a combustion spoon, light it and notice how it burns in air.

After the spoon has cooled, and not before, put another piece of dry phosphorus in it, ignite it and insert in a bottle of oxygen. Do not let the burning phosphorus get against the sides of the bottle. Compare with phosphorus burning in air.

4. Action on iron. Place sand in a jar of oxygen sufficient to form an even layer over the bottom. Heat the end of a piece of iron picture wire about 30 cm. long and dip it into powdered sulfur. Light the sulfur and note whether the iron will take fire in air. Repeat and insert the lighted end into the bottle of oxygen. The heat from the burning sulfur causes the iron to catch fire. (Repeat if not successful at the first trial.) Describe what happened.

What can you say of the chemical activity of oxygen at ordinary and at more elevated temperatures?

EXP. 11. OXYGEN CONSUMED DURING COMBUSTION

Instructor's Experiment

Materials. Potassium chlorate. Manganese dioxid. About 1 m. of iron picture wire. Powdered iron. Yellow phosphorus.

Apparatus. A stoppered bell jar or a two-liter bottle cut off * near the bottom, or simply with a hole broken in the bottom. Two one-hole stoppers to fit the bottle. Two pieces of glass tubing fitting the stoppers, about 20 cm. long. A short piece of rubber tubing. A pinchcock. An oxygen generator. Two closed tubes, preferably graduated, 30 cm. or more in length and about 2 cm. in diame-

* To cut a glass bottle, see p. 28.

ter. Stout wire about half as long as the tubes. Two tall beakers or cylinders. Ring stand and large clamp. Tank or pail for holding water.

Procedure. (a) Rapid combustion.

Slip one piece of glass tubing through one of the stoppers, put the stopper tightly in the bottle and connect the rubber tube to the end of the glass tube projecting from the stopper. Set the bottle in a tank or pail of water and fill it by sucking on the rubber tube; then close the rubber tube with a pinchcock. Support the bottle by a clamp and ring stand and fill it with oxygen. Raise or lower the bottle to make the water level the same inside as outside, and mark the level with chalk or an elastic.

Wrap the picture wire around a pencil in a close spiral. Remove the pencil, and tip the end of the wire with sul-



FIG. 10.

fur as in Exp. 10. Fuse a copper wire loop into one end of the second piece of glass tubing, seal the other end, and slip it through the hole in the second rubber stopper. Hang the coil of wire from the loop.

Light the sulfur, then quickly removing the first stopper insert the other stopper tightly so that the picture cord may burn in oxygen, as shown in Fig. 10. Slip the glass tube up or

down through the stopper and lower the bottle in the water if necessary to prevent ingress of air.

The picture wire is now burning in a closed space filled with oxygen. As the combustion progresses, what change is seen in the water level inside the bottle? What must have become of the oxygen? What must be

EXPERIMENTS

the composition of the black substance at the bottom of the tank?

(b) Slow combustion.

1. Of iron. Fill one of the tubes with water and invert it in a dish of water. Then fill the tube with oxygen. Close the end of the tube with the thumb and remove from the water. Carefully remove the thumb for a moment and introduce 5–10 g. of powdered iron. Cover with the thumb and shake the tube until the iron distributes itself over the inside of the tube. Then invert in a tall beaker of water and stand aside for a day or two. Explain what happens. Is there any significant difference between this reaction and that in the last experiment?

2. Of phosphorus. Keeping the phosphorus under water, impale a piece about the size of a pea on the end of the wire. Place the other end of the wire in a beaker of water and slip over the phosphorus the closed tube, so arranging the quantity of water in the beaker that the phosphorus shall be out of water but inclosed in the tube full of air over water. Stand aside for three or four days until action ceases. Explain what happens. What is the difference between this experiment and that with the iron?

This reaction offers a method for determining the proportion of oxygen in air. When the water ceases to rise inside the tube, showing that the reaction is complete, make the level of the water inside and outside the tube the same, either by raising the tube or by adding water to the beaker. Then ascertain the proportion of the gas absorbed. This is the proportion of oxygen in the atmosphere. What do you find it to be?

EXP. 12. HEAT EVOLVED DURING SLOW OXIDATION LEADS TO SPONTANEOUS COMBUSTION

Instructor's Experiment

Materials. Yellow phosphorus. Carbon disulfid. Cotton.

Procedure. Dry a small piece of phosphorus and lay it on a glass plate. Notice how it smokes after a few seconds. How do you explain this action ?

Dissolve a piece of dried phosphorus as large as a pea in about 5 ccm. of carbon disulfid in a test tube. By the aid of pincers or a piece of wire (CAUTION: Do not handle with the fingers!) dip a little cotton in the solution of phosphorus and spread it out on the ring of an iron stand. The carbon disulfid evaporates quickly, leaving the phosphorus in finely divided condition, so that a large surface is exposed to the action of the air. Note what happens presently. How do you explain it?

EXP. 13. KINDLING TEMPERATURE

Materials. Yellow phosphorus. Sulfur. Paraffin. Sulfur matches. Parlor matches.

Apparatus. A small pan, such as is used for a sand bath, or piece of sheet iron. Gauze. Tripod or ringstand.

Procedure. (a) A study of a match.

At equal distances from the center of the pan or piece of sheet iron and as far as possible from each other, place a small piece of dried phosphorus, of sulfur, of paraffin, and a match from which the head has been removed. Set the pan on a tripod, and under its center place a burner. Presumably the substances are heated equally rapidly. Notice the order in which the substances catch fire. That which takes fire first has the lowest kindling temperature. Examine a sulfur match. Do you get any indication of the substances composing it? (Match heads formerly contained phosphorus, but because of its poisonous properties, phosphorus sulfid is now used, its kindling temperature being about the same as for phosphorus.)

Rub a similar match against a slightly roughened surface. What substance first catches fire? Whence does it derive the heat necessary to raise it to its kindling temperature? Which substance burns next? Whence does it derive the heat necessary to raise it to its kindling temperature? Which substance burns last?

Repeat the work of the last two paragraphs, using parlor matches. What difference in the composition of parlor and sulfur matches is evidenced?

What would be the effect if a match were tipped with sulfur alone? (Try to ignite a piece of sulfur by friction.)

If wood had a lower kindling temperature, which part of a match could be left out?

In burning iron in oxygen, why was the picture cord tipped with sulfur?

(b) The kindling temperature of gases.

Light a Bunsen burner, and bring down over the flame a piece of wire gauze. Why does the flame not burn above the gauze? Is there unburned gas above the gauze? To answer this question hold a lighted match above the gauze and explain the result. What cools the gas above the gauze below its kindling temperature?

Remove the gauze from the flame for a moment and, while it is still hot, bring it down into the flame again. Why does the gas above the gauze now catch fire?

Turn off the gas in the burner. Then turn it on again but do not light it, and bring down a piece of cold wire gauze to a distance of about 5 cm. above the top of the

burner. Bring a lighted match above the gauze. Under proper conditions the gas burns above but not below the gauze. Why not?

Note. The safety lamp, invented by Sir Humphry Davy and used by miners, depends on the principle illustrated in the preceding experiments. It consists of a small lamp surrounded by fine wire gauze. When brought into a mine where combustible gas is present, the gas passes readily through the wire gauze and burns brightly on the inside. The gauze conducts the heat of the flame away, and prevents the gas outside the gauze from becoming heated to its kindling temperature. Thus the miner is warned that a dangerous gas is present.

EXP. 14. DIFFUSION

Instructor's Experiment

Materials. Bromin.* Cupric chlorid. Ammonia water (ammonium hydroxid). Alcohol. Alcoholic solution of phenolphthalein. Dilute sodium hydroxid solution.

Apparatus. Fruit jar, or wide-mouth bottle, covered with glass plate. A thistle tube. Two cylinders covered with glass plates or two large test tubes fitted with corks. A separating funnel or a pipette.

Procedure. (a) Gases.

Through the thistle tube deposit about 1 ccm. of bromin inside on the bottom of a fruit jar. Remove the thistle tube and cover with a glass plate. Notice how, as the bromin vaporizes, it rises through the air until it soon fills the whole jar. Are the particles of which gases are composed capable of moving with great freedom?

*Avoid inhaling the fumes of bromin, as it attacks the nose and throat. The antidote is to inhale alcohol poured on a handkerchief. Do not allow liquid bromin to touch the skin, as it produces severe burns. If it does get on the skin, wash quickly with water and then with a dilute solution of baking soda.

(b) Liquids.

1. Make a concentrated solution of cupric chlorid in a little cold water. To 1 ccm. of the solution in a test tube add a little ammonia water, and notice the deep blue color produced when the liquids mix.

By means of a thistle tube pour the cupric chlorid solution into a cylinder or large test tube until there is a layer 2–3 cm. thick, taking care not to spatter the sides of the cylinder. On the cupric chlorid solution float water. To float one liquid on another, cut a thin section from the top of a large cork, the thinner the better. Place the section of cork on the heavier liquid, and deliver the lighter liquid slowly upon the center of the cork, using a separating funnel or a pipette. When the test tube is about three fourths full, float a layer of ammonia water on the top of the others. Remove the section of cork and without disturbing the layers of liquid, cover or cork the cylinder and set it where it can remain quiet for several days. It now contains cupric chlorid and ammonia separated by a considerable layer of water.

From day to day notice the rise of the blue copper chlorid through the tube until it meets the ammonia diffusing down through the water, as is indicated by the deep blue color.

2. Try the effect of bringing together a few drops of an alcoholic solution of phenolphthalein and a little dilute sodium hydroxid solution. Then repeat the work outlined in the preceding paragraphs, placing in the bottom of the cylinder sodium hydroxid solution, on it floating a mixture of alcohol and water in equal proportions until the test tube is nearly full, and lastly 1 ccm. of an alcoholic solution of phenolphthalein. Remove the section of cork, close the test tube, and stand aside as in (1). Explain the final result.

Are the particles of which liquids are composed capable of moving with considerable freedom? How does this freedom compare with the freedom of motion of the particles of gaseous bodies?

(c) Diffusion takes place with solids just as with gases and liquids, only much more slowly.

In a previous exercise it was found that gases and liquids react chemically much more readily than solids. Suggest an explanation of this fact.

EXP. 15. DECOMPOSITION OF WATER BY ELECTRICITY

Instructor's Experiment

Materials. Sugar. Conc. sulfuric acid. Sodium hydroxid. Sodium sulfate.

Apparatus as shown in Fig. 11, consisting of two graduated test tubes or measuring glasses. The bowl



FIG. 11.

may be made by cutting a bottle. To do this, tie a string wet with kerosene tight about the bottle where it is desired to cut it. Set fire to the oil, rotating the bottle slowly as the oil burns. As soon as the string burns through, plunge the bottle in water.

Fit the mouth of the bottle with a solid rubber stopper. Drive a wire nail through this stopper in two places, thus making holes through which the wires leading to the electrodes may pass.

The electrodes consist of two pieces of platinum foil

about 2 cm. long and 1 cm. wide, each welded * on to a platinum wire about 7 cm. long. If these wires are pushed through the holes in the stopper, it will close about them water-tight. Bend the platinum wires into the form of hooks and hang into them similarly hooked copper wires leading to the battery. When the bowl is filled with solution, the circuit is complete.

Procedure. (a) Non-electrolytes.

1. Remove the measuring tubes and rinse the whole apparatus with distilled water. Then pour distilled water into the bowl until it is nearly full. Connect the electrodes with a battery or some other source of electricity and attempt to pass a direct current through the water. Do you notice any indication that a current is passing? Is pure water an electrolyte or a non-electrolyte?

2. Dissolve 5 g. of sugar in the water in the bowl. Does the current now flow through the solution? Is sugar an electrolyte?

(b) Electrolytes.

1. Empty the sugar solution from the bowl, rinse with distilled water and refill, adding about 5 g. of sodium hydroxid to the water in the bowl and stirring to mix thoroughly. What difference do you notice? Is sodium hydroxid an electrolyte or a non-electrolyte?

2. Empty the contents of the bowl, rinse with distilled water and refill as before, adding sodium sulfate to the water. Describe what happens. Is sodium sulfate an electrolyte or a non-electrolyte?

* To weld platinum, lay the foil on a piece of flat iron, hold the wire in the position desired and heat both with a fine flame from a blowpipe. When they become red-hot, a light blow with a hammer will produce a firm weld. 3. Empty, rinse and refill the bowl as before, adding to the water about 10 ccm. of conc. sulfuric acid, stirring well as before. Is the acid an electrolyte?

(c) Electrolysis of water.

Disconnect the wires and stop the current. Fill the graduated test tubes or measuring glasses with solution from the bowl, close with the thumb, and invert in the bowl, placing one test tube over each electrode. See to it that no bubbles of air have gained access to these measuring tubes. Start the current again, so regulating its strength as to cause a steady liberation of bubbles without any undue heating of the wires or the solution. Note the rate at which the gases are liberated.

When a sufficient amount of the gases has been collected, break the circuit, and measure the amount of gas in each tube to the nearest cubic centimeter, first adjusting the tubes so that the water level is right. If this cannot be satisfactorily accomplished in the bowl, close the tubes with the thumb and remove to a vessel of water of sufficient depth. No correction for pressure, temperature or moisture is necessary, as the gases are under the same conditions when measured. What is the relation between the volumes of the two gases ?

Slip the thumb over the mouth of the tube containing the smaller amount of gas, remove it from the water, turn it mouth up, and test its contents with a glowing spark. What gas does it contain?

The gas in the second tube is called *hydrogen*. In a similar way remove the second tube from the solution, and test its contents with a lighted match or other flame. What happens?

In each of the above instances the current will continue to flow through the solution until the water is used up and the other substance is left. Furthermore, by evaporating any of the three solutions mentioned the original substance, sodium hydroxid, sodium sulfate or sulfuric acid may be recovered. This shows that the catalytic agents are not used up but that the gases come from the decomposition of the water. What can be said, then, as to the composition of water?

(d) Calculations of results.

1. Multiply the volume of oxygen by 0.00143 g., the weight of 1 ccm. of oxygen. Multiply the volume of hydrogen by 0.00009 g., the weight of 1 ccm. of hydrogen. Divide the weight of the oxygen by the weight of the hydrogen. How many parts by weight of oxygen are present in water for every 1 part of hydrogen? How many parts by weight of water contain 1 part by weight of hydrogen?

2. Add the weights of oxygen and hydrogen. The sum is the weight of the water decomposed. The weight of 1 ccm. of steam is 0.0008 g. Calculate the volume which this water would have occupied if, instead of being decomposed, it had been converted into steam.

3. Fill in the blanks in the following statements :

By weight, 1 part of hydrogen unites with () parts of oxygen to give () parts of water.

By volume () parts of hydrogen unite with 1 part of oxygen to give () parts of steam.

EXP. 16. DECOMPOSITION OF WATER BY METALS

Instructor's Experiment

Materials. Iron filings, tacks or nails. Sodium. Potassium.

Apparatus as shown in Fig. 12, consisting of a flask for

generating steam connected with a piece of iron pipe about 50 cm. long and 2-3 cm. in diameter, containing iron filings or nails. From this pipe an exit tube leads into a bottle, as shown in the figure, in which the excess steam condenses. From this a delivery tube dips under the surface of a pneumatic trough. If a furnace is not available, heat the tube with several Bunsen burners. A



FIG. 12.

metallic capsule for sodium and a stiff wire for a holder (see Suggestions to Teachers). Wide-mouth bottle.

Procedure. (a) Decomposition of water by iron. First performed by Lavoisier in 1783 with a gun barrel, furnishing the first proof that water is not an element.

Heat the middle of the iron pipe to redness and pass in steam from the flask. After the air has been driven from the apparatus, collect some of the gas in an inverted bottle filled with water. When the bottle is full cover with a glass plate and remove the bottle from the water. Keeping the bottle inverted bring to a flame and remove cover. Describe what happens.

Is the gas apparently like either gas formed when water is decomposed by means of electricity?

The gas is hydrogen, its odor being due to impurities. Iron is an element. Where must the hydrogen come from?

Fill a bottle half full of hydrogen, raise from the water, thus allowing air to enter and mix with the hydrogen. Bring to a flame and note result. Bring out clearly the effect of mixing air with hydrogen.

Iron and some other metals decompose water at high temperature; magnesium at boiling temperature; others at ordinary temperature.

(b) By sodium or potassium.

1. Throw a piece of sodium about the size of a small pea on a dish of water, holding a sheet of glass between you and the dish to prevent spattering. What happens?

2. Throw a similar piece of potassium into water. What happens?

The action with potassium is similar to that of sodium except for the flame. Either the metal burns or else a gas is given off which burns.

3. To settle this point, fill a capsule with sodium, twist it firmly into the wire holder. Fill a bottle with water, cover with a glass plate and invert in a pan of water, being sure that no air gets into the bottle. Lean the bottle against one edge of the pan and quickly thrust the capsule, mouth down, under the mouth of the bottle (see Fig. 29). Regulate the flow of gas by slightly inclining the capsule as the action slackens. If careless handling allows the sodium to escape from the capsule, stand back until all action ceases. Describe what happens.

When action is complete, cover the mouth of the bottle with a glass plate, remove from water and stand bottle upright. Bring a flame to the mouth of the bottle and remove the glass cover. What happens?

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The gas is hydrogen, colored yellow by sodium. Sodium and potassium are elements. Where does the hydrogen come from?

Dip the fingers in the water into which the sodium and potassium were thrown and note the effect. This is due to compounds of these metals dissolved in the water (see Exp. 34).

EXP. 17. THE ACTION OF METALS ON ACIDS

Materials. Small pieces of zinc, magnesium, copper, tin and of iron, such as coarse filings or small nails. Sulfuric, hydrochloric and acetic acids.

Apparatus. Test tubes and an evaporating dish.

Procedure. (a) Fill a test tube one quarter full of sulfuric acid, then fill to the top with water. Close the tube with the thumb and invert in an evaporating dish containing water. Roll about 7 cm. of magnesium ribbon into a ball, slip it under the mouth of the test tube and note action. When the tube is full of gas, close with the thumb and bring to a flame. What happens?

The gas is hydrogen. Magnesium is an element. Where must the hydrogen have come from? (See if magnesium acts on water to liberate a gas.)

(b) Without attempting to collect the gas as m(a) see if there is any action between the following metals and acids by adding to 5 ccm. of acid in a test tube a piece of the metal. Warm the acid if necessary to get action but do not heat to boiling. State whether the action is slow, medium or rapid in the following cases:

Zinc and sulfuric acid. Zinc and hydrochloric acid. Zinc and acetic acid. Iron and sulfuric acid.

EXPERIMENTS

Tin and hydrochloric acid. Magnesium and acetic acid. Copper and sulfuric acid.

Copper and hydrochloric acid.

Many metals (not all) decompose many acids (not all) with the liberation of hydrogen.

(Set up the apparatus for the next experiment so that it will be ready at the beginning of the period.)

EXP. 18. PREPARATION AND PROPERTIES OF HYDROGEN

Materials. Sulfuric acid. Granulated zinc or small pieces of sheet zinc. Solutions of potassium permanganate and copper sulfate. Wooden splints. Cobalt chlorid test paper (see Exp. 26).

Apparatus as shown in Fig. 13 consisting of a bottle whose capacity is about 300 ccm. The thistle tube

reaches almost to the bottom of the bottle. A piece of clay pipestem. Two 250 ccm. flasks closed with corks. A test tube. 4-6 fruit jars or wide-mouth bottles.

Procedure. (a) Preparation.

Put 20-25 g. of granulated FIG. 13. zinc into the bottle, insert the stopper (fitted with the thistle tube and delivery tube as shown) tightly, and through the thistle tube pour in enough sulfuric acid to cover the end of the thistle tube.

If the action is slow, add a little copper sulfate solution and shake the bottle. What is the effect? The copper sulfate is decomposed by the zinc, the copper being deposited on the zinc as is shown by the dark coloration. The copper then acts as a catalytic agent.

FIG. 13.



CAUTION: The hydrogen liberated is at first mixed with air. A hydrogen and air mixture is explosive; hence keep all flames away from the apparatus at this stage of the work.

To ascertain when the hydrogen is sufficiently pure, collect a test tube of the gas over water, cover with the thumb and, keeping the test tube inverted remove it to a flame. If a whistling noise is heard, the hydrogen is still mixed with air. When it burns with only a quiet puff at the beginning, it is sufficiently pure.

Collect six bottles of hydrogen gas, after sealing allowing them to stand inverted until used. If the flow of gas slackens, introduce through the thistle tube additional acid. Keep the apparatus for the next experiment.

(b) Properties.

1. Place one jar of hydrogen mouth upward on the table and another mouth downward. Remove the seal from second jar and hold it mouth downward above the table. Then remove the cover from the upright jar. After about a minute bring both jars to a flame. Do you detect hydrogen in both bottles? If not, from which one has it escaped? How did it escape from this bottle? What does this indicate as to the relative weights of hydrogen and air?

2. Note whether hydrogen has any color, taste or odor. If impurities give to the gas an odor, they may be removed by treating with a dilute solution of potassium permanganate acidified with sulfuric acid. Slide the cover glass partly from a jar of hydrogen and quickly (Why use haste?) introduce the permanganate solution. Shake the covered jar for a few minutes and then test odor again.

3. Remove the seal from a jar of hydrogen and, hold-

ing it mouth downward, thrust well into the jar a lighted splinter of wood and hold it there until all action ceases (see Fig. 14). What do you observe?

Does hydrogen support combustion as well as burn?

4. Slip into the end of the rubber tube attached to the outlet tube of the generator a piece of clay pipestem and light the escaping hydrogen. Hold in the flame a wire or





piece of glass tubing. Does the flame seem to be very hot?

Hold a clean, well-corked flask filled with cold water 20-30 cm. above the hydrogen flame. What happens?

Blow out the hydrogen flame and hold the second clean, well-corked flask filled with cold water in the hydrogen as it escapes unburned. What happens?

A test for water is given on page 48. Rub off the deposit on the first flask with a piece of cobalt chlorid paper, made blue by warming. What happens? What was the deposit on the first flask? Explain its formation.

(c) Another product of the reaction remains dissolved in the liquid in the hydrogen generator. It may be recovered as follows: Allow the acid to stand in contact with zinc until all action ceases, then pour it off into a beaker. Allow the particles of copper and other matter to settle, then carefully pour off the clear liquid into another beaker or evaporating dish. If crystals do not form on standing overnight, evaporate part of the liquid and try again. The crystals are zinc sulfate.

EXP. 19. OXIDATION AND REDUCTION

Materials. Small bits of copper or lead.

Apparatus. A piece of hard glass tubing about 30 cm. long by 1 cm. in diameter bent nearly at a right angle, as shown in Fig. 15. A hydrogen generator connected to a drying tube filled with granulated calcium chlorid kept in position by plugs of cotton in either end of the tube. Wire.

Procedure. (a) Oxidation.

Fill the middle of the hard glass tube with pieces of copper or a few pieces of lead. Support this tube on a wire gauze placed on an iron ring, keeping the short end of this tube nearly vertical by means of a piece of wire fastened to the ring stand. Keep the vertical tube heated somewhat so as to cause a current of air to pass through the apparatus. Heat the metal in the tube, gently at first, then to a low red heat. Notice the change in color of the copper or lead. How do you explain this action?

Continue the heating for 10–15 minutes, then allow the apparatus to cool. Complete the reactions :

 $\begin{array}{l} \text{Copper} + \text{oxygen} \rightarrow \\ \text{Lead} + \text{oxygen} \rightarrow \end{array}$

(b) Reduction.



While the tube is cooling start the hydrogen generator and see that all air has been driven out. Then connect the drying tube of the generator to the tube containing the oxi-

FIG. 15.

dized metal as shown in Fig. 15. While the hydrogen is passing through this tube, gently heat it as before, allowing the upright tube to remain cool. Describe and explain what takes place.

What collects in the cool portion of the exit tube? Test it and see if your answer is correct.

Copper oxid + hydrogen \rightarrow Lead oxid + hydrogen \rightarrow

Consult the text, page 61, and see how this experiment might be made of use in determining quantitatively the composition of water.

EXP. 20. THE NASCENT STATE

Materials. 200 ccm. of distilled water, to which is added just enough potassium permanganate to impart a decided color. Conc. sulfuric acid.

Apparatus. Four beakers. A hydrogen generator.

Procedure. Divide the water containing potassium permanganate solution into four portions.

1. To one portion add 5 ccm. of conc. sulfuric acid.

2. Into another portion drop a few grams of zinc.

3. Into a third portion drop a similar amount of zinc, and then add 5 ccm. of conc. sulphuric acid.

4. Through the last portion allow hydrogen from the generator to bubble.

After about 20 minutes compare the results in the four solutions. In which case is there a chemical change resulting in the bleaching of the potassium permanganate? Can sulfuric acid alone cause the bleaching? Can zinc alone? What do they produce when they react? Does ordinary hydrogen cause the bleaching? In what condition is hydrogen most active?

EXP. 21. DECANTATION, FILTRATION AND DISTILLATION

Materials. Filter papers. Sand. Salt. A light insoluble powder, such as precipitated chalk (calcium carbonate) or magnesia (magnesium oxid). Red ink or potassium permanganate solution.

Apparatus. Funnel, ring stand, beakers and stirring rods. Distillation apparatus as shown in Fig. 18, consisting of a condenser, boiling flask of about a half liter capacity fitted to it and a receiving flask of about half this capacity. If a condenser is not available, a long piece of glass tubing, a meter or more in length, will serve the purpose if the boiling is sufficiently slow to allow the air to cool and condense the steam.

Procedure. (a) Decantation.

Stir a little sand into a beaker half full of water, then allow the mixture to stand for a few minutes. To what extent does the sand settle out of the water? See how completely you can separate the sand by carefully pouring off the water. This process of pouring off is called *decantation*; the sand is typical of any heavy insoluble substance.

Mix about 50 g. of sand with 10 g. of salt, add 50 ccm. of water, stir well, then allow to settle and see how completely you can decant into a beaker the liquid without allowing any sand to be poured off. Taste and measure the liquid. Where is the salt? Calculate how much of the salt was removed from the sand by decanting the liquid. Add 50 ccm. of water to the beaker containing the sand, stir, settle and decant as before. Calculate how much salt is left with the sand after the second decantation.

Continue the process until the taste of salt is no longer perceptible. How many decantations were necessary to separate the salt and sand? What class of substances can be separated from what other class of substances by this method? Suggest practical uses of the process of decantation.

(b) Filtration.

To 50 ccm. of water add about 5 g. of salt and some

precipitated chalk or magnesia, stir well and allow to settle for a few moments. Do you judge that decantation would be as successful now as in (a)? Why?

Fold a filter paper as shown in Fig. 16 and open it into a cone having three thicknesses of paper on one side and one on the other. Fit it into a funnel, moisten with a little water and



FIG. 16.

press the paper snugly against the sides of the funnel. Sup-



FIG. 17.

port the funnel in a ring stand, placing a beaker beneath it.

Stir up the mixture previously prepared and pour it gradually upon the filter paper in the funnel, always keeping the liquid well below the top of the paper. To avoid spattering and prevent drops running down the sides of the beaker; hold the middle of a glass rod against the edge of the beaker and pour the liquid along the rod on to the side, not the tip, of the filter paper, as shown in Fig. 17.

This process is called *filtration*. The liquid which runs through it is called the *filtrate*: the substance remaining in the filter is the *residue*. Ascertain by tasting whether the salt is in the residue or the filtrate. Can dissolved substances be separated from liquids by filtration? What substances can be separated by filtration? Suggest practical uses of the process of filtration.

(c) Distillation.

Add sufficient salt and red ink or permanganate to about 150 ccm. of water to give it a distinct taste and



FIG. 18.

color. Place the liquid in the boiling flask and connect the flask with the condenser as shown in Fig. 18. Connect the lower opening of the jacket of the condenser to a water tap, so that the flow of water will be in the direction indicated by the arrows. Turn on the water, and as it issues from the upper outlet, conduct it by means of rubber tubing to the sink. Heat the flask on a wire gauze until the water boils. Note carefully and describe what happens. This process is called *distillation*. Allow the first few ccm. of the *distillate*, *i.e.* the liquid that condenses, to go to waste, as this may contain dust and impurities washed out of the condenser. Collect the remainder of the distillate in a clean flask or beaker, continuing the process until there is enough to test. Note the color, odor, and taste of distilled water. Where is the salt and coloring matter? What class of substances may be removed by distillation? What class might not be removed in this way? For drinking purposes is distillation a better method of purifying water than filtration? State several reasons why. Suggest practical uses of the process of distillation.

EXP. 22. HOW HEAT AFFECTS SOLUBILITY

Materials. Crystallized copper sulfate (blue vitriol), salt, potassium chlorate, and alum.

Apparatus. Mortar and pestle. Test tubes. A beaker of boiling water. Four small 1 gram measures,* one for each of the substances mentioned above.

Procedure. Put exactly 10 ccm. of cold water into a test tube, and to it add very finely powdered copper sulfate in successive portions of 1 g. each, as long as it dissolves. Shake the test tube continually to aid solution. When no more will dissolve, the solution is said to be *saturated*. What is the solubility of copper sulfate in water at room temperature ?

Heat the saturated solution of copper sulfate by placing the test tube in boiling water and again add powdered copper sulfate in portions of 1 g. each until no more will dissolve. How many additions of copper sulfate are required to saturate the solution? What is the solubility of copper sulfate in water at 100°? How is the solubility of copper sulfate affected by rise in temperature?

* See Suggestions to Teachers.

Cool the solution of copper sulfate by holding the test tube in running water. Explain the result.

Repeat the above procedure, some students using salt, others potassium chlorate, others alum in place of copper sulfate. Does heat affect the solubility of all substances alike ?

NOTE. When heat increases solubility, cooling of a saturated solution causes *supersaturation*. The normal tendency of a supersaturated solution is to separate out immediately the excess of the solute, *i.e.* of the dissolved substance. With certain substances, however, this separation seems to "stick" and some supersaturated solutions may exist for some time before separation of the excess of the solute takes place, as is shown in the next experiment.

EXP. 23. SUPERSATURATION

Instructor's Experiment

Materials. Sodium sulfate (Glauber's salt), or sodium thiosulfate (hypo).

Apparatus. A beaker. 2-3 test tubes with tightly fitting corks. A thermometer.

Procedure. Pulverize about 100 g. of sodium sulfate, place it in the beaker, add about 25 ccm. of water, warm to $30^{\circ}-35^{\circ}$,* and maintain at this temperature for about 15 minutes, stirring to hasten solution. If all of the sodium sulfate dissolves readily, add more until the solution is saturated. If some remains undissolved, add water, 1 ccm. or so at a time, until everything has gone into solution. Rinse out the test tubes, including the corks, with water and invert them so that they may drain. Pour the solution of sodium sulfate, saturated at $30^{\circ}-32^{\circ}$, into the test tubes, cork immediately, and let stand undisturbed until thoroughly cold. The water then holds in

* The solubility of sodium sulfate is greater at 32° than at any other temperature.

solution an excess of sodium sulfate beyond the amount which would dissolve at room temperature. The solution is supersaturated, yet no crystals of sodium sulfate ordinarily appear.

Shake one of the test tubes and describe what happens. When supersaturated solutions are disturbed, separation of the solid usually takes place.

Cautiously remove the cork from another test-tube and drop a minute crystal of sodium sulfate into the solution. Notice how from this as a nucleus the crystals grow in all directions until the contents of the test tube become practically solid.

Sodium sulfate is one of the common substances present as dust in the atmosphere. Why was it necessary to keep the tubes corked?

Repeat the whole experiment, if time permits, using 50 g. of sodium thiosulfate (hypo). Heat the crystals gently in a test tube and they will dissolve in the water which they contain, forming a saturated solution. Cool the test tube in running water to room temperature, introduce a small crystal of hypo and watch the crystals grow.

EXP. 24. SOLUTIONS OF GASES

Materials. A syphon or bottle of carbonated water. Apparatus. Thermometer. Beakers. Gauze. Ring stand.

Procedure. (a) How pressure affects the solubility of gases.

Press the lever and draw about 50 ccm. of water from the syphon into a beaker. What causes the water to leave the syphon? Note the separation of gas from the solution, causing the bubbling or effervescence. Was the water when drawn from the bottle saturated, unsaturated, or supersaturated with gas? How is the pressure on the solution remaining in the bottle affected when liquid is removed from the syphon? Do you see any indication of this? How does change of pressure affect the solubility of the gas?

(b) How temperature affects the solubility of gases.

Set the beaker and its contents on a gauze on a ring stand and warm with a burner. Test the temperature with the thermometer and do not heat to the boiling point of water. How does the rise in temperature affect the solubility of the gas? Contrast this effect with the effect of a rise in temperature on the solubility of solids.

Draw 100 ccm. of cold water from the tap and warm it over the burner. How do you account for the formation of small bubbles on the inside of the beaker.

When a glass of cold water is allowed to stand for some time, small bubbles appear, clinging to the glass. Explain their formation.

EXP. 25. CRYSTALLIZATION

Materials. Copper sulfate. Potassium dichromate. Alum. Salt.

Apparatus. Beakers. Funnels. Filters.

Procedure. From the results of Experiment 22 calculate how much water will be needed to make a solution of 50 g. of copper sulfate saturated at 100°. Put together the water and copper sulfate, heat to boiling, and see whether your calculation was approximately correct. If some solid remains undissolved, add a little more water. If all the copper sulfate goes into solution, and you have reason to believe that you added too much water, evaporate the solution over a Bunsen burner or on a water bath until a solid begins to form around the edges or in the solution.

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A water-bath consists of a vessel containing water that is kept boiling by a burner placed beneath. The steam from this vessel transmits heat at 100° to another vessel placed in or on the first. A dish containing the liquid to be evaporated set on top of a beaker of water, boiling slowly, is a simple form of water-bath. A common household form is the double boiler used for cooking substances that readily burn.

If the saturated copper sulfate solution is not clear, it may be filtered, but the addition of a few drops of sulfuric acid will usually render the solution perfectly clear without interfering with the resulting product.

Put a little of the liquid into a test tube, cool it in running water, and shake.

Cover the rest of the liquid in the beaker and set it away to cool. How do the resulting crystals differ in the two cases? How does rapidity of formation seem to influence their size? (The smaller crystals are likely to be purer.)

Prepare hot solutions of 50 g. of alum, 30 g. of potassium dichromate, and 20 g. of salt, each in 50 ccm. of water, some students preparing one, others another. Cover the beakers containing the solutions and set them away to cool. After some time examine the resulting crystals. Remove some of the most perfect crystals, placing them on pieces of filter paper to dry. Describe any differences in form which you notice. Remove covers from the beakers and allow the solutions to evaporate until the original substances are recovered.

EXP. 26. WATER IN CRYSTALS

Materials. Crystals of copper sulfate, sodium sulfate, potassium chlorate, sodium carbonate, alum, salt. Calcium chlorid. Sodium hydroxid. Cobalt chlorid solution.

Apparatus. Dry test tubes. Glass plates or watch glasses.

Procedure. (a) Water in crystals.

1. Examine carefully a crystal of copper sulfate (blue vitriol). Do you notice any evidence of moisture about it?

Place a copper sulfate crystal weighing about 1 g. in a clean dry test tube, and heat it gently. Describe what happens. What appears on the cool sides of the test tube? Test it and see if it is water. From what source must this have come? Do you think it was chemically combined or just mixed with the copper sulfate?

When the copper sulfate undergoes no further change on gently heating allow the residue to cool and moisten it with a drop or two of water. What effect do you notice? The change in color is due to the formation of minute crystals of the original copper sulfate. The heated copper sulfate * unites with water to form blue vitriol or crystallized copper sulfate.

To obtain larger crystals, dissolve the residue in a little warm water, and allow the liquid to evaporate spontaneously. Compare the crystals which form with the original substance. Can you detect any difference?

2. Moisten some filter papers with a little cobalt chlorid solution and allow them to dry spontaneously. The pink color is due to crystals of cobalt chlorid which contain water. Warm three or four of the filter papers over a burner and note the change of color due to driving out the water from the crystals.

Moisten one of the blue papers with a drop of water and explain the action. Breathe upon another and allow a third to stand exposed to the air. This procedure is used as a *test for water*.

Blue cobalt chlorid paper acts as a barometer to indicate * This procedure, as well as that given in 2, is used as a test for water. whether the air contains much or little water vapor. In hot dry regions it will be blue, while a moist atmosphere is indicated by the pink color. Try it for your locality at various times of the year.

3. Heat dry crystals of the other substances and ascertain whether they contain water. Many (not all) crystals contain water.

(b) Water in crystals held chemically.

Note the taste of alum. Heat gently on an iron pan or on an evaporating dish a crystal of alum about the size of a hazelnut. When the alum undergoes no further change, turn it over so as to heat the other side. Then remove the flame.

When the piece of "burnt alum" is cool, break it open, and taste a bit of the inside. Is it any longer alum? What kind of a change was occasioned by the loss of the water? Was the water in the crystals chemically combined or were the crystals merely a mixture of water and "burnt alum"?

Put the "burnt alum" in a test tube, add to it about ten times its volume of water, and keep the whole near the boiling point for about five minutes. Then cool by holding the test tube in running water, and taste the liquid. What substance does the liquid now contain? How was it formed?

What distinction can you draw between the expressions "blue vitriol" and "copper sulfate"? Between "Glauber's salt" and "sodium sulfate"? Between "soda crystals" and "sodium carbonate"? Is there any difference between "salt" and "sodium chlorid"?

(c) Efflorescence.

On clean glass plates expose clear crystals of sodium sulfate and sodium carbonate, allowing the crystals to

E

stand several days if necessary. Then note what has happened. What becomes of the water which the crystals contained? Why should bottles containing crystals be kept corked?

(d) Deliquescence.

Expose on glass plates overnight pieces of calcium chlorid and of sodium hydroxid. Examine the substances the next day. From what source must the water have come? Calcium chlorid is sprinkled on roads to prevent them from getting dusty. Explain its action.

EXP. 27. PURIFICATION BY CRYSTALLIZATION

Materials. Alum. Copper sulphate. Potassium sulfate. Aluminum sulfate.

Procedure. (a) The separation of alum from copper sulfate.

Take 50 g. of alum, and for an impurity mix with it 10 g. of copper sulfate. Powder the mixture and dissolve it in 50 ccm. of boiling water. Filter the solution if it is not clear. Set the beaker containing the solution in a dish of cold water. Stir the contents of the beaker with a glass rod, and change the water in the dish if it becomes warm. When the liquid is cool, pour the contents of the beaker upon a filter. What is the color of the filtrate? What does this color indicate?

Wash the crystals on the filter by pouring over them several times 10 ccm. of cold water, allowing each portion to drain off entirely before adding another portion. Continue washing until the wash water is no longer blue. Then lay 3-4 filter papers, one on top of the other, on the table, spread out the crystals on the upper one and dry them by pressing with 2-3 other filter papers. When the moisture has been absorbed by the paper, examine the crystals and taste them. Of what do they seem to consist? Can you see any of the other substance?

A copper compound mixed with ammonium hydroxid gives a blue liquid. Dissolve a little of the crystallized substance in a little warm water and add ammonium hydroxid. If the *test for copper* indicates its presence, dissolve the crystals in as little hot water as possible, and recrystallize the alum.

NOTE. While many substances form pure crystals in solutions containing more than one substance, and, therefore, may be separated from each other by crystallization, others may not be separated in this way because they form mixed crystals or crystals of new substances, as shown in (b).

(b) Substances not separable by crystallization.

Dissolve 3.5 g. of potassium sulfate and 13 g. of aluminum sulfate in about 40 ccm. of hot water, and set the solution away to cool. When crystals have formed, remove them and by their taste identify them. Are these crystals a new substance or are they the same as one of the ingredients in the mixture ?

EXP. 28. REACTIONS RUN TO EQUILIBRIUM UNLESS PREVENTED BY SOME FACTOR

Materials. Conc. nitric (HNO₃), sulfuric (H₂SO₄), and hydrochloric (HCl) acids. Potassium nitrate, KNO₃. Potassium hydrogen sulfate, KHSO₄. Potassium chromate, K₂CrO₄. Sodium carbonate, Na₂CO₃. Lead nitrate, Pb(NO₃)₂. Pieces of copper.

Apparatus. Beakers. Test tubes. Stirring rods.

Procedure. (a) Reactions running to equilibrium, not to completion.

1. Test and see that solutions of potassium nitrate, potassium hydrogen sulfate and sulfuric acid do not attack copper but that nitric acid does.

2. To 10 ccm. of water in a beaker add cautiously 10 ccm. of conc. H_2SO_4 and mix thoroughly. Make a saturated solution by heating 20 g. of potassium nitrate with 10 ccm. of water and divide into two equal portions.

To half of the potassium nitrate solution add 5 ccm. of the H_2SO_4 (1:1) solution. Pour a few drops of the mixture upon copper and note the action, warming if necessary. What causes this action? Stand the remainder aside to cool and crystallize. Note the formation of clear needles of potassium nitrate.

3. Heat the other half of the potassium nitrate solution (prepared above) if necessary to keep the KNO_3 in solution and add 10 ccm. of the H_2SO_4 (1:1) solution. Pour a few drops of the mixture upon copper and note the action. What causes this action? Stand the remainder aside to cool and crystallize (if necessary overnight). Note the formation of opaque crystals of potassium hydrogen sulfate.

Discussion. Between potassium nitrate and sulfuric acid the only reaction which can take place under the condition of these experiments is represented by the equation:

potassium hydrogen hydrogen potassium hynitrate + sulfate \iff nitrate + drogen sulfate $KNO_3 + H_2SO_4 \iff HNO_3 + KHSO_4$ That some HNO_3 is formed in (2) is indicated by the fact that the mixture attacks copper; for, of the four substances represented in the equation, HNO_3 is the only reagent which will attack copper. This proves that some $\rm KNO_3$ has reacted with $\rm H_2SO_4$ to form $\rm HNO_3$. Yet, on cooling, potassium nitrate separates from the solution, proving that *all* of the $\rm KNO_3$ has not reacted with the $\rm H_2SO_4$ present. In other words, the reaction does not run to completion but to equilibrium, all four of the substances being present in solution.

In (3) twice the proportion of sulfuric acid is added that was present in (2). The mass action of the H_2SO_4 on the KNO₃ causes the reaction to run much further toward completion than in (2), as is indicated by the crystallization of KHSO₄ instead of KNO₃ from the solution. All four substances are still present in the mixture but there is more KHSO₄ than KNO₂.

When none of the substances present in a reacting mixture are removed from the sphere of action, reactions run to equilibrium.

Nitric acid boils at 120.5° , H_2SO_4 at 330° ; KNO₃ and KHSO₄ are volatile only at higher temperatures. By heating up the mixtures in (2) and (3) water will boil away first. When the temperature rises above 121°, HNO₃ will become a gas and boil away. What effect will this have on the reaction? Why? Before answering this question perform (b) if necessary.

(b) Reaction running to completion.

1. Equilibrium prevented by the formation of a gaseous substance.

To 10 ccm. of water add a drop or two of HCl. Taste the solution and note one of the characteristic properties of acids.

Dissolve 15 g. of sodium carbonate crystals (or 8 g. of anhydrous Na_2CO_3) in 25 ccm. of boiling water. Dilute 10 ccm. of conc. HCl with an equal volume of water and

heat to boiling. Remove from the flame and add gradu ally the sodium carbonate solution until a further slight addition of the latter produces no effervescence. Then cool the liquid and taste it. Is HCl any longer present? What common substance seems to have taken its place? Do you judge that any gaseous substance was formed during the reaction? Why?

The equation representing the reaction which has taken place is

sodium		hydrogen		sodium		water		carbon
carbonate	+	chlorid	\rightarrow	chlorid	+		+	dioxid
Na2CO3	+	2 HCl	\rightarrow	2 NaCl	+	H ₂ O	+	CO_2

Which of the reaction products escaped as a gas? Explain in detail how the formation of a gaseous substance causes the reaction to run to completion.

2. Equilibrium prevented by the formation of an insoluble substance.

Dissolve about 1 g. of potassium chromate in 100 ccm. of water and 2 g. of lead nitrate in about 20 ccm. of water and heat both solutions to boiling. Add half of the lead nitrate solution to the potassium chromate solution, stir thoroughly and allow the mixture to stand for a few minutes. When the precipitate has settled, add to the clear liquid above it a few drops of the lead nitrate solution. If a precipitate forms, add 1-2 ccm. more, stir and allow to settle once more. Test the clear liquid as before and add the lead nitrate solution, a little at a time, as long as a precipitate forms. Then filter the solution. If the filtrate is colored, add more lead nitrate and filter again.

All chromates are intensely colored. Judging from the color of the filtrate, does it contain any chromate? Judging by the color, where is the chromate? Did the reaction run to equilibrium or to completion? By evaporating the filtrate, needle-like crystals of potassium nitrate could be obtained. Where did the potassium come from? Where the nitrate? The equation representing the change is :

potassium lead lead potassium chromate + nitrate \rightarrow chromate + nitrate K_2CrO_4 + Pb(NO₈)₂ \rightarrow PbCrO₄ + 2 KNO₈

EXP. 29. HYDROGEN CHLORID, HCl

Materials. Salt, NaCl. Conc. sulfuric acid, H_2SO_4 . Magnesium ribbon. Manganese dioxid, MnO_2 . Copper oxid, CuO. Nitric acid, HNO_3 . Silver nitrate (AgNO₃) solution. Ammonia water (ammonium hydroxid), NH_4OH . Litmus paper.

Apparatus. A 250 ccm. flask fitted with thistle and exit tubes as shown in Fig. 19. A bottle. Piece of cardboard with hole in center.

Procedure. (a) Preparation.

To 10 ccm. of water in a beaker add cautiously 15 ccm. of conc. H_2SO_4 and allow the mixture to cool. Set up the apparatus as shown in Fig. 19. Place about 25 g. of salt in the flask, insert the stopper, and



FIG. 19.

through the thistle tube pour in the H_2SO_4 solution previously prepared. Hydrogen chlorid is evolved at once but the flask may be warmed if necessary to hasten action.

Complete the equation : $H_2SO_4 + 2 NaCl \rightarrow$

(b) Properties.

Is the gas colorless and invisible while (1) inside the flask; (2) outside the flask or bottle as it escapes into the air? Blow gently across the top of the bottle from which the gas is escaping. What causes the change in the appearance of the gas? In answering this question recall what happens to a cold window pane when breathed upon. Note the odor of the gas. Hold a moistened finger for an instant in the bottle of gas, and then touch the finger to the tongue. Bring a lighted match into the gas. Does it burn or support combustion?

Withdraw the delivery tube from the bottle and insert it as far as possible in a test tube and collect some gas by *downward displacement*. Close the test tube with the thumb and invert in a dish of water. Remove thumb and explain the result. Why not collect the gas over water?

(c) Hydrochloric acid, HCl.

Put about 20 ccm. of water into the bottle and again introduce the delivery tube, keeping the end just *above* the surface of the water. Continue the action as long as gas is readily absorbed.

Prove the identity of the solution of hydrogen chlorid thus obtained with the hydrochloric acid of the laboratory bottle by making the first four of the following tests on each liquid.

1. Note the effect of each on blue and red litmus paper.

2. Dilute considerably and note the taste.

3. Put 10 ccm. in a small test-tube and fill with distilled water. Invert in a dish of water and test the action of magnesium on each liquid, as directed in Exp. 17, (a). Are the reactions the same in each case? What is one constituent of hydrochloric acid?
4. Heat 10 ccm. in a test tube with a little manganese dioxid. Are the reactions the same in each case? The other constituent of hydrochloric acid, thus liberated, is *chlorin*.

5. Shake about 1 g. of copper oxide with 10 ccm. of hot water. Is it soluble? Add 5 ccm. of HCl and heat if necessary. Is there any reaction? How do you know? The substance formed is copper chlorid, $CuCl_2$. Write an equation showing its formation.

6. To a dilute solution of hydrogen chlorid or other chlorid, add a little nitric acid and a few drops of silver nitrate solution. Note the character of the precipitate which appears. To one portion of the liquid containing the precipitate add ammonia water until the solution will turn red litmus paper blue. What happens to the precipitate?

Other soluble chlorids besides hydrogen chlorid give these same reactions. This procedure is used as a *test* for chlorids.

The taste of hydrochloric acid, its action on litmus paper and on many metals and oxids are properties possessed by acids in general. State these general properties of acids.

EXP. 30. CHLORIN

Materials. Conc. hydrochloric acid, HCl. Manganese dioxid, MnO₂. Yellow phosphorus. Thin copper foil. Charcoal. Turpentine. A candle twisted into a wire for a holder. Potassium hydroxid (KOH) solution.

Apparatus as shown in Fig. 20. 3-4 bottles having the same size neck interchangeable with A. Wooden block. Beaker. A hydrogen generator with a clay pipestem tip.

Combustion spoon. Filter paper. Pieces of cardboard or glass plates.

NOTE. Chlorin is a very irritating gas, the effects of which may be counteracted by inhaling the fumes of alcohol sprinkled on a hand-



FIG. 20.

kerchief. This whole experiment should be performed under a hood or in a good draft.

Procedure. (a) Preparation.

Place about 30 g. of manganese dioxid in the flask and set up the apparatus as shown in Fig. 20. Through the thistle tube introduce about 50 ccm. of conc. HCl. Chlorin will be evolved at once but the flask may be warmed when necessary to hasten the reaction. To prevent chlorin from get-

ting into the room, cover the end of the exit tube in the beaker with water. When this becomes yellowish, showing that it is nearly saturated, add potassium hydroxid solution until the color disappears. Keep the resulting liquid for the next exercise.

Fill 6-8 bottles with the gas by downward displacement, removing A as soon as it takes on a yellowish color and substituting one of the other bottles. Cover each bottle, when removed, with a cardboard or glass plate. As soon as you have collected one or two bottles of gas, begin to perform the experiments, refilling these bottles for the later experiments.

Complete the equation: $MnO_2 + 4 HCl \rightarrow$

Is MnO_2 an oxidizing or reducing agent? What other substances that you have worked with do you think might be substituted for MnO_2 in this reaction?

(b) Physical properties.

Note the color and odor of chlorin. Compare with the color and odor of the water in the beaker in which the exit tube dips. Is chlorin soluble in water?

(c) Chemical properties.

1. Dry a small piece of phosphorus, observing the precautions mentioned on page 15. Place it in a combustion spoon and, without lighting it, introduce it into a bottle of chlorin. What happens? Phosphorus trichlorid, PCl_3 , is formed. Write an equation for the reaction taking place.

2. Heat the strip of copper foil and before it has had time to cool introduce it into another bottle of chlorin. What happens? Copper chlorid, $CuCl_2$, is formed; write the equation.

3. After testing its purity, the instructor will light the hydrogen as it issues from the clay tip attached to a hydrogen generator, and introduce the flame into another bottle of chlorin. Does chlorin support the combustion of hydrogen? Notice any change in the character of the flame. Allow the burning to continue for some time, and then withdraw the flame if it is not already extinguished. Hold a moistened finger in the gas in the bottle and touch it to the tongue. Note the effect of this gas on a moistened piece of blue litmus paper and blow gently into the bottle. What happens? What is the ash, *i.e.* the product formed, when hydrogen burns in chlorin? Write an equation expressing the reaction.

4. Set fire to a small piece of charcoal, noting how it burns in the air. Then introduce it, still glowing, into a bottle of chlorin. Does chlorin support the combustion of carbon?

5. The substances ordinarily burned for light or heat consist largely or entirely of carbon and hydrogen. A candle and turpentine are of this nature. To ascertain the action of chlorin on these substances containing both hydrogen and carbon proceed as follows:

Light a candle, and notice how it burns in the air. By means of a wire lower it, still lighted, into a bottle of chlorin. Though the candle continues to burn for a time, how does the character of the flame change? What becomes of the carbon in the candle? Blow gently across the mouth of the bottle. What becomes of the hydrogen in the candle?

If time permits, make a similar test with a small flame of illuminating gas burning in chlorin.

6. Heat 5 ccm. of turpentine to boiling in a test tube, taking care that it does not catch fire. Pour it over a large piece of filter paper, then drop into a bottle of chlorin before the turpentine has time to cool. Explain what happens.

When a substance consisting of carbon and hydrogen burns in chlorin what is the chemical action? Compare combustion in air with combustion in chlorin.

Is chlorin an element that is very active chemically?

EXP. 31. BLEACHING WITH HYPOCHLOROUS ACID, HC10

Materials. Calcium hypochlorite (contained in "bleaching powder" or "chloride of lime"). Hydrochloric acid, HCl. Potassium carbonate (K_2CO_3) solution. Litmus solution. Indigo solution. A bottle of dry chlorin. Pieces of calico or other colored cotton cloth.

Apparatus. Funnel and ring stand. Beakers. Watch glasses or evaporating dishes.

Procedure.

1. Notice the odor of fresh bleaching powder. Of what does it remind you?

2. Mix 10 g. of bleaching powder with 150 ccm. of water. In a short time filter the resulting liquid, and notice the odor of the filtrate. Is bleaching powder soluble in water?

3. To 5 ccm. of the solution on a watch glass add a few drops of HCl. Judging by the odor, what gas is liberated? What can you say of the stability of hypochlorous acid?

4. To 10 ccm. of the bleaching powder solution add a drop or two of litmus solution. Make a similar test with indigo solution and with bits of the colored cloth. Allow to stand if no result appears at first.

Make similar tests with the bleaching powder solution to which a little HCl has been added. Is the action quicker with or without the acid? Why?

5. To 20 ccm. of bleaching powder solution add potassium carbonate solution until a precipitate no longer forms. Filter off the precipitated calcium carbonate. The resulting solution, often called Javelle water, contains potassium hypochlorite and is frequently used in the home to remove stains. Test its power to bleach colored cloth, with and without the assistance of hydrochloric acid. Why do directions for home use often say to add vinegar to the Javelle water?

Write an equation indicating the formation of potassium hypochlorite.

Test similarly the solution in the beaker into which the exit tube of the chorin generator dipped in the last experiment. Does it seem to be identical with Javelle water? From what was it formed? Write an equation showing the reaction, consulting, if necessary, the reaction given below.

6. Into the bottle of dry chlorin introduce a piece of moist colored cloth and a similar piece that is thoroughly dry. Do not allow the two to come in contact. Which piece is bleached more rapidly? See whether pure water will bleach another piece of the cloth. Judging from the effect on the dyed cloth, what seems to be formed when chlorin and water are brought together? The equation expressing the reaction of chlorin and water is

$2 \text{ Cl} + \text{H}_2\text{O} \rightarrow \text{HCl} + \text{HOCl}$

What industrial use for chlorin and hypochlorous acid do you know of?

EXP. 32. THE PERCENTAGE OF OXYGEN IN POTASSIUM CHLORATE

THE WEIGHT OF ONE LITER OF OXYGEN

Materials. Potassium chlorate, KClO₃, dried on a radiator or in an oven.

Apparatus as shown in Fig. 21. A is a test tube of hard glass, connected by a one-holed rubber stopper and



a delivery tube to the liter bottle B. It is fitted with a tight rubber stopper pierced with two holes. Through one hole passes the tube leading from A. Through the other hole passes a tube, D, shaped like an inverted U, one arm of which reaches to the

bottom of B. C is a 500 ccm. beaker. Barometer and thermometer for general class use.

Procedure. Set up the apparatus as shown and see that all joints are tight by sucking on the end of the

U-shaped tube; then remove the test tube and weigh it carefully on a delicate balance. Into the tube put 1-1.5 g. of potassium chlorate. Jar the tube so that the potassium chlorate collects at the closed end, and then weigh again. Weigh the 500 ccm. beaker to a tenth of a gram on a platform scale. Fill the bottle nearly full of water and cause the tube D to fill with water by blowing gently into the delivery tube leading from A and plug the end of the U-tube with a squill of filter paper. Insert the stopper and delivery tube tightly into the test tube containing the potassium chlorate. Remove plug from Utube and place weighed beaker under the open end.

Heat the tube A in the flame of the burner until no further change in its contents takes place. Move the burner back and forth at first so as to make a gentle heat, but afterwards make it intense enough to decompose all of the potassium chlorate. If the molten mass froths and leaves a deposit high up in the tube, heat this deposit until it melts and runs back to the bottom.

When all the oxygen has been driven off and only molten potassium chlorid remains in the tube A, allow it to cool. Raise or lower the beaker so that the inverted U-tube dips as far into the water in the beaker as it does into the water in the bottle. Then remove the beaker and the test tube in the order named and weigh the test tube again on the delicate balance. Take the temperature of the water as that of the gas and note the barometric pressure. Tabulate results thus:

Weight of tube and potassium chlorate .					
Weight of tube empty \ldots		•2		• .	
(a) Weight of potassium chlorate taken				•	
Weight of tube and potassium chlorate .					
Weight of tube and potassium chlorid .	•	0	•		
(b) Weight of oxygen lost \ldots					-111

(c) Find how much oxygen would be obtained from 1 g. of potassium chlorate thus:

 $\begin{array}{c} \text{The weighed amount} \\ \text{of potassium chlo-} \\ \text{rate } (a) \end{array} \} : \left\{ \begin{array}{c} \text{the weight of} \\ \text{oxygen} \\ \text{lost } (b) \end{array} \right\} :: \left\{ \begin{array}{c} 1 \text{ g. of potas-} \\ \text{sium chlo-} \\ \text{rate} \end{array} \right\} : \left\{ \begin{array}{c} x \text{ g. of oxy-} \\ \text{gen.} \end{array} \right\} \\ \end{array}$

What is the percentage of oxygen in potassium chlorate?

(d) Figure out the weight of potassium chlorid left in the tube and calculate how much is formed from 1 g. of potassium chlorate.

(e) Weigh the beaker and the water it contains to the tenth of a gram and calculate the volume of water in the beaker, remembering that 1 ccm. weighs 1 g. The volume of the water is the same as the volume of the oxygen evolved. Why? Subtract the pressure due to water vapor (see text, p. 410) from the barometric reading to get the pressure of the oxygen, and reduce the volume to standard conditions.

What is the weight of this volume of oxygen?

Then, if the volume of oxygen in cubic centimeters has the weight in grams just recorded, 1000 ccm. (1 l.) of oxygen has a weight that is in the same ratio. Calculate thus the weight of 1 l. of oxygen under standard conditions.

Note. The instructor should average the results of the whole class, and should explain why the average result of careful work should be more nearly correct than most single experiments. He should also call attention to the way in which one careless bit of work may affect the results of a series of painstaking experiments.

EXP. 33. DETERMINATION OF THE VOLUME RATIO IN WHICH HYDROGEN AND OXYGEN COMBINE

Instructor's Experiment

Material. Mercury.

Apparatus. A eudiometer, coil, dry battery, beaker, and standard, as shown in Fig. 22. A hydrogen generator and oxygen generator, or

better hydrogen and oxygen drawn from gas holders. A cork to fit the eudiometer.

Procedure. Set up the apparatus as shown and fill the eudiometer completely, even the tip of the tube above the stopcock, with mercury. Introduce into the eudiometer about 20 ccm. of pure hydrogen, taking care that the tubes leading from the generator or gas holder are full of hydrogen (not of air) before connecting with the tube above the stopcock. In order



that gas may enter the apparatus it will be necessary to draw off some mercury into the beaker. When approximately 20 ccm. have entered, shut the stopcock and make the level the same in both arms by drawing off or adding mercury to the tube B. Measure the volume of hydrogen in the graduated tube A. After the volume of hydrogen has been noted, introduce about the same volume of oxygen, observing all the directions given for hydrogen. Note the volume of oxygen added.

Pour mercury into B until a quantity is present greater

F

than is necessary to fill completely one side of the apparatus, and press a cork into the open end of the tube B. Connect the wires fused in the top of the tube A with the coil and pass a spark through the mixed gases. What happens? Write an equation representing the reaction which takes place.

Remove the cork from B. Explain what happens?

The amount of water formed in the reaction condenses and occupies no volume worth consideration. Pour mercury into B until the level is the same in both sides of the apparatus, and measure the volume of the remaining gas, which in the present instance is oxygen, as may be proven by testing its effect on a glowing splint. Record your results as follows and calculate the volume ratio in which hydrogen and oxygen combine.

ccm. volume of hydrogen, ccm. volume of or	xygen introduced.
ccm. volume of oxygen, ccm. volume of ox	xygen left over.
ccm. total volume, ccm. volume of or	xygen used.
ccm. of hydrogen gas unite with ccm. of oxy	ygen gas to form
water. The ratio is : :	

Repeat the experiment, using about 30 ccm. of hydrogen and 10 ccm. of oxygen in order to show that it makes no difference which element is present in excess. Avoid using the exact ratio of 2:1, lest the violence of the explosion wreck the apparatus. Results varying from the correct ratio are caused usually by the inclusion of some air during the manipulation, or by impure gases.

EXP. 34. BASES

Materials. Solutions of sodium hydroxid, NaOH (89 g. in 1 l. of solution); of hydrochloric acid (165 ccm. of conc. HCl in 1 l. of solution); of copper sulfate, CuSO₄; of ferric chlorid, FeCl₈; and of magnesium sulfate,

 $MgSO_4$. Sodium. Lime (calcium oxid), CaO. Litmus paper. A solution of 1 g. of phenolphthalein in 100 ccm. of dilute alcohol (half water).

Apparatus. Beakers. Stirring rods. Test tubes. Evaporating dishes.

Procedure. (a) Methods of formation.

1. Action of strong metals on water.

Remove the oil from a piece of sodium about as large as a pea by pressing between filter papers. Throw it into an evaporating dish half full of water, watching the action through a piece of glass or standing at some distance to avoid being hit by spattering drops. What happens? When the action is over try the effect of the solution on the fingers. Touch a finger to the tongue. What is its taste? What is its effect on pieces of red and blue litmus paper? Add a few drops of phenolphthalein solution. What happens?

The substance contained in the water is sodium hydroxid. It is a typical strong base or alkali. Write an equation indicating its formation from sodium and water.

2. Action of the oxids of some metals on water.

Place a piece of lime as big as a hazelnut in a small beaker and pour upon it about 5 ccm. of water. What happens after standing 10 minutes or less? The substance formed is calcium hydroxid, $Ca(OH)_2$. Write an equation showing how it is formed from calcium oxid and water. Put some of the resulting substance in a test tube of water, shake thoroughly and allow to settle. Decant the clear liquid and test as you did the sodium hydroxid in (1). Is calcium hydroxid a strong base?

3. Insoluble bases by precipitation.

Place 2-3 ccm. of copper sulfate, ferric chlorid, and magnesium sulfate in separate test tubes and dilute each

to 10 ccm. with water. Add a few drops of sodium hydroxid to each solution and note the precipitation of the insoluble bases, $Cu(OH)_2$, $Fe(OH)_3$, and $Mg(OH)_2$. Write equations for their formation. Do the reactions run to equilibrium or to completion? Why?

Insoluble bases have no taste and no effect on the fingers or on indicators that is appreciable. They react with acids, however, just as soluble bases do.

(b) Neutralization.

Place 20 ccm. of NaOH solution in a beaker, add a few drops of phenolphthalein, and add, with stirring, HCl until the color just disappears. To be sure that no excess of acid is present, add NaOH drop by drop until the pink color reappears, then finally bleach with a single drop of acid.

Dip the fingers into the resulting solution, taste and smell it. Can you detect either base or acid? To be sure that the solution is not merely a mixture in which the properties of the acid hide those of the base, and vice versa, evaporate the solution to dryness and taste the solid left. What is it? Has a new substance been formed? To what class of substances does it belong? Write an equation expressing its formation.

(c) Methods of forming salts.

The several methods of forming salts illustrated in the laboratory work so far may be summarized as follows :

- 1. By the interaction of elements, as —— on ——
- 2. By the action of acids on metals, as on —
- 3. By the action of acids on oxids, as ---- on -----
- 4. By the action of acids on bases, as on —
- 5. By metathesis when there is formed
 - (a) a gaseous substance, as on —
 - (b) an insoluble substance, as on —

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EXP 35. NITROGEN — ITS PREPARATION AND PROPERTIES

Instructor's Experiment

Materials. Yellow phosphorus. Sodium nitrite, NaNO₂, or potassium nitrite, KNO₂. Ammonium chlorid, NH₄Cl.

Apparatus. A tin box-cover or some similar object that will float on water. A stoppered bell jar or a two-liter bottle cut off near the bottom and fitted with a stopper. A pneumatic trough. Apparatus as shown in Fig. 23, consisting of a half-liter flask fitted with a two-hole stopper through which pass a small separating funnel* and exit tube as shown. Ring stand and gauze. 5–6 fruit jars or bottles. Combustion spoon.

Procedure. (a) Natural nitrogen from air.

Float the cover hollow side up on water in a tank. Place in it a piece of phosphorus the size of a pea and light the phosphorus. Quickly cover the burning phosphorus with the bottle, pressing the bottle down into the water so that air is imprisoned inside the bottle over the water. Describe and explain what happens. What is the "smoke"? What constituent of the atmosphere is used up? The remaining gas is mostly nitrogen.

Allow the bottle to stand until the "smoke" has dissolved in the water and the gas in the bottle is clear; then make the water level the same inside and outside the bottle. The nitrogen in the bottle is sufficiently pure for most experiments. Does it have any color, odor, or taste? Determine by proper experiments whether nitrogen burns or supports combustion.

* A thistle tube will serve the purpose if a separating funnel is not available.

(b) Chemically pure nitrogen.

Set up the apparatus as shown in Fig. 23. Dissolve 16 g. of ammonium chlorid in 50 ccm. of water and intro-



duce the solution into the flask through the separating funnel. Dissolve 20 g. of sodium nitrite or 25 g. of potassium nitrite in 25 ccm. of water and put the solution into the separating funnel. Allow 5 ccm. of the nitrite solution to run into the flask, then close the stopcock. Heat the mixture with a burner, so regulating the heating

that the liberation of gas does not become too rapid. If the action slackens, add more nitrite solution, a few cubic centimeters at a time. When the air has been driven out of the apparatus collect several bottles of gas.

Determine by proper experiments whether this gas pure nitrogen — has color, odor, or taste; whether it burns or supports combustion. Ignite a piece of phosphorus in a combustion spoon and put it into a bottle of gas, replacing the cover to prevent the rapid escape of nitrogen. What happens? Does the gas seem to have the same properties as that prepared in (a)? Does nitrogen seem to be an inert or an active gas?

The liberation of nitrogen is due to the decomposition of ammonium nitrite, formed by metathesis from the reagents used. Water is another decomposition product. Write equations expressing the formation and decomposition of ammonium nitrite. What substance remains dissolved in the liquid in the flask?

EXP. 36. NITRIC ACID

Materials. Sodium nitrate, NaNO₃. Conc. H_2SO_4 . A conc. solution of ferrous sulfate, $FeSO_4$. Wooden splints. Small pieces of iron and zinc. Hair or wool.

Apparatus. A small glass retort. A flask into which the neck of the retort fits. Ring stand. A glass tube. Test tubes and a beaker. A dish or beaker full of water.

CAUTION: Do not allow nitric acid to come into contact with the skin or the clothes.

Procedure. (a) Preparation.

Place 30 g. of sodium nitrate in the retort, and set up the apparatus as shown in Fig. 24. Remove the stopper

from the retort, pour in 20 ccm. of conc. H_2SO_4 by means of a funnel and replace the stopper. Heat the contents of the retort gently, so that a liquid distills over into the flask. If the latter becomes hot, rotate it in the dish of water to keep it cool. Continue the heating with a small flame until no more nitric acid passes into the receiver. Why does the nitric acid cease



to distill? Does the reaction in the retort run to equilibrium or to completion? Why? Write the equation for the reaction. Consult the text and find two reasons why HCl could not be used in place of H_2SO_4 in making HNO₃. When cold, extract contents of retort with water.

(b) Properties.

1. Notice the color of the nitric acid; the yellowish tint is caused by gaseous impurities dissolved in the liquid.

Put a few ccm. of HNO_3 in a test tube, and heat to boiling. What colored gas is given off at first? What is the color of the vapor of HNO_3 later? What is the color of the HNO_3 left in the test tube?

2. Keep some HNO_3 boiling in a test tube and introduce into the fumes a glowing spark on the end of a long wooden splint. Does it continue to burn? What makes wood burn? Where does the wood get this substance? A decomposition product of HNO_3 may be seen in the test tube above the glowing spark. What is its color? Where did you see it before? Note color given to the uncharred wood by the HNO_3 .

3. Boil some HNO_3 in a test tube in the mouth of which is placed a loosely fitting plug of hair or wool. (Hood.) What happens? From the last two tests what conclusions do you draw as to the oxidizing action of HNO_3 ?

4. Pour some HNO_3 upon bits of iron and zinc in separate test tubes. Compare the action with that of HCl on these same metals.

5. To 5 ccm. of a conc. solution of ferrous sulfate, add a minute crystal of sodium nitrate and shake to mix thoroughly. Hold the test tube in an inclined position and pour down the side of the test tube about 5 ccm. of conc. H_2SO_4 so that it will underlie the solution without mixing. Note the formation of the dark ring on the line of contact. This procedure is used as a test for nitrates, for any other nitrate will give the same reaction.

EXP. 37. NITROGEN MONOXID, OR NITROUS OXID, N20

Materials. Ammonium nitrate, NH_4NO_3 . Phosphorus. Wooden splints. Cobalt chlorid solution.

Apparatus as shown in Fig. 25. A large beaker of about 1 l. capacity. Three bottles. Combustion spoon.

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While setting up the apparatus, place the large beaker full of water on a gauze over a burner and heat for use later.

Procedure. (a) Preparation.

Place about 15 g. of ammonium nitrate in the test tube and set up the apparatus as shown in Fig. 25. Heat

gently until the ammonium nitrate melts and gas is evolved, but do not cause the reaction to go too rapidly by overheating. Fill the bottles in which the gas is to be collected with warm water and collect three bottles of the gas.



The gas liberated is nitrogen monoxid, often called "laughing gas." Test the liquid that condenses in the side-neck test tube and see if it is water. Write the equation expressing the reaction taking place. How does it differ from the similar reaction used to prepare nitrogen (Exp. 35 b)?

(b) Properties.

1. Note the color, odor and taste of the gas. Fill a test tube with the gas by downward displacement, close with the thumb and invert in a dish of water. What happens? Why use hot water in collecting the gas?

2. Insert a burning splint into a bottle of the gas. Does nitrogen monoxid burn or support combustion?

3. Place a piece of phosphorus in a combustion spoon, ignite and insert in a bottle of the gas. What happens?

Nitrogen monoxid is decomposed into its elements at the temperature of burning wood or phosphorus. On this

basis explain why these substances should burn better in this gas than in air.

EXP. 38. NITROGEN DIOXID, OR NITRIC OXID, N202

Materials. Copper. Conc. nitric acid, HNO₃. Phosphorus. Wooden splints. Oxone (fused sodium peroxid), Na₂O₂.

Apparatus. Same as for making hydrogen, Fig. 13. Bottles. Combustion spoon. Small oxygen generator consisting of a side-neck test tube and medicine dropper as shown in Fig. 26.

Procedure. (a) Preparation.

Place about 20 g. of copper in the bottle, insert the stopper and through the thistle tube add about 20 ccm. of water and 10 ccm. of conc. HNO_3 . Action will begin at once, but additions of HNO_3 will be required later.

Collect three or four bottles of gas by displacement of water.

(b) Properties.

1. Note the color of the gas that first fills the generator. After it has bubbled through the water, note the color of the nitrogen dioxid that collects in the bottles. What must have become of the brown gas?

Remove the cover from a bottle of nitrogen dioxid and expose it to the air. What happens? How was the brown gas formed that first filled the generator?

2. To ascertain which constituent of the air causes nitrogen dioxid to turn brown, collect a half bottle of gas from the generator. Place a piece of oxone as big as a hazelnut in the little oxygen generator shown in Fig. 26. Fill the medicine dropper with water, insert the stopper and by gently squeezing let a couple of drops of water fall

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on the oxone. This generates oxygen according to the equations:

$$\begin{array}{c} \mathrm{Na_2O_2} + 2 \operatorname{H_2O} \rightarrow 2 \operatorname{NaOH} + \operatorname{H_2O_2} \\ \mathrm{H_2O_2} \rightarrow \mathrm{H_2O} + \mathrm{O} \end{array}$$

Fill the other half of the bottle containing nitrogen dioxid with oxygen. What happens? The brown gas is *nitro* genetetroxid, N_2O_4 . What constituent of the air is it that causes nitrogen dioxid to turn frown?

3. Insert a burning splint into a jar of gas, removing the cover only as little as is necessary. Does nitrogen dioxid burn or support combustion?

Place a piece of phosphorus in a combustion spoon, ignite and insert in a bottle of nitrogen dioxid. What happens?

Nitrogen dioxid is decomposed into its elements at the temperature produced by burning phosphorus, but not at the temperature of a burning splint. Explain the preceding results on this basis.

(c) The reactions.

Several different reactions take place at the same time when nitric acid acts on copper. The metal is oxidized by nitric acid to the oxid which then dissolves in more acid to form a salt as the equations show.

$$\begin{array}{c} 2 \operatorname{HNO}_3 \to \operatorname{H}_2 \operatorname{O} + \operatorname{N}_2 \operatorname{O}_2 + - \operatorname{O} \\ 3 \operatorname{Cu} + - - \to - \\ 3 - - + - \operatorname{HNO}_3 \to 3 \operatorname{Cu}(\operatorname{NO}_3)_2 + - - \end{array}$$

What colors the liquid in the generator blue?



FIG. 26.

EXP. 39. AMMONIA, NH₃

Materials. Ammonium chlorid, NH_4Cl . Freshly slaked lime (calcium hydroxid) $Ca(OH)_2$. Conc. HCl. Litmus paper. Wooden splint.

Apparatus. As shown in Fig. 27. Three bottles.

Procedure. (a) Preparation.

Take a quarter of a test tube full of ammonium chlorid and the same amount of calcium hydroxid. Smell of each and note whether either has an odor. Mix the two substances thoroughly on a paper and cautiously smell again. What is the odor? How could it have been produced? What would be the effect of raising the temperature? Complete the equation

$Ca(OH)_2 + 2 NH_4Cl \rightarrow CaCl_2 + ---+$

Put the mixture into the test tube and set up the apparatus as shown in Fig. 27 and heat gently. Turn the



outlet tube up and collect a bottle full of gas by *upward displacement* as shown by the dotted lines. What does this indicate as to the relative density of air and ammonia?

(b) Properties.

1. Hold a moistened finger in the gas and touch to the tongue. What is the taste of ammonia?

FIG. 27.

2. Thrust a lighted wooden splint

up into the bottle of ammonia. Does it burn or support combustion?

3. Put 3-4 drops of conc. HCl in a bottle, cover with a glass and shake thoroughly. Lift the bottle containing

 $\rm NH_3$ from over the outlet tube of the generator, cover with a glass plate and then place mouth upward on the table. Place the bottle containing the HCl mouth downward over the bottle containing the $\rm NH_3$ and remove both glass plates. Note what happens. Stand the bottles aside in order that the substance may settle. After it has settled remove the upper bottle and allow any ammonia to escape.

4. Collect some ammonia in a test tube by upward displacement, cover with the thumb and invert in a pan of water. Remove thumb and explain results. Why not collect ammonia over water?

(c) Ammonium hydroxid, NH₄OH.

Turn the outlet tube of the generator down and insert it in a bottle containing about 20 ccm. of water, keeping the end of the tube just *above* the surface of the water as shown in Fig. 27. Why not dip it into the water? Shake the bottle occasionally to mix the liquid thoroughly and continue the reaction as long as the gas is readily absorbed by the water. Satisfy yourself that the liquid in the bottle is identical with the ammonium hydroxid of the laboratory by noting the odor and effect on red litmus paper of each solution. A reaction must have taken place between the gas and the water. Complete the equation

 $---+--\rightarrow NH_4OH$

1. Heat in a test tube about 10 ccm. of the solution in the bottle, noting by the odor what comes off first. Continue the heating until about half of the liquid is boiled away. Can you now detect any ammonia? What must have happened to the ammonium hydroxid? Is the reaction written above reversible or not?

2. Add dilute HCl to the remainder of the liquid in

the bottle until it will just turn blue litmus paper red. Evaporate to dryness and compare the product obtained with that formed in (b) 3, as to taste, odor, effect of heat, and of sodium hydroxid upon each. The substance is in each case ammonium chlorid, NH₄Cl. Write equations expressing its formation in each case.

3. Any ammonium salt heated with a strong base, such as NaOH, KOH or $Ca(OH)_2$, will liberate ammonia, which may be detected by the odor or by red litmus paper. This is a *test for ammonium salts*.

EXP. 40. DECOMPOSITION OF AMMONIA BY ELECTRICITY Instructor's Experiment

Materials. Conc. ammonia water (ammonium hydroxid), NH₄OH. Salt. Wooden splints.

Apparatus. Same as used in the decomposition of water by electricity, Fig. 11.

Procedure. Into the bowl of the apparatus place a sufficient quantity of a mixture of conc. ammonium hydroxid and water (equal volumes) to cover the electrodes and dissolve in the mixture about 25 g. of salt. Fill the graduated test tubes or measuring glasses with the solution and invert over the electrodes. Connect the wires to a dynamo or battery and note the evolution of gas. At what rate does it collect in each test tube? When one of the tubes is full of gas remove it from the bowl and holding it mouth down thrust up into it a lighted wooden splint. What gas does it contain?

When the other tube is full of gas, test it similarly. What happens? This gas is nitrogen.

How many volumes of hydrogen combine with one volume of nitrogen to form ammonia?

NOTE. The reaction which takes place is not as simple as it appears. Investigation shows that it is the salt which is decomposed

by the electric current, sodium being liberated at the cathode and chlorin at the anode. Consult Exp. 16 (b) and explain why hydrogen is collected at the cathode in this experiment. Recalling the action of chlorin on compounds containing hydrogen (see Exp. 30 (c)), explain why nitrogen collects at the anode. What other product is formed by the sodium; by the chlorin? What becomes of these two products when they come together in the solution? What then is the action of the salt in the present experiment? Write equations for all the reactions involved.

EXP. 41. THE EQUIVALENT OF MAGNESIUM

Materials. Conc. HCl. Magnesium ribbon.

Apparatus. As shown in Fig. 28 consisting of a half liter flask, small evaporating dish and pan of water. Stiff

card or small square of glass. Graduate, thermometer, and barometer for general class use.

Procedure. Weigh accurately not more than 0.5 g. of magnesium ribbon.* Roll or tie it into a little ball that will readily slip into the neck of the flask. Place the ball of magnesium in the dish and the dish in the pan containing suf-



FIG. 28.

ficient water to cover the dish to a depth of 3 to 4 cm. Put 25 ccm. of conc. HCl into the flask and fill with distilled water. Cover the mouth of the flask with a card or small square of glass and invert in the pan of water outside the dish. Quickly bring the inverted flask over the magnesium in the dish so as to catch all the hydrogen liberated but allowing no air to enter the flask.

When the metal is completely dissolved, cover the mouth of the flask with the glass plate and remove to sink or deep vessel previously filled with water and adjust the flask so as to make the level of water the same inside

* See Suggestions to Teachers.

and outside the flask. Cover the mouth of the flask with the glass plate, remove from the bowl and place upright on the table. By pouring from a graduate, ascertain the volume of water necessary to fill the flask even full. The volume of water added equals the volume of hydrogen liberated.

Note the temperature of the water (equal to the temperature of the gas) and read the barometer. Subtract the pressure due to water vapor (see text, p. 410) from the barometric reading to get the real pressure of the hydrogen. Reduce the volume of hydrogen to standard conditions and calculate the weight of magnesium necessary to liberate 1 g. of hydrogen. This result is the equivalent of magnesium as obtained in your experiment. Repeat with another piece of magnesium, if time permits, and average the results. Record your results as follows:

		Exp. 1	Exp. 2
Weight of magnesium taken .		1 g. Ma.	g.
Volume of hydrogen obtained		v 6 ccm.	ccm.
Temperature		19 0	0
Barometer		77/ mm.	mm.
Water vapor pressure		16.35 mm.	mm.
Pressure of hydrogen		75465 mm.	mm.
Volume hydrogen (standard con	n-		
ditions)		ccm.	ccm.
Equivalent of magnesium			
Average			

EXP. 42. THE EQUIVALENT OF SODIUM

Apparatus. Wide mouth bottle of 250-500 ccm. capacity, pan of water, capsule (for holding sodium) and wire for holder as shown in Fig. 29. Wire nail to fit the capsule. Stiff card or small square of glass. Graduate, thermometer and barometer for general class use. **Procedure.** See that the capsule is thoroughly clean and dry and weigh it accurately. Cut off any crust from some pieces of sodium and remove any oil with filter paper. Fill the capsule even full with clean sodium, using the head of the nail to press it tightly into the capsule, and weigh. Twist the wire firmly about the capsule, being sure that it cannot drop out. Fill the bottle with water and invert in the pan of water, taking care that no air enters. Lean the bottle against the side of the pan as shown in Fig. 29, and quickly thrust the cap-

sule, mouth downward, under the mouth of the bottle so as to catch all the gas liberated. When the flow of gas slackens, tilt the capsule slowly so that water may again come in contact with the sodium, and finally turn the capsule mouth upward. If care-



FIG. 29.

less handling causes the sodium to escape from the capsule, stand back until all action ceases.

When the action is complete, measure the hydrogen evolved and take the temperature and pressure. Calculate and record the results as directed in Exp. 41. Repeat if time permits and average the results.

EXP. 43. PREPARATION OF AN ACID SALT

Materials. Conc. H₂SO₄. Solid KOH. Phenolphthalein solution. Litmus paper. Zinc.

Apparatus. Beakers. Evaporating dish. Stirring rod. Graduate or 20 ccm. pipette.

Procedure. Dissolve 7 g. of solid KOH in 20 ccm. of water. Add 10 ccm. of cone. H_2SO_4 to 40 ccm. of water. Measure 20 ccm. of the H_2SO_4 solution into an evaporating dish, add 15 ccm. of the KOH solution, and cool. To the

G

mixture add a drop of phenolphthalein solution and, stirring constantly, neutralize with the remaining KOH solution added drop by drop, until a faint pink color is produced. Write an equation indicating what has been formed. What kind of a salt is it? What other methods of forming salts do you know?

To the neutralized solution now add 20 ccm. of the H_2SO_4 solution and evaporate until a little of the substance removed on a glass rod solidifies on cooling; then remove from the flame and cool.

Note the properties of the hydrogen potassium sulfate thus formed. Dissolve a bit of it in water and cautiously taste the solution and note its action on blue litmus paper. Put some of the salt in a test tube with a piece of zinc, add a little water and notice the result. Is hydrogen potassium sulphate an acid as well as a salt? Write an equation indicating its formation.

EXP. 44. CARBON

Materials. Coal, wood, paper, bread, potato, vegetables, starch, cotton, wool, meat, candle. Sand. Kerosene. Powdered wood charcoal. Animal charcoal. Hydrogen sulfid water. Dark brown sugar. Copper oxid, CuO. Lime water, Ca(OH)₂.

Apparatus. Clay crucible or iron dish. Ring stand or tripod. Beakers. Test tube with cork. Funnel. A hard glass test tube fitted with stopper and exit tube as explained in (c). Pincers.

Procedure. (a) The occurrence of carbon.

1. (Hood.) Place a thin layer of sand in a clay crucible or small iron pan and on it put small pieces of some of the animal and vegetable material first listed above, different students using different materials. Cover this material with sand to protect it from the action of the air (Why?), or else place a cover on the crucible or pan. Support on a ring stand and heat strongly until all smoking ceases; then cool and examine.

One of two things will happen. The material will sometimes be volatilized; more often it will be decomposed, leaving a residue of charcoal. What happens with the substances you tried? Hold some of the black residue in the top of a Bunsen flame. Does it burn up as charcoal does?

2. Hold a cold glass tube in the yellow Bunsen flame and the flame of a candle and of kerosene. What is deposited on the cold glass? Where did it come from, the air or the burning substance? Name one element that the common liquid or gaseous fuels contain as shown by your experiments. Point out one similarity in composition between the fuels we burn and the foods we eat.

(b) The properties of amorphous carbon.

1. Fill a test tube one quarter full of powdered wood charcoal and pour into it 5 ccm. of water containing hydrogen sulfid. Cork the tube and shake thoroughly from time to time for 15 minutes. Then note whether the odor is gone. If not, add more charcoal and repeat the shaking until the odor disappears. State one reason why charcoal is used in water filters.

2. Dissolve 25 g. of brown sugar in 100 ccm. of water and note color of solution. Add 10 g. of animal charcoal and boil for 15 minutes; then filter. Note color of filtrate and taste it. Is the sugar still there? Repeat the treatment, if necessary, until the solution is nearly colorless. Why is animal charcoal used in refining sugar and other substances?

(Use the sugar solution for Exp. No. 51, and start the

fermentation to-day, allowing it to continue until you are ready to distill the mixture.)

(c) Carbon as a reducing agent.

Mix 5 g. of copper oxid intimately with an equal bulk of powdered charcoal, and heat the mixture strongly in a hard glass test tube, fitted with an exit tube so arranged that the gas liberated may bubble into 5 ccm. of lime water in a test tube. The precipitate obtained in the lime water indicates carbon dioxid (see Exp. 46 d). Sometimes a red glow appears inside the test tube as though its contents were on fire. After heating 5–10 minutes, cool, and pour out the contents upon the desk. What is the reddish material? What becomes of the charcoal? Write an equation expressing the reaction between charcoal and copper oxid.

EXP. 45. MANUFACTURE OF ILLUMINATING GAS

Instructor's Experiment

Material. Soft coal. Lead acetate. Litmus paper. Apparatus as shown in Fig. 25, except that the test tube must be of hard glass and the exit tube bent at a right angle, allowing the test tube to lie horizontally.

Procedure. Fill the hard glass test tube half full of chunks (not dust) of soft coal and set up the apparatus. Place pieces of moist red litmus paper and of filter paper moistened with lead acetate solution on opposite sides of the side-neck test tube, the ends projecting from the mouth of the tube so that they will be held in position by the stopper when inserted. Heat the coal gently at first, then strongly, and collect the gas evolved over water, noting its properties. Does it seem similar to the gas coming from the gas pipes? Different processes for making gas for fuel or illumina tion are used in different localities. How is it made in your city and from what material? Why is this process or material used rather than another?

What collects in the side-neck test tube? Note and explain any change in the papers. What makes the red litmus turn blue? Hydrogen sulfid is usually present, causing the formation of black lead sulfid when it comes in contact with lead acetate. Complete the equation and name the other product of the reaction.

$$Pb(C_2H_3O_2)_2 + H_2S \rightarrow PbS + ----$$

EXP. 46. CARBON DIOXID, CO_2 , AND CARBONIC ACID, H_2CO_3

Materials. Marble or limestone, CaCO₃. Conc. HCl. Lime water, Ca(OH)₂. Wooden splint. Bread. Candle. Apparatus such as was used in making hydrogen, Fig. 13. Test tubes. Beaker. Wire. Litmus paper.

Procedure. (a) Preparation.

Put 10-20 g. of marble or limestone into the generating bottle, add 50 ccm. of water, insert the stopper and through the thistle tube pour 5 ccm. of conc. HCl. Collect three bottles of gas, adding more acid from time to time if necessary to hasten the action. Complete the equation:

 $CaCO_3 + - HCl \rightarrow - + H_2CO_3 \rightarrow - + CO_2$ Any of the common acids may take the place of HCl in this reaction. Why?

(b) Physical properties.

Note the color, odor, and taste of carbon dioxid and ascertain whether it will burn or support combustion.

Set a lighted candle in a bottle of air and invert over it a bottle of carbon dioxid. Explain the result.

(c) Chemical properties.

1. Put a piece of blue litmus paper into a little water and let carbon dioxid bubble into it. Explain what happens. Is carbon dioxid an acid anhydrid?

2. Add an equal volume of lime water to the solution just formed and explain what happens.

3. Shake the mixture obtained in (2) and pour out all but 5 ccm. and continue to pass the carbon dioxid through this volume. What happens? Acid calcium carbonate, soluble in water, is formed.

Complete the following equations expressing the changes which have taken place and write similar equations showing the products formed when CO_2 is absorbed by NaOH.

$$CO_{2} + H_{2}O \rightarrow \underline{\qquad}$$

$$Ca(OH)_{2} + H_{2}CO_{3} \rightarrow \underline{\qquad} + \underline{\qquad}$$

$$CaCO_{3} + H_{2}CO_{3} \rightarrow CaH_{2}(CO_{3})_{2}$$

(d) Testing for carbon dioxid.

When treated with HCl, any carbonate liberates carbon dioxid just as marble does. The formation of a precipitate in lime water is a *test for carbon dioxid and carbonates*. Carbon dioxid is so heavy that it may be poured from one bottle to another just like water, as the following experiment shows.

1. Pour about 20 ccm. of lime water into a bottle and shake it. No significant change should occur, though, on long standing, lime water shows the presence of small amounts of carbon dioxid in the air. (Try it.) Pour a bottle of carbon dioxid into the lime water and shake again. What happens?

2. Hold a burning wooden splint in a bottle of air

until it is extinguished, add a little lime water and shake. What is the result? Where did the carbon dioxid come from?

3. Stick a piece of bread on a wire, hold it in a flame until it takes fire, then lower it into another bottle of air. When the flame goes out, test with lime water as before and explain where the carbon dioxid comes from.

4. Blow the breath through a glass tube into a little lime water. Explain the result. How was the carbon dioxid in the breath produced? What similarity is there in the burning of coal or wood under the boilers of an engine and the use of food in the body? Why is the temperature produced higher in one case than the other? What causes the body temperature to be higher than that of the atmosphere?

EXP. 47. CARBON MONOXID

Instructor's Experiment

Materials. Charcoal. Oxygen. NaOH. Lime water. Apparatus as shown in Fig. 12, except that the iron pipe is replaced by a combustion tube of hard glass. This is supported in the furnace on a strip of sheet iron to prevent sagging of the tube when heated. In place of the boiling flask a 2-liter bottle is attached to the combustion tube by means of an exit tube, passing through a 2-hole rubber stopper fitting the neck of the bottle. Through the other hole in the stopper passes an inlet tube reaching to the bottom of the bottle, the outer end being connected to the water tap. Short piece of rubber tubing. Pinch cock.

Procedure. Fill the 2-liter bottle with oxygen by displacement of water and insert the stopper carrying the inlet and exit tubes. Close the inlet tube with a piece of

rubber tubing and a pinch cock. Fill the combustion tube with small pieces of wood charcoal* (no dust) and connect it to the oxygen bottle. Pour NaOH solution into the wash bottle until the end of the inlet tube is covered to the extent of 1 cm.

Heat up the combustion tube moderately to begin with, lighting the center burners first. All joints must be tight, as will be indicated by bubbles passing through the NaOH solution. When the tube is hot, connect the inlet tube of the oxygen bottle to the water tap, so that as water enters, oxygen will be forced slowly over the heated charcoal. Regulate the size of the stream so that, it takes from 10–15 minutes to fill the bottle with water. Collect two or three bottles of carbon monoxid over water as it escapes from the apparatus. When the oxygen bottle is full of water, shut off the water, disconnect the combustion tube at both ends, and turn off the gas in the furnace.

(CAUTION: Carbon monoxid is poisonous. Avoid breathing the gas!)

Note the color and odor of the gas and ascertain whether it will burn and support combustion. Describe the flame produced.

Introduce 25 ccm. of lime water into a bottle full of carbon monoxid and shake. Is there any reaction? How could you tell the difference between carbon monoxid and carbon dioxid? Set fire to this same bottle of gas and shake the lime water with the products of combustion after the flame has gone out. What happens? To what does carbon monoxid burn?

* Ordinarily charcoal has not been distilled at a sufficiently high temperature to remove all volatile material. Before performing this experiment it is well to heat the charcoal in the combustion tube in the furnace while passing a slow current of hydrogen through the tube. In this way considerable tarry matter will be removed. *Discussion.* When oxygen and carbon come together inside the combustion tube, the latter burns just as it does in air, forming carbon dioxid. As this gas passes over the hot carbon it is reduced to carbon monoxid. The sodium hydroxid wash bottle is to remove any unchanged carbon dioxid. Write equations indicating all reactions taking place.

Explain in detail how in a similar way carbon monoxid is produced by coal stoves or charcoal braziers, sometimes causing death to the inmates of the house. If carbon monoxid is burned, is this danger removed?

EXP. 48. BURNING AND SUPPORTING COMBUSTION

Instructor's Experiment

Materials. Wooden splints.

Apparatus. A piece of moistened cardboard having a hole in the center, placed on top of a lamp chimney fitted

with a stopper pierced by two tubes as shown in Fig. 30. The tube A is wider and shorter than G. Clamp and ring stand.

Procedure. Support the chimney in a vertical position by means of a clamp and ring stand. Connect the tube G to a gas jet and allow illuminating gas to flow at full pressure into the apparatus for a moment. Then turn the gas partly off and light it as it escapes from the hole in the cardboard. Thrust a



lighted wooden splint up into the tube A. Describe and explain what happens. What difference is there in the chemical reaction taking place within and without the chimney? What other gases which you know might be substituted (a) for the illuminating gas; (b) for the air? Under what conditions is it said that a substance burns

and another supports combustion? Under what conditions could you reverse the statement?

EXP. 49. FLAMES

Materials. Powdered charcoal. Piece of cardboard 10-15 cm. square. HNO₃.

Apparatus. Bunsen burner. Copper wire. Blowpipe, or glass tube 50 cm. long from which to make it. Short piece of glass tube.

(a) Why a flame gives light.

1. Notice the blue flame of a Bunsen burner. Does it give considerable light?

2. Hold a piece of wire or glass tube in the flame, and notice how it emits light as it becomes hot. What would be the effect on the luminosity of the flame if it should become filled with hot solid particles? By knocking together two blackboard erasers, fill the flame with fine dust and note the effect. Throw some powdered charcoal into the flame and note the effect.

3. Close the air holes at the bottom of the Bunsen burner and notice the change in luminosity of the flame. Are there any visible solid particles which can cause it to emit light? Hold in the flame a cold glass tube. What solid material is deposited? Could it have been the source of the light of the flame? What becomes of it when the flame is not interfered with? From what is it formed in the flame? Why is an ordinary flame luminous?

(b) The structure of a flame.

1. Note the structure of a Bunsen flame as shown in Fig. 31.

2. Using a Bunsen flame with a sharp inner cone, G, about an inch high, hold a piece of cardboard in the center of the flame, first vertically, resting the cardboard on the top of the burner tube; secondly, horizontally, about an inch above the top of the tube. As soon as the cardboard begins to char, remove it quickly from the flame before it takes fire. The charred outlines are cross-sections of the flame, indicating the hotter parts.

3. Prove that the inner cone, G, consists of unburned gas by holding a glass tube in it as shown in Fig. 32, and light the gas as it issues FIG. 31. from the tube.

4. Clean a piece of copper wire by dipping it in HNO₂ for a moment, then washing in water. Hold one end of

> this wire in the top of the flame and explain why it turns black. Lower the wire to a point just above the inner cone and observe and explain how the original copper color returns. Remove the wire from the flame and note how the hot copper turns black in the air before it cools. Explain.

(c) The blowpipe flame.

If one is not already available, make a piece of glass tubing of the size and shape shown in D, Fig. 5, and draw out the shorter end as shown in Fig. 3. Cut the

slender part straight across so as to form a tip with an opening not larger than the diameter of a pin. Thus you have made a blowpipe. Preserve it for use in later experiments.

Turn down the gas in a Bunsen burner until a yellow flame about 4 cm. high results. Hold the blowpipe as

FIG. 32.







shown in Fig. 33, and blow gently. When the breath is nearly exhausted, use the cheeks as a bellows while inhaling a fresh breath through the nose. With a little practice a steady, narrow, very hot flame may be obtained. Note that the blowpipe flame has the same structure as the blue Bunsen

flame. The oxidizing flame may be increased in size and the reducing flame diminished by blowing somewhat stronger, and vice versa.

EXP. 50. HYDROCARBONS

Materials. Kerosene. Candle. Cobalt chlorid solution. Calcium carbid, CaC_2 . Lime water, $Ca(OH)_2$. Red litmus paper. Gasoline or benzine. Lard, tallow, or butter. Filter paper.

Apparatus. Small flask with stopper. Two wide-mouth bottles. Glass plate. Two watch glasses or evaporating dishes.

(a) Composition.

Procedure. 1. Fill a small flask full of cold (ice) water and cork it tightly. Wipe the flask dry and hold it over the flame of burning gas, kerosene, and a candle. Wipe off the moisture in each case with a piece of cobalt chlorid test paper. What is indicated in each case? How was it formed? Where must the hydrogen have come from, the air or the burning material? Outline a general
method for ascertaining whether a burning substance contains hydrogen.

2. Hold a piece of cold glass tubing in the yellow flame of gas, kerosene, and a candle. What is deposited in each case?

The common gaseous and liquid fuels consist almost wholly of the two elements indicated. What are the final products of combustion when carbon and hydrogen burn? Write equations indicating the products formed when illuminating gas (consisting mainly of CH_4 and H_2) burns. Write a similar equation for the burning of kerosene, assuming it to have the formula $C_{10}H_{22}$.

(b) Common gaseous hydrocarbons.

1. Fill a wide-mouth bottle one seventh full of illuminating gas by displacement over water. Raise the bottle from the water for a moment and allow air to displace the remaining water. Cover with a glass plate and stand upright on the table. Remove the plate and bring a flame to the mouth of the bottle. What happens? Why does the gas explode now though it burns quietly in the burners?

2. Fill a test tube and invert in a dish of water. Drop a small piece of calcium carbid into the dish and quickly collect the liberated acetylene. Note its odor. Close the test tube with the thumb and bring to a flame. Describe how acetylene burns. What is deposited by the flame? Calculate the percentage composition of acetylene and of methane, CH_4 , the chief constituent of illuminating gas, and see if you can get an idea why one deposits more soot than another. Test the water left in the dish with red litmus paper. Complete the equation and figure out what is present.

 $CaC_2 + 2 H_2O \rightarrow C_2H_2 + ----$

(c) Common liquid hydrocarbons.

CAUTION: Keep the bottles containing gasoline and benzine away from all flames !

1. Pour 1 ccm. (25 drops) of kerosene into a watch glass or evaporating dish and the same volume of gasoline or benzine into another and note the time it takes each to evaporate. Which is the more volatile?

2. Pour not more than 25 drops of gasoline or benzine into a wide-mouth bottle, shake well, stand upright on the table and bring a flame to the mouth of the bottle. Explain what happens. Treat similarly 25 drops of kerosene and explain the difference in the results. Why is it that explosions happen so frequently with benzine and gasoline?

3. Try the solubility of fats, such as lard, tallow, or butter, in benzine or gasoline. Why are these liquids used in "dry cleaning"?

EXP. 51. ALCOHOL AND ACETIC ACID BY FERMENTA-TION

Materials. Brown sugar, or sugar solution from Exp. 44. Yeast cake. Sodium acetate, $NaC_2H_3O_2$. Conc. H_2SO_4 . Alcohol, C_2H_5OH . Lime water, $Ca(OH)_2$.

Apparatus consisting of a liter flask fitted with an exit tube leading into a small flask used as a wash bottle. This wash bottle containing lime water is protected from the air by having its exit tube dip into water in a test tube. Distillation apparatus as shown in Fig. 18. Evaporating dish.

Procedure. (a) Alcohol

With a little water rub into a thin paste a quarter of a cake of yeast and add it to the sugar solution used in

Exp. 44, adding more sugar until you have about 50 g. in 500 ccm. of water. Set up the apparatus as directed above, placing the sugar solution in the large flask and about 50 ccm. of lime water in the small flask. Add just enough water to the test tube to cover the end of the exit tube. Allow to stand in a warm place ($25-30^{\circ}$ C.) for about a week.

Fermentation is soon indicated by bubbles of gas appearing in the sugar solution and later passing into the lime water. What is the gas liberated, judging by the reaction in the lime water? (If no reaction is noted, the apparatus leaks.)

Leaving the sediment in the bottom of the fermentation flask, decant two thirds of the liquid, and distill it at as low a temperature as possible until about 20 ccm. of distillate are obtained. This should consist of alcohol mixed with some water. A second distillation and collection of the first half of the distillate will reduce the proportion of water, which may be removed completely by allowing the mixture to stand for some time over quicklime. This treatment will ordinarily not be necessary to get evidence of the presence of alcohol by the following tests.

Pour the distillate into an evaporating dish and dip a piece of filter paper in it. Set fire to the paper, drop it into the dish, and note the character of the alcohol flame. If the filter paper will not take fire, too much water is present. Pour the distillate into a test tube, warm nearly to boiling, and bring the mouth of the tube to a flame. The alcohol will be volatilized (b. p. 78°) and burn at the mouth of the tube.

(b) Acetic acid.

1. Allow the remainder of the mixture in the fermentation flask to stand open to the air for several weeks,

noting the odor, and testing with blue litmus from time to time. The characteristic sour smell of acetic acid, the active constituent in vinegar, will develop eventually.

2. Add 5 ccm. of conc. H_2SO_4 to 1 g. of any acetate, and warm. Note the sour odor of the acetic acid that is liberated. Add 3 ccm. of alcohol and warm again. Note the sweet odor of ethyl acetate. Both of these reactions are used as *tests for acetates*. Complete the equations:

EXP. 52. SOAP

Materials. Lard, tallow, or cottonseed or olive oil. Solid NaOH; also solution. Alcohol. HCl.

Apparatus. A porcelain dish and ring stand.

Procedure. In the porcelain dish dissolve 2 g. of NaOH in 20 ccm. of water, add about 10 g. of the fat and 20 ccm. of alcohol, and boil gently until the odor of alcohol is gone and the contents of the dish is a pasty mass.

The alcohol takes no part in the chemical action, but serves to dissolve both the fat and the sodium hydroxid, so that they act more rapidly on each other. In practical soap-making no alcohol is used. Complete the equation, and write the name of each substance beneath its formula:

$\begin{array}{c} \overline{\mathrm{C}_{3}\mathrm{H}_{5}(\mathrm{OOC}\cdot\mathrm{C}_{17}\mathrm{H}_{35})_{3}} + - \mathrm{NaOH} \rightarrow \mathrm{C}_{3}\mathrm{H}_{5}(\mathrm{OH})_{3} + \\ - \mathrm{NaOOC}\cdot\mathrm{C}_{17}\mathrm{H}_{35} \end{array}$

What is soap, stated in chemical terms? Do you see any similarity between the reaction by which soap is made and the reaction of alkalies on ammonium salts (p. 78)?

Dissolve some of the resulting substance in warm water and test it with the fingers. Does it appear to be soap? To some of the soap solution add HCl, and shake vigorously. The curdy substance that forms is a mixture of stearic with other organic acids. Remove the curdy material to a test tube, add NaOH solution, and warm. Explain why the precipitate dissolves, and what is formed.

EXP. 53. CONSTITUENTS OF FOODS

Materials. Cottonseed or olive oil, or lard or butter. Corn meal. Raw egg. Hard boiled egg. Meat. Milk. Nuts. Cheese. Peas or beans. Starch. Flour. Bread. Cane sugar. Glucose or some sirup. Fruit. Potato. Vegetables. Filter paper, cotton, linen, hemp, silk, wool. Mixed cotton and woolen goods. Ether or benzine. H_2SO_4 . HNO₃. NaOH. NH₄OH. Water solution of iodin. Fehling's solution made from tablets or prepared by dissolving 9 g. of copper sulfate crystals, 45 g. of Rochelle salt, and 20 g. of sodium hydroxid in 1000 ccm. of water.

Apparatus. Unglazed paper. Litmus paper. Test tubes. Beaker. Funnel. Watch glass or evaporating dish. Porcelain crucible. Pipestem triangle.

Procedure. (a) Fats.

1. (CAUTION: Keep ether or benzine away from flames!) To an eighth of a test tube full of corn meal, add 10 ccm. of ether or benzine, cork, and allow to stand 10–15 minutes. Then filter into a watch glass or evaporating dish and stand in a draught of air to evaporate. Note what is left. Does it seem to be a fat or oil? This method is used as a *test for fat*.

2. A simpler method of detecting considerable fat is as follows: Put a drop of fat on a sheet of unglazed paper, hold to the light, and note the effect. Press a piece of the

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yolk of hard-boiled egg upon the paper; after a moment scrape it off and hold to the light as before. Does yolk give a test for fat? Warming will sometimes help this test by melting the fat contained in the substance.

3. Test 2 or 3 other foods for fat, different students using different foods.

(b) Carbohydrates.

1. Sugar. Examine and taste *glucose*, noting its properties. It is difficult to crystallize and is not usually seen in solid form. Most sirups consist mainly of glucose.

Heat 10 ccm. of Fehling's solution to boiling, add a little glucose, boil again, and note the result. This procedure is used as a *test for sugar*. (Several sugars do not give this test, though the common ones do, when properly treated.) If much sugar is present, it can be detected by the taste.

Examine and taste *cane sugar*, noting its properties. It crystallizes readily. Heat 10 ccm. of Fehling's solution to boiling as before and add a few grains of cane sugar. Does it give the test?

Dissolve 1 g. of cane sugar in 10 ccm. of water, add 1 drop of H_2SO_4 and boil for 5–10 minutes. Then add 10 ccm. of Fehling's solution and boil again. Does it give the test for sugar now?

Test 2 or 3 foods for sugar, boiling with acid if it does not give the test without this treatment.

When boiled with any acid, cane sugar is converted into glucose, and levulose, a body resembling glucose closely. Explain how a food may taste sweet, yet not give Fehling's test for sugar until after it is boiled with acid.

In making taffy, caramels, butter scotch, etc., what is the use of the vinegar or other acid added before boiling? If you wanted the candy to "sugar" quickly, *i.e.* crysEXPERIMENTS

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tallize well, would you boil the mixture with vinegar or other acid? Why?

Fehling's solution consists essentially of cupric oxid dissolved in a solution of an alkaline tartrate. When boiled with glucose the cupric oxid, CuO, is reduced to cuprous oxid, Cu₂O, which is not soluble in the tartrate solution and is precipitated as a red powder.

2. Starch. Notice the properties of starch. Moisten it with a water solution of iodin and describe the result. Grind 1 g. of starch to a fine powder, make into a thin paste with 10 ccm. of water, then pour the mixture into 100 ccm. of boiling water and boil 5 minutes. Cool a portion of the boiled *starch paste* in water and then add a drop of iodin solution. Result? Does the iodin produce a more striking effect on boiled starch than when it is not boiled? The blue color given by iodin is a *test for starch*. Heat destroys the color.

Test two or three foods for starch, both before and after boiling.

3. Cellulose. Examine filter paper, cotton, linen, hemp, etc., and note the properties of cellulose, especially its toughness. Note how readily it burns when dry, without fumes or much smoke and without swelling up.

The fibrous matter in vegetable material is cellulose. Wood and grass contain much, sometimes also celery and parsnips. Other vegetables contain little unless they have become "pithy," *i.e.* full of cellulose. It is not digested by human beings, hence is of no value as a food constituent. Many animals utilize it as food, however.

(c) Proteid.

1. Heat some silk, wool, meat, peas or beans and notice how the material swells up as it burns. Note the smoke and the odor produced. Compare with cellulose.

2. Heat one of the above in a test tube, noting the odor produced. Hold a piece of red litmus in the fumes in the tube. What happens? Explain why.

3. Shake a little albumen (raw white of egg) with water in a test tube. It goes into solution, does it not? (Many proteids are soluble in water.) Heat the mixture to boiling and note result. Proteids are coagulated by boiling. What is the scum that forms when milk, meat, and many vegetables and fruits are boiled? This is frequently rejected. Do you think it contains nutrient material?

4. Warm some of the white of a hard-boiled egg with HNO_8 for a moment, pour off the acid into another test tube, and note the color produced. Add NH_4OH to the egg and note the effect. The yellow color produced by nitric acid is a *test for proteid*.

5. Test 2 or 3 other foods for proteid, different students using different materials.

(d) Mineral matter.

(Hood.) Heat some one of the foods you have been testing in a porcelain crucible without cover over the Bunsen flame until the black color due to carbonization has disappeared. Is there much ash left? An incombustible residue left after burning is a *test for mineral matter*.

(e) The amount of water contained in a substance may be determined by heating in a drying oven to 105° C. for several hours.

After discussion in the class room, record the results obtained by the different students as follows:

FOOD FAT SUGAR STARCH PROTEID MINERAL WATER Potato None None Much Little Little Much *Clothing.* How could you tell the difference between linen or cotton on the one hand and silk or wool on the other by burning each? Boil a little cotton cloth,

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all wool, and a mixed wool and cotton goods one after another for 5 minutes in NaOH solution. What is the result. Outline another method of distinguishing between wool and cotton.

EXP. 54. THE HALOGEN GROUP

Materials. Powdered fluorspar, calcium fluorid, CaF₂.
Salt, NaCl. Potassium bromid, KBr. Potassium iodid,
KI. Conc. H₂SO₄. Manganese dioxid, MnO₂. Paraffin.
Apparatus. Test tubes. Lead dish. Glass plate.
Procedure. (a) Hydrids of the halogens.

Place 1 g. of calcium fluorid in a test tube, add 3 ccm. of conc. H_2SO_4 , and note what happens. Warm if necessary to hasten the reaction. Treat in the same way 1 g. each of sodium chlorid, potassium bromid, and potassium iodid in separate test tubes. Blow across the mouth of each tube. In each case what causes the fuming? Hold blue litmus in the fumes. What similarities do you observe in the four reactions? Write equations showing what is formed in each case.

Does the mixture in any of the tubes become colored? If so, what colors are produced and which one changes most quickly? This change is due to oxidation of the hydrogen compound first formed by air or H_2SO_4 . To increase this effect, add a pinch of manganese dioxid to each test tube and note what happens in each case. To prevent fumes getting into the room, wash out the test tubes as soon as the results are noted.

Tabulate your results as follows:

Compound HF	Oxidized by Air Not at all	Oxidized by MnO2 Not at all	STABILITY Greatest	
HCl				
HBr				
HI				

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Hydrogen fluorid is not affected by the most vigorous oxidizing agents. Hydrogen chlorid is oxidized by strong oxidizing agents, hydrogen bromid by mild oxidizing agents, and hydrogen iodid is readily oxidized even by the air. Write equations indicating the products formed in each case.

(b) Etching glass by hydrofluoric acid.

1. Put a drop of conc. H_2SO_4 on a piece of glass, sprinkle into it a pinch of powdered fluorspar, and after some minutes note the action of the liberated hydrofluoric acid on the glass.

2. Warm a glass plate about 10 cm. square and cover one side of it with a thin coating of paraffin. Through this wax, when cold, scratch letters or figures with a blunt instrument, taking care that the lines are not



too fine, and that the wax is removed through to the glass. In a lead dish mix about 5 g. of powdered fluorspar with enough conc. H_2SO_4 to make a thin paste, lay the glass plate face

down on the dish (see Fig. 34), and allow the exposed places to be acted upon for 24 hours by the fumes of hydrofluoric acid.

Scrape off the wax as much as possible, removing the last of it with a cloth moistened with alcohol or benzine (Avoid flames !) and note how the glass is etched.

EXP. 55. BROMIN

Materials. Bromin water (Br₂) containing a little liquid bromin at the bottom of the bottle. Chloroform,*

* Carbon disulfid, CS_2 , may be substituted for chloroform, though it should be distilled to render it colorless. Carbon disulfid must be kept away from all flames.

CHCl₃. Alcohol, C_2H_5OH . Colored calico. Potassium bromid (KBr) solution. Chlorin water, Cl_2 .

Apparatus. Test tubes.

Procedure. (a) Examine bromin water and state the properties of the liquid bromin at the bottom of the bottle. Does bromin appear to be considerably soluble in water? Cautiously smell of the solution but avoid inhaling much of the vapor, as bromin attacks the membranes of the eyes, nose, and throat even more than chlorin does. Inhaling the fumes from a handkerchief moistened with alcohol will partially overcome the effects.

Allow a piece of colored calico to lie in bromin water for some time. Do you note any bleaching action similar to chlorin?

(b) 1. To 10 ccm. of water in a test tube add about 2 ccm. of chloroform, shake thoroughly, then allow the tube to stand for a few moments. Are the liquids apparently soluble in each other?

Add 2 ccm. of bromin water, shake again, then stand as before. Judging by the color, where is the bromin, in the water or dissolved in the chloroform?

The fact that bromin will collect in and give color to chloroform is used as a *test for free bromin*.

2. To see whether bromin in combination with other elements will give this test, add 5 drops of KBr solution to 10 ccm. of water and shake with chloroform as before. The result which you get is typical of all *compounds* containing bromin. Make a general statement covering your results.

3. To the mixture used in (2) add 5 ccm. of chlorin water and shake again. Result? Do you get a test for free bromin? What must have set the bromin free? Complete the following equation:

 $- \text{KBr} + \text{Cl}_2 \rightarrow - \text{KCl} + ---$

Shake chlorin water with chloroform and see if chlorin possibly gives the same color as bromin.

Another method of liberating bromin was used in Exp. 54. Make a general statement covering this method. Outline a method of preparing bromin from KBr based on the experiments you have made.

EXP. 56. IODIN

Materials. Iodin. Alcohol. Carbon disulfid. Chloroform.* Starch paste.[†] Potassium iodid (KI) solution. Chlorin water, Cl₂. Bromin water, Br₂. Phosphorus.

Apparatus. Test tubes. Tile or brick. Pincers.

Procedure. (a) Examine iodin and state its physical properties, comparing it with bromin and chlorin.

Place a few crystals of iodin in a test tube and heat gently, noting what happens. What condenses on the cool parts of the glass? Define *sublimation*.

Test the solubility of iodin in water, alcohol, carbon disulfid and chloroform, noting the relative degrees of solubility and the colors produced.

(b) Add a drop of iodin solution to some very dilute starch paste and note result. This is used as a *test for free iodin*. What other use have you made of this same reaction? To see whether iodin combined with other elements will produce the same color, add a drop of KI solution to starch paste. Result?

(c) 1. Shake 2 ccm. of chloroform with 10 ccm. of a water solution of iodin. Result? This procedure is used as a test for free iodin.

2. Shake similarly 2 ccm. of chloroform with 20 ccm. of water containing 5 drops of KI solution. The result is typical of all *compounds* of iodin. Does iodin combined with other elements give the iodin test?

* See footnote, p. 102. † Prepared as directed, p. 99.

3. Pour half of the KI solution from (2) into another test tube, add 2 ccm. of chloroform and 5 ccm. of chlorin water and shake thoroughly. Result? What must have set the iodin free?

4. Add to the remaining KI and chloroform mixture from (2) a few drops of bromin water and shake. Result? What must have set the iodin free?

Write equations showing the action of chlorin and bromin on potassium iodid. Write an equation showing the action of metals, such as zinc, on acids. Compare this equation with the two preceding equations and see if you can note any similarity in the reactions.

(d) Put a little iodin on a tile or brick, and with a glass rod or pincers press gently upon it a small piece of phosphorus, dried between filter papers. Result? How did chlorin act with phosphorus? Bromin acts similarly. Would you say that the halogen elements are very active chemically?

Outline a method for preparing iodin from KI based on the experiments you have made.

EXP. 57. SULFUR

Materials. Sulfur. Carbon disulfid, CS_2 . Strip of copper foil that will slip into a test tube. Powdered iron. Zine dust.

Apparatus. Test tubes. Test tube holder. Watch glass or small beaker. Magnifying glass. 15 cm. filter paper. Funnel. Beaker of water.

Procedure. (a) Physical properties.

1. Examine a piece of ordinary sulfur and note its properties. Shake 1 g. with 3-5 ccm. of carbon disulfid for 5-10 minutes, pour off the liquid into a small beaker or watch glass and allow to evaporate. (Keep carbon

disulfid away from flames !) Make a sketch of the crystals as seen under a lens. This is *rhombic* sulfur. 2. Fold a 15 cm. filter paper and place it in a small funnel. Fill a test tube about half full of sulfur and melt it at as low a temperature as possible by holding it over a flame and shaking constantly. Pour the melted sulfur into the filter paper in the funnel and watch it cool. As soon as a few crystals have formed, pour out upon a pan the sulfur which is still melted. Open the filter paper, examine and draw the crystals of *monoclinic* sulfur thus formed. Are they clear and transparent? Keep them until the next exercise and note any change.

3. Melt a half test tube full of sulfur as before, and heat until it boils, noting the changes of color that occur. From time to time during the heating, tilt the test tube and note the changes in viscosity indicated by the readiness with which it flows. Note the color of sulfur vapor and pour the boiling liquid in a thin stream into a beaker of water. Where the sulfur vapor comes in contact with the cold beaker and surface of the water, note and describe the "flowers of sulfur" which form. Examine the properties of the liquid sulfur suddenly cooled by the water, noting its color and hardness and whether it is elastic or brittle. Keep this *plastie* sulfur until the next exercise and see if there is any change in properties.

State in detail the changes in properties of sulfur accompanying a change in temperature.

(b) Chemical properties.

1. Burn a piece of sulfur and cautiously note the odor of the gas produced. State in chemical terms what you mean by the "odor of burning sulfur."

2. Heat 5 g. of sulfur to boiling in a test tube until sulfur vapor fills the tube. Warm a strip of copper foil

in the burner and thrust it far into the sulfur vapor, noting carefully what happens. After a minute or two, withdraw the strip and examine the properties of the copper sulfid that has formed.

3. Recall or repeat the experiments showing how iron and zinc combine with sulfur, Exps. 2(d) and 4(e).

Would you say that sulfur is chemically active at ordinary temperatures; at high temperatures? Write equations for the four reactions noted above. Compare the action of sulfur and of oxygen on these metals.

EXP. 58. HYDROGEN SULFID, H₂S

Materials. Iron sulfid, FeS. Conc. HCl. Hydrogen peroxid, H_2O_2 . Bromin water. Solutions of copper sulfate, $CuSO_4$; arsenic chlorid, $AsCl_3^*$; antimony chlorid, $SbCl_3$; stannous chlorid, $SnCl_2$; zinc sulfate, $ZnSO_4$.

Apparatus. A generator such as was used in preparing hydrogen, Fig. 13, but the gas must be collected by downward displacement and not over water. Glass rod or piece of porcelain. Test tubes. Strip of copper. Silver coin (dime).

CAUTION: Hydrogen sulfid is a poisonous gas, the first effects being dizziness and headache. Avoid breathing it. As an antidote inhale chlorin from a handkerchief on which chlorin water has been sprinkled; or add HCl to a solution of "chloride of lime" and inhale the fumes.

Procedure. (a) Preparation. Fit up the generator as shown in Fig. 13. Put 10-20 g. of iron sulfid in the bottle and through the thistle tube add about 25 ccm. of conc. HCl, diluted with an equal volume of water. Allow the gas to bubble into 100 ccm. of water in a beaker until the water smells strongly of hydrogen sulfid;

* Made by dissolving As₂O₃ in HCl.

then collect 2-3 bottles by downward displacement. Add conc. HCl through the thistle tube if necessary to hasten the action. Complete the equation:

$$\text{FeS} + -\text{HCl} \rightarrow \text{H}_2\text{S} + ---$$
.

(b) Physical properties.

Note the color, odor, and taste of hydrogen sulfid. Is it soluble in water? (Smell of the water into which the gas has been passing before answering this question.)

Test the action of hydrogen sulfid water on blue litmus paper and explain.

(c) Chemical properties.

1. Does hydrogen sulfid support combustion? Does it burn? Light the gas as it escapes from the generator and cautiously smell the fumes. What is one product of combustion of H_2S ? Hold a cold glass rod or piece of porcelain in the flame. What deposits on it? Where did it come from? Why do you get no deposit when the gas burns freely? Compare the formation of a deposit here with the deposit of soot in an ordinary flame.

2. Add a few drops of hydrogen peroxid to 10 ccm. of H_2S water. Explain what happens. Is H_2S an oxidizing or a reducing agent? Complete the equation:

$H_2O_2 + H_2S \rightarrow S + ---$

3. Add a few drops of bromin water to 10 ccm. of H_2S water and explain what happens. All the other halogen elements will act similarly. Complete the equation :

$Br_2 + H_2S \rightarrow S + ---$

4. Add to 10 ccm. of H_2S water a few drops of copper sulfate solution and describe what happens. In a similar way add arsenic chlorid, antimony chlorid, stannous

EXPERIMENTS

chlorid, and zinc sulfate, to separate portions of H_2S water. Describe the sulfids formed and complete the equations :

$$\begin{array}{c} \mathrm{CuSO}_4 + \mathrm{H}_2\mathrm{S} \rightarrow \mathrm{CuS} + ----\\ -- \mathrm{AsCl}_3 + -- \mathrm{H}_2\mathrm{S} \rightarrow \mathrm{As}_2\mathrm{S}_3 + ---\\ -- \mathrm{SbCl}_3 + -- \mathrm{H}_2\mathrm{S} \rightarrow \mathrm{Sb}_2\mathrm{S}_3 + ----\\ \mathrm{SnCl}_2 + \mathrm{H}_2\mathrm{S} \rightarrow \mathrm{SnS} + ----\\ \mathrm{ZnSO}_4 + \mathrm{H}_2\mathrm{S} \rightarrow \mathrm{ZnS} + ----\end{array}$$

5. Hold a strip of copper and a silver coin in H_2S water and note the effect. Complete the equations:

 $\begin{array}{c} \mathrm{Cu} + \mathrm{H_2S} \rightarrow \mathrm{CuS} + - - - \\ - \mathrm{Ag} + \mathrm{H_2S} \rightarrow \mathrm{Ag_2S} + - - - \end{array}$

No considerable action of this kind takes place except when an oxidizing agent (such as air) is present to combine with the nascent hydrogen as it is liberated. This action is mainly responsible for the tarnishing of silver.

The odor of hydrogen sulfid and the blackening of silver caused by its solutions are *tests for hydrogen sulfid*.

EXP. 59. SULFUR DIOXID, SO₂, AND SULFUROUS ACID, H₂SO₃

Materials. Sulfur. Wooden splint. Flowers, or piece of calico. Solutions of potassium permanganate, KMnO₄; sodium sulfite, Na₂SO₃; barium chlorid, BaCl₂. Litmus paper. HCl. NaOH. Charcoal or copper. Conc. H₂SO₄.

Apparatus. Combustion spoon. Wide mouth bottle. Oxygen generator as shown in Fig. 9 without the pneumatic trough.

Procedure.

(a) Preparation by oxidation of sulfur.

1. Place a piece of sulfur in a combustion spoon, ignite it and lower it into a wide mouth bottle. Cover the bottle and allow the sulfur to burn until the flame is

extinguished. Note the color, odor, and taste of sulfur dioxid. Will it burn or support ordinary combustion?

Prepare another bottle of sulfur dioxid in a similar way. Moisten a flower or piece of calico, and allow it to stand in the sulfur dioxid for some time. What seems to be the action of sulfur dioxid on coloring matter?

2. Pour 10-20 ccm. of water into a bottle and shake it about so that the bottle is wet all over. Burn sulfur above the water in the bottle as before, and shake so that the gas produced may be absorbed. Pour the water from the bottle and smell of it. Is sulfur dioxid soluble in water? Test the water with blue litmus paper, noting the first and also the latter effects. Is sulfur dioxid an acid anhydrid? Compare the following equation with the action of carbon dioxid on water.

$SO_2 + H_2O \rightarrow H_2SO_3$, sulfurous acid.

3. Add NaOH solution, drop by drop, to the sulfurous acid solution until it turns litmus paper blue. Does the solution smell of sulfur dioxid any longer? Complete the equation and name the product formed.

 $H_2SO_3 + - NaOH \rightarrow Na_2SO_3 + ----$

(b) Preparation by reduction of sulfuric acid, H₂SO₄.

1. Fit up a test tube with an outlet and delivery tube as shown in Fig. 9. Fill the test tube one eighth full of pieces of charcoal or copper, and add 10 ccm. of conc. H_2SO_4 . Suspend the apparatus from a ring stand and heat with a small flame as the gas is evolved. If white fumes appear above the liquid, moderate the heat immediately.

2. Repeat the experiments of (a) sufficiently to assure yourself that the same gas is produced in (b) as in (a).

3. Allow the gas to bubble into about 10 ccm. of water

in a test tube until it smells strongly of sulfur dioxid. Use this solution for (e). Complete the following equations:

 $\begin{array}{c} \mathrm{H}_{2}\mathrm{SO}_{4} + \mathrm{C} \rightarrow \mathrm{CO} + \underbrace{\longrightarrow} + \underbrace{\longrightarrow} + \underbrace{\longrightarrow} + \mathrm{CuO} + \underbrace{\longrightarrow} +$

(c) The odor of sulfur dioxid and the fact that it will bleach potassium permanganate are used as *tests for sulfur dioxid and sulfurous acid*.

1. Test part of the solution obtained in (b) by adding a few drops of potassium permanganate solution. What happens? Do you get a test for SO₂ and H₂SO₃?

2. To a solution of sodium sulfite, add HCl and note the odor; then add KMnO_4 as in (1). Do you get the same test? Why is it necessary to add HCl in (2) but not in (1)?

3. Add barium chlorid and HCl to (1) and (2). The formation of a white precipitate is a *test for sulfates*. Did you get the test?

Permanganates are substances very rich in oxygen which is liberated readily and oxidizes sulfurous acid. Complete the equation:

$H_2SO_3 + O \rightarrow ---$

EXP. 60. SULFUR TRIOXID, SO₃, AND SULFURIC ACID, H_2SO_4

Instructor's Experiment

Materials. Scraps of platinum. Conc. HNO_3 and HCl. Asbestos. Conc. solution of sulfur dioxid. Barium chlorid (BaCl₂) solution.

Apparatus. A 2-liter bottle fitted with inlet and exit tube as used in Exp. 47, connected with a wash bottle which is connected with a glass tube about 30 cm. long filled with asbestos containing finely divided platinum. Test tube. Porcelain crucible. Pipestem triangle,

Procedure. Dissolve 0.5–1 g. of old platinum foil or wire in aqua regia, evaporate to 2–3 ccm., then add about 10 ccm. of water. Shred some asbestos board, soak the shreds in the platinum solution just prepared, and heat to redness in a porcelain crucible or dish. By this treatment the platinum chlorid is decomposed, leaving finely divided platinum deposited throughout the asbestos.

Set up the apparatus as directed above, connecting the inlet tube of the large bottle to the water tap, and the outlet tube to the wash bottle which contains a strong solution of sulfur dioxid in water. Loosely fill the center third of the glass tube with the platinized asbestos, support it on a ring stand, and connect it to the exit tube of the wash bottle. Arrange the apparatus so that when water enters the large bottle, air will be forced through the solution in the wash bottle and, mixed with sulfur dioxid, will pass over the platinum. Regulate the stream of water so that about one bubble a second passes through the wash bottle. Note that no interaction between the sulfur dioxid and oxygen takes place at ordinary temperature. When the tube is moderately heated, sulfur trioxid is formed. State what you observe. Rinse out a test tube with water and slip it over the end of the tube containing the platinum so that the fumes may come in contact with the moisture on the sides of the tube. After a few moments rinse out the test tube with water and test the solution for sulfuric acid.

EXP. 61. PHOSPHORUS

Materials. Yellow and red phosphorus. Phosphorus pentoxid, P_2O_5 . HCl. Conc. HNO₃. Solution of ammonium molybdate, $(NH_4)_2MoO_4$. Commercial fertilizers. Ferrous sulfate, FeSO₄. Conc. H₂SO₄. NaOH.

Apparatus. Tile or brick. Large beaker. Blue lit mus paper. Glass rod. Cobalt glass. Platinum wire.

Procedure. (a) Note the physical difference between yellow (observe the cautions given on p. 15) and red phosphorus. Place a little of each upon a tile or brick and touch with a warm glass rod. What happens? Heat the glass rod and try to ignite the red phosphorus. If you do not succeed, ignite it with a burner. Compare the way red and yellow phosphorus take fire and note the product formed in each case.

(b) Dry a piece of phosphorus about the size of a pea, and place it upon a tile or brick. Ignite it, cover with a large beaker and allow to stand until the white "smoke" has settled. What is the "smoke"? Describe it.

Press a moistened piece of blue litmus paper upon some of the deposit. Explain what happens. Allow the deposit to stand in the air for about an hour and note what happens; test it now with a piece of dry blue litmus paper and explain why phosphoric pentoxid is one of the best drying agents. Complete the equation:

 $\mathrm{P_2O_5} + - \mathrm{H_2O} \mathop{\longrightarrow} - \mathrm{H_3PO_4}$

(c) Place about 1 g. of phosphorus pentoxid in a test tube and add a few drops of water. Is the combination with the water vigorous or mild? Add 10 ccm. of water and boil for 5 minutes. Add one drop of this phosphoric acid solution to 5 ccm. of ammonium molybdate solution to which a few drops of conc. HNO_3 have been added. The formation of a yellow precipitate on standing 5–10 minutes is a test for phosphates.

(d) To 1 g. of commercial fertilizer add 10 ccm. of water and a few drops of HCl, heat to boiling and filter. Test the solution for salts of phosphoric and nitric (p. 72) acids and of ammonium (p. 78) and potassium

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(p. 126). Does this fertilizer contain all the foods neces sary for a plant?

EXP. 62. PHOSPHIN, PH₃

Instructor's Experiment

Materials. Solid NaOH or KOH. Yellow phosphorus. Apparatus. As shown in Fig. 35.

Procedure. Put about 50 g. of solid NaOH or KOH, 200 ccm. of water and 2-3 pieces of phosphorus about the size of a pea in a quarter or half liter flask and set up the



FIG. 35.

apparatus as shown in Fig. 35. Connect the inlet tube of the flask with the gas pipe and allow gas to flow at full head into the flask for a minute until the air is replaced; then turn down the gas so that about 1 bubble in 5 seconds issues from the exit tube. Heat up the solution in the

flask until it nearly reaches the boiling point, and maintain this temperature by a small flame. Phosphin and other hydrids of phosphorus will be generated and escape through the water into the air. Describe what happens. What can you say about the kindling temperature of some of the gases formed? What difference do you notice between PH_3 and NH_3 ? What is the "smoke" formed by burning PH_3 ? Why is it necessary to remove the air from the flask? What would happen if air got into the flask?

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Before taking the apparatus apart, allow the liquid to cool and sweep out all phosphids by turning on the illuminating gas full head for a moment. Pour out the solution, wash any remaining phosphorus with water and replace it in the phosphorus bottle.

EXP. 63. ARSIN, AsH₃, AND STIBIN, SbH₃ Instructor's Experiment

Materials. Zinc. HCl. Solutions of any arsenic compound, of any antimony compound, and of "chloride of lime."

Apparatus. 2 hydrogen generators. 2 clay pipe stem jets for burning the gas. 3 porcelain plates or evaporating dishes.

Procedure. Start both generators and ascertain that all air has been driven from each apparatus by testing the purity of the gas as directed on page 36. When the gas collected burns quietly, replace the delivery tube with the pipe stem and ignite the hydrogen as it issues. When the hydrogen burns quietly, introduce into one generator through the thistle tube a few drops of any arsenic compound and into the other the same amount of any antimony compound. Describe the changes in the flames.

Any soluble compound of arsenic or antimony is reduced by the nascent hydrogen to arsin and stibin. Write equations for this change, assuming that you started with $AsCl_a$ and $SbCl_a$.

Move a porcelain plate slowly about in the flame in which arsin is burning and describe the effect. Get a similar deposit from the stibin flame on a second plate. This procedure is known as the Marsh *test for arsenic and antimony*. On a third plate trace the symbol Sb by means of the stibin flame and then obliterate it by means of the deposit from the arsin flame.

Treat all three plates with "chloride of lime" solution and state the results.

What differences do you notice between AsH_3 and SbH_3 on the one hand and NH_3 on the other? Write equations showing what is formed when arsin and stibin burn. As_2O_3 and Sb_2O_3 are white solids; do you get any evidence of them? When the flame is cooled, elementary arsenic or antimony is deposited. Compare with the deposit of sulfur from burning H_2S and of carbon from H_4C and other hydrocarbons.

The deposit of arsenic and antimony cannot be distinguished with certainty except by chemical means. Arsenic is readily dissolved in hypochlorites, being oxidized to arsenic pentoxid, which unites with water to form arsenic acid, H_8AsO_4 . Antimony is not easily oxidized by this reagent, hence remains on the plate. Complete the equations and compare with phosphoric acid (p. 113).

 $\begin{array}{c} -\operatorname{As}+-\operatorname{O} \twoheadrightarrow ---\\ \operatorname{As}_2\operatorname{O}_5+-\operatorname{H}_2\operatorname{O} \twoheadrightarrow -\operatorname{H}_3\operatorname{As}\operatorname{O}_4 \end{array}$

EXP. 64. ELEMENTS THAT ACT BOTH AS ACID-FORMERS AND BASE-FORMERS

Materials. Solutions of lead nitrate, $Pb(NO_3)_2$; sodium carbonate, Na_2CO_3 ; sodium sulfate, Na_2SO_4 ; potassium chromate, K_2CrO_4 ; stannous chloride, $SnCl_2$; zinc sulfate, $ZnSO_4$; aluminum sulfate, $Al_2(SO_4)_3$; antimony chlorid, $SbCl_3$. Arsenic oxid, As_2O_3 . Bits of tin, zinc, and aluminum. NaOH. KOH. NH₄OH. HCl.

Procedure. (a) Lead as a base-former.

Dilute 10 ccm. of lead nitrate solution to 50 ccm. with water. To 5 ccm. portions of this solution add a few drops of the reagents specified : (1) NaOH; (2) Na₂CO₃; (3) Na₂SO₄; (4) K₂CrO₄. Note the result in each case.

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Write equations and name each lead compound formed. What uses are made of any of these compounds? Acting as a base-former, lead unites with what kind of radicals?

(b) Lead as an acid-former.

1. To 5 ccm. of your lead nitrate solution add NaOH until the precipitate, first formed, redissolves. Write equations showing what lead compound you now have in solution. Name it. Test this solution with a drop of each of the reagents used in (a). Do you get any of these indications of the presence of lead? Why not? Lead as an acid-former unites with what kind of elements?

2. Use KOH as in (1). Does it act similarly?

3. Use NH_4OH as in (1). Does it act similarly?

What makes lead act as an acid-former? (NH_4OH is not sufficiently strong as a base to cause any element to act as an acid-former.)

(c) Tin, zinc, aluminum, antimony, and arsenic as well as lead readily act both as base-formers and as acid-formers.

1. Precipitate the hydroxid of one of the elements mentioned above and note that it will dissolve either in HCl or in NaOH. Some students choose one, others another element. (For arsenic hydroxid use the solid As_2O_3 .)

2. Heat a bit of tin, zinc, or aluminum with NaOH and note that it will dissolve in this as it will in HCl, hydrogen being liberated in both instances. Write equations expressing the reaction.

EXP. 65. REDUCTION OF METALS FROM THEIR ORES

Materials. Piece of wood charcoal or thick bark. Some of the following oxids; As_2O_3 , Sb_2O_3 , Bi_2O_3 , CuO, PbO, SnO_2 , ZnO. Anhydrous sodium carbonate, Na_2CO_3 .

Apparatus. Blowpipe (made in Exp. 49 (c)).

Procedure. Scrape a shallow depression in the surface of the charcoal or bark. Put in it about 1 g. of lead oxid and direct upon it the reducing flame of the blowpipe. If the material blows away, place a little Na_2CO_3 on it and moisten with a drop or two of water. Sodium carbonate takes no part in the reaction; it melts and holds the material together and protects it somewhat from the air. After heating for a short time, allow to cool. What change has taken place in the lead oxid? How was this change brought about? Did the charcoal have any influence in the reduction?

There is a coating formed on the charcoal about the depression. Note its color when hot and when cold. Does it resemble lead oxid? Its formation is due to the fact that a little metal is volatilized, oxidized by the air, and deposited on the charcoal.

Repeat the above with 2-3 of the other oxids, different students using different oxids. Tabulate as follows:

Oxid	COATING		METAL	D. D. on Mamir	Proving
	Hot	Cold	READILY OR NOT	D. F. OF MEIAL	TEMARK
As ₂ O ₃	-	Rie 1		5	1. 112
Sb ₂ O ₃					
$\mathrm{Bi}_2\mathrm{O}_3$			1		
CuO					
PbO					
SnO_2	1 1 1 1				
ZnO	1				

The oxids of copper and tin are difficult to reduce in this way, requiring long, steady heating and probably mixing with powdered charcoal. After cooling, turn the material over with a knife and look for these metals on the bottom. Did you obtain globules of metallic arsenic and zinc? Did you obtain heavy coatings in these cases? Note the boiling points of these metals (see text, p. 407) and see if you can explain these results.

Blowpipe tests of this character are often used to determine what metal is present in a compound, for when heated with sodium carbonate on charcoal, most compounds of the elements listed above will be reduced to the metals.

EXP. 66. BORIC ACID, H₃BO₃, AND BORAX BEAD TESTS

Materials. Borax, $Na_2B_4O_7$. Conc. H_2SO_4 . HCl. NaOH. Alcohol. Asbestos shreds. Turmeric paper. Some solid compound of cobalt, manganese, chromium, and iron, such as $Co(NO_3)_2$, MnO_2 , $Cr_2(SO_4)_3$, and Fe_2O_3 or iron rust.

Apparatus. Beaker and test tubes. Pincers. Platinum wire about 5 cm. long. Short glass tube. Blowpipe.

Procedure. (a) Dissolve by heating to boiling 10 g. of borax in 25 ccm. of water. Add 3 ccm. of conc. H_2SO_4 to an equal volume of water and mix thoroughly with the hot borax solution. Note what happens on cooling and describe the boric acid which crystallizes. Pour the mixture through a filter and wash the crystals. Touch to the tongue. Is boric acid a strong acid? Complete the equation:

$Na_2B_4C_7 + H_2SO_4 + - H_2O \rightarrow 4H_3BO_9 + - - -$

(b) 1. Place some of the boric acid in an evaporating dish and add 3-5 ccm. of alcohol. Set fire to the alcohol and note the color of the flame. If the color is not distinct, soak a shred of asbestos in the mixture and heat in the hottest part of a Bunsen flame. This green flame is used as a test for boric acid.

2. Dissolve a crystal of borax in a little water and set free the boric acid by adding a little HCl. Dip a piece of turmeric paper in the solution and dry it by placing it on the outside of a beaker or test tube of boiling water, where it will be heated to 100° but not much higher. The red color which appears on drying is a *test for borates*. (With dilute solutions it may be necessary to wet the turmeric paper and dry it repeatedly to get a good test.) Moisten the red paper with an alkali and note the change of color. Will HCl restore the color?

(c) Rotate one end of a piece of glass tubing in a Bunsen flame and just as the glass is running together insert a platinum wire about 5 cm. long heated red hot. The glass tube will serve as a handle and prevent loss of the wire.

Make a loop in the end of the platinum wire about 2–3 mm. in diameter by wrapping the wire once around the point of a lead pencil. Heat the loop and while hot press it against a small crystal of borax, thus causing it to adhere. Heat the borax in the hottest part of the Bunsen flame or better in the flame of a blowpipe, noting how it swells up as the water in the crystals boils away. Note also the clear bead finally produced.

While this bead is hot, press it against a mere trace of a cobalt compound and heat again. Note the color produced.

To remove the bead, heat it red hot and shake it out of the loop or plunge it into water, thus causing it to disintegrate. Make a new bead and see that it is colorless. If not, shake out again and start anew.

Make similar beads containing manganese, chromium, and iron compounds. Heat all the beads in the oxidizing flame and record the colors produced.

EXP. 67. ALUM FROM CLAY

Materials. Clay. Conc. H_2SO_4 . Potassium carbonate, K_2CO_3 . NH_4OH . Solutions of cobalt nitrate, $Co(NO_3)_2$, and alum, $KAl(SO_4)_2$.

Apparatus. Evaporating dish. Beakers and funnel. Plaster of Paris block * or piece of charcoal or bark.

Procedure. (a) Mix thoroughly 50 g. of dry, finely pulverized clay soil with 30 ccm. of conc. H₂SO₄ in an evaporating dish, and heat for 20 minutes (Hood) over a low flame to such a temperature that white fumes just begin to appear. When cool, pour the mixture into a beaker, add 100 ccm. of boiling water, stir thoroughly, allow to settle, and while still hot decant the liquid as completely as possible into a second beaker. Treat the residue in the same way with 50 ccm. and then with 30 ccm. of boiling water. Heat the combined water extracts to boiling, and add slowly with constant stirring, 12 g. of solid potassium carbonate. After all action has ceased, filter the boiling liquid. Evaporate the filtrate to half its original volume and set aside to cool until the next exercise. Remove the crystals obtained and dry them between filter papers.

More crystals may be obtained by further concentration of the remaining liquid. They will not be as pure and may need recrystallization to get them colorless.

The composition of clay may be taken as $HAlSiO_4$, the reaction on heating with H_2SO_4 being :

 $2 \operatorname{HAlSiO}_4 + 3 \operatorname{H}_2 \operatorname{SO}_4 \rightarrow \operatorname{Al}_2 (\operatorname{SO}_4)_3 + - \operatorname{SiO}_2 + - \operatorname{H}_2 O$

In the preceding equation fill in the numbers necessary to make it balance. The effervescence produced on add-

* Mix plaster of Paris with one third its weight of water and pour it upon a sheet of paper laid upon the desk. When it has partially hardened, cut it into pieces about 10×2 cm. and allow to dry. ing K_2CO_3 is due to the excess of H_2SO_4 not used in decomposing the clay. Write the equation for the formation of alum.

(b) Dilute 5 ccm. of alum solution with 10 ccm. of water and add NH_4OH . Notice the character of the aluminum hydroxid precipitated. Heat the mixture to boiling and filter. Heat some of the precipitate with the blowpipe upon charcoal or plaster of Paris and note the character of the aluminum oxid formed. Moisten the aluminum oxid with a drop of cobalt nitrate and heat strongly again. A blue color is a *test for aluminum*. (Zinc gives a green color when similarly treated.)

(c) Shake some pulverized clay with water and allow the heavier material to settle out for ten minutes. With 100 ccm. of the turbid water mix 5 ccm. of alum solution. Add a piece of litmus paper and make just alkaline with ammonium hydroxid. Note the time it takes this solution to settle clear as compared with some of the water that has not been so treated. This illustrates a method for making turbid water clear, except that the cheaper calcium hydroxid is used instead of ammonium hydroxid. What causes the clay to settle?

EXP. 68. DYES AND MORDANTS

Materials. Pieces of white cotton cloth about 2 by 10 cm. Conc. HCl. $\rm NH_4OH$. Solution of Congo Red containing 1 g. of dye, 1 g. of $\rm Na_2CO_3$, and 2 g. of $\rm Na_2SO_4$ in each 100 ccm. of water. Solution of alizarin containing 1 g. of dye in each 100 ccm. of water and enough $\rm NH_4OH$ to make it go into solution. Solution of aluminum sulfate, $\rm Al_2(SO_4)_3$. Solution of cochineal * — boil 10 g. in 100 ccm. of water. Extract of logwood made by covering logwood with water and bringing it to boiling. Do this four times. Reject first three; keep fourth.

* Cochineal contains carminic acid.

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Apparatus. Evaporating dish, test tubes, and beakers.

Procedure. (a) To remove the "sizing" from the cloth.

Add 5 ccm. of conc. HCl to 100 ccm. of water in a beaker, place 3-5 pieces of cloth in this solution, and heat to boiling; then wash the cloth thoroughly in running water \cdot and finally dip in water containing a few drops of NH₄OH.

(b) 1. Dilute 5 ccm. of the Congo Red solution to 50 ccm. with water in an evaporating dish. Keep the solution boiling gently and immerse a piece of cloth in it for 3-5 minutes, stirring constantly. Remove the cloth, wash thoroughly, dry and put in your notebook. Congo Red dyes cotton without a mordant.

2. Dilute 5 ccm. of the alizarin solution to 50 ccm. with water. Boil a piece of cloth in it; remove, wash, and dry as before. Does alizarin dye cotton without a mordant?

(c) 1. Add a few drops of NH_4OH to 5 ccm. of a solution of aluminum sulfate. What is precipitated?

2. Pour 5 ccm. of your alizarin solution into a test tube, dilute with 10 ccm. of water, heat to boiling and add 5 ccm. of aluminum sulfate solution. If a precipitate does not form immediately because of the NH_4OH in the alizarin solution, add a few drops of NH_4OH . Allow to stand until the precipitate settles. Where is the color, in the liquid or in the precipitate? An insoluble compound formed by the union of a base (or an acid) with a dye is called a *lake*.

3. Soak a piece of cloth in the aluminum sulfate solution, squeeze it dry and attempt to dye it in your alizarin solution as before. Is the result any better? Where is the lake in this case? Aluminum hydroxid acts as a mordant for alizarin; also for other dyes including logwood and carminic acid (cochineal). If you have time, try one of these also.

EXP. 69. HARD WATER AND HOW TO SOFTEN IT

Materials. Soap cut into pieces about the size of a pea. Solutions of calcium sulfate, $CaSO_4$; sodium carbonate, Na_2CO_3 ; borax, $Na_2B_4O_7$; and sodium phosphate to which 1 g. of NaOH is added for each 5 g. of Na_2HPO_4 , thus forming Na_3PO_4 . Lime water, $Ca(OH)_2$. HCl. NaOH. NH_4OH . Litmus paper. Several different kinds of washing powders or "boiler compounds." Marble, $CaCO_3$. Ammonium molybdate, $(NH_4)_2MOO_4$. Turmeric paper.

Apparatus. Carbon dioxid generator (p. 85).

Procedure. (a) Hard waters.

Dilute 40 ccm. of lime water with 60 ccm. of water, pass in carbon dioxid until the precipitate first formed is redissolved. Write equations expressing the change taking place. What substance is in solution? Call this No. I.

Add 20 ccm. of calcium sulfate solution to 80 ccm. of water. Call this No. II.

1. Take half a test tube full of distilled water, add a piece of soap, cover with the thumb, and shake, noting the readiness with which a lather is formed.

2. Treat half a test tube of No. I in the same way. Does it lather readily?

3. Treat half a test tube of No. II just as in (2). Does it lather readily?

Solutions I and II are typical hard waters. Magnesium salts act similarly to calcium salts and both are commonly present in water.

4. Remove some of the precipitate ("scum") appearing in (2) and (3) to a watch glass and treat with 2-3 ccm. of water and 5 drops of HCl. The precipitate dissolves, but a new one (stearic acid) forms immediately. Scrape this on to the edge of the watch glass, drain off the liquid, wash with a few drops of water and add 5 drops of NaOH. Does the precipitate dissolve? Stir it and see it lather. What is in the solution? What is the "scum"?

5. Boil half a test tube of No. I. Do you note any change? Shake with soap as before. Is there any difference now? Explain why. Water that is softened by boiling is called "temporarily hard water."

6. Treat half a test tube of No. II as in (5), answering the same questions. What is the difference between the two hard waters? Water not softened by boiling is called "permanently hard water."

(b) Chemical methods of softening hard water.

1. Add NH_4OH to half a test tube of I until red litmus turns blue. Shake with soap as before. Does NH_4OH soften the water? In softening temporarily hard water, any base will act similarly. Usually the calculated amount of lime, the cheapest base, is added to it. Complete the equation :

$CaH_2(CO_3)_2 + Ca(OH)_2 \rightarrow ---$

2. Add 2-3 ccm. of sodium carbonate solution to half a test tube of either I or II. Do you note any change? Shake with soap. Is the water soft? Write equation.

3. Add 2-3 ccm. of borax solution as in (2). Result?

4. Add 2-3 ccm. of sodium phosphate solution as in (2) Result?

(c) Analysis of washing powders.

Make a solution of the powder, or "boiler compound," and test for carbonates (p. 86), borates (p. 120), phosphates (p. 113), and ammonium salts (p. 78). Test also for soap by acidifying with HCl and see if a precipitate of stearic acid, etc. forms (p. 97). What does the powder contain? Let different students analyze different powders and thus find out what is on the local markets.

EXP. 70. FLAME TESTS - COLORED FIRE

Materials. Solutions of the chlorids of calcium $(CaCl_2)$, strontium $(SrCl_2)$, barium $(BaCl_2)$, potassium (KCl), and sodium (NaCl). Sugar. Strontium chlorate, $Sr(ClO_3)_2$. Barium chlorate, $Ba(ClO_3)_2$. HCl.

Apparatus. Mortar and pestle. Tile or brick. Platinum wire. Test tubes. Blue cobalt glass.

Procedure. (a) Clean your platinum wire by alternately dipping in HCl and heating until it gives no color to the flame. Then dip the wire in a solution of calcium chlorid, and note the color given to the flame.

Clean your wire between each test. Note and record the color produced by chlorids of strontium, barium, potassium, and sodium.

Dip your wire into a solution of sodium chlorid and then of potassium chlorid, and note which of the two you can detect in the flame. Dip the wire into both solutions again, and look at the flame through a piece of blue cobalt glass. Which color do you see now? What use could you make of the blue cobalt glass?

Compounds of the elements mentioned above, when moistened with HCl, generally give their characteristic flame tests.

(b) In separate mortars * pulverize 5 g. of cane sugar and an equal volume of strontium chlorate. Mix thoroughly on a filter paper (Do not grind together), place on a tile or brick, and ignite with a burner. Why does the combustion take place so vigorously? What compound gives color to the flame?

Mix similarly 5 g. of sugar with an equal volume of barium chlorate and ignite.

* Three students should work together on (b), one powdering the sugar, one the strontium chlorate, and one the barium chlorate.

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EXP. 71. MANUFACTURE OF POTASSIUM NITRATE

Materials. Sodium nitrate, NaNO₃. Potassium chlorid, KCl. Potassium nitrate, KNO₃.

Apparatus. Beakers and test tubes. Cobalt glass. Platinum wire.

Procedure. Dissolve by heating 28 g. of $NaNO_3$ in 20 ccm. of water. Put 25 g. of KCl in 30 ccm. of water and heat to boiling; then add the $NaNO_3$ solution. Continue to boil the mixture until its volume is reduced nearly one third, remove from the gauze and allow to settle; then *immediately* decant the clear liquid from the crystals as completely as possible into another beaker and allow it to crystallize also.

Wash the crystals in the first beaker with 5 ccm. of boiling water and decant the washings. Taste the substance and apply the flame test. What is it? Write an equation indicating how it was formed.

Cool the second beaker, if possible in ice water, decant the liquid from the crystals, dissolve in the least possible amount of boiling water, and recrystallize. Note the form of the crystals, taste them and apply the flame test using the blue glass. Potassium nitrate crystallizes in needles. Taste it. What is the second substance obtained in this experiment?

Consult the text (p. 64) and ascertain which of the four substances present in the solution is least soluble at 100° and which at 0°. Which is most likely to separate from the boiling solution therefore? What effect on the reaction would this have? Which substance is most likely to separate from the cold solution? State two reasons why. Can you suggest any changes in this process that would make it more efficient on a commercial seale?

EXP. 72. WASHING SODA, BAKING SODA, AND BAKING POWDER

Materials. Washing soda, Na_2CO_3 . Baking soda, HNaCO₃. Several different brands of baking powder. Some cleaning compounds. HCl. NaOH. NH₄QH. Lime water, Ca(OH)₂. Solutions of iodin; barium chlorid, BaCl₂; ammonium molybdate, (NH₄)₂MoO₄; cobalt nitrate, Co(NO₃)₂. Marble, CaCO₃.

Apparatus. Carbon dioxid generator (see p. 85). Test tubes. Beakers. Evaporating dish.

Procedure. (a) Washing soda and baking soda.

1. Touch some baking soda and then some washing soda to the tongue and try to distinguish them by taste.

2. Moisten the hands with a solution of washing soda and then wash them in water. Is washing soda a good cleaning agent? It is the principal ingredient in most washing and cleaning compounds. Add HCl to some of them and see if they do not effervesce.

3. Pass carbon dioxid from a generator into 2 g. of washing soda in 10 ccm. of water. Taste the solution resulting after the liquid is apparently saturated with gas. Is the product baking soda? How may washing soda be converted into baking soda?

4. Put 5 g. of baking soda into a test tube and heat gently. What do you observe? Put 5 ccm. of lime water into a small beaker and tilt the test tube so that if a heavy gas is liberated by the baking soda it may pour into the beaker. Shake the lime water frequently. What inference do you draw from the result? What condenses on the cold parts of the test tube? Taste the product left in the test tube. Is it washing soda? Write an equation expressing the change of baking soda into washing soda.
5. To get an idea at what temperature baking soda is converted into washing soda, put 5 g. of baking soda into 10. ccm. of water and heat gently. Is gas liberated before the water boils? What is the purpose of baking soda in making bread or cake? What product is left in the bread? See if you can't taste it in "soda biscuits" or cake made with soda.

(b) Baking powder.

1. Examine some baking powder and see if you notice any action taking place. Pour a little water upon it. What happens? Test the gas with lime water. What is it? What causes the dough to begin to rise as soon as it is mixed? What causes it to rise in the oven?

Calculate how much (a) baking soda and (b) washing soda will be necessary to liberate 100 ccm. (standard conditions) of carbon dioxid, when treated with HCl. Write equations showing the products formed in each of these cases. If mixed in the proper proportions, could these mixtures be used to raise bread? Why is baking soda. always used as a leavening agent instead of washing soda?

(c) Analysis of baking powders.

All baking powders contain baking soda, but many different substances are used to unite with it and liberate the gas, such as an acid salt of tartaric, phosphoric, or sulfuric acid. Ammonium carbonate and ammonium alum, $\rm NH_4Al(SO_4)_2$, are sometimes present, and starch is frequently added to prevent the ingredients from acting on each other.

1. Pour 25 ccm. of water and 10 drops of cone. HCl upon 10 g. of baking powder, shake thoroughly, then settle and decant the clear liquid through a filter.

2. Boil the residue with a little water, cool and add a drop of iodin solution. Do you get a test for starch?

K

3. Evaporate 5 ccm. of the filtered liquid to dryness in an evaporating dish. If a tartrate is present, it will turn black and smell like burned sugar.

Test a few drops of the filtered liquid (4) for sulfates (p. 111), and (5) for phosphates (p. 113).

6. Test 5 ccm. for ammonium salts (p. 78).

7. Make 5 ccm. of the filtered solution alkaline with NH_4OH . Is gelatinous aluminum hydroxid precipitated? If you are in any doubt, test for aluminum in the precipitate by the cobalt nitrate test (p. 122). What substances did you find in the baking powders?

EXP. 73. THE REPLACEMENT OF ONE METAL BY ANOTHER

Materials. Solutions of zinc sulfate, $ZnSO_4$; copper sulfate, $CuSO_4$; ferric chlorid, $FeCl_3$; mercuric chlorid, $HgCl_2$; silver nitrate, $AgNO_3$. Zinc strips. Copper wire or strips. Iron nails. Mercury. Lead acetate, $Pb(C_2H_3O_2)_2$. Stannous chlorid, $SnCl_2$. Acetic acid.

Apparatus. 10 test tubes. Test tube rack or beakers to hold the test tubes. 2 large beakers.

Procedure. (a) Using separate test tubes put the following metals into 5 ccm. of a solution of the salts indicated. In each case record whether a deposit appears on the metal and what it seems to be, rubbing the metal with the finger in 2 and 5 to get evidence on this point. In 5 and 6 what does the blue color of the resulting solution indicate? Write equations expressing all reactions.

- 1. Zn in CuSO4.
- 2. Zn in HgCl₂.
- 3. Fe in CuSO₄.
- 4. Fe in ZnSO4.
- 5. Cu in HgCl₂.

- 6. Cu in AgNO₃.
- 7. Cu in FeCl₃.
- 8. Hg in AgNO₃.
- 9. Hg in ZnSO4.
- 10. Hg in CuSO4.

(b) For the instructor.

1. Dissolve 50 g. of lead acetate in 250 ccm. of water, add 1-2 ccm. of acetic acid and by a string suspend in the solution a bunch of zinc turnings or strips. Allow to stand overnight and note the replacement of the lead by the zinc.

2. Add 25 ccm. of conc. HCl to 500 ccm. of water and dissolve in it 30 g. of stannous chlorid. In a similar way allow zinc to hang in the solution overnight. Note the replacement of tin by zinc.

From the experiments you have seen, arrange the metals in the order in which they stand in the Potential or Replacement Series, the highest coming first.

EXP. 74. REACTIONS OF SILVER SALTS IN PHO-TOGRAPHY

Materials. Solutions of silver nitrate, $AgNO_8$ (2 g. in each 100 ccm.); of potassium bromid, KBr (4 g. in each 100 ccm.); of "hypo," sodium thiosulfate, $Na_2S_2O_8$ (125 g. in 500 ccm.). Developer made as follows: 2 g. hydrochinon, 8 g. sodium sulfite crystals, 8 g. sodium carbonate crystals, 5 ccm. of oxalic acid solution in each 250-300 ccm. of water.

Apparatus. Test tubes. Cork. Black paper.

Procedure. Clean 4 test tubes thoroughly. Wrap black paper about one and fit it with a clean cork. Put exactly 5 ccm. of $AgNO_3$ solution and 10 ccm. of water into this test tube. Measure out exactly 5 ccm. of potassium bromid into another. Carry all of the test tubes into a room darkened as much as possible. In the dark add the KBr to the $AgNO_3$, cork the tube and invert once or twice but do not shake it. Pour one third of the mixture into each of the two clean test tubes. Keep one third

in the test tube wrapped in black paper in the dark room. What is the precipitated material in the test tubes? Write an equation for its formation.

1. Add 10 ccm. of hypo to one portion of silver bromid. What happens? What is the use of hypo in photography?

2. Add 5 ccm. of developer to the silver bromid in the dark room. After 2-3 minutes add 10 ccm. of hypo, shake, bring to the light, and remove black paper. There should be no difference between the results in (1) and (2) if the latter has not been exposed to the light. A *developer* is a reducing agent not strong enough to reduce silver salts unless they have been exposed to light.

3. Expose the third portion of silver bromid 2-3 minutes to direct sunlight, shaking the tube to expose all portions equally. Add 5 ccm. of developer. Explain what happens. After 2-3 minutes add 10 ccm. of hypo and shake. What is the difference between the results in (2) and (3)? What is the precipitate in (3)?

EXP. 75. EFFECT OF OXIDATION ON THE PROPERTIES OF ELEMENTS

Materials. Solutions of chromium sulfate, $Cr_2(SO_4)_3$; sodium chromate, Na_2CrO_4 ; potassium permanganate, $KMnO_4$; manganese sulfate, $MnSO_4$; barium chlorid, $BaCl_2$; lead nitrate, $Pb(NO_3)_2$; oxalic acid, $H_2C_2O_4$. Alcohol, C_2H_5OH . Sodium peroxid, Na_2O_2 . Lead dioxid, PbO_2 . NaOH. HCl. HNO₃. H_2SO_4 . Acetic acid, $H(C_2H_3O_2)$.

Procedure. (a) Chromium compounds.

1. Add NaOH to 10 ccm. of $Cr_2(SO_4)_3$ and describe the precipitate of $Cr(OH)_3$ which forms. Dissolve a little of it in HCl and in HNO₃. In the compounds

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formed, is chromium acting as a base-former or an acidformer? (The color of these compounds sufficiently indicates the base-forming condition of chromium.)

2. Treat most of the $Cr(OH)_3$ with 2-3 g. of Na_2O_2 , add 20 ccm. of water and boil. The color produced is a sufficient indication that chromium is acting as an acid-former.

3. To 5 ccm. of the solution from (2) add NaOH. Is there any precipitate now? Why? Add BaCl₂ and Pb(NO₃)₂ to separate 5 ccm. portions of the solution and make just acid with acetic acid. Describe the precipitates of BaCrO₄ and PbCrO₄ which form. Either precipitate is a test for chromates. The same reactions may be used as tests for barium and lead. State how.

4. Add 10 ccm. of conc. HCl and 5 ccm. of alcohol to the rest of the solution from (2), boil and note change of color. What does it indicate? Write equations for all the reactions which have taken place.

(b) Manganese compounds.

1. Add NaOH to 5 ccm. of $MnSO_4$ and describe the precipitate of $Mn(OH)_2$ which forms. Try to dissolve a little in HCl and in NaOH. Result? In these compounds is manganese acting as an acid-former or a base-former?

2. Dilute 10 ccm. of dilute HNO_3 with an equal volume of water, add some $Mn(OH)_2$ and 2-3 g. of lead dioxid and boil 5 minutes. Allow the PbO₂ to settle and note the color of the solution, which is sufficient indication that manganese is acting as an acid-former.

3. To 5 ccm. of KMnO_4 solution add 5 ccm. of H_2SO_4 and oxalic acid until the color is bleached. Oxalic acid is a reducing agent. What has happened to the permanganate? Add NaOH to the bleached solution until

it is alkaline. Is $Mn(OH)_2$ precipitated now? Why? Write equations for all reactions.

How does oxidation affect the properties of an element? Reduction of a compound has what effect on its properties?

(c) Testing water.

To 15 ccm. of H_2SO_4 add 5 drops of $KMnO_4$ and divide into three test tubes. Fill (1) with distilled water, (2) with tap water, (3) with water in which flowers or grass have been standing. Heat all three to boiling and allow to stand 10–15 minutes. The water in (3) contains organic matter; (1) contains none. Does (2) contain organic matter? Water containing much organic matter is not wholesome.

The water analysis may be continued further by testing it for chlorids (p. 57), sulfates (p. 111), and "hardness" with soap (p. 124). The presence of much "chlorids" sometimes indicates contamination by sewage. The presence of sulfates means little except when accompanied by magnesium and calcium, due to "hardness." The tests for nitrates and ammonia are significant but can not be made quickly or simply.

EXP. 76. BLUE PRINTS AND THEIR REACTIONS

Materials. Solutions of ferric chlorid, FeCl_3 ; ferrous sulfate, FeSO_4^* ; potassium ferricyanid, $K'_3(\text{FeC}_6N_6)^*$; potassium ferrocyanid, $K_4(\text{FeC}_6N_6)$; potassium thiocyanate, KSCN; oxalic acid, $H_2C_2O_4$; hydrogen peroxid, H_2O_2 . Conc. HCl. Chlorin water, Cl_2 . Shingle nails or tacks. Smooth-surfaced letter paper. Cotton.

Apparatus. Test tubes. Glass plate.

* Solutions of FeSO₄ and K₃(FeC₆N₆) must be freshly prepared.

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Procedure. (a) Ferric salts.

Add 5 ccm. of ferric chlorid solution and 5 drops of conc. HCl to 10 ccm. of water and divide into 3 test tubes. To (1) add a drop of potassium ferricyanid, to (2) potassium ferrocyanid; to (3) potassium thiocyanate. Note and record your results.

(b) Ferrous salts.

Add 5 drops of conc. HCl to 5 ccm. of ferrous sulfate solution, dilute with 10 ccm. of water and divide into 3 test tubes. To (1) add a drop of potassium ferrocyanid; to (2) potassium ferricyanid; and to (3) potassium thiocyanate. Note and record your results.

Because of its ready oxidation even by the air, it is almost impossible to get a ferrous solution free from ferric ions. Pure ferrous solutions give colorless test in (1) and (3). Explain your results.

The preceding reactions are used as *tests for iron*; (a) 2 and 3 being used for the ferric condition and (b) 2 for the ferrous condition.

(c) Change from ferrous to ferric.

Pour 2 ccm. of ferrous sulfate into a test tube, add 5 ccm. of chlorin water or hydrogen peroxid, and heat to boiling. Test a drop of the solution for ferrous and ferric iron; if the former is still present, add more oxidizing agent and boil again. Explain what has happened and write equations to express the change, assuming that you used ferrous chlorid instead of sulfate.

(d) Change from ferric to ferrous.

1. Pour 5 ccm. of conc. HCl, 2 ccm. of ferric chlorid solution, and 10 ccm. of water into a test tube, add 4-6 shingle nails and allow to stand for some time, testing a few drops every 5 minutes for ferrous and ferric iron until

the latter disappears. Explain what has happened and write equations expressing the change.

In a room that can be darkened considerably, mix 10 ccm. of each of the following solutions: ferric chlorid, potassium ferricyanid, and oxalic acid.

2. To 5 ccm. of the solution add 10 ccm. of water and see if you have any test for ferrous iron. Let this test tube stand in the dark until the end of the exercise, then note again.

3. Put 5 ccm. of the solution in a test tube and stand in the direct sunlight for 10–15 minutes, then dilute with water. Do you get any test for ferrous iron? What has happened to the ferric iron? Oxalic acid is a reducing agent. Can you explain? Does this change take place in the dark?

4. Dip a wad of cotton in the solution and squeeze almost dry. Rub a sheet of writing paper with the moistened cotton, coating one side evenly and lightly, then place in a dark drawer to dry. Lay the paper on a book, place some opaque object upon it and expose it to the sun until the paper assumes a bronze color. If a fern, flower, or head of grass is used, cover with a glass plate to get sharp outlines. Wash the print in water 2–3 times, dip for a minute into very dilute HCl, wash again, dry and put into your notebook. What is the compound on the paper?

EXP. 77. IDENTIFICATION OF SIMPLE SUBSTANCES

Materials. Water-soluble salts of the different metals.

(a) Determination of the metal.

Dissolve most of the salt (Always keep some for reference) in water. Lay a sheet of white paper on the desk and over it place a piece of clean glass. With a glass rod put 5–10 drops of the solution in different places on the glass. Dip a clean glass rod into NaOH and touch the edge of one of the drops with it. Note any action. In a similar way test other drops of the solution with NH_4OH , $(NH_4)_2S$, and K_2CrO_4 . Using the Table and notes for help, draw inferences as to the metal present in your solution. Confirm your inferences by making the tests for that metal given elsewhere in the manual. If no indication is obtained, examine the flame, heat on charcoal with Na_2CO_3 , and make test for NH_4 . What metal is present in the compound you have? How do you know?

(b) Determination of the acid.

If any metal other than potassium, sodium, and ammonium has been found, make the remainder of your solution alkaline with Na_2CO_3 * solution, boil for 10 minutes, then filter. Acidify 1–2 ccm. of the filtrate with HNO₃ and test for HCl. If the test is negative, acidify the whole filtrate with HCl and boil to decompose the carbonate, noticing whether H₂S or any other gas which you can recognize by the odor is liberated. Using 1–2 ccm. portions of this boiled solution, make tests for the other acids which may be present, referring to the statement of Solubilities, to ascertain which acids are possible. Make the test for carbonates (if necessary) on some of the dry substance. What acid radicle is present in the compound which you have? How do you know?

Boiling with Na_2CO_3 transforms the salt which you had into a sodium salt of the same acid and precipitates the metal as a carbonate, as the following typical equation shows:

$2 \operatorname{AgNO}_3 + \operatorname{Na_2CO}_3 \rightarrow \operatorname{Ag_2CO}_3 + 2 \operatorname{NaNO}_3$.

* This sodium carbonate must be the purest obtainable.

	PRECIPITATES	Caran			
K2CrO4	(NH ₄) ₂ S	NH40H	NaOH	OF SALTS	Territoria (
red red yellow	black black black black	black white white	brown yellow yellow white ¹	white yellow white white	Ag Hg ous Hg ic Pb
yellow olive	brown black yellow	white blue ¹ white ¹	white blue white	white blue white	Bi Cu Cd
	yellow ² orange ² brown ² yellow ²	white white white	white 1 white 1 white 1	white white white white	As Sb Sn ous Sn ic
inter la	white gray black black	white gray green red	white ¹ gray green red	white green green reddish	Al Cr Fe ous Fe ic
	black black buff white	pink ¹ green ¹ buff white ¹	lavender green buff white ¹	red green pink white	Co Ni Mn Zn
yellow		white	white	white white white white	Mg Ba Sr Ca
				white white white	K Na NH ₄

¹ Soluble in excess.

² Sometimes difficult to obtain; add drop of HCl also.

SOLUBILITY OF COMPOUNDS

Potassium, sodium, and ammonium compounds all soluble in water. Nitrates and acetates all soluble.

Chlorids all soluble except silver, mercury (ous), and lead.

Sulfates all soluble except barium, strontium, calcium, and lead.

Carbonates, phosphates, and borates all insoluble except potassium, sodium, and ammonium salts.

Oxids and hydroxids and sulfids all insoluble except potassium, sodium, ammonium, barium, strontium, and calcium.

LISTS OF CHEMICALS AND SUPPLIES

A liberal allowance for a class of 10 students doing all the experiments. All chemicals are C. P.

1 lb. Acid, acetic, 30 % 12 lb. Acid, hydrochloric, Sp. Gr. 1.19 6 lb. Acid, nitric, Sp. Gr. 1.42 1 lb. Acid, oxalic 8 lb. Acid, sulfuric, Sp. Gr. 1.84 2 qt. Alcohol, 95% 1 oz. Alizarin 2 lb. Alum 1 oz. Aluminum, sheet 8 oz. Aluminum sulfate 1 lb. Ammonium carbonate 1 lb. Ammonium chlorid 10 lb. Ammonium hydroxid, Sp. Gr. 0.9 1 oz. Ammonium molybdate 1 lb. Ammonium nitrate 1 oz. Antimony chlorid 1 oz. Antimony oxid 1 oz. Arsenious oxid 8 oz. Barium chlorate 8 oz. Barium chlorid 8 oz. Barium nitrate 1 oz. Bismuth chlorid 1 oz. Bismuth oxid 8 oz. Bromin 1 oz. Cadmium nitrate 1 lb. Calcium carbid 10 lb. Calcium carbonate, marble chips 8 oz. Calcium carbonate precipitated chalk 1 lb. Calcium chlorid, granules 1 lb. Calcium fluorid, fluorspar, powder 8 oz. Calcium nitrate 2 lb. Calcium oxid, lime, in tins 1 lb. Calcium sulfate, gypsum

Paris 1 lb. Carbon disulfid 1 oz. Cochineal, ground 1 lb. Charcoal, animal. Boneblack. 1 lb. Charcoal wood 1 lb. Charcoal wood blocks 1 lb. Chloride of lime 1 lb. Chloroform 4 oz. Chromium sulfate 4 oz. Cobalt chlorid 4 oz. Cobalt nitrate 1 oz. Congo red 1 lb. Copper, foil, 1 in. 2 lb. Copper turnings 4 spools Copper wire, Nos. 16, 18, 24, 30. 1 oz. Copper oxid, black powder 3 lb. Copper sulfate 1 lb. Ether 1 oz. Fehling's solution tablets 15 packs Filter papers, 4 in. 2 packs Filter papers, 6 in. 1 pack Filter papers, 12 in. 1 oz. Hydrochinon 1 oz. Indigo 4 oz. Iodin, resublimed 1 lb. Iron powder "alkoholized " 2 spools Iron wire, Nos. 16 and 24 1 lb. Iron chlorid (ic) 1 lb. Iron sulfate (ous) 2 lb. Iron sulfid (ous) for H₂S 1 lb. Lead, sheet 8 oz. Lead, acetate 4 oz. Lead dioxid 1 lb. Lead nitrate 1 lb. Lead oxid, litharge

2 lb. Calcium sulfate, plaster of

1 oz. Litmus cubes 25 books Litmus paper, red 25 books Litmus paper, blue 4 oz. Logwood, ground 4 oz. Magnesium, ribbon 8 oz. Magnesium nitrate 4 oz. Magnesium oxid, wet process 8 oz. Magnesium sulfate 2 lb. Manganese dioxid, powdered 8 oz. Manganese nitrate 8 oz. Manganese sulfate 1 lb. Mercury 4 oz. Mercury chlorid (ic) 4 oz. Mercury nitrate (ic) 4 oz. Mercury nitrate (ous) 4 oz. Mercury oxid 4 oz. Nickel sulfate 1 lb. Oxone. (Fused sodium peroxid) 1 oz. Phenolphthalein 1 oz. Phosphorus, red 4 oz. Phosphorus, yellow 1 oz. Phosphorus pentoxid 1 oz. Potassium, metal 8 oz. Potassium bromid 8 oz. Potassium carbonate 3 lb. Potassium chlorate 1 lb. Potassium chlorid 4 oz. Potassium chromate 1 lb. Potassium dichromate 8 oz. Potassium ferricyanid 4 oz. Potassium ferrocvanid 1 oz. Potassium acid sulfate 2 lb. Potassium hydroxid, sticks 2 lb. Potassium nitrate

4 oz. Potassium iodid 4 oz. Potassium nitrite 8 oz. Potassium permanganate 8 oz. Potassium sulfate 1 oz. Potassium thiocyanate 1 lb. Rochelle salt 4 oz. Silver nitrate 4 oz. Sodium, metal 4 oz. Sodium acetate 1 lb. Sodium carbonate, purest, drv 5 lb. Sodium chlorid, fine 4 oz. Sodium chromate 2 lb. Sodium hydroxid, sticks 1 lb. Sodium nitrate 4 oz. Sodium nitrite 4 oz. Sodium peroxid, powder 8 oz. Sodium phosphate 2 lb. Sodium sulfate, crystals 8 oz. Sodium sulfite, crystals 2 lb. Sodium thiosulfate, hypo 8 oz. Strontium chlorate 4 oz. Strontium chlorid 3 lb. Sulfur 1 oz. Tartar emetic 1 lb. Tin, granulated 8 oz. Tin chlorid (ous) 1 oz. Tin oxid (ic) 1 sheet Turmeric paper 1 lb. Zinc, dust 3 lb. Zinc, mossy 1 lb. Zinc. sheet 4 oz. Zinc nitrate 4 oz. Zinc oxid, wet process 8 oz. Zinc sulfate

MATERIALS TO BE OBTAINED LOCALLY AS NEEDED

4 oz.	Baking powders,	several	dif-	Blue print
	ferent kinds			1 lb. Borax
1 lb.	Baking soda			4 doz. Candles.

1 pt. Benzine

4 oz. Beans

2 Carbonated water, syphons or bottles

paper

Christmas

INDIVIDUAL APPARATUS

4 oz. Cheese 10 yd. Cheesecloth (towels) 4 oz. Coal (soft) 4 oz. Corn meal 4 lb. Cotton batting 1 yd. Cotton cloth, colored 1 yd. Cotton cloth, white 4 oz. Cream of tartar 1 Egg, raw 1 Egg, hard boiled 4 oz. Fertilizers, several different kinds 4 oz. Flour Fruit 4 oz. Glucose sirup 4 oz. Hydrogen peroxid 1 box Iron picture wire 1 lb. Iron shingle nails 1 pt. Kerosene 4 oz. Lard Matches, parlor Matches, sulfur

4 oz. Meat 4 oz. Milk 4 oz. Nuts 1 pt. Olive oil 1 lb. Paraffin 4 oz. Peas 1 lb. Rock candy 10 sheets Paper, black 10 sheets Paper, writing 1 Potato 4 oz. Starch 1 cake Soap 1 pt. Turpentine 1 lb. Sugar, dark brown 1 lb. Sugar, granulated Vegetables 4 oz. Vinegar 1 oz. Wool or felt 4 oz. Washing powder, several different kinds 5 lb. Washing soda

3 Yeast cakes.

INDIVIDUAL APPARATUS

For class of 10 students. Liberal allowance for breakage has been made.

- 12 Beakers, 500 ccm.
- 24 Beakers, 250 ccm.
- 24 Beakers, 125 ccm.
- 12 Brushes, test tube
- 15 Burners, Bunsen with wing tips
- 12 Calcium chlorid tubes, straight, 1 bulb
- 12 Capsules for sodium (see p. x)
- 12 Clamps for condensers
- 12 Clamps for test tubes
- 12 Condensers (Liebig), 15 in.
- 12 Combustion spoons, $\frac{1}{2}$ in. bowl
- 15 Crucibles, porcelain, No. 0, with covers
- 25 Dishes, evaporating, No. 1
- 12 Dishes, lead
- 12 Files, 3-cornered

- 5 packs filter paper, 9 cm.
- 5 packs filter paper, 15 cm.
- 12 Flasks, flat bottom, 500 ccm., with 2-hole rubber stoppers to fit
- 25 Flasks, flat bottom, 250 ccm., with 2-hole rubber stoppers to fit
- 12 Flasks, round bottom, 500 ccm., with 1-hole rubber stoppers to fit
- 12 Flasks, round bottom, 250 ccm., with 1-hole rubber stoppers to fit
- 12 Frying pans, iron, 5 in.
- 12 Funnels, 21 in.
- 5 doz. Fruit jars, pint
- 50 Glass plates, 3×3 in.
- 12 Glass plates, 6×6 in.
- 10 Glass plates, 3×4 in. Cobalt glass

- 100 ft. Glass tubing, 5 mm. outside diameter. For bending
- 25 ft. Glass tubing, 3 mm. outside diameter. For stirring rods.
- 12 Graduates, 25 ccm.
- 12 Medicine droppers
- 12 Mortars with pestles, porcelain, 4 in.
- 12 Pincers, iron, 4 in.
- 12 Pipe stems, clay
- 15 Pipe stem triangles, small
- 12 Pneumatic troughs (see p. vii)
- 12 Retorts, 125 ccm., with ground glass stoppers

- 50 ft. Rubber tubing, ³/₁₆ in. inside diameter
- 12 Stand irons with 2 rings
- 20 doz. Test tubes, soft, $6 \times \frac{3}{4}$ in.
- 20 Test tubes, side-neck, $6 \times \frac{3}{4}$ in. 12 1-hole rubber stoppers to fit
- 24 Test tubes, ignition, $6 \times \frac{3}{4}$ in.
- 12 Test-tube holders, brass wire
- 12 Test-tube racks for 12 test tubes
- 15 Thistle tubes, $10 \times \frac{3}{15}$ in.
- 20 Wire gauze, 5×5 in.
- APPARATUS FOR GENERAL USE
- 1 doz. Asbestos sheets
- 2 Balances, platform, weights 500 g. to 1 g.
- 5 Balances, horn pan, 7 in. beam weights 100 g. to 0.01 g.
- 1 Barometer
- 5 Beakers, 1000 ccm.
- 3 Dry cells
- 4 Bottles, 2 liter (Acid bottles)
- 12 Bottles, ground glass stoppers, 1 liter
- 100 Bottles, ground glass stoppers, 250 ccm.
- 50 Bottles, salt mouth, 8 oz.
- 10 Bottles, salt mouth, 16 oz.
- 4 Burettes, 50 ccm., graduated
- 1 Combustion furnace
- 15 ft. Combustion tubing, 1 cm. inside diam. Exps. 19 and 47
- 1 set Cork borers (6 in set)
- 1 gross Corks, assorted
- 1 Eudiometer and standard, coil and mercury (10 lb.) Exp. 33
- 2 Dishes, evaporating, 6 in.
- 1 Dish, evaporating, 10 in.
- 2 Files, round
- 6 Flasks, flat bottom, 1000 ccm., 1and 2-hole rubber stopper to fit

- 4 Funnels, glass, 4 in.
- 1 Funnel, glass, 6 in.
- 3 Gas measuring tubes, graduated, 12 in. long, ½ in. inside diameter
- 4 Gas measuring tubes, not graduated, 12 in. long, $\frac{1}{2}$ in. inside diameter
- 1 Gas pipe, iron, $20 \times \frac{1}{2}$ in.
- 2 Graduates, 1000 ccm.
- 2 Graduates, 500 ccm.
- 4 Graduates, 100 ccm.
- 1 Hammer
- 3 Lamp chimneys (student)
- 2 Magnets
- 2 Magnifying glasses
- 1 Mortar and pestle, iron, 6 in.
- 2 Pinch cocks, Mohr's, medium
- 2 Pipettes, 100 ccm.
- 2 Pipettes, 20 ccm.
- 6 Plates, porcelain, 6 in. (Buy in crockery store)
- Platinum foil, 1000 in., 2 sq. in.
- Platinum wire, No. 27, 25 in.
- 2 lb. Rubber stoppers, 1 and 2 hole, Nos. 0-5
- 2 Separating funnels, 100 ccm.
- 2 Thermometers, 0-200°

NOTE ON APPARATUS AND CHEMICALS 143

NOTE ON PURCHASING APPARATUS AND CHEMICALS

The preceding lists of apparatus and chemicals include everything needed for a class of ten students working individually and performing all the experiments, and for all the instructor's experiments, in a school where chemistry has never been taught before. For the second and all subsequent years, the lists may be greatly cut down. The quantities of chemicals specified will last, in many instances, several years. It is not economical to buy in smaller quantities, however, because the prices charged are proportionately much higher. With careful handling, much of the apparatus for general use need never be replaced.

The attention of instructors in schools handicapped by lack of funds is called to the following suggestions for minimizing the expense for chemicals and apparatus. When unusual or expensive apparatus is called for in the Manual, suggestions for substituting other materials are frequently made (see p. x, xi, xii, 13, 16, 21, 26, 28, 32, 40, 69). The barometer, thermometers, dry cells, induction coil and mercury may be borrowed generally from the physics department (possibly the eudiometer, also). Test-tube racks may be constructed in the manual training department. Lead dishes may be made out of sheet lead; blowpipes from glass tubing (see p. 91).

For satisfactory results, Exp. 47 requires a combustion furnace, useful also in Exp. 16 a. By omitting Exp. 47 and (if the apparatus cannot be borrowed or devised from available material) Exps. 16 a and 33, a relatively great saving can be effected. Condensers and clamps to hold them are relatively expensive and are used only in Exps. 21 and 51. By having students use this apparatus in turn, the number may be cut down to three if necessary (or entirely eliminated, see p. 40). Similarly, six retorts may be made to serve for Exp. 36. Though it is not recommended, students may work in pairs. Much of the apparatus listed for general use is for the instructor's use and convenience about the laboratory. It is possible, however, to get along with one half of the bottles, evaporating dishes, funnels, gas measuring tubes and graduates mentioned in this list.

If all the above devices are adopted, a saving of from one half to one third of the cost of the lists as printed may be accomplished. This apparent saving will be at the expense of the instructor, from whom additional labor will be necessitated. It is to be regarded as doubtful economy, for the efficiency of his teaching is likely to be lessened thereby.

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