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(PRACTICAL.)

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CHAPTER I.

INTRODUCTORY EXPERIMENTS.

To bore a cork.—The cork must first be softened by being rolled gently but firmly under the foot. It will shrink somewhat during this process, so that a cork somewhat wider than the neck of the bottle should be chosen. Take now a round file and press the sharp end gently into the cork, turning it round all the time. The file will gradually penetrate to the far side, leaving a small hole, which can be enlarged by filing away different parts of the wall, until it is slightly less than the tube it has to contain.

More regular holes can be made by means of proper cork borers, which are brass tubes of different sizes, with one end sharpened and the other perforated to admit a steel rod. The borer having been chosen of slightly less diameter than the tube for which the hole is intended, the sharp end is dipped in water, and the rod inserted in the hole at the other end. The edge is pressed gently into the cork, and the borer twisted (always in the same direction) under gentle pressure until it emerges at the other side. It should *not* be pressed up against a hard object as this will blunt the borer. Well sharpened borers can also be used for indiarubber stoppers. In this case they are moistened either with alcohol or glycerine, and pressed through more slowly. Tubes should always be dipped in water before being pushed into the hole.

To bend a piece of glass tubing.—Take a piece of $\frac{3}{16}$-inch soft tubing, and hold it horizontally in the upper part of an ordinary gas flame, with its length in the plane of the flame. Turn the tubing slowly round and round so as to heat it uniformly. When it begins to soften remove it from the flame and bend it slowly and carefully to the required shape. Allow it to cool, and *then* remove the coating of soot from the bend.

To cut narrow glass tubing with a file.—Take a piece of glass tubing and lay it on a table. Taking hold of it with the finger and thumb of the left hand near the point where it is to be cut, draw one edge of a sharp triangular file *once* across it, from heel to point. The tube can now be broken by forcibly attempting to bend it away from the file-scratch, and at the same time trying to pull it apart. Round off the sharp edges by holding the tube vertically in the Bunsen flame, until the flame is coloured strongly yellow by the sodium of the glass.

To draw out a piece of glass tubing; to make a jet.—Take a short piece of tubing and heat the middle portion in a small blowpipe flame, supporting each end of the tube with the fingers, and continually turning it round so as to heat all sides equally; when the tube is quite soft, remove it from the flame and slowly draw it out. The more slowly it is drawn out, the more gradually will the tube taper towards the centre. Then with a file cut the tube at a suitable point, round off the cut end in the flame, and the jet is ready for use.

To make a bulb tube.—Take a piece of $\frac{3}{16}$-inch tubing, about 6 inches long, and heat one end in the blowpipe flame until it is completely closed and rounded off. Before this rounded end becomes too thick, take the tube out of the flame, and, holding it vertical, blow into it steadily, but not too strongly, until a small bulb is blown at the heated end.

Use of Spatula.—It is often necessary to take a small quantity of some solid substance out of a bottle. For this purpose the spatula—an instrument very like a small table-knife—is used. A small bone or ivory paper-cutter answers the purpose very well. The blade of a penknife is sometimes used, but the use of iron or steel is objectionable. A platinum spatula is the best.

To fix a platinum wire in a glass holder.—Take six inches of narrow glass tubing and draw it out a little in the middle as described above. Cut in two at the middle of the drawn-out part. Into the narrow cut end insert a piece of platinum wire about two inches long; then hold this end in the flame until the glass closes round the wire. A piece of glass rod makes a better holder. Draw the rod out and cut in two as described above. Then hold the narrow end of the rod in the flame until it is quite soft, and insert the piece of platinum wire into the soft end. The free end of the wire should be bent into a loop or coiled into a flat spiral.

The platinum wire thus mounted will be in frequent use for flame tests, and should be kept dipped in hydrochloric acid in a test tube. This can be done by fitting the holder with a cork which fits into the mouth of the test tube. Kept in this way the wire is always clean.

To make a wash-bottle.—Take a 16-oz. flask, either of the ordinary round form or, better, of the conical shape, fit it with a good cork or indiarubber stopper, in which bore two holes (for the cork use a round file; for the rubber, a sharp cork-borer moistened with alcohol). Then fit two pieces of tubing, bent as shown in the figure, first rounding off the ends in the flame, and see that the tube *a* reaches very nearly to the bottom of the flask; to the end *a* of the longer tube fit a piece (about 1½ inches) of narrow (⅛ in. diam.) indiarubber tubing, carrying at the other end a short piece (about 1 inch) of glass tubing drawn out to a jet. The wash-bottle is to be nearly filled with distilled water, which can be expelled when required by blowing down the tube *m*, and the fine stream issuing from the jet can be discharged in any desired direction by the flexibility of the rubber tubing. The wash-bottle is used chiefly for washing precipitates on filters, and serves as a reservoir of pure water for general purposes.

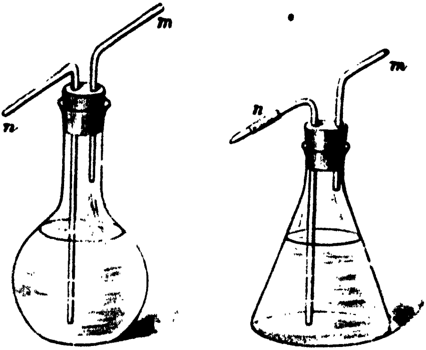


Fig. 1.

To make a desiccator.—In some of the experiments to be described later it will be found necessary to prevent the substances taking up moisture from the air. In order to secure this, we place them in a dry atmosphere enclosed in a vessel known as a desiccator. Take a piece of thick plate glass about five or six inches square, and select a stout-walled cylindrical glass vessel about six inches high and about four inches in diameter. A small gas jar or wide-mouthed bottle serves very well. The edge of the mouth must be ground quite flat by rubbing it with a little water on a flat piece of sandstone. Take a shallow, fairly wide beaker, of such a size that the gas jar can pass over it easily, and half fill it with either calcium chloride (anhydrous) or concentrated sulphuric acid. Over the mouth of the beaker place a pipe-clay triangle, to form a rest, and bend down the three ends of the wires over the edge. Clean and dry the plate and gas jar. Place the beaker containing the desiccating agent on the plate, and cover it with the jar. In order to have close contact between the jar and the plate, it is advisable to grease the edge with a little lard, or stiff grease. If sulphuric acid is used, care must be taken that no splashing is caused by shaking.

The water bath.—It is frequently necessary to evaporate liquids over the water bath, for if they are heated with the naked flame, the temperature may become too high and the substance be spoiled. A simple form of water bath consists of a beaker which is partially filled with water, kept boiling by the flame of a Bunsen burner. The liquid to be evaporated is put into a porcelain basin placed on the top of the beaker. The steam rising from the boiling water heats the basin and produces the required evaporation. More serviceable water baths, of various shapes, are made of copper, generally with moveable rings at the top so that basins of various sizes can be fitted in.

CHAPTER II.

CHARACTERISTIC PROPERTIES OF SOME COMMON SUBSTANCES.

In the preceding chapter, we have described some of the more important operations, with which the student must become familiar, before he can conduct the experiments to be described later on. To be successful in these he must carefully carry out the instructions given—even in regard to what may appear unimportant details. In many cases the accurate boring of a cork will mean the difference between the success and failure of an experiment, *e.g.* in the preparation of gases. Again, awkward accidents from sharp tubes can be prevented by spending a few minutes in holding them in the gas flame, as directed above. Apart from this, however, he must also proceed to his work with the determination to observe as much as possible, and deduce from the observed facts, their causes. It is not always possible to give reasons for the things he sees, but it *is* possible to notice what takes place. An account of every experiment should be written out—concisely, and in such cases as the collecting of oxygen, hydrogen, etc., a sketch should be made of the apparatus used. Whenever experiments appear to fail, although carried out carefully according to the instructions, a note of the fact should be made. The explanation may occur to the student later when his knowledge is more complete.

It is often of great importance to the chemist to be able to identify quickly some common substance. An elaborate analysis would take too long, so he has to rely on the simpler characteristics. The student should cultivate the habit of noting the more apparent properties of the materials he uses and sees used, he should observe the appearance of the substance, notice if it has a characteristic smell, test its hardness, and observe the effects of heat on it and if it sublimes the characters of the sublimate. Below is a short scheme for such an examination.

First.—Observe whether the substance is a *solid*, *e.g.* common salt, a *liquid*, *e.g.* ordinary water, or a *gas*, *e.g.* air.

Second.—Note the *colour* carefully. By simple inspection we can distinguish between bromine and alcohol, copper and zinc, ferric oxide and sulphur, etc.

Third.—*Smell* the substance—not too vigorously or the results may be unpleasant. Many substances have very characteristic odours and can be recognised by them. Take for instance a solution of sulphur dioxide in water, alcohol, bisulphide of carbon, bromine, etc.

Fourth.—Note the *lustre*—whether metallic, glassy, resinous, etc., or its absence. Thus ordinary wood charcoal is quite dull, whilst graphite has a distinct metallic lustre. Quartz again has a glassy lustre, whilst that of rock sulphur is resinous.

Fifth.—Test the *hardness* and *malleability* of the substance. Diamond is harder than any other body, and can always be distinguished from another substance by means of that property. Aluminium again is harder than tin, iron than cadmium, etc. The simplest manner in which to test the relative hardness of two substances, is to find out which will scratch the other. The one which sustains the scratch is the softer. To test the malleability we usually place a small portion of the body on a small iron block, and strike it smartly with a hammer. In this way we can distinguish, say, antimony and lead. The antimony is brittle and breaks into pieces; the lead is malleable, and flattens out.

Sixth.—If the substance is a solid, heat a little in a dry test tube. This test would enable us to distinguish at once between iodine and graphite, which in most respects resemble each other very closely. The iodine volatilises to a beautiful violet vapour, the graphite remains unaltered. Common salt and ammonium chloride, again, resemble each other in many respects. They are both white solids, soluble in water, and possessing a sharp taste. On heating a little of each in separate tubes, we can distinguish at once. Ammonium chloride sublimes, that is, it passes at once into vapour and condenses in the cool part of the tube to a white solid. We get a white crusty deposit in the upper part of the tube. Common salt, on the other hand, does not volatilise.

Seventh.—If the substance is a liquid, boil a small portion in a test tube and note the colour of the vapour. On boiling, *e.g.* a strong solution of hydrochloric acid and a little strong nitric acid in separate test tubes, clouds of white irritating fumes are given off in each case, but if a sheet of white paper is held behind the tube containing the nitric acid, the vapour inside is seen to have a distinct reddish tint. This is due to the slight decomposition of of nitric acid into oxides of nitrogen, oxygen, and water. A drop of bromine, again, when warmed gives off very dark reddish-brown vapour, of a pungent, irritating odour.

Eighth.—Try the action of water on a little of the substance. This test will at once enable us to distinguish quick-lime (calcium oxide) from common salt. The quick-lime will combine with the water, swell up, and become hot. The salt will simply dissolve to a clear liquid. When a a drop or two of water is added to a small portion of sulphur trioxide, there is a hissing sound, as of water dropped on hot iron. Great heat is evolved, and the white solid dissolves to a clear liquid. If the solid had been the metal sodium, we should have had the hissing sound, and effervescence of a gas, hydrogen, which would ignite with a sharp pop when we place a lighted match at the mouth of the tube.

Some of the commoner substances used by the chemist are briefly described below. The student should examine these in the manner mentioned in the foregoing scheme.

Ammonia Solution.—A colourless liquid with the characteristic smell of ammonia. If a little of it be heated in a test tube ammonia gas is given off, which we can detect by holding a piece of red litmus paper near the mouth of the tube; the colour of the paper is changed to blue.

Ammonium Chloride (*Sal ammoniac*).—A white solid, soluble in water, volatilises when heated, and condenses in the cool part of the tube.

Ammonium Nitrate.—A white solid, very soluble in water with considerable fall of temperature. When heated in a dry tube it first fuses and then gives off bubbles of gas—nitrous oxide. It should not be heated too strongly, or a slight explosion may occur.

Bromine.—A heavy liquid, almost black, which gives off reddish-brown vapours of an offensive and irritating smell. It is sufficiently soluble in water to give a coloured solution. When warmed it gives off red vapours. All work with bromine should be performed in the draught chambers.

Calcium Oxide (*quick-lime*).—A hard greyish solid in its common form, white when pure. It combines with water, giving out much heat, and falling to a powder it is then said to be slacked. Remains unchanged when heated.

Calcium Chloride.—White or colourless solid. When left exposed to moist air it absorbs the moisture, and dissolves in it, forming a clear liquid. It is extremely soluble in water. On account of its attraction for water it is often used for drying gases.

Calcium Sulphate.—A white solid, usually in the form of powder, when it is known as "Plaster of Paris." When mixed with a little water Plaster of Paris forms a paste which sets to a hard mass on standing, combining with the water. Calcium sulphate is almost insoluble in water, and remains unchanged on heating.

Carbon.—(1) *Charcoal.* A dull, soft, black solid, insoluble in water, unchanged by heating in a tube.

(2) *Graphite.* Greyish black solid, with bright metallic lustre, soft and easily split into small plates. Insoluble in water, and unchanged on heating.

(3) *Diamond.* Bright, glassy crystals. Harder than any other known substance. Accordingly, it readily scratches glass. Insoluble in water, and unchanged by heating in a tube. Brittle.

Carbon Bisulphide.—Colourless liquid, of offensive odour. Heavier than water, with which it does not mix, but sinks as oily drops to the bottom of the tube. When warmed it simply gives off its offensive vapour, which is inflammable, burning with a bluish flame.

Caustic Potash and Caustic Soda.—These are both white solids. They cannot be distinguished from one another by their appearance, but if a clean platinum wire be drawn over the surface of a piece of caustic soda and then placed in the Bunsen flame, the flame is coloured an intense yellow; while, with caustic potash, the flame is coloured a faint violet. Both substances are sold in round sticks, which dissolve in water with the production of much heat. The solution turns red litmus blue.

Chalk.—An opaque white solid which effervesces when a few drops of hydrochloric acid are poured upon it. It is insoluble in pure water.

Copper.—A reddish metal, capable of taking a high polish. Tarnishes when heated. Malleable and fairly hard.

Copper Oxide.—A black solid, dull, usually in the form of powder. Insoluble in water, and unaltered by heat.

Copper Nitrate.—Green crystals, very soluble in water. When heated in a dry tube the crystals give off reddish brown fumes of nitric peroxide mixed with oxygen, and leave a black residue of copper oxide.

Copper Sulphate.—Fine blue crystals of glassy lustre, known as "blue vitriol." Soluble in water. When heated in a dry tube water is given off which condenses in the cool portion of the tube, whilst the colour of the residue gradually changes to white. On adding water again to the white residue it assumes its original blue colour.

Iodine.—Greyish black solid of bright metallic lustre, fairly soft and brittle. Almost insoluble in water. When heated in a dry tube it gives off beautiful violet vapours.

Iron.—Greyish white solid of bright metallic lustre. Hard and malleable. Not soluble in water, tarnishes when heated in a tube.

Ferrous Sulphate.—Green, glassy crystals, soluble in water. Heated in a dry tube, water is first given off, which condenses in the cool portions of the tube, the crystals turning to a lighter colour. On further heating, a residue of ferric oxide remains.

Ferric Chloride.—Yellow solid, usually of a dull appearance. Readily takes up water, giving a clear yellow solution. Very soluble in water.

Ferric Oxide.—Reddish brown solid, usually seen as a dull powder. Insoluble in water, and unchanged on heating.

Indigo Solution.—Dark blue liquid, bleached by concentrated nitric acid, but unchanged by alkalies and dilute acids.

Litmus Solution.—Blue or red solution, according as it is alkaline or acid.

Lead.—A greyish solid, usually dull, but a freshly cut surface shows a bright metallic, almost silvery, lustre. Soft and malleable; can be scratched by the finger nail, and easily flattened under the hammer. Insoluble in water. When heated in a dry tube it melts readily, and tarnishes, taking up oxygen and forming a coating of lead oxide.

Lead Nitrate.—A white crystalline solid, the crystals usually somewhat opaque. Glassy lustre. Soluble in water. When heated it first crackles, and then gives off reddish brown fumes of nitric peroxide mixed with oxygen. The residue, lead oxide, is yellow when hot, but assumes a lighter colour on cooling.

Lime Water.—A colourless liquid which turns red litmus paper blue. If a glass tube, open at each end, be inserted into some of the liquid in a test tube, and air from the lungs be blown through the liquid, it becomes milky, the carbonic acid in the breath combining with the lime to form insoluble calcium carbonate.

Litharge.—A yellow powder which becomes brown red when heated. It is insoluble in cold water.

Magnesium.—Silvery white metal. Usually dull because coated with the oxide, magnesia, but if this is scraped off it is seen to have a bright metallic lustre. Insoluble in water. Becomes dull when heated in the tube, and if heated strongly enough, will take fire. The best way to show this latter property is to take a short piece of magnesium ribbon, hold one end in a pair of tongs, and bring the other into the flame. It will burn with a dazzling light.

Magnesium Sulphate (*Epsom salts*).—White or colourless crystals with glassy lustre. Soluble in water. When heated they become opaque and lose their water, which condenses in the cool part of the tube.

Manganese Dioxide.—Usually seen as a dull black powder. Insoluble in water. When moderately heated in a dry tube remains unchanged, but when *strongly* heated gives off oxygen.

It is impossible to distinguish by the ordinary simple tests between powdered charcoal, oxide of copper, and powdered manganese dioxide. The charcoal could be identified by its comparative lightness. By adding a few c.c. of hydrochloric acid and warming, we can easily distinguish them. The charcoal remains unaltered, the copper oxide gives a solution which is green when allowed to settle, the manganese dioxide gives off a yellow gas, chlorine, of offensive, irritating odour. The white fumes given off in each case are only hydrochloric acid gas.

Mercury.—Silvery white *liquid* of metallic lustre. Very heavy, and insoluble in water. When heated in a dry tube it tarnishes slightly and boils, the vapour condensing on the sides of the tube in the form of minute silvery globules.

Mercuric Oxide.—A bright red powder. Heavy, and insoluble in water. When heated in a dry tube it first darkens, and on stronger heating decomposes, giving off oxygen, which can be identified by its causing a spark on a piece of wood to burst into flame, the mercury being deposited as a bright mirror on the sides of the tube.

Nitric Acid.—A colourless liquid (if exposed to bright light slightly yellow). Mixes readily with water, and when heated gives a reddish vapour of a very irritating odour.

Phosphorus.—(1) *Yellow.* A waxy-looking solid, yellow, and if freshly cast into sticks, transparent. It is always kept under water, because of the ease with which it combines with oxygen and becomes ignited. Cut a small piece under water, dry, and place it on a small plate under a jar. It will smoulder and gives off white fumes of oxide of phosphorus. If it does not burst into flame, lift off the jar, touch with a hot piece of iron wire, and replace the jar. It will now burn giving off dense white fumes. When it has burnt out, a red residue will remain on the plate. This contains red phosphorus amongst other things.

(2) *Red Phosphorus.* This is a dark-red solid, usually seen as a dull powder. In the lump form it has a glassy lustre. It is insoluble in water. When heated in a dry tube it partly changes to the yellow form, which combines with the oxygen of the air producing white fumes of oxides. It can be handled more easily than the yellow phosphorus as it is stable in air at the ordinary temperature.

Potassium.—A silvery solid when preserved from the action of the air by being kept in naphtha. When cut with a knife it has a metallic lustre, but the surface rapidly tarnishes and becomes coated with a bluish film of oxide. When a small piece is treated with a few drops of water, there is a brisk effervescence of hydrogen. When a small portion is heated in a dry tube, it first melts, and then takes fire, and burns with a blue flame, the white residue being oxides of potassium.

Potassium Chlorate.—White crystals with glassy lustre. Soluble in water. When heated in a dry tube the crystals at first crackle and break up (this is due to drops of water which are inside the crystals expanding into steam and so breaking the crystals open), then melt, and give off bubbles of oxygen. This can be identified as in the experiment with mercuric oxide. The white residue is potassium chloride.

Potassium Nitrate (*Saltpetre or Nitre*).—White or transparent crystals with glassy lustre. Soluble in water. When heated in a dry tube the crystals first crackle and fly about, then fuse, and finally on being strongly heated give off bubbles of oxygen, and leave a white residue of potassium nitrite.

Sodium.—A silvery metal resembling potassium in most respects. It burns, however, with a *golden yellow* flame.

Sodium Chloride (*common salt*).—A white crystalline powder; when large enough, the crystals are seen to be colourless cubes which have a glassy lustre. They crackle when heated. Soluble in water.

Sodium Sulphate (*Glauber's salt*).—A white crystalline solid; when fresh, the crystals are transparent and have a glassy lustre. When exposed to the air they *effloresce*, *i.e.*, give up their water of crystallisation, and become powdery on the surface. When heated the salt fuses, then loses its water, which condenses again on the sides of the tube, and leaves a white residue of the *anhydrous* sulphate.

Sodium Nitrate.—A white crystalline solid which takes up water from moist air, and dissolves in it forming a clear solution. When heated it behaves like potassium nitrate, giving off oxygen, and leaving a residue of sodium nitrite.

Sodium Carbonate (*washing soda*).—When freshly prepared it is obtained as large transparent crystals of glassy lustre, but on exposure to the air, these *effloresce*, like sodium sulphate, and become coated with an opaque powder. When heated, it loses water and leaves the anhydrous carbonate.

Sulphur.—A yellow solid. Generally seen as a powder (flowers of sulphur) or as lumps (rock or roll sulphur). It has a resinous lustre, and is insoluble in water. When a piece of roll sulphur is held in the hand, near the ear, it is heard to crackle, the heat of the hand causing the outside to expand and crack. When heated over the flame it melts to an amber-coloured liquid. This, on further heating, darkens and becomes viscous, until it is as thick as treacle; on still further heating it becomes less viscous and finally boils, giving off a dark brown vapour, which takes fire with a slight explosion, burning with a pale blue flame.

Sulphur Trioxide.—A white solid, usually in silky needles. Combines eagerly with water, great heat being evolved. If a little is dropped into water, there is a hissing sound, and sulphuric acid is formed. When gently heated it sublimes.

Sulphuric Acid (*oil of vitriol*).—An oily liquid, colourless when pure. When mixed with water the temperature rises considerably. When heated it gives off irritating fumes of sulphur trioxide.

Nordhausen Sulphuric Acid.—Resembles sulphuric acid, but contains in addition a large amount of dissolved sulphur trioxide. On warming, this readily distils away. On dropping a little of the acid into water there is the hissing sound noticed in the case of sulphur trioxide.

Water.—Pure water has no taste or smell, and, except in very thick layers, it is colourless; when heated in a porcelain dish it evaporates and leaves no residue. It gives no turbidity or colouration with the following reagents, barium chloride, silver nitrate, Nessler's reagent; the reason for applying these tests will be understood after reading chapter vii.

Zinc.—Bright bluish-white solid, with metallic lustre. As generally seen it is slightly tarnished and therefore dull. Hard and brittle. Not acted upon by water at the usual temperatures. When heated in a dry tube it melts, and becomes covered with a coating of zinc oxide.

Zinc Oxide.—A white flaky solid, which becomes pale yellow on heating. It is insoluble in water.

Zinc Sulphate.—White or colourless crystals with glassy lustre. Soluble in water. When heated in a dry tube, it first fuses, and then gives up part of its water of crystallisation, which condenses in the cool parts of the tube. The residue still contains water which can only be expelled by strongly heating.

CHAPTER III.

REPARATION AND PROPERTIES OF SOME COMMON ELEMENTS AND COMPOUNDS.

Section I.—Oxygen.

Preparation.

Exp. 1.—Fit up a small hard glass flask with cork and bent delivery tube and arrange the pneumatic trough for the collection of the gas as shown in Fig. 2.

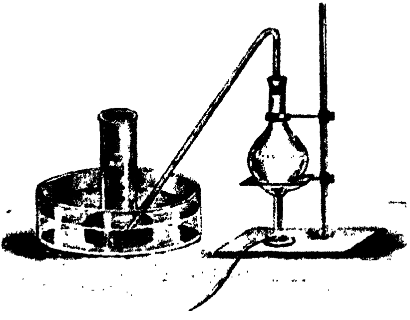


Fig. 2.

Several gas jars must be quite filled with water and a greased plate placed over the mouth of each; the jars can then be turned mouth downwards without the water running out. Now place the jars in the trough (partly filled with water) with their mouths under water and remove the plates; the water will still remain in the jars. Place one of the jars on the beehive shelf, which is simply an inverted earthenware dish with one hole at the side for the delivery tube, and another at the top to allow the gas to pass into the jar. Next take about 20 grammes of potassium chlorate; powder, and mix it intimately with about one-fifth of its weight of black oxide of manganese. Put the mixture in the flask, replace the tube and cork, and heat gently over a Bunsen flame. At first bubbles of air, afterwards bubbles of oxygen, will be driven over. When this is the case, place the delivery tube under the beehive shelf; the oxygen will bubble through and displace the water in the jar. When it is full, cover the mouth with a glass plate, and remove from the trough. In a similar manner fill the remaining jars, and then remove the end of the tube from the water. One jar should be set aside, mouth downwards, in a small dish of water, for a future lesson.

Observe that the gas is colourless, without smell, and tasteless. It is also but slightly soluble in water, or we could not have collected it in the manner described.

The manganese dioxide remains unaltered after the experiment. The reaction is represented by the equation—

$$2KClO_3 = 2KCl + 3O_2.$$

By treating the black residue, which remains in the retort, with hot water and afterwards filtering, the KCl can be dissolved and separated from the manganese dioxide which remains on the filter paper.

Properties of Oxygen.

Exp. 2.—Place a small piece of sulphur on a deflagrating spoon which is provided with a disc. Ignite the sulphur in the lamp flame, and notice how feebly it burns. Now remove the plate from a jar of oxygen and rapidly insert the spoon, lowering the disc to the top of the jar. The sulphur at once begins to burn with a much brighter, more luminous flame, and a suffocating gas—sulphur dioxide, SO_2—is produced. Remove the spoon when the flame seems to be dying out, and add a little water. Shake vigorously to dissolve the gas. We have now a solution containing sulphurous acid, which will turn blue litmus red.

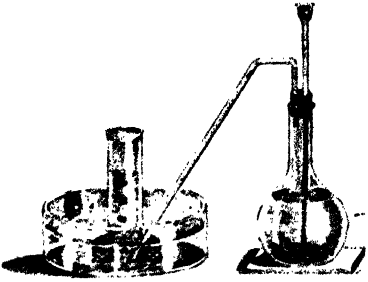
Exp. 3.—Fill a small glass dish with water, and, using a pair of tongs, place in it a piece of ordinary phosphorus. Cut off a small piece of the size of a pea, and rapidly dry it between two pieces of filter (*Great care must be exercised in handling as it ignites with the warmth of the hand; it should always be kept under water*). Place the small piece of phosphorus on a deflagrating spoon, touch it with a hot wire to ignite it, and rapidly remove the plate from the second jar of oxygen and insert the spoon. Very vivid combustion ensues, the phosphorus combining with oxygen, and giving white clouds of the oxides. Remove the spoon, and shake up with water. On adding blue litmus we see that the solution is strongly acid, as in the sulphur experiment.

Exp. 4.—Take a splinter of wood, ignite it and allow to burn for a few seconds, then blow out the flame. The splinter will have a spark on the end. Insert it

Exp. 6.—Take one of the remaining jars of oxygen, rapidly introduce some sand, and replace the lid. Shake the jar so that you have a complete layer of sand on the bottom. Now twist together loosely a few strands of fine iron wire, and attach one end to a cork which will cover the mouth of the jar. Around the other end wrap a small piece of fine string, ignite this in the lamp flame, and rapidly introduce it into the oxygen. The wire burns with bright scintillations, forming "magnetic" oxide of iron, which drops in molten globules upon the sand.

SECTION II.—HYDROGEN.

Exp. 1.—Preparation.—Introduce into a twelve-ounce flask, fitted with funnel and delivery tube as shown in the figure, 10 grammes of zinc, and add 180 c.c. of



dilute sulphuric acid (prepared by previously mixing the concentrated acid with eight times its volume of water). Bubbles of gas will be observed to rise at the surface of the zinc, and the gas passing out of the delivery-tube may be collected in strong glass cylinders as shown. Some cylinders of the gas may be obtained and reserved for an experimental investigation of the properties of hydrogen. When the action has ceased, the clear liquid from the flask may be poured into a porcelain basin, and evaporated until it has been reduced to about 20 c.c. in bulk. On allowing the liquid to cool, crystals of a white salt will be observed to separate out; the zinc has displaced the hydrogen of the sulphuric acid and formed sulphate of zinc.

$$Zn + H_2SO_4 = ZnSO + H_2.$$

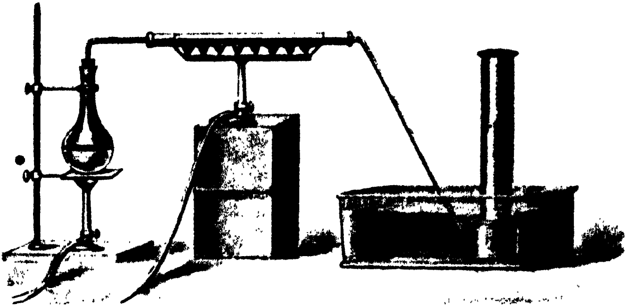
Properties of Hydrogen.

Exp. 2.—Take the cylinder of gas first collected, and holding it mouth downwards, apply a light—the hydrogen will burn instantaneously throughout the vessel with explosion, owing to its being intimately mixed with air carried over from the flask in which the hydrogen was generated. The second cylinder, treated in the same way, will probably be found to burn quietly, as it contains very little air. Notice that in this instance the hydrogen burns only where it has access to air, namely, at the mouth of the cylinder; also that a lighted taper pushed up into the cylinder whilst the hydrogen is burning, will be extinguished. We see, therefore, that hydrogen burns where it comes into contact with the air, but will not support the combustion of a taper.

Exp. 3.—Take a *dry* cylinder, somewhat smaller than those used for collecting the gas, and holding it mouth downwards transfer the hydrogen from one of the cylinders into it by pouring upwards. The hydrogen will rise in the dry cylinder and displace the air from it. Now apply a light to the mouth of this cylinder and there will be a slight explosion, owing to a small admixture of air during the transference, whilst the gas will burn and moisture appear on the sides of the cylinder. We learn from this experiment that hydrogen is lighter than air, and that, when it burns, water is formed. The burning consists in the combination of hydrogen with the oxygen of the air.

Reserve a jar of hydrogen for an experiment to be described later, with its mouth under water, as described under oxygen.

Exp. 4.—Hydrogen can also be prepared by passing steam over red-hot iron. The oxygen of the steam combines with the iron and the hydrogen is liberated.



Fit up the apparatus shown in the figure. It consists of a flask in which water can be boiled to produce steam. This flask is fitted with a cork through which passes a delivery tube leading to a porcelain or hard glass tube containing iron filings. The other end of the porcelain tube is also fitted with a cork and bent tube leading to a pneumatic trough for collecting the hydrogen. After the apparatus is arranged, light the Bunsen burner under the porcelain tube and wait until the iron has become red-hot, next heat the water and allow the steam to pass over the iron,—hydrogen will be evolved, and a jar of it can be collected at the pneumatic trough.

SECTION III.—WATER.

Exp. 1.—Take a strong soda-water bottle and fit it with a good tight cork. Graduate it in the following manner:—fill it with water, and pour into a measuring cylinder; read off the volume of the water, and take two-thirds of it and pour back into the bottle; carefully mark the level of the liquid with a slight file scratch, holding the bottle vertical. Now fill up the bottle, and inverting it in the pneumatic trough, carefully bring under its mouth the jar of hydrogen. By carefully tilting the jar, hydrogen can be passed into the bottle, bubble by bubble. When the bottle is two-thirds full of hydrogen (down to the file mark) fill up the remainder with oxygen in the same manner, and replace the cork whilst the mouth is still under water. We have a mixture of two volumes of hydrogen with one of oxygen. Take the bottle, carefully wrap it in a towel, and light a taper. Hold the bottle in one hand, remove the cork with the other, and get someone to bring the light immediately to the mouth of the bottle. There will be a loud report, due to the combination of the hydrogen with the oxygen. Water will be formed, but its formation here is not evident because the bottle was already wet. If we had taken *dry* gases and placed them in a dry bottle, on igniting, water would have been seen as a dew on the sides of the vessel.

Exp. 2.—The apparatus shown in Fig cept the wires bearing strips of foil fused into each limb of the U-tube near the bend: these are of platinum, and are called the electrodes. The apparatus is filled up to the bulb with water containing a little sulphuric acid, and each electrode is connected by a copper wire with a pole of a battery of four Bunsen's or Groves's cells. As soon as the connection is made, gas is seen to rise from the electrodes, and to collect in the tubes. The volume of gas collected in one tube will be observed to be twice as great (or rather more, owing to the greater solubility of oxygen in

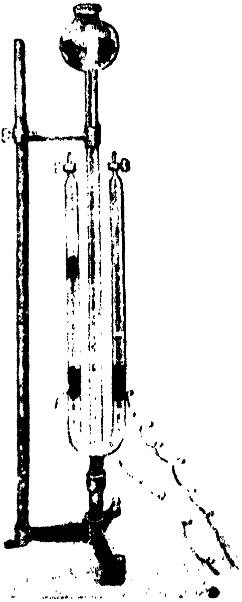


Fig. 5.

water) as that in the other, and on examination it will be found that this gas is inflammable, and shows the properties of hydrogen, whilst the other will prove to be oxygen.

Exp. 3.—With a clean knife cut a small piece of sodium and place it in the pneumatic trough, holding it down with a wire net below the surface of the water and under a gas jar filled with water. Hydrogen will be given off and will rise in bubbles into the jar.

$$2Na + 2H_2O = 2NaOH + H_2.$$

Exp. 4.—Another experiment by which water may be shown to be a compound of oxygen and hydrogen, is to pass dry hydrogen over heated copper oxide. The hydrogen unites with the oxygen of the oxide forming water, and metallic copper is left behind. Fit up a Woulff's bottle with a funnel and delivery tube as shown in the figure. Place about twenty grs. of zinc in it and make the connections with the wash bottles and U-tube. The first bottle should contain permanganate, the second concentrated

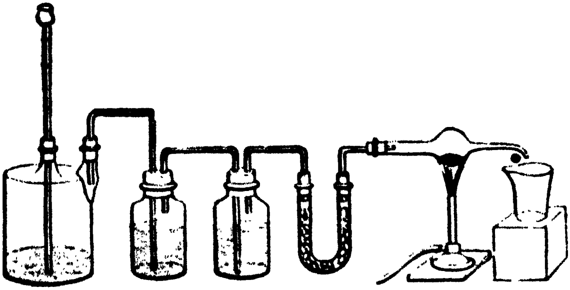


Fig. 6.

sulphuric acid, and the U-tube calcium chloride. Connect the U-tube with a bulb tube of hard glass containing a few grains of black oxide of copper. Light the Bunsen burner under the bulb tube, and when the copper oxide has become hot, pour dilute sulphuric acid down the funnel of the Woulff's bottle from time to time to keep up a steady evolution of hydrogen. In a short time water will be found in the end tube, which, condensing into drops, may be collected in a small beaker placed to receive it.

Exp. 5.—When hydrogen is burnt in oxygen (or air) the gases combine and form water. To show this, take the hydrogen apparatus used above, replace the long delivery tube by a shorter one leading to a calcium chloride tube, the end of which is drawn out to a point as shown in the figure. Place some granulated zinc (about 10 grms.) in the flask, add a little dilute sulphuric acid,—a gentle effervescence of hydrogen will take place, and the air will be driven out. Before we light the hydrogen we must be sure that all the air has been expelled from the apparatus. We can

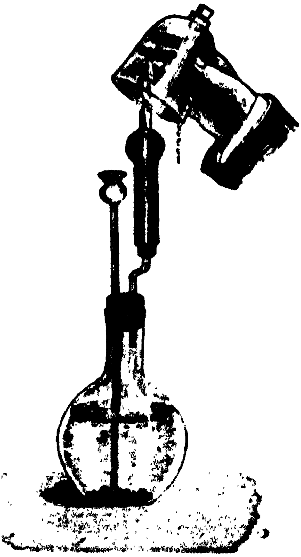


Fig. 7.

tell when this is the case by holding a test tube over the jet (which should be pushed well to the top of the tube) for a few minutes, then raising it gradually, and when clear of the jet rapidly bringing the thumb over its mouth. On bringing the mouth of the test tube to a flame the gas should burn quietly, and not with a sharp explosion. When we have got rid of the air, and not until then, we can safely light the hydrogen issuing from the jet. It will be seen to burn with a pale bluish flame, scarcely luminous. Now bring over the jet a dry gas jar for a few minutes. Moisture will be condensed on the walls of the vessel, showing that water has been produced.

SECTION IV.—CHLORINE.

Preparation.

Exp. 1.—Place a little black oxide of manganese in a test tube, add a few c.c. of concentrated hydrochloric acid, and warm gently. Notice that the air in the tube is replaced by a greenish-yellow gas of a pungent, irritating odour. Take a piece of litmus paper, moisten, and introduce it into the mouth of the tube. The chlorine will gradually bleach it.

Exp. 2.—On account of the disagreeable effects resulting from breathing chlorine, large quantities of the gas should always be prepared in the draught closet. Fit up the apparatus depicted on page 29; into the flask introduce about 40 grammes of manganese dioxide (in the form of lumps), and about 150 c.c. of strong commercial hydrochloric acid. In the smaller flask, which will serve as a wash-bottle for the gas, place concentrated sulphuric acid so that there is about half an inch of it above the end of the tube. Place the large flask on a sand bath and warm gently,—a current of gas will pass through the wash-bottle by the delivery tube, and into the jar. The latter will gradually fill with the yellow gas, for the chlorine being heavier than air sinks to the bottom of the jar and displaces the air. This mode of gases is known as "*downward displacement*." This cannot be collected by the pneumatic trough it is very soluble in water. Fill six jars with chlorine in this manner.

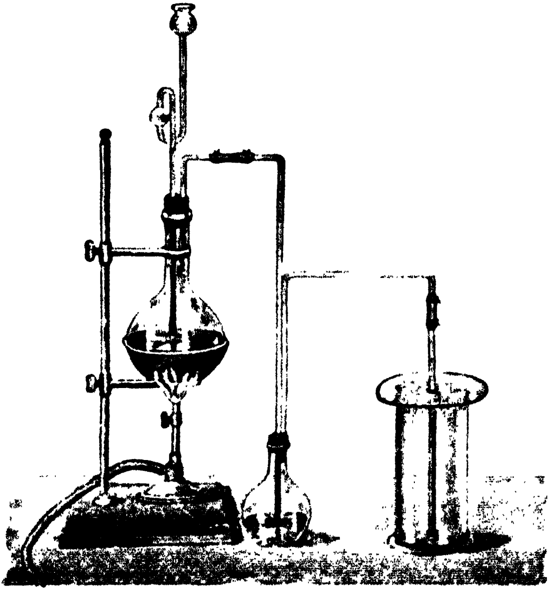


Fig. 8.

Properties of Chlorine.

Exp. 3.—Light a taper, and introduce it into a jar of the gas. The taper still burns, but with a changed flame. The colour becomes redder, and volumes of sooty smoke are evolved. The chlorine combines with the hydrogen in the wax, but not with its carbon, which is consequently set free as soot.

Exp. 4.—Throw a few grains of powdered antimony into another jar. The metal combines with a flash, giving white antimony chloride.

Exp. 5.—Using the same jar of gas, take a piece of sodium and heat it on a deflagrating spoon until it takes fire, then place it in the chlorine,—it will burn vigorously, giving white deposits of sodium chloride.

Exp. 6.—Invert a jar of chlorine in a trough of water. The water will gradually rise, showing that it dissolves the gas.

Exp. 7.—Fill a Cowper's tube with chlorine water (see figure) and expose to direct sunlight; bubbles of gas will be seen to rise in the liquid. When sufficient gas has collected it may be tested with a glowing splinter, and will be found to be oxygen.

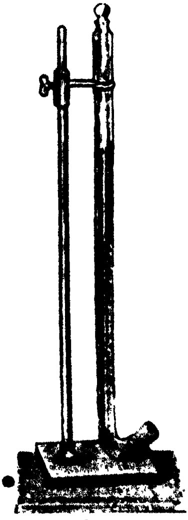


Fig. 9.

The change which has taken place is represented by the equation—

$$2Cl_2 + 2H_2O = 4HCl + O_2$$

Exp. 8.—Pass chlorine for some minutes through about 50 c.c. of water, and to about 20 c.c. of this add a solution of sulphuretted hydrogen; hydrochloric acid is formed, the liquid becoming turbid owing to the separation of sulphur, according to the equation—

$$Cl_2 + H_2S = 2\,HCl + S.$$

Exp. 9.—Introduce a lighted jet of hydrogen into a jar of chlorine. It continues to burn with the production of fumes of hydrochloric acid. The fumes become denser if a drop of ammonia liquor be brought to the mouth of the jar.

Exp. 10.—Place a piece of dry cloth dyed with turkey-red in a stoppered jar of dry chlorine, and leave it some minutes; no decolourization will occur. If the cloth be now moistened and replaced in the jar it will be bleached.

Exp. 11.—Place a printed label, smeared with writing ink, in a jar of moist chlorine; in a little while the ink stains will disappear whilst the printing ink will be unaffected.

Exp. 12.—Make some starch-iodide paste as follows. Place a few crystals of potassium iodide in 50 c.c. of water in a beaker, and heat until the water boils. Crush a small piece of starch in a porcelain basin, make it into a thin paste with cold water, and add with stirring to the boiling liquid mentioned above. When cold take some narrow strips of filter paper and dip into the liquid, drain, and allow to dry; these can be kept for later experiments. Take one of the wet papers and put it into a jar containing chlorine. A dark colouration is produced, showing that iodine has been liberated.

$$2\,KI + Cl_2 = 2\,KCl + I_2.$$

Section V.—Hydrochloric Acid.

Preparation.

Exp. 1.—Place about two grs. of common salt in a test tube, and add a few c.c. of concentrated sulphuric acid. Warm gently, and note the white fumes given off and their irritating smell. The gas evolved is hydrochloric acid, which fumes in moist air.

Exp 2.—Set up a similar piece of apparatus to that used in making chlorine, and place about 50 grammes of common salt (in lumps) in the large flask. (These can be made by sawing a block of salt into pieces of about a cubic inch volume.) In the wash-bottle place a little water. Take 60 c.c. of conc. sulphuric acid and stir it gradually into an equal bulk of water. When cool add gradually to the salt, pouring it through the funnel. The gas is evolved and being heavier than air, can be collected by downward displacement, just as in the case of chlorine. Fill two jars, and the bolt-head to be used in Exp. 5, ascertaining when they are full by means of a lighted match, which will be extinguished. It will be found advisable to collect the gas in the draught chamber. The flask containing the salt and acid must be gently heated if the gas does not come off rapidly enough. When the vessels are filled, the gas can be passed into about 100 c.c. of distilled water, until the bubbles no longer disappear as they pass into the liquid. The apparatus can then be disconnected and the large generating flask filled up with water.

Properties of Hydrochloric Acid.

Exp. 3.—Fix a small piece of candle on a deflagrating spoon, light it, and carefully lower it into a jar of hydrochloric acid. It will be immediately extinguished, proving that the gas does not support combustion; nor does the gas itself ignite.

Exp. 4.—Into the same jar pour a few drops of blue litmus. The colour will be changed to red, indicating the acid nature of the gas.

Exp. 5.—To show the solubility of hydrochloric acid, fill a large bolt-head, of at least two or three litres content, with the gas by displacement. Fit it with an india-rubber cork, through which passes a tube with stop-cock, and drawn out into a jet as shown. Dip the extreme end of the tube into water and open the

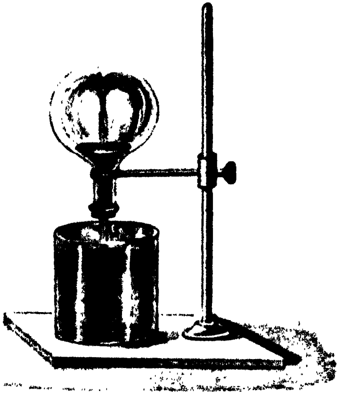


Fig. 10.

Under these conditions the gas at first comes into contact with the water very slowly by a process of diffusion, and it is desirable to bring about contact with a larger surface of water by cooling the flask; this may be done by pouring a few drops of ether over its surface. When the jet once comes into play, the water continues to rise into the flask until all the hydrochloric acid is absorbed.

Exp. 6.—To a few c.c. of the strong solution of hydrochloric acid add a few pieces of zinc. Brisk effervescence takes place. If a light is brought to the mouth of the tube there is a sharp explosion. On evaporating the liquid to dryness a white solid is obtained,—zinc chloride. The gas evolved is hydrogen. $Zn + 2HCl = ZnCl_2 + H_2$.

Exp. 7.—Take a few drops of the solution of hydrochloric acid made when preparing the gas, dilute with ten or twelve times its bulk of distilled water, and add a few c.c. of silver nitrate solution. A white curdy preparation of silver chloride will be produced. Divide it into two portions, and boil one with strong nitric acid. It will remain unaltered. To the other add strong ammonia solution. The precipitate will be dissolved. $AgNO_3 + HCl = AgCl + HNO_3$.

SECTION VI.—NITROGEN.

Preparation.

Exp. 1.—Our chief source of nitrogen is the air, which contains nitrogen and oxygen with a little argon. By removing the oxygen we obtain a mixture of nitrogen with the small amount of argon. Float a small porcelain crucible containing red phosphorus, on water, and place a large bell-jar with a narrow neck (Fig. 11) over it, so that the depth of the water is about one-third the height of the bell-jar. Now ignite the phosphorus by touching it with a hot wire, and close the bell-jar by means of a cork or stopper. The phosphorus will burn brightly at first, and the heat evolved will expand the gas and depress the water inside the jar. After a little time the combustion will cease, and the water will ultimately rise above its original level. The fine white powder which is formed during the combustion, consisting of phosphorus pentoxide, will gradually settle down, and dissolve in the water. When the water has ceased to rise within the jar, pour more water into the vessel in which it stands until the levels

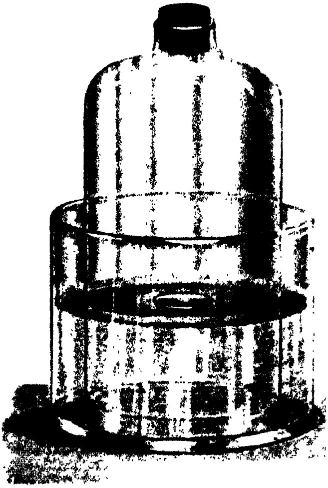


Fig. 11.

inside and outside are the same. The gas has diminished in volume and altered in character. It will be found to extinguish a lighted taper, and to be quite inert towards chemical reagents. When air is deprived of its oxygen, the residual gas, which we have now in the jar, is nitrogen.

$$5(4N_2, O_2) + P_4 = P_4O_{10} + 20N_2.$$

Exp. 2.—Take a glass tube about 700 m.m. high and 15 m.m. diameter, closed at one end, and of as even bore as possible. Invert this, filled with air, over water, note the volume of the air, and pass up into it a piece of phosphorus attached to a stout copper wire. The phosphorus will slowly combine with the oxygen of the air, and the water will rise in the tube. Allow it to stand in a shaded place until the water ceases to rise. Now remove the phosphorus, and adjust the level of the water to the same height inside and outside the tube, and note the volume of residual nitrogen. The volume (correction may have to be made for variation of temperature and pressure during the experiment) occupied by the original air and the residual nitrogen may be ascertained with tolerable accuracy by seeing what volume of water is required to fill the tube to the two levels noted.

Exp. 3.—Connect a gas holder containing air with a small wash bottle containing conc sulphuric acid, and provided with a delivery tube bent once at right angles (Fig. 12). Now take a hard glass tube of about 18 in. length, fill it with copper turnings, which should be held in their place by two loose plugs of asbestos fixed at about 3 in. from each end. Fit with two corks and into one introduce the delivery tube from the wash-bottle. Into the other fit a delivery tube, which should dip into a trough of water. Heat the copper turnings to redness by means of a long Bunsen burner, place a jar of water over the end of the delivery tube. The gas which is expelled from the gas holder, will, if the corks are tight and well

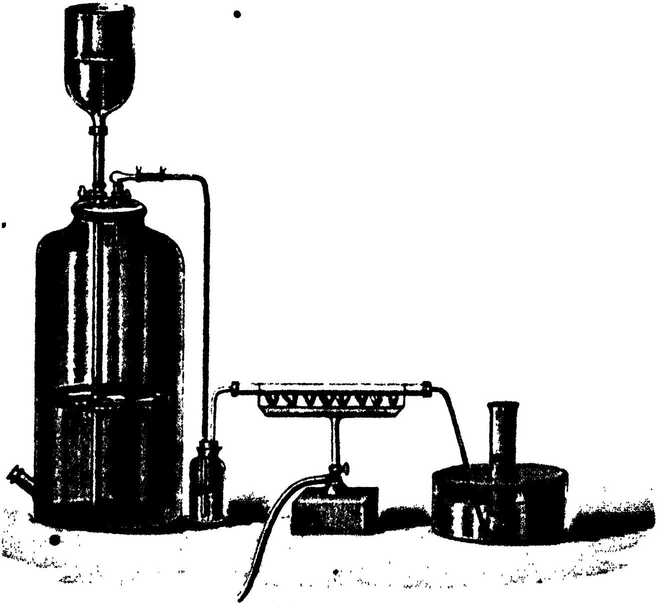


Fig. 12.

bored, pass through the drying bottle, over the copper and be there deprived of its oxygen. Note that the nitrogen is colourless, practically insoluble in water, without taste or smell, and a non-supporter of combustion. Shake up another jar containing nitrogen, with a little lime-water, and observe that no turbidity is

Properties of Nitrogen.

Exp. 4.—Take a piece of hard glass tubing about a yard long and $\frac{3}{8}$ in. diameter, seal off one end in the blowpipe flame and bend it at about 3 in. from that end so as to make an acute angle. Fill it with mercury, and, using a small basin or mortar for a pneumatic trough, invert it and fill with nitrogen from the apparatus described under the previous experiment. Close the open end with a tight cork and remove from the trough. Cut off a strip of magnesium ribbon about 6 in. long, scrape it to remove oxide, coil it into a small narrow loop, and drop it into the end of the tube, quickly replacing the cork. Fix the tube in a vertical position with the corked end under mercury, and heat the magnesium at the other end, first gently and gradually more strongly. Magnesium is one of the few elements with which nitrogen can combine, so on allowing to cool, and then opening the corked end, the mercury will rush in to take the place of the gas now combined with the magnesium as nitride.

SECTION VII.—AMMONIA.

Preparation.

Exp. 1.—Heat a little ammonium chloride and quick-lime together in a test tube, after having powdered them. Notice the pungent smell of the gas which is evolved, and that a piece of moist red litmus paper is turned blue by it.

Exp. 2.—Fit up the piece of apparatus shown below. Powder 20 grammes of ammonium chloride and about 50 grammes of quick-lime. Mix about 30 grammes of the lime with the ammonium chloride intimately and introduce the mixture into the flask. Place the remainder of the lime on the top of this so as to form a drying layer. In the U-tube place pieces of fresh quicklime to further dry the gas. (The ordinary drying agents, sulphuric acid and calcium chloride, are inadmissible in this case because they combine with ammonia.) The jars used for storing the gas should be carefully dried before experimenting. The gas comes off on gently heating the sand bath on which the flask is placed, and being

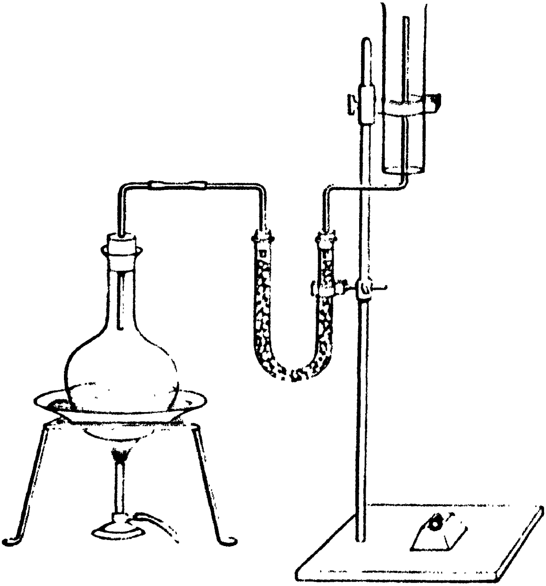


Fig. 13.

lighter than air, rises to the top of the jar. This method of collecting gases is spoken of as *upward displacement.* Several jars can be filled, and covered with greased glass plates.

$$2\,NH_4Cl + CaO = CaCl_2 + H_2O + 2\,NH_3.$$

Properties of Ammonia.

Exp. 3.—Invert one of the jars in a trough full of water which has been coloured with a little red litmus. The gas will be absorbed rapidly, the water rushing into the jar, and the red litmus will be turned blue.

Exp. 4.—Introduce a lighted taper into another jar of the gas. Where the flame meets the ammonia and air together there will be a pale greenish mantle, showing that ammonia is combustible.

Exp. 5.—Place some strong ammonia solution in a small flask and bubble a rapid stream of oxygen through it from a gas-holder. Bring to the mouth of the flask a lighted taper. There will be no doubt in this case as to the combustibility of the ammonia. It may be found necessary to warm the flask slightly to get a good evolution of gas, but with the strongest solution of ammonia it works quite well in the cold.

Exp. 6.—Fill two similar jars by displacement with ammonia and hydrochloric acid gas respectively, and cover the mouth of each jar with a glass plate. Now bring them mouth to mouth and withdraw the glass plates. The gases, as they come into contact, will combine, forming a fine white powder, which remains for some time diffused throughout the jars. This substance is ammonium chloride, NH_4Cl.

$$NH_3 + HCl = NH_4Cl.$$

Section VIII.—Nitric Acid.

Preparation.

Exp. 1.—Introduce 20 grammes of potassium nitrate into a stoppered retort, and as much concentrated sulphuric acid as will just cover it. Apply a moderate heat, and presently the vapour of nitric acid will pass over and condense in the neck of the retort. The liquid may be collected in a small flask slipped over the mouth of the retort, and kept cool by means of a stream of cold water, or a wet cloth. When about 10 c.c. have condensed, or so soon as whitish fumes of sulphuric acid begin to appear, the experiment should be stopped.

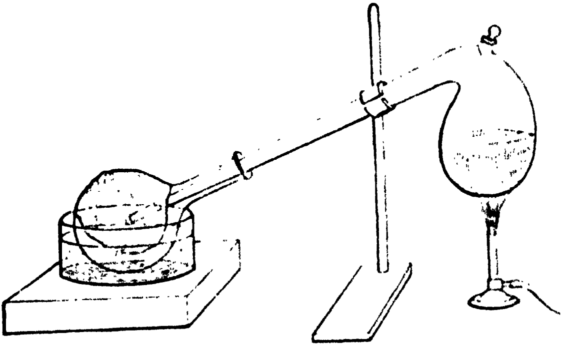


Fig. 14.

The reaction is represented by the equation

$$\underset{\text{Potassium nitrate.}}{KNO_3} + H_2SO_4 = \underset{\text{Acid potassium sulphate.}}{KHSO_4} + HNO_3.$$

Care must be taken that the mixture of nitrate and sulphuric acid does not cake to the glass. A piece of wire gauze would be useful to prevent this. Place it on a tripod so that it is just in contact with the glass of the retort, and shake the contents from time to time. If fumes begin to appear, remove the burner from the retort—you are probably getting more nitric acid off than you can conveniently condense. Observe that the liquid is of a yellowish colour (due to small quantities of oxides of nitrogen which are dissolved in it), and fumes in air. When pure it is colourless.

Properties of Nitric Acid.

Exp. 2.—Dry some sawdust thoroughly in a small basin before a fire. Take about 20 c.c. of fuming nitric acid and stir it into the middle of the sawdust. The nitric acid will rapidly oxidize the wood and the whole will become so hot ultimately that it will burst into flame. Suitable precautions should be taken to prevent any inflammable material coming into contact with the burning mass. A wet towel is an effectual extinguisher.

Exp. 3.—Place about 20 c.c. of fuming nitric acid in a small round-bottomed flask and warm it gently in the draught-chamber over a tripod and gauze. Fasten a long pencil of wood charcoal to a copper wire, heat the pointed end in the basin until it glows strongly, and then introduce it into the acid. It will continue to burn vigorously, taking oxygen from the acid and causing it to evolve copious fumes of oxides of nitrogen.

$$2\,HNO_3 + C = H_2O + NO_2 + NO + CO_2.$$

Exp. 4.—To a few c.c. of indigo solution contained in a test tube add a few c.c. of concentrated nitric acid.

The acid oxidizes the indigo to a substance called isatin. The blue colour is consequently bleached to a brownish yellow.

Exp. 5.—Place a few pieces of granulated tin in a small porcelain crucible in the draught-chamber, warm slightly, and pour on them a few drops of concentrated nitric acid. Ruddy fumes will be evolved and the bright metal converted into a white powder, which is a compound of oxide of tin with water. The tin takes oxygen and water from the nitric acid and the nitrogen is evolved combined with oxygen.

Exp. 6.—Take a piece of soft glass tubing of about [illegible] in. diameter and 18 in. long. Blow a bulb on one end, using the gas blow-pipe if necessary, bend the tube twice at right angles, so that while the bulb is directed downwards, the opposite end is directed upwards. Allow a few c.c. of nitric acid to run into the bulb, and place a dry, clean gas jar over the other end. Now heat the horizontal part of the tube by means of a long burner, gently at first, and then more strongly. When it is fairly hot, warm the bulb carefully so as to drive a slow current of nitric acid vapour through the hot part of the tube. It will be there broken up into oxides of nitrogen, water vapour, and oxygen. Place a piece of white paper behind the gas jar,—the contents will gradually assume a ruddy tint, due to the presence of nitrogen peroxide.

$$4\,HNO_3 = 2\,H_2O + 4\,NO_2 + O_2.$$

Exp. 7.—Take a few c.c. of a solution of potassium nitrate in a test tube and add about an equal volume of ferrous sulphate solution. Mix well and cool. Now pour carefully down the side of the tube a few c.c. of concentrated sulphuric acid. The heavy acid will sink to the bottom of the tube, forming an oily layer, and where it comes in contact with the upper liquid there will be a dark brown ring. The sulphuric acid reacts with the nitrate, liberating free nitric acid, which is reduced by the ferrous sulphate with formation of nitric oxide. This again combines with more ferrous sulphate to form a dark brown compound, which is seen in the ring. Being unstable it is destroyed on warming. This is a very delicate test for nitric acid and nitrates.

Exp. 8.—To a few c.c. of concentrated solution of potassium nitrate add a few bright pieces of copper, and warm gently. Nothing happens. Now add carefully a few c.c. of strong sulphuric acid. This liberates nitric acid, which at once begins to attack the copper, forming a green solution of copper nitrate and liberating brown fumes of nitric peroxide. (Nitric oxide which is colourless, is first formed, but it combines with the oxygen of the air to form nitric peroxide.)

$$3\,Cu + 8\,HNO_3 = 3\,Cu(NO_3)_2 + 2\,NO + 4\,H_2O.$$

Exp. 9.—To a few cubic centimetres of *dilute* nitric acid in a porcelain basin add fragments of lead, and digest on a water-bath until the acid is saturated, and no more of the metal will dissolve. Evaporate the clear liquid to dryness, a white salt, nitrate of lead, remains.

Exp. 10.—Dilute 5 c.c. of nitric acid with an equal bulk of water and add a little litmus solution, which will become of a bright red colour. Now add ammonia solution, little by little, until the last drop turns the litmus blue, and concentrate the liquid to a point at which, when a drop of it is allowed to cool on the end of a glass rod, it crystallizes. On standing, crystals of ammonium nitrate will be obtained.

Exp. 11.—Heat a few grammes of potassium nitrate in a test-tube until it fuses, and then drop into it one or two fragments of dry charcoal. The charcoal will ignite and burn with violence, being oxidized by the nitrate to CO_2.

Exp. 12.—Repeat the experiment, introducing a few small shavings of lead; substituting shavings of lead for the charcoal, the lead will be oxidized at the expense of the nitrate and transformed into a yellowish powder of oxide of lead.

SECTION IX.—NITROUS OXIDE.

Exp. 1.—Preparation.—Fit up a small, round-bottomed flask with cork and delivery tube, as shown in Fig.

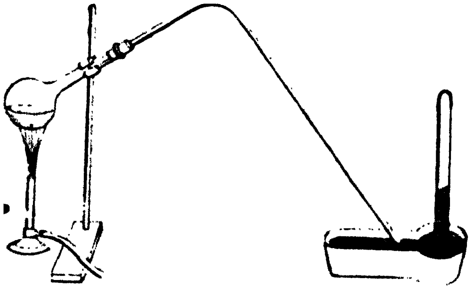


Fig. 15.

15. Place in the flask about 30 grammes of ammonium nitrate, replace the cork and tube, and let the end of the latter dip into the pneumatic trough. As the gas, though fairly soluble in cold water, is almost insoluble in hot water, the trough and collecting jars must be filled with the latter, or, better still, with mercury, as

shown in the figure. Warm the nitrate gently to expel the moisture, and then more strongly so as to fuse it. Bubbles of gas will rise through the melted mass and expel the air. When you think the air has been expelled, bring a jar over the end of the delivery tube. A *gentle* evolution of gas must be kept up by using only a moderate temperature. If heated too strongly the nitrate will decompose with explosive violence. Collect several jars of the gas, and then raise the end of the tube out of the liquid in the trough, so that, on cooling, the liquid will not be sucked back into the still heated flask.

$$NH_4NO_3 = 2\,H_2O + N_2O.$$

Properties of Nitrous Oxide.

Exp. 2.—Take a jar of the gas—notice that it is colourless and of slightly sweetish taste and smell. Light a wooden spill and thrust it into the gas, observing the change in the character of the flame. The gas itself does not burn, but the spill burns with a much brighter flame than in air.

Exp. 3.—Place a small piece of sulphur in the bowl of a deflagrating spoon and ignite it by inserting it for a moment into the flame of the Bunsen. While it is only feebly burning, depress it into a jar of nitrous oxide. The flame will be extinguished. Withdraw the spoon, heat the sulphur until it burns with a brighter flame, and replace it in the gas. The flame will become much brighter. Shake up the contents of the jar with water when the combustion has ceased, and test the liquid with a few drops of blue litmus. It will be turned red, showing that the sulphur has formed an acid substance. It is the same compound that was produced by the burning of sulphur in oxygen. The oxygen in the nitrous oxide has combined with the sulphur, but as it was previously in combination with nitrogen, additional energy was required in this case to decompose the oxide, hence the need of a brightly burning flame to begin with.

$$2\,N_2O + S = SO_2 + 2\,N_2.$$

p. 4.—Before filling one of the jars, fit it with a tight cork. Heat the end of the rod of a deflagrating spoon in the Bunsen flame until it is almost red hot, and push it through the cork from the inside until the bowl will just be opposite the middle of the jar. Fill the jar with gas, rapidly slide the cover off, and introduce the spoon, containing a few small pieces of ignited phosphorus, and push the cork well in. Notice that the phosphorus burns almost as brightly as in oxygen, and with the production of dense white fumes. Light a taper and, removing the cork and spoon, lower it into the residual gas in the jar. The taper is extinguished, the gas being nitrogen. Shake up the white fumes with water, and test, as in the previous experiment, with a few drops of blue litmus. A red colour will be produced,—the white fumes being oxides of phosphorus, which combine with water forming the corresponding acids.

$$P_4 + 10\,N_2O = P_4O_{10} + 10\,N_2;$$
$$P_4O_{10} + 2\,H_2O = 2\,H_2P_2O_6.$$

Exp. 5.—Fill a large dish with cold water, and after inverting one of the jars of gas with its mouth under water, slide away the glass plate and tilt the jar so that a few bubbles of gas escape and are replaced by the cold fluid. Insert your hand under water and cover the mouth of the vessel by firmly pressing it. Remove from the dish and shake vigorously; the water present will dissolve some of the gas and cause a diminution of pressure, shown by the suction on your hand. Still keeping your hand tightly fixed over the mouth of the jar, bring it under water again, and allow more water to be drawn in, taking care, however, that no air is drawn in at the same time. Replace your hand and shake up again. By repeating the process described, if the gas is pure, it can be completely dissolved and its place taken by the water. This experiment further emphasises the need for using hot water instead of cold in collecting the gas.

Exp. 6.—Take a jar of air and place it with its mouth open and turned upwards. Take, again, a jar of nitrous oxide and, sliding off its cover, pour out the gas from it into the jar of air, as though you were pouring a liquid. Test the jar which originally contained air with a glowing splinter. It will be re-ignited, showing that the air has been displaced by the heavier nitrous oxide. Test the jar which originally contained the nitrous oxide in the same manner. It will be found to have no effect on the splinter—being now full of air. Nitrous oxide is about one and a half times as heavy as air.

SECTION X.—NITRIC OXIDE.

Exp. 1.—**Preparation.**—Take the apparatus used in making hydrogen (see Fig. 16) and introduce into the flask about 20 grammes of small pieces of copper. Allow the end of the delivery tube to dip under the water in the trough, and pour just sufficient water through the funnel to conveniently cover the end of it. If your apparatus is quite tight bubbles of air will be forced out of the delivery tube. Now mix about 50 c.c. of concentrated nitric acid with an equal bulk of water, and pour a little of the mixture through the funnel. The acid and the copper will react, and bubbles of gas will be given off, the solution turning green from formation of copper

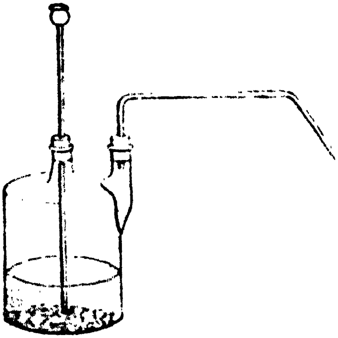


Fig. 16.

Red fumes will at first be seen in the flask. When these have disappeared, place a jar full of water (which need not be warm) over the end of the delivery tube, and fill it with gas. Collect several jars, and cover them with wet glass plates in the usual way.

If the flask gets hot, cool it by pouring cold water on the outside. If gas does not come off rapidly enough, add more acid. The reaction is represented by the following equation:

$$3\,Cu + 8\,HNO_3 = 3\,Cu(NO_3)_2 + 2\,NO + 4\,H_2O.$$

Properties of Nitric Oxide.

Exp. 2.—Fix a small piece of candle to the bowl of a deflagrating spoon by heating the latter slightly in the flame of the burner and holding the candle on it until it is set. Light it, and, after removing the cover, lower it into the jar of nitric oxide. It will be immediately extinguished. Notice that red fumes are produced near the mouth of the jar on removing the plate. Nitric oxide evidently does not itself burn, nor does it support the combustion of a candle.

Exp. 3.—Place a piece of sulphur in the bowl of a deflagrating spoon and heat it fairly strongly in the flame until it burns brightly. Lower it into a jar of the gas. It will be at once extinguished. Nitric oxide does not give up its oxygen even to *brightly* burning sulphur. (Compare the behaviour of nitrous oxide in this respect.)

Exp. 4.—Cut off a piece of yellow phosphorus of the size of a pea, dry it quickly with a piece of filter paper, place it in a deflagrating spoon, and touch it with a hot wire. As soon as it begins to burn lower it into a jar of the gas. It will be extinguished. Remove it rapidly and replace the glass plate. Ignite it as before, but allow it to attain to vigorous combustion this time before inserting it into the jar. It will now burn with a much brighter flame than in air. Nitric oxide is a more stable compound than nitrous oxide; hence a higher temperature is required to decompose it. The white fumes observed are due to oxides of phosphorus. Dissolve them by shaking up with water, and test the solution with a drop or two of blue litmus. The liquid has a strong acid reaction.

Exp. 5.—For this experiment you will need a small supply of oxygen. Take the apparatus you used in preparing oxygen (Section I. Exp. 1), and charge it with about 10 grammes of the mixture of potassium chlorate and manganese dioxide. Heat it until all the air has been expelled by oxygen, and then fill a jar with it by displacement of water. Invert one of the jars of nitric oxide in the trough, remove the glass cover, and allow about a quarter of the gas to escape, taking care that no air enters during the process. Bring the mouth of the jar of oxygen under that of the jar of nitric oxide, and carefully tilting, pass a few bubbles of oxygen into it. Where the two gases come into contact ruddy brown fumes of nitrogen peroxide will be seen. Shake the water round the jar so as to offer as large a surface as possible to the gas, but be careful to prevent any air entering. The fumes will dissolve in the water, the contents of the jar becoming quite colourless again. Pass in a few more bubbles of oxygen and repeat the above process. The nitric oxide will gradually combine with the oxygen, and the product will dissolve in the water. Water will be drawn in to take its place, and if your gases are pure you will ultimately have nothing in the jar but water. By reversing the process, and passing nitric oxide, bubble by bubble, into a measured volume of air over water, we can withdraw the oxygen, and measure the residual nitrogen. This was one of the first methods used in the analysis of air.

$$2\,NO + O_2 = 2\,NO_2;\quad 2\,NO_2 + H_2O = HNO_2 + HNO_3.$$

Exp. 6.—Prepare a concentrated solution of ferrous sulphate, by powdering 10 grammes of the crystals in a small mortar and shaking up in a flask with about

50 c.c. of water. Pour off the clear liquid rapidly into a jar of nitric oxide, and cover the mouth with your hand, which should be wet. Shake up well, and notice that the green solution of sulphate turns dark brown. It has dissolved the nitric oxide, forming the compound which served as a test for nitric acid. (See Section VIII. Exp. 7). Now open the mouth of the jar under water—if the gas was pure, and the ferrous sulphate solution strong, the vessel will immediately fill with water.

Exp. 7.—Pour a few drops of carbon bisulphide into a jar of the gas and rapidly replace the cover. Shake well, so that the liquid exposes a larger surface, and becomes vaporized. Light a taper, remove the glass plate, and bring the light to the mouth of the jar. There will be a brilliant flash, due to the combination of the carbon and sulphur with the oxygen of the gas.

$$6\,NO + CS_2 = CO_2 + 2\,SO_2 + 3\,N_2.$$

Exp. 8.—Pour off the greenish-blue solution of copper nitrate from the flask used in the preparation of the gas, dilute it, and filter it free from particles of copper, carbon, etc. Evaporate it in a porcelain basin over a naked flame, on a tripod, taking care that the flame does not play above the surface of the liquid. When most of the water and nitric acid have been driven off, evaporate to dryness on a water bath. The green solid remaining is copper nitrate. Break off a small piece and heat it in a dry tube. Dense brown fumes of nitric peroxide are driven off, mixed with oxygen, and black copper oxide is left in the tube.

$$2Cu(NO_3)_2 = 2CuO \quad 4NO_2 + O_2.$$

Exp. 9.—Heat a few crystals of potassium nitrate strongly in a dry test tube. First they crackle and fly about, then fuse and give off a colourless gas. Test it with a glowing splinter. It is oxygen. The residual salt is potassium *nitrite*. When cool, dissolve it in a few drops of water, add a drop or two of dilute hydrochloric acid, and insert a starch-iodide paper. The nitrous acid liberates iodine, which colours the paper a dark blue.

$$2KNO_3 = 2KNO_2 + O_2; \quad KNO_2 + HCl = HNO_2 + KCl;$$
$$HNO_2 + KI = KOH + NO + I.$$

SECTION XI.—SULPHUR.

Properties.

Exp. 1.—Take a few pieces of the brassy mineral, iron pyrite; powder it well and introduce it into a narrow tube of hard glass, closed at one end. Heat it gradually until it is red hot. The substance will part with some of its sulphur, which will settle on the walls of the tube as a light yellow deposit. This will melt to a light-coloured liquid on further heating. The residue, even after being strongly heated, still contains sulphur—being a lower sulphide of iron called "magnetic pyrites."

$$3FeS_2 = Fe_3S_4 + S_2.$$

Exp. 2.—Take a round-bottomed flask of about 250 c.c. capacity, clean and dry it. Break up about 20 grammes of roll sulphur in a mortar until the pieces are smaller than peas, and transfer them to the flask. Grasp the neck of the flask in a small clamp or test tube holder, and warm the contents gently over the flame of a Bunsen burner. Note carefully all that takes place. At first there is a slight crackling caused by the uneven expansion of the sulphur, and then the portions near the glass melt to a dark liquid. The flask should be shaken gently to allow the temperature to become equalised, sulphur being a bad conductor of heat, and then heated again. When the solid has just disappeared we shall have a thin, mobile, amber-coloured liquid, at a temperature of about 115°C. Continue heating,—the colour will gradually become darker and the liquid more viscous, until at a temperature of about 220°C, it is of a dark brown colour and of the consistency of treacle, so that the flask may safely be inverted without loss of the contents. On applying more heat the colour does not alter much, but the liquid again becomes more mobile. At 440°C, it boils and gives off a dark orange-coloured vapour, which will probably take fire in the air with a slight report. Allow the flask to cool slightly (to about 350°C), and slowly pour the dark mobile liquid in a thin stream into a large dish of water. Take out the yellow solid and note that it is semi-transparent and plastic. It can be stretched and twisted in the hands like a piece of indiarubber.

Exp. 3.—Heat gently a few pieces of sulphur in a test tube until they are just melted, and then pour as before into water. Take out the solidified mass, and observe that it is yellow, hard, and brittle. In making "plastic" sulphur, it is necessary to heat it to the temperature mentioned above, viz., about 350°C.

Exp. 4.—Take a fairly large clay crucible and fill it with pieces of roll sulphur. Place the crucible on a sand-bath and heat it up gradually until the solid has melted to the thin liquid, at a temperature of about

120° C. Remove the flame from the sand-bath and allow the crucible to cool slowly until a crust has formed on the surface of the still molten liquid. Pierce the crust in two places by means of a sharp iron rod, raise the crucible with a small pair of tongs and pour out the fluid into water, carefully allowing the last drops to drain out. Remove the crust, and notice the shape of the crystals of sulphur in the crucible. They are long and needle-shaped. This is known as the *prismatic* modification.

Exp. 5.—Reduce a few pieces of ordinary roll sulphur to a fine powder in a mortar and introduce it into a test tube. Add about 20 c.c. of carbon bisulphide (take care that you keep it away from the neighbourhood of any flame), keep your thumb over the mouth of the tube and agitate well. Warm the tube gently in a beaker of hot water, shake again, and filter from undissolved sulphur, etc., into a small glass evaporating basin. Cover with a large watch glass, and leave in the draught chamber to evaporate. If the carbon bisulphide has escaped fairly slowly, beautiful transparent crystals of a honey-yellow colour will be left. This form of sulphur is known as *octahedral*.

Exp. 6.—Take about a gramme of "flowers of sulphur" and place it in a dry test tube. Add about 20 c.c. of carbon bisulphide, shake well, and warm gently. You will notice that there is still undissolved sulphur present and that its colour is paler than before. Has *any* dissolved? To ascertain this, filter off the clear liquid after letting the particles of solid settle to the bottom, and allow the solution to evaporate, as before, in the draught chamber. It will deposit yellow crystals, hence some sulphur has been dissolved. Treat the insoluble portion in the same way with more carbon bisulphide. It still refuses to dissolve. This almost white form, insoluble in carbon bisulphide, is known as "*amorphous*" sulphur.

Exp. 7.—Take a small piece of your freshly prepared specimen of "plastic" sulphur, put it in a test tube and pour on to it about 10 c.c. of carbon bisulphide, and warm the mixture by placing the test tube in a beaker of hot water. Filter the liquid on to a dry glass plate and allow it to evaporate in the draught chamber. It will leave practically no residue, as plastic sulphur is almost insoluble in carbon bisulphide.

Exp. 8.—Put aside specimens of your plastic and prismatic sulphur and examine them from time to time. After a few days the plastic form will become hard and brittle, and will lose its transparency. The transparent prismatic crystals will also turn yellow and opaque. Both forms are unstable and pass gradually into octahedral sulphur.

Exp. 9.—Fuse a little "flowers of sulphur" with an equal quantity of sodium carbonate in a small iron or porcelain crucible. Allow to cool, and dissolve the solid mass in a little water. Clean a shilling by first scouring with sand which has been moistened with a few drops of caustic soda, washing, dipping in a little warm dilute nitric acid, and finally rinsing with water. Dip the clean shilling in the solution in the crucible. It will be turned black, becoming coated with silver sulphide. This reaction is used in testing for sulphur.

Exp. 10.—Take the flask you used for melting sulphur in Exp. 2, and place a few pieces of sulphur in it. Bend a piece of stiff copper wire round a glass tube of about ¼ in. diameter, so that it will easily slip into the flask, and bend a piece at right angles so that the spiral will be supported on the mouth of the flask with its end just over the sulphur. Remove the copper and carefully heat the sulphur until the flask is full of dark-red vapour. Now heat the spiral in the Bunsen flame until it is almost red hot, holding it with a pair crucible tongs, and rapidly introduce it into the sulphur vapour. Combination will ensue and the vigour of the reaction will keep the spiral at a red heat. When the copper ceases glowing remove the flame from the flask and take out the wire. It will be found to be almost wholly converted into a black brittle mass of cuprous sulphide, which can be easily broken away from the residual thin thread of copper. This and the following experiment sufficiently demonstrate the affinity of sulphur for the metals. $2Cu+S=Cu_2S$.

Exp. 11.—Mix intimately in a mortar 7 grammes of iron filings and 4 grammes of "flowers of sulphur." The iron and sulphur do not combine, but can be separated by means of a magnet. Now heat the mixture strongly in a clay crucible. The mass will glow brightly when combination takes place. On cooling, a friable, black mass will be seen, on which the magnet has no action. This is ferrous sulphide, and it should be put aside for a future experiment.

$$Fe+S=FeS.$$

SECTION XII.—SULPHURETTED HYDROGEN.

Preparation.

Exp. 1.—Fit up a small hydrogen apparatus as shown in the figure on the next page, and place in it about 10 grammes of granulated zinc. Connect it with a small wash bottle containing a little lead acetate solution. Pour on to the zinc a little dilute sulphuric acid. Gas will bubble through the lead acetate and

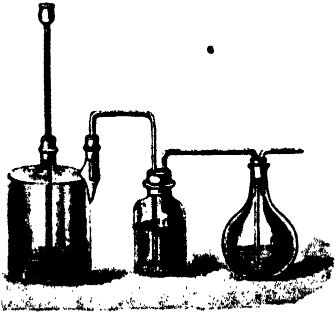


Fig. 17.

be there deprived of any sulphuretted hydrogen it may contain. Now heat a few pieces of roll sulphur in a small round-bottomed flask until it is just below boiling point. Warm the end of the glass tube leading from the wash-bottle, and insert it in the molten sulphur. Hydrogen will bubble through the sulphur and there combine to form sulphuretted hydrogen. This can be tested for by holding a small piece of filter paper, moistened with lead acetate solution, over the mouth of the flask. It will be turned black, owing to the production of lead sulphide.

Exp. 2.—Take a few pieces of the ferrous sulphide prepared in Exp. 11, Section XI., place it in a test tube and add a few c.c. of dilute hydrochloric acid. There is immediate evolution of a gas, which has a peculiar, offensive odour, resembling that of rotten eggs. Test the gas by holding a piece of lead acetate paper over the mouth of the tube. It will be blackened.

$$FeS + 2HCl = FeCl_2 + H_2S.$$

Properties of Sulphuretted Hydrogen.

Exp. 3.—Thoroughly wash out the hydrogen generator and wash-bottle used in Exp. 1, and bring into the large flask several lumps of ferrous sulphide, and into the wash-bottle a little water. Mix about 100 c.c. of concentrated commercial hydrochloric acid with an equal bulk of water, and pour a little of the mixture into the funnel of the generator. Sulphuretted hydrogen will bubble out from the wash-bottle, and several dry gas jars can be filled with it by downward displacement of air. (The preparation of this gas should be effected in the draught chamber.) When the evolution of gas slackens pour in more acid. The jars must be covered with greased plates.

Exp. 4.—Remove the glass cover from a jar of the gas and bring a lighted taper to the mouth; it will burn with a pale blue flame, sulphuretted hydrogen being combustible in air, and a slight yellow deposit of sulphur will be produced on the walls of the jar. Note (by its smell) the presence of the same gas that was formed when sulphur burnt in air. On thrusting the lighted taper down into the gas it should be extinguished, as sulphuretted hydrogen does not support combustion. If it is not, the reason probably is that the air in the jar was not completely displaced by the gas.

$$2\,H_2S + 3O_2 = 2\,H_2O + 2\,SO_2.$$

Exp. 5.—Take one of the jars which have been filled by displacement of air, introduce about 100 c.c. of cold water, and close the mouth firmly with your hand. Shake vigorously. Your hand will be sucked in, indicating diminution of the pressure of the gas in the jar. Open the mouth under water, taking care that no air is drawn in at the same time. Water will rise into the jar to take the place of the gas which has been dissolved. Sulphuretted hydrogen is fairly soluble in cold water, but only slightly so in hot water. To obtain the gas free from air, we collect it over hot water as in the case of nitrous oxide. Pass some of the gas through a little water in a flask and add a few drops of neutral litmus. It will be reddened, indicating the presence of an acid.

Exp. 6.—Replace the delivery tube of the generator by the drying tube and glass jet used in Exp. 5, Section III., keep up a good gas evolution, and when all the air has been driven out of the tube, light the gas. Bring a dry gas jar over the flame and allow it to remain there for a minute or so. Notice that moisture is condensed on the walls of the vessel. This is produced from the hydrogen of the gas, by its union with the oxygen of the air. From Exp. 1 we also know that sulphur is a constituent. Test the moisture in the jar with a piece of neutral litmus paper. It will be reddened by the sulphurous acid dissolved in it.

Exp. 7.—Take five large test tubes. Into the first bring a few crystals of copper sulphate, dissolve in about 50 c.c. of water, and acidify with a few drops of hydrochloric acid. Into the second bring a little powdered white arsenic and dissolve in dilute hydrochloric acid. Place a few crystals of zinc sulphate, ferrous sulphate, and sodium chloride in the third, fourth, and fifth tubes respectively, dissolve each in about 50 c.c. of water, and acidify with a little hydrochloric acid. Now pass a stream of sulphuretted hydrogen through the liquid in each tube. The copper solution will give a dense black precipitate of copper sulphide; the arsenic solution a bright yellow precipitate of arsenious sulphide. In the other tubes there will be no apparent change. To them add a few c.c. of strong ammonia solution. The zinc solution will give a white precipitate of zinc sulphide, the iron a black mass of ferrous sulphide. The sodium chloride solution will still remain unchanged. These facts are used in identifying the different metals, and form part of the basis of qualitative analysis. The sulphides of copper and arsenic are insoluble in dilute acids; those of zinc, iron and sodium are soluble. Zinc and iron sulphides, however, are insoluble in alkalies, while sodium sulphide is soluble.

$$CuSO_4 + H_2S = CuS + H_2SO_4;$$
$$2\,AsCl_3 + 3\,H_2S = As_2S_3 + 6\,HCl.$$

SECTION XIII.—SULPHUR DIOXIDE.

Preparation.

Exp. 1.—Place a little sulphur on a deflagrating spoon and heat it until it burns with a pale blue flame. Observe the characteristic suffocating smell of sulphur dioxide, and also the peculiar taste it leaves.

$$S + O_2 = SO_2.$$

Exp. 2.—Heat a few bright copper turnings in a test tube, with a few c.c. of concentrated sulphuric acid, until brisk evolution of gas takes place. Observe the smell, taste, etc. of the gas. It is sulphur dioxide. The reaction may be regarded as taking place between the copper and sulphuric acid in the first place, with evolution of hydrogen, the hydrogen then reacting with more sulphuric acid, breaking it up into water and sulphur dioxide.

$$Cu + H_2SO_4 = CuSO_4 + H_2;$$
$$H_2 + H_2SO_4 = 2\,H_2O + SO_2.$$

Exp. 3.—Fit up a small flask (about 500 c.c. volume) with a good cork, thistle funnel and delivery tube, as shown in Fig. 18. Introduce about 50 grammes of copper clippings, and about 100 c.c. of concentrated sulphuric acid. Warm the flask with the Bunsen flame until the gas is evolved briskly, and then turn the flame low. Collect a few jars of the gas by downward displacement of air, sulphur dioxide being heavier than air. The jars should be dry, and

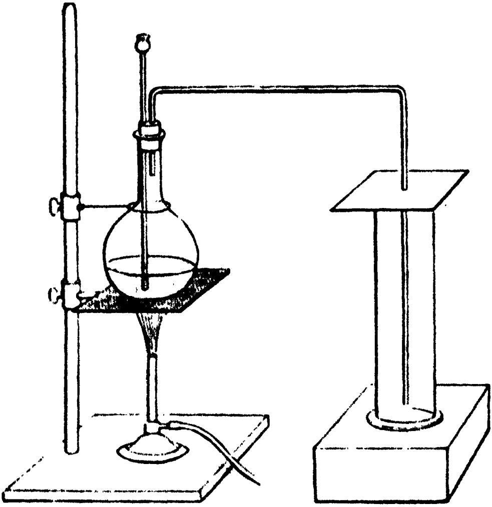


Fig. 18.

covered with greased plates, and the gas should be collected in the draught chamber on account of its unpleasant effects. When the gas has been collected, turn out the burner and leave the apparatus for a future experiment.

Properties of Sulphur Dioxide.

Exp. 4.—Remove the cover from one of the jars of gas, and lower a lighted candle into it. The candle will be immediately extinguished; sulphur dioxide neither burns nor supports combustion.

Exp. 5.—Add a few drops of blue litmus solution to about 50 c.c. of water, remove the plate from a jar of gas, rapidly pour in the coloured water, and close the mouth of the jar tightly with your hand. Shake vigorously. The gas will dissolve in the water, turning the litmus red, and your hand will be sucked into the jar. Open the mouth of the jar under water—it will rapidly fill if the gas was pure. The water contains sulphurous acid in solution.

$$H_2O + SO_2 = H_2SO_3.$$

Exp. 6.—Rose-leaves thrown into a solution of sulphur dioxide are bleached, the colouring matter, however, is not destroyed as when chlorine is used, and the colour may even be restored again by adding a few drops of strong sulphuric acid, or by exposure to air for some time.

Exp. 7.—Liberate iodine from a solution of potassium iodide by adding a *few drops* of chlorine water; now add sulphurous acid, and the brown colour of the iodine will disappear.

$$2\ KI + Cl_2 = 2\ KCl + I_2.$$

$$I_2 + HO_2 + H_2SO_3 = H_2SO_4 + 2\ HI.$$

The sulphurous acid reduces water, and is itself oxidized to sulphuric acid, whilst the hydrogen which is liberated combines with the iodine to form hydriodic acid, which is colourless. The reaction which takes place with chlorine water is explained by the equation.

Exp. 8.—To a solution of potassium chromate add sulphurous acid, the *yellow* colour of the chromate will change to *green* owing to the reduction of CrO_3 to Cr_2O_3, the salts of the former being red or yellow, whilst those of the latter are green. This change from yellow to green affords a means of testing for the presence of sulphur dioxide either in the gaseous state or in solution.

Exp. 9.—Take 50 c.c. of a solution of caustic soda, and pass sulphur dioxide into it till it is saturated with the gas. On allowing the solution to evaporate at ordinary temperatures (it decomposes when heated), or on adding alcohol to it, the *acid salt*, sodium hydrogen sulphite, separates out—

$$NaOH + SO_2 = NaHSO_3.$$

If we add a second 50 c.c. of the same solution of caustic soda we shall then obtain on evaporation or treatment with alcohol the *normal* salt—

$$NaOH + NaHSO_3 = Na_2SO_3 + H_2O.$$

Exp. 10.—Powder a crystal or two of sodium sulphite, place the powder in a test tube, and add a few drops of dilute hydrochloric acid. Sulphur dioxide is given off and can be recognised by its smell and its action on a piece of filter paper which has been dipped in a solution of potassium chromate (see Exp. 8 above.) This is a convenient method for the production of pure sulphur dioxide.

$$Na_2SO_3 + 2\ HCl = 2\ NaCl + H_2O + SO_2$$

Exp. 11.—Dissolve a bright, clear crystal of sodium sulphite in a few c.c. of distilled water in a test tube, and add a few drops of barium chloride solution. There will be a white precipitate of barium sulphite.

$$Na_2SO_3 + BaCl_2 = 2\ NaCl + BaSO_3;$$

Add a few drops of hydrochloric acid; the precipitate will dissolve with slight effervescence.

$$BaSO_3 + 2\ HCl = BaCl_2 + H_2SO_3.$$

Now add a few c.c. of chlorine water—a permanent white precipitate will slowly form, the sulphurous acid being oxidised by the chlorine to sulphuric acid, which gives a precipitate of barium sulphate, insoluble in hydrochloric acid.

$$H_2SO_3 + H_2O + Cl_2 = H_2SO_4 + 2\ HCl;$$

$$H_2SO_4 + BaCl_2 = BaSO_4 + 2\ HCl.$$

SECTION XIV. SULPHUR TRIOXIDE.

Exp. 1.—Prepare a little platinised asbestos by dipping asbestos wool in a strong solution of platinic chloride, allowing it to soak well, and then into a strong solution of ammonium chloride. This action causes a precipitate of the double ammonium platinum chloride to be deposited in the fibres. Heat the wet mass (after removing from the ammonium chloride) in a water oven until it is quite dry, and then over the naked Bunsen flame until no more white fumes of ammonium chloride are evolved. The asbestos will then be coated with a thin layer of finely-divided platinum. Draw out one end of a piece of hard glass tubing of about ½ in. diameter in the blow-pipe flame and cut it off in the narrow portion. Make the tube about 9 in. long, and fit the wide end with a cork. Bore the cork and fit into it a narrow tube. Place the platinised asbestos about half way along the tube. Fit up a wash-bottle, as shown in the figure, with a cork through which three tubes pass, two nearly to the bottom, the other just through the cork, connect one long tube to a small gas holder containing oxygen, and the other to a small sulphur dioxide generator containing, instead of copper, sodium sulphite. To obtain sulphur dioxide, allow a little oxygen and sulphur dioxide to bubble up together through

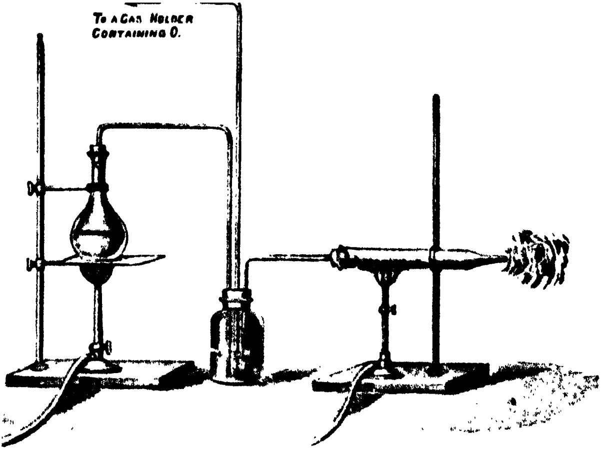


Fig. 19.

sulphuric acid in the wash-bottle, and then to pass through the tube containing asbestos. Nothing will be noticed. Now heat the portion of the tube containing the asbestos by means of the Bunsen burner. White clouds of sulphur *trioxide* will be seen streaming from the narrow opening. When the dioxide and oxygen are heated together in the presence of platinum they unite to form the trioxide.

Note the suffocating smell. The reaction is represented by the equation $2\,SO_2+O_2 = 2\,SO_3$.

Connect a small delivery tube to the contracted end of the heated tube by means of a short piece of indiarubber tubing, and pass the white fumes into a little distilled water contained in a small beaker. After five or ten minutes, add a few drops of hydrochloric acid, and then a few drops of barium chloride solution. A white ppt. of barium sulphate will be produced, showing that sulphuric acid was present.

$$H_2O+SO_3 = H_2SO_4.$$

Exp. 2.—Take a small stoppered retort and introduce about 10 c.c. of Nordhausen sulphuric acid. This contains about 30 per cent. of free sulphur trioxide in solution, which is readily driven off on heating. At the end of the neck place a flask as a receiver, and let a gentle stream of cold water trickle over it to keep it cool. Gently heat the retort—dense white fumes of the trioxide will be evolved, and will condense as white silky needles in the flask. Place of the trioxide in some water, a hissing sound ensues and sulphuric acid is produced.

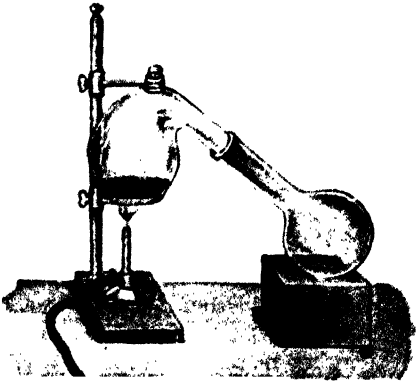


Fig. 20.

SECTION XV.—SULPHURIC ACID.

Exp. 1.—Preparation.—Fit up the apparatus as shown in the figure. It consists of a flask of about five litres capacity fitted with a cork provided with five holes through which pass tubes delivering—

(1) Sulphur dioxide, (2) Nitric oxide,
(3) Steam, (4) Oxygen from a gasholder.

The fifth hole is provided with a tube opening into the air.

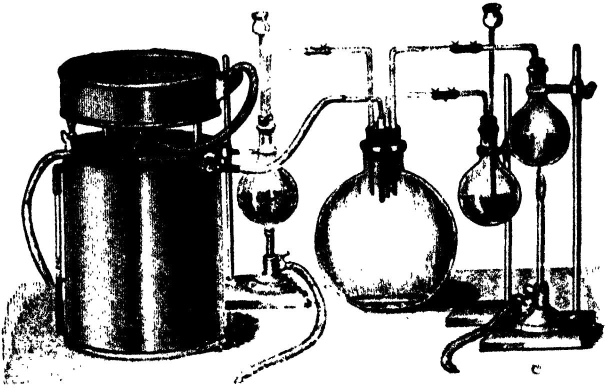


Fig. 21.

Pass some sulphur dioxide, nitric oxide, steam, and oxygen into the flask, then shut off the steam supply: crystals of nitrosulphonic acid (lead chamber crystals) may be seen to form. On clearing the flask of red fumes by a current of oxygen, and then passing in more steam, these crystals will dissolve with the evolution of red fumes. The liquid condensed in the flask may be tested for sulphuric acid.

Properties of Sulphuric Acid.

Exp. 2.—Sulphuric acid when concentrated has great affinity for water. Place about fifty c.c. of water in a small flask and pour in about ten c.c. of strong sulphuric acid, keeping the liquid stirred. Note the great evolution of heat.

Exp. 3.—The affinity of sulphuric acid for water is so great that it can abstract it even from its compounds. Cane sugar for instance is a compound of carbon, hydrogen, and oxygen, the two last elements being present in the same proportion as in water. Make a few c.c. of a very strong solution of sugar in a beaker, place the beaker in a saucer, and pour into the syrup about twenty c.c. of concentrated sulphuric acid. The latter will extract the hydrogen and oxygen in the form of water and immense volumes of carbon will be disengaged.

$$C_{12}H_{22}O_{11} = C_{12} + 11\ H_2O.$$

Exp. 4.—Dip a match-stalk into strong sulphuric acid. The white wood is almost immediately blackened. Wood is of similar composition to sugar, and on treatment with the strong acid the water is partially withdrawn and carbon left, as shown by the charring.

Exp. 5.—Dilute a few c.c. of the strong acid with about ten times its volume of water, and write with the solution, using a stick, on a white sheet of paper. There will be no apparent design. Now warm before the fire (or burner). The design will gradually become darker until it is quite black. The heating causes the weak acid to lose water and so become stronger. The concentrated acid then attacks the paper, taking the water and leaving carbon—the charred design.

SECTION XVI.—CARBON.

Exp. 1.—Place a few splinters of wood in a test tube of hard glass and heat strongly, keeping your thumb loosely fixed on the end. Test the gas with a lighted match. After some time it will burn with a bright luminous flame—it is the chief constituent of ordinary coal gas. Observe that an oily brown liquid distils up the sides of the tube. Test it with a piece of neutral litmus paper—the paper will be reddened showing that the liquid is acid. The black residue, after continued heating, will be fairly pure charcoal. Many substances which contain carbon, when heated out of contact with the air, lose their volatile components and leave the more inert carbon.

Exp. 2.—Heat a piece of bone in the same manner. It chars, showing that carbon is present and gives off a pungent oil. The residue after continued heating is a very impure form of carbon known as *bone black*. It has the valuable property of absorbing various colouring matters from their solutions when they are boiled with it.

Exp. 3.—Various substances containing carbon burn in an insufficient supply of air with a smoky flame. This is the source of that form of carbon known as *lamp black*. Place a little oil in a small dish, apply a light to it, and lower a cool white plate into the bright smoky flame. A black deposit of impure carbon will be obtained.

Exp. 4.—Cane sugar is a very convenient substance to use in the preparation of pure carbon, because of the ease with which it can be decomposed by sulphuric acid. We have already alluded to the great affinity which exists between water and sulphuric acid.

Make about 100 grammes of sugar into a thick syrup by dissolving it in a small quantity of hot water, and place it in a deep glass cylinder, then pour in about 100 c.c. of concentrated sulphuric acid. Presently the liquid will blacken and froth considerably, and a mass of black charcoal, much more bulky than the sugar originally taken, will be formed. Wash this thoroughly with water till free from acid, and there remains carbon in a granular, amorphous condition. By drying this, and then heating it in a stream of chlorine to remove hydrogen or other gases, a very pure specimen of carbon is obtained.

Exp. 5.—Compare specimens of charcoal and graphite. Charcoal is black and dull; graphite is grey, and has a metallic lustre. Try the streak, by writing on a piece of white paper with each. Charcoal leaves a dull black mark like ordinary artist's crayons, whilst graphite leaves the bright grey line characteristic of black lead pencils.

Exp. 6.—Collect two jars of oxygen in the manner described in Section I., Exp. 1. Place a piece of charcoal on a deflagrating spoon and ignite it by heating it in the Bunsen flame. Lower it into the jar of oxygen. It will burn vigorously with bright scintillations. Remove the spoon, and when the jar is cool, add about 50 c.c. of clear lime-water to the contents, and shake. The lime-water will be rendered quite turbid owing to the formation of insoluble calcium carbonate, indicating the presence of the gas carbon dioxide. (If large excess of carbon dioxide is present the white turbidity may re-dissolve). Now repeat the experiment, using graphite instead of charcoal. You will find that the graphite is very difficult to ignite in the Bunsen, so use the mouth blowpipe. When the graphite glows brightly, insert it quickly into the second jar of gas. It will burn more vigorously, but not nearly so brilliantly as the charcoal. Remove the spoon, and test with lime-water as before. The same turbidity will be produced, showing that graphite is composed of the same element as charcoal. In a similar manner we can show that diamond is nothing more nor less than carbon.

Exp. 7.—Take a narrow tube about 18 in. long and ½ in. diameter, and seal off one end. Clean and dry it, fill with mercury and insert in a small basin of mercury. Now fill it with dry ammonia gas by means of the apparatus described in Section VII., Exp. 2, using a strong solution of ammonia instead of the mixture of ammonium chloride and lime. Expel all the air from the apparatus before collecting any gas. Heat strongly a few pieces of wood charcoal (preferably cocoa-nut charcoal), and a piece of graphite, in a small crucible filled with sand. When cool take the graphite and allow it to rise through the mercury into the tube. Nothing will occur. Now insert in the same way the piece of charcoal. Rapid absorption of the ammonia will take place, the gas being condensed in the pores of the charcoal. By using a few pieces of the freshly ignited charcoal, the gas if pure can be almost wholly absorbed. Other gases behave in the same manner, notably hydrochloric acid, sulphur dioxide, sulphuretted hydrogen, nitrous oxide, and carbon dioxide. This property is not possessed by graphite or diamond.

p. 8.—The absorption of gases by charcoal is probably due to its porous character. This is also the reason of its action on dilute solutions of colouring matters and other substances. Dilute solutions of strychnine, for instance, can be deprived entirely of that substance by boiling with freshly prepared animal charcoal. Take about a quarter of a litre of water and add indigo solution until it is of a dark blue colour. Now add about five grams of animal charcoal, boil for ten minutes, allow to settle for a few seconds, and then pour through a paper filter. The filtrate will be quite colourless, shewing that the indigo has been entirely removed. Repeat the experiment with a solution of blue litmus—it will behave *in a similar* manner. Graphite and diamond do *not decolorise* these solutions, but bone black, being especially porous, possesses this property in a greater degree than the other forms of amorphous carbon.

Exp. 9.—Make an intimate mixture of about a gramme of finely-powdered oxide of lead (litharge) with about one-tenth its weight of charcoal, and heat to redness in a hard glass tube, or porcelain crucible, for five minutes. Now throw some of the powder into a mortar with a little water and rub it up, using pressure, with the pestle, and then wash away the charcoal by means of a stream of water. Pellets or plates of metallic lead will be obtained—

$$2\,PbO + C = 2\,Pb + CO_2.$$

Exp. 10.—Rub together in a small mortar about a gramme of black copper oxide, and about an eighth of that amount of fine charcoal. Mix intimately and place in a test tube. Fit the tube with a cork and delivery tube, the latter bent twice at right angles, so that it can be dipped into clear lime-water contained in another test tube. Heat the tube containing the copper oxide in the Bunsen flame. At first air will bubble through the liquid, and then a gas which turns it milky. This latter is carbon dioxide. Heat until no more gas is disengaged. Remove the cork and delivery tube, cool, and shake out the copper oxide on to a piece of white paper. Note that it is of a much lighter colour than the original copper oxide, this difference being due to the presence of reduced copper. Place a little in a test tube and add a few c.c. of dilute nitric acid. Reddish fumes of nitric peroxide will be evolved, the copper reacting with the nitric acid (see Section X., Exp. 1). What action has *copper oxide* on nitric acid?

$$2\,CuO + C = 2\,Cu + CO_2.$$

SECTION XVII.—CARBON MONOXIDE.

Exp. 1.—Fit an iron gun-barrel with a cork at each end, and pack it tightly with pieces of charcoal of the size of a nut, holding them in their places by means of loose plugs of asbestos. Through one cork arrange a delivery tube leading to a small pneumatic trough, and through the other cork a tube connected to a wash-bottle containing water, connect the wash-bottle by means of another tube to a gas-holder filled with carbon dioxide (as shown in the figure), or with a carbon dioxide generator like that described in Section XVIII., Exp. 1. Wrap pieces of wet filter paper around the ends of the iron barrel near the corks, and see that they are kept moist. Heat the barrel to bright redness by means of a small tube furnace, and pass over the charcoal a slow stream of carbon dioxide. Collect a jar of the gas at the trough, and cover the mouth with a plate. Disconnect the carbon dioxide apparatus, and apply a light to the jar of gas. It burns with a bluish flame, but does not allow the taper to burn in it. The gas is carbon monoxide.

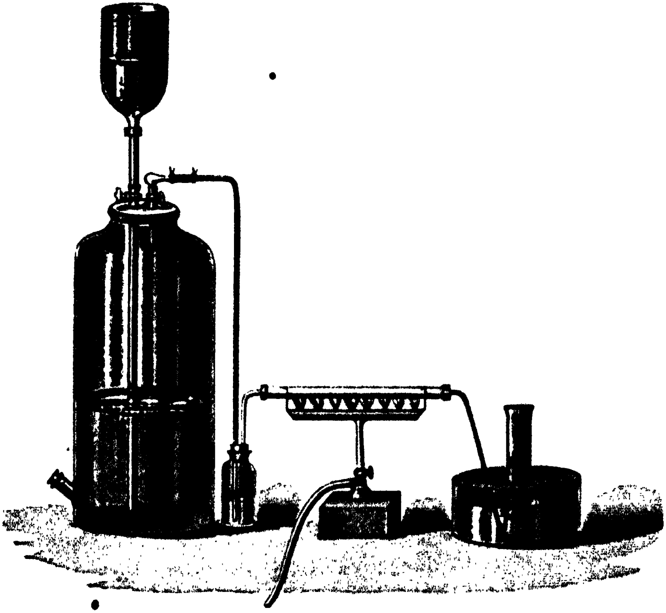


Fig. 22.

$$CO_2 + C = 2CO.$$

Exp. 2.—About 20 grammes of crystallized oxalic acid are put into an eight-ounce flask provided with thistle funnel and delivery-tube, and as much concentrated sulphuric acid as will cover it. Heat is applied steadily until effervescence begins, and is then moderated so as to secure a regular and not too rapid evolution of the gas. Collect over water, avoiding any escape of the gas, as it is very poisonous. The reaction which takes place is—

$$\left.\begin{matrix}\mathrm{COOH}\\ \mathrm{COOH}\end{matrix}\right\} + H_2SO_4 = CO + CO_2 + H_2O + H_2SO_4.$$

The sulphuric acid removes the elements of water from the oxalic acid without itself undergoing any chemical change.

Exp. 3.—Pour lime-water into a jar of the gas, and shake up; the lime-water will become turbid owing to the formation of calcium carbonate—

$$CaO + CO_2 = CaCO_3.$$

The presence of carbon dioxide is thus indicated.

After the experiment has been performed, it is well to burn the gas rather than let it escape directly into the air.

Exp. 4.—Pour a few cubic centimetres of caustic soda into a jar of the gas and shake up well, the carbon dioxide will combine with the caustic soda, forming sodium carbonate—

$$2\ NaOH + CO_2 = Na_2CO_3 + H_2O.$$

Bring the mouth of the jar under water, the water will rise, and it will be seen that about half the volume of the gas is left. This is the carbon monoxide. Transfer some of this gas to a smaller vessel by decanting it over water, and shake up with lime-water; no turbidity will be produced, the carbon dioxide having been removed. Now apply a light to the gas; it will burn with a beautiful blue lambent flame. When the combustion is finished, again shake up the vessel, and marked turbidity will then be produced, showing that carbon dioxide has again appeared. The carbon monoxide has united with oxygen during the process of combustion, with the formation of carbon dioxide—

$$2\,CO + O_2 = 2\,CO_2.$$

It may also be noticed that carbon monoxide does not itself support combustion, for if, whilst it is burning, the lighted taper be plunged into the vessel, the flame will be extinguished.

Exp. 5.—Remove the carbon dioxide from a second jar of the collected gas, and then decant into the jar sufficient air to fill the vessel.

We have now a mixture of equal volumes of carbon monoxide and air, and if, after allowing the gases to stand for two or three minutes to mix properly, the mouth of the jar be held towards the flame of a Bunsen burner, it will be seen that carbon monoxide and air form an explosive mixture.

Exp. 6.—Carbon monoxide can be prepared quite free from carbon dioxide, however, in several ways. Heat a little coarsely powdered Potassium ferrocyanide with a few c.c. of concentrated sulphuric acid in a test tube. Gas will be evolved, which can be shown to be carbon monoxide by applying a light. It will burn with a blue flame. Before doing this, however, we can show that carbon dioxide is absent by passing the gas into clear lime-water.

$$K_4FeC_6N_6 + 6\,H_2SO_4 + 6\,H_2O = 6\,CO + 2\,K_2SO_4 + FeSO_4 + 3\,(NH_4)_2SO_4$$

Exp. 7.—Heat a few pieces of potassium formate with a few c.c. of strong sulphuric acid in a test tube. Gas will be evolved as in the previous case. Test it first for carbon dioxide by passing into lime-water, and then for carbon monoxide by applying a light. It will burn with the flame characteristic of carbon monoxide, and will not render lime-water milky.

$$KHCO_2 + H_2SO_4 = KHSO_4 + H_2O + CO.$$

Exp. 8.—By passing steam over strongly heated charcoal we get a mixture of carbon monoxide and hydrogen which is known as *water gas*, and is often used on a large scale as fuel. Bore a hole of about ¼ in. diameter in the bottom of a large clay crucible by means of a sharp file, and fill it with pieces of charcoal of about the size of a nut. Cover the crucible with a similar one, inverted, and also perforated. Fit a small 8 oz. flask with a cork and delivery tube bent upwards, and with its end drawn out to a fine point. Place some water in the flask and keep it gently boiling so that you have a fine jet of steam. Now turn the blow-pipe flame into the lower hole of the crucible and heat the charcoal to redness. Remove the flame and direct the jet of steam into the crucible. A mixture of hydrogen and carbon monoxide will escape from the top and can be lit by means of a lighted taper.

$$H_2O + C = CO + H_2.$$

Exp. 9.—Carbon monoxide, by virtue of the fact that it combines with oxygen forming carbon dioxide, acts as a good reducing agent. Take the tube used in preparing sulphur trioxide (Section XIV., Exp. 1) or a similar one, and instead of asbestos place in it a thin layer of copper oxide. Connect the corked end with a wash-bottle containing a strong solution of caustic soda, and that again with your carbon monoxide generator. Heat the copper oxide gently and pass a slow stream of the monoxide over it. It will be gradually reduced to reddish brown metallic copper. You should of course first see that all the air is driven out of the apparatus. That carbon dioxide is produced in this reaction can be shown by passing the gas, after leaving the copper oxide, into lime-water. The lime-water will become milky.

$$CuO + CO = CO_2 + Cu.$$

Exp. 10.—Cuprous chloride, when dissolved in hydrochloric acid, possesses the property of dissolving carbon monoxide. Dissolve copper oxide in hot dilute hydrochloric acid until no more will dissolve. Pour off the clear liquid after allowing the undissolved oxide to settle, and boil it with some of the reduced copper prepared in the last experiment. Cork it up in a bottle to keep it out of contact with the air. Next collect a tube full of carbon monoxide, which has been separated from the carbon dioxide by means of caustic soda solution, by decanting from a larger vessel in the pneumatic trough. Pour in about 20 c.c. of your acid solution of the cuprous chloride, close the mouth of the vessel lightly with your hand, shake well, and open under water. The gas will dissolve in the cuprous chloride, forming a compound with it, and water will be sucked in to take its place.

SECTION XVIII.—CARBON DIOXIDE.

Exp. 1.—**Preparation.**—Carbon dioxide is most conviently prepared by acting on a carbonate with dilute mineral acids. Take a Woulff's bottle (a stout glass bottle with two narrow necks) and charge it with about 50 grammes of marble, broken into pieces of the size of a nut. Fit the bottle with two tightly fitting corks, and through one introduce a thistle funnel which will barely touch the bottom of the bottle as shown in Fig. (23). In the other cork place

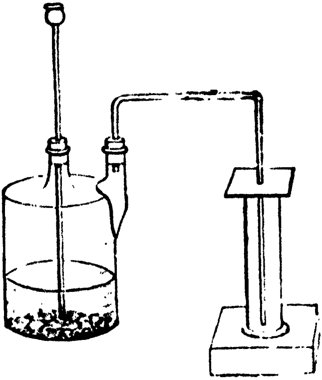


Fig 23.

a delivery tube bent twice at right angles so as to make three sides of a rectangle, one end just dipping below the cork, the other almost reaching to the table. If the gas is required free from hydrochloric acid it must first be passed through a wash-bottle containing water. Place about 50 c.c. of water in the apparatus, see that all joints are tight, and add a little dilute hydrochloric acid (made by adding an equal bulk of water to strong acid). Brisk effervescence will ensue, carbon dioxide being evolved. Collect the gas by downward displacement of air, placing the jars under the delivery tube, and covering with greased glass plates. The jar is full when a lighted match held at the mouth of the jar is extinguished. Note that the gas thus collected is colourless and without taste or smell.

$$CaCO_3 + 2\,HCl = CaCl_2 + H_2O + CO_2.$$

Properties of Carbon Dioxide.

Exp. 2.—Fill a large beaker of three or four litres capacity, by downward displacement, with carbon dioxide which has been bubbled through water to remove hydrochloric acid, and plunge a taper into the gas; it will be immediately extinguished. Now detach a soap-bubble charged with air into the beaker, and it will be found to float on the surface of the heavier carbon dioxide. The density and other properties of the gas may further be illustrated by pouring it over a lighted candle, and thus extinguishing it, or by ladling out the gas with a smaller beaker, and showing the presence of carbon dioxide in the beaker by its extinguishing a taper.

Exp. 3.—To a few cubic centimètres of lime water add four or five times the volume of distilled water, and pass a stream of carbon dioxide through the clear liquid. At first a turbidity is produced, owing to the formation of calcium carbonate—

$$Ca(HO)_2 + CO_2 = CaCO_3 + H_2O.$$

Continue to pass the gas, and the liquid will become quite clear again, because we have now carbon dioxide in solution in the water. Divide the clear liquid into two portions, and boil one portion for a little time; to the other add a volume of lime water equal to that originally taken. In each case the turbidity first noticed will be reproduced, since in each case we have got rid of the excess of carbon dioxide; in the first case we expelled it by heat, in the second we added sufficient lime to combine with it to form calcium carbonate.

Exp. 4.—Pass a stream of carbon dioxide through a few cubic centimètres of water to which some drops of litmus solution have been added, and note that the litmus assumes a claret tinge; contrast this with the effect of adding litmus to water containing a little hydrochloric or sulphuric acid. Notice also that by boiling, the carbon dioxide is expelled, and the litmus assumes its original colour. A solution of carbon dioxide in water is, therefore, very unstable, and possessed of a feebly acid character.

Exp. 5.—Pass the expired air from the lungs, or carbon dioxide (washed) from marble, through a slightly alkaline pink solution of phenol-phthalein,—the pink liquid becomes colourless. This change from a pink to a colourless solution may be used as a means of indicating the presence of carbon dioxide.

Exp. 6.—Simple proof that carbon dioxide contains carbon.—Although carbon dioxide has been shown by Exp. 2 above to be incapable of supporting the combustion of a candle, it can be decomposed by some few substances which have great affinity for oxygen. Magnesium, sodium, and potassium are metals of this nature. Take a strip of magnesium ribbon about 9 in. long and scrape it well with a knife to remove magnesia. Hold one end in a pair of crucible tongs and ignite the other in the Bunsen flame. Insert it immediately into a jar of carbon dioxide. It will continue to burn brilliantly, white oxide of magnesium and carbon deing deposited on the sides and floor of the vessel. In order to see the carbon more plainly, dissolve away the magnesia by means of a little dilute hydrochloric acid, and filter. Black specks of charcoal will be left on the filter paper.

$$2\ Mg + CO_2 = 2\ MgO + C.$$

Exp. 7.—Carbon dioxide is fairly soluble in water at the ordinary temperature, water dissolving its own

volume of gas at 15° C. Take one of the jars filled with the gas, introduce about 200 c.c. of cold water, place your hand tightly over the mouth and shake well. There will be strong suction on the hand, showing that gas has been absorbed by the water. Open the mouth under water, the liquid will rise in the jar.

Exp. 8.—Shake up in a similar manner a jar of the gas with dilute solution of caustic soda, and rapidly bring the mouth under water and open it. If the gas is pure the vessel will immediately fill with water, the caustic soda combining with the carbon dioxide to form sodium carbonate.

$$2\,NaHO + CO_2 = Na_2CO_3 + H_2O.$$

Exp. 9.—Pass carbon dioxide into a solution of caustic soda until the liquid is saturated, and then evaporate down to dryness on a water-bath. A residue will be obtained consisting of the monohydrated normal carbonate of soda, $Na_2CO_3 \,.\, H_2O$.

Exp. 10.—**Test for carbonates.**—Add dilute hydrochloric acid to the solid carbonate, or to an aqueous solution of a carbonate in a test-tube. An effervescence will be observed, and, on decanting the gas downwards into a second tube containing lime-water, and shaking up, a turbidity will be produced in the lime-water owing to the formation of calcium carbonate.

Exp. 11.—Take the liquid remaining in the generator at the close of Exp. 1. Evaporate it to dryness in a porcelain basin. The white solid which is obtained is calcium chloride. Dissolve a little in water and prove that a chloride is present by the silver nitrate test. (See Section V., Exp. 7).

SECTION XIX.—FLAME.

FLAME.—Whenever a gas or vapour is brought into an atmosphere with which it can react chemically, and the heat generated is sufficient to bring about incandescence of the particles, flame is produced. The heat is generated, and the incandescence effected, in the region where the reaction is carried on, that is, at the surface where contact occurs between the two gases, as is seen when a jar of hydrogen burns mouth downwards. When we speak of hydrogen or coal gas as being a combustible gas, and of air as being the supporter of combustion, we imply that hydrogen or coal gas, when once ignited, burns in air. And in flames under ordinary circumstances this is the case; if, however, we were to lead a stream of air from a jet into an atmosphere of coal gas, the flame would attach itself to the jet, and might be described as air burning in coal gas. In either case the flame marks the surface of contact between the air and coal gas, and is the region where the chemical changes take place, which transform the hydrogen and carbon in the coal gas into water and carbon dioxide as ultimate products.

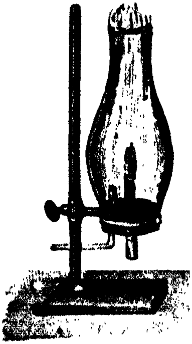


Fig. 24.

Exp. 1.—Fit into the bottom of an ordinary lamp chimney a cork through which passes a short piece of straight wide tubing, and a second narrow piece bent at right angles as shown in Fig. 24, and connected with the supply of coal gas. Close the aperture at the top of the chimney, and allow the gas to escape by the straight tube until the air is displaced, then light it at the lower extremity of this tube and uncover the aperture at the top of the chimney. The flame will then pass up the tube and attach itself to the inner opening where the air and coal gas meet, the flame area being air and the surrounding atmosphere coal gas. The gas escaping at the top of the chimney may also be ignited, combustion occurring here in the ordinary manner.

In any case before flame can be produced at all, the temperature of the combustible body must first reach a certain limit known as the *point of ignition.* This temperature varies with different bodies; the vapour of carbon bisulphide may be ignited by a glass rod heated only to 150°C., whilst with hydrogen or coal gas a dull red heat (600°C.) is insufficient. Conversely, a flame is extinguished if its temperature is by any means reduced below the point of ignition of the vapours consumed in it (see Exp. 3).

Exp. 2.—Hold a piece of wire gauze (about thirty meshes to the inch) horizontally over a Bunsen burner and about an inch above the orifice (Fig. 25). Turn on the gas and light it on the upper side of the gauze, the flame will not be communicated to the stream of gas on the underside of the gauze. Much heat is carried off by the gauze, and the part of the flame in contact with the gauze where it meets the upward current of gas, is so far cooled in conquence of this that its temperature falls below the point of ignition of the gas.

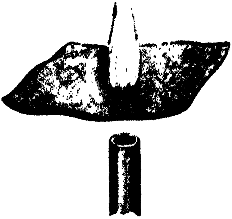


Fig. 25.

Exp. 3.—Light the Bunsen burner and depress the gauze into the flame. The cold gauze will conduct away the heat from the portion of flame in contact with it, and lower its temperature below the ignition point of the gas, which will now only burn below the wire. After a time the gauze will also become heated if no special precaution is taken to keep it cool. When it becomes quite red hot, the ignition point of the gas is reached, and the flame will strike through and burn above the gauze again.

Exp. 4.—Make a piece of the wire gauze into a cylindrical roll and place a candle within it. Now direct the flame of a Bunsen burner against the outer surface of the gauze; the wax may be melted, but the candle cannot be lighted unless the gauze is heated to redness. The reason for this will be gathered from the explanation given in the previous experiment.

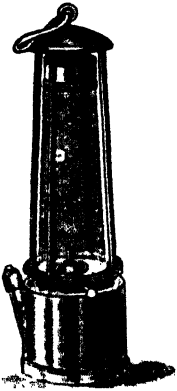
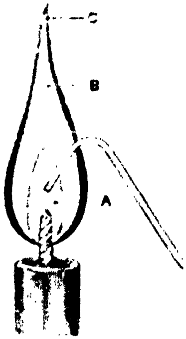


Fig. 26.

The *Davy Lamp* (Fig. 26) is such an arrangement, in which an oil lamp is shut in by a layer of gauze, and even if such a lamp be entirely surrounded with inflammable gas, this will not become ignited, although the inflammable gas which passes through the gauze may burn inside it and fill the space above the oil lamp with flame. If, however, the gauze becomes strongly heated, or if the flame should be mechanically driven through the meshes, communication with the inflammable atmosphere outside may be established, and ignition will then take place.

The candle flame.—The inflammable matter in a candle is the wax or tallow, consisting essentially of carbon and hydrogen. This is melted round the wick, which becomes charged and serves as a still, from which the vapours of hydrocarbons are supplied into the area immediately surrounding it. That such an area exists containing combustible vapour may easily be shown.

Exp. 5.—Depress a sheet of stout paper quickly into a candle flame to the level of the top of the wick, and hold it steadily there for about a second. On withdrawing it, a ring of sooty deposit will be seen,



enclosing a clear area. Secondly, take a glass syphon tube, as shown in Fig. 27, and bring the shorter limb into the centre of the flame; presently yellowish-brown vapours will be seen to pass down the tube and issue at the other end. These vapours will be found to be inflammable, and may be burnt at the exit of the tube.

Exp. 6.—Make a short coil of stout copper wire by giving it half-a-dozen turns round a piece of glass rod about 5 m.m. in diameter, and so that only a small space is left between one coil and the next. Bring the coil into the upper part of the luminous zone of a candle flame; the flame will become smoky: if it be quickly depressed to the level of the wick, the flame loses its luminosity, and indeed may be extinguished altogether. Copper, being a good conductor and radiator, carries off heat and lowers the temperature to such an extent that the particles no longer maintain the white heat which imparts the luminosity to the flame, and the combustion is rendered so incomplete that carbonaceous matters escape combustion and pass off as smoke. The vapours may in this way be cooled down even below their point of ignition, and the flame is then extinguished altogether.

SECTION X.—DIFFUSION OF GASES.

The particles of gases possess a much greater freedom of motion than do those of liquids or solids, for the excursions which they may make are confined in the case of the latter to the space occupied by the liquid or solid itself; in the case of gases, however, the extent of these excursions is only limited by the walls of the vessel in which they are contained. Furthermore, if the walls are loose and open in texture, that is, porous, then it is possible for the gaseous particles to pass through them.

Exp. 1.—A stout glass collecting jar of about one quarter litre content is filled with hydrogen and held steadily, mouth downwards, for three or four minutes. A lighted taper is then brought into it, and it will be found that instead of the gas burning quietly, as pure hydrogen does, there will be an explosion. This shows that, during the period of exposure, the heavier air particles have diffused into the jar, the explosion being due to admixture of air with the hydrogen.

Exp. 2.—A porous cell such as is used in voltaic batteries, is fitted with an indiarubber stopper, and a glass tube about half a meter long, furnished with a bulb, is passed through the stopper; this is bent and drawn out into a jet as shown in the figure. Before inserting the cork, fill the bulb and lower part of the tube with water. Now place a beaker filled with hydrogen over the porous cell, and the water will be immediately depressed and driven out of the jet in a fine stream. This is evidence that the light gas hydrogen passes through the walls into the porous cell more quickly than the air is able to pass out of it. A greater volume of gas collects within the space of the cell and tube and forces the water out before it.

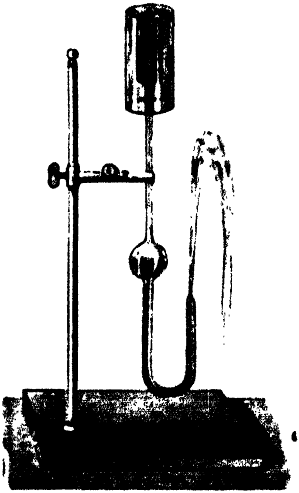


Fig. 28.

Exp. 3.—Do a similar experiment with the arrangement shown in the figure below, in which it is possible to observe, by bubbles passing through the water in the bottle, that air is finding its way into the porous cell through the glass tube. In this case the cell containing air is surrounded with carbon dioxide in a beaker. Under such circumstances the air diffuses out of the cell more quickly than the heavier gas, carbon dioxide, passes into it. This tends to produce a partial vacuum within the cell, and air enters in bubbles through the liquid in the bottle.

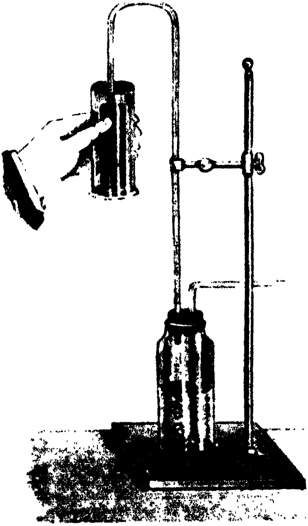


Fig. 29.

Exp. 4.—Repeat the last experiment, using oxygen instead of carbon dioxide, and note that in this case the bubbles pass in much more slowly, and altogether a much smaller volume of air is required to restore equilibrium in the cell.

The relative densities of the gases employed in the above experiments are—

Hydrogen, 1. Air, 14·4. Carbon dioxide, 22.
Oxygen, 16.

The greater the difference in density, the more rapid is the diffusion. By measuring the amount of different gases which diffused through a porous plug in a given time, under like conditions, Graham found that *the relative rates of diffusion are inversely proportional to the square roots of their densities.* Thus in relation to air—

		$\frac{1}{\sqrt{\text{density}}}$.	Velocity of diffusion observed.
Density of hydrogen	= 0·0695	= 3.792	3·830
,, oxygen	= 1·1043	= 0·951	0·949
,, carbon dioxide	= 1·5180	= 0·812	0·812

DETECTION OF A GAS.

Lower a lighted taper into the gas.

THE GAS IS IGNITED.

H_2S, CO, H.

1. It burns with a very pale blue flame and a deposit of sulphur is seen on the sides of the jar after the combustion.

The gas was **Sulphuretted hydrogen.**

Confirm by now testing for SO_2 with K_2CrO_4 paper.

2. It burns with a bright blue flame and no sulphur is deposited.

Pour clear limewater into the jar.

The limewater is turned milky.

3. The gas was **Carbon monoxide.**

The gas burns with a nearly nonluminous flame and after combustion limewater is not turned milky by the resulting gas

The gas was **Hydrogen.**

THE GAS IS NOT IGNITED BUT THE TAPER CONTINUES TO BURN.

O, N_2O, Cl.

The taper burns more brightly than in air.

The gas is O or N_2O.

Withdraw the taper quickly so that as little air is admitted as possible, or if another jar of the gas is at hand, use it instead, for the following test.

1. Take a jar containing nitric oxide gas and place it mouth to mouth under water with the jar of gas to be examined, and mix the gases.

Red fumes are produced.

The gas is **Oxygen.**

2. No red fumes are seen (except a little due to the air admitted if the first jar is used).

The gas is **Nitrous oxide.**

3. The taper burns with a smoky flame.

The gas is **Chlorine.**

Chlorine is easily recognised by its green colour and irritating odour.

THE GAS IS NOT IGNITED AND THE TAPER IS EXTINGUISHED.

NH_3, SO_2, NO, HCl, N, CO_2.

Smell the gas cautiously, NH_3 and SO_2 have characteristic odours.

1. Leave a piece of red litmus paper, previously damped, in the jar for some minutes.

The paper is turned blue.

The gas is **Ammonia.**

2. Pour a little dilute solution of $KMnO_4$ into the jar, if the solution is decolorised,

The gas is **Sulphur dioxide.**

3. Red fumes are formed when air is admitted to the jar.

The gas is **Nitric oxide.**

4. Pour some clear limewater into the jar.

The limewater is turned milky.

The gas is **Carbon dioxide.**

5. Leave a piece of blue litmus paper, previo damped, in the jar.

The paper

The gas is **Hydrochloric acid.**

6. If the gas answers to none of the above tests, it is

If more than one jar of the gas is at hand, confirm the above tests by performing some of the experiments with it described in Chapter III.

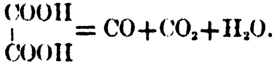
CHAPTER IV.

EXPERIMENTS ON THE ACTION OF HEAT, WATER, AND ACIDS ON SOME FAMILIAR SUBSTANCES.

THE following experiments are arranged for the purpose of training the student's powers of observation, and confirming his knowledge of certain important chemical processes. Accurate notes should be taken, recording the facts observed, and the equations representing the reactions carefully worked out.

Exp. 1.—In a dry test tube place about two grammes of oxalic acid and add rather more than an equal quantity of concentrated sulphuric acid. Nothing happens apparently in the cold. Fit the mouth of the tube with a cork and delivery tube; the latter should be bent so as to make three sides of a rectangle, one side being much shorter than the opposite one and passing just beneath the cork. In another test tube place about five c.c. of clear lime water. Heat the mixture of oxalic and sulphuric acids gently over a Bunsen; the crystals dissolve and then give off gas, with a certain amount of effervescence. If gentle heat is applied, the evolution of gas will be quite steady. Bubble a little of the colourless gas through the lime water. The latter will become quite milky. What does this indicate? Now remove the cork and tube and apply a lighted match to the mouth of the test tube. A beautiful blue flame is obtained showing that the gas burns. But the match is extinguished when pushed into the tube; the gas does not support combustion. What common gas, burning in air with a blue flame, are you acquainted with? Why do we test with lime water *before* applying a light, rather than after?

The following equation explains the reaction:—



$$\begin{matrix} COOH \\ | \\ COOH \end{matrix} = CO + CO_2 + H_2O.$$

Oxalic acid.

Exp. 2.—Take a dry test tube of the same diameter as that used in the previous experiment, so that the same cork and delivery tube will do again. Place in it a few crystals of the reddish compound, ammonium dichromate, fit the cork and delivery tube, and heat gently. Suddenly a brisk agitation will be set up, the larger red crystals rapidly disintegrating into finer green particles with increase of volume. A gas is also evolved which is colourless. Bubble a little through lime water. No milkiness is produced. Remove the cork and test the gas with a lighted match. The gas does not burn, nor does it support combustion. It has neither taste nor smell. What common colourless gas has these properties? Examine the curious green powder left in the tube. Is it soluble in water? Is the ammonium dichromate soluble? The equation of the reaction is:—

$$(NH_4)_2Cr_2O_7 = N_2 + 4H_2O + Cr_2O_3.$$

Ammonium dichromate. Chromium sesquioxide.

Exp. 3.—In a test tube place about half a gramme of red lead, add about five c.c. of fairly concentrated hydrochloric acid, and warm gently, keeping the thumb loosely covering the mouth of the tube. The air in the tube is gradually replaced by a gas having a greenish yellow colour. What gases are you acquainted with, which have this character? Moisten a strip of iodide-starch paper and bring it to the mouth of the tube. A blue colour is produced. What substance has the blue colour, and why is it produced? Moisten a piece of litmus paper and insert it just inside the mouth of the tube, but above the level of the liquid. Replace your thumb and allow the gas to act thoroughly on the litmus. It will be gradually bleached. Observe the pungent, irritating odour. Test with a lighted match—it is extinguished by the gas, which does not burn. On allowing the liquid to cool, white needle-shaped crystals separate out, some floating on the surface. What are they? The equation is:—

$$\underset{\text{Red lead.}}{Pb_3O_4} + 8HCl = \underset{\text{Lead chloride.}}{3PbCl_2} + 4H_2O + Cl_2.$$

Exp. 4.—Heat strongly in a dry test tube about a gramme of barium peroxide, and pass the evolved gas into clear lime water by means of the delivery tube. No turbidity is produced. Remove the cork and test the gas by means of a lighted match. The gas does not burn, but the match when thrust into the gas burns much more vigorously than before. Collect more of the gas by loosely closing the mouth of the tube with the thumb, and test with a glowing splinter. It at once bursts into flame, however feeble the spark. What is the gas evolved? The equation is:—

$$\underset{\text{Barium peroxide.}}{2BaO_2} = \underset{\text{Barium monoxide.}}{2BaO} + O_2.$$

Warm with dilute hydrochloric or sulphuric acid a little of the original peroxide, and then some that has been strongly heated. Do you notice any difference in their behaviour?

Exp. 5.—To a few crystals of sodium sulphite contained in a tube, add a few c.c. of dilute hydrochloric acid. Brisk effervescence takes place. Smell the gas. Do you recognise it? Moisten a small piece of filter paper with a solution of potassium chromate; it will be stained a faint yellow. Bring it into the gas; it is changed to a green, indicating the presence of a strong reducing agent. What is the gas? The equation is:—

Sodium sulphite.

Sulphur dioxide.

Exp. 6.—Mix together in a small mortar about equal quantities of solid ammonium chloride and quicklime. Place the powdered mixture in a dry tube and heat gently over a Bunsen flame. Do you recognise the smell of the gas evolved? Close the mouth of the tube with the thumb and collect a little of the gas; test it with a lighted match. The gas *may* burn with a yellowish flame, but it extinguishes the match. Moisten a piece of red litmus paper and bring it to the gas. It is rapidly turned blue, indicating that the gas has a strong alkaline character. Remove the stopper from a bottle of hydrochloric acid and bring it opposite the mouth of the tube. Marked white fumes are produced, showing that the hydrochloric acid reacts with the gas. What is the gas, of alkaline character, which possesses the properties detailed above? The equation is:—

$$2NH_4Cl + CaO = 2NH_3 + CaCl_2 + H_2O.$$

Exp. 7.—Fill a test tube to a depth of half an inch with black antimony sulphide, and add about 5 c.c. of fairly concentrated hydrochloric acid. Warm gently. An offensive smell is noticed, resembling that of rotten eggs. Apply a lighted match to the gas; it burns with a pale bluish flame, and deposits a yellow substance on the sides of the tube, but the match is put out. Place a drop of lead acetate solution on a piece of filter paper and allow the gas to come in contact with it. It is rapidly blackened. What colourless gas possesses these properties? What is the yellow deposit noted above?

$$Sb_2S_3 + 6\,HCl = 2\,SbCl_3 + 3\,H_2S.$$

Exp. 8.—Heat a few pieces of copper nitrate in a dry tube. Note the brown colour of the gas evolved. The green nitrate gradually turns black as it loses the gas. What is the black substance produced? Test the gas with a lighted match—the match is extinguished and the gas does not burn. What is the gas evolved? Equation:—

$$2\,Cu(NO_3)_2 = 2\,CuO + 4\,NO_2 + O_2.$$

Exp. 9.—Place a few pieces of calcium chloride in a test tube and cover them with concentrated sulphuric acid. Heat gently; copious white fumes are evolved of very irritating odour. Test the gas with a piece of moist, blue litmus paper. It is rapidly turned red, indicating the presence of a strongly acid gas. Collect a little of the gas in the tube by closing the mouth with the thumb, and apply a lighted match. The match is extinguished, and the gas does not burn. Fit the cork and delivery tube in the mouth of the test tube and drive off more of the gas. Dip the end of the delivery tube under water. The gas dissolves and the water rises in the tube. What colourless soluble gas has the properties observed? Note the white residue left in the tube. Test it with water and see if it dissolves. Is the original calcium chloride soluble? Equation:—

$$\underset{\text{Calcium chloride.}}{CaCl_2}+H_2SO_4 = 2HCl+\underset{\text{Calcium sulphate.}}{CaSO_4}.$$

Exp. 10.—Mix a little black oxide of copper with about a sixth part of its weight of charcoal in a small mortar. Heat the mixture in a dry test tube fitted with cork and delivery tube, and pass the gas evolved through a little clear lime water. The liquid is rapidly turned milky. What does this show? If passed through long enough, the gas will gradually re-dissolve the precipitate which causes the milkiness. Why? Test the gas with a lighted match; the match is extinguished. After heating strongly for some time, shake out the contents of the tube into a mortar. Observe the change in colour. Rub well with a pestle and note that some of the particles can be readily burnished, and have a distinct red colour. To a portion of the changed powder add a few drops of dilute nitric acid. Red fumes are evolved. What are they? Would copper oxide behave in the same manner if similarly treated? What is the action of the carbon in this reaction? The equation representing the reaction is,

$$2CuO+C = 2Cu+CO_2.$$

Exp. 11.—Dissolve a lump of sugar in the least possible quantity of water, add three or four times the bulk of strong sulphuric acid, and warm slightly. The liquid rapidly chars, turning black. What can the blackening be due to? Pour out the frothy mess into a basin and note the solid black substance which has been produced. What is it? In what notable points does it differ from the sugar originally taken? What is the action of the sulphuric acid on the sugar?

$$C_{12}H_{22}O_{11} = 12\,C + 11\,H_2O.$$

Exp. 12.—Heat a few pieces of thin zinc foil in a tube with about 10 c.c. of fairly concentrated caustic soda solution. After a short time brisk effervescence will take place. Test the gas, which is colourless, by passing through clear lime water. No turbidity is produced. Apply a lighted match. The gas extinguishes the match, but itself burns with a faintly luminous flame. Has it any smell? Equation:—

$$Zn + 2\,NaOH = Na_2ZnO_2 + H_2.$$

Exp. 13.—Roughly powder a piece of potassium ferrocyanide, place in a test tube, and add double the bulk of concentrated sulphuric acid. Heat gently. Effervescence takes place, the gas evolved being colourless. Pass a few bubbles through clear lime water; no turbidity is produced. Test with a lighted match; the gas burns with a brilliant blue flame. What is it? In what respects does it differ from the gas obtained in Exp. 1? The reaction is somewhat complicated.

$$K_4FeC_6N_6 + 6\,H_2SO_4 + 6\,H_2O = 2\,K_2SO_4 + FeSO_4$$
$$+ 3\,(NH_4)_2SO_4 + 6\,CO.$$

Exp. 14.—Powder about equal proportions of potassium nitrate and ammonium chloride together in a mortar, dissolve in the least possible quantity of water, and heat gently in a test tube. A colourless gas is steadily evolved. Pass a little into lime water—no milkiness is produced. Apply a light to the mouth of the tube—it is extinguished and the gas does not burn. Also it has no taste or smell. The reaction is the same as if we had heated ammonium nitrite. Equation :—

$$NH_4NO_2 = N_2+2\,H_2O.$$

Exp. 15.—Heat a few crystals of potassium nitrate ("saltpetre") with a few c.c. of concentrated sulphuric acid. Note the irritating fumes, with their faint reddish colour, in the tube. The faint reddish tinge is due to oxides of nitrogen, the presence of which distinguishes the fumes from those of hydrochloric acid. What vapour can it be? Equation :—

$$\underset{\text{Potassium nitrate.}}{KNO_3}+H_2SO_4 = KHSO_4+\underset{\text{Nitric acid.}}{HNO_3}$$

Exp. 16.—Mix together about equal proportions of iron filings and yellow "flowers of sulphur," and heat the mixture strongly in a test tube. Suddenly the mixture will glow strongly, showing that some reaction is taking place. When the mass is cool, break it up, and place in a fresh tube. Add a few c.c. of dilute hydrochloric acid and warm gently. A gas of offensive smell and taste is evolved. Test with a lighted match; it burns with a pale bluish flame and deposits a light yellowish powder on the walls of the tube, but does not allow the match to burn in it. Test the gas with a piece of lead acetate paper; the paper is immediately blackened. What gas is indicated by these tests? The reactions are represented by :—

$$Fe+S = \underset{\text{Ferrous sulphide.}}{FeS};\ FeS+2\,HCl = FeCl_2+\underset{\text{Sulphuretted hydrogen.}}{H_2S}.$$

Exp. 17.—Pour a few drops of mercury into a test tube and add a few c.c. of concentrated sulphuric acid. Warm gradually; after a short time gas will be evolved. Test with a lighted match; the match will be extinguished and the gas will not burn. Note the characteristic taste and smell. Test again with a piece of potassium chromate paper. The paper will be turned green. After the reaction a white substance will settle from the liquid. What is it?

$$Hg + 2\,H_2SO_4 = \underset{\text{Mercuric sulphate.}}{HgSO_4} + 2\,H_2O + SO_2.$$

Exp. 18—Mix a little salt with about 100 c.c. of water. To obtain pure water from the mixture, fit up the apparatus as shown in the figure. Place the mixture of salt and water in the flask connected to a Liebig condenser, through the outer tube of which a stream of cold water is passed. Boil the mixture: the water is driven off as steam, which passes into the condenser, where it is liquefied, and the pure water produced runs into the flask placed to receive it.

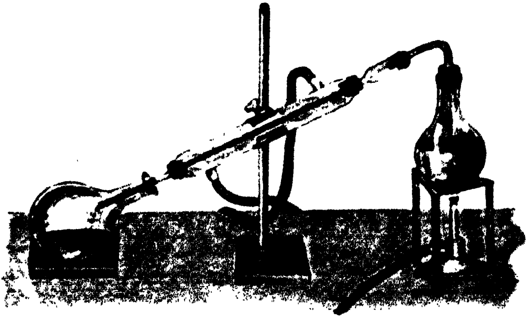


Fig. 30.

The salt remains in the heated flask. This process is called *distillation.*

Exp. 19.—Mix intimately about equal portions of sugar and sand. To separate the mixture into its constituents again, first place the mixture in a small beaker, and add water as long as any diminution occurs in the bulk of the solid. Fold a small filter paper, place it in a small glass funnel and pour on the clear liquid from the insoluble sand. Collect the filtrate in a small basin and evaporate it almost to dryness on a tripod. When almost dry, allow the basin to cool. Crystals of sugar will separate out. Now wash the sand by decantation several times with a little water, and finally pour it upon the filter paper. Place the funnel, filter and sand in a water oven until dry. The sand can then be removed. Thus by combining the processes of *solution, filtration, and crystallization*, we have recovered our original substances sugar and sand from the mixture.

CHAPTER V.

SIMPLE QUANTITATIVE EXPERIMENTS.

On Weighing.—In performing the experiments to be described in the following pages, we shall find it necessary to weigh accurately some of the substances which take part in the reactions. The quantities used are, as a rule, small, hence the need for a more delicate balance than those used hitherto. A beam is poised at its centre on a knife-edge, and has at each end a similar knife-edge on which is balanced a pan. When the balance is not being used the beam and pans are supported on rests, but when we wish to weigh, we place the object to be weighed on one pan and the weights on the other, and by *gently* turning a metal axle (generally provided with a milled head

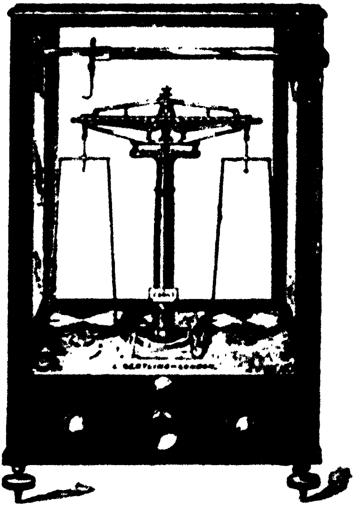


Fig. 31.

and placed beneath the pillar of the balance) cause the long needle attached to the beam to swing across a small ivory scale. If the needle swings to points at equal distances from the zero of the scale, the object and the weights are of equal mass, and the process of weighing has been accomplished. Our first business is to see that the needle swings equal distances from the zero of the scale when the pans are unloaded; to ensure which the screw at the end of the right arm of the beam must be adjusted if necessary. (In some balances a vane is attached to the needle, by moving which to the right or left weight is added to the corresponding portion of the beam.) As a rule this adjustment should only be made by one with some practical knowledge of the balance. Having proved the balance to be in equilibrium about its zero reading, we place the object to be weighed on the left pan, take the largest weight from the box, and place it on the right pan (using a pair of forceps). Next we *slowly* turn the axle and allow the beam to swing. The needle probably moves at once to the left and shows no tendency to swing back. This indicates that the right pan is too heavy. Try the next weight, replacing the first. At length we shall find a weight which is too light, the one before it having been too heavy. Keep the light weight still on the pan and place beside it the next smaller one. If these are too heavy, remove the smaller one and try still smaller ones in succession. We shall gradually work down from the grammes to the decigrammes, then to the centigrammes and finally to the milligrammes. As we get to the smaller weights, we must allow the needle to swing once or twice to each side before taking the readings. (If we read first, say on the left, we must take the *next* reading on the right.) At last we shall find that a certain weight on the right pan is too large, whilst a milligramme less makes it too small.

The actual weight obviously lies between these, but for our purpose it will be sufficiently accurate to take the weight which gives the nearest approach to equal swings on each side of the zero. Now we count up the gramme weights on the pan, write the total down, then the decigrammes, centigrammes, etc., in order. Having written down the weight of the object, we restore the weights to the box, checking them over to see if the total agrees with the weight we have noted down. This precaution is very necessary, especially to beginners—it prevents many erroneous weighings which would simply undo all the other work of the experiments. Articles with any liquid attached to them should never be placed on the pans; loose substances should always be weighed in glass vessels, *e.g.* beakers, watch glasses, etc.; no crucible, tube, etc., should be weighed when hot. It is advisable to have a small camel-hair brush for removing dust from the pans and weights. A pair of forceps is usually provided with

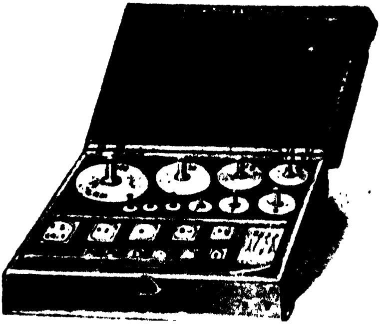


Fig. 32.

box of weights for lifting them from the box to the pan, etc. Weights should never be lifted with the fingers. As the student becomes more practised he will be able to save much time in weighing, by choosing a weight somewhere near that of the object instead of working down from the largest in the box.

Determination of the amount of water of crystallization contained in copper sulphate.—Take about 20 grammes of ordinary copper sulphate crystals ("blue vitriol"), powder in a mortar, and then dissolve in the least quantity of boiling water. Filter the hot solution through a small filter paper placed, if possible, in a funnel surrounded by a steam jacket. Allow the clear liquid to run into a clean porcelain basin surrounded by cold water, and stir it rapidly with a glass rod. Fine crystals will separate out. Pour off the mother liquor and place the fine wet powder on a watch glass inverted in a small funnel, in order to drain off as much of the liquid as possible. (This can be greatly accelerated by connecting the end of the funnel with a small water pump.) Press the drained crystals between blotting paper, turning them frequently. Change the blotting paper and after pressing well and turning, leave in a warm room for some hours. Heat a small porcelain crucible, cool it in a desiccator, and weigh. Add now about half a gramme of the fine crystals, and weigh carefully again. The difference between the weighings gives us the amount of crystallized copper sulphate taken. Place the crucible and contents in an air oven provided with a thermometer, and by regulating the burner, heat up the oven until the thermometer remains fairly steady at about 110°C. After it has remained at this temperature for about half an hour, raise the flame of the burner until the temperature is about 250°C. Keep it fairly steady between 250° and 260° for about twenty minutes. Take out the crucible, place it in the desiccator for fifteen minutes and weigh again. Place again in the oven, which should be still *at 250—260° C. and heat for* another quarter of an hour, cool as before, *and weigh. If* the two weighings do not differ by more than 1 or 2 milligrammes, we can assume that all the water has been driven off. The difference between the second and last weighings gives us the amount of water contained in the weight of sulphate taken.

1.	Weight of crucible	8·672 grms.	
2.	,, ,, ,, +$CuSO_4.5\,H_2O$	9·183 ,,	
	Copper Sulphate taken		·511 grms.
3.	,, ,, ,, +$CuSO_4$	8·998 ,,	
4.	,, ,, ,, ,,	8·997 ,,	
	Water driven off	·186 ,,	

511 parts contain 186 of water ∴ 100 contain 36·4. (Theoretically 100 should contain 36·0).

Determination of the amount of iron contained in ferrous ammonium sulphate.—When certain crystalline bodies are dissolved together in water and allowed to crystallize out again, it is possible to obtain crystals of a substance containing equal numbers of molecules of the respective substances originally taken. These complex bodies are often termed "double salts." Ferrous ammonium sulphate is such a double salt, $Fe(NH_4)_2(SO_4)_2.6\,H_2O$, formed by the union of ferrous sulphate, $FeSO_4.7\,H_2O$, and ammonium sulphate, $(NH_4)_2SO_4$, with the elimination of one molecule of water. Take about 50 grammes of good ferrous sulphate crystals and 25 grammes of ammonium sulphate—mix well and powder in a mortar, then dissolve in the least possible quantity of boiling water. Filter into a clean basin, observing the same precautions as in the previous experiment, drain the fine crystals and dry between blotting paper as there described. The substance, if kept in a well-stoppered bottle in the dark, keeps much better than ferrous sulphate.

Heat, cool, and weigh a small porcelain crucible and lid. Add about ·3—·4 of a gramme of the dry substance prepared above and weigh exactly again. The difference is the weight of sulphate taken. Place the crucible on a pipeclay triangle, so adjusting the lid that there is a very small opening between it and the edge of the crucible. Bring under it a small Bunsen flame fixing the triangle about 6 in. above. Heat the contents of the crucible gradually, so that no spurting occurs from the sudden expansion of the water in the crystals. When the greenish colour has disappeared, being replaced by a dirty white, increase the supply of gas slightly; white fumes will be slowly evolved, due to the escape of the ammonia and sulphur trioxide. On more strongly heating the white colour will be replaced by a brown, showing that the ferrous sulphate is being decomposed, leaving reddish brown ferric oxide. Heat up to bright redness, remove the lid so that the contents of the crucible can become thoroughly oxidized, and continue the heating for about ten minutes. Cool and weigh. Repeat the heating for another ten minutes, cool and weigh again. (Of course the lid should also be strongly heated in order to decompose any sulphate that may have been spurted on to it.) If the weighings do not differ by more than a milligramme or so, we can assume that the experiment is completed.

The difference between the first and final weighings gives us the amount of ferric oxide present. $\frac{7}{10}$ths of this is the amount of iron contained in the sulphate. It should amount to 14·2 theoretically.

$$4Fe(NH_4SO_4)_2.6H_2O+O_2=2Fe_2O_3+8NH_3+8SO_3+10H_2O.$$

1.	Weight of crucible + lid		14·562 grms.	
2.	,, ,, ,,	,, + sulphate	15·094 ,,	
		Sulphate taken		·532 grms.
3.	,, ,, ,,	,, + Fe_2O_3	14·668 ,,	
4.	,, ,, ,,	,, ,,	14·670 ,,	
		Ferric oxide		·108 grms.

Amount of iron present = $\frac{7}{10}$ × ·108 = ·075 grms., *i.e.* 14·1 %.

Determination of the amount of silver contained in silver nitrate.—Take a bulb tube of combustion glass similar to that described on p. 2, about six inches in length and about $\frac{3}{8}$ of an inch in diameter. Wash it out carefully, and dry it. (This can be done by heating it, and slowly sucking a current of air through by means of a straight piece of narrow glass tubing.) Take some crystals of silver nitrate, powder them, and press well between blotting paper to remove any water which may be included. Now carefully weigh the bulb tube, and add about 1·5 grammes of the nitrate, taking care that none remains attached to the outside of the tube. Weigh accurately again; the difference is the amount of nitrate taken. Wrap a piece of filter paper round the upper part of the tube, and fix the tube at this part in a clamp stand. So arrange it that the tube is in a sloping position. Bring a small Bunsen flame under the bulb (where the nitrate should be) and gradually heat it. The substance will first melt to a clear liquid and then simmer, giving off red fumes of nitric peroxide and oxygen. So regulate your supply of heat that the decomposition goes on quietly and without any violent spitting. The liquid will gradually become turbid owing to the deposition of metallic silver.

$$2\,AgNO_3 = 2\,Ag + 2\,NO_2 + O_2.$$

Finally a white crust of silver will adhere to the bulb;

heat it more strongly to be sure of complete decomposition, and then carefully heat the upper portions of the tube to convert any spittings of nitrate into metal. Allow to cool and weigh carefully. To make certain that no nitrate remains unchanged, heat the tube thoroughly, cool, and weigh again. There should be practically no difference between the two weighings.

The difference between the first and final weighings gives the weight of silver present.

1. Weight of tube		10·372 grms.	
2. ,, ,, ,,	+nitrate	11·984 ,,	
	$AgNO_3$ taken		1·612 grms.
3. ,, ,, ,,	+silver	11·398 ,,	
4. ,, ,, ,,	,,	11·397 ,,	
	Silver obtained		1·025 grms.

1·612 of nitrate contains 1·025 of silver $\therefore$ 100 contain 63·6. Theoretically 100 parts should contain 63·5 of silver.)

Determination of the amount of carbon dioxide contained in calcium carbonate.—When calcium carbonate is strongly heated in a current of air it loses carbon dioxide and becomes converted into calcium oxide or lime. This fact is made use of in the conversion of limestone into lime in an ordinary lime-kiln. We can also apply it to the estimation of the percentage weight of carbon dioxide in a specimen of pure carbonate. Take a small porcelain crucible and lid (platinum is better if it can be had), clean thoroughly, and heat to redness on a pipeclay triangle over a Bunsen flame. Remove the flame and allow to cool partially. While still warm, transfer by means of a clean pair of tongs to a desiccator and allow to cool completely for fifteen minutes or longer. Weigh accurately, and bring into the crucible about half a gramme of pure precipitated calcium carbonate, heat gently over the Bunsen flame, but not to perceptible redness, to remove moisture, cool as before in the desiccator and weigh accurately again. The difference gives us the weight of carbonate taken. Now place the crucible on a triangle, with the lid almost covering the mouth, but leaving a small opening for the escape of carbon dioxide, heat it up to redness with a Bunsen flame, and then turn on a small blowpipe flame. Gradually increase the supply of gas until you have the hottest flame possible, and heat with this for a quarter of an hour, then slowly diminish the flame and allow the crucible to cool down gradually. Transfer to the desiccator, cool for fifteen minutes, and again weigh accurately. The contents will have diminished in weight owing to the loss of carbon dioxide, but may still contain undecomposed carbonate. To test whether this is the case or not, heat exactly as before but only for five minutes, cool, and weigh again. If the further loss is only a milligramme or so we can conclude that all the carbonate is practically decomposed. The difference between the final weighing, and the weight of the crucible, lid, and contents, gives us the weight of carbon dioxide contained in the weight of carbonate taken. Calculate what weight 100 parts of carbonate would contain. It should be 44.

$$\underset{40+12+48}{CaCO_3} = CaO + \underset{44}{CO_2}.$$

Determination of the amount of carbon dioxide contained in sodium carbonate.—Many carbonates cannot be decomposed by heat at all readily, and consequently demand for their analysis some method differing from that adopted in the last experiment. Sodium carbonate is one of the most typical and best known of these. Take a small flask holding 150—200 c.c. and having a fairly wide mouth. Fit it with a good cork or indiarubber stopper. Take one of those straight bulb tubes known as calcium chloride tubes, fill it with pieces of granulated calcium chloride, and fit the wider end with a small cork perforated by a short piece of glass tubing. This latter should be closed by a short piece of indiarubber tubing containing a piece of glass rod, so that the calcium chloride can be prevented from picking up moisture from the air when the apparatus is not in use. Bore two holes in the cork selected for the flask: one to take the narrow end of the bulb tube, the other to take a short straight piece of quill tubing closed by a small piece of cork. (A boring does very well.) Take a very short test tube—of such a length that it will readily go into the flask—and fasten round the open end a piece of thread. Place in the flask about 50 c.c. of dilute sulphuric acid (about one of sulphuric acid to four of water), replace the cork with its two tubes and indiarubber cap, place the dry test tube with its thread on the balance pan with the flask, and weigh carefully. Now pick out some bright crystals from some washing soda, introduce about one gramme into the test tube, and by passing the thread through the short tube in the cork of the flask, allow the tube to dangle just out of contact with the acid. Fix the thread in the tube by means of the small cork (which should fit tightly) and weigh the whole apparatus again. The difference in weight gives us the amount of soda crystals taken. Now remove the apparatus from the pan and take off the indiarubber cap. Remove the small cork and lower the tube slightly in the flask replacing the cork. By tilting the flask and gently shaking we can gradually decompose our carbonate by bringing small quantities of acid to it. The moist carbon dioxide has all to pass through the drying tube, which prevents loss in weight from the escape of moisture. Hence all we lose is carbon dioxide. When no further decomposition ensues on addition of the acid, lower the tube bodily into the acid, replace the cork, and gradually heat the liquid almost to boiling on a tripod over a Bunsen burner. This will liberate any gas dissolved by the liquid. To the end of the calcium chloride tube attach a long piece of indiarubber tubing and, after removing the little cork, *gently* draw air through the apparatus to displace the carbon dioxide. Shut off the calcium chloride, by means of the indiarubber cap, from communication with the outside air, allow the apparatus to cool, replace the cork, and weigh again. The difference between the second and final weighings gives us the amount of carbon dioxide contained in the carbonate.

1. Weight of flask + tubes	56·562 grms.	
2. ,, ,, + crystals	57·583 ,,	
Weight of crystals taken		1·021 grms.
3. Weight of flask + tubes + crystals — CO_2	57·123 ,,	
Weight of CO_2		·160 grms.

1·021 of $Na_2CO_3.10H_2O$ contain ·160 of CO_2. ∴ 100 would contain 15·6

(Theoretically it should contain 15·3 °/ₒ of CO_2.)

This method can also be used with calcium carbonate, using hydrochloric acid; lead carbonate, using nitric acid, etc.

Determination of the equivalent of magnesium by weighing the hydrogen evolved. The equivalent of magnesium may be shortly defined as that weight, which will replace unit weight of hydrogen in one of its compounds.

A wide-mouthed weighing bottle of thin glass is fitted with a rubber stopper and tubes, as shown in the figure, and about 5 c.c. of water run into it. One of the tubes is simply bent at right angles, and closed with a short piece of rubber tubing and glass rod. The other is furnished with a bulb lightly packed with dry fibrous asbestos or glass wool, and concentrated sulphuric acid is introduced into its lower part, as shown, by dipping it up to its orifice, in a test tube containing the acid. The outside is then rinsed with water (from a wash-bottle), so as to remove any acid adhering to it. A quantity of magnesium (not more than 0·2 gramme) is accurately weighed and dropped into the bottle, and the indiarubber cork fitted into its place. The whole apparatus is then weighed.

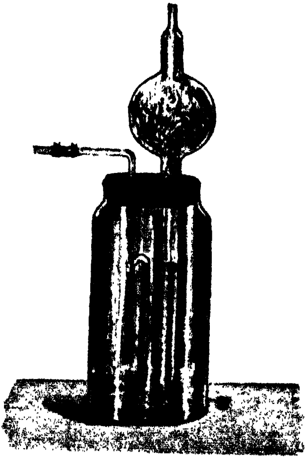


Fig. 33.

Momentarily removing the glass rod, blow gently down the bulb tube till about half the acid is expelled, and quickly replace the glass rod. The acid is acted upon by the metal, hydrogen is evolved and passes out by the only exit left for it, viz. the bulb tube; the concentrated acid still remaining there serves to dry the gas. When the metal is all dissolved, remove the glass rod and draw a gentle current of air through the apparatus, so as to remove any hydrogen in the bottle, replace the rod, and again weigh. A decrease of weight will be found to have occurred, owing to the discharge of hydrogen from the apparatus, *e.g.*

Weight of apparatus before reaction 35·534 grammes.
" " after " 36·520 "

Loss of weight 0·014 "

This is the weight of the hydrogen evolved. The weight of the magnesium taken was 0·168 gramme. The equivalent of magnesium is then—

$$\frac{0·168}{0·014} = 12$$

The equation representing the reaction is

$$Mg + H_2SO_4 = MgSO_4 + H_2.$$

Determination of the equivalent of magnesium from the volume of hydrogen evolved.—Take a long measuring tube closed at one end and graduated in c.c. One graduated up to 100 c.c. is preferable. Take a narrow beaker, pour in about half a litre of water and add about 30 c.c. of concentrated sulphuric acid, stirring briskly. When cool, fill up the tube completely with the acid, taking care that no air bubbles are allowed to enter, close the open end by means of the thumb and invert in the beaker, which contains the greater portion of the diluted acid. Fix the tube in a small clamp stand. Draw out a piece of soft glass tubing of about ⅜ in. diameter (it should slide comfortably in the graduated tube) in the blowpipe flame and round off the end. Cut it off at about 1¼ in. from the closed end and round off the edges. Next scrape a piece of magnesium ribbon to remove all oxide, and weigh out a piece of about ·1 gramme (it should not be more). Drop the coiled ribbon into the small tube and fill up with water, shaking well, if necessary, to disengage any air bubbles. Close the end by means of the thumb and bring it under the mouth of the graduated tube (which is still under the dilute acid). Force the small tube up into the wider one and lower the latter until it almost touches the bottom of the beaker and completely encloses the other. Remove your hand and rinse off the acid. Soon the heavier acid will diffuse to the magnesium and begin to dissolve it. Hydrogen will be evolved and will be collected in the tube.

When the magnesium has completely dissolved, adjust the tube so that the liquid inside is level with that outside, more water being poured into the beaker if necessary. Do not touch the tube with your hand but let it remain in the clamp, upon which hang a thermometer close to the tube in order to read the temperature of the air in the neighbourhood of the gas. In a few minutes the temperature of the hydrogen will be sensibly the same as that indicated by the thermometer. Take a reading also, of the height of the barometer. Now carefully measure the volume of the gas, raising the tube (still in the clamp) until the liquid in it is slightly above that outside. Having thus read the volume, temperature, and pressure of the *wet* gas, we can deduce the weight of *dry* gas, *i.e.* the hydrogen in the manner indicated below.

The volume of a moist gas having been measured at a given temperature and pressure it is necessary to first find what volume the *dry* gas would occupy at 0°C. and 760 m.m. pressure. Allowance must be made

(*a*) for the temperature of the gas (*i.e.* of the room);
(*b*) for the pressure to which it is subject (that of the atmosphere at the time of the experiments);
(*c*) the tension of the water-vapour.

This involves a somewhat complex calculation, and a table has therefore been drawn up to enable the student to make the correction by using the factor given in the table.

Thus, suppose the temperature of the laboratory to be 10° C. and the atmospheric pressure 740 m.m.; the tension of aqueous vapour with which the gas is saturated is for 10° C., 9·1 m.m. Now if v is the volume of the moist gas as observed, then V the volume of the dry gas at 0° C. and 760 m.m. pressure is given by the expression—

$$V = \frac{v \times 273 \times (740 - 9{\cdot}1)}{283 \times 760} = 0{\cdot}928\,v.$$

bserved volume of gas, 120 c.c.
he factor under 16° C. and 750 m.m. is 0·0823.

$$\text{Weight of hydrogen} = \frac{0·0823 \times 120}{1000} = 0·00988 \text{ grm.}$$

Determination of the equivalent of zinc.—Use the same apparatus as above and proceed in exactly similar manner. Weigh about ·2 gramme of bright zinc foil, roll it up and place it in the short tube full of water and introduce as before into the measuring tube.

$$Zn + H_2SO_4 = ZnSO_4 + H_2.$$

Determination of the equivalent of iron. Dilute hydrochloric acid will be found to act more rapidly than sulphuric in this experiment. Weigh out about ·2 gramme of fine iron wire which has been scrubbed with a piece of sand paper to remove rust, and follow the same method as in the magnesium experiment.

$$Fe + 2\,HCl = FeCl_2 + H_2.$$

Indirect determination of the equivalent of silver. In the above experiments we have directly measured the quantity of hydrogen replaced by a given weight of metal, and from the two numbers calculated the amount of metal equivalent to one gramme, or unit weight in any system, of hydrogen. In some cases such a method is impossible because the metal will not act on the acid and evolve hydrogen. Then we have recourse to indirect methods, such, for instance, as the following. Silver does not cause evolution of hydrogen from the common acids, but magnesium does; we can therefore determine the equivalent of magnesium. Now magnesium can readily replace silver in silver nitrate, giving free silver and magnesium nitrate.

$$2\,AgNO_3 + Mg = Mg(NO_3)_2 + 2\,Ag.$$

Obviously, if we can determine the weight of silver liberated by the equivalent of magnesium, we shall have the equivalent of silver.

Take a large porcelain crucible—one that will hold 25 c.c. comfortably—wash it carefully, dry it in the hot water oven, and allow it to cool. Place it on one pan of the balance and on the other a light, small beaker. Into the beaker drop shot, one by one, until the counterpoise is just too heavy; remove one shot and place instead small pieces of paper until the counterpoise is just equal in weight to the crucible. Now take about two grammes of silver nitrate crystals, dissolve in 20 c.c. of distilled water and place the solution in the crucible (now removed of course from the balance pan). Put the counterpoise aside for a short time and weigh out accurately somewhere about ·1 gramme of bright magnesium ribbon. The weight of metal need not be precisely ·1 gramme, but whatever it is, it should be accurately known. Place the ribbon in the solution—it will soon become coated with a grey deposit of metallic silver. Stir it about with a short glass rod in order to loosen the silver, which will fall to the bottom of the vessel, and expose a fresh surface of magnesium to the action of the silver nitrate. Do not remove the rod from the crucible until the end of the experiment. Gradually the magnesium will disappear and be replaced by a greyish powder. By gently tapping the sides of the crucible we can get this to collect on the bottom, the clear solution of the nitrates remaining above. Carefully pour this off from the silver, down the glass rod, into the bottle kept for silver residues. (If there was silver on the glass rod it should have been rinsed back into the crucible by a few drops of distilled water from a wash-bottle). Fill up again with distilled water, stir well, and again allow the precipitated metal to settle, tapping the crucible gently. Pour off the supernatant liquid, taking care that no metal is lost. This washing operation should be repeated five or six times in order to remove all silver and magnesium nitrates from the metal. Finally wash with a little rectified spirits, also removing all solid matter from the outside of the crucible. Dry in the water oven for twenty minutes, and gradually heat the crucible to redness on a pipeclay triangle, over a Bunsen flame, a lid being placed over it to throw back any spittings. Allow to cool, remove the lid and place on the balance pan. On the other pan place the counterpoise and determine the extra weight needed to bring the two pans into equilibrium. This will be the weight of silver displaced by the known weight of magnesium taken. The problem is to find the weight of silver replaced by the equivalent weight of magnesium. The equivalent of magnesium is 12·1—by a simple sum in proportion we can find from the above data the weight of silver it corresponds to. This is the weight of silver which would replace unit weight of hydrogen in any acid with which it reacted.

Indirect determination of the equivalent of copper.—Copper, again, is a metal which does not cause evolution of hydrogen from the common mineral acids. We must adopt a method similar to that described in the above experiment. Weigh accurately a rather deep porcelain crucible of about 50 c.c. content. Into this bring about 40 c.c. of a solution containing not less than 2 grammes of copper sulphate, and then about 0·15 gramme of magnesium ribbon, the exact weight of which has been determined. The magnesium will slowly disappear, and a heavy powder will be precipitated to the bottom of the vessel. When on stirring with a glass rod there is no longer any sign of the foil, the reaction is complete. The magnesium has replaced the copper in the salt originally taken, and we have now, in the vessel, metallic copper and a solution containing magnesium sulphate and excess of copper sulphate. Now place the porcelain crucible in the centre of a basin of about the same depth, and direct a *very gentle* stream of water on the surface of the liquid, as shown in the figure below. Adjust the stream so that little if any of the particles are so disturbed as to be carried out of the crucible, and allow it to continue some minutes and

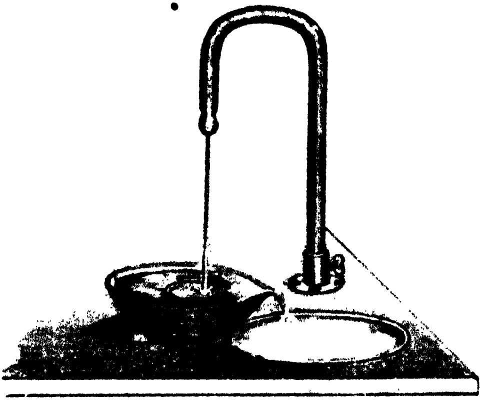


Fig. 34.

so long as the faintest trace of blue colour is to be seen. On adding ammonia to a little of the liquid in a test tube the blue colour is intensified, and if then, on looking down the tube, the colour is no longer apparent, the washing may be discontinued. Now take out the crucible, pour off the liquid carefully, so as to avoid the loss of any copper, and if some granules are found at the bottom of the basin, pour off the liquid in it and wash these into the crucible. After the water has been drained away as completely as possible from the copper, add a few cubic centimetres of alcohol, pour off again and repeat this washing with alcohol once or twice more. Now place the crucible in a water oven, allow it to remain till quite dry, cool, and then weigh it. The increase in weight will represent the amount of copper it contains.

We have now (*a*) the weight of magnesium used, (*b*) the weight of copper which this magnesium has displaced, and if the experiment has been carried out with due care these weights will be found to show very nearly the relation 12·1 : 31·6. Thus if 12·1 be accepted as the equivalent of magnesium, 31·6 must be taken as that of copper.

An experiment of the same kind may be performed with metallic iron and copper sulphate.

Direct determination of the volume of oxygen contained in a weighed amount of potassium chlorate.—Select a thick-walled bottle with a fairly wide mouth, and of about

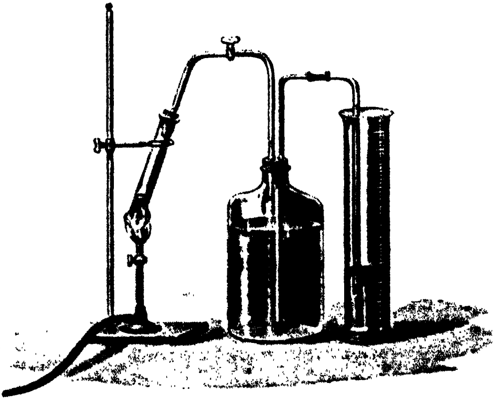


Fig. 35.

one litre capacity. Fit it with a good sound cork, previously softened. Take a piece of narrow quill tubing of suitable length, and bend it twice at right angles so as to make three sides of a rectangle; one side should be slightly longer than the bottle, and the one parallel slightly less. Take a piece of the same tubing about 6 in. long and bend it in the middle at an obtuse angle. Round the ends of the tubes in the flame of the burner, and perforate the cork in two places. Through one hole pass the longer limb of the first tube until, when the cork is pushed home, the end almost touches the bottom of the bottle. Through the other hole push the short tube until the end just appears below the cork. To the other end of this latter tube attach a short piece of sound indiarubber tubing, fitted with a brass clip by means of which it can be completely closed. We have yet to make the vessel in which to heat our potassium chlorate. It must be of fairly infusible glass in order to stand the temperature needed for the complete decomposition of the substance. The glass known as "combustion" tubing is convenient. Heat a piece about 18 in. long at the middle in the hottest blowpipe flame, and when fairly soft draw it rapidly apart. Snap it in the middle and take one portion; where it tapers most rapidly, soften it and draw off the long narrow portion, and seal up the end. Now heat this thoroughly in the hottest flame, and blow it out into a small bulb. When cool wash out carefully and dry, fit with a good cork, and bend a piece of quill tubing at a right angle, rounding the ends. Bore the cork and insert the glass tube so that the end just appears below the cork. Before proceeding to weigh out the potassium chlorate we must test our apparatus in order to see whether it is quite tight. Fill the bottle completely with water, clip the indiarubber tube so that it is closed, and carefully force in the cork. As it is pushed into the bottle, water will be forced out of the tube which projects. Now connect the indiarubber tube with the glass tube of the bulb, having previously pushed in the cork until it was quite tight, Release the clip, so that there is a perfectly free passage. If the apparatus is quite tight, little or no water should run out of the side tube. We shall suppose that all is in perfect working order. Replace the clip and remove the bulb gently from the cork. Shake the bulb to be quite sure that no particles of cork have fallen into it, and carefully weigh it. Introduce about 1·5 grammes of crystals of potassium chlorate, and weigh again. The difference gives the exact weight of substance we have taken. Replace the cork carefully in the bulb tube and release the clip from the indiarubber. Observe that the apparatus is quite air-tight, and place under the tube projecting from the bottle a tall graduated measuring cylinder of 500 c.c. content so as to catch the fluid expelled. Now gradually heat the chlorate contained in the bulb, by means of a Bunsen flame—not too strongly at first. The bulb is better fixed in a small clamp stand so as to slope slightly upwards. The chlorate will first fuse, and then give off bubbles of oxygen. Keep the liquid gently simmering until very little more water is expelled from the bottle, and then heat it more strongly. The side tube should now dip some distance below the surface of the water in the graduated cylinder. When no more liquid is expelled, even on strongly heating, the flame can be gradually lowered, and finally removed altogether, and the apparatus allowed to assume the temperature of the room. When the level of the liquid in the cylinder is practically stationary, we carefully adjust the cylinder so that the water in it is level with that in the bottle, observe the volume of water and read the temperature of the surrounding air by means of a thermometer hung near. The height of the barometer must also be read to give us the pressure, and the aqueous tension for the observed temperature noted and deducted. The volume of water read off, which is of course the volume of oxygen expelled, can then be corrected to 0°C. and 760 m.m. pressure in the usual manner (see the section on the equivalent of magnesium, p. 116). From our data we can calculate the volume of oxygen measured at 0°C. and 760 m.m. evolved by 122·5 grammes of $KClO_3$. It ought to be about 33·5 litres.

$$2\,KClO_3 = 2\,KCl + 3\,O_2$$
$$2 \times 122{\cdot}5 \qquad\qquad 3 \times 22{\cdot}32 \text{ litres.}$$

On acids, alkalies, and salts.—*Acids* may be broadly defined as those substances which contain hydrogen which is replaceable by a metal. Thus hydrochloric acid when treated directly with zinc, for example, gives off its hydrogen, the place of which in the acid is now occupied by the zinc, giving us zinc chloride. Many acids, however, are too weak to give off hydrogen and dissolve the metal when treated directly with it; the hydrogen in them *can* be replaced by metal, but indirectly. If we present the metal to such in the form of an oxide, we can in many cases get it to dissolve, the hydrogen of the acid uniting with the oxygen of the oxide as water, the metal combining with the remaining part of the acid. A reaction of this nature is seen in the following equation.

$$PbO + 2\,HCl = PbCl_2 + H_2O.$$

Even weak acids can often be recognised as such by their action on certain colouring matters. A solution of litmus for example is turned a bright red by most acids. Pour a drop or two of hydrochloric, nitric, and sulphuric acids into separate beakers, dilute considerably, and add a drop or two of neutral litmus solution to each. A bright red colouration will result in each case.

Bases may be defined as those substances which react with acids to produce water, the hydrogen of the acid being replaced by metal. They are the oxides and hydrates of the metals. Many of them form insoluble compounds with certain acids, and consequently appear to have no action on them. Before deciding that a substance is *not* a base, we must therefore try it with several acids. We can recognise the presence of a base again, in many cases, by the action with certain colouring matters like litmus. Only those which are soluble, or slightly soluble, in water exhibit this property. This action is shown also by certain salts in which we have what is called a strong base united with a weak acid, *e.g.* sodium carbonate. All these substances can, when dissolved in water, turn red litmus blue, and are said to possess an *alkaline* reaction. Pour a few drops of caustic soda solution, NaOH, limewater, $Ca(OH)_2$, and sodium carbonate solution, Na_2CO_3, into separate beakers, and add a few drops of red litmus solution to each. The solution will be immediately turned blue.

Finally, *salts* are the compounds produced by the union of an acid with a base. They may be regarded, quite as correctly, as acids in which the hydrogen has been replaced by a metal. Acids turn litmus red; bases restore the blue colour; salts have no action on litmus —they are said to be neutral. Take about a quarter of a stick of solid caustic soda, dissolve it in about 20 c.c. of water in a beaker, and add a few drops of litmus. Pour about 20 c.c. of concentrated hydrochloric acid into a small flask and add about twice its volume of water; mix well, and pour it gradually into the sodium hydrate solution. By cautious addition of the acid, the blue colour of the litmus can be made to merge into a purple—neither red nor blue. The alkali will then be completely neutralised by the acid and we shall have a salt, neither free acid nor alkali being present. Pour the neutral liquid into a small porcelain basin and evaporate to dryness on a tripod. We shall obtain a white solid, coloured slightly by the litmus.

Taste it. Do you recognise it? Compare the taste with that of a little *dilute* caustic soda solution. Is there any difference? Scrape out a little of the solid substance into a dry test tube, pour a few c.c. of strong sulphuric acid upon it, and warm gently. What are the white fumes produced? Would caustic soda give them when similarly treated?

The reaction which has taken place is represented by the following equation:—

$NaOH$	$+$	HCl	$=$	$NaCl$	$+$	$H_2O.$
Sodium hydrate		Hydrochloric acid		Common salt		

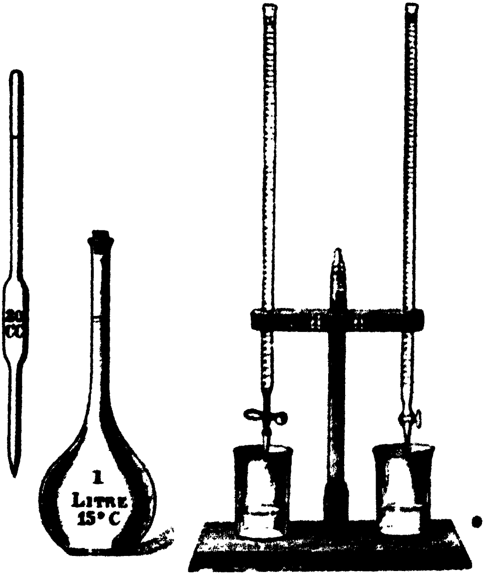


Fig. 36.

In order to carry out the following

the graduated vessels shown in Fig. 36, viz., a pipette, a stoppered flask and two burettes.

The amount of a given acid required to neutralise a fixed weight of a certain alkali is also fixed. This being the case, if we take double the amount of alkali, we should require double the amount of acid to completely neutralise it. Take a stoppered measuring flask, graduated to contain 250 c.c. when filled up to a certain mark on the neck, and counterpoise it on the balance. On the pan which supports the counterpoise, place in addition weights making up 16 grammes; we wish to add to the flask approximately 16 grammes of nitric acid. Clean and dry a 10 c.c. pipette, suck up into it pure nitric acid to the mark. (Care must be taken that no acid can enter the mouth. It is advisable to attach a long piece of india-rubber tubing to the end of the pipette and suck from the end of that.) Remove the flask from the balance and add the acid; replace and see if the acid weighs enough—it should not. Remove the flask and add a few more drops of acid. When about 16 grammes of acid have been taken, weigh exactly. Then fill up the flask with distilled water to the mark and mix thoroughly. We have now 250 c.c. of dilute acid containing a known weight of pure nitric acid; each c.c. consequently contains a known weight of acid. Now take a stick of solid caustic soda, dissolve it in about 250 c.c. of water—there is no need to measure it very exactly—and stir the solution thoroughly. Measure out, by means of a 10 c.c. pipette, 10 c.c. of the soda solution into one beaker, and 20 c.c. into another. Note that in measuring out liquids from pipettes, the last drop should not be blown out, but allowed to run out by holding the nozzle of the pipette on the side of the beaker for a few seconds.) Fill up a burette, with the acid prepared as above, to the zero mark, and see that the nozzle below the tap is quite full of liquid. To the 10 c.c. of caustic soda solution taken, add enough litmus to give it a distinct blue colour, and then add the acid from the burette gradually until the blue colour becomes a purple, indicating that the alkali has been neutralised. After a little practice the student will be able to tell when this point is being approached, and he can then add the acid drop by drop. Carefully read the level of the liquid in the burette—it gives us the volume of acid added. Now repeat the process with the 20 c.c. of soda solution. As double the volume of solution is taken, it will contain double the weight of soda, and consequently should require double the amount of acid to neutralise it. When the purple colour is obtained, subtract the second reading of the burette from the third; the difference is the volume of acid added. It should be double the first volume taken. In reading the level of the liquid it is advisable to take the lowest point of the curved surface.

From the volume of acid of known strength required to neutralise a given volume of alkali solution we can determine the strength of the latter. By the strength of a solution of acid or alkali, we mean the weight in grammes of the pure acid or alkali contained in one cubic centimetre of the solution. If we know the strength of the acid and the number of c.c. of it taken, we can, by multiplying, determine the total weight of *pure, undiluted* acid contained in that volume. If we know the equation which represents the reaction we can calculate from the weight of pure acid taken, the weight of pure alkali needed to neutralise it. Now this weight of alkali is contained in the number of c.c. of *solution* of alkali taken; if we divide this weight by the number of c.c., we consequently obtain the weight of alkali contained in one c.c. of solution, which is its strength.

To make the above reasoning clearer let us consider the experiment described in the foregoing paragraph. Suppose that our 10 c.c. of soda solution required for neutralisation 9·2 c.c. of the nitric acid solution, and the 20 c.c. required 18·3 c.c. Then 30 c.c. of soda needed 27·5 c.c. of acid. Now 250 c.c. of acid solution contained, say 16 grammes of nitric acid, the remainder being, of course, water; 1 c.c. of solution therefore contains $\frac{16}{250}$ grammes of nitric acid, 27·5 c.c. will contain $27{\cdot}5 \times \frac{16}{250}$ grammes. According to the equation of the reaction,

$$\underset{63}{HNO_3} + \underset{40}{NaOH} = \underset{85}{NaNO_3} + \underset{18}{H_2O},$$

63 grammes of HNO_3 need 40 grammes of NaOH for complete neutralisation. What weight will $\frac{27{\cdot}5 \times 16}{250}$ grammes require?

$$63 : \frac{27{\cdot}5 \times 16}{250} :: 40 : x, \quad \therefore x = 1{\cdot}117 \text{ grammes.}$$

30 c.c. of caustic soda solution must therefore contain 1·117 grammes of solid caustic soda. 1 c.c. will contain $\frac{1{\cdot}117}{30} = {\cdot}037$ grammes. This is the *strength* of the unknown solution of soda we began with. The example illustrates the fact that by neutralising with solutions of known strength, we can find the unknown strength of a solution of acid or alkali, if we know what acid or alkali we are dealing with.

The determination of the amount of acid in a solution by means of Iceland spar.—Clean a small flask and place in it exactly 50 c.c. of the solution of nitric acid, prepared as directed above, using a 50 c.c. pipette. Pick out a piece of Iceland spar of 5 or 6 grammes weight, and of such a size that it will readily pass down the neck of the flask. Weigh it accurately. Now attach a piece of stout thread to it, and suspend it in the acid. Brisk effervescence will take place, the spar dissolving with formation of calcium nitrate and evolution of carbon dioxide. Most of the particles of liquid thrown up by the effervescence will be reflected into the solution by the sides of the vessel, so there will be very little loss from that cause. The solution should be agitated from time to time to bring fresh portions of acid in contact with the spar. When all effervescence has ceased, take out the spar, remove the thread, wash, and dry between blotting paper, and finally weigh again. The difference between the two weighings gives the weight of spar dissolved. From the equation representing the reaction, we can calculate the amount of nitric acid necessary to dissolve that amount of calcium carbonate.

$$\underset{100}{CaCO_3} + \underset{126}{2\,HNO_3} = \underset{164}{Ca(NO_3)_2} + \underset{18}{H_2O} + \underset{44}{CO_2}.$$

If 100 grammes of $CaCO_3$ need 126 grammes of HNO_3, what weight will x grammes of $CaCO_3$ need? The answer to that gives the weight of nitric acid contained in the 50 c.c. of nitric acid solution taken.

1. Weight of spar before solution 6·984 grms.
2. ,, ,, ,, after ,, 4·450 ,,
,, ,, ,, dissolved 2·534 grms.

100 : 2·534 :: 126 : x. $x = 3·193$ grms. of HNO_3.

The strength of the solution of nitric acid can of course be obtained by dividing x by the volume, viz., 50 c.c. This experiment cannot be applied to determine the amount of sulphuric acid in a solution, because insoluble calcium sulphate is formed in that case, which coats the crystal and protects it against further action.

CHAPTER VI.

INTRODUCTION TO SYSTEMATIC ANALYSIS.

QUALITATIVE Analysis has for its object the determination of the number and nature of the constituents of substances, without reference to their proportions.

Usually it has reference to the examination of *salts*, *i.e.* combinations of *acids* and *bases*, and in such cases the problem involved is the determination of the particular base and acid present in any given substance. Thus we examine substances to determine, not directly what *elements* are present, but what metals and what acid radicles—the latter, of course, being usually groups of elements.

In determining the metal or metals present in a given substance, we submit a portion of the substance to the action of various reagents. These reagents are applied in the dry way, *e.g.* before the blowpipe, or in the wet way, *i.e.* in the state of solution. The latter method is the more important. Usually, the phenomenon which we take advantage of in detecting any substance is the formation of a *precipitate*, when solutions of certain bodies are brought together. The formation of a precipitate, of course, is due to the interaction of the substances in solution giving rise to the production of some new compound which is insoluble, or only slightly soluble, in the liquid present.

Reagents are divided into *group reagents* and *special* The former are those which produce a similar general result with several metals—*e.g.* hydrochloric acid gives a white precipitate with either silver, mercury, or lead, and is, therefore, the group reagent for those metals.

Special reagents are those which we make use of for confirming the presence or absence of a particular metal. In applying reagents to a body to be analysed, it is of the utmost importance that they should be used in a systematic order; otherwise altogether erroneous results may be obtained.

The group reagents usually employed are the following:—hydrochloric acid; sulphuretted hydrogen in presence of hydrochloric acid; ammonia in presence of ammonium chloride; ammonium sulphide in presence of ammonia and ammonium chloride; ammonium carbonate in presence of ammonia and ammonium chloride. These are applied in the above order.

The metals are usually divided into six groups, according to their behaviour with the above reagents.

Group I. comprises those metals whose chlorides are insoluble in water, and which, therefore, are precipitated by hydrochloric acid (or, indeed, by any soluble chloride). They are silver, lead, and mercury (ous).

Group II. comprises *those of the remaining metals* whose sulphides are insoluble in hydrochloric acid, and which, therefore, are precipitated by sulphuretted hydrogen in presence of that acid. They are mercury (ic), lead, bismuth, copper, cadmium, tin, antimony, and arsenic.

Group III. includes *those of the remaining metals* whose hydrates are insoluble in water and in ammonium chloride solution, and which, therefore, are precipitated by ammonia in presence of ammonium chloride. They are iron, chromium, and aluminium.

Group IV. consists of *those of the remaining metals* whose sulphides are insoluble in water, and which, therefore, are precipitated by ammonium sulphide in alkaline solutions. They are nickel, cobalt, manganese, and zinc.

Group V. includes, *of the remaining metals*, those whose carbonates are insoluble in water and ammonium salts, and which, therefore, are thrown down by ammonium carbonate in presence of ammonium chloride. They are barium, strontium, and calcium.

Group VI. comprises *the remaining metals*. They are magnesium, potassium, sodium, and ammonium.

Special notice should be taken of the words "of the remaining metals," which appear so often in the above account. Group IV., for example, does not consist of *all* the metals which are precipitated by ammonium sulphide in alkaline solutions, but of such of these only as are not included in Groups I., II., III.

As we are only concerned with, at most, two metals in a group, and generally only one, our attempts to identify a metal present in one of its salts are much simplified.

After determining the group to which a metal belongs, the next thing is to identify the particular metal present. Advantage is taken of the slight differences in the properties of the similar compounds of different metals existing in the same group—*e.g.* as to colour, solubility in various reagents, etc.—in order to settle this question; and, finally, the answer is confirmed and substantiated by the application of a *special* or *confirmatory* test. In testing for the acid radicles the splitting up of the various acids into groups cannot be so fully carried out, and more importance is to be attached to confirmatory or special reactions.

It must be carefully borne in mind that analysis is founded upon a knowledge of the properties of the metals and acids, and is really an application of such chemical knowledge to the question in hand. The student should, therefore, in all cases endeavour to clearly understand the chemical changes which are produced in the various operations of chemical analysis. To this end he will derive great advantage by making it a rule to express by means of equations the various reactions which occur. These equations should be written in a note-book at the time the experiments are being made in the laboratory.

It will be found that almost all reactions occurring in analysis are examples of metathesis or double decomposition, *e.g.* the interchange of acids and bases—thus barium chloride and sodium sulphate give us sodium chloride and barium sulphate; the latter, being insoluble in water, is thrown down as a white precipitate.

$$Na_2SO_4 + BaCl_2 = BaSO_4 + 2\,NaCl.$$

Such reactions, of course, present little difficulty, but the practice of writing the equations should be adhered to, even in simple cases, as by this means the student acquires a valuable familiarity with the valency of metals and the basicity of acids.

Flame tests.—(1) Place a small quantity of solid calcium chloride, $CaCl_2$, on a watch-glass. Lift a small portion of it with the loop of the platinum wire, and hold in the lower outer part of the Bunsen flame. The flame shows an intense orange-red colour characteristic of calcium compounds. Clean the wire by holding it in the flame until all trace of the orange coloration disappears. Then wash it in dilute HCl and again heat it.

(2) Take some copper sulphate (or any copper salt) and heat it in the same way. The flame is of a bluish green colour, but possibly not very intense. Pour a few drops of HCl into a watch-glass, dip the loop of the wire in it, and take up with the moistened wire a few particles of the salt. When the wire is now placed in the flame the bluish

green coloration will be much more intense. Now clean the wire by alternately dipping it in HCl and heating it in the flame. The first effect of this is merely to increase the intensity of the coloration, but after some time it becomes fainter, and by using fresh acid once or twice we can thoroughly cleanse the wire.

(3) Take some potassium nitrate and treat in the way described above. The flame is coloured a beautiful violet. Moistening with HCl makes the flame more intense. Clean the wire, wash it, and put it away in a corked test tube or bottle.

Borax bead reactions.—Borax, being an acid borate, $Na_2B_4O_7 = 2NaBO_2 . B_2O_3$, has the power of displacing other (volatile) acids from metallic salts, so forming fusible borates. These borates, in the case of many metals, possess distinctive colours, and so afford valuable evidence.

(1) Take a piece of platinum wire fitted with a glass handle, as already described, but without a loop at the end, heat in the Bunsen flame, and dip into powdered borax. Some of the salt will adhere to the wire. Again heat the wire, when the borax upon it will first "intumesce," *i.e.* swell up from the escape of its water of crystallization, and then fuse quietly to a clear, colourless bead. Now take a small quantity of an iron compound, *e.g.* $FeSO_4$ or Fe_2O_3, and bring it into contact with the bead, and again heat in the *outer* part of the Bunsen flame; a characteristic reddish-brown colour will be produced. If now the air-holes at the base of the lamp be partially closed, so as to produce a slight luminous appearance in the upper central portion of the flame, and the bead be heated in this flame ("reducing flame"), since there is an excess of coal-gas there, the oxide of iron, existing as borate in the bead, will be reduced to a lower state of oxidation, and the bead will, when cooled (best in the

coal-gas at the centre of the base of the Bunsen flame), be found to be pale green. Treated in the same way, copper compounds give a blue bead in the oxidizing flame, and a red in the reducing. In all cases only a very small quantity of the substance to be tested must be used, otherwise the colour produced is too dark to be recognisable.

The blowpipe.—To acquire the proper use of the blowpipe will necessitate a good deal of practice. A *continuous* blast of air must be kept up; for this purpose it is necessary to blow without interfering with the breathing. The secret of doing so is to use the cheeks to blow with, and to breathe through the nose. If the cheeks be blown out, it will be found that air can be forced out of the mouth by contraction of the cheeks at the same time that inspiration is effected through the nose. With practice this operation may be prolonged indefinitely. When blowing continuously, however, since the air is forced out of the mouth by the pressure of the cheeks—not by their contraction—they must be kept distended. It is not necessary to blow hard: a very gentle blast suffices.

To obtain the blowpipe flame, the air-holes at the bottom of a Bunsen burner are closed so as to give a smoky luminous flame, and the blast of air from the nozzle of the blowpipe is then directed across this flame so as to produce a long straight pointed flame. It is used for observing the effects of a very high temperature on different substances, and in some cases to effect their oxidation or reduction.

To oxidize a substance it is heated in the outer flame—that is, at the tip of the blowpipe flame. To effect reduction, the inner flame must be used—that is, the substance is heated in the centre of the blowpipe flame, which for this purpose must show a small luminous streak in the centre.

Experiments with the blowpipe.—(1) Take a piece of hard charcoal and scrape a small groove near one end. In this groove place a small quantity of litharge (lead oxide) and direct on it a reducing flame. In a short time the oxide will melt, and if the heating be continued long enough a globule of lead will remain on the charcoal. This is an example of reduction effected by the reducing-flame and the charcoal.

(2) Take some potassium nitrate, KNO_3, and, having placed it on charcoal as described in (1), bring the blowpipe flame to bear on it. It suddenly flares up, and causes ignition of the charcoal. This is *deflagration*.

(3) Take weights of powdered sodium carbonate, Na_2CO_3, and potassium carbonate, K_2CO_3, proportionate to their molecular weights, and mix them thoroughly. This mixture is known as *fusion mixture*, and should be kept ready for use. Take a small quantity of litharge (lead oxide), mix it with three or four times its weight of *fusion mixture*, and heat the mixture on charcoal before the blowpipe. The mass fuses, and in a short time small globules of lead appear and are readily fused into one large globule. The use of the fusion mixture is twofold—it prevents the fused litharge from being absorbed by the charcoal, and the fusion and reduction are more easily effected than in Experiment 1. The fusion mixture here acts as a *flux*.

Note.—When a substance is to be ***reduced***, it should be heated ***on charcoal***, with some suitable flux, in the ***reducing flame***. If ***oxidation*** is to be effected, it should be heated on the platinum wire, or on a piece of platinum foil in the oxidizing flame.

(4) Take some zinc oxide, ZnO, mix it carefully with fusion mixture, and heat on charcoal before the blowpipe. The oxide is reduced to the metallic state, and zinc,

volatile, burns on passing through the outer flame with a bluish green flame, forming the oxide which is deposited on the charcoal in the form of an *incrustation*, which is yellow when hot and white when cold. Now moisten the mass with a few drops of cobaltous nitrate, and again heat in the outer flame. The mass now assumes a fine green colour.

(5) Take a small quantity of tin oxide, and mix it with powdered potassium cyanide, KCy; place the mixture on charcoal, and cover it over with a little KCy, then heat in the reducing blowpipe flame. The tin oxide is quickly reduced, and small globules of metallic tin are obtained. Substances that are not easily reduced by charcoal alone are readily reduced in the presence of KCy, owing to the tendency of that compound to take up oxygen or sulphur to form KCNO or KCNS.

(6) Powder some copper sulphate, and mix it with five or six times its weight of fusion mixture. Heat the mixture on charcoal before the inner blowpipe flame. The mass fuses, and the $CuSO_4$ is reduced to metallic copper, which is too infusible to be obtained as a globule, and therefore remains on the charcoal as small reddish particles or scales which are not easily detected. Allow the mass to cool, remove it from the charcoal together with the crust of underlying charcoal, and place the whole in a small mortar. Powder it very finely, and transfer the powder to a small beaker. Pour on some water, and stir the whole for some time; on standing, the metallic particles sink quickly, so that, if the water be poured off after a short time, the copper will be found as a powder at the bottom of the beaker, and may be recognised by the effect of adding a few drops of nitric acid. Iron compounds may be similarly dealt with, and the iron particles are readily detected by means of a magnet.

Sublimation.—Take some ammonium carbonate, and place a small quantity in the bulb of a bulb tube. Holding the stem of the tube with a pair of crucible tongs, heat the substance in the Bunsen flame. It gradually disappears from the bulb and condenses as a *white sublimate* in the upper part of the tube.

To test the solubility of substances.—(1) Take some Epsom salts, $MgSO_4$, and with a spatula place a very small quantity of it in a dry test tube. Half fill the test tube with water from the wash-bottle, using the jet of water to wash all traces of the salt down the sides of the tube. Close the tube with the thumb, and shake up the solution. It will then be found that all the substance has dissolved.

(2) Take a small quantity of lead chloride, $PbCl_2$, and attempt to make a solution of it in the way described above. It will be found that this salt does not dissolve readily in cold water. Now hold the test tube in the Bunsen flame so as to boil the water. The lead chloride soon dissolves, and a clear solution is obtained. Allow this solution to stand and cool—the lead chloride again crystallizes out.

(3) Take some calcium hydrate and attempt to make a solution as above. It will be found that this substance is apparently insoluble in both hot and cold water. Take some red litmus paper and test the solution, it will be found to give a strong alkaline reaction, thus showing that a solution has been obtained, and that calcium hydrate is slightly soluble in water. Also, place a few drops of the solution on a piece of clean platinum foil and heat over the Bunsen flame. A small white speck is left on the foil, showing that the solution contained some solid substance.

(4) Take some pure calcium carbonate and treat as above. It will be found to be insoluble in water. Take a fresh quantity of the substance in a test tube and pour in

some dilute hydrochloric acid, HCl. The carbonate rapidly dissolves with *effervescence*, carbonic acid gas, CO_2, being evolved.

Precipitation is the basis of testing in the wet way. One of Berthollet's laws of double decomposition may be enunciated thus:—

"Two substances in solution will always decompose each other, if it be possible, by double decomposition, to produce another substance less soluble than either of the original substances."

This law expresses the principle of testing by precipitation. For example, take a solution of barium chloride in a test tube and add to it a small quantity of a solution of magnesium sulphate, $MgSO_4$. The following double decomposition is possible:—

$$BaCl_2 + MgSO_4 = BaSO_4 + \underset{\text{(soluble)}}{MgCl_2}.$$

Now barium sulphate, $BaSO_4$, is quite insoluble in water and acids, and therefore the reaction takes place, and a white precipitate of $BaSO_4$ is produced.

This reaction now furnishes us with a test for the detection of barium compounds, and of sulphates; for the test is based on the insolubility of $BaSO_4$, and may therefore indicate Ba or H_2SO_4, but has nothing to do with **Mg** or HCl. A consideration of the reaction given below will further explain this:—

$$Ba(NO_3)_2 + Na_2SO_4 = BaSO_4 + \underset{\text{(soluble)}}{2\,NaNO_3}.$$

Substances employed in systematic analysis for the detection of other substances are called *reagents*.

Solutions of gases or of solids, with other liquids so employed, are called "*wet*" reagents; those used in the solid form, "*dry*" reagents. Solutions used as wet reagents should not be too concentrated.

Filtration.—*To fold a filter paper*—If, as is usually the case, the precipitate is required as well as the clear liquid, an ordinary plain filter answers best. In cases where the amount of precipitate is but small, and the filtrate is the portion desired, a ribbed or folded filter should be used, as the liquid flows through such a paper much more quickly than through a plain filter.

To fold a plain filter.—Take a circular piece of filtering paper of the requisite size, and fold it in half. Then again fold it in half, so as to form a quadrant. Now open it in such a way that a cone of paper is formed having one thickness of paper on one side and three thicknesses on the other. Take a plain funnel (*i.e.* not a fluted one) having an angle of 60° at the apex, and fit the filter paper carefully into it, and moisten the paper with water. The paper should be in contact with the glass in every part, and ought in all cases to be small enough to go entirely into the funnel, and leave a small portion of the glass uncovered all round its upper edge.

If, as sometimes happens, the angle of the funnel is not exactly 60°, it will be found that the paper will not fit accurately to the glass. In this case, fold the filter paper the second time, not exactly in half, and open the smaller or larger side according as the angle of the funnel is less or greater than 60°.

A little care in folding and fitting the paper to the funnel is well repaid by the greater rapidity with which the liquid will flow through; and, since filtration is the most tedious operation in analytical work, any means by which it can be facilitated is well worth attention.

Certain precipitates—*e.g.* silver chloride—filter easily and rapidly in the usual way, whilst others—*e.g.* ferric hydrate—filter very slowly by the ordinary method. In order to increase the rapidity of filtration in such cases, the following arrangement may be used:—

The funnel and filtering paper are arranged as already described, but, instead of allowing the filtrate to drop directly from the funnel into the test tube or other vessel, fit to the lower end of the funnel, by means of a short length of indiarubber tubing, a piece of narrow glass tube bent round upon itself, near its upper extremity.

The bend insures that the liquid shall entirely fill the tube, and there is therefore formed an unbroken column of liquid in the lower part of the tube, and this column by its weight, reduces the pressure on the lower side of the filter paper, and hence very greatly increases the rate of filtration.

In using this arrangement it is important that the paper should fit closely to the funnel, otherwise channels are formed for the admission of air, which, of course, altogether spoils the action of the long tube in reducing the pressure below the paper. The tube should be of as great a length as the filter-stand will allow, but a length of a few inches has a remarkable effect in accelerating filtration, especially of gelatinous precipitates like ferric hydrate, a precipitate which it is most tedious to filter by ordinary means.

In making the bend in the tube, it will be found best to heat the tube at the proper place in a large flat flame, and, holding each end of the tube in the fingers, when the portion in the flame becomes quite soft, to bring the hands near together, at the same time giving one hand a slight twist. A loop will thus be formed, in a similar manner to the forming of a loop in a short piece of string, if it be held by one hand at each end and the hands be then brought nearer together. A little practice will make this clearer than any amount of description.

To fold a pleated or folded filter paper.—Take a circular piece of filtering paper and fold it in half, then again, and again in half, then open so that its outline is a semicircle; the creases produced by the other foldings are merely to serve as guides. Then, beginning at the straight side (*i.e.* the diameter of the circle of which the paper now forms half), fold the paper from the centre to the circumference as far as the first crease; then turn the whole paper over, and fold the folded portion back an equal amount on the other side; and so on, until the whole paper is folded into a sector-shaped piece. Press the paper well so as to render the creases distinct, and then open it. A conical, pleated filter will thus be formed which, when supported by a funnel, will allow a liquid to flow through with great rapidity.

Filtration.—Take a solution of barium chloride $BaCl_2$, and add a solution of sodium carbonate, Na_2CO_3, slowly, and finally drop by drop, until no further precipitate is formed.

Fit a filter-paper into a funnel, and moisten it with a jet of water from the wash-bottle to make it fit closely to the glass. Then support the funnel on a suitable stand, and, having placed a test-tube or small beaker below to receive the *filtrate*, pour a portion of the contents of the test tube gently on to the side of the filter-paper, taking care not to fill it too full. When this portion has filtered through, add another portion, and so on until the whole is filtered.

To *wash* the precipitate on the filter-paper, direct a jet of water on to it so as to wash it down into the apex from the upper edge of the paper, fill up with water, and allow to stand until this water has all filtered through. Then fill up again several times until the washings show no alkaline reaction.

Testing solubility of precipitates.—Take a small quantity of a solution of silver nitrate, $AgNO_3$, dilute it with water, and add dilute hydrochloric acid, HCl; a white curdy precipitate of silver chloride, AgCl, is obtained. Add a solution of ammonia, AmHO, in excess, and shake up the mixture: the precipitate dissolves, showing that AgCl is soluble in AmHO.

This is a convenient method of procedure applicable in most ordinary cases, but it is sometimes advisable to separate the precipitate from the accompanying liquid. This can be done thoroughly by filtration and washing, but more readily thus:—Allow the precipitate to subside, pour off the liquid and fill up with water. Again allow the precipitate to settle down and pour off the water. Repeat this once or twice. This method of washing by *decantation* is applicable only to heavy precipitates.

Take, for example, a crystal or two of copper sulphate in a clean test tube, and dissolve in about 20 c.c. of distilled water. Add a few drops of hydrochloric acid and then pass a slow stream of bubbles of sulphuretted hydrogen through the liquid as long as any change seems to be produced. Fill up the tube with water, and boil; the black precipitate will separate fairly readily and we can then pour off the top liquid. Repeat the washing once or twice, finally pouring away from the precipitate as much of the liquid as possible. Now add a few drops of dilute nitric acid and warm gently; the precipitate will dissolve to a clear bluish solution with perhaps a yellow globule of sulphur floating in it. Pour off from the sulphur into a small test tube, and divide into two portions. In the ordinary course of events, the precipitate obtained with sulphuretted hydrogen might contain either **lead or copper**; we accordingly test the solution of the precipitate for both of these. To one of the portions, add a few drops of dilute sulphuric acid and a bulk of methylated spirit equal to that of the solution. (Note that only as much dilute nitric acid should be used for dissolving the black precipitate as is absolutely necessary.) Shake well—no white precipitate of lead sulphate will be obtained; hence lead is absent. To the other portion add a few drops of ammonia solution; a fine blue colour is obtained, indicating the presence of copper. By washing a heavy precipitate and decanting off the washings, much of the time which would be occupied by tedious filtrations can be saved.

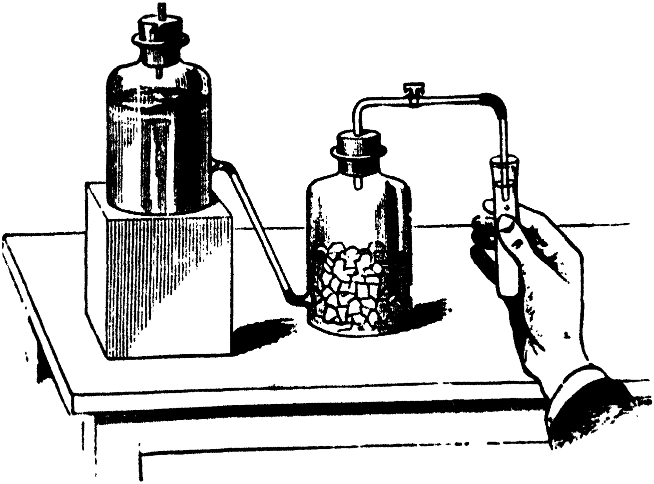


Fig. 37.

Sulphuretted hydrogen.—This is best used in the form of gas, though a solution in water will suffice if the apparatus for the manufacture of the gas be not readily available. The best form of apparatus consists of two bottles with necks in the side, connected together by a piece of indiarubber tubing. One bottle is partially (about half) filled with a mixture of fragments of iron sulphide and glass marbles, the latter being used to allow of a free passage of a liquid in the interstices between the pieces of iron sulphide; the other bottle contains dilute hydrochloric acid (one of strong acid to about two or three of water). The upper neck of the bottle containing the iron sulphide is fitted with a cork carrying a tube which is bent at right angles, and fitted either with a piece of indiarubber tubing and a pinch-cock, or, better, with a glass stop-cock. When in use, the acid bottle is raised on a block and a piece of straight glass tube is fitted, by means of a short length of indiarubber tubing, to the delivery tube of the other bottle, and the flow of the gas governed by the stop-cock or pinch-cock. When not in use, the position of the bottles is reversed, when the acid runs away from the iron sulphide, and all action ceases. This arrangement is shown in figure 37.

The following should be taken as the golden rules of analysis :—

Use small quantities of the substance to be tested, even if you have a large stock of it at your disposal; otherwise reactions will often be masked, especially those performed in the dry way. If you have only a small allowance, treasure it, and always keep some in reserve.

Work systematically, avoid making random shots, and **record your processes and results** in an orderly way.

Do not assume what you think **ought** to have happened if it does not; the basis of your expectation may be erroneous, and in laboratory practice should be confirmed, though it is foolish to follow up an unsatisfactory result in an examination.

Make a note of seeming discrepancies of results: you will probably find the explanation later.

Get some one to test you frequently: any one may do this roughly by furnishing you with a numbered test tube or packet, of which the contents are known to him. Remember that it is permissible for an examiner to try your skill on a base *or* an acid alone, or even on *aqua purissima*.

Keep your test tubes and vessels scrupulously clean. Wash them and your platinum wire at the end of your work each day; try the wire alone in the flame before using it.

Be exact in carrying out the directions given.

CHAPTER VII.

SYSTEMATIC ANALYSIS.

WHEN a substance is given for analysis it should first be examined in the dry way by the tests described on pp. 149-152. If the substance is a solid a solution of it should next be made as described, pp. 153-154, and the various reagents applied in the order given in the table, p. 155, and afterwards the metals confirmed by the tests given on pp. 157-161. Another portion of the substance should be examined for the acid by the methods given on pp. 156, 157, 161-163.

Preliminary Examination in the Dry Way.

If the substance is in solution, a portion should be evaporated to dryness on a water-bath while the examination in the wet way is being proceeded with. The residue after evaporation should then be examined as below, I.—VII.

I. Note the general physical characteristics of the substance.

On this point the previous experience of the student must be his guide, and for this purpose he should note the appearance, colour, lustre, crystalline form, etc., of all substances with clearly marked characteristics which come under his notice. It is useless to give any list of such substances, as they must be seen to be recognised.

II. Heat a small portion of the powdered substance in a bulb-tube in the Bunsen flame.

OBSERVATION.	INFERENCE.
1. *The substance fuses and solidifies on cooling.*	Salts of the alkalies or alkaline earths.
2. *The substance changes colour—*	
(*a*) *Yellow* while *hot*, *white* on *cooling*.	ZnO.
(*b*) *Dark red* while *hot*, *yellowish brown* on *cooling*.	Fe_2O_3
(*c*) From *yellow* to *reddish brown* while *hot*, *yellow* when *cold*, fusible at a red heat.	PbO.
3. *The substance gives off water with or without fusion.*	Salts containing water of crystallization.
4. *The substance volatilizes and condenses as a sublimate in the upper part of the tube—*	NH_4.
5. *A gas is evolved—*	
(*a*) O (test with glowing splinter).	Nitrates.
(*b*) Brown fumes of N_2O_4.	Nitrates of heavy metals, *e.g.* Pb, Cu, etc.

VI. Heat a small quantity of the substance in a test tube with a little dilute HCl.

OBSERVATION.	INFERENCE.
A gas is evolved— CO_2 (lime-water test).	Carbonates.

IV. Heat a portion of the substance on charcoal in the inner blowpipe flame.

OBSERVATION.	INFERENCE.
1. *It decrepitates.*	KCl, KNO_3, etc.
2. *It fuses and sinks into the charcoal.*	KCl, KNO_3, etc.
3. *It deflagrates.*	Nitrates.
4. *A white mass remains, highly luminous while heated.*	ZnO, CaO.
5. *The cold residue of 4 when moistened with cobaltous nitrate,* $Co(NO_2)_2$ *and strongly heated, is coloured green.*	ZnO.
6. *A coloured residue is left on the charcoal.*	Cu, Fe.
7. *An incrustation is formed on the charcoal—*	
(*a*) *White* incrustation.	Am.
(*b*) *Yellow* when *hot, white* when *cold.*	Zn.

V. Mix a portion of the substance carefully with fusion mixture and a trace of KCy, and heat on charcoal under the inner blowpipe flame.

OBSERVATION.	INFERENCE.
1. *A metallic bead is obtained.*	
A white malleable bead which marks paper (*yellow incrustation* on charcoal).	Pb.
2. *Red infusible scales.*	Cu.
3. *Magnetic metallic particles.*	Fe.
4. *When the fused mass is placed on a bright silver coin and moistened with a drop of water, a black stain of* Ag_2S *is produced.*	Sulphates.

VI. If the substance is coloured, heat on borax bead.

OBSERVATION.		INFERENCE.
Oxidizing Flame.	*Reducing Flame.*	
Yellow (cold), brown (hot).	Dirty green, olive.	Fe.
Green (hot), blue (cold).	Colourless or red.	Cu.
Black (too much of the substance has been used).		

VII. Hold a piece of the substance moistened with HCl on the loop of a CLEAN platinum wire in the Bunsen flame.

1. The flame is *violet*,	K.
2. ,, ,, *orange-red*,	Ca.
3. ,, ,, *green*,	Cu.
4. ,, ,, *blue*,	Pb, $CuCl_2$.

EXAMINATION FOR BASE.

Preparation of Solution.

1. If the substance is solid, it should be finely powdered, then try its solubility in water, (i) cold, (ii) boiling. If insoluble in water, try first dilute HCl, then concentrated HCl. If insoluble in HCl, try *aqua regia*.

Note.—It will generally be found unnecessary to employ *aqua regia*, but, if used, the solution should be evaporated to dryness with excess of HCl, and the residue dissolved in warm water before proceeding with the examination.

2. If the substance is insoluble in the above solvents, powder it very finely and mix thoroughly with five or six times its weight of pure fusion mixture. Heat the mixture on a boat made of a large piece of platinum-foil, first over the Bunsen flame, and then over the blowpipe. When cold, treat the foil and the fused mass with boiling water until the whole mass is completely broken up and separated from the foil. The *solution* now contains the *acid* of the substance combined with K and Na, while the *residue* contains the *base* as an oxide or carbonate. Filter off and wash the residue with hot water until the washings give no alkaline reaction. Then—

(*a*) Test *filtrate* for *acid*, as directed in examination for acid.

(*b*) Test residue for base by dissolving in dilute HCl and proceeding as detailed below.

3. If the substance is provided in solution, or is soluble in water, test with litmus paper. If the reaction is—

(*a*) *Acid.* Free acid, acid salt, or a normal salt having an acid reaction, may be present.

(*b*) *Alkaline.* Free alkali, or alkaline earth, or a strong alkali (*e.g.* KOH united with a weak acid—*e.g.* H_2CO_3 may be present.

(*c*) *Neutral.* Water, or one of a large number of salts, may be present.

When the solution is prepared, the examination must be carried on in accordance with the following table. When the base is found by this method, its presence should be further confirmed by the special tests given in pp. 157-161.

ERAL TABLE for the ANALYSIS of a SUBSTANCE CONTAINING ONLY ONE BASE.

<table>
<tr><td colspan="6">To a portion of the solution add a few drops of dil. HCl.</td></tr>
<tr><td rowspan="5">Precipitate, if produced, is probably — $PbCl_2$ (white). Filter, wash precipitate with a little cold water, scrape a little out and dissolve in boiling water. Test the solution in the manner indicated under Lead, p. 157.
the
ilute,
t be
this</td><td colspan="5">If no precipitate is produced, to the same portion of the solution add H_2S water.
If a precipitate is indicated, pass a current of H_2S gas through the solution.</td></tr>
<tr><td rowspan="4">Precipitate, if produced, may be—CuS (black), or PbS (black).
Wash the precipitate, dissolve in dil. HNO_3 to the solution add a few c.c. o dil. H_2SO_4 and an equal bulk of methylated spirits, shake, and allow to stand half an hour. White precipitate indicates Pb.
Confirm in the original solution by tests given under Lead, p. 157.
If no precipitate, boil off the alcohol and add excess of ammonia. Deep blue colouration indicates Copper. Confirm in original solution by tests given under Copper, p. 158.</td><td colspan="4">If no precipitate is produced, to a fresh portion of the solution add AmCl and AmHO in excess, after boiling with a few drops of HNO_3 to oxidize Ferrous Salts, if present, to Ferric Salts.</td></tr>
<tr><td rowspan="3">Precipitate, if produced, is probably—
$Fe_2(OH)_6$ (reddish-brown).
Test the original solution in the manner indicated under Iron (ferrous and ferric), pp. 158, 159.</td><td colspan="3">If no precipitate is produced, to the same portion of the solution add Am_2S.</td></tr>
<tr><td rowspan="2">Precipitate, if produced, is probably—ZnS (white).
Test the original solution in the manner indicated under Zinc, p. 159.</td><td colspan="2">If no precipitate is produced, to a fresh portion of the solution add AmCl, AmHO in excess, and Am_2CO_3.</td></tr>
<tr><td>Precipitate, if produced, is probably—$CaCO_3$ (white).
Test the original solution in the manner indicated under Calcium, p. 160.</td><td>If no precipitate is produced by above reagents, base must be either NH_4 or K. Boil original with NaOH.
Strong pungent smell, and gas which turns red litmus paper blue, indicates NH_4. Confirm by ammonium tests, p. 160. If no ammonia is given off, base is probably potassium.
Test the original solution in the manner indicated under Potassium, p. 161.</td></tr>
</table>

EXAMINATION FOR ACID.

In testing for acids, the student should be guided by—

(*a*) *The results of the preliminary examination.* In many cases the acid will have been detected in this examination, and it is only necessary to confirm the result in the wet way.

(*b*) *The result of the examination for the base.* The base being now known, and the properties and solubility of the substance being determined, the field of search is often considerably narrowed. If no acid is found, the substance may be an oxide or hydrate, and here, again, a knowledge of the nature of the base helps to decide whether such a compound is likely to occur.

A knowledge of the commoner compounds of each metal is often suggestive, but it should not encourage random shots.

Preparation of Solution.

1. *The substance is in solution or soluble in water.*

If metals of Groups I. or II. be present, remove them by boiling with a slight excess of pure sodium carbonate, filter, and use the filtrate for the examination for acids. If Groups I. and II. be absent, use the original solution.

2. *The substance is insoluble in water, but soluble in acids.*

Boil a portion of the solid with a strong solution of sodium carbonate, filter, and use the filtrate.

3. *The substance is insoluble.*

Treat as described for the preparation of the solution for the examination for bases, using the aqueous filtrate.

Before adding the reagents, exactly neutralize a portion of the solution with HNO_3, or, if acid, with ammonia. If the solution has been boiled with sodium carbonate, the liquid will be strongly alkaline. In this case, make it distinctly acid with HNO_3, boil to expel CO_2, and then neutralize again with ammonia.

Test and Observation.	Inference.
1. *To neutral solution add* $BaCl_2$.	
White precipitate, *insoluble* in HCl.	Presence of H_2SO_4.
White precipitate, *soluble* in HCl, dissolves with effervescence.	Presence of H_2CO_3.
2. *To another portion add* $AgNO_3$. *White* precipitate, *insoluble* in HNO_3, *soluble* in NH_3.	Presence of HCl.
If no precipitate is produced by $BaCl_2$ or $AgNO_3$—	
3. *To a portion of solution add a solution of* $FeSO_4$, *and then pour down the side of the test-tube strong* H_2SO_4. See Note, p. 163.	
A *brown ring* formed at junction of fluids indicates	HNO_3.

LEAD, Pb.

Solution—Lead Nitrate, $Pb(NO_3)_2$.

1. Group-reagent, HCl, gives a *heavy white crystalline* precipitate of lead chloride, $PbCl_2$: *soluble in hot water*, but crystallizing out on cooling; *insoluble in ammonia.* No precipitate is formed in dilute solutions.

2. Potassium chromate, K_2CrO_4, gives a *bright yellow* precipitate of lead chromate, $PbCrO_4$: insoluble in dilute nitric acid and acetic acid; soluble in sodium hydrate, NaHO.

3. Potassium iodide, KI, gives a *golden yellow* precipitate of lead iodide, PbI_2: soluble in boiling water, but crystallizing out in brilliant irridescent scales on cooling.

COPPER, Cu.

Solution—Cupric Sulphate, $CuSO_4$.

1. H_2S gives a *brownish-black* precipitate of cupric sulphide, CuS: insoluble in hot dilute sulphuric acid.

2. Ammonia, $AmHO$, or ammonium carbonate, Am_2CO_3, when added in small quantities, produces a *greenish-blue* precipitate of a basic salt, which is readily dissolved in excess of the reagent, forming a splendid *azure-blue liquid*; this coloration is easily perceptible, even if the solution contains only slight traces of copper. The blue solution contains ammonio-cupric sulphate, $(NH_3)_2Cu''SO_4$.

3. Potassium ferrocyanide, K_4FeCy_6, gives a *reddish-brown* precipitate of cupric ferrocyanide, Cu_2FeCy_6, in neutral or slightly acid solutions, but not in alkaline solutions. A *brownish colour* is produced in very dilute solutions of copper salts; it is best seen when the reaction takes place in a watch-glass placed on a sheet of white paper. K_4FeCy_6 supplies one of the most delicate reactions for copper salts.

IRON (*Ferric*), Fe'''.

Solution—Ferric Chloride, Fe_2Cl_6.

1. Ammonium hydrate, $AmHO$ gives a *reddish-brown* precipitate of ferric hydrate, $Fe_2(HO)_6$.

2. Potassium ferrocyanide, K_4FeCy_6, gives a *dark blue* precipitate of *Prussian blue*, $Fe_4(FeCy_6)_3$.

3. Potassium ferricyanide, $K_6Fe_2Cy_{12}$, gives *no* precipitate, but a *deep green* (*very dilute solutions*) or *brown coloration*.

IRON (*Ferrous*) Fe''.

Solution—Ferrous Sulphate, $FeSO_4$.

1. AmHO gives a *light coloured* precipitate of ferrous hydrate, $Fe(HO)_2$, which rapidly turns *dirty green*, and more slowly *reddish-brown*, by reason of the absorption of oxygen: complete precipitation of ferrous hydrate by AmHO is prevented by the presence of ammonium salts.

2. Potassium ferrocyanide, K_4FeCy_6, gives a *bluish-white* precipitate, which speedily turns blue by absorption of oxygen.

3. Potassium ferricyanide, $K_6Fe_2Cy_{12}$, gives a *dark blue* precipitate of $Fe_2Cy_6\,3\,FeCy_2$ (*Turnbull's blue*). [*Distinction from ferric salts.*]

ZINC, Zn.

Solution—Zinc Sulphate, $ZnSO_4$.

1. Am_2S gives a *white* precipitate of zinc sulphide, ZnS

2. Sodium hydrate, NaHO, precipitates zinc hydrate, $Zn(HO)_2$, as a *white gelatinous* precipitate, soluble in excess. Addition of AmCl does not reprecipitate the zinc hydrate.

3. Potassium ferrocyanide, K_4FeCy_6, gives a *white* precipitate of Zn_2FeCy_6: insoluble in dilute acids.

CALCIUM, Ca.

Solution—Calcium Chloride, $CaCl_2$.

1. Ammonium Carbonate, Am_2CO_3 gives a *white* precipitate of calcium carbonate, $CaCO_3$.

2. Ammonium oxalate, $Am_2C_2O_4$, give a *white powdery* precipitate of calcium oxalate even in very dilute solutions. The precipitate is insoluble in acetic and oxalic acids, but is readily soluble in nitric and hydrochloric acids.

3. Dilute sulphuric acid or soluble sulphates give, *from concentrated solutions*, a white precipitate of calcium sulphate, $CaSO_4$, which is slightly soluble in water.

AMMONIUM, NH_4(Am).

Solution—Ammonium Chloride, AmCl.

1. Platinic chloride, $PtCl_4$, produces in neutral and concentrated acid solutions a *dense yellow* precipitate of the double chloride, $PtCl_4 . 2\,AmCl$.

2. Add NaHO or KHO, and heat gently. Ammonia, NH_3, is given off, and is readily recognised by its smell, action on litmus paper, and by fuming when a glass rod dipped in HCl is brought near it.

3. Heat some dry ammonium chloride in a test tube: a white sublimate is formed in the upper part of the tube. All ammonium salts are volatilized or decomposed by heat.

POTASSIUM, K.

Solution—Potassium Chloride, KCl.

1. Place a few drops of the solution on a watch-glass, and add to it two or three drops of platinic chloride, $PtCl_4$, a little HCl, and then a few drops of alcohol. If the solution is concentrated, a *dense yellow crystalline* precipitate of the double chloride, $PtCl_4 . 2 KCl$, is at once formed; if the solution be dilute, the precipitate is obtained only after some time. In this case, if allowed to stand quietly for half-an-hour or so, a yellow crystalline precipitate forms on the bottom of the glass, where it is readily detected by stirring it with the point of a thin glass rod. In very dilute solutions no precipitate is obtained.

2. Tartaric acid gives in concentrated neutral or slightly acid solutions, a *white crystalline* precipitate of cream of tartar. Shaking or stirring favours the precipitation.

3. Potassium compounds colour the flame *violet*. Owing to the presence of traces of sodium in all potassium salts, this flame test is not always distinct. By looking at the flame through a piece of blue cobalt glass the potassium flame can be distinguished in the presence of the sodium, but this method is not very reliable, and, unless the potassium flame is very distinct, the presence of this element will need careful confirmation in the wet way.

SULPHURIC ACID, H_2SO_4.

Solution—Potassium Sulphate, K_2SO_4.

1. *Barium chloride, $BaCl_2$, gives a *heavy white powdery* precipitate of barium sulphate, $BaSO_4$, insoluble in HCl.

2. Lead acetate, $Pb(C_2H_3O_2)_2$, gives a *heavy white* precipitate of lead sulphate, $PbSO_4$.

---

* Use Barium Nitrate $Ba(NO_3)_2$ if

F. St. Prec.

HYDROCHLORIC ACID, HCl.

Solution—Sodium Chloride, $NaCl$.

1. Silver nitrate, $AgNO_3$, gives a *white curdy* precipitate of silver chloride, $AgCl$: insoluble in strong boiling nitric acid: soluble in solution of ammonia.

2. Mix with MnO_2* and H_2SO_4, and heat: chlorine gas is given off, and may be recognised by its colour, odour, and bleaching action.

3. Strong sulphuric acid, when added to a strong solution of a chloride or to the dry salt, produces acid fumes of HCl.

CARBONIC ACID, H_2CO_3.

1. All carbonates are decomposed by dilute acids with the evolution of CO_2. This may be detected by suspending a drop of lime water on a glass tube over the gas, when the drop soon turns milky.

Note.—Acid carbonates, or bi-carbonates, evolve CO_2 on boiling, and are converted into carbonates; *e.g.*

$$2KHCO_3 = K_2CO_3 + CO_2 + H_2O.$$

2. Add $NaHCO_3$ to a solution of $MgSO_4$: no precipitate is formed, but on boiling the solution a white precipitate of $MgCO_3$ is quickly formed. The normal carbonate, gives a precipitate of $MgCO_3$ at once.

---

* Common MnO_2 frequently contains chlorine; the best is "needle" or native "manganese."

NITRIC ACID, HNO_3.

Solution—Potassium Nitrate, KNO_3.

1. Take a weak solution of the nitrate, and dissolve in it a crystal of ferrous sulphate, $FeSO_4$. When quite cold, pour into the tube enough concentrated H_2SO_4 to form a distinct layer at the bottom. A deep purple or brown-coloured ring will appear where the liquids meet.

Note.—When the addition of $FeSO_4$ produces a precipitate (*e.g.* with lead nitrate), a portion of the solution should be boiled with sodium carbonate, then filtered, concentrated, and the filtrate acidified with dilute H_2SO_4. The test may then be applied to this solution which contains the acid as sodium nitrate, the base having been precipitated as a carbonate.

2. Heat with concentrated sulphuric acid and copper turnings: nitric oxide, NO, is given off, which becomes nitric peroxide, N_2O_4, in the upper part of the tube from absorption of oxygen.

3. A nitrate heated on charcoal deflagrates.

Note.—All nitrates are soluble in water; hence we have no precipitation reactions.

On the following pages will be found some model analyses. These the student should study as a guide to the best method of writing out the results of his work.

WHITE CRYSTALLINE SUBSTANCE. SOLUBLE IN WATER. NEUTRAL.

Experiment.	*Observation.*	*Inference.*
	PRELIMINARY EXAMINATION.	
Heated in a dry bulb tube.	Brown oxides of N evolved.	Nitrate of heavy metals.
„ with HCl.	White insoluble substance formed on addition of acid; dissolves on heating.	[Grp. I. (Pb)]
„ „ H_2SO_4.	Oxides of N.	HNO_3.
„ „ Na_2CO_3.	Deflagrates: metallic malleable bead which marks paper, blue flame.	Pb.
„ on platinum wire.	Blue flame.	Pb, As, Sb.
	WET EXAMINATION FOR BASE.	
Added HCl.	White pp.: dissolves on boiling: SH_2 added to solution thus obtained: *reddish-brown* (colour due to excess of HCl), finally black pp. is obtained.	**Pb.**
„ KI.	Yellow pp.	„
„ K_2CrO_4.	„	„
	EXAMINATION FOR ACID.	
Boiled with Na_2CO_3 to remove Pb, neutralized with HNO_3, and added $BaCl_2$.	No pp.	—
Added $AgNO_3$.	„	—
Pptd Pb from sol. with Na_2CO_3, filtered, acidified with H_2SO_4, and added $FeSO_4$, and H_2SO_4.	Brown ring formed.	HNO_3.

$FeSO_4$ pptates Pb as $PbSO_4$. See p. 64, Note.

Base, PbO. **[Lead Nitrate, $Pb(NO_3)_2$].** **Acid, HNO_3.**

WHITE AMORPHOUS POWDER. SLIGHTLY SOLUBLE IN WATER. NEUTRAL.

Experiment.	Observation.	Inference.
PRELIMINARY EXAMINATION.		
Heated in dry bulb tube.	—	—
" with HCl.	—	—
" " H_2SO_4.	—	—
" on charcoal with Na_2CO_3.	—	—
Placed residue on silver coin.	Dark stain on coin.	Sulphur compounds.
Heated on charcoal.	—	—
" " " with $Co(NO_3)_2$.	—	—
" " platinum wire.	Red flame (traces of Sr flame).	Ca, Sr.
WET EXAMINATION FOR BASE.		
Added HCl.	No pp.	Absence of Grp. I.
" SH_2.	" "	" " II.
" AmCl, AmHO.	" "	" " III.
" " " Am_2S.	" "	" " IV.
" " " Am_2CO_3.	White pp.	Ba, Sr, Ca.
" $CaSO_4$.	No pp.	Ca.
" $Am_2C_2O_4$.	White pp.	"
EXAMINATION FOR ACID.		
Added $BaCl_2$.	White pp., insoluble in HCl.	H_2SO_4.
	Confirmed by Preliminary Examination.	
Base, CaO.	**[Calcium Sulphate, $CaSO_4$.]**	**Acid, H_2SO_4.**

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