PRACTICAL ORGANIC CHEMISTRY

TURPIN

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BY

G. S. TURPIN, M.A., D.Sc.

HEAD-MASTER OF THE INTERMEDIATE AND TECHNICAL SCHOOL,
SWANSEA

London
MACMILLAN AND CO.
AND NEW YORK
1895

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APPARATUS REQUIRED

A. By each Student.

20 oz. Flask, fitted with Rubber Cork with two holes.
2 oz. Flask, fitted with Rubber Cork with one hole.
One medium-sized Flask.
Retort Stand with Rings.
Bunsen Burner.
2 feet Gas Tubing.
1 foot small Rubber Tubing.
Porcelain Crucible and Lid.
Pipe-clay Triangle.
2 Evaporating Basins.
Pestle and Mortar.
4 oz. Retort.
20 Test-tubes and Test-tube Stand.
Test-tube Brush.
Two Funnels.
One Thistle-headed Funnel.
Three Beakers.
Wire-Gauze.
Sand-Bath, or piece of Asbestos Paper.
Blowpipe.
Spring Clip (Mohr's).
Crucible Tongs.

B. For General Use.

(*The largest class is supposed to consist of* 24 *students.*)

24 Gas Cylinders, 8 in. by 2 in., or wide-mouthed Bottles.
24 Glass Plates to cover the mouths of the Cylinders.
6 Deflagrating Spoons.
4 Balances (weighing 100 grams accurately to 5 milligrams) and Sets of Weights.
24 Glass Tubes, closed at one end, 50 cm. by 2 cm.
24 Glass Tubes, closed at one end, 75 cm. by 1·2 cm., fitted with Indiarubber Stoppers, each with one hole.
24 Glass Tubes, closed at one end, of Hard Glass, 13 cm. by 1·2 cm., fitted with Rubber Corks, each with one hole.
12 Large Basins.
12 Beehive Shelves.

12 Half-metre Rules.
12 Burettes.
12 500 cc. Measuring Flasks.
12 100 cc. Measuring Cylinders.
12 Sets of Pipettes, 50 cc., 25 cc., and 10 cc.
12 Round-bottomed Flasks—400 cc.—fitted with Rubber Corks, each with one hole.
12 Wurtz's Distillation Flasks, 200 cc.
12 Liebig's Condensers.
12 Centigrade Thermometers, reading up to 200° C.
2 Sets good Cork-Borers.
12 Bell-jars, small size, with open neck fitted with Rubber Stoppers, each with one hole.
12 Metres thick-walled Rubber Tubing.
24 Small Rubber Bands.
12 Clamps for use with the Retort Stands.
12 Gas-washing Bottles.
12 Bulb-tubes of Hard Glass.
A supply of Glass Tubing, mostly of 4-5 mm. internal diameter.

N.B.—Somewhat larger quantities than the above should be ordered to provide against breakages.

CHAPTER I

WEIGHING AND MEASURING SOLIDS AND LIQUIDS

Weighing.—The operation of weighing consists in a comparison of the quantity of matter in the substance to be examined, or in other words its *mass*, with that of a certain standard. The standards both of weights and of measures which are in general use in this country are excessively arbitrary and complicated; in this book we shall always employ the *metric system*, which has many advantages over the British system, and is universally employed for scientific purposes.

In the metric system the unit of mass is the *gram* or *gramme* which is defined as *the mass of one cubic centimetre of water* at the temperature of 4° C., and in thus basing the unit of mass upon those of volume and length the metric system is following as simple a plan as could be invented; chemistry, however, is much more concerned with weights than with measurements of length, area, or volume, and we commence accordingly with mass and its measurement.

Units of Mass or Weight.—The *gram* is then the primary unit of mass or weight, but it is frequently inconvenient to be compelled to express a given weight always in grams, as this may lead to the use of very large numbers for a large weight, and of many decimals when a very small weight has to be expressed; to meet this difficulty the metric system employs other units which, however, are always connected with the gram by a ratio which is a multiple of ten, and it is on this account that the system is also known as the decimal system. Thus for larger weights we may employ the units

Decagram = 10 grams
Hectagram = 100 grams
Kilogram = 1000 grams

but of these only the kilogram is in frequent use.

For smaller weights the units carry Latin prefixes, and are as follows :—

Decigram = $\frac{1}{10}$ gram
Centigram = $\frac{1}{100}$ gram
Milligram = $\frac{1}{1000}$ gram

The Balance.—The balance is the instrument generally employed for comparing the masses of two bodies, and though its fuller description is left to the science of Physics, it is necessary to describe it here very briefly. The ordinary balance is essentially a lever with two arms of equal length, which swings about a central fulcrum at O; the two masses to be compared are attached to the two ends of the beam A, B, at equal distances from O, generally by the help of pans such as are shown in the figure at C, D; if the two masses are equal the attraction of the earth upon them, which we call their weight, and is due to gravity, will be the same for each, and the balance will be in equilibrium; but if the mass placed in the pan C be

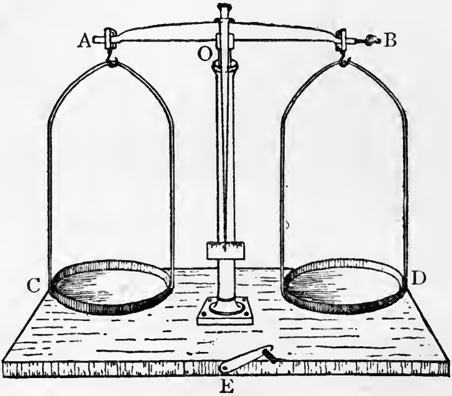

FIG. 1.—The Balance.

greater than that in D, then its weight will also be greater, and the action of gravity will cause that pan to descend, thereby pulling upwards the other pan D.

In order to measure the mass (or weight) of a given substance it is placed in one of the pans C or D, and into the other we put masses of known amount until they are found to balance exactly that of the body to be weighed; a set of masses, or *weights* as they are always called, for this purpose is necessary for use with any balance, and on their correctness depends ultimately the exactitude of any measurement of weight which can be made. This exactitude is also affected by the sensitiveness of the balance, that is, by the amount necessary to make the beam turn.

EXERCISE I.—Apparatus needed: a balance and set of weights, a number of copper coins, or discs of metal of equal weight.

A. Place a coin or one of the metal discs in the left-hand pan of the balance; raise the beam by the handle E and you will at once notice that the pan in which the coin is placed over-balances the empty one; now turn back the handle E so that the beam is again supported, and selecting from the box a weight which seems to you likely to be near the mark, place it in the empty pan; let us suppose you have taken the 5 gram weight: then steadily turn the handle E again so that the beam may swing, and watch which way it turns; this will tell you whether the 5 gram weight is heavier or lighter than the coin you are weighing; if it is too light to balance the coin we will try the 10 gram weight next, and this we find to be too heavy.

Note.—Always turn the handle E so that the beam is firmly supported before removing weights from the pans or placing weights in them.

We have found the coin to weigh between 5 and 10 grams; put back the 5 on the pan in place of the 10 gram weight and add the 1 gram; if this is not enough add another 1 gram weight, making 7 altogether.

We will suppose it is finally found that the proper weight is between 8 and 9 grams; it is next necessary to bring out the smaller weights, and it is a good plan for any one beginning to work with a balance to keep ready for use a piece of cardboard or stout paper about 4 inches by 2, marked out as shown in Fig. 2 :—

.5 grams	.2	.1	.1
.05	.02	.01	.01

FIG. 2.

The several weights being placed each on its proper square, you must next add the .5 gram to the 8 gram weight, and on trying find (we suppose) that this is too small ; then add the .2 gram weight, and you find the total to be somewhat too large —so try next the .1 gram in place of the .2 gram : you find this to be almost right, but on carefully watching the swing of the needle find the coin to be slightly heavier than the weights in the other pan.

You must now proceed in similar fashion with the smaller set of weights which give the hundredths of a gram or centigrams. When the balance shows you have the right weights in the pan, you must first read off their amount by noticing the gaps in the box of weights and on the card which you use for the subdivisions of the gram ; then carefully replace the weights which are on the pan, and while putting them back add up their amounts, comparing this with the total obtained by noticing the gaps left in the box and on the cardboard : the two must agree exactly.

Thus the following weights were found to be missing from their places :—

5.0 gram
2.0 ,,
1.0 ,,
.5 ,,
.1 ,,
.02 ,,
8.62 grams

On replacing the weights the same total was obtained, and this amount is therefore correct.

B. Find the weight in the same way of 2 coins, of 5 coins, and of 10 coins.

Calculate in each case what the weight of a single coin should be, and compare with the result obtained in A.

Measuring.—The unit of volume in the metric system is based upon the *unit of length*, which is the primary unit of the system; this is *the metre*, and was originally intended to be equal to one ten-millionth part of a quadrant of the earth's surface, drawn from the North Pole to the Equator in such a manner as to pass through Paris. It is plain that this distance is not one that can be easily measured with great accuracy, and more recent investigations have shown that the original measurements on which the metre was based were not perfectly exact. Thus it follows that the metre as we use it does not conform quite strictly to its original definition, and the fundamental unit of the metric system is really an arbitrary one, being the distance between two fine lines on a bar of metal carefully preserved as a standard in the archives of the French Government.

The length of the metre is 39.37 inches in English measure, and from it the other units of length in the metric system are derived; the larger ones are obtained by multiplication, and the smaller ones by division by some multiple of ten, thus:—

Metre = 39.37 inches or 1.094 yards

for the smaller lengths—

Decimetre = $\frac{1}{10}$ metre = 3.937 inches
Centimetre = $\frac{1}{100}$,, = .3937 ,,
Millimetre = $\frac{1}{1000}$,, = .03937 ,,

and for the multiples of the metre—

Decametre = 10 metres = 10.94 yards
Hectametre = 100 ,, = 109.4 ,,
Kilometre = 1000 ,, = 1094 ,,

EXERCISE 2.—In order to familiarise yourself with the measurement of lengths in the metric system do the following:—

(1) Mark on paper two dots a foot apart, and measure with the half-metre rule what this is equal to in decimetres, in centimetres, and in millimetres.

(2) Now mark off on paper the length of your foot; try to

estimate what this will be in centimetres ; then measure it with the half-metre rule and compare with your estimate.

(3) First estimate in the same way in centimetres and then measure (*a*) the width of the bench at which you work, (*b*) its height from the floor, (*c*) the height of this page.

(4) Mark off an inch on paper ; now measure this in millimetres, and see how this agrees with your measurement of a foot in centimetres.

(5) First estimate in millimetres and then measure (*a*) the thickness of the wooden top of your working-bench, (*b*) the inside diameter of the tube of your Bunsen burner.

Measurement of Volume.—The measurement of volume in the metric system is based upon the measurement of length, the ordinary units being two in number, the *cubic centimetre* (cb.c., or cc., as it is written for shortness) and the *cubic decimetre* or *litre*.

The *cubic centimetre* is plainly the volume of a cube which measures one centimetre in the side ; now a centimetre is approximately .4 inch, but of course a cubic centimetre is not .4 cubic inch ; just as a cubic foot is not 12 cubic inches, but $12 \times 12 \times 12$ or $1728 \ (= 12^3)$ cubic inches, so a cubic centimetre is $.4 \times .4 \times .4$ (or $.4^3$) or .064 cubic inch.

More exactly 1 cb.c. = .061 cubic inch.

The *cubic decimetre*, generally called the *litre*, is the larger unit of volume which is in general use where the metric system is employed ; as 1 decimetre = 10 centimetres, it follows that 1 cubic decimetre = $10 \times 10 \times 10 = 1000$ cubic centimetres.

One *litre* then = 1000 cb.c.
= 61 cubic inches.

In Chemistry we seldom measure volumes except of liquids or gases, and then usually as a means of finding the weight by calculating it from the volume ; in this course of exercises the volume of a gas will generally be measured by that of some liquid (water) which the gas has been made to displace in such a way that the volume of water collected is equal to that of the gas whose volume it is desired to know. We are chiefly concerned, therefore, with liquids so far as measurement of volume is to be performed, and for this purpose several kinds of graduated measuring instruments are used. Measuring flasks are employed when we wish to measure an exact litre or some simple fraction of it; those most often in use are the litre, 500 cc. and 250 cc. flasks, and each bears a fine circular mark somewhere on the neck of the flask which indicates the point to which the flask must be filled in order that it may hold the volume for which it is graduated. Pipettes (Fig. 3) are employed to measure out exactly some smaller volume than the flasks are used for, and the ordinary sizes are the 100 cc., 50 cc., 25 cc., and 10 cc. pipettes; but those measuring 20 cc., 5 cc., and 2 cc., or even 1 cc., are sometimes used; in these the graduation mark is on the narrow stem above the wider bulb, and it must be remembered that pipettes are graduated *to deliver* the volume marked on them; that is to say, if you fill up a 50 cc. pipette by sucking water into it with the mouth until the water rises above the mark on the stem, then *quickly* remove the lips and close the upper end of the pipette with the forefinger (this requires a little practice, but the knack is soon learnt), and now slightly relieve the pressure of the finger so that the water flows from the pipette drop by drop until the level of the water coincides with the mark on the stem of the pipette, then the 50 cc. pipette so filled to the exact mark will hold rather more than 50 cc.; but if the water contained in it be allowed to flow out into a beaker, the volume of water collected in this will measure exactly 50 cc., while the slight excess remains adhering to the walls of the pipette.

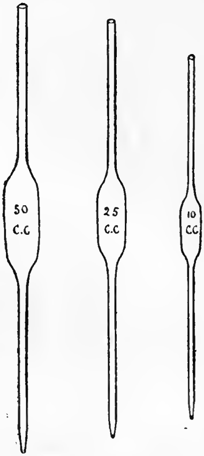

FIG. 3.—Pipettes.

EXERCISE 3.—Apparatus required: a 500 cc. or other measuring flask and some pipettes; a dish to hold water.

(1) Practise filling a pipette in the way described above; you will notice that the surface of the water in the stem of the pipette is not flat but curved, and may be puzzled which part of this curve should be brought to the level of the mark on the stem of the pipette; the right plan is to work so that the *lowest*

part of the curve just touches the level of the mark as shown in Fig. 4.

(2) Compare a 50 cc. pipette with the 500 cc. flask and find whether it requires exactly 10 times the volume of the pipette to equal that of the flask. If they do not agree, we shall see later how we can find which of them is in error.

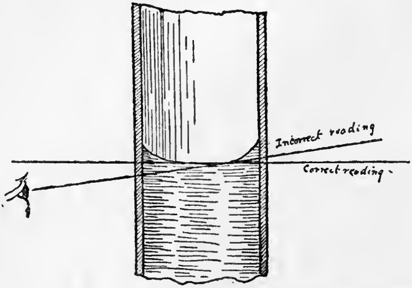

Fig. 4.—Cause of possible error in reading Burettes.

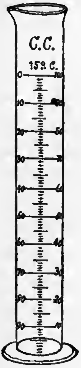

Fig. 5.—Measuring Cylinder.

Measuring Cylinders (Fig. 5) are used for measuring any required volume of water or other liquid, up to a limit fixed by the size of the cylinder selected: *burettes* (Fig. 6) are more accurate instruments, intended for a similar purpose, but dealing with smaller volumes of liquid. In reading the level of the water in a cylinder or burette it is necessary to take considerable care if large errors are to be avoided, and the two following points must be carefully observed: (1) place the eye at the same level as the surface of the liquid in the cylinder or burette; (2) always read by the same portion of the curved surface which the liquid has, preferably by the lowest portion of this curve (Fig. 4).

Exercise 4.—Apparatus required: a graduated burette and a measuring cylinder, some pipettes.

(1) Fill the burette with water above the zero mark of the scale; open the clip (Fig. 6, *a*) wide by pressing it firmly, and let the water run out into a dish or beaker, until there is no more air left in the jet of the

burette; then fill it up again, this time exactly to the zero mark, noting the precautions mentioned above; run out part of the contents of the burette into a dry measuring cylinder. Read off carefully both the burette and the cylinder; do they agree? Then run in the remainder of the contents of the burette down to the end of the graduations, and again compare the burette and cylinder.

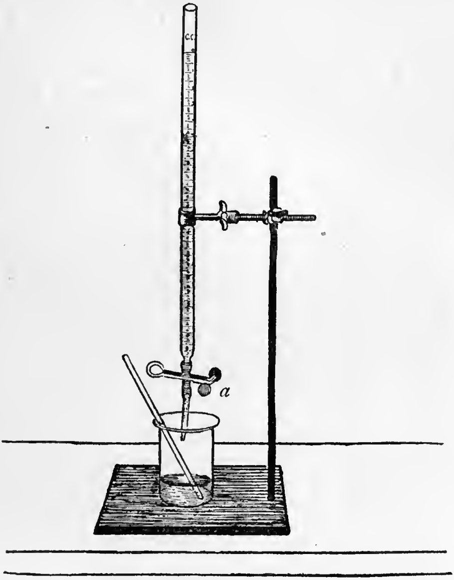

FIG. 6.—Burette arranged for use.

(2) Fill a 25 cc. pipette with water, and run this into the dry measuring cylinder; read off the volume shown by the graduations on the cylinder; fill the pipette again, run the water into the cylinder and again read the united volume. Continue until the limit of the graduations is reached.

Measuring the Volume of a Solid.—The volume of a solid can be measured directly only when it is of some simple geometrical shape such as a cube or sphere; it can be measured indirectly by noticing the volume of water or other liquid which the solid is able to displace.

EXERCISE 5.—Apparatus required: 100 cc. cylinder, 50 cc. pipette, some marbles or small glass stoppers.

(1) Put about 50 cc. of water in a 100 cc. cylinder, and read off its volume accurately; now carefully slide a marble or glass stopper into the water, taking care not to lose any water by splashing; the water rises, why? Read the level of the water accurately, and calculate the volume of the marble or glass stopper.

(2) Do the same experiment in a modified form; put the marble in a dry measuring cylinder, and run in 50 cc. of water from a pipette; read the level of the water in the cylinder and calculate the volume of the marble.

(3) Find in the same way the volume of a piece of iron or other metal which is of convenient size and shape. Another and more accurate method of measuring the volume of a solid will be described in the next chapter.

CHAPTER II

RELATIVE DENSITY OR SPECIFIC GRAVITY

IT is obvious from common experience that different substances are not alike in the volume occupied by a given weight of the substance; iron is called heavy and wood light, because a pound, say, of iron takes up very much less space than the same weight of the lighter material wood. The relation between volume and weight for various substances is of great importance, and it is essential that we should have some method of expressing this relation clearly and accurately.

Density.—The *density* of a substance is a means of expressing this relation, and is defined to be "the mass of a given quantity of the substance divided by its volume." Let us find what the density of water is.

EXERCISE 6.—Apparatus required: balance, a beaker, some pipettes, a burette, a 250 cc. measuring flask (dry).

(*a*) Put the beaker in the left-hand pan of the balance, weigh it, and make a note of the weight in your notebook; now run in 25 cc. of water from a 25 cc. pipette, and weigh again; the difference between the two weighings will give the weight of 25 cc. of water; calculate the density of water from the formula

$$\text{Density} = \frac{\text{Weight (in grams)}}{\text{Volume (in cb.c.)}}$$

(*b*) Pour out the water from the beaker, again weigh (why?) and repeat the experiment, using a 50 cc. pipette.

(*c*) Repeat with a volume of water run out at random (but carefully measured) from the burette.

(*d*) Weigh the dry 250 cc. flask; fill up with water to the mark, and weigh again.

On calculating the results of your experiments you will in each case find the density of water to be expressed by unity, and this follows directly from the original definition of the gram, referred to on p. 1, as the weight of 1 cb.c. of water. The slight divergencies from the number 1, which will be found even when your weighings are very carefully done, are due partly to the want of perfect accuracy in the weights you use, and much more to inaccuracy in the pipettes and other measuring vessels.

Relative Density.—For other solids and liquids it is more usual to speak of their *relative density* or *specific gravity* than of their density; by the relative density we mean (for solids and liquids) the density relative to water, or the number which tells us the proportion between the weight of a given volume of the substance and the same volume of water. We have just seen that in the metric system the density of water is expressed by unity, and therefore in that system the relative density or specific gravity of a substance is expressed by the same number as its density.

EXERCISE 7.—Apparatus required: pipettes, a burette, a 250 cc. measuring flask, balance, some common salt.

(*a*) Prepare a 20 per cent solution of salt in water as follows:—weigh out 100 grams of salt, put this in a fairly large beaker, add about 300 cc. of water and stir until dissolved; pour the solution into a 500 cc. measuring flask, rinse out the beaker twice with a small quantity of water, adding the rinsings to the solution in the measuring flask, and finally fill this up to the mark: then mix thoroughly by shaking well. (This solution need not be prepared by the student.)

(*b*) Determine the density of this solution as in Exercise 6, *a*, *b*, *c*, and *d*. N.B.—A pipette, or other measuring vessel, which is not thoroughly clean and *dry*, must first be washed out with some of the solution which is to be measured with it.

The number which you find for the density of this 20 per cent salt solution should be 1.15; this means that 1 cc. of the solution weighs 1.15 gram, but as 1 cc. of water weighs exactly 1 gram, it follows that the solution is 1.15 times heavier than water; in other words, its relative density or specific gravity is also 1.15.

Exercise 8.—Apparatus required: the same, with some copper sulphate in place of the common salt.

(*a*) Prepare a 10 per cent solution of copper sulphate by dissolving 50 grams of crystallised copper sulphate in 500 cc. of water in the same way as described above (Exercise 7, *a*).

(*b*) Determine the density of this solution.

More accurate methods for determining the specific gravity of liquids are described in books on Physics; in Chemistry, and more especially in its technical applications, a method is often required by which the density can be quickly determined with the least possible trouble and with fair accuracy; such a method is furnished by the use of instruments called Hydrometers.

The Hydrometer.—The use of the hydrometer is based upon a principle of which the explanation must be left to books on Physics; we will, however, construct a simple form of hydrometer in order to see how the instrument is used.

Exercise 9.—Take a piece of glass tubing about 30 cm. long and 7-10 mm. external diameter: close up one end (for the method of doing this, see Chapter VI.) and load the tube with leaden shot so that it will float in water with about $\frac{2}{3}$rds of its length immersed; then cut a narrow strip of cardboard about 20 cm. long and of such width as will just allow it to be pushed inside the tube, mark on this a scale in millimetres numbered from 50 at the bottom to 250 at the top; push the scale into the glass tube until the lowest graduation of the scale—the 50 mm. mark—is just 50 mm. from the bottom of the tube. Now close up the top of the tube by means of the blowpipe, or with a sound cork cut off flush with the edge of the glass and then covered with sealing-wax.

(*a*) Weigh the hydrometer you have made: measure its length; then measure its diameter by placing the tube between two blocks of wood (Fig. 7), and with your millimetre scale reading off carefully the distance between the two blocks. Calculate the cross-section of the tube from the formula

$$s = \pi r^2,$$

where π denotes the ratio of the circumference of a circle to its diameter $(= \frac{22}{7})$, and r denotes the radius or half the diameter. Next calculate the volume of the hydrometer by multiplying its length in centimetres by its cross-section in square centimetres, so that the volume may be expressed in cubic centimetres.

(*b*) Float the hydrometer in water contained in a tall jar, and notice on the scale the level at which it floats. Calculate what portion of the length is immersed in the water, and then find the volume of this portion; you will then know the volume of water displaced by the instrument. What is the weight of this amount of water? Compare it with the weight of the hydrometer itself.

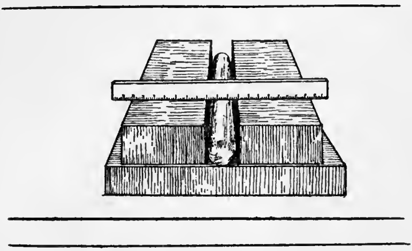

FIG. 7.—Measuring Diameter of Tube.

You will find, if your measurements are all carefully made, that the hydrometer sinks in the water to such a depth as to displace a quantity of water equal in weight to the hydrometer itself; let us check this result by another experiment.

EXERCISE 10.—Take a round glass vessel with a narrower neck at one end; the vessel should be about 20 cm. long and 6 cm. in width, but a wider one will answer, though the results will be less accurate. Fit the neck of the vessel with a tight-fitting indiarubber cork pierced with one hole; take a piece of ordinary glass tubing long enough to project a little distance below the cork while reaching up to the top of the glass vessel, and bend this tube (for the method of doing this, see p. 29) so as to follow the curve of the vessel and bring the tube when in position near to the side. About 2 cm. from the upper end of the glass tube make a small hole in its wall by means of a blowpipe and a piece of platinum wire as described on p. 31. Fit the stopper into the vessel, and carefully push the tube through the hole so that the end of the tube projects a short distance below the stopper; clamp the whole firmly so that it may not get tilted to one side or the other while being used (Fig. 8).

(*a*) Place a beaker or basin under the end of the tube: then fill the vessel nearly to the top with water and let the excess run out through the small opening down the glass tube into the beaker; it is best to have the lower end of the tube ground aslant as shown in the figure, as otherwise some of the water may remain inside the tube (for the method of doing this, see p. 31). Empty the water out of the beaker, and then weigh it: put it under the glass tube again; now take the hydrometer and place it carefully in the water in the vessel. Some of the water overflows—why? Weigh this water, and compare its weight with that of the hydrometer; what do you find?

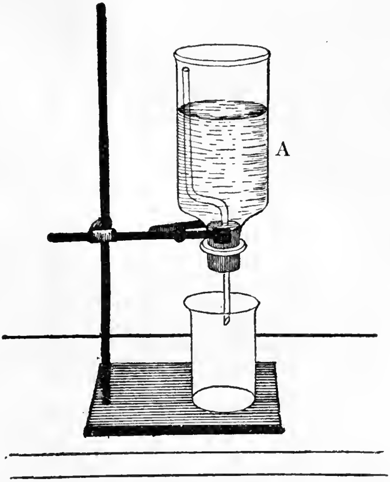

FIG. 8.—Apparatus for Exercise 10.

(*b*) Repeat experiment (*a*), using the 20 per cent solution of salt in place of water; what do you find?

(*c*) Repeat with the 10 per cent solution of copper sulphate in place of the solution of salt; what do you find?

You find that the hydrometer floats at different levels in the three liquids, but in each case the weight of the liquid displaced is equal to that of the hydrometer.

EXERCISE 11.—Notice carefully the levels at which the hydrometer floats in the solutions of salt, and of copper sulphate, using each in turn in a tall cylinder. To obtain a good result it is necessary to take the reading when the hydrometer is not touching the side of the cylinder to which you will notice it is always attached; this can be prevented by using a wire ring shown in Fig. 9, made up by twisting together two short pieces of copper wire (the length of each wire should be about twice the diameter of the cylinder) so as to form a loop; the loop is fixed in the cylinder below the water, and to avoid any error through friction between it and the hydrometer a few taps on the side of the cylinder will be found sufficient.

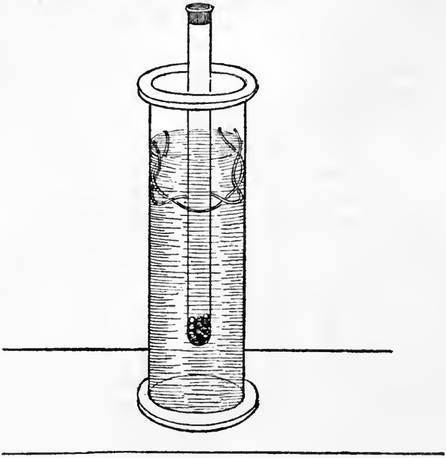

FIG. 9.—Hydrometer floating in a Cylinder of Liquid.

What is in each case the volume of liquid displaced by the hydrometer? What do you know, from Exercise 10, to be the weight of this volume? Hence calculate the density of each liquid and compare with the results obtained in Exercises 7 and 8.

Determination of the Relative Density of Solids.—To find the relative density or specific gravity of a solid we need to know, just as for a liquid, the volume and the weight of a given quantity of the substance. The weight of a solid is easily found, but the volume cannot be measured so readily as in the case of a liquid; in fact, direct measurement of the volume is only possible in the case of solids of regular shape, such as the sphere, cube, or cylinder, and even in such cases the best way of determining the volume of the solid accurately is an indirect one.

Exercise 12.—(*a*) Take the instrument fitted up in Exercise 10 and arrange it full of water with a measuring cylinder underneath; take a 100 gram weight, tie a fine thread (silk is best) round it, and lower it carefully into the glass vessel A, Fig. 8; some water overflows—why? What is the volume of the 100 gram weight, and what is its relative density or specific gravity?

(*b*) Repeat (*a*), using in place of the 100 gram brass weight a large iron weight, a large glass stopper (choose one without any hollow cavities inside), or any other object of suitable size that is not affected by the water. Find in each case the volume of the object used, and its relative density or specific gravity.

This is, however, at best, a single method; a far more accurate one is based on a principle to which the following exercise will lead us:—

Exercise 13.—We have found in Exercises 9 and 10 that when a body floats in water, it displaces a volume of water equal in weight to the floating body. The water then buoys up this floating body with a force equal to the weight of water displaced. Is there any such buoyant action on a body which is too heavy to float? Let us try.

(*a*) Take the 100 gram weight whose volume we have found in Exercise 12: hang it by a silk thread from the hook above the left-hand pan of the balance (Fig. 10); over the pan itself put a wooden bridge, and on this a beaker arranged so as to enclose but not touch the hanging weight; balance the 100 gram weight by another one in the other pan (if the two weights do not balance accurately, add smaller weights to the pan which needs them); then nearly fill the beaker with water (taking great care not to spill any on the pans of the balance) so that the weight hangs in water instead of in air. Try whether the balance is still maintained; if not, what do you conclude? Measure the buoyant action of the water on the weight by adding weights to the left-hand pan until the two pans again balance; compare this weight with that found in the preceding exercise to be the weight of water displaced by the 100 gram weight. What do you find?

(*b*) Repeat with the glass stopper or other article used in Exercise 12.

In each case we find that, so far as the character of the experimental methods used in Exercise 12 allows us to tell, the buoyant action of water on a body immersed in it is equal to the weight of the volume of water displaced, and as this gives us the volume of the immersed body, we have a new and accurate method of determining that volume. It is only necessary to measure the loss of weight of the body when immersed in water to know the weight of an equal volume of water; if this is expressed in grams, the same number will give the volume of the solid in cubic centimetres; dividing this into the weight of the body in air, we have its relative density

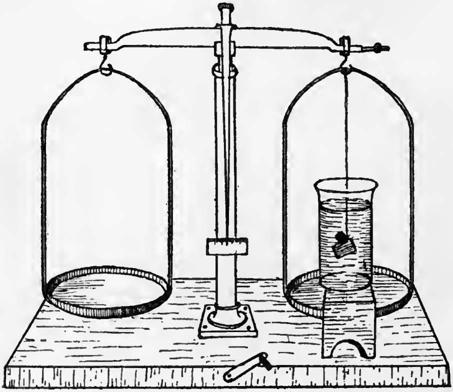

FIG. 10.—Balance arranged for Determination of Relative Density.

$$D = \frac{W}{W - W'}$$

where W = weight in air
W′ = weight in water

(*c*) Find in this way, using the apparatus and arrangement described in Exercise 12, the specific gravity of brass, iron, glass, etc.

The Measurement of Gases

It has been seen in the course of the foregoing experiments that it is easier to weigh a solid than to measure its volume; with a liquid either can readily be done, but in the case of a gas the measurement of its weight is rendered difficult by the necessity of enclosing it in a vessel whose weight must always be large compared with that of the gas enclosed. Here in our simple experiments gases will be measured, not weighed.

But the measurement of volume is also attended with certain difficulties in the case of gases which are scarcely noticed in the case of solids or liquids, except in some exact experiments: the volume of a given solid or liquid is well defined and subject only to slight variation with ordinary changes of pressure or of temperature, while the volume of a gas is so largely influenced by these conditions that they must always be taken into account in any but the very roughest experiments.

Influence of Pressure—Boyle's Law.—Under ordinary circumstances gases are subject to the pressure of the atmosphere, that is, to the pressure caused by the weight of the enormous thickness of air that lies above us; this pressure can be measured by means of a *barometer*.

EXERCISE 14.—Take two pieces of glass tubing, each 80 or 85 centimetres long, of which one must be closed at one end; connect them together by means of a piece of thick-walled indiarubber tubing about 10 or 12 cm. long, and with a pair of pliers twist pieces of copper wire tightly round the places where the indiarubber overlaps the glass tubing.

Let the closed end of the tube A rest on the floor, and holding B with the open end upwards pour clean dry mercury (see Note) into it through a small funnel with a drawn-out neck, until it is filled about 5 cm. above the indiarubber; if you look at the mercury in the tube you will notice there are a number of air-bubbles still left in the tube, and these must be removed as far as possible by carefully tapping the lower end of A on the floor; then place the tubes as shown in Fig. 11, in which *c* and *d* are two corks with grooves cut in their sides to receive the tubes; these are kept firm by string tied round them or by indiarubber bands.

What happens when the tube A is lifted to its final position? Why does the mercury fall?

Measure the distance between the levels of the mercury in the two tubes, keeping the barometer fixed as exactly vertical as you can make it; what is this distance in inches? What is it in millimetres? In chemistry we generally express the height of the barometer in millimetres, and the pressure of the atmosphere as equal to so many millimetres of mercury.

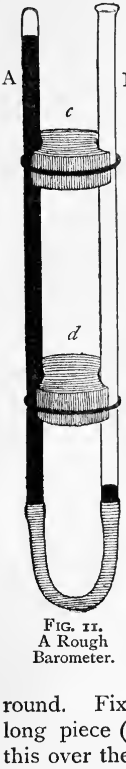

FIG. 11.
A Rough
Barometer.

(*a*) Attach a piece of thick-walled indiarubber tubing to the open end of B; blow into this—what happens? Suck air out through this tube—what happens? Why?

(*b*) If you have an air-pump that you can use (one of the cheap filter-pumps which have to be connected to the water-tap will be the most convenient), attach the piece of indiarubber tubing to it, and pump out the air from the tube B. What happens? How does this give you a method of finding out whether your air-pump works properly?

EXERCISE 15.—Take a piece of glass tubing about 25 cm. long, closed at one end, and about 8 mm. inside diameter; attach to the open end a piece of thick-walled indiarubber tubing about a metre in length, and to the other end of this attach another piece of glass tubing B (Fig. 12), of which the mouth has been expanded by the method described on p. 31; make both connections firm by means of copper wire tightly twisted round. Fix A firmly in the clamp of a retort stand; tie a long piece (4 metres) of string round the neck of B and pass this over the arm of a gas-bracket above your bench, fixing the loose end of the string tightly round a nail in the wood-work of your shelves, or round the end of the clamp used for supporting A.

Put the lower ends of A and B on a level: pour clean dry mercury into B until it can be seen above the indiarubber on this side, then carefully remove A from its clamp and lower it to let a little of the imprisoned air escape; pour more mercury into B, and let a little more air out of A, until finally you get the mercury at the same level in B as in A, and standing about half-way up this latter tube.

(*a*) Now mark the level of the mercury in A by a little indiarubber band slipped over the tube; what is now the pressure on the air in A? Raise B, what effect has this, and why? Continue to raise B until the volume of the air in A is as nearly as you can tell half what it was at first (remember that A is somewhat narrow just at the closed end, and make some allowance for this), and measure the difference in level of the mercury in the two tubes. What is now the pressure on the air in A expressed in millimetres of mercury?

(*b*) Lower B until the volume of the air in A is, as nearly as you can tell, $\frac{2}{3}$rds of what it was at first; again measure the difference in level of the mercury in the two tubes.

(*c*) Lower B until the volume of the air in A is again equal to the original volume. What is now the pressure? Lower B still further until this volume increases by a half; measure the difference of level of the mercury and so find the pressure. What is it?

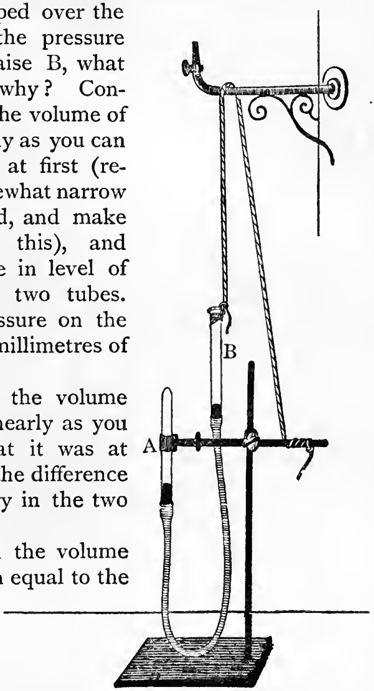

FIG. 12.—Apparatus for illustrating Boyle's Law.

(*d*) Finally lower B until the volume of the air is double what it was under the atmospheric pressure. What is now the pressure on the gas?

Tabulate your results in the following manner:—

Volume = V	Pressure = P	VP
1	? mm. of mercury	?
$\frac{1}{2}$	?	?
$\frac{2}{3}$	?	?
$\frac{3}{2}$	?	?
2	?	?

What do you find about the numbers in the third column under the heading VP?

The law you have just verified for yourself in Exercise 15 was originally discovered by Robert Boyle in 1660, and is called after him Boyle's Law; it is generally stated in this form:—*The volume of a gas is inversely proportional to its pressure.* It is true not only for air, but for other gases within certain limits.

Influence of Temperature—Dalton's Law.—We have found that the volume of a gas changes very considerably when the pressure upon it is altered: let us see what effect change of temperature has upon the volume of a gas; but first we must have a means of measuring temperatures. This is done by using thermometers.

EXERCISE 16.—Examine a thermometer; what is it made of? Hold the bulb in your hand so as to warm it; what happens, and why? There is a graduated scale on the thermometer; notice the reading of the thermometer when the bulb is (1) held in the hand, (2) put in cold water from the tap.

(*a*) Take some ice and break it up into small lumps: put these in a large funnel, and then support the thermometer with its bulb well buried in the ice; let the whole stand for a short time, then read the thermometer; wait a minute, then read again, and so on until six or eight readings have been taken. What do you find?

(*b*) Fit the thermometer cautiously into one of two holes in a cork, taking care not to break the thermometer ; a piece of glass tubing bent at right angles should be fixed in the second hole of the cork, and then this placed firmly in the neck of a large flask containing about 100 cc. of water. The whole arrangement should be such that the bulb of the thermometer is an inch or two above the water. Put the flask over the burner and heat the water till it boils ; take readings of the thermometer at intervals of a minute until a good part of the water has been boiled away. What do you find ?

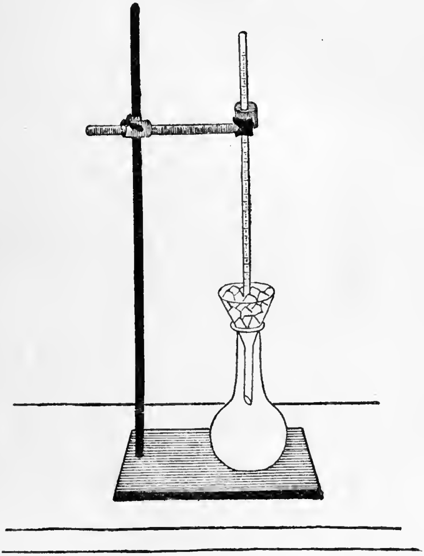

FIG. 13.—Apparatus for Exercise 16 (*a*).

The thermometer, supposing it to be a Centigrade one, registers 0° in the melting ice, and 100° C. in the vapour of boiling water ; these are the two fixed points of the thermometer, and the interval between them is divided into 100 degrees. Other thermometers than the Centigrade have the same fixed points, namely the melting point and boiling point of water, but use other numbers: thus the Fahrenheit thermometer which is in common use in this country calls the freezing point 32° F. and the boiling point 212° F. In this book we shall use the Centigrade thermometer exclusively.

EXERCISE 17.—Take a round-bottomed glass flask, large enough to hold 400 or 500 cc., but not too large to go inside the pan of water; set the pan over a flame to boil the water, and in the meantime dry the flask thoroughly inside by blowing air from a foot-bellows into the flask while it is kept hot by a flame (keep turning the flask round so that it may not crack): next fit an indiarubber cork with two holes to the neck of the flask, pass the thermometer through one hole, and a short piece of glass tubing through the other; to this tube fix a short piece of indiarubber tubing with a spring clip by which the tube can easily be closed or opened at will. The cork, thermometer, and tubes must all be thoroughly dry.

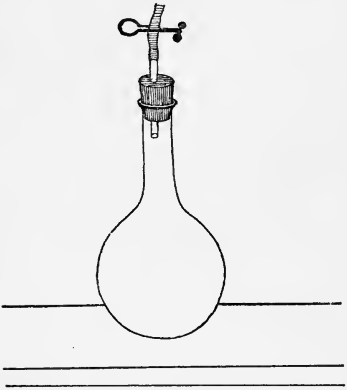

FIG. 14.—Round-bottomed Flask fitted up for Exercise 17.

Put the flask in the pan of boiling water, first holding it for a minute or two in the steam; open the spring clip and read the thermometer, repeating both these at short intervals until the readings of the thermometer become constant. Then see that the clip is properly closed, and remove the flask from the pan. Let it cool for a minute, then put it neck downwards in a large basin of cold water and open the clip. What happens? Why?

Shake the flask, keeping its neck all the time under water: open the clip, then close it again; remove the flask, turn its neck upwards and read the thermometer; take out the cork and measure the amount of water *v* which has entered. Then measure the volume of the flask up to the level which the cork occupies when in the neck, call this V. The experiment tells us that a volume of air which measures $V - v$ at the lower temperature fills V cc. at the higher temperature of the experiment; calculate (1) the increase in volume of 1 cc. of air at the lower temperature when raised to the higher temperature, and (2) the rate of this increase per degree.

(*b*) Dry the flask, cork, and tubes thoroughly: then fill the flask with coal gas by displacement; cork up at once, and repeat the experiment. Compare the result with that got when you used air.

You cannot expect by this method to obtain very accurate results; more careful investigations have proved that the expansion of gases by heat can be represented by the statement "a volume of gas measuring 273 at 0° C. becomes $273 + t$ at t° C." How do your results obtained in Exercise 16 agree with this law?

CHAPTER III

MIXTURES AND COMPOUNDS

We now proceed to the more purely chemical parts of our study, and must first try to form clear ideas as to what is meant by a chemical *compound* and how we distinguish it from a mixture.

Exercise 18.—Mix together about equal quantities of sugar and sand: taste a little of the mixture; how are the properties of the mixture related to those of the ingredients?

(*a*) How can you separate the sugar from the sand? can you pick out any bits of sugar or sand with your fingers? Rub some of the mixture in a mortar; can you separate the sugar and sand now?

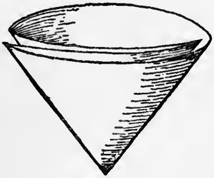
Fig. 15.—Folded Filter Paper.

(*b*) Put what you have powdered in the mortar into a beaker, add water and stir well or heat on the sand-bath; what happens? How can you separate the solution of sugar from the insoluble sand? Take a piece of filter paper, fold it as shown in Fig. 15, then put it in a funnel and fit the paper carefully to the sides of the funnel; place the funnel in the mouth of a flask and now pour the contents of the beaker into the funnel, taking care not to let the liquid fill the funnel up to the top of the filter paper. What is the result?

(*c*) Mix some salt and chalk: taste a little; how do the properties of the mixture compare with those of the ingredients? Separate them in the same way as before; is the separation complete? Try this by taking a weighed quantity of salt and of chalk (both should be dry), mixing, and then separating as before; dry the funnel with the chalk and filter paper in a steam-oven, then weigh the chalk and the filter paper (but without the funnel), putting an equal piece of filter paper in the other pan of the balance so as to get the weight of the chalk itself. To find the weight of the salt, take a weighed evaporating dish, put the solution of salt into it (or if there is too much liquid put in only part at first and afterwards add more) and evaporate it to dryness on the sand-bath. Let the dish cool and weigh it with the salt. How does the weight of salt obtained compare with the amount used?

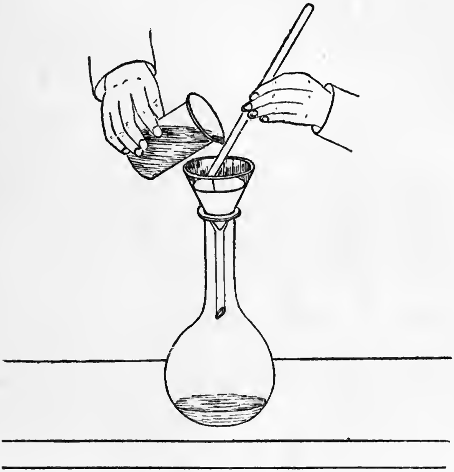

FIG. 16.—Filtering.

(*d*) Mix some fine iron-filings with about twice the weight of flour of sulphur: examine the properties of the mixture; is it attracted by a magnet like iron, and will it burn like sulphur? Try to separate the iron and sulphur again (1) by the magnet, (2) by shaking with water. You find that neither the iron nor the sulphur will dissolve in the water, but other liquids could be used to separate the sulphur by dissolving it, just as the salt and sugar were separated by solution in water; such a liquid is carbon disulphite, but its use by a class is dangerous and objectionable on account of its inflammability and bad smell.

(*e*) Put a little of the dry mixture of salt and chalk in a test-tube and heat it for a few minutes in the flame of your burner; let it cool, then shake out the contents and examine them to see whether any change has occurred.

(*f*) Put some of the mixture of iron-filings and sulphur in a dry test-tube, and heat it in the same way; what happens? Let it cool, then break open the tube and examine its contents; powder them as well as you can in the mortar, and try to separate it into iron and sulphur by the methods used in (*d*).

In the case when iron and sulphur are heated together, a compound is formed called sulphide of iron; its properties are quite different from those of its constituents. What then is the difference between a compound and a mixture? It was Robert Boyle who first clearly distinguished between the two in the seventeenth century, and he gave as the characteristic of a compound that its properties are quite different from those of the substances composing it. A compound is also generally much more difficult to split up into its constituents than is the case with a mixture.

All substances are not included in the two classes of mixtures and compounds; there is a third very important class, that of the *elements*. These are substances which cannot be split up or decomposed by any known method. As you have not time to apply all the methods, even in the case of any one substance, we must be content with this definition; you will learn more about the elements and their number elsewhere (Roscoe and Lunt, p. 21), and you will presently have to prepare some of the most important among them and examine their properties.

CHAPTER IV

ON SETTING UP APPARATUS

IN what follows you will often have to fit together glass flasks and tubes by means of corks, to bend glass tubes, and do other things for which you will need some directions.

Corks.—When fitting a cork to a flask or tube, choose one which seems rather too large for the opening it has to fit; soften it by rolling it under your foot, and you will then find, if your choice has been a good one, that the cork fits tightly, and is easily put into its place. If it is wished to keep the cork clean and the floor is dirty, wrap the cork in a piece of paper before rolling it.

To bore holes in corks, instruments called cork-borers are used; these are brass tubes, sharpened at one end and provided at the other with holes through which a stout wire can be put to serve as a handle. Select a borer of the exact size of the tube which has to fit the hole, or failing this the borer should be rather smaller than the tube; in boring press steadily, but not too heavily, and keep turning the borer round; when the borer is nearly through the cork finish by pressing against a *wooden* surface.

Indiarubber corks may be used, and for some purposes are almost a necessity; they are expensive if many are lost; each student should be provided at the commencement with two of different sizes, the larger one with two holes to fit a 600 cc. (= 20 oz.) flask, the smaller one with one hole to fit a 75 cc. flask (the size called 2 oz. by the dealers).

To work Glass Tubing.—To *bend* glass tubing an ordinary flat-flame burner must be used, not the Bunsen; heat the glass in the flame, holding the tube in line with the width of the flame and turning it slowly round all the time until it is soft enough to bend easily, then remove it from the flame and bend to the angle required.

To *draw out* glass tubing, either the Bunsen flame may be used, or better, the foot blowpipe arranged to give a pointed flame a few inches long ; the glass should be allowed to become much softer than is necessary when bending a tube, then remove it and slowly draw the two pieces apart.

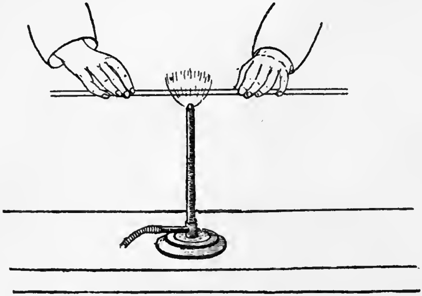
FIG. 17.—Bending Glass Tubing.

To *cut* glass tubing a triangular file is used : make a scratch with this at the point where the tube is to be cut, then break it by a pull and bend combined, taking care to have the file-mark uppermost and to bend downwards. The sharp edges of the cut can be roughened by the flat surface of the file, or can be rounded off by heating in a Bunsen or blowpipe flame until the glass just melts at the edges and colours the flame distinctly yellow.

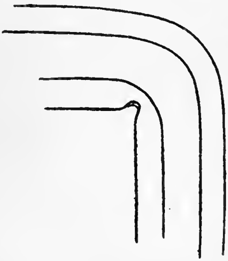
FIG. 18.—Good and Bad Bends.

If it is required to *seal* one end of a glass tube, it is more convenient to start with a tube a couple of inches longer than the one wanted : heat the tube in the blowpipe flame at the point where it is to be sealed, and keep the glass in rotation until it is quite soft ; then remove from the flame and draw off the short piece. Cut the narrow drawn-out tube across, and heat the tube at the beginning of the narrower part in the blow-pipe flame, which must now be small and pointed; draw off the fine tube slowly as the glass softens, and finally seal the tube. To make the end round and of uniform thickness, it must now be heated in a rather large flame, always keeping the tube in rotation, until the glass has thickened considerably; then blow gently into the open end so as to expand the thickened part slightly and give it the desired shape.

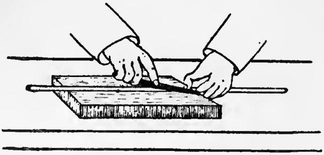

FIG. 19.—Cutting Glass Tube: First Operation.

To *widen the opening* of a glass tube we require a piece of charcoal with a rather blunt conical point; the end of the tube for about half an inch is heated in the large flame of the blowpipe until it becomes soft, the charcoal is then pushed in with a steady pressure and rotary motion combined.

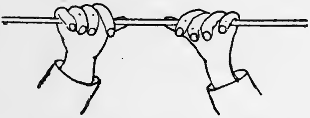

FIG. 20.—Cutting Glass Tube: Second Operation.

To *grind* glass it should be kept moistened with a solution of camphor in turpentine: this solution should be made in small quantities at a time as it does not keep very well; thus if it be wished to grind a slanting end to a glass tube as required in Exercise 10, p. 15, it is easily done by rubbing the glass against an ordinary file kept moist with this solution.

To *make an opening in the side of a glass tube*, heat the tube at the point where the opening is to be made with the blowpipe flame until the glass is quite hot; then push the end of a piece of thin platinum wire into contact with the hot glass and draw out slowly the wire and the glass adhering to it.

CHAPTER V

THE AIR

Action of Air on Metals.—You know that many changes occur to substances exposed to the air, which do not take place when the air is excluded; we must now examine some of these changes, with the hope of finding out something as to the nature of air and of the part it plays in these processes, taking first of all the case of the more common metals.

EXERCISE 19.—(*a*) Heat a small porcelain crucible supported on a pipe-clay triangle in the Bunsen flame, let it cool and then put in it some pieces of tolerably clean lead; weigh the crucible with the lead in it; now heat the crucible again with the lid on until the lead is melted, then allow to cool and weigh again. Is there any change of weight? Again heat

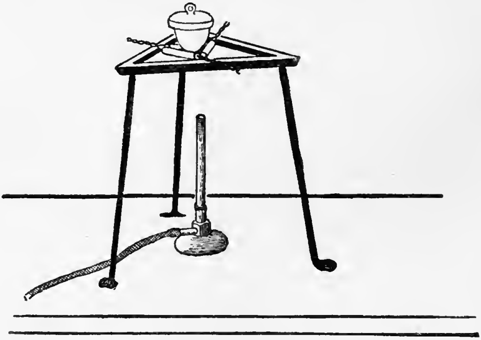

FIG. 21.—Heating a Porcelain Crucible.

the crucible with the lid off and keep the lead melted for half an hour, let it cool and weigh again—is there now any change of weight? If so, try to suggest the reason.

(*b*) Repeat the above experiment, using some clean copper turnings in place of the lead (fresh copper turnings are often greasy, but are easily freed from grease by washing with a little alcohol). Then do the same with some iron-filings, which should also be freed from grease in a similar way before being used. Try any other metals that are easily to be obtained, and compare the results, drawing what conclusions you can.

You will now have discovered that when metals are heated in air they are in many cases changed into new substances, and that the new substance obtained weighs more than the metal from which it was got. The following experiment should enable you to come a step nearer to a satisfactory explanation of the fact you have just ascertained:—

EXERCISE 20.—Take a glass cylinder or wide-mouthed glass bottle, pour some water into it and shake well in order to wet the inner surface of the cylinder; now put in some fresh iron-filings and distribute them over the inside surface by shaking; place the cylinder mouth downwards in a basin of water and leave for a day or more. Notice what has happened in the interval; leave again for a day or more until finally the water rises no further inside the cylinder, then put a glass plate to the mouth of the cylinder and remove it with its contents, taking care not to allow any air to enter; turn the cylinder mouth upwards, remove the glass plate and at once test the gas left with a lighted taper. What happens? Try to suggest an explanation.

Action of Air on Phosphorus.—There are very many other substances besides the metals which undergo similar change in air, especially when heated; thus anything that burns in air would come under this head, but generally these instances are less simple to examine and the results more difficult to explain; we shall therefore choose for our next experiment not a substance like coal or wax, but the less common body phosphorus, which has a great advantage for our purpose over coal and wax, both because it is an element, while coal and wax are compounds undergoing change when heated without access of air, and also because it burns far more readily than these substances do.

EXERCISE 21.—Phosphorus has to be kept under water on account of its great inflammability; you must take the greatest care in using it, and on no account touch it with the fingers unless under water. Cut off a small piece of phosphorus the size of half a pea; dry it carefully by pressing between filter paper, then put it in a *dry* porcelain dish; light the phosphorus by touching it with a hot wire, then immediately place over it a large thoroughly dry glass bottle. Notice what happens: when the phosphorus goes out, remove the bottle, pour some water into it and shake.

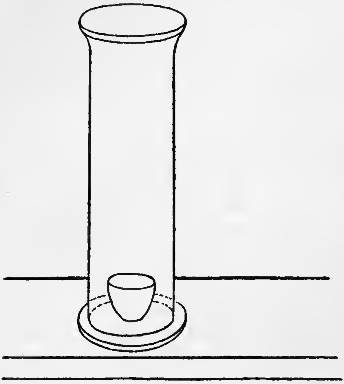

FIG. 22.—Apparatus for Exercise 21.

This last experiment shows that when phosphorus burns in air, it is converted into a white solid substance, which dissolves in water; remembering this fact, let us now go on to another experiment similar to the last, but arranged to bring out another and separate point.

EXERCISE 22.—Take a piece of phosphorus the size of a large pea, dry it, put it in a porcelain dish floating on water; light the phosphorus and then at once place over it, with the mouth under water, a glass cylinder or wide-mouthed bottle. Wait until the phosphorus has gone out and the fumes have nearly or quite disappeared, then shake the bottle so as to fill the porcelain dish with water and sink it; put a glass plate to the mouth of the bottle and remove this with its contents: about how much air is left in the bottle? Turn the cylinder mouth upwards, remove the plate, and test the gas with a taper; what happens, and what are your conclusions?

The Inactive Constituent of Air.—You have seen from Exercises 20 and 22 that when iron rusts or phosphorus burns in air a gas is left which is apparently the same in each case; moreover, in both experiments you have found that, so far as you can tell by the rough methods employed, there is the same proportion of gas left from the air whether iron is made to rust in it or whether we burn phosphorus. It seems then likely that this gas which puts out a taper is present originally in the air along with some other gas which is removed by the action of the rusting iron or the burning phosphorus; this opinion has been confirmed by many other experiments, and the inert gas present in air is called nitrogen; we shall learn more about it later on.

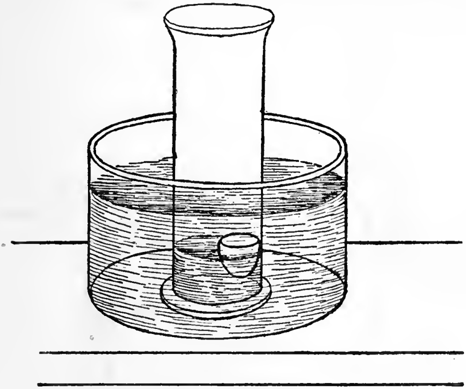

FIG. 23.—Apparatus for Exercise 22.

The Active Constituent of Air.—But what is the other gas which is removed in combination with the iron or phosphorus? In order to get this gas we might make experiments, hoping to obtain it from the iron rust or from the white substance formed by the burning of the phosphorus; the compounds formed in these two cases are, however, not readily decomposed, and we must choose some other substance than these in order to be successful. When the metal mercury is heated in air it is slowly converted into a red substance which may be regarded as the "rust" of mercury, and if this action be made to occur under suitable experimental conditions (see

Roscoe and Lunt, p. 45) it is found that the same gas nitrogen is left as in the two cases we have studied ; evidently the mercury has united with the other and active constituent of air to form this red rust of mercury. Now try the following experiment with some of it :—

EXERCISE 23.—Take a clean dry test-tube, or better, a piece of hard glass tubing closed at one end ; fit it with a sound cork through which is fixed a glass delivery tube bent as shown in Fig. 24 ; put about 4 or 5 grams of the red rust of mercury into the hard glass tube, then weigh it without the cork ; now replace the cork, and support the tube either by a clamp or by two rings connected with a retort stand so that the lower portion can be strongly heated without burning the cork ; let the farther end of the bent delivery tube dip under water in a dish or other suitable vessel. Heat the oxide of mercury in the tube by means of the Bunsen burner, moving the flame about at first in order to heat the tube gradually and lessen the risk of cracking it. Notice carefully what happens.

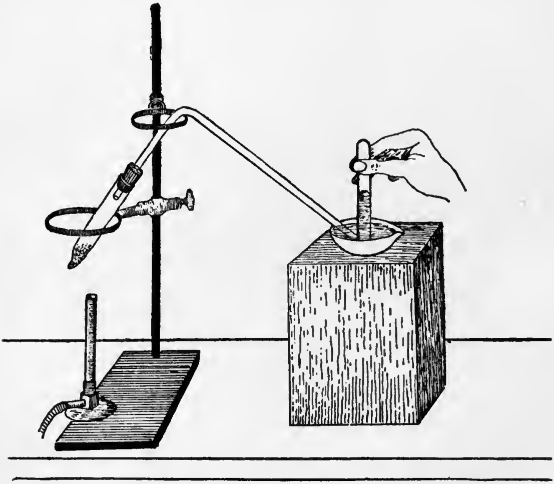

FIG. 24.—The Decomposition by Heat of Oxide of Mercury.

Collect the gas which comes off in a test-tube; this must be filled with water, the thumb placed firmly over the mouth of the tube, which is then turned downwards and placed under the water in the dish; the thumb may then be removed, and on holding the test-tube over the end of the delivery tube the bubbles of gas will rise up and displace the water. When the test-tube is full of gas, close the mouth of it with your thumb, remove the tube from the basin and test the gas in it by plunging into it a splint of wood which has been lighted and then the flame blown out, leaving a glowing spark at the end of the wooden splint.

What happens when the first test-tube of gas is tested? What with the second and third?

Continue to heat, until no more gas comes off; then let the tube cool, and weigh it with its contents; what is the substance left, does it weigh more or less than the rust of mercury taken? How can the difference be explained?

Oxygen and the Proportion of it in Air.—The gas obtained in the last experiment is called oxygen, and was first made by Priestley in 1774 by the same method as you have used; it was this discovery which first made possible a satisfactory explanation of what happens when metals rust or are calcined in air and when combustible substances burn; Priestley himself was unable to see the correct explanation even after he had discovered oxygen, but he carried the news to a French chemist, Lavoisier, who very soon came to the conclusion that air is a mixture of the two gases which we call oxygen and nitrogen, and that the oxygen alone takes part in rusting or in the burning of bodies, combining with them to form compounds called oxides. You will find more about this very important subject in your text-book on theoretical chemistry; we now go on to try two simple methods by which we can find roughly the proportion in which oxygen and nitrogen are mixed in the air.

EXERCISE 24.—(*a*) Take a glass tube closed at one end, about 50 cm. long and 2 cm. in diameter; measure its volume —v_1—by filling it with water from a graduated cylinder. Take a piece of copper wire about 50 cm. long, bend a small loop at one end, and at the other turn back about 10 cm.; put a piece of phosphorus, the size of a small nut, in a test-tube, and just cover it with water: heat some water in another test-tube to boiling and pour some of the hot water over the phosphorus until this melts completely; put the hooked end of the copper wire into the molten phosphorus, and cool the test-tube containing it under the tap until the phosphorus becomes quite solid again.

Withdraw the wire with the piece of phosphorus on the end and put it in the long glass tube, which must be full of water; let this water run out into a cylinder such as is used for collecting gases, emptying the tube completely, then at once put the tube, with its mouth still downwards and the phosphorus on its wire still inside, into the cylinder of water (Fig. 25). The phosphorus soon begins to give off white fumes—what is happening to it? In the dark it would be seen to glow, and it is really burning very slowly and combining with the oxygen of the air. Take the temperature, t_1, of the water which has run out of the tube; this we may take to be the temperature of the air which is now inside the tube. Read also the height of the barometer, p_1.

Put away the whole arrangement for a day or more, keeping careful record in your notebook of the observations you have made so far. Then go on with Exercise 25.

(*b*) When you return to this experiment, you notice that the phosphorus has stopped fuming, showing that it has used up all the oxygen in the tube; we must now go on to measure the volume of nitrogen left, and this will tell us how much nitrogen there was in the volume of air taken.

FIG. 25.—Rough Analysis of Air by means of Phosphorus.

You must first make the level of the water the same, inside and outside the long tube, so that the gas may be measured under the pressure of the atmosphere; do this by pouring water into the outer cylinder, or by raising the inner tube as the case may require. Slip a narrow indiarubber band over the tube and place it at the level of the water inside the tube; the volume—v_2—of the tube between the rubber band and the closed end is the volume of the nitrogen left. Measure this by pouring water into the tube from a graduated cylinder.

It is, however, necessary to take account of the temperature and pressure at which this volume v_2 is measured; take the temperature of the water left in the cylinder (not the graduated one) as the temperature t_2, and read the height of the barometer to get p_2.

Then if the nitrogen had been measured at the same temperature t_1 and pressure p_1 as those at which the air was measured, its volume would have been found as

$$v_2 \times \frac{p_2}{p_1} \times \frac{t_1 + 273}{t_2 + 273}$$

Work out what this is equal to, but do not go beyond the first place of decimals (supposing you are measuring the volumes in cubic centimetres); call the result v_2^1. Then a volume v_1 of air has been found to contain a volume v_2^1 of nitrogen, and the percentage of nitrogen in the air is

$$\frac{v_2^1}{v_1} \times 100$$

Find from this the percentage of oxygen in the air, and compare your numbers with those of other students; they should all be nearly alike, proving that, as far as this not very accurate method will show, the proportion of oxygen to nitrogen in the air is constant.

Rough Analysis of Air by means of Pyrogallic Acid.—Another and quicker method makes use of the fact that a solution of pyrogallic acid in water containing some caustic soda or caustic potash rapidly takes up oxygen from the air.

EXERCISE 25.—(*a*) Put a little pyrogallic acid—which you see is a white crystalline solid and is obtained from oak-apples—into a test-tube half full of water; shake until the pyrogallic acid is dissolved. You notice that the solution is at first nearly colourless, but gradually becomes darker; pour half into another test-tube, and to this add a few drops of sodium hydrate (caustic soda) solution from the bench bottle; the latter half darkens much more rapidly than the other, and in each case the darkening is due to absorption of oxygen from the air, whereby the pyrogallic acid is converted into various dark-coloured substances.

(*b*) Take a tube closed at one end, 70 or 80 cm. long and about 12 or 15 mm. inside diameter, fitted at the open end with a good indiarubber stopper. Weigh out about half a gram (not more) of pyrogallic acid, put it in a test-tube, add half a test-tube full of water and shake until solution is effected; pour this into the long tube and mark the level of the liquid by an indiarubber band—the volume from this band up to the open end of the tube is the volume of air taken for analysis.

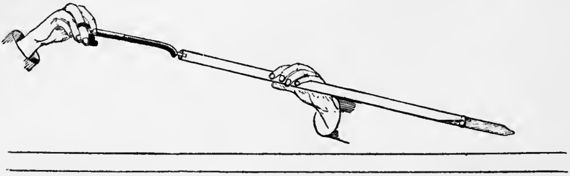

FIG. 26—Introducing the piece of Sodium Hydrate in Exercise 25 (*b*).

Now slant the tube, and slide into it (but not into the liquid) a piece of solid caustic soda about the size of a large pea; at once insert the rubber stopper and then let the lump of caustic soda fall down into the solution of pyrogallic acid; shake the tube and its contents occasionally during ten minutes, putting the tube flat on the bench when you are not shaking it.

At the end of these ten minutes the whole of the oxygen in the air enclosed in the tube will have been absorbed; slip a second indiarubber ring over the stoppered end of the tube, then put it mouth downwards in a cylinder of water and remove the stopper, taking care that no air is at this point allowed to enter the tube. Raise or lower the tube until the level of the water is the same inside and out, then mark this level by means of the second indiarubber band (do not disturb the first). The volume from this second band to the closed end of the tube measures the amount of nitrogen in the air used.

You have then to measure v_1, the volume from the first rubber band to the open end of the tube, and v_2, the volume from the closed end of the tube to the second rubber band; then v_2 is the amount of nitrogen in a volume v_1 of air, and the percentage of that gas is $\frac{100\,v_2}{v_1}$, while that of oxygen is

$\frac{100\,(v_1 - v_2)}{v_1}$. Calculate these from your measurements of v_1 and v_2, and compare the results with those of other students as well as with those obtained in the preceding exercise.

The Composition of Air.—What then do you now conclude as to the composition of air? You have shown that it contains two gases which we call oxygen and nitrogen, and that of these oxygen makes up about one-fifth, and nitrogen the other four-fifths of the volume of air; in oxygen, you have found, a match burns more brightly than in air, while the nitrogen puts out the flame entirely; both gases are colourless and have no smell. The properties of air are accordingly not markedly different from those of its constituents, but are intermediate between them, so that you would judge that air is a *mixture* of the two gases and not a compound if you refer to the distinction originally given by Boyle and mentioned on p. 28: this conclusion is confirmed by many other considerations, for which you must consult your teacher or some other book (Roscoe and Lunt, p. 134).

CHAPTER VI

OXYGEN

Oxygen from the Rust or Scale of Metals.—You have found by previous experiments that air contains as its active constituent a gas called oxygen which unites with many metals when these are exposed to the action of the air, and converts them into their oxides ; these oxides are, in the case of some of the common metals, ordinarily called rust or scale, and it was further seen that oxygen could be obtained by merely heating the oxide formed by the action of air on hot mercury: we must now find some less expensive method of making the gas and then proceed to examine its properties in more detail.

EXERCISE 26.—Try whether you can obtain oxygen from any of the following oxides by heating them in a test-tube (best of hard glass) fitted with cork and delivery tube as in Fig. 24 :—Iron oxide, copper oxide, litharge, red lead, manganese dioxide. Note carefully in each case any change that occurs, and if a gas is given off collect it in test-tubes and test whether it is oxygen (if you have forgotten how to do this look at p. 37).

Oxygen from Potassium Chlorate.—Although some of these oxides give off oxygen when strongly heated, none of them affords a very convenient method of preparing the gas ; a much better substance to use for this purpose is potassium chlorate, a white crystalline solid of somewhat more complex composition than the oxides.

EXERCISE 27.—(*a*) In a test-tube, filled as in the preceding exercise, put about 2 grams of dry potassium chlorate ; heat it, noticing carefully what happens, and collect any gas which may be given off. Put the test-tube with the white residue on one side for use in the next exercise.

(*b*) Repeat this experiment, adding a weighed quantity of manganese dioxide, about half that of the potassium chlorate used; what gas is evolved on heating the mixture—does it come off more readily than in (*a*)?

When no more gas is evolved let the test-tube cool and then treat the residue with hot water; filter to separate the insoluble residue, and collect the whole of this residue on the filter paper; let the clear filtrate run into a clean vessel and keep this for further examination: wash the residue twice with hot water poured over the paper so as nearly to fill it, then put the funnel and paper into a steam-oven to dry; or carefully spread the filter paper with the residue on a piece of wire gauze, which is supported at least six inches above a rather small flame. When dry (at least half an hour will be needed) weigh the black residue, putting an unused filter paper of the same size in the pan with the weights to balance that on which the residue is supported: compare the result with the weight of the manganese dioxide used. What do you conclude?

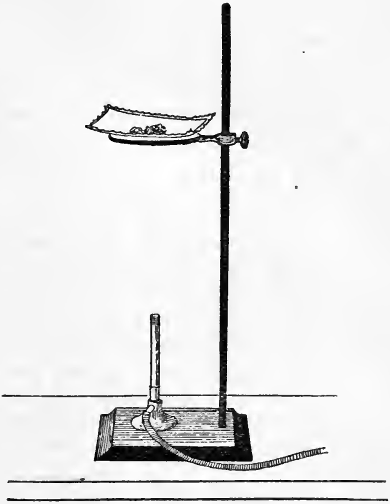

FIG. 27.—Drying a Solid Residue over a small Bunsen Flame.

The Decomposition of Potassium Chlorate by Heat.—From these experiments you conclude that when potassium chlorate is heated it is decomposed and gives off oxygen gas, a reaction which takes place more readily when manganese dioxide is mixed with the potassium chlorate, although the former substance is left unaltered at the end of the process. What is formed from the potassium chlorate besides the oxygen which is evolved? Evidently it is the white solid which is left in the test-tube in Exercise 27 (*a*), and is dissolved in the clear filtrate obtained when the unaltered manganese dioxide is separated in Exercise 27 (*b*).

Exercise 28.—Dissolve a few crystals of potassium chlorate in water and add some solution of silver nitrate; test in the same way a solution of some of the white solid left in Exercise 27 (*a*), and some of the clear filtrate obtained in Exercise 27 (*b*). What is the result in each of these cases?

We find that when heated either alone or with manganese dioxide, potassium chlorate is converted into a new substance which is a white solid like the chlorate, but differs from it in that the solution of the chlorate gives no precipitate with silver nitrate, while a solution of the new substance gives a thick white precipitate with that re-agent. This new substance is called by chemists potassium chloride; you have found that it gives off no oxygen when heated, and as a matter of fact it contains no oxygen at all, but is a compound of two elements, potassium and chlorine, with the formula KCl; while potassium chlorate contains oxygen besides these two elements, and has the formula $KClO_3$.

The Properties of Oxygen.—You must next prepare oxygen in larger quantity with a view to a more complete examination of its properties:—

Exercise 29.—Mix about 12 grams of dry $KClO_3$ with half the weight of dry manganese dioxide; put the mixture into a rather large test-tube fitted with an air-tight cork and a delivery tube, arranged as shown in Fig. 24. Heat the mixture in the tube, beginning near the cork and gradually moving the flame towards the closed end whenever the evolution of gas becomes too slack; do not use too large a flame, and turn the air-regulator at the bottom of the Bunsen burner so that the flame is just slightly luminous—it is then not so hot, and less likely to destroy the tube.

Collect 5 jars of the gas and then make the following experiments:—

(i.) Test the gas in the first jar with a glowing splint.

(ii.) Take a small piece of phosphorus the size of half a pea, dry it *cautiously* by pressing between filter paper, then put it in a dry deflagrating spoon, and immediately place this in the second jar of oxygen (Fig. 28). What happens? Then remove the spoon, light the phosphorus, and at once put it back again in the jar.

When the phosphorus has burnt out, take away the spoon, pour a little water in the jar, cover it with a glass plate and shake thoroughly for two or three minutes until the thick white fumes have dissolved. Take out a little of the solution on the tip of your finger and taste it; to the rest add some blue litmus solution—what happens?

Fig. 28.—Burning Phosphorus in Oxygen.

(iii.) Burn a small piece of sulphur in a jar of oxygen; notice that the product is gaseous and is characterised by a peculiar smell; test its solution in water as before.

(iv.) Burn a piece of charcoal in gas; the product is again gaseous, but has no smell; test its solution in water by taste and with litmus.

(v.) Burn a piece of magnesium ribbon—held by crucible tongs—in oxygen; the product is a white solid; try whether this is soluble in water, and whether it has any action on blue litmus.

The substances burnt in the last four experiments are all of them elements, and the product is in each case a compound of the element with oxygen, called an *oxide;* the properties of the several oxides are very different, and while several of them when dissolved in water form *acids* able to turn blue litmus red, that is not the case with the oxide of magnesium. The name oxygen, which means acid-producer, is in fact a misnomer, as while some of the oxides form acids with water, others do not: indeed many of the oxides have opposite properties to those of acids and are able to neutralise them; but the further consideration of this matter is left until a later chapter.

Quantitative Examination of the Decomposition of Potassium Chlorate by Heat.—We will now go on to an experiment to find out how much oxygen can be got from a given weight of potassium chlorate.

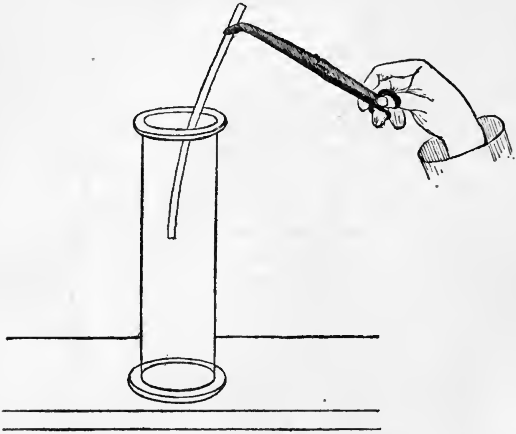

FIG. 29.—Burning Magnesium in Oxygen.

EXERCISE 30.—Take a flask holding about 400 to 500 cc. fitted with a rubber cork through which pass two holes; one of them carries a short tube *a* with the end bent downwards, while through the other one is fixed a tube *b* reaching quite to the bottom of the flask; to the free end of *a* is attached a short tube of hard glass *c* by means of a rubber stopper, and on *b* there is put a piece of indiarubber tubing which can be kept closed by a spring clip; this piece of rubber tubing leads by a straight piece of glass tubing into a large beaker or other vessel *d* capable of holding at least 300 cc.

Fill the large flask with water; replace the stopper and tubes, but without *c*; open the spring clip and blow into *a* until the tube *b* is full of water, then close the clip; put the empty vessel *d* in position.

Clean and dry the hard glass tube *c*, then weigh it; next put into it about 1 gram (not more than 1·1 gram) of dry $KClO_3$, and weigh again. Attach *c* to the tube *a* by the rubber stopper; open the spring clip and move it into such a position that it will be kept open by the unyielding glass tube.

Heat *c* cautiously at first to avoid cracking the glass, but strongly at the end, until no more gas is evolved; then remove the burner and leave the whole apparatus for at least ten minutes to cool; shake the flask gently so that the gas in it may take the temperature of the water left in the flask, then raise this or the vessel *d*, as may be required, until the level of the water is the same in both; when this is the case, close the spring clip, remove *d*, and measure the water collected in it by means of a graduated cylinder.

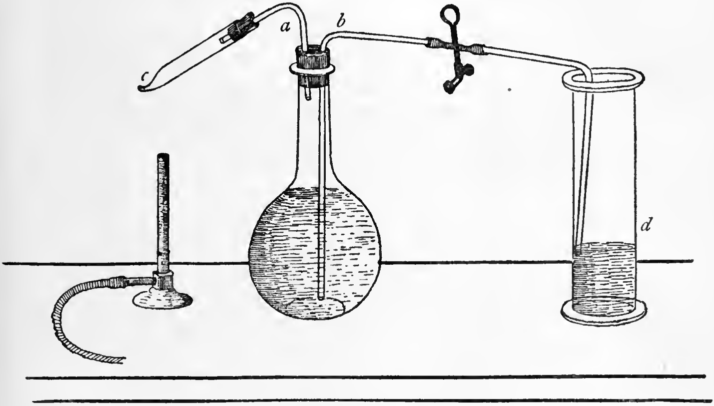

FIG. 30.—Apparatus for Exercise 30.

This volume of water is plainly equal to the volume occupied in the flask by the oxygen which has been evolved; read the temperature of the water in the flask, taking this as the same as the temperature of the gas in it, and also read the height of the barometer. Calculate what the volume of oxygen evolved would be equal to if measured at 0° C. and 760 mm. pressure.

What volume of oxygen, measured under the standard conditions of temperature and pressure, would be evolved from 1 gram of potassium chlorate according to your result? Compare with the results. obtained by other students, and finally with that calculated from the equation (you will have learned something about these by now)—

$$\begin{array}{ccccc} \mathrm{2KClO_3} & = & \mathrm{2KCl} & + & \mathrm{3O_2} \\ 2\left\{39+35.2+48\right\} & & 2\left\{39+35.2\right\} & & 3\times 32 \\ 244.4 & & 148.4 & & 96 \\ \text{grams} & & \text{grams} & & \text{grams} \end{array}$$

making use of the fact that 32 grams of oxygen fill 22¼ litres at the normal temperature and pressure.

Finally weigh the hard glass tube with the residue of potassium chloride; calculate the amount of potassium chloride left when one gram of the chlorate is decomposed by heat, and compare your result with those of other students and with the number calculated from the above equation.

CHAPTER VII

NITROGEN

Nitrogen from Air.—We have already seen that when air is deprived of its oxygen, a gas is left which puts out a taper; we have done nothing to find out whether this gas is an elementary gas, a mixture of gases, or a compound, and the experiments which would be needed to give an answer to this question are too protracted for us to enter upon them; you must be content with the statement that up to a very recent time all experiments which had been made pointed to the conclusion that this residual gas obtained when pure dry air is deprived of oxygen was an element, to which the name nitrogen is given; and though some very recent experiments have shown the presence of small quantities of another gas, possibly more than one, mixed with the nitrogen, yet our ideas as to the composition of the great bulk of this residual gas remain the same.

The element nitrogen, mixed with a small proportion of the newly discovered gas argon, can then be obtained from the air by depriving it of oxygen; in experiments that profess exactitude, it would of course be necessary also to remove moisture and the small quantity of carbon dioxide always present in atmospheric air; the removal of oxygen is most conveniently done by means of some substance which readily combines with oxygen to form a compound such as can easily be separated from the remaining nitrogen, that is, one which is either solid or else soluble in water. Phosphorus is such a substance; it burns readily in air, uniting with the oxygen to form the solid phosphorus pentoxide, P_2O_5, which is easily soluble in water.

EXERCISE 31.—Repeat Exercise 22; test the nitrogen first with clear lime-water, then with a lighted match.

Other substances besides phosphorus might be used, such as metallic copper; this must, however, be kept red-hot in a furnace, or be moistened with solution of ammonia, in order that it may be able to withdraw the oxygen, and the experiment is best done by your teacher in class.

Nitrogen from Ammonia.—We can, however, obtain nitrogen in quite a different way; instead of starting with the air and removing the other gases (or most of them) that are there mixed with the nitrogen, we take a compound of nitrogen and liberate the element from this by chemical action. One of these methods starts from ammonia, a compound of nitrogen with hydrogen having the formula NH_3, and removes the hydrogen by oxidation with bleaching powder.

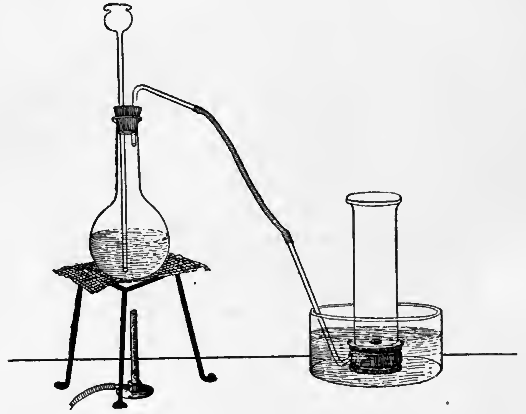

FIG. 31.—Preparation of Nitrogen from Ammonia and Bleaching Powder.

EXERCISE 32.—In a large flask fitted with thistle funnel and delivery tube, arranged as in Fig. 31, put 100 cc. of solution of ammonia from the bench bottle. Rub about 40 grams of bleaching powder into a thin paste with water, and add this gradually through the thistle funnel while the flask is gently heated, and collect the gas evolved over water; test it with lime-water and with a lighted match.

Nitrogen from Ammonium Nitrite.—In another method we make use of the fact that when a solution of ammonium nitrite $NH_4 NO_2$ is heated, this compound decomposes into nitrogen and water.

EXERCISE 33.—It is cheaper and more convenient to use a mixture of ammonium chloride and sodium nitrite instead of ammonium nitrite itself. Fit a flask (which need not be very large, one of about 250 cc. capacity is best) with cork and delivery tube arranged to collect the gas over water; put about 5 grams each of ammonium chloride and sodium nitrite in the flask with 50 cc. of water; heat gently over wire gauze, and remove the flame when the evolution of gas becomes rapid.

Collect two jars of the gas and test them respectively with lime-water and with a lighted taper.

CHAPTER VIII

WATER

SOLUTION, DISTILLATION, CRYSTALLISATION

Water as a Solvent.—Before making experiments with a view to gaining what knowledge we can of the composition of water, it will be well to examine those of its properties which are of the greatest importance in the study of chemistry; and the first step in this direction will be to find out something as to the differences between the various kinds of natural waters —such as rain water, spring water, sea water, etc.

EXERCISE 34.—Weigh two porcelain dishes: in one of them put 50 cc. of rain water, in the other 50 cc. of spring or river water (does tap-water come under this head?); evaporate the contents of each dish to dryness, and if there is any solid residue left in them, weigh the dishes again, and so find the amount of solid residue from 50 cc. of rain water and spring water respectively; suggest a reason for the difference in the two cases.

Evidently water has the power of holding solid substances in solution, and the following experiments will tell you more about this property:—

EXERCISE 35.—Put 3 beakers side by side and pour 50 cc. of water into each of them; in each beaker should be a glass rod for stirring, and in order to lessen the risk of making a hole in the beaker when stirring it is a good plan to slip a short piece (1 inch) of rubber tubing over the lower end of the stirring-rod; weigh out 10 grams each of salt, copper sulphate, and chalk, all of them in powder, and put them in the beakers. Stir for a few minutes: you will soon find that the whole of the salt dissolves, but not the copper sulphate or the chalk; it is, however, evident that some of the copper sulphate has dissolved, from the blue colour of the water. Plainly salt is more soluble than copper sulphate.

Put the beaker containing the copper sulphate on a sand-bath and heat it, stirring the while; soon the whole dissolves, showing that copper sulphate is more soluble in hot water than in cold, and the same is true of nearly all solids.

Do the same with the third beaker: the whole of the chalk does not dissolve, but you cannot tell whether some of it may not have done so; the best way of testing this is to separate the excess of chalk from the liquid and then find out whether the liquid leaves any solid residue on evaporation. How can you separate the liquid and solid? Partly by letting the chalk settle and pouring off the clear liquid above, but more completely by *filtration:* in this process the liquid makes its way through the fine pores of the paper used, while the solid substance is kept back.

Take a piece of filter paper, and fold it as shown in Fig. 15: place it in a glass funnel, wet it under the tap, draining off the excess of water, and support the funnel in the neck of a clean flask; pour the milky liquid from the third beaker into the funnel, using a glass rod as a guide (Fig. 16), and taking care never to fill the funnel to above three-quarters of the height of the filter paper. The filtrate which runs through into the flask, and should be quite clear, is to be poured into a clean evaporating dish, and the water boiled away; notice what residue is left. Is chalk soluble in water?

Separation of Substances by Crystallisation from Water.—In this way it is found that while some solid substances are apparently insoluble in water, the solubility of others varies considerably; and in general the amount of this solubility is increased by raising the temperature. It is easy to see how these facts can be utilised for the separation of an insoluble substance like chalk or sand from those which are readily soluble like salt or sugar; can you see how it would be possible to separate two substances which are both soluble, but to different extents? The following experiment will show you:—

EXERCISE 36.—Take 10 grams each of salt and of copper sulphate, both in powder, put them in a beaker, add 50 cc. of water and stir well; after a time the salt is dissolved, but some of the copper sulphate is left and can be separated by filtration. The same result is more thoroughly obtained by heating the mixture until both substances are dissolved and then putting the solution away to cool: some of the sulphate of copper separates out in crystals with sharp edges and smooth faces.

It is, however, difficult in this way to effect anything like a complete separation; a portion of the less soluble substance is easily obtained in purity, but it is generally impossible entirely to free the more soluble body from its companion.

Try to separate other mixtures in this way—such as (*a*) potassium chloride and sulphate, (*b*) salt and sugar, etc.

Water of Crystallisation.—You will have noticed in the previous experiment the formation of crystals—geometrical solids with characteristic shapes: about these latter we cannot concern ourselves here, but one thing should be noticed by you which the next experiment will serve to illustrate.

EXERCISE 37.—(*a*) Weigh a porcelain dish, put some crystals (about 10 grams) of washing-soda (sodium carbonate) in it, and weigh again. Heat the dish on a sand-bath; the crystals very soon melt, then the liquid boils and gives off vapour; hold a clean dry funnel in the vapour, examine the drops of liquid to which the vapour is condensed: what do you think the liquid is? Continue to heat until no more vapour is evolved, then let the dish cool and weigh again. What proportion of their weight have the crystals lost, and to what is this loss due? Compare your results with those of other students. Dissolve the residue in as little hot water as will suffice, and put away to cool: you should obtain crystals like those from which you started.

(*b*) Repeat this experiment with the same quantity of crystals of copper sulphate, and afterwards of common salt.

You find that crystals often, though not always, contain water which can only be driven out by considerable heat; for the same substance the proportion of this "water of crystallisation" is constant, but it is different for different substances.

Solubility of Gases in Water.—Water can dissolve not only solids but also liquids and gases; we shall investigate the latter case only in this book, and at present only the air dissolved in spring water.

EXERCISE 38.—(*a*) Put some water in a beaker or flask and heat it; long before the water is near the boiling point you notice bubbles of gas appearing on the sides of the beaker; these must come from the water, and evidently heat does not increase the solubility of gases in water as it does that of solids—the opposite is in fact the case.

(*b*) The following method may be used in order to measure roughly how much gas is dissolved in ordinary tap-water: fit a gallon tin can with indiarubber cork and delivery tube (Fig. 32),

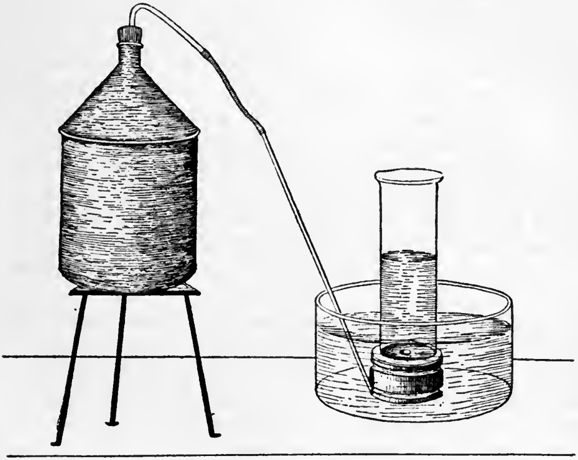

FIG. 32.—Apparatus to collect Dissolved Air expelled from Water by Heat.

the tube being just flush with the narrow end of the cork; fill the can quite full of water and then press in the cork with delivery tube, so that the excess of water is forced into the tube and drives out the air from it.

Heat the can with a powerful burner, and support a small gas cylinder over the mouth of the delivery tube, which must be arranged to dip below water in a basin; after a time bubbles of gas are given off, and when the water boils these are carried by the steam into the cylinder; when the water has boiled for a few minutes take the flame away. How much gas do you get? Put a glass plate over the mouth of the cylinder, remove this from the basin, invert, and test the gas collected. Is it air?

Distillation.—You have seen in several of the preceding experiments that when a solution of a solid in water is evaporated, the solid is left behind: we have also found that the steam given off carries none of the solid with it, for in Exercise 18 (*c*) the amount of salt left was equal in weight to that used. It is plain that by condensing the steam given off we shall get water free from the solid impurities dissolved in it, provided they behave like salt and do not evaporate along with the steam.

EXERCISE 39.—(*a*) Fit a 600 cc. flask with a good cork and a bent piece of fairly wide glass tubing; it is best to have the end of the tube which is inside the flask ground aslant; the tubing should be chosen of such a size as to go readily, but with not much to spare, into the inner tube of a Liebig's condenser, and the joint can then easily be made sufficiently air-tight by means of a strip of clean glazed paper wrapped round the smaller tube; fill the flask about half full of water, and add a few crystals of potassium permanganate, which will colour the water intensely purple.

Heat the flask on wire gauze or a sand-bath; connect the lower side-tube of the condenser with the tap, and the upper side-tube to the sink: turn on the tap gently until the outer jacket of the condenser is full and water issues steadily from the waste tube. Collect the condensed water in a flask or beaker. Test this water; does it contain any of the potassium permanganate?

(*b*) Repeat the experiment, using water to which some strong ammonia solution has been added in place of water coloured with potassium permanganate. Collect the distillate in several portions and test each of them by smell for the presence of ammonia: if a more sensitive test than the sense of smell is wanted, you may use paper coloured red with litmus, which you will find on trial is turned blue by ammonia.

Fractional Distillation.—This last experiment will have shown that it is sometimes difficult to purify water by distillation from a volatile impurity; the separation can, however, generally be effected more or less completely in such a case by the process known as *fractional distillation.*

EXERCISE 40.—Mix equal volumes of water and methylated spirit, and put the mixture in a 200 cc. distillation flask of the kind shown in Fig. 33. In the neck of the flask fit a cork, through which passes a thermometer whose bulb should be just below the opening of the side tube; a cork will be needed to connect this side tube with the condenser.

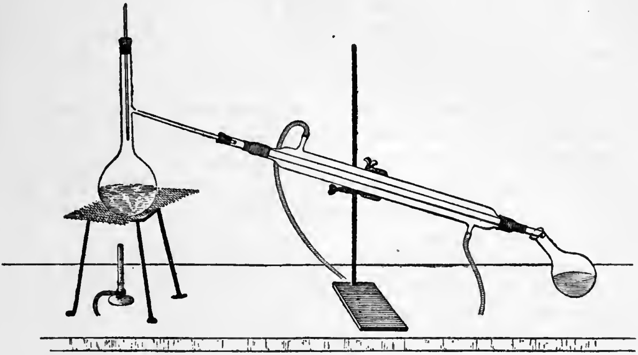

FIG. 33.—Fractional Distillation.

Heat the flask on wire gauze, and collect the distillate in two approximately equal portions; test these by trying whether a little of each will burn, also by taking their specific gravities with a hydrometer. Which contains more alcohol?

Empty the distillation flask, then pour back into it the first half A of the distillate: distil until half of A has been collected, and test this by taking its specific gravity. Compare the numbers obtained with the specific gravity of the methylated spirit itself. What do you find?

Notice also the temperatures indicated by the thermometer throughout the distillation. What do you conclude as to the boiling point of methylated spirit? Then try the next experiment.

EXERCISE 41.—Empty the distillation flask, and pour in some undiluted methylated spirit; heat this to boiling and notice carefully the temperature indicated by the thermometer during the distillation. Has methylated spirit a constant boiling point? If not, you may conclude that it is not a single substance, but a mixture of two or more.

Action of Metals on Water.—We discovered important facts about the nature of air by examining the chemical changes which metals undergo in its presence; let us see whether anything can be learned about water in a similar way. You know that some metals are unaltered by long exposure to water, while others like iron become gradually corroded; it would be possible for you to learn much by examining the reaction between iron and steam, but the experiment is more suitable to be shown to you in the class-room; and for our purpose it is better to take a much less familiar metal, *i.e.* sodium.

EXERCISE 42.—(*a*) Put some water in an evaporating dish and throw a small piece of sodium on it; a violent reaction evidently takes place.

(*b*) This can be moderated and made easier to investigate by alloying the sodium with mercury—a metal which has no action upon water; this alloy is called sodium amalgam. Prepare some by putting about 10 or 15 grams of mercury in a dry mortar, and adding in small pieces about 1 gram of sodium, pressing each piece into contact with the mercury by means of the pestle until the two metals unite with a slight flash of light.

Put the sodium amalgam thus prepared in a small flask of about 75 cc. capacity fitted with a cork and delivery tube arranged to collect the gas evolved over water; fill the flask up to the neck with water and replace the cork and tube. A

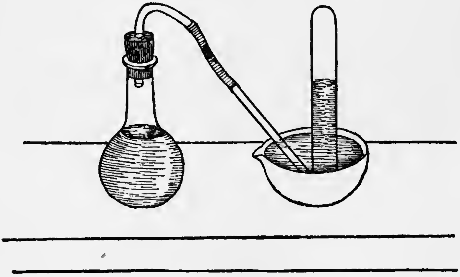

FIG. 34.—Hydrogen from Sodium Amalgam and Water.

moderate reaction now occurs, and bubbles of gas are given off, which are collected in test-tubes (Fig. 34); when each test-tube is full, remove it and apply a lighted match to the gas collected in it; this is the gas formerly known as *inflammable air*, but now called *hydrogen*. Where do you suppose it comes from?

Notice what is left in the flask at the end of the reaction; pour off the watery liquid into a basin, and evaporate on the sand-bath until no more water can be expelled; then take off the dish and let it cool. What is left? This solid residue contains the sodium used in combination with a part of the water from which the hydrogen you collected has been expelled.

You will be able to learn more about the composition of water in the next chapter, where we shall prepare this gas hydrogen in larger quantity and examine it more carefully.

CHAPTER IX

HYDROGEN

Action between Metals and Acids.—You have already learned something about the nature of acids; the three strong acids so much used in chemical manufactures—sulphuric, hydrochloric, and nitric acids—are able to act upon and dissolve many of the common metals, and at the same time certain gases are evolved whose study is of great importance.

EXERCISE 43.—Take a small flask holding about 75 cc. fitted with a cork and delivery tube so arranged that any gas evolved can be collected over water. Try the action of the three acids mentioned (in each case use the acid diluted with twice its bulk of water) upon the following metals, taking about 2 grams of the metal in each experiment:—copper, lead, iron, tin, zinc. If any gas is evolved, collect it in test-tubes over water, and test it with a lighted match; draw up a table showing what results you get with the several metals and acids.

You will find that in every case where there is any action with sulphuric or hydrochloric acid, a gas is evolved which is readily inflammable and burns with a pale flame scarcely visible in bright daylight; this gas is the same gas, hydrogen, as was obtained in the last exercise by the action of the metal sodium on water; it is placed amongst the elements because no experiments hitherto made have succeeded in decomposing it; in the case of nitric acid we do not obtain hydrogen, but other gases which will be more fully examined later on.

Preparation and Properties of Hydrogen.—We now proceed to prepare hydrogen in larger quantity and examine its properties in detail.

EXERCISE 44.—Fit up a flask with thistle funnel and delivery tube arranged to collect the gas over water: in the flask put about 15 grams of granulated zinc, replace the cork, and then add through the funnel about 20 cc. of dilute sulphuric acid (1 of acid to 3 of water); collect the first portions of gas evolved in test-tubes and try each tube at the flame of the Bunsen burner, carrying the tube to it with the mouth downwards; when the gas burns quietly inside the test-tube, it is safe to collect in larger quantity, and the cylinders should be taken in place of the test-tubes. When the evolution of gas slackens add some more dilute acid through the funnel. Do the following experiments with the gas:—

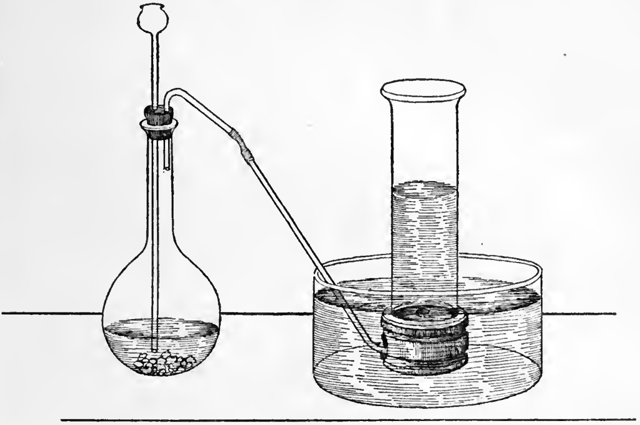

FIG. 35.—Apparatus for preparing Hydrogen from Zinc and Sulphuric Acid.

(i.) Remove the glass plate from a jar of the gas, holding over it a lighted taper, or match held in crucible tongs.

(ii.) Remove the glass plate from the mouth of the second jar, and after a few seconds test it with a lighted match.

(iii.) Try to pour the gas from one cylinder into another full of air. Which way must you pour the gas, up or down?

(iv.) Holding a jar of the gas mouth downwards, remove the glass plate, and at once push upwards into the jar a lighted match held by crucible tongs.

(v.) Remove the delivery tube and replace it by a vertical delivery tube similar to that shown in Fig. 36; the wider portion of the delivery tube is packed with small lumps of calcium chloride (placed between plugs of cotton wool), the object of which is to dry the gas thoroughly for the next two experiments, calcium chloride being a substance with great affinity for water.

Take a clean dry flask of the largest size you can weigh on the balance, and fit it with a good cork; place the flask over the vertical delivery tube from which hydrogen is being rapidly evolved, and hold it there for several minutes; then lift off the flask from the tube, cork it firmly, taking care until the cork is in place to keep the mouth of the flask downwards and balance the flask and cork together; take out the cork, blow dry air through the flask from a bellows, and then replace it: is there any gain of weight? If so, determine its amount and say to what it is due. Measure the volume of the flask, and then see if your result agrees roughly with the more exact measurements which show that a litre of hydrogen weighs .085 gram at the ordinary temperature, whilst a litre of air weighs about 14½ times as much.

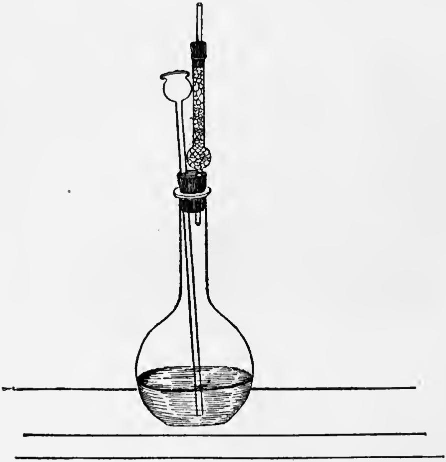

Fig. 36.—Apparatus for Exercise 44 (v.).

(vi.) Place a dry test-tube over the vertical delivery tube, leave it for a minute, then remove it with the mouth downwards and bring to a flame; if the gas burns quietly in the tube as it should do, you may safely put a light to the gas where it issues from the delivery tube, but be very careful not to do so before having tested the gas in the way just described, as otherwise a serious accident may be the result.

Hold a clean dry beaker over the flame of burning hydrogen: what happens, and what conclusion do you draw?

(vii.) Remove the cork and tubes, and filter the liquid left in the flask into a beaker; put the clear filtrate away until your next lesson, and then examine it again; you should find colourless crystals of zinc sulphate, $ZnSO_4.7H_2O$.

Quantitative Examination of the Reaction between Zinc and Dilute Sulphuric Acid.—The reaction by which we have prepared hydrogen must next be examined with a view to discovering how much of the gas is obtained from a given quantity of zinc; this is readily done with a fair degree of accuracy by the following simple method:—

EXERCISE 45.—Fit up an apparatus similar to that shown in Fig. 37; the flask A must hold not less than 500 cc., and must be full of water at the commencement of the experiment; the flask B should be small (one of 60 cc. capacity is quite large enough), and is two-thirds filled with dilute sulphuric acid (1 to 3), to which a few drops of solution of copper sulphate are added (this makes the reaction between the zinc and acid more rapid through deposition of metallic copper on the zinc and consequent galvanic action); both corks should be of india-rubber, and all the connections must be quite air-tight.

Open the clip D and blow into the tube C until water flows from the end of the tube E into the cylinder or other vessel placed there, then close the clip at once. Weigh accurately a piece or pieces of pure granulated zinc of between .8 and 1.0 gram; drop it into the flask B. At once place this firmly on the rubber stopper at C, and open the clip D.

When all the zinc is dissolved, raise either the flask A or the cylinder at E as may be required, until the level of the water is the same in both (why is this done?); then close the clip D, take away the cylinder E and measure the amount of water which has collected in it; take the temperature of the water in A, and read the height of the barometer; these give approximately the temperature and pressure under which the volume of hydrogen evolved is equal to that of the water collected in E.

Calculate what this volume becomes at 0° C. and 760 mm. pressure, and then find the weight of this volume of hydrogen, having given that 1 litre of hydrogen has been found by very accurate experiments to weigh .0899 gram (you may take .09 as sufficiently accurate for your purpose) at the standard temperature and pressure.

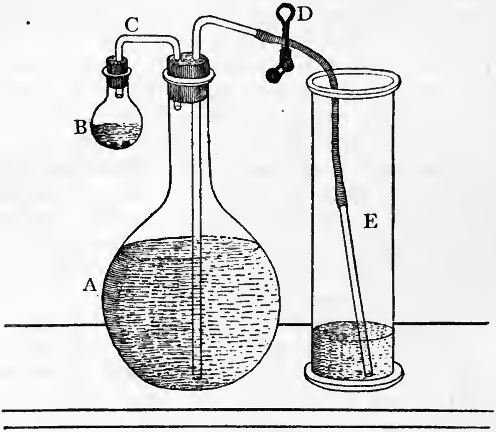

FIG. 37.—Apparatus for Exercise 46.

Divide the weight of zinc used by the weight of hydrogen evolved in order to find how much zinc is needed to expel 1 gram of hydrogen.

Then repeat the above experiment, using dilute hydrochloric acid (1 of strong acid to 3 of water) in place of sulphuric acid; work out the result in the same way.

Compare your two results together, and also with those of other students who have used somewhat different weights of zinc.

Equivalents.—The number which you have found in these experiments expresses how many parts of zinc are needed to displace one part of hydrogen, and is called the *equivalent* of zinc; the knowledge of the equivalents of the different elements is of the very greatest importance.

EXERCISE 46.—Determine in the same way the equivalents of some other metals, such as iron (.9 gram), magnesium (.4 gram), tin (2 grams), or aluminium (.3 gram); the numbers in brackets give the largest quantities to be used; in the case of tin, hydrochloric acid diluted with its own bulk of water must be used, a few drops of a weak solution of platinum chloride may be added to facilitate the reaction, and heat must be applied to aid the chemical change.

Determination of Equivalents of Metals in their Oxides.—We can also determine the equivalent of a metal in other less direct methods which are often more exact; and in the case of those metals which do not easily evolve hydrogen in contact with an acid, some other method must of necessity be used. One simple method is to convert a weighed quantity of the metal into oxide, and from the increase in weight to calculate the equivalent of the metal *compared to oxygen*, and as the equivalent of oxygen has been found by careful experiments on the composition of water to be 8, taking hydrogen as the standard, it is easy to calculate the equivalent of the metal compared to hydrogen.

EXERCISE 47.—(*a*) Weigh a porcelain crucible and lid: take a piece of magnesium ribbon weighing about a gram, clean it with sand-paper and roll it into a ball; place this in the crucible and weigh again; heat the crucible with the lid on for five minutes, then take off the lid and heat with the full flame for ten minutes longer; allow to cool and weigh; heat again for five minutes and weigh, repeating this process until the weight becomes constant. Calculate how much magnesium combines with one gram of oxygen, and from this how many grams of magnesium are equivalent to one of hydrogen, taking the equivalent of oxygen as 8; compare with the result obtained in Exercise 46.

(*b*) Clean a porcelain dish, dry and weigh it; put in it about 2 grams of pure tin in foil or small pieces, and weigh again; put the dish on a sand-bath in a good draught cupboard, cover the tin with an inverted funnel (Fig. 38) and pour into the dish half a test-tube full of strong nitric acid; heat if necessary to start the reaction, then turn out the flame: when the action is over, remove the funnel and wash any white powder adhering to it into the dish; heat this on the sand-bath until the residue appears quite dry, then take the dish from the sand-bath and support it by a pipe-clay triangle over the direct flame of the burner; after five minutes turn out the flame, and when the dish is cold weigh it with its contents. The residue is tin oxide.

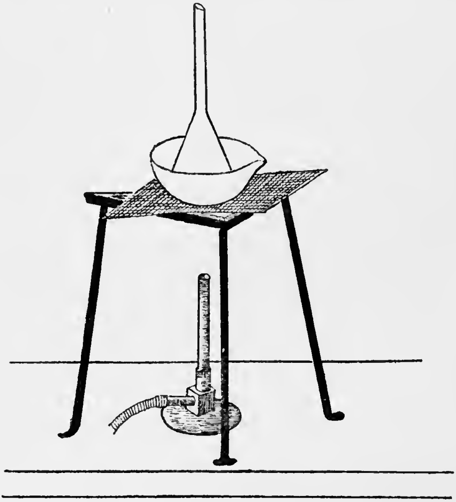

Fig. 38.—Oxidation of Tin with Nitric Acid.

Calculate how much tin combines with 1 gram of oxygen, and hence the equivalent of tin; compare with the number found in Exercise 46.

Determination of Equivalents by Displacement of one Metal with another.—Another method of finding the equivalent of a metal is by comparing it with another metal whose equivalent is known, using a reaction in which one of the metals displaces the other from combination; thus if a piece of iron be placed in a solution of copper sulphate the iron gradually dissolves and an equivalent amount of copper is precipitated, so that by weighing both the iron and the copper we can calculate the equivalent of either metal when that of the other is known.

Exercise 48.—(*a*) In a beaker put about 100 cc. of a strong solution of copper sulphate with some clean French nails or other pieces of scrap iron (it is a good plan to wash the iron with dilute acid and then with water before placing it in the copper sulphate solution); cover the beaker with a piece of paper and put away until the liquid loses its blue colour entirely. Then pour off the liquid from the solid matter left in the beaker, evaporate the liquid in a dish to obtain crystals (what are they?), and also examine the residue left in the beaker.

(*b*) In another beaker put 100 cc. of copper sulphate solution: take a piece of iron wire (piano wire is purest), clean it with sand-paper and cut into short lengths; weigh accurately a quantity of this iron wire between 1.5 and 2 grams, and place it in the solution of copper sulphate. Cover the beaker with a piece of paper and put away for several hours at least.

Weigh a circular filter paper and then fit it in a funnel; filter the contents of the beaker through this paper, washing every particle of the precipitated copper out of the beaker and into the filter paper. Wash the precipitate by pouring water over it, and finally wash again with alcohol, if a supply of that liquid or of methylated spirit can be had. Take out the filter paper from the funnel very carefully, open it out and spread it on the top of several thicknesses of dry filter paper: support this on wire gauze at least six inches above a small flame.

When quite dry weigh the filter paper (the top sheet only, of course) with the copper; from your numbers calculate what weight of copper is equivalent to 1 gram of iron. From this, since you have found in Exercise 46 the equivalent of iron compared to hydrogen, you can readily calculate the equivalent of copper.

(*c*) In the same way you can find the weight of copper equivalent to 1 gram of magnesium or to 1 gram of zinc; also by using a solution of silver nitrate you can find the weight of silver equivalent to 1 gram of magnesium, of zinc, or of iron; or again, using a solution of lead nitrate you can find the weight of lead equivalent to 1 gram of either of the same three metals.

CHAPTER X

NITRIC ACID

Preparation and Properties.—Nitric acid is manufactured in very large quantity from a substance found in great beds in the countries on the west coast of South America, which is known as Chili saltpetre, and is sodium nitrate $NaNO_3$; we will now make some of the acid in a similar way, but using ordinary saltpetre—potassium nitrate KNO_3—and then examine its properties.

EXERCISE 49.—Put 25 grams of powdered potassium nitrate in a 4 oz. retort; calculate from the equation

$$KNO_3 + H_2SO_4 = KHSO_4 + HNO_3$$

how many grams of sulphuric acid must be taken, and then find how many cc. this quantity of acid will measure, taking the specific gravity of concentrated H_2SO_4 as 1.8.

Add the sulphuric acid to the potassium nitrate, pouring it through the tubulus if a tubulated retort is used, or down the neck through a thistle-headed funnel if the retort is plain and without a tubulus; heat the retort over a *small* flame or on the sand-bath and collect the acid which distils over in a small dry flask kept cold with water.

The arrangement shown in Fig. 39 dispenses with the use of a retort stand and clamp, and is quite safe if ordinary care be used.

Then do the following experiments with the acid:—

(i.) Add two drops of the acid to half a test-tube full of water: test the diluted acid by tasting and with litmus paper.

(ii.) Put some small pieces of marble or limestone in a test-tube, add a little water and then a few drops of the nitric acid;

what happens? Try the same experiment with any other acid you can find in the bottles above your bench.

(iii.) Put 50 cc. of solution of potassium hydrate, KOH (1 in 5), in a beaker and add dilute HNO_3 (1 in 4) from your bench bottle until the liquid, after being well stirred, is just acid to litmus paper; evaporate the liquid in a porcelain basin to about 25 cc., then pour into a small beaker, and put away to cool.

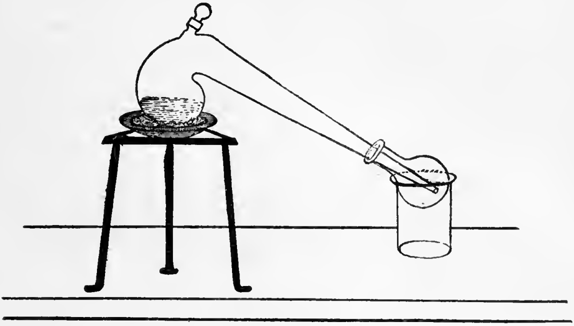

FIG. 39.—Preparation of Nitric Acid.

If there is too much liquid to go into the basin, fill this two-thirds, boil over the flame and fill up from the beaker as the liquid evaporates.

(iv.) Put a little sawdust in a porcelain dish and heat on a sand-bath until the sawdust begins to char; then remove the dish to a fume cupboard and pour a few drops of the strong nitric acid you have made over the dry sawdust.

(v.) Put two or three copper turnings in a flask, add a few drops of water and then some nitric acid. Do the same experiment with a few grains of tin in place of the copper.

Action of Nitric Acid on Metals—Nitric Oxide.—The action of nitric acid on metals appears totally different from that of hydrochloric and sulphuric acid; while the latter gives hydrogen the former gives several gases, prominent amongst which is the "nitric air" of Priestley, now known as Nitric Oxide: this is a compound of nitrogen and oxygen with the formula NO, and we will now prepare and examine the gas.

EXERCISE 50.—Put 25 grams of copper turnings in a flask fitted with thistle funnel and delivery tube arranged to collect the gas over water as in Fig. 35: mix 50 cc. of strong HNO_3 with the same volume of water in a beaker and add a portion of this through the funnel; wait a few minutes for the reaction to begin and then proceed to collect the gas, throwing away the first cylinder of gas collected (why?); when the reaction slackens add some more of the nitric acid, but do not add too much at once. Make the following experiments with the gas:—

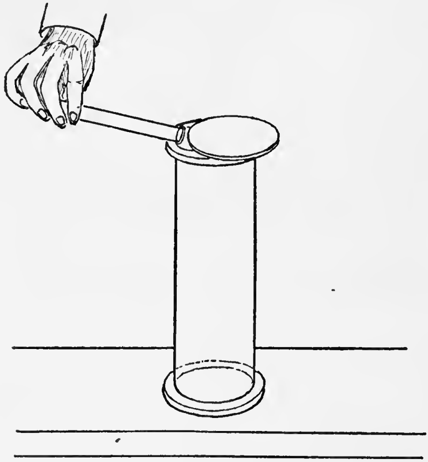

FIG. 40.—Pouring Carbon Disulphide into the Jar of Nitric Oxide—Exercise 50 (v.).

(i.) Remove the glass plate from a jar of the gas; notice the brown fumes produced: what are they?

Pour some water into the jar and shake with the plate on the mouth; the red fumes dissolve: test the solution with litmus.

(ii.) Take another jar of the gas, and put a lighted taper into it.

(iii.) Into another jar of the gas put a deflagrating spoon with some burning sulphur.

(iv.) Try the same experiment with phosphorus instead of sulphur; first with the phosphorus just burning and then with the phosphorus burning brightly.

(v.) Partly slide off the cover from another jar and, through the narrow opening left, pour five or six drops of carbon disulphide CS_2 (this should be first poured from the bottle into a dry test-tube); immediately replace the cover, shake well, remove the cover and apply a light.

If too much carbon disulphide be taken this experiment will not be successful.

(vi.) Remove the cork and tubes from the evolution flask; filter the blue liquid into a beaker; pour some of it into a porcelain dish, evaporate to half the bulk, and then put aside for a day or two.

Nitric Oxide from Nitric Acid and Ferrous Sulphate; its Use to analyse Air.—This same gas nitric oxide can be got from nitric acid by the action of several other substances which are not metals; in fact anything which can take up oxygen will generally react with nitric acid to form nitric oxide under suitable conditions; such a substance is one of the two sulphates of iron, ferrous sulphate, or green vitriol $FeSO_4$.

EXERCISE 51.—Fit a fairly small flask (100 or 150 cc.) with cork and delivery tube to collect the gas evolved over water; put 20 grams of ferrous sulphate in the flask, add 15 cc. of water, and then the same bulk of concentrated nitric acid; replace the cork and delivery tube, and heat the flask gently on a sand-bath.

(i.) Collect a jar of the gas and notice that it gives red fumes in the air; it is nitric oxide.

(ii.) Take a glass tube closed at one end of the same size as that used in Exercise 24 (*a*); fill it about one-third with water and put a small dish over the mouth of the tube; invert the tube and transfer it with the contained air to a basin of water. Mark the level of the water in the tube with an indiarubber band.

Now allow the nitric oxide which is being evolved in great purity from the $FeSO_4$ and nitric acid to bubble up into the tube, removing the tube to one side after each bubble until the red fumes which are formed have dissolved in the water.

As soon as more red fumes cease to be formed on addition of a bubble of nitric oxide, put the tube aside for a few minutes; then mark the level of the water inside the tube with another rubber band. You should find that a portion of the air has disappeared: how is this?

This method was the first devised for the analysis of air; in Priestley's hands it gave only rough results, but shortly afterwards Cavendish improved it in several points, and working with all his usual exactness, obtained numbers which are surprisingly accurate when we consider the imperfections of the method; other methods of greater ease and accuracy are now known.

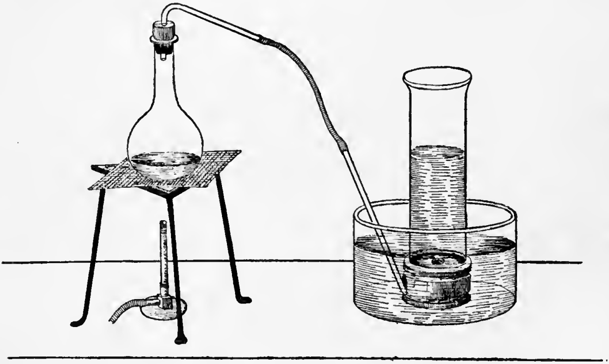

FIG. 41.—Preparation of Nitric Oxide from Ferrous Sulphate and Nitric Acid.

CHAPTER XI

NITRATES AND THEIR BEHAVIOUR WHEN HEATED

Action of Heat on Lead Nitrate.—You have already seen how potassium nitrate and copper nitrate can be prepared; you will require some lead nitrate for the next experiment, and can make it as follows:—

EXERCISE 52.—Put 10 cc. of concentrated nitric acid in a beaker, with the same bulk of water, and add little by little 15 grams of litharge PbO; stir well, and then cool the beaker by placing it in a dish of cold water. When cold, filter off the lead nitrate which will have separated, let the crystals drain, then carefully take out the filter paper from the funnel, open it out and support on wire gauze six inches at least above a small flame, Fig. 27.

(While the lead nitrate is being dried you may go on to the next exercise.)

When dry, put the lead nitrate in a short tube of hard glass fitted with a cork and delivery tube, and supported with a clamp, or in the way shown in Fig. 24, so that the gas evolved can be collected in test-tubes over water. Heat the tube at first cautiously, then more strongly.

Notice the brown fumes evolved (what happens to them?), also the colourless gas which is collected over water; test this gas with a glowing splint. Pour part of the water from the dish into a test-tube and try whether it contains an acid; pour another portion into a second test-tube and add a few drops of a solution of potassium iodide. The brown coloration shows that iodine has been liberated from the potassium iodide, in this case by the action of nitrous acid HNO_2, which is produced by the reaction of the brown fumes of NO_2 with the water in the dish. Try to work out the equations: when in difficulties consult your teacher, or refer to Roscoe and Lunt, Lesson XVI.

Action of Heat on Potassium Nitrate.—For this exercise you will require some potassium nitrate, and may use that prepared in Exercise 49 (iii.), but must first dry it carefully.

EXERCISE 53.—Take a piece of glass tubing about 5 mm. internal diameter and 7 cm. long, heat it at the middle in a Bunsen flame until quite soft and make it into two tubes of half the length, each sealed at one end (these are called ignition tubes); fill one of these tubes about one-fifth with roughly powdered potassium nitrate, and holding it with crucible tongs heat as strongly as possible in the Bunsen; the nitrate first melts, and then at a higher temperature gives off a gas with effervescence; test the gas by holding a glowing splint at the mouth of the tube.

Keep the tube in the flame for a few minutes after the oxygen begins to come off; then allow to cool and break the tube to extract the solid residue.

Put a portion of this in another test-tube and add a few drops of dilute H_2SO_4, the brown fumes evolved indicate the presence of a nitrite, in this case potassium nitrite KNO_2.

Take the remainder of the residue and dissolve it in a little water in a test-tube; add a few drops of solution of potassium iodide and then of dilute H_2SO_4; a brown coloration is produced: what does this indicate?

EXERCISE 54.—**Preparation of Ammonium Nitrate.**—Put 10 cc. of strong HNO_3 in a beaker with the same bulk of water and add solution of ammonia in small quantities at a time until a drop of the liquid taken out on the end of a glass rod is distinctly alkaline to litmus paper. Evaporate the liquid in a porcelain dish until a sample taken out with a glass rod solidifies as it cools; then take away the flame.

During the time required for the evaporation go on with the next exercise.

EXERCISE 55.—**Action of Heat on various Nitrates.**—Prepare four ignition tubes, and in separate tubes try what happens when the following nitrates are heated:—mercuric nitrate, copper nitrate, calcium nitrate, sodium nitrate. Is their behaviour like that of lead nitrate or that of the potassium salt?

EXERCISE 56.— **Decomposition of Ammonium Nitrate—Nitrous Oxide N_2O.**—Put the ammonium nitrate prepared in Exercise 54 in a small flask of about 60 cc. capacity, which is supported at a convenient height above a burner in the way shown in Fig. 42; B is a dry flask of any convenient size fitted with a doubly bored cork (the large 600 cc. flask with its indiarubber cork does very well) through which pass the inlet tube C, reaching to the middle of the flask, and the outlet tube D; the flask B is kept steady by a ring of the retort stand, pressing it down against a folded piece of paper which is placed as a pad on the base of the retort stand; in this way the use of a clamp is not necessary, but if there is a sufficient supply of these forthcoming it is easy to adopt a different arrangement. The exit tube D is connected with a delivery tube leading to a basin, which is best filled with warm water, as the gas is somewhat soluble in hot.

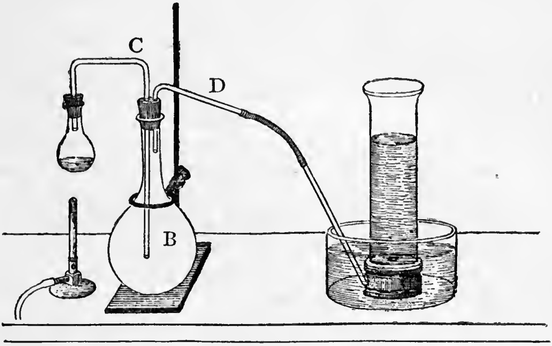

FIG. 42.—Decomposition of Ammonium Nitrate by Heat.

Heat the small flask carefully until the ammonium nitrate melts and then begins to decompose: if the evolution of gas becomes too rapid take care to lower the flame at once, and if necessary remove it altogether. Make the following experiments with the gas:—

(i.) Plunge a glowing splinter into a jar of the gas.

(ii.) Try to burn sulphur in the gas (*a*) with the sulphur only just alight, (*b*) with the sulphur burning brightly.

(iii.) Burn a small piece of phosphorus in the gas.

(iv.) Fill a cylinder half full of the gas: fit up an apparatus for producing nitric oxide (one apparatus will generally be enough for the class), and bubble this gas into the cylinder which is half full of nitrous oxide. Are there any brown fumes produced?

You will find at the end of the experiment that a small quantity of liquid has collected in the flask B: test this liquid to find out whether it is water as it appears to be, one of the best tests being to take the boiling point of the liquid; this can be done roughly if the liquid collected is poured into a test-tube and boiled while the bulb of a thermometer is immersed in the boiling liquid.

The decomposition of ammonium nitrate by heat is found to be different from that of the metallic nitrates, and the difference is due to the fact that what we call ammonium is not an element at all, but a group of elements for which it is convenient to have a special name; this is shown by the formula $NH_4.NO_3$; when this compound is heated, the group or "radicle" ammonium NH_4 is decomposed, and the reaction is therefore entirely different from what can occur when the nitrate of a metallic element such as potassium nitrate KNO_3 is heated.

CHAPTER XII

AMMONIA

Ammonia produced by Heating Organic Matter containing Nitrogen.—Ammonia NH_3 is much the most important compound of nitrogen and hydrogen; it is generally formed whenever animal or vegetable matter containing nitrogen is decomposed either by heat or by natural processes such as putrefaction.

EXERCISE 57.—(*a*) Try the action of heat on small quantities of the following, all of which contain nitrogen:—horn clippings, feathers, hair, cheese. Heat each substance separately in a dry test-tube, avoiding so much heat as will produce charring: test the gas given off by smell, and with red litmus paper (which is turned blue by ammonia).

(*b*) Fit a test-tube with cork and bent tube, the latter connected, as shown in Fig. 43, with a small wide-mouthed bottle by a cork through which passes also the straight tube drawn out to a jet at its upper end; fill the test-tube three-quarters full with small pieces of coal, and heat the test-tube as strongly as possible without melting the test-tube. The coal-gas issuing from the jet may be lit after the air has been driven out from the small bottle. When no more gas comes off, remove the cork from the bottle and test its contents by smell, and with litmus paper, for ammonia.

Ammonia prepared from Ammonium Chloride; its Properties.—Practically all the ammonia now used is obtained as a by-product in the manufacture of coal-gas, in which process it is produced in large quantity and collects in the ammoniacal liquor; from this a solid compound ammonium chloride or sal-ammoniac NH_4Cl is got by neutralising the ammonia with hydrochloric acid HCl; it is from this compound that we shall now prepare ammonia in sufficient quantity for the examination of its properties.

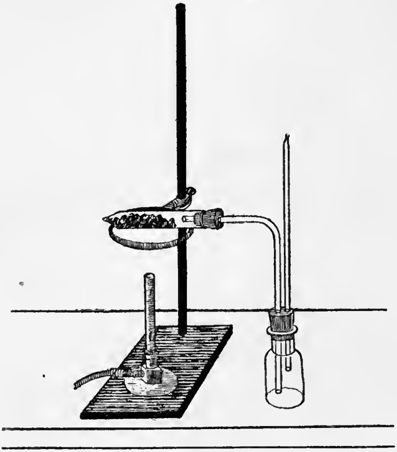

FIG. 43.—Ammonia from Coal.

EXERCISE 58.—Fit a dry flask with cork and delivery tube arranged as shown in Fig. 44; powder separately 20 grams of sal-ammoniac and 40 grams of dry quick-lime, mix thoroughly, and at once place in the flask, which is then nearly filled with coarsely powdered quick-lime placed above the mixture. Heat the flask on a sand-bath, and collect the gas evolved in dry cylinders by upward displacement, which method is used because the gas is very soluble in water and is lighter than air. Make the following experiments:—

(i.) Notice the smell of the gas, but take care not to breathe it unless largely diluted with air. Try also its action on red and blue litmus paper, and on turmeric paper.

(ii.) Push a lighted match into a jar of the gas held with its mouth downwards.

(iii.) Put water coloured red with litmus and a drop of dilute acid in a basin; fill a dry cylinder with ammonia, cover its mouth (kept downwards of course) with a piece of dry paper and plunge in the water.

(iv.) Dip a glass rod into concentrated hydrochloric acid, and hold it in a jar of ammonia.

(v) Take away the bee-hive shelf and lead the gas into water in a beaker or small bottle: what happens to the gas?

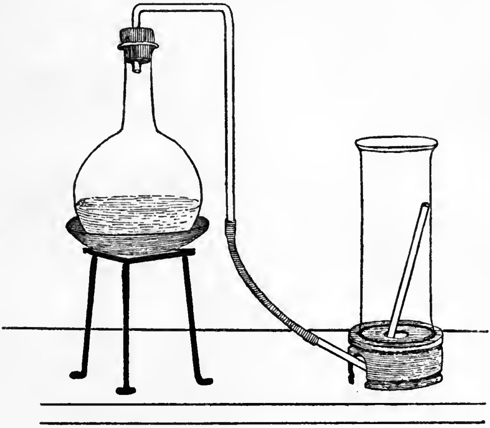

FIG. 44.—Preparation of Ammonia from Ammonium Chloride and Lime.

Take care to remove the delivery tube from the water before the evolution of gas slackens, as otherwise the water will be sucked back into the flask.

The Proportion in which Nitrogen and Hydrogen are combined in Ammonia.—To find out in what proportion by volume the two gases nitrogen and hydrogen combine to form ammonia, you may make the following experiment :—

EXERCISE 59.—Take the tube used in Exercise 25 (*b*), dry it thoroughly and fill it with dry chlorine by downward displacement, leading the gas in through a long narrow tube reaching to the bottom of the wider tube. (If an apparatus for evolving chlorine, which should be washed with water and then dried by bubbling through concentrated sulphuric acid, is set up in a draught cupboard, all the students can fill their tubes from it in turn.) Gradually withdraw the leading tube, and at once close the wider tube with a rubber stopper smeared in vaseline to protect it from the action of the chlorine.

Take a small test-tube of thin glass and fit it with a good soft cork: fill the test-tube half full of ammonia solution and cork it, taking care that none of the liquid is on the outside of the tube or cork; open the tube which is full of chlorine, holding it in a slanting position, and slide in the test-tube; at once cork up the chlorine tube, then let the test-tube fall suddenly to the bottom and break.

A chemical change takes place between the ammonia and the chlorine by which the nitrogen is liberated, while the hydrogen of the ammonia combines with the chlorine to form hydrochloric acid.

When the reaction has been completed by shaking the tube, open it under a basin of water: mark the height to which the water rises by an indiarubber band; then put your thumb over the mouth of the tube, remove it with its contents from the basin, invert and test the gas left with a lighted match. It is nitrogen: what fraction of the original volume of chlorine is this volume of nitrogen which is left? It is known from other experiments that hydrogen and chlorine combine in equal volumes: what then is the proportion by volume in which nitrogen and hydrogen are combined in ammonia?

CHAPTER XIII

ACIDS, BASES, AND SALTS

Acids.—In the previous exercises you have repeatedly had to use certain substances called acids, and have learned to recognise them by their action upon the vegetable colouring matter litmus ; this is almost blue in colour, though with a slight purple tinge, when in its natural state, but is turned to a bright red colour by the action of even a very small quantity of an acid, such as sulphuric, hydrochloric, or nitric. You have also found that these acids possess other properties in common, which we must now examine more fully and accurately than in what has gone before.

EXERCISE 60.—(*a*) Fill several test-tubes about half full of a weak solution of neutral litmus, and add to separate test-tubes a few drops of any acid you can find on your bench. Note what the effect is in each case.

(*b*) Put a small piece of zinc in each of four test-tubes, and add about 5 cb.c. of one of the above acids (diluted with water in the proportion of 1 of acid to 3 of water, the strength of acid with which your bench bottles are filled), putting dilute sulphuric acid in the first test-tube, hydrochloric in the second, and so on. Notice the result in each case, and then try similar experiments with other metals in place of zinc, such as magnesium, iron, copper, lead, and tin. Tabulate your results thus :—

Metal	Sulphuric Acid	Hydrochloric Acid	Nitric Acid	Acetic Acid
Zinc	Colourless inflammable gas evolved, and metal dissolves	—	—	—
Magnesium	—	—	—	—

(*c*) Put a small piece of marble (calcium carbonate) in each of four test-tubes; to the first add about 10 cb.c. of dilute sulphuric acid, to the others the same quantity of one of the other three acids; if any gas is evolved test it by holding a lighted match in the mouth of the test-tube, and also by making the gas bubble through some clear lime-water in another test-tube. Try a similar experiment with other carbonates in place of the marble, such as soda crystals (sodium carbonate), magnesite (magnesium carbonate), and white lead (lead carbonate). Embody your results in a table similar to that given above.

Bases.—These are substances which are able to combine with and neutralise acids, taking away their essential acid properties, such as those of turning blue litmus red and of expelling carbon dioxide from carbonates; the bases are mostly oxides of metals, or compounds of these oxides with water which are known as hydrates.

EXERCISE 61.—(*a*) Fill several small beakers or test-tubes about one-third full of dilute sulphuric acid, and add enough litmus solution to each to produce a distinct red colour; to the separate beakers or test-tubes add (1) solution of sodium hydrate, (2) solution of potassium hydrate, (3) solution of ammonium hydrate, (4) magnesium oxide, (5) zinc oxide, (6) iron oxide; the liquids must be added drop by drop, the solids must be in fine powder, and are to be added in small quantities at a time, and the mixture is in each case to be well stirred or shaken after each addition of one of the above six substances.

Notice the result in each case and draw up your observations in the form of a table.

(*b*) Try the same experiment with hydrochloric or nitric acid in place of the sulphuric.

(*c*) Finally try the action of the six bases mentioned above upon a neutral solution of litmus.

These experiments show that the bases you have tried differ considerably in the strength with which the reactions characteristic of bases are exhibited: the most pronounced bases are often spoken of as *alkalies*, and sodium and potassium hydrates, as well as ammonia, come under this head. The alkalies are not only able to take away from acids their acid character, but themselves have properties which are in some respects the opposite of these possessed by acids; thus they turn red litmus blue, as you have just seen, and later on in Exercise 82 you will see that they are able to absorb carbon dioxide, forming carbonates, whereas the acids decompose carbonates and evolve carbon dioxide.

Neutralisation.—The next point to be taken up is the quantitative examination of the reaction between acids and bases.

EXERCISE 62.—(*a*) Measure 10 cb.c. of a dilute sulphuric acid from your bench bottle (using a pipette, if you are sufficiently accustomed to the use of that instrument to have no fear of sucking up the acid into your mouth, or otherwise a measuring cylinder or burette) and put them in a beaker, with enough litmus solution to produce a distinct red tinge. Fill a burette with a solution of sodium hydrate, hold it over the mouth of the bottle from which the solution has been taken, and open the spring clip quite wide to let the liquid drive out all the air from the jet of the burette; then close the clip, and fill up the burette with sodium hydrate solution to the zero mark.

Now place the beaker containing the 10 cb.c. of dilute acid under the burette, and opening the clip slightly, run the solution of sodium hydrate into the beaker by small quantities at a time, until the red colour of the litmus is *just* turned blue. Read off on the scale of the burette the volume of alkali added, then pour the solution into a weighed porcelain dish and evaporate the liquid to complete dryness on the sand-bath; weigh the dish together with the solid residue left in it, then examine the residue by tasting it and try its action upon litmus; is it acid or alkaline? It is called a *salt*, this particular salt being sodium sulphate; what weight of it have you obtained from the 10 cb.c. of dilute sulphuric acid?

(*b*) Repeat the experiment, taking, however, 20 cb.c. of dilute sulphuric acid, using both this and also the sodium hydrate solution of the same strength as in (*a*). Compare the quantity of alkali required for neutralisation, and also the weight of sodium sulphate formed with the corresponding results in (*a*).

(*c*) Try similar experiments with other acids than sulphuric, and other alkalies than sodium hydrate; very much weaker solutions than those kept in the bench bottles may be used with advantage if it is not desired to determine the weight of the salt formed: that can also be done with the weaker solutions, of course, if larger volumes of liquid are used.

Salts.—The experiments just made have shown you that when an acid is neutralised by a base a substance is formed which is a crystalline solid and is generally neutral to litmus; these substances are called salts, and are to be regarded as derived from the acid by replacement of its hydrogen—for all acids are found to contain hydrogen—with a metal; this is represented in the equations for the various reactions by which salts are formed, such as the following:—

$$\underset{\text{acid}}{HCl} + \underset{\text{alkali}}{NaOH} = \underset{\text{salt}}{NaCl} + H_2O$$

which applies to the case of hydrochloric acid and sodium hydrate; this also represents the fact that water is formed together with a salt when an acid and an alkali react, a fact which is not seen from the experiments you have just done, for you have always used mixtures of acid or alkali with a large proportion of water.

A Law of Chemical Combination.—The experiments in this chapter also serve as instances of what has been found to be a general law of chemical combination: you have found that each acid when neutralised with a base combines with it in a fixed proportion and yields a fixed proportion of the salt which is the chief product of the reaction; the general law is that *when substances combine chemically they do so in fixed proportions*, and is applicable to all kinds of chemical combination, and not only to that between acids and alkalies.

All the quantitative experiments with regard to chemical action which are described in this book will give further evidence of the truth of this general law, if the experiments are carefully made and accurate results obtained; some have already been performed in Exercises 30 and 45.

CHAPTER XIV

HYDROCHLORIC ACID AND THE CHLORIDES

The Action of Strong Sulphuric Acid on Common Salt: Hydrochloric Acid.—Common salt was found many years ago to yield a gas of peculiar properties when treated with strong sulphuric acid; this gas was called "marine acid air," but is now known as hydrochloric acid. Common salt is not by any means the only substance which will give this gas, for there is a whole series of compounds—the chlorides—all of which evolve this same gas when acted upon by concentrated sulphuric acid.

EXERCISE 63.—(*a*) Put a little common salt in a dry test-tube and pour over it some strong sulphuric acid; try the same experiment with any other chloride, such as calcium chloride, sal-ammoniac (ammonium chloride), magnesium chloride, etc.

(*b*) Fit up a flask with thistle funnel and delivery tube, arranged as shown in Fig. 45, so that the flask can be heated on a sand-bath, and the gas collected by downward displacement; put 20 grams of salt in the flask; in a beaker put 15 cc. of water, and pour into this in a thin stream 45 cc. of strong sulphuric acid; allow the mixture to cool and then pour it on to the salt.

Heat the flask on the sand-bath and regulate the rate of evolution of the gas by raising or lowering the flame: make the following experiments:—

(i.) Test a jar of the gas with a lighted taper; notice also the white fumes which the gas gives in contact with moist air.

(ii.) Cover the top of the second jar of the gas with paper; invert, and put the mouth in a basin of water coloured blue with litmus.

(iii.) Lead the gas into water in a beaker or bottle; after some time test portions of the solution obtained for the presence of an acid by trying its action on (*a*) blue litmus paper, (*b*) lumps of marble or limestone, (*c*) bits of zinc or iron. To the rest of the solution add some litharge and boil; filter whilst hot and let the clear filtrate cool: the crystals which form are lead chloride $PbCl_2$, formed by the action of the acid HCl upon the base PbO. Try to work out the equations.

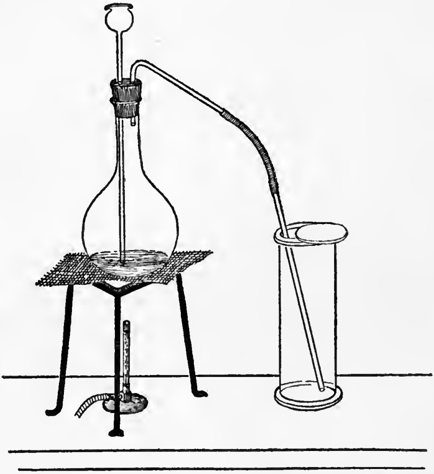

FIG. 45.—Preparation of Hydrochloric Acid Gas.

(iv.) Dry thoroughly the long glass tube used in Exercise 36, and fill it with dry HCl by downward displacement; the gas should be led in through a long, narrow, and dry tube, and is best bubbled through concentrated sulphuric acid in a small gas-washing bottle on its way (Fig. 46); when full, close the tube with a dry indiarubber cork.

Put 100 grams of mercury in a dry mortar; add to it several small pieces of sodium (each the size of a pea), pressing each piece with the pestle until it combines with the mercury with a flash of light; when the amalgam begins to turn pasty do not add any more sodium.

Pour the sodium amalgam thus prepared down the side of the tube containing the HCl gas, and at once replace the cork; shake the tube vigorously for five minutes, then open under a basin of water. What happens?

Put your thumb over the mouth of the tube, remove it from the basin and test the contained gas with a lighted taper. What gas is it?

Hydrochloric acid is decomposed by sodium amalgam, and in this reaction the mercury plays no part beyond putting the sodium in the form of a liquid alloy, which can be brought into more intimate contact with the gas than would be possible with the solid metal sodium; the action of the sodium is to combine with the chlorine of the hydrochloric acid and liberate the hydrogen, and the above experiment shows that the volume of this hydrogen is one-half that of the hydrochloric acid.

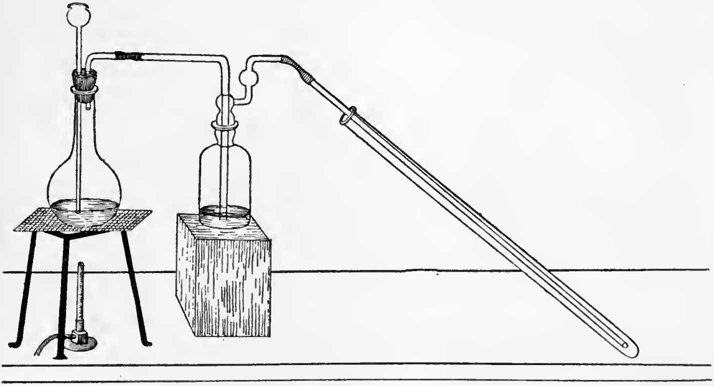

FIG. 46.—Filling a Tube with dry HCl.

Other experiments which are not of a kind that you can very well make for yourself show that the volume of chlorine is equal to that of the hydrogen with which it combines; see Roscoe and Lunt, pp. 179-181.

The Chlorides.—The salts of hydrochloric acid would be called *hydrochlorates* if we followed in this case the general plan of naming salts after the acids from which they are derived; these salts of hydrochloric acid are, however, identical with the compounds of the metals with chlorine (see next chapter), and hence the shorter name *chlorides* is always used. They can be obtained from hydrochloric acid in several ways, as by acting with it on a metal, or on a base, or on the carbonate of a metal.

EXERCISE 64.—(*a*) Put 50 cb.c. of dilute hydrochloric acid in each of three small beakers: to the first add about two grams of magnesium, to the second about three grams of magnesium oxide (magnesia usta), and to the third about 6 grams of magnesium carbonate (magnesite); what happens in each case? Pour off the clear liquid from the residue, or filter if necessary, and then evaporate each solution separately to a small bulk; allow the liquid to cool and examine the crystals which are formed to see whether the same substance is produced in each case.

(*b*) Prepare potassium chloride from pearl-ash (potassium carbonate); copper chloride from copper oxide.

Double Decomposition.—There is another and very convenient method by which those chlorides can be prepared which are insoluble, or only slightly soluble, in water; that is, by double decomposition between two solutions, one containing a soluble chloride and the other a soluble salt of the metal whose chloride is to be obtained; when these solutions are mixed there is a mutual interchange between the two salts, the metal of each combining with the acid radicle of the other.

Lead chloride is such a chloride, being only slightly soluble in water; and if a solution of a soluble chloride like sodium chloride or hydrochloric acid (hydrogen chloride) is mixed with a solution of lead nitrate, a double decomposition occurs and lead chloride separates out as a white precipitate, while sodium nitrate (or nitric acid) remains in solution:—

Lead Nitrate (soluble)		Lead Chloride (insoluble)
and	give	and
Sodium Chloride (soluble)		Sodium Nitrate (soluble)

EXERCISE 65.—(*a*) Pour some solution of lead nitrate in a beaker and add solution of sodium chloride or of hydrochloric acid until no more precipitate is formed; what is this precipitate, and how is it formed? Put some of it in a test-tube with a

fair proportion of water and heat to boiling; if the precipitate does not dissolve pour half of it away, add more water and boil again. Then allow the solution of the lead chloride in hot water to cool, and notice that the chloride separates in small crystals; this behaviour is fairly characteristic of lead chloride.

(*b*) Put a little solution of silver nitrate in a test-tube and add some solution of sodium chloride or any other soluble chloride; the precipitate which forms is silver chloride, a substance almost entirely insoluble in water whether hot or cold.

Pour half of the precipitate into another test-tube; to one portion add nitric acid, to the other solution of ammonia, and notice what happens in each case.

Tests for Chlorides.—The last reaction with silver nitrate is very generally used as a test for the presence of chlorides in a solution; another test for a chloride when a solid substance is to be examined is to put some of it in a test-tube and pour strong sulphuric acid over it when, as you have seen, hydrochloric acid is evolved.

CHAPTER XV

CHLORINE

The Oxidation of Hydrochloric Acid: Chlorine.—Chlorine was first prepared by the Swedish chemist Scheele, in 1774, by heating hydrochloric acid (the solution of the gas) with a certain mineral now known as manganese dioxide; the action may be regarded as an oxidation of the hydrochloric acid to water and chlorine, and many other oxidising agents may be used in place of the manganese dioxide with almost equal advantage: still Scheele's process remains the best, and is worked on a very large scale in preparing chlorine for the manufacture of bleaching-powder.

EXERCISE 66.—(*a*) Try what happens when a few cc. of strong hydrochloric acid (the solution of the gas) are heated in a test-tube with each of the following oxidising agents—manganese dioxide, red lead, potassium bichromate. Be cautious not to inhale more than a trace of the vapours given off, as they are very irritating.

(*b*) Take a flask fitted with a doubly-bored cork, which should be well smeared with vaseline to protect it from the action of the gas: through the cork pass a thistle funnel and a delivery tube leading to a small gas-washing bottle half filled with water (if this is dispensed with the gas given off will contain a good deal of HCl, which interferes with some of the experiments); from this the delivery tube is so arranged that the gas can be collected by downward displacement (Fig. 47). The whole should stand in a draught cupboard which is in good working order.

Wash out the flask with water, then put in it 40 grams of manganese dioxide and 120 cc. of concentrated hydrochloric acid (the crude acid is quite suitable) and mix thoroughly by shaking. Heat the flask gently on a sand-bath or wire gauze until the gas is evolved in a steady stream: collect in jars and do the following experiments:—

(i.) Notice the colour of the gas, but be very cautious about smelling it; then put a lighted taper into the jar of chlorine (if there is HCl mixed with the chlorine the taper will be put out).

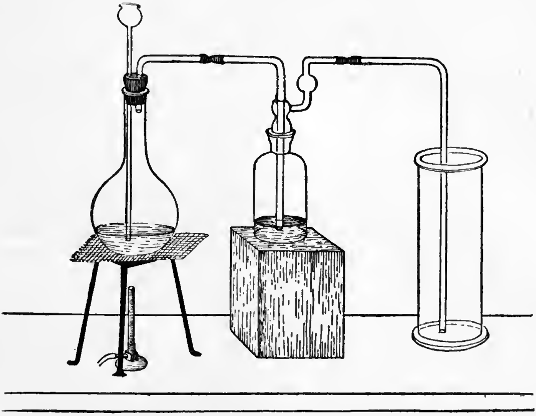

FIG. 47.—Preparation of Chlorine.

(ii.) Heat a little turpentine in a test-tube and pour a few drops on to a piece of filter paper: drop this into a jar of chlorine.

(iii.) Powder a little antimony very finely in a mortar: then sprinkle it from a piece of paper into a jar of chlorine.

(iv.) Fit up a small hydrogen apparatus, with a tube bent as shown in Fig. 48, and drawn out to a jet at the end; after testing to find whether it is safe to do so, light the hydrogen at the jet and then lower the flame into a cylinder of chlorine.

(v.) Try to burn a piece of charcoal in the gas.

(vi.) Drop into a jar of the gas a piece of red Turkey twill and a piece of printed paper smeared with writing ink.

(vii.) Pour about 20 cc. of strong sulphuric acid into a jar of chlorine, replace the cover, and carefully incline the jar, at the same time rotating it so that the acid is spread over nearly the whole inner surface; the gas will then be dry in a few minutes: take a piece of Turkey red twill which has been dried in a steam-oven or on wire gauze above a flame, and put it into the jar of dry chlorine with one corner left underneath the cover; allow to stand and then notice the difference in the result of this experiment and that with the moist gas.

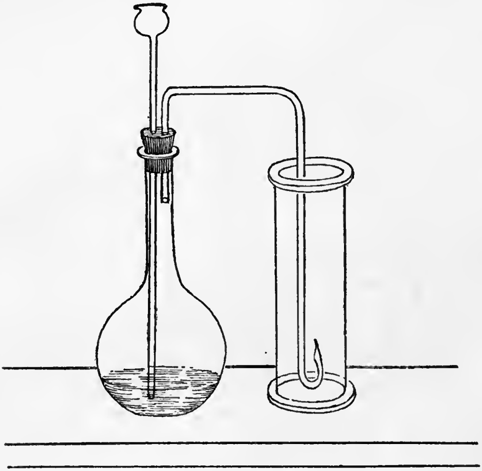

FIG. 48.—Burning Hydrogen in Chlorine.

(viii.) Take out the solution of chlorine—or chlorine water—from the gas-washing bottle (take care not to breathe the fumes) and try its action on solutions of indigo or of any other dye.

CHAPTER XVI

SULPHUR, THE SULPHIDES, AND SULPHURETTED HYDROGEN

SULPHUR is obtained from two chief sources—(*a*) native sulphur found in volcanic districts, and (*b*) iron pyrites FeS_2, of which immense quantities are used in the manufacture of sulphuric acid; a large proportion of this acid is used up by the alkali makers, and the sulphur of the acid remains in combination with lime in the "alkali waste"; from this the sulphur is now generally recovered by Chance's process.

The Properties of Sulphur.—Sulphur is remarkable as occurring in several "allotropic" forms with strikingly different properties; three of these modifications are very distinct and easily prepared.

EXERCISE 67.—(*a*) Put some flour of sulphur in a dry test-tube and half fill it with carbon disulphide, *taking great care not to bring this very inflammable liquid near a light;* shake well, and then filter through a dry filter paper into a small dry beaker; cover this over with a piece of paper and put away in a good draught cupboard or in the open air for the carbon disulphide to slowly evaporate. In the meantime go on with the next experiments.

When all the carbon disulphide has evaporated, take out the crystals of sulphur and examine the best of them carefully, if possible with a lens: make a drawing of the shape of the crystals, and note whether they are light or dark yellow, transparent or only translucent, brittle or elastic.

(*b*) Put some lumps of sulphur in a dry porcelain dish and heat over a small flame until the sulphur boils or takes fire, noting carefully the changes in appearance and consistency of the sulphur as its temperature rises; then take firm hold of the dish (Fig. 49) with crucible tongs at a point opposite the spout and pour the melted sulphur in a thin stream into cold water in a large beaker (the dish to which some of the burning sulphur still adheres should be put away at once in a draught cupboard, *not* in the one where the carbon disulphide is evaporating, and the flame extinguished by stretching a damp duster over the mouth of the dish).

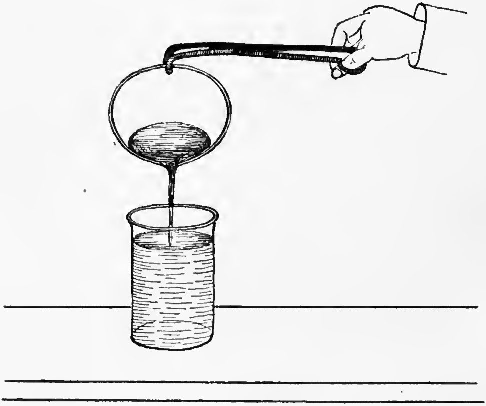

FIG. 49.—Preparing Plastic Sulphur.

Take out the sulphur from the beaker of water and examine its properties, then put away until your next lesson and examine again.

(*c*) Put some more lumps of sulphur in the porcelain dish and heat on the sand-bath until the sulphur is all melted: then turn out the flame, take off the dish and allow it to cool. As soon as a crust has formed on the top of the dish pierce two holes at opposite sides of the crust, and pour out the still liquid portion of the sulphur into a beaker of cold water. Let the dish stand for a few minutes more, then cut round the top crust with a knife and remove the whole of the crust: examine the crystals carefully with a lens, make a drawing of their shape, and note whether they are light or dark yellow, transparent or translucent, brittle or elastic; then put away until the next lesson and examine the crystals again.

The Sulphides.—Sulphur combines readily with almost every element: you have already seen how easily it takes fire in the air, burning to produce a gas with the well-known smell of burning sulphur; it also enters into combination with the metals, forming the sulphides, and one instance of this you have already noticed—that of iron; see Exercise 18.

EXERCISE 68.—(*a*) Heat strongly in a dry test-tube a small quantity of copper filings mixed with twice the bulk of flour of sulphur.

(*b*) Put a small lump of sulphur in a test-tube; roll a piece of tin foil into a loose ball and push it to within an inch of the bottom of the tube; heat the sulphur until it boils and the vapour reaches the ball of tin foil.

Action of Acids on Sulphides — Sulphuretted Hydrogen.—When a metallic sulphide is acted upon with hydrochloric or sulphuric acid, it generally happens that a gas is evolved which can be recognised by its smell and other properties as sulphuretted hydrogen H_2S; some sulphides are not acted upon, whilst nitric acid never liberates sulphuretted hydrogen, as that acid is decomposed by the gas with separation of sulphur.

EXERCISE 69.—(*a*) Try the action of dilute H_2SO_4 (1 of acid to 2 of water) and of dilute HCl (1 of strong acid to 1 of water) upon the following sulphides:—ferrous sulphide FeS, iron pyrites FeS_2, antimony sulphide Sb_2S_3; if there is no reaction in the cold, heat.

(*b*) Fit a flask with thistle funnel and delivery tube arranged to collect the gas over water; it is advisable to use hot water as the gas is fairly soluble in cold, but much less in hot water. If preferred, the gas, being heavier than air, can be collected by downward displacement, but then more of it escapes into the room. Do the following experiments:—

(i.) Light a jar of the gas: notice the colour of the flame and that part of the sulphur is deposited on the sides of the jar, while part of it burns to produce sulphur dioxide; the deposit of sulphur is very difficult to remove if once allowed to dry, and should therefore be rubbed off at once with water and a duster.

(ii.) Take a cylinder full of H_2S, and a second cylinder full of chlorine or, as being more accessible, of bromine vapour: put the two cylinders mouth to mouth and then draw away the glass plates; mix the two gases by inverting the pair of cylinders several times, always keeping them pressed firmly together. The chlorine or bromine unites with the hydrogen of the H_2S and sets free the sulphur.

(iii.) Put a little strong nitric acid in a beaker, and bubble sulphuretted hydrogen into it; try the same with nitric acid diluted with its own bulk of water, and finally with the dilute nitric acid (1 in 4) from your bench bottle: what happens in each case?

(iv.) Pass the gas into water in a large beaker; some of the gas is dissolved, forming a solution which may be used in the the next experiment.

(v.) In four separate test-tubes put about 5 cc. of solutions of the following:—(*a*) lead nitrate, (*b*) mercuric chloride, (*c*) copper sulphate, (*d*) zinc sulphate, (*e*) calcium chloride. Fill up each test-tube nearly to the top with solution of sulphuretted hydrogen and shake; note what happens in each case; the precipitates which are formed in some cases are sulphides of the respective metals.

Use of Sulphuretted Hydrogen in Analysis.—On account of the striking differences in colour and in other properties, such as solubility in water and in acids, of the various metallic sulphides, sulphuretted hydrogen is largely used in chemical analysis as a re-agent; for this purpose it is convenient to have an apparatus from which a stream of the

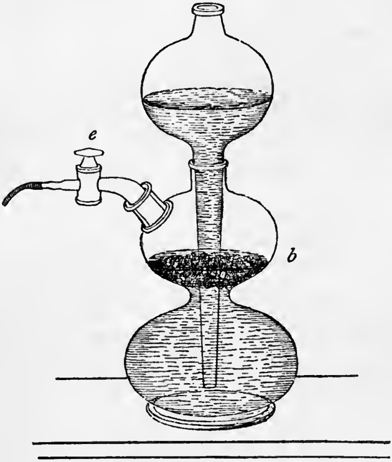

FIG. 50.—Kipp's Apparatus for generating Sulphuretted Hydrogen.

gas can be obtained at will; one well-known arrangement is that invented by Kipp and shown in Fig. 50, which has lumps of ferrous sulphide in the middle globe and hydrochloric acid diluted with its own bulk of water in the other two; on opening the tap *e* the acid is forced by its own pressure into the middle bulb, and there acting on the ferrous sulphide liberates sulphuretted hydrogen: when the tap is closed, the gas accumulates in *b* and gradually forces the acid back again out of contact with the lumps of sulphide, and thus stops the action. A newer form of apparatus, much more convenient when a large supply of the gas is needed, is that of de Koninck, shown in Fig. 51; this is on a similar principle to the Kipp's apparatus, but holds a much larger quantity of acid, and uses it up far more completely, giving a steadier current of gas and for a much longer time without the necessity of the unpleasant operation of refilling.

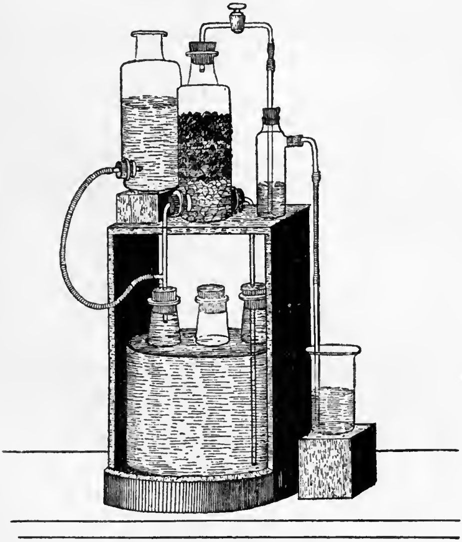

FIG. 51.—De Koninck's Apparatus for generating Sulphuretted Hydrogen.

CHAPTER XVII

SULPHUR DIOXIDE

SULPHUR dioxide is formed when sulphur burns in air or oxygen, and also when a sulphide is "roasted"—that is, heated in a current of air; large quantities of the gas are required for the manufacture of sulphuric acid, and are obtained either by burning sulphur or by roasting iron pyrites FeS_2.

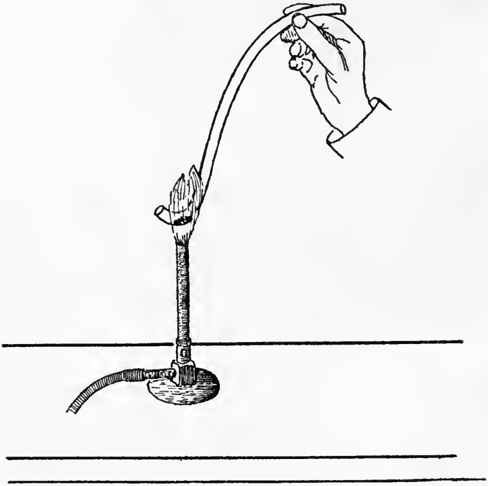

FIG. 52.—Roasting Iron Pyrites.

EXERCISE 70.—(*a*) Take a piece of hard glass tubing about 30 cm. long and bend it as nearly as you can to the shape shown in Fig. 52; put a small piece of sulphur in the lower bend, and then support the tube in the flame of a Bunsen burner so that the vertical portion of the tube is kept hot: in this way a draught of air will be caused and the products of oxidation will issue from the upper opening; test these by smelling, and by holding a piece of damp litmus paper in the fumes.

(*b*) Do the same experiment with some coarsely powdered iron pyrites in place of the sulphur.

Preparation from Sulphuric Acid.—In order to prepare a sufficient quantity of the gas to examine its properties it is more convenient to start from sulphuric acid and recover from it the sulphur dioxide used in its manufacture; this can be done by heating the strong acid with certain metals or other substances, such as carbon or sulphur itself, which readily take up oxygen.

EXERCISE 71.—(*a*) Put a few copper turnings in a test-tube, cover them with concentrated sulphuric acid and heat.

(*b*) Put a piece of charcoal in a mortar, pour a little strong sulphuric acid on to it, and rub into a paste; add a little more acid and then pour into a test-tube; heat the mixture.

(*c*) Heat a small lump of sulphur with some strong sulphuric acid in a test-tube.

(*d*) To prepare the gas in larger quantity, fit up the same apparatus as was used in making HCl (Fig. 45); put 15 grams of copper turnings in the flask, and add through the funnel 25 cc. of strong sulphuric acid; heat carefully on wire gauze, and as soon as the gas begins to come off fairly rapidly turn down the flame to prevent too rapid evolution of gas. Collect by downward displacement and do the following experiments:—

(i.) Test a jar of the gas with a lighted match; light the match again and pour the gas downwards over the flame.

(ii.) Fill a jar with the gas, cover the mouth with a piece of paper, and then put the jar mouth downwards in a basin of water coloured blue with litmus.

(iii.) Fill a jar with SO_2, and another jar, which should first be washed out with water to wet the sides thoroughly, with H_2S from the apparatus fixed for general use in the laboratory: place the two jars mouth to mouth, the one which contains the

SO_2 being above, and slip out the two glass plates ; allow the two gases to mix and notice the result.

(iv.) Fill a jar with water and lead the gas into it ; after a few minutes the solution will smell strongly of the gas, and may be used for the following experiments :—

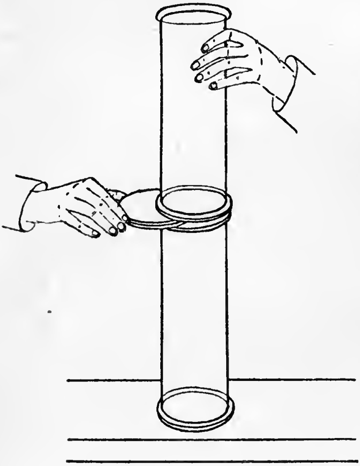

Fig. 53.—Exercise 71 (iii.).

(v.) If a red flower can be obtained, dip a petal of it in the solution of sulphur dioxide, and notice the effect on the colour of the petal ; an infusion, made from dried flowers (rose leaves) by pouring hot water over them, may be used if fresh flowers are not available.

(vi.) Put a little carbonate of soda in a test-tube, and pour some of the solution of sulphur dioxide over it : what happens, and what do you conclude from the result ?

The Sulphites.—The last experiment shows that the solution of SO_2 in water contains an acid strong enough to expel CO_2 from a carbonate ; this acid is called sulphurous acid, but is only known in solution, though its salts, which are termed sulphites, are much more stable ; the formula of the acid is H_2SO_3, and as each of the two hydrogen atoms can be replaced by metals, we can get two series of sulphites and the acid is called dibasic. Thus there are two sodium sulphites, one—$NaHSO_3$—in which only half of the hydrogen in the acid has been replaced, and the other—Na_2SO_3—in which the whole of the hydrogen has been replaced by sodium.

Exercise 72.—(*a*) Put 20 cc. of a solution of sodium hydrate NaOH in a beaker and add solution of SO_2, or lead SO_2 into the soda, until it smells strongly of the gas after being well stirred; then pour into a dish and evaporate on the water-bath until a white solid remains; this is sodium hydrogen sulphite or bisulphite $NaHSO_3$. Put half of the salt in a specimen tube, and leave the other half in the dish for the next experiment.

(*b*) To half of the sodium bisulphite obtained in the previous experiment add 10 cc. of the same solution of NaOH; again evaporate to dryness on the sand-bath. The salt thus obtained is sodium sulphite Na_2SO_3.

(*c*) Put some sodium sulphite, or any other sulphite, in a test-tube and pour dilute sulphuric acid over it; test the gas evolved by smelling it, and by holding a piece of blue litmus paper in the fumes.

(*d*) Put some freshly prepared solution of SO_2 in a test-tube, and add barium chloride solution; divide into two halves, add ammonia to one half and dilute HCl to the other.

CHAPTER XVIII

SULPHUR TRIOXIDE AND SULPHURIC ACID

Formation of Sulphur Trioxide.—Sulphur trioxide SO_3 is not formed in appreciable quantity when sulphur is burned in air or oxygen under ordinary circumstances, the oxidation stopping at the lower stage corresponding to the oxide SO_2. Under certain conditions, however, sulphur dioxide SO_2 is able to combine with more oxygen to form the trioxide; the presence of hot platinum, especially when in a finely-divided state, is found to be peculiarly favourable to this action, though the precise way in which the platinum influences the chemical change is not thoroughly understood.

EXERCISE 73.—Cover some shreds of asbestos with finely-divided platinum by dipping them several times in a solution of platinum chloride and heating strongly in the Bunsen flame after each immersion; put the shreds of platinised asbestos in a bulb tube of hard glass, and then connect this with the apparatus shown in Fig. 54; the tube A is partly filled with a mixture of dry $KClO_3$ and MnO_2, and the flask B is half filled with a strong solution of SO_2, while at C is the bulb containing the platinised asbestos. Heat the mixture in A so that a slow stream of oxygen is evolved, which passing through B takes up SO_2; let the issuing gases pass into a test-tube D half full of water, and after a few minutes test the solution in this test-tube by adding $BaCl_2$ solution; if there is no precipitate, pour in a few drops of ammonia; then add dilute HCl; does the precipitate dissolve? What does your result indicate?

Now heat the bulb C while the stream of mixed gases is passing through it; notice the white fumes evolved and then put another test-tube half full of water at D and make the white fumes bubble through the water; in a few minutes take away the two burners and remove the tube A from its connection with the flask B before the solution in this is sucked back into the tube. Test the liquid in D by adding $BaCl_2$ solution and then dilute HCl as before; is the result different? Apply the same test to some dilute sulphuric acid from the bench bottle.

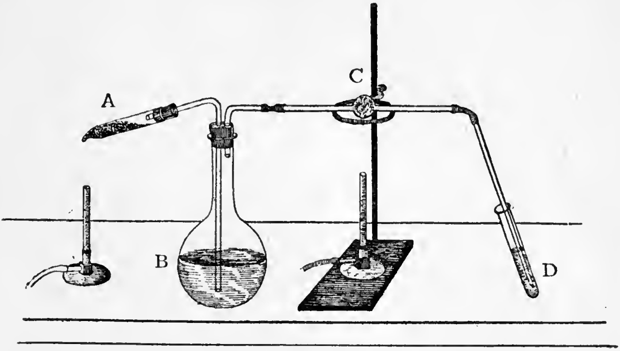

Fig. 54.--Apparatus for making Sulphur Trioxide.

Sulphuric Acid

Manufacture of Sulphuric Acid.—Sulphuric acid is manufactured in immense quantities by the oxidation of SO_2, in presence of water, by means of the oxygen of the air; this is a change which, under ordinary circumstances, takes place only slowly, but if a small quantity of the vapour of nitric acid be mixed with the air and SO_2 it is found that the oxidation occurs far more readily; the following experiments should help you to see in what way the nitric acid works:—

EXERCISE 74.—(*a*) Put some strong solution of SO_2 in a small flask, add half the bulk of strong nitric acid, and heat until the liquid boils; is any gas evolved from the mouth of the flask? If so, what gas is it? Test the liquid left in the flask for sulphuric acid.

(*b*) Put a few copper turnings in a large flask and pour a little nitric acid over them; lead the red fumes into another large flask, in the bottom of which some strong solution of SO_2 has been placed; cork this flask and shake it well; what happens? Take out the cork and blow a little air into the flask through a tube; are there any red fumes formed? If so, how do you explain their formation? Then test the solution in the flask for sulphuric acid.

Upon the results of these experiments it should be possible for you to give an explanation of the mode of action of the nitric acid in the manufacture of sulphuric acid; write down what happens in the following cases:—

(i.) HNO_3 acts upon SO_2 in presence of water

(ii.) The gas generated in (i.) mixes with air.

(iii.) The gas produced in (ii.) comes in contact with SO_2 and water, and you will find that at the end of (iii.) the same gas is set free as in reaction (i.), so that the changes (ii.) and (iii.) may occur many times over without any more nitric acid being used.

The apparatus needed to represent on a small scale the process used in manufacturing sulphuric acid is complicated, and is better shown to the students in class, rather than set up by each separate student; such an apparatus is described in Roscoe and Lunt's *Inorganic Chemistry for Beginners*, p. 208.

Properties of Sulphuric Acid.—For the following experiments with the acid the manufactured acid may be used:—

EXERCISE 75.—(i.) Put some water in a beaker, and then pour into it in a thin stream about the same bulk of concentrated sulphuric acid; is there any rise of temperature?

(ii.) Pour enough of the strong acid into a small beaker to fill about one-third of it; mark the level of the liquid with a small piece of gummed paper stuck on the outside of the beaker, and put away for a day or more in a place where the beaker will not be upset; then notice whether the volume of the liquid has undergone any change.

(iii.) Dip a glass rod or glass tube closed at the end in dilute sulphuric acid and trace characters on a piece of paper; dry the paper at a safe distance above a flame; what happens, and how do you explain it? Note that cellulose, which forms the bulk of paper, is a compound of the three elements C, H, and O, having the formula $C_6H_{10}O_5$.

(iv.) Put 15 grams of powdered sugar in a small beaker;

heat half a test-tube full of water to boiling, and pour it over the sugar; when the sugar is dissolved, fill a test-tube nearly full of strong sulphuric acid and pour this into the syrup (the beaker should stand in an evaporating basin). Notice the smell of SO_2, as well as the black mass produced, and consult Exercise 71 (*b*) if you are unable to explain the formation of that gas.

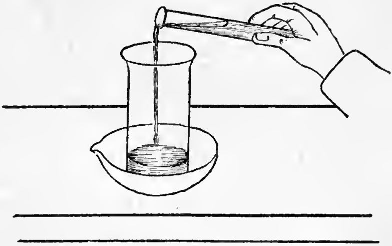

FIG. 55.—Exercise 75 (iv.).

(v.) Devise for yourself such experiments as you can, to show the *acid* character of sulphuric acid.

The Sulphates.—Like sulphurous acid, sulphuric acid is dibasic, and forms both acid salts and normal salts; these sulphates can be obtained by the action of the acid on the metals, on their oxides or hydrates, or on a salt with some other acid which the sulphuric acid is able to displace.

EXERCISE 76.—(*a*) Prepare zinc sulphate by dissolving the metal in dilute sulphuric acid, and evaporating the filtered solution until it crystallises on cooling.

(*b*) Put 20 cc. of dilute sulphuric acid in a beaker, and add solution of potassium hydrate from a burette until the liquid after stirring is just alkaline to litmus, noticing what volume of the alkaline solution has to be added; if no crystals of potassium sulphate separate at once, evaporate the liquid to half the bulk and put away to cool.

In another beaker put 20 cc. of the same sample of dilute acid, and add only half the volume of the same sample of potassium hydrate solution that was found to be necessary to neutralise the acid. Evaporate this solution in the sand-bath to a small bulk, and then put away to cool.

Examine the two salts with respect to their solubility in water, crystalline form, effect on litmus solution.

(*c*) Prepare magnesium sulphate from magnesite, a natural carbonate of the metal, by adding excess of the powdered mineral to 25 cc. of dilute sulphuric acid; filter the liquid whilst still warm, and evaporate the liquid to half the bulk; then put the liquid in a beaker and allow to cool.

(*d*) Make some lead sulphate by adding about 20 cc. of dilute sulphuric acid to 20 cc. of solution of lead nitrate in a beaker; filter the white precipitate of lead sulphate, wash it with hot water and then dry it in a steam-oven or in the manner shown in Fig. 27. Examine the solubility of the $PbSO_4$ in water and in nitric acid. The reaction by which the lead sulphate is formed is another instance of "double decomposition" (p. 88).

(*e*) Prepare $BaSO_4$ in a similar way from barium chloride solution and sulphuric acid; examine its solubility in nitric and in hydrochloric acids. Is this reaction also a case of "double decomposition"?

Tests for Sulphates.—We have already (p. 103) made use of the insolubility of barium sulphate both in water and in dilute hydrochloric acid as a test by which to discover the presence of sulphuric acid; the same result is, of course, obtained when any other soluble sulphate is present instead of hydrogen sulphate or sulphuric acid.

Another test for sulphates depends on a "dry" reaction, that is, one which takes place between solid bodies at a high temperature and not in solution; when a sulphate is mixed with carbon and strongly heated, the carbon withdraws oxygen from the sulphate and reduces it to a sulphide, which is then easily detected either by the evolution of H_2S when treated with an acid, or by the formation of black silver sulphide when the mass containing a soluble sulphide is placed on a clean silver coin and moistened with water.

EXERCISE 77.—(*a*) Mix a little dry sodium sulphate with charcoal in a mortar and put some of the mixture in an ignition tube; heat strongly in the Bunsen flame, then cool and break open the tube. Put part of the residue on a watch glass and pour a little dilute HCl over it; is there any H_2S evolved? Put the rest on a bright silver coin, moisten with a drop of water and leave for a few minutes, then wash the coin and examine it for a brown stain (this can be removed by rubbing with a cloth and some moistened lime-powder).

(*b*) Try the same experiment with any other sulphate. With some sulphates this test in the above form does not work satisfactorily: some of the sulphides are insoluble in water and therefore do not react with silver, and are also insoluble in dilute acids so that no H_2S is evolved when dilute hydrochloric acid is poured over them; the following modification is the one generally used in analytical work and succeeds with any sulphate:—

EXERCISE 78.—Mix some lead sulphate with dry sodium carbonate, and put the mixture in a small cavity scooped out of a piece of charcoal. Turn off the air supply at the base of the Bunsen burner, and having adjusted the flame to a height of about 2 inches, direct it with the mouth blowpipe on to the charcoal; as your object is to reduce the sulphate it is best to heat the mass not with the extreme tip of the flame where oxygen is present in excess, but with the central or reducing portion where the coal-gas, being as yet only partly burned, aids the charcoal in withdrawing oxygen from the sulphate. The first reaction may be regarded as one of double decomposition between the lead sulphate and sodium carbonate:—

$$PbSO_4 + Na_2CO_3 = Na_2SO_4 + PbCO_3$$

and then the sodium sulphate is reduced to sulphide, while the lead carbonate is also reduced to metallic lead with production of CO_2.

Look for shining metallic globules on the charcoal; then remove the fused mass, place it on a silver coin, moisten with water and press into powder with a pestle; after a few minutes wash the coin and examine it for a brown stain of silver sulphide.

CHAPTER XIX

CARBON AND CARBON MONOXIDE

Varieties of Carbon.—Carbon occurs in several allotropic forms, which are described in Roscoe and Lunt's *Inorganic Chemistry for Beginners*, pp. 217-221; we shall refer you to that book for an account of these allotropic forms, and give only a few experiments bearing on the formation and properties of some of the chief varieties of amorphous carbon. These are formed by the action of heat upon organic substances containing carbon, and according to the substance acted upon, and to some extent according to the precise conditions under which the carbonisation is effected, we obtain different varieties of amorphous carbon, such as wood charcoal, animal charcoal, etc.

Carbon a Reducing Agent.—All of these are alike in some important respects, more particularly in the power of reducing, or withdrawing oxygen from, various compounds when heated with them, but in appearance and physical properties there is considerable difference between them.

EXERCISE 79.—(*a*) Put some sawdust or small pieces of wood in a porcelain crucible covered with a lid, and heat strongly with the Bunsen flame; when no more inflammable gases are given off allow to cool, then remove the lid and examine the residue; it is wood charcoal.

(*b*) Mix this wood charcoal with some powdered oxide of copper, which should be dry; put the mixture in a tube of hard glass (Fig. 56) and heat strongly, passing any gas evolved into a small beaker of lime-water; notice the effect on the lime-water, and when the reäction is over remove the beaker, *then* the flame, and examine the residue in the tube as soon as this is cold.

(*c*) Take a piece of charcoal, make a small hollow in it, and in this put some lead oxide (litharge); heat in the blowpipe flame until a metallic globule is formed; take this out, and note whether the metal is soft or brittle, and whether it marks paper.

(*d*) Mix a little white arsenic (oxide of arsenic) with powdered charcoal; and put the mixture in a dry ignition tube; heat and notice the mirror formed in the cold part of the tube by the condensation of metallic arsenic which is given off in vapour.

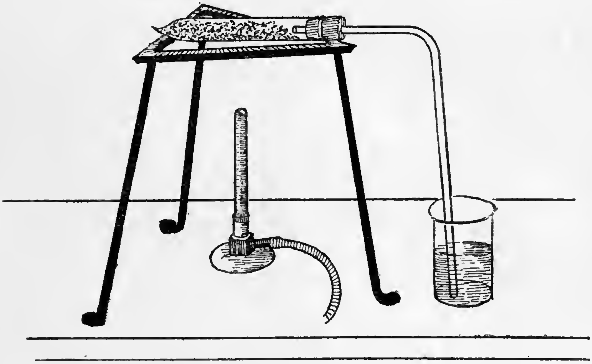

FIG. 56.—Reduction of Copper Oxide by ignition with Charcoal.

(*e*) Prepare some animal charcoal from bone meal in the same way as wood charcoal was prepared in (*a*); when no more gas is evolved, take away the flame and prepare a weak solution of indigo; put this in a small beaker, add the contents of the crucible and boil for a short time; then filter the liquid and notice whether the animal charcoal has had any effect.

Carbon Monoxide (CO)

Preparation of Carbon Monoxide from Formic Acid.—This gas is formed when the dioxide CO_2 is passed over red-hot carbon, and in this way very large quantities of it are formed in fires and furnaces; for laboratory purposes other methods are more convenient, and we shall examine two, both of which consist in acting upon an organic acid with strong sulphuric acid; you have already seen that the great affinity of this acid for water enables it to withdraw the elements of water from certain organic substances, and in the case of cellulose $C_6H_{10}O_5$ and cane-sugar $C_{12}H_{22}O_{11}$, both of which contain hydrogen and oxygen in the exact proportion in which they combine to form water, the product of the reaction is a black mass of carbon; but if we take formic acid CH_2O_2 and act upon it with strong sulphuric acid we obtain CO carbon monoxide.

EXERCISE 80.—Fit up a fairly small flask with cork and delivery tube arranged to collect the gas over water; put 10 cc. of formic acid in the flask, add 40 cc. of strong sulphuric acid, and, if necessary, heat gently; collect all the gas given off, and burn each jar of gas before allowing it to escape into the air. Do the following experiments:—

(i.) Remove the plate covering a jar of the gas just sufficiently to allow you to pour in some lime-water; shake and notice the result. Then set fire to the gas, and replace the cover immediately the flame dies out; shake again and note the result.

(ii.) Connect the flask from which CO is being evolved with a piece of hard glass tubing packed with copper oxide between two asbestos plugs; as soon as the air has been displaced from the tube light the gas which issues from the end, and then heat the tube at first cautiously to avoid cracking it, then strongly. What happens to the flame of burning CO? Lead some of the gas now coming from the end of the tube into lime-water; what happens and what conclusion do you draw?

Preparation from Oxalic Acid.—Carbon monoxide can also be obtained by heating oxalic acid $C_2H_2O_4$ with strong sulphuric acid, but in this case it is mixed with carbon dioxide.

EXERCISE 81.—In a flask of the same size as was used in the previous exercise put 10 grams of oxalic acid, and add 40 cc. of strong sulphuric acid: heat until gas is evolved, then lower the flame to prevent the reaction from becoming violent; collect over water.

(i.) Pour lime-water into a jar of the gas, and shake: then set fire to what is left.

(ii.) Fill the long tube used in Exercise 25 (*b*), with the gas evolved from oxalic acid, leaving, however, about 10 cc. of water in the tube: cork the tube under water, and then remove it from the basin; take a piece of solid sodium hydrate about an inch long, remove the cork from the tube and push in the caustic soda, not allowing it to fall into the water until after the cork has been replaced; shake well for a few minutes and open again under water. What do you conclude from the result, knowing that CO_2 is absorbed by a solution of sodium hydrate, while CO is not?

CHAPTER XX

CARBON DIOXIDE AND THE CARBONATES

Formation and Properties of Carbon Dioxide.—This gas is formed when carbon burns in a sufficient supply of air or oxygen, and can also be obtained by the action of heat or acids upon the carbonates, several of which occur in large quantities in nature; the most abundant of these is calcium carbonate, which is found as limestone, marble, and in other forms, and it was by the action of acids upon this substance that the gas was first prepared and described under the name of "fixed air."

EXERCISE 82.—(*a*) Heat strongly in ignition tubes small quantities of various carbonates such as those of potassium or sodium, calcium (limestone), magnesium (magnesite), zinc (calamine), iron (spathose iron ore), copper (malachite), lead (white lead), etc.; and notice in each case whether there is any change and whether a gas is evolved which puts out a burning match.

(*b*) Try the action of dilute acids upon the same carbonates, using ordinary test-tubes.

(*c*) Prepare carbon dioxide, using the apparatus shown in Fig. 57; put several small lumps of marble or limestone in the flask, taking care not to drop them heavily into the flask, but to slide them gently down the side of the neck, which should be held nearly horizontal; pour a little water down the thistle funnel, and then small quantities of concentrated hydrochloric acid as required: the gas may be collected either over water or by downward displacement, as it is much heavier than air; make the following experiments with the gas:—

(i.) Test a jar of the gas with a lighted match.

(ii.) Pour some of the gas downwards over a burning match.

(iii.) Hold a piece of burning magnesium ribbon held with crucible tongs in a jar of the gas; shake up the products of the combustion with dilute hydrochloric acid, which dissolves both magnesium and its oxide; are there any black particles left, and, if so, what are they?

(iv.) Put some blue litmus solution in a beaker and bubble carbon dioxide through it for several minutes; notice the change in colour, and then heat the beaker on wire gauze until the liquid boils.

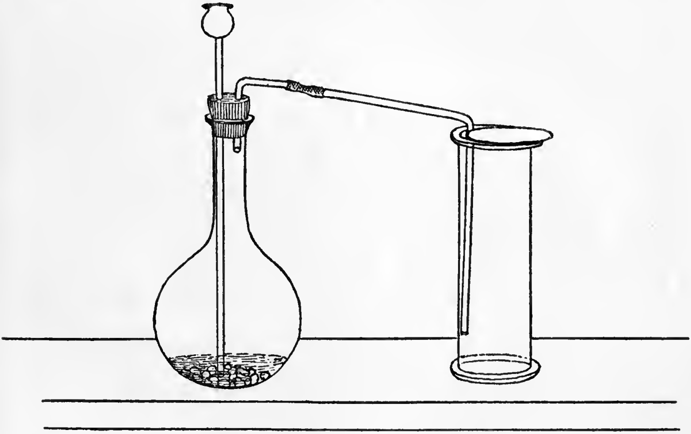

FIG 57.—Preparation of Carbon Dioxide.

(v.) To show the absorption of the gas by alkaline liquids take the long glass tube used in Exercise 25 (*b*); fit the india-rubber cork with a plug made of a short piece of glass tubing drawn out to a fine jet and then sealed, the closed end of this plug being placed so that it may be outside the tube when the cork is in position.

Fill the tube with water and then displace this with CO_2 at the pneumatic trough, leaving, however, about 10 cb.c. of water in the tube, then cork the tube and remove it from the trough; take a piece of solid sodium or potassium hydrate about an inch long and slide it a short distance inside the tube, which is held nearly horizontal: at once replace the cork, let the piece of alkali fall into the water at the bottom of the tube and shake well for a few minutes; put the mouth of the tube under water in a basin and break off the sealed top of the glass plug with crucible tongs.

(vi.) Measure the density of the gas in the following way:—Take a round-bottomed flask of about 400 cb.c. or 500 cb.c. capacity, fitted with a rubber cork through which passes a short piece of glass tubing; on the outer end of this tube fit a short piece of good rubber tubing with a spring clip attached.

Fill the flask with dry CO_2 by downward displacement, the gas being dried by means of a bulb tube packed with small pieces of calcium chloride; let the gas pass for at least five minutes in a steady stream through the flask, then carefully replace the cork and close the rubber tube with the spring clip. Weigh the flask and its contents.

Attach the flask by the rubber tubing to a filter pump in good working order; open the clip for about ten seconds, then close it, disconnect the flask and weigh it again; what is the loss in weight due to?

Put the mouth of the flask under water in a basin and open the spring clip: when no more water enters remove the flask, and measure the water which has entered. Calculate from your results the weight of a litre of CO_2 at the temperature and pressure of the laboratory, and compare with the numbers obtained by other students as well as with the exact number which can be ascertained from your teacher.

The Carbonates

Formation of Carbonates and their Properties.—You have seen by your experiments that carbon dioxide when bubbled into water produces an acid which is only strong enough to turn litmus to a port-wine red, quite distinct from the bright red produced by a drop of the dilute acid such as sulphuric; this weak acid is called carbonic acid, and is so unstable that it is not possible to determine its composition by direct analysis; its salts, the carbonates, are, however, much more stable, and their composition shows that the carbonic acid from which they are derived must be regarded as a dibasic acid with the formula H_2CO_3.

EXERCISE 83.—**The two Carbonates of Sodium.**—(*a*) Put 20 cc. of sodium hydrate solution in a beaker, and bubble CO_2 through the liquid until a drop of it taken out on the end of a glass rod no longer turns red litmus blue; measure 20 cc. of sodium hydrate solution from the same bottle, and add to the contents of the beaker; evaporate the liquid in a dish to about half the bulk, and then put it away to cool.

When cold, examine the liquid for crystals of the carbonate $Na_2CO_3.10H_2O$; notice their form, and test their solubility in water, and the behaviour of the solution towards litmus, and towards solution of gypsum (calcium sulphate).

Put the remainder of the crystals in a porcelain dish and heat on the sand-bath; the crystals first melt and then give off steam, finally leaving a white residue of the anhydrous carbonate Na_2CO_3; compare Exercise 37.

(*b*) In another beaker put 50 cc. of sodium hydrate solution (which should be of at least the strength 1 in 5), and pass a rapid current of CO_2 into it, keeping the liquid cool by placing the beaker in cold water; after a time minute crystals will form in the liquid, and when these do not increase in amount filter the liquid and collect the precipitate; press the crystals between filter paper and then complete the drying by exposure to the air; the substance is sodium bicarbonate $NaHCO_3$, and must not be dried by heat, as it is easily decomposed. Examine its solubility in water and the behaviour of the solution towards litmus and towards a solution of gypsum (calcium sulphate).

(*c*) Weigh a porcelain crucible and lid: nearly fill it with sodium bicarbonate which has been thoroughly dried and weigh again; heat the crucible and its contents over the Bunsen flame for ten minutes and again weigh; heat for five minutes more and weigh, repeating this process until the weight is found to remain constant. Compare the loss of weight with that calculated from the equation for the change which has occurred.

Dissolve the residue in a small quantity of hot water, and then allow to cool, first taking a little of the solution and diluting it in order to test its action on litmus and on solution of calcium sulphate. When the major part of the solution has deposited crystals, examine these and compare them with those of sodium carbonate $Na_2CO_3.10H_2O$.

(*d*) Finally draw up a table in which the properties of the two carbonates of soda are contrasted.

EXERCISE 84.—**The two Carbonates of Calcium.**—(*a*) Put a little water in a beaker and then add 20 cb.c. of lime-water, which is a solution of calcium hydrate $Ca(OH)_2$; pass carbon dioxide until the precipitate which forms at first is re-dissolved and no more CO_2 is absorbed; what do you suppose will be present in the solution now, judging from what happened in the case of sodium hydrate in Exercise 83 (*b*)? Now measure out 20 cb.c of lime-water and add to the liquid in the beaker; what happens, and what do you suppose is the composition of the substance formed, judging again from the previous exercise? There are two carbonates of calcium just as there are two of sodium; they are the carbonate $CaCO_3$, and the bicarbonate $Ca(HCO_3)_2$.

Can you now advance any explanation of the different behaviour of solutions of Na_2CO_3, and of $NaHCO_3$ when added to one of calcium sulphate?

(*b*) Prepare some solution of calcium bicarbonate as in (*a*); boil this solution for several minutes and note what happens.

(*c*) Finally draw up a table in which the properties of the two carbonates of lime are contrasted.

Measurement of the Volume of CO_2 from a weighed quantity of Marble.—We next examine quantitatively the reaction by which carbon dioxide is produced from an acid and a carbonate; in the first method to be described the amount of gas evolved is determined by measuring its volume, and in the second method by finding the loss of weight which the apparatus suffers in consequence of the escape of the gas.

EXERCISE 85.—Fit up the same apparatus as that used in Exercise 45; since carbon dioxide is distinctly soluble in water, it is best to saturate the water placed in the large flask with the gas before commencing the experiment proper, and this can be done either by bubbling carbon dioxide through the water for several minutes, or by adding to the water first about 5 cc. of sodium carbonate solution, and then enough dilute sulphuric acid to make the liquid distinctly acid to litmus after being well shaken.

When the whole apparatus is connected, the syphon tube in E being full of water, the cylinder empty, and the clip D closed, fill the small flask B two-thirds with dilute hydrochloric acid, and drop into it a *very small* lump of marble, not more than .3 gram, which shall dissolve while your first weighing is being done, and saturate the liquid with carbon dioxide.

Weigh exactly a small piece of marble, of weight from 1 to 1.5 gram; drop it into the small flask and *at once* attach this to its indiarubber cork fixed on the tube C. Open the spring clip D, and wait until the whole of the marble is dissolved; then raise the large flask A or the cylinder E, as may be required, until the level of the water is the same in both, close the clip and measure the volume of water collected in E.

From your knowledge of the density of carbon dioxide calculate the weight of this volume of the gas, and from this what weight of CO_2 could be obtained from one gram of marble; compare your result with those of other students.

EXERCISE 86.—**Determination of Loss of Weight when a Carbonate is dissolved in Acid.**—Take the small flask used in the last exercise, and fit a bulb tube packed with small pieces of calcium chloride into the indiarubber stopper as shown in Fig. 58. Fill the flask two-thirds with dilute hydrochloric acid (1 of strong acid to 3 of water), drop in a small lump of marble weighing about .75 gram (it need not be weighed exactly), and replace the cork and bulb tube.

Weigh accurately a lump of marble between 2 and 2.5 grams in weight; when the small piece of marble dropped into the flask is all dissolved, but not before, weigh the flask with its contents and tube attached; note that this is the weight of the flask full of CO_2 and with the liquid in it saturated with the gas.

Remove the cork and tube from the flask, and down the neck, held as nearly horizontal as possible, slide in the lump of marble, taking care that none of the liquid is splashed out of the flask and lost; at once replace the cork and tube.

When all the marble is dissolved, weigh the flask again with its contents; compare this with the sum of the weights of the flask before the last piece of marble was put in, and the weight of this piece of marble. Why is there a loss in weight? Obviously because the CO_2 has escaped which was evolved by the action of the acid on the calcium carbonate. Calculate what your result indicates as the weight of CO_2 evolved when one gram of marble is dissolved in acid, and compare with the numbers found by other students.

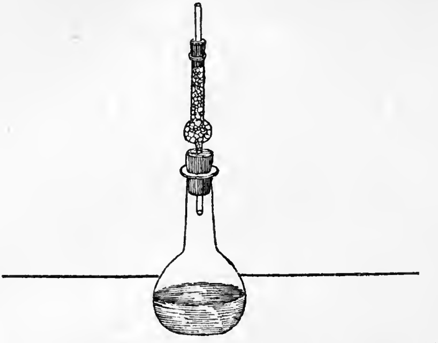
FIG. 58.—Flask with Calcium Chloride Tube attached.

The object of the bulb tube filled with calcium chloride is to prevent the CO_2 which escapes carrying water vapour along with it. The error due to this is, however, less than 1 per cent of the weight of CO_2, and may be disregarded in any but the most exact experiments; the calcium

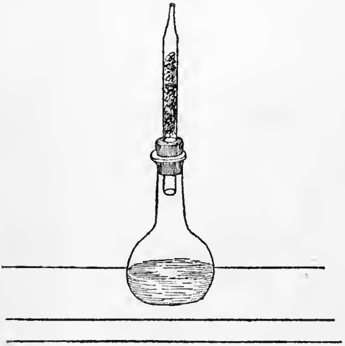
FIG. 59.—Flask with Tube attached containing Glass-Wool.

chloride tube may therefore be replaced by a piece of fairly wide glass tubing drawn out to a fine jet at the upper end and loosely packed with cotton, or better, glass-wool in the middle half (Fig. 59); the purpose of the glass-wool is to stop the finely divided spray which comes off with the gas, and whose weight is much more considerable than that of the water vapour.

EXERCISE 87.—Repeat the two preceding exercises with other carbonates, such as magnesite $MgCO_3$, witherite $BaCO_3$, dry sodium carbonate Na_2CO_3, etc.

When the carbonate is in powder or small pieces it should be wrapped in paper before being dropped into the acid; in repeating Exercise 86 the weight of this paper must of course be known, and must be taken into account in making the calculation.

Determination of Loss in Weight when a Carbonate is decomposed by Heat.—You have already tried the action of heat on various carbonates in Exercise 82 (*a*), and have found that some of them evolved carbon dioxide when heated; the complete decomposition of calcium carbonate by heat requires a high temperature such as cannot be obtained with an ordinary Bunsen burner, unless some arrangement be used to diminish the great loss of heat by radiation and convection, which occurs when a crucible is heated over a flame in the usual manner. The best furnace for the purpose is that of Hempel, in which the heat of the flame is thoroughly utilised before the hot gases are allowed to enter the short chimney; the simpler construction of furnace made by Griffin fails to give the same high temperature as is reached even with a small Bunsen burner in the Hempel furnace, but with this a gram of calcium carbonate can be decomposed in about twenty minutes. In place of using either of these furnaces the crucible may be heated with the flame of a foot-blowpipe, which effects the decomposition in from ten to fifteen minutes.

EXERCISE 88.—Weigh a porcelain crucible and lid; put in it from 1 to 1.5 gram of calcium carbonate either as powdered marble or precipitated chalk; the latter is somewhat more readily decomposed, but is not as a rule quite pure; weigh the crucible and its contents exactly, then heat strongly by one of the methods mentioned above for fifteen minutes; allow to cool and weigh; heat again for ten minutes and repeat until the weight becomes constant.

Calculate the weight of CO_2 evolved on heating one gram of calcium carbonate, and compare with the results obtained in Exercises 85 and 86.

CHAPTER XXI

CHEMICAL ANALYSIS

THE object of Qualitative Chemical Analysis is to determine the nature and identity of substances by examining their properties; but since the obvious properties, such as colour and form, are not usually of sufficient definiteness to enable us to pronounce at once upon the nature of whatever we have to investigate, recourse must be had to other properties which differ more from one substance to another and are of wider range of variation than those of colour and form; we must then examine not only the general appearance of the substance, but also its solubility in water and other liquids, its behaviour when heated, and—the most valuable means of all—its chemical behaviour under various conditions of experiment.

Chemical analysis includes within its scope the investigation of every substance, organic or inorganic, found upon the earth, and includes a multitude of problems of the greatest difficulty and complexity; it is therefore only a very small portion of this wide province upon which we can enter here, but the methods used are similar in character to some of those employed in more difficult tasks, and will only require to be extended and amplified, not reversed or thrown aside, when entering upon a less restricted field of operation.

The substances to which we shall here confine our attention are the salts formed by the combination of the four acids—carbonic, nitric, sulphuric, and hydrochloric—with the following bases:—the oxides of lead, copper, iron, zinc, calcium, and potassium; and ammonia. These salts have now to be prepared and their properties and chemical behaviour examined;

they will be taken first in groups arranged under the base contained in them.

Lead

The metal Lead is easily recognised by its properties, it is very heavy, has a bluish-gray metallic lustre, is easily hammered into thin sheets, and is so soft as to leave a mark upon paper when drawn over a piece. Take a piece of lead and examine these properties for yourself, then prepare the four salts of lead as described in the following exercises.

Lead Carbonate is not readily prepared in the laboratory in the manner used on the large scale in the manufacture of white-lead, which you may regard as an impure carbonate of lead; the manufacturing method consists in exposing sheets of lead to the joint action of the air, of carbon dioxide and water, and of acetic acid, but the slowness of the process makes it quite unsuited for laboratory purposes. The two methods we shall try depend the first on the reaction between lead oxide and carbonic acid, the second on double decomposition between a soluble salt of lead and a soluble carbonate of some other metal.

EXERCISE 89.—(*a*) Put about 5 grams of powdered litharge, PbO, in a mortar and rub it into a thin paste with a little water: wash the contents of the mortar into a small beaker, filling this two-thirds full; set up an apparatus for evolving CO_2, and bubble the gas through the contents of the beaker for at least ten minutes; while this is going on you may do the next part of the exercise.

After passing the gas for some time, filter the contents of the beaker, and dry a portion of the solid residue in the way shown in Fig. 27: put other small portions of the residue in three test-tubes, and try the action of hydrochloric, of sulphuric, and of nitric acid upon the substance obtained. Has the lead oxide been converted into carbonate? Work out the equation.

(*b*) Put about 20 cb.c. of a solution of lead nitrate in a beaker and add to it solution of sodium carbonate until the amount of the precipitate which is formed does not increase; the best way to discover when this point has been reached is to let the precipitate settle and then to pour a drop or two of the sodium carbonate solution down the side of the beaker; it is then easy to see whether any more precipitate is produced. Filter the contents of the beaker.

While this is being done, work out the equation for the reaction which has occurred; does this show that anything else is formed besides lead carbonate, and if so, where is this substance? It is dissolved in the water, and therefore to obtain the lead carbonate in a state of purity it is necessary to wash the precipitate with clean water poured over it from a test-tube, or, better, directed on to it from a wash-bottle (Fig. 60).

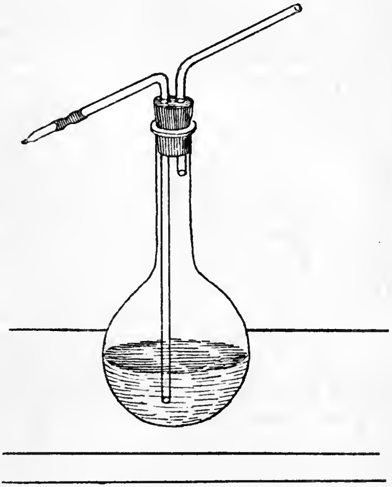

FIG. 60.—Wash-Bottle.

Dry one portion of the washed precipitate, and on other portions (which need not be dried) try the action of dilute hydrochloric, sulphuric, and nitric acids.

(*c*) Heat a portion of the dried lead carbonate strongly in an ignition tube.

(*d*) Place a little of the dried lead carbonate in a small cavity scooped in charcoal, and heat in the blowpipe flame; remove the metallic globules obtained, and try whether they are malleable and soft enough to mark paper.

(*e*) Draw up a statement of the properties of lead carbonate, mentioning its appearance, solubility, and insolubility in water, action of acids, and the result of heating it in an ignition tube, or on charcoal in the blowpipe flame.

Lead Nitrate can be obtained by acting with nitric acid upon metallic lead, the oxide, or the carbonate; you have already prepared it from the oxide in Exercise 52, and it will be sufficient now to prepare some from the metal itself.

Exercise 90.—(*a*) Cut a piece of lead into thin shavings, and weigh about 10 grams of these into a beaker: fill a test-tube half full of strong nitric acid, fill up with water, and then pour the mixture over the lead. Put the beaker in a fume cupboard and heat gently to start the reaction, and again when it slackens; add a test-tube full of water and heat to the boiling-point, when the lead should dissolve completely, and if any white insoluble residue is left it is tin oxide from tin present in the lead used; pour the clear liquid whilst still hot into another beaker and leave to crystallise. Meanwhile make the following experiments with any sample of lead nitrate that can easily be obtained:—

(*b*) Heat some lead nitrate in an ignition tube.

(*c*) Heat powdered lead nitrate on charcoal in the blowpipe flame; the first effect noticed is due to the fact that it is a nitrate you are examining (see p. 148), but on further heating metallic globules will be obtained, which on examination can be shown to be lead, and thus prove the presence of that metal in the salt taken.

(*d*) Draw up a list of the properties of lead nitrate.

Lead Sulphate is not easily got by the action of sulphuric acid either on the metal or its oxide, from the reason that the sulphate is nearly insoluble in water, and the first portion of this substance formed therefore acts as a protective coating and prevents further action of the acid. In concentrated sulphuric acid, lead sulphate is, however, fairly soluble, and accordingly the strong acid is not without action on the metal. The easiest way of preparing the sulphate is by mixing a solution of a salt of lead with one of a sulphate of some other metal.

Exercise 91.—(*a*) Put some thin shavings of lead in a test-tube, add some concentrated sulphuric acid, and heat carefully in the Bunsen flame: does any action show itself? After allowing the test-tube to cool in the air for some minutes, hold it under the tap with the water turned full on, and when the acid is quite cool (but not before, as otherwise an accident will occur) pour it into a beaker of cold water. The white precipitate is lead sulphate.

(*b*) Put about 20 cb.c. of a solution of lead nitrate in a beaker and add dilute sulphuric acid in excess, that is until more of the acid has been added than is needed to convert the whole of the lead nitrate in the solution into sulphate (see Exercise 89, *b*). Filter the precipitate, and wash it while still on the filter paper, supported in the funnel; then dry the precipitate.

(*c*) Put some lead sulphate in an ignition tube and heat.

(*d*) Heat some lead sulphate on charcoal in the blowpipe flame; then try a mixture of lead sulphate with dry sodium carbonate.

(*e*) Try whether you can dissolve lead sulphate in hydrochloric or nitric acid.

(*f*) Draw up a list of such properties of lead sulphate as you have examined.

Lead Chloride is most readily prepared, like the sulphate, by double decomposition; it is, however, somewhat more soluble in water than the sulphate, and in hot water dissolves to a considerable extent.

EXERCISE 92.—(*a*) Heat some lead shavings with hydrochloric acid in a test-tube.

(*b*) Boil some litharge, PbO, with dilute hydrochloric acid and filter whilst still hot; allow the clear filtrate to cool and then examine it for crystals of lead chloride.

(*c*) Put 20 cb.c. of lead nitrate solution in a beaker and add dilute hydrochloric acid in excess; filter and wash the precipitate; dry a portion of it, and in the meantime go on to the next experiment.

(*d*) Put a little of the precipitated lead chloride in a test-tube, fill this two-thirds with water, and heat to boiling; if the chloride does not dissolve completely, pour some of it away, add more water and boil again; when solution is complete let the liquid cool.

(*e*) Heat lead chloride in an ignition tube.

(*f*) Heat lead chloride on charcoal on the blowpipe flame; then treat similarly a mixture of lead chloride and dry sodium carbonate.

(*g*) Draw up a list of properties.

Tests for Lead

We must now select certain reactions, from those just investigated, as being suitable for use as tests by which to discover whether an unknown substance contains lead. The best are the following :—

(i.) A solid substance containing lead if mixed with dry Na_2CO_3 and heated on charcoal in the blowpipe flame gives beads of metallic lead, recognised by their malleability and power of marking paper.

(ii.) If a clean platinum wire (which should be mounted in a glass handle in the way shown in Fig. 61) be used to hold a little of a lead salt in the Bunsen flame, the flame will be coloured a livid blue; this coloration is most distinct in the case of the chloride, and it is best to moisten the substance with hydrochloric acid before holding it in the flame.

FIG. 61.—Platinum Wire mounted in Glass Handle.

(iii.) **HCl** added to a solution containing a salt of lead gives a white precipitate of $PbCl_2$ which is soluble in hot water.

(iv.) **H_2SO_4** gives a white precipitate of $PbSO_4$, which is insoluble in hot water and also in HCl and HNO_3.

(v.) Another important test for lead depends on the properties of its sulphide, which is insoluble in water, but is dissolved by hot dilute nitric acid and converted by it into lead nitrate; the test then is as follows :—

H_2S gives a black precipitate, which, if filtered and washed, can be dissolved by boiling with dilute nitric acid; this solution gives a white precipitate with dilute sulphuric acid.

Copper

The Metal Copper is easily recognised by its red colour; it is not so heavy as lead, and is much less easily fusible; when heated in air the metal becomes oxidised on the surface, and is converted more or less completely into the black oxide CuO.

EXERCISE 93.—Examine some copper turnings; heat a weighed quantity in a porcelain crucible, noticing any change in appearance or alteration in weight.

Copper Carbonate is found in nature as a mineral malachite, which is, however, of somewhat more complex composition than the simple carbonate $CuCO_3$; it is most easily prepared by double decomposition between a copper salt and a carbonate of some other metal.

EXERCISE 94.—(*a*) Put 25 cb.c. of a solution of copper sulphate in a beaker and add sodium carbonate solution in excess; filter and wash the precipitate; dry a portion of this and use the undried portion for (*b*).

(*b*) Try the action of acids on the carbonate.

(*c*) Heat some dried copper carbonate in an ignition tube.

(*d*) Heat some of the dried salt on charcoal in the blowpipe flame and examine the residue for metallic copper; this is best done by cutting out with a knife the portion of charcoal containing the fused mass and pressing this into powder in a mortar, afterwards washing away the light particles of charcoal from any heavier ones of metallic copper by means of a gentle stream of water.

If you are unsuccessful in obtaining metallic copper from its carbonate by this method, repeat the experiment, using a mixture of copper carbonate with dry sodium carbonate and a little potassium oxalate; the latter salt is added to assist the reducing action of the flame and charcoal.

(*e*) Hold a little copper carbonate on a loop of platinum wire in the Bunsen flame; then moisten the residue on the wire with a drop of hydrochloric acid and again hold in the flame: notice the effect in each case.

(*f*) Heat another loop of platinum wire in the Bunsen flame: dip it into some powdered borax and again heat, repeating, if necessary, until the loop is filled by a clear bead of molten borax: let this cool, and then pick up with it a little copper carbonate, and again heat the bead either in the upper oxidising portion of the Bunsen flame, or in the oxidising blowpipe flame; note the colour of the borax bead when hot, and also when cold; if the colour is too deep to be clearly distinguished, this can be remedied by heating the bead until it melts, shaking off the greater part of the borax with a sharp jerk of the hand, and then picking up some more borax with the wire and heating until the whole fuses to form a fresh bead; if the colour is too faint, more of the copper carbonate must be melted into the bead.

When a satisfactory bead has been obtained, and its colour after being heated in the oxidising flame carefully noted, it should be heated again, this time in the reducing flame of the blowpipe, and the colour of the bead noted.

The colours are due to the formation of borate of copper (borax is a borate of sodium); and the different colours of the bead according to the flame, whether oxidising or reducing, in which it has been tested are due to the difference in colour between cupric borate and cuprous oxide; the latter substance being formed by the action of the reducing flame.

Copper Nitrate is a blue crystalline solid easily soluble in water; it has already been prepared by the action of nitric acid on metallic copper (Exercise 50 (vi.)), and can also be readily obtained by dissolving either the oxide or the carbonate in the same acid.

EXERCISE 95.—(*a*) Dissolve about 5 grams of copper oxide in hot dilute nitric acid, evaporate to a small bulk and then allow the liquid to cool.

(*b*) Heat some copper nitrate in an ignition tube; the black residue is copper oxide: what are the red fumes evolved, and of what are they an indication?

(*c*) Heat copper nitrate on charcoal; then treat in the same way a mixture of copper nitrate with several times its bulk of a mixture of sodium carbonate and potassium oxalate; examine the residue for metallic copper as in Exercise 94 (*d*).

(*a*) Test copper nitrate by holding it on platinum wire in the Bunsen flame; moisten the residue on the wire with hydrochloric acid and heat again.

(*e*) Prepare a clean borax bead and use it to test some of the copper nitrate.

(*f*) Prepare a solution of copper nitrate in water and pass H_2S gas into this; filter the precipitate of copper sulphide and wash it, then take some of it on a spatula, place it in a test-tube, add dilute nitric acid and boil. Does the precipitate dissolve?

(*g*) Put some more solution of copper nitrate in a test-tube; in another test-tube put a few drops of ammonia and then fill up nearly to the top with water and shake; now add a little of this very dilute ammonia to the solution of copper nitrate: what happens? The precipitate is copper hydrate $Cu(OH)_2$. Add to it some strong solution of ammonia and shake: what happens is denoted by saying that the precipitate of copper hydrate dissolves in the excess of ammonia, forming a dark blue solution.

Copper Sulphate is a blue crystalline solid, commonly known as blue vitriol, not unlike the nitrate in appearance; it has already been prepared from the metal and sulphuric acid (Exercise 71), and can also be readily obtained by the action of the same acid on the oxide or carbonate.

EXERCISE 96.—(*a*) Dissolve about 5 grams of copper oxide in dilute sulphuric acid, evaporate to a small bulk and then allow the liquid to cool.

(*b*) Heat copper sulphate in an ignition tube: explain what happens.

(*c*) to (*g*) Repeat experiments (*c*) to (*g*) of Exercise 95, using copper sulphate in place of copper nitrate.

Copper Chloride is not easily made by the action of hydrochloric acid on copper, as that acid does not readily attack the metal; it can be made by dissolving the oxide or carbonate in hydrochloric acid, or by acting with chlorine upon metallic copper. It is generally met with as a green solid easily soluble in water, but these green crystals contain water of crystallisation like the blue ones of the nitrate and sulphate.

EXERCISE 97.—(*a*) Dissolve about 5 grams of copper oxide in dilute hydrochloric acid, evaporate the liquid to a small bulk and then allow to cool.

(*b*) to (*g*) Repeat experiments (*b*) to (*g*) of Exercise 95.

Tests for Copper

(i.) The salts of copper are mostly blue or green in colour, but the anhydrous salts are often colourless.

(ii.) Heated on platinum wire in the Bunsen flame the salts of copper colour the flame either green, or blue followed by green; the latter coloration is observed in the case of the chloride, or of any other salt after being moistened with hydrochloric acid.

(iii.) Salts of copper melted in a borax bead colour it blue after being heated in the oxidising flame; in the reducing flame the bead is either colourless, or red and opaque.

(iv.) Heated on charcoal in the blowpipe flame, the salts of copper are reduced only with difficulty; the reduction is made easier by mixing sodium carbonate and potassium oxalate with the salt.

(v.) **H_2S** with a solution of a copper salt gives a black precipitate of CuS, soluble in hot nitric acid to a green solution, which becomes blue when diluted with water, and intensely blue on addition of ammonia.

(vi.) **NH_3** in solution added to one of a copper salt gives a greenish-blue precipitate if the solution of ammonia is very weak; the precipitate dissolves easily in an excess of ammonia to form a dark blue solution.

Iron

Metallic Iron has a gray lustre, is fusible only with great difficulty, and is distinguished very easily from other metals by the readiness with which it is attracted by a magnet: iron forms two distinct series of salts—the *ferrous* salts in which the iron atom may be regarded as divalent or equivalent to two atoms of hydrogen, and the *ferric* salts in which the atom of iron is to be considered as trivalent or equivalent to three atoms of hydrogen. Thus ferrous chloride is $FeCl_2$, and ferric chloride $FeCl_3$; ferrous sulphate is $FeSO_4$, and ferric sulphate $Fe_2(SO_4)_3$; and ferrous oxide is FeO, while ferric oxide is Fe_2O_3.

In consequence of this fact the number of compounds of iron which should be described is double that required in the case of the other metals we are taking in this book, but since some of these iron compounds are seldom met with, they will be omitted and only the most important ones described.

Ferrous Carbonate $FeCO_3$ occurs in very large quantity in nature as spathic iron ore, and mixed with clay as clay ironstone; it constitutes the most important source from which both the metal iron and its derivatives are obtained.

EXERCISE 98.—(*a*) Heat some powdered spathose iron in an ignition tube.

(*b*) Try the action of dilute acids on ferrous carbonate; the solution obtained is in each case one of a ferrous salt, but oxidises readily in air to a ferric salt, especially if warm.

(*c*) Mix some powdered ferrous carbonate with sodium carbonate and heat strongly on charcoal in the reducing flame of the blowpipe; in this case, as with copper, addition of potassium oxalate makes it easier to effect the reduction. Remove the fused residue from the charcoal to a mortar, press it into powder with the pestle and wash away the lighter particles of charcoal; test the fine powder which is left with a magnet (the blade of a knife may conveniently be magnetised for this purpose) and notice whether any of it is attracted.

(*d*) Heat a very small quantity of the substance in a borax bead, first in the reducing flame and afterwards in the oxidising flame of the blowpipe.

Ferrous Chloride $FeCl_2$ is formed when either metallic iron or ferrous carbonate $FeCO_3$ is dissolved in hydrochloric acid; from this solution green crystals of the chloride can be obtained which contain water of crystallisation.

EXERCISE 99.—(*a*) Dissolve about 5 grams of iron in dilute hydrochloric acid and concentrate the liquid by evaporation until crystals of ferrous chloride are obtained on cooling.

(*b*) Dissolve some ferrous carbonate in dilute hydrochloric acid and use this solution for the tests (*e*) to (*g*).

(*c*) and (*d*) Repeat experiments (*c*) and (*d*) in the preceding exercise, using ferrous chloride in place of the carbonate.

(*e*) Pass H_2S gas into a solution of $FeCl_2$ which has been made slightly acid with hydrochloric acid; what happens? Now add sufficient ammonia to make the liquid alkaline, and pass more H_2S to complete the precipitation; the black precipitate is ferrous sulphide FeS.

Filter this and wash it well with water: then try whether it is soluble in acids; why was no precipitate obtained with H_2S before adding the ammonia?

(*f*) Add ammonia to another portion of the solution of ferrous chloride; note the appearance of the precipitate and its changes; these latter are due to oxidation by the air.

Filter the liquid; does the filtrate contain any iron still in solution? To test this, pass H_2S gas through it; if there is any black precipitate that will show that the ammonia does not precipitate the iron completely from a solution of ferrous chloride.

Wash the precipitate with water; test its solubility in dilute acids.

(*g*) Repeat experiment (*f*), adding a solution of sodium hydrate NaOH in place of one of ammonia. The composition of the precipitate formed is the same in both cases, and it is generally found that a solution of ammonia behaves as if it contained a substance $NH_4.OH$ or AmOH (in which Am is an abbreviation for NH_4), similar to NaOH or KOH in composition; this substance is often spoken of by the name ammonium hydrate.

Ferric Chloride $FeCl_3$ is formed when iron is acted upon with chlorine, and when a solution of ferrous chloride is treated with chlorine; it is by the second of these two methods that we shall prepare it, and the chlorine may for this purpose be most conveniently liberated in the solution itself by oxidising hydrochloric acid with nitric acid; this mixture of the two acids is known as *aqua regia*, a name due to the power of dissolving gold which it possesses in consequence of the formation of chlorine when the mixture is gently heated, and the combination of this with the gold.

EXERCISE 100.—(*a*) To a solution of ferrous chloride add a small quantity of *aqua regia* prepared in a test-tube by mixing one volume of strong nitric acid with three volumes of concentrated hydrochloric acid; boil the liquid for several minutes. The solution now contains ferric chloride $FeCl_3$; is there any change in its colour?

(*b*) Take some of the solution of ferric chloride and pass H_2S gas through it; what happens? After the liquid is saturated with the gas, and smells strongly of it, heat it to boiling and filter; if the filtrate is not clear, run it through the filter paper a second or third time; examine the colour of the filtrate; is it like that of a ferrous or of a ferric salt? Explain the action of the H_2S.

(*c*) To some solution of ferric chloride add ammonia in excess; what is the colour of the precipitate? It is ferric hydrate $Fe(OH)_3$. Filter the liquid, and test the filtrate by adding H_2S to find out whether it still contains any iron.

(*d*) Examine some solid ferric chloride, noticing its appearance and colour; dissolve a little in water, and with this solution repeat experiments (*b*) and (*c*).

(*e*) Heat some solid ferric chloride in an ignition tube.

(*f*) Mix some solid ferric chloride with dry Na_2CO_3 and potassium oxalate, and heat on charcoal in the reducing flame of the blowpipe.

(*g*) Examine the behaviour of ferric chloride when fused in a borax bead, and heated first in the oxidising and then in the reducing flame of the blowpipe.

(*h*) Ferric chloride can be reduced to ferrous chloride by the action of "nascent" hydrogen, that is, by the action of hydrogen which is evolved in contact with the solution to be reduced; any reagents capable of producing hydrogen readily may be used, such as zinc and dilute sulphuric acid, but the best to use in this case are iron and dilute hydrochloric acid, as there will then be no contamination of the liquid.

Put some solution of ferric chloride in a small flask, add dilute hydrochloric acid and several grams of iron wire; notice the change in colour of the solution as the reduction proceeds, and when it is complete test portions of the liquid to find out whether it contains a ferrous or a ferric salt.

Ferrous Sulphate $FeSO_4$ is easily made by dissolving iron or ferrous carbonate in dilute sulphuric acid; when anhydrous it is white, but is generally found in green crystals, having the composition $FeSO_4 . 7H_2O$.

(*a*) Dissolve about 5 grams of iron in dilute sulphuric acid, filter whilst still hot and before the last portion of iron has been dissolved; cool the filtrate quickly to prevent as far as possible oxidation by the air.

(*b*) to (*e*) Repeat experiments (*c*) to (*f*) of those done with ferrous chloride in Exercise 99.

(*f*) To some solution of ferrous sulphate add dilute sulphuric and then some strong nitric acid: boil the mixture; the ferrous sulphate is oxidised to ferric sulphate according to the equation

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

the oxygen required being supplied by the nitric acid.

Notice the changes of colour during the process.

Ferric Sulphate $Fe_2(SO_4)_3$ is formed by the oxidation of ferrous sulphate as described in the last experiment; when anhydrous it is white in colour, but also occurs in reddish crystals containing water of crystallisation.

A well-known substance is *Iron Alum*, formed by bringing together ferric sulphate and potassium sulphate: it crystallises from water in pink crystals closely resembling those of ordinary alum, and possessing the composition $FeK(SO_4)_2 . 12H_2O$.

EXERCISE 101.—(*a*) Prepare some iron alum by putting 6 grams of green vitriol and 2 grams of potassium sulphate in a dish: adding 10 cc. of dilute sulphuric acid, 10 cc. of dilute nitric acid, and enough water to fill the dish two-thirds full; then heating the dish until the contents have boiled vigorously for several minutes, and allowing the liquid to cool.

(*b*) to (*h*) Use iron alum for tests (*b*) to (*h*) given under Ferric Chloride in Exercise 100.

Tests for Iron

A. Tests for Ferrous Salts

(i.) **H_2S** passed into an acid solution gives no precipitate.

(ii.) **Am_2S**, or **H_2S** in an alkaline solution, gives a black precipitate of FeS, soluble in dilute HCl.

(iii.) **Ammonia** gives a dirty white precipitate of $Fe(OH)_2$, which rapidly oxidises in the air and is converted into brown $Fe(OH)_3$. Ammonia does not completely precipitate iron from a solution of a ferrous salt, since the ferrous hydrate $Fe(OH)_2$ is somewhat soluble in water containing dissolved ammonia.

(iv.) **Potassium Thiocyanate KCNS** gives no coloration with a solution of a ferrous salt, if this contains no ferric salt.

(v.) **Potassium Ferrocyanide $K_4Fe(CN)_6$** gives a light-blue precipitate with a solution of a ferrous salt.

B. Tests for Ferric Salts

(i.) **H_2S** passed into an acid solution gives a milky-white precipitate of sulphur, which is often so finely divided that it passes through the pores of a filter paper.

(ii.) **Am_2S** or **H_2S in an Alkaline Solution** gives a black precipitate of FeS mixed with free sulphur; when this precipitate is treated with a dilute acid the FeS dissolves, but the free sulphur remains.

(iii.) **Ammonia** gives a reddish-brown precipitate of ferric hydrate $Fe(OH)_3$; this is insoluble in water containing ammonia in solution, and therefore iron can be completely precipitated by ammonia from a solution which contains iron in the ferric state.

(iv.) **Potassium Thiocyanate KCNS** gives a dark red coloration with a solution of a ferric salt.

(v.) **Potassium Ferrocyanide $K_4Fe(CN)_6$** gives a dark blue precipitate of Prussian blue with a solution of a ferric salt.

C. Tests applicable both to Ferrous and Ferric Salts

(i.) Heated in a borax bead, either a ferrous or ferric salt gives a green bead in the reducing flame, and one which is either colourless or tinged with yellow in the oxidising flame.

(ii.) Heated with Na_2CO_3 on charcoal in the reducing flame of the blowpipe, both ferrous and ferric salts give a residue containing magnetic particles.

Zinc

The Metal Zinc is a bluish-white metal, not very heavy; it oxidises on the surface when exposed for a long time to air, but the oxidation is only superficial under ordinary conditions; if, however, the metal be strongly heated in air it takes fire and burns, forming a white oxide ZnO.

Zinc Carbonate $ZnCO_3$ occurs as the mineral calamine, a fairly abundant ore of zinc; it can be prepared artificially by adding a solution of sodium carbonate to one of a zinc salt.

EXERCISE 102.—(*a*) Prepare some zinc carbonate by precipitating a solution of zinc sulphate with one of sodium carbonate; filter and wash the precipitate, and dry part of it.

(*b*) Examine the action of dilute acids on zinc carbonate.

(*c*) Heat some dry zinc carbonate strongly in an ignition tube, noticing the colour of the residue in the tube when hot, and also after it has become cold.

(*d*) Heat some zinc carbonate on charcoal in the reducing flame; what appearance does the heated mass present? Moisten the residue with a few drops of a solution of cobalt nitrate and heat again; what is now the colour of the mass?

Zinc Chloride $ZnCl_2$ can be made by dissolving the metal, its oxide or carbonate, in hydrochloric acid; it is a white solid in the anhydrous state, in which it is generally seen, but also forms colourless crystals containing water of crystallisation; the salt is very soluble in water, and the anhydrous compound takes up water with great eagerness.

EXERCISE 103.—(*a*) Dissolve about 5 grams of metallic zinc in a mixture of 20 cb.c. of concentrated HCl with the same bulk of water, and evaporate the liquid on the sand-bath until it becomes solid on cooling.

(*b*) Heat some solid $ZnCl_2$ in an ignition tube.

(*c*) Heat some of the chloride on charcoal in the blowpipe flame; moisten the residue with a few drops of a solution of cobalt nitrate and heat again.

(*d*) To some solution of $ZnCl_2$ in a test-tube add a little dilute HCl, and then pass H_2S gas through the solution.

(*e*) Make another portion of the solution alkaline by adding ammonia (does this give any precipitate?) and then pass H_2S; the precipitate is zinc sulphide ZnS: notice its colour and then examine whether it is soluble in dilute HCl.

Zinc Sulphate $ZnSO_4$ is easily made by the action of sulphuric acid upon the metal, its oxide, or carbonate. It is generally met with in colourless crystals of the composition $ZnSO_4 . 7H_2O$, which are easily soluble in water.

EXERCISE 104.—(*a*) Zinc sulphate may be prepared by dissolving the metal in dilute sulphuric acid, as already done in Exercise 45.

(*b*) to (*e*) Repeat experiments (*b*) to (*e*) of the preceding exercise, using the sulphate in place of the chloride.

Zinc Nitrate $Zn(NO_3)_2$ can be prepared by dissolving zinc, the oxide or carbonate, in nitric acid.

EXERCISE 105.—(*a*) Put about 40 cb.c. of dilute nitric acid in a beaker and add zinc oxide until some remains undissolved (from 5 to 10 grams will probably be required according to the strength of the acid); filter the liquid and evaporate the clear filtrate to about half the bulk, then allow to cool.

(*b*) to (*e*) Repeat experiments (*b*) to (*e*) of Exercise 104, using the nitrate in place of the chloride.

Tests for Zinc

(i.) **H_2S** added to an acid solution of a zinc salt produces no precipitate.

(ii.) **Am_2S or H_2S in an Alkaline Solution** of a zinc salt produces a white precipitate of ZnS, which is easily soluble in dilute HCl. This precipitate is liable to be discoloured by the presence of traces of other metals.

(iii.) Some zinc salts when heated on charcoal in the blowpipe flame give a residue of zinc oxide, showing brilliant luminosity when strongly ignited; if such a residue is moistened with a few drops of a solution of cobalt nitrate and again heated it becomes green in colour.

The test is most readily made applicable to all zinc salts by employing a mixture of the salt with dry Na_2CO_3: a certain amount of zinc oxide is then left on the charcoal as an incrustation round the heated cavity; this incrustation is yellow when hot and white when cold, but becomes bright green in colour if moistened with cobalt nitrate solution and re-heated.

Calcium

The metal Calcium is itself rarely seen, although its compounds are very abundant; the chief of these is the carbonate $CaCO_3$, which occurs in very large quantity as marble, limestone, chalk, and other forms.

Calcium Carbonate $CaCO_3$ is the source from which most of the compounds of calcium are prepared: it is insoluble in water, but easily soluble in dilute acids, with the exception of sulphuric; a high temperature is required to effect its decomposition into lime CaO and carbon dioxide.

EXERCISE 106.—(*a*) Heat some calcium carbonate in an ignition tube; is there any change?

(*b*) Heat some calcium carbonate on charcoal in the blowpipe flame; moisten the residue with water and place it on a piece of red litmus paper.

(*c*) Hold some calcium carbonate on a clean piece of platinum wire in the Bunsen flame: repeat the experiment, but first moisten the platinum wire with strong HCl before using it to take up the calcium carbonate.

(*d*) Try the action of dilute acids on calcium carbonate.

Calcium Chloride $CaCl_2$ is easily made by dissolving either lime CaO or calcium carbonate $CaCO_3$ in hydrochloric acid; when anhydrous it is a white solid, which is largely used to dry gases on account of its great attraction for water; it is also found in crystals of the composition $CaCl_2 . 7H_2O$, which are very soluble in water.

EXERCISE 107.—(*a*) Dissolve 10 grams of calcium carbonate in 30 cb.c. of strong HCl mixed with an equal bulk of water; evaporate the liquid on the sand-bath until no more water is expelled, and then allow it to cool.

(*b*) Put a piece of anhydrous calcium chloride in a beaker or dish and expose it to damp air.

(*c*) Heat some calcium chloride in an ignition tube.

(*d*) Heat some of the chloride on charcoal in the blowpipe flame.

(*e*) Hold a little of the chloride on a clean platinum wire in the Bunsen flame.

(*f*) Make a solution of calcium chloride and add to this in turn HCl and H_2S: boil the liquid until it no longer smells of H_2S, then add ammonia, and next pass H_2S gas into the alkaline solution.

(*g*) To a fresh solution of $CaCl_2$, or to that used in (*f*), add a solution of ammonium carbonate; filter the precipitate and wash it; take up a portion of this precipitate on platinum wire moistened with dilute HCl, and hold it in the Bunsen flame.

Calcium Sulphate $CaSO_4$ occurs in nature as alabaster and gypsum, both of which contain water of crystallisation; when most of this water is expelled by heat a useful product known as plaster of Paris is obtained. Calcium sulphate dissolves only to a slight extent in water, one part of the sulphate requiring about 500 of water to dissolve it.

EXERCISE 108.—(*a*) to (*c*) Repeat experiments (*a*) to (*c*) of Exercise 107, using calcium sulphate in place of the carbonate.

(*d*) Shake up some calcium sulphate with water in a test-tube and filter; to the clear filtrate add solution of ammonium carbonate, filter and wash the precipitate, then test it on platinum wire in the Bunsen flame.

Calcium Nitrate $Ca(NO_3)_2$ is prepared by dissolving the oxide or carbonate in dilute nitric acid: it is readily soluble in water.

EXERCISE 109.—(*a*) Dissolve 10 grams of calcium carbonate in 50 cb.c. of dilute nitric acid: filter if necessary, and evaporate the clear filtrate to about half the bulk, then put it in a beaker to cool.

(*b*) to (*f*) Repeat experiments (*c*) to (*g*) of Exercise 107, using calcium nitrate in place of the chloride.

Tests for Calcium

(i.) A calcium salt held on platinum wire in the Bunsen flame colours the flame orange-red; this reaction is best shown by the chloride and nitrate, and with the other salts is made more evident if the platinum wire is first dipped in hydrochloric acid before using it to take up the salt.

(ii.) A solution of a calcium salt gives no precipitate either with HCl or with H_2S whether in acid or alkaline solution; ammonia also fails to give a precipitate.

(iii.) **Ammonium Carbonate Am_2CO_3** gives with a solution if a calcium salt a white precipitate of $CaCO_3$, which is easily soluble in dilute HCl or dilute HNO_3.

That this precipitate contains calcium is best shown by filtering it, and after washing testing it by (i.).

(iv.) Dilute **H_2SO_4** gives a white precipitate of $CaSO_4$ with a solution of a calcium salt, provided this is not too weak.

The precipitate appears more readily if the liquid is boiled.

Potassium

The chief source of potassium compounds is now the deposits at Stassfurt in Germany, where large quantities of the chloride KCl and the sulphate K_2SO_4 are found.

Potassium Carbonate K_2CO_3 used formerly to be made by treating wood-ashes with water, which dissolved the soluble K_2CO_3 present in the ashes (hence the name potash); it is now generally made from the chloride by a process too complicated for description in this book.

EXERCISE 110.—(*a*) Examine some potassium carbonate: test its solubility in water, the action of the solution on litmus, and of acids on the solid carbonate.

(*b*) Heat some potassium carbonate in an ignition tube.

(*c*) Heat some of the carbonate on charcoal in the blowpipe flame.

(*d*) Hold some of the carbonate on platinum wire in the Bunsen flame, then repeat after dipping the wire in hydrochloric acid.

(*e*) To a solution of potassium carbonate add HCl, H_2S, ammonia, more H_2S, and ammonium carbonate in succession.

(*f*) To some solution of the carbonate add an equal bulk of a strong solution of sodium hydrogen tartrate and shake well; if no precipitate is formed try again, using a stronger solution of the potassium carbonate.

Pour part of the liquid and precipitate into another test-tube: fill this nearly full with water and shake; what happens?

Potassium Chloride is found in large quantity at Stassfurt, both as the pure chloride KCl, and in loose combination with other salts.

EXERCISE 111.—(*a*) Examine some potassium chloride and test its solubility in water.

(*b*) to (*f*) Repeat the corresponding experiments of the preceding exercise, using potassium chloride in place of the carbonate.

Potassium Sulphate K_2SO_4 is found at Stassfurt in large deposits of kainite; it is much less easily soluble in water than the carbonate and chloride, from either of which the sulphate can be obtained by the action of sulphuric acid.

EXERCISE 112.—(*a*) Put 5 grams of potassium carbonate in a beaker and add gradually dilute sulphuric acid until *nearly* the whole of the carbonate is dissolved; filter the hot liquid, and let the filtrate cool to deposit crystals. There are two sulphates of potassium, which is it you have prepared in this experiment? How could the other one be made?

(*b*) to (*f*) Repeat experiments (*b*) to (*f*) of Exercise 110, using potassium sulphate in place of the carbonate.

Potassium Nitrate KNO_3 was formerly made by a process which is still used to some extent, depending on the slow putrefaction of organic matter containing nitrogen; it is, however, now most largely made from sodium nitrate or Chili saltpetre $NaNO_3$; this substance is found in large deposits in several parts of the west coast of South America, and is very soluble in water; when a strong solution of this sodium nitrate is mixed with potassium chloride KCl, the less soluble potassium nitrate KNO_3 is precipitated.

(*a*) Dissolve about 10 grams of KCl and 1·2 grams of $NaNO_3$ in a small quantity of hot water and cool the liquid (filtered if necessary); the crystals which are obtained consist of KNO_3, but require to be re-crystallised in order to obtain that substance in a state of purity; this is done as follows: filter the liquid, and when the crystals have been freed as completely as possible from the mother-liquor by allowing them to drain on the filter paper, wash them with a very little cold water; remove the washed crystals to a beaker, add about a test-tube full of water and heat this until it boils, adding more water if required to dissolve the crystals; when the solution cools pure KNO_3 will be deposited in crystals, which vary in size according to the rate at which the liquid has been cooled; they are purest when small, and it is therefore best to effect the cooling as rapidly as possible.

(*b*) to (*f*) Repeat experiments (*b*) to (*f*) of Exercise 110, using potassium nitrate in place of the carbonate.

Tests for Potassium

(i.) Potassium salts held on platinum wire in the Bunsen flame produce a lilac coloration; this is liable to be masked by other flame colorations, especially by the intense yellow flame caused by the presence of even a small quantity of a sodium compound; such a masked potassium flame when examined through blue glass shows a distinct crimson-red flame (the blue glass stops the yellow light of the sodium flame).

(ii.) Potassium salts heated in an ignition tube become fused.

(iii.) Potassium salts heated on charcoal in the blowpipe flame are fused and melt into the charcoal.

(iv.) A solution of a potassium salt gives no precipitate with any of the following:—HCl, H_2S in acid solution, ammonia, H_2S in alkaline solution, ammonium carbonate.

(v.) Sodium hydrogen tartrate added to a solution of a potassium salt gives, when this is strong enough, a white crystalline precipitate of potassium hydrogen tartrate.

The precipitate forms more readily when the liquid is well stirred or shaken.

Ammonium (NH_4)

The salts formed when ammonia NH_3 combines with an acid are regarded as derived from a compound radicle NH_4, to which the name ammonium is given, and for which the abbreviation Am is generally used; this radicle fills the same place in the ammonium salts as the metal K or Na in those of potassium or sodium.

The only important source of the ammonium compounds is the ammonia evolved in the distillation of coal; this is to a large extent converted at the gasworks either into ammonium chloride AmCl or the sulphate Am_2SO_4.

Ammonium Carbonate Am_2CO_3 is made by heating a mixture of powdered calcium carbonate $CaCO_3$ with ammonium chloride or sulphate; it is a white solid, which smells strongly of ammonia, and is easily soluble in water.

EXERCISE 113.—(*a*) Mix 10 grams of finely powdered marble or limestone with 5 grams of dry ammonium chloride, also in fine powder; place the mixture in a porcelain dish covered with a glass funnel and heat the dish, using a small flame, and having the dish supported on wire gauze or on a pipe-clay triangle.

The ammonium carbonate is given off as vapour, and condenses for the most part on the sides of the glass funnel; this process is called *sublimation.*

(*b*) Notice the appearance, smell, and solubility in water of ammonium carbonate; try the action of dilute acids upon it.

(*c*) Heat some ammonium carbonate in an ignition tube.

(*d*) Put a little ammonium carbonate in a porcelain dish and heat it strongly with the Bunsen flame, the dish being supported on a pipe-clay triangle. Is there any residue left in the dish?

(*e*) Put some solution of ammonium carbonate in a test-tube and add to it an equal bulk of a solution of sodium hydrogen tartrate; shake vigorously for a minute or more. Is there any precipitate? What other solution used instead of ammonium carbonate would give a similar result?

(*f*) Put a little solution of ammonium carbonate in a test-tube, add about an equal volume of sodium hydrate solution, and heat the mixture until it is on the point of boiling; then remove the test-tube from the flame and smell the mouth. Is there any smell of ammonia? Hold a piece of red litmus paper inside the mouth of the test-tube; does it turn blue?

Ammonium Chloride AmCl is prepared by neutralising the ammonia liquor of the gasworks with hydrochloric acid, and evaporating the liquid to dryness. It can be purified either by sublimation when it is obtained as a tough fibrous mass, or by re-crystallisation from water.

EXERCISE 114.—(*a*) Examine some ammonium chloride; has it any smell?

(*b*) to (*e*) Repeat experiments (*c*) to (*f*) of the preceding exercise, using ammonium chloride in place of the carbonate.

Ammonium Sulphate Am_2SO_4 is prepared in a similar way to the chloride, using sulphuric acid in place of hydrochloric; it is purified by recrystallisation from water.

EXERCISE 115.—(*a*) to (*d*) Repeat experiments (*c*) to (*f*) of Exercise 113, using ammonium sulphate in place of the carbonate.

Ammonium Nitrate $AmNO_3$ has been prepared in Exercise 54 by neutralising ammonia with nitric acid; it is distinguished amongst the ammonium salts by undergoing complete decomposition into water and nitrous oxide when the salt is strongly heated.

EXERCISE 116.—(*a*) to (*d*) Repeat experiments (*c*) to (*f*) of Exercise 113, using ammonium nitrate in place of the carbonate.

Tests for Ammonium Salts

(i.) An ammonium salt when strongly heated in an ignition tube generally gives a white sublimate.

(ii.) An ammonium salt when strongly heated in a porcelain dish volatilises completely.

(iii.) An ammonium salt when gently heated in a test-tube with solution of sodium hydrate evolves NH_3, which can be easily recognised by its smell and by its action on red litmus paper.

(iv.) A strong solution of an ammonium salt gives a white crystalline precipitate with sodium hydrogen tartrate.

The Acids

The four acids whose salts are included in our list are carbonic, hydrochloric, sulphuric, and nitric acids; these have been examined, and tests for their salts described, in the earlier portions of this book; the tests will for convenience be recapitulated at this point.

Carbonates

The carbonates are for the most part insoluble in water; amongst those for which we have to test only the carbonates of potassium and ammonium dissolve in water.

Tests for Carbonates

(i.) A carbonate when acted upon with dilute acids evolves CO_2, which is easily detected by its property of turning lime-water milky.

Dilute HNO_3 is the best acid to use, since $PbCO_3$ is not so readily acted upon by HCl or H_2SO_4, and $CaCO_3$ does not easily react with H_2SO_4.

The gas evolved may be brought into contact with the lime-water by pouring it into another test-tube in the bottom of which the lime-water has been placed, and then shaking up the contents of this second test-tube with the thumb placed firmly over its mouth.

Chlorides

The chlorides of the metals in our list are all readily soluble in water with the exception of lead chloride, which is only slightly soluble in cold water but dissolves more easily in hot.

Tests for Chlorides

(i.) Strong H_2SO_4 poured over a chloride causes HCl to be evolved; this is recognised by its giving white fumes in the air, and by turning blue litmus paper red.

(ii.) A solid chloride when gently heated with MnO_2 and strong H_2SO_4 evolves chlorine, recognised by its smell and bleaching action upon damp litmus paper.

(iii.) A solution of a soluble chloride gives a white curdy precipitate of AgCl when a solution of silver nitrate $AgNO_3$ is added; this precipitate is insoluble in HNO_3, but readily soluble in ammonia; it darkens on exposure to light.

Sulphates

The sulphates for which we have to test are all soluble in water with the exceptions of lead sulphate $PbSO_4$, which is almost quite insoluble, and calcium sulphate $CaSO_4$, which is slightly soluble in water.

Tests for Sulphates

(i.) A solid sulphate mixed with dry Na_2CO_3 and heated on charcoal in the reducing flame, gives a residue containing sodium sulphide Na_2S. If this residue is placed on a clean silver coin and moistened with water, the coin will be found after a short time to be stained brown owing to the formation of silver sulphide Ag_2S.

(ii.) A solution of a sulphate gives a white precipitate of barium sulphate $BaSO_4$ when barium chloride $BaCl_2$ is added. This precipitate is insoluble in HCl.

Note.—If lead is present it must be removed before applying this test, as otherwise $PbCl_2$ may be precipitated and lead to a mistaken conclusion; this is effected by boiling the solution with excess of sodium carbonate and filtering off the precipitated $PbCO_3$. The clear filtrate must then be made acid with HCl before adding the $BaCl_2$, to prevent precipitation of $BaCO_3$.

Nitrates

The nitrates are soluble in water without exception; it is consequently impossible to detect the presence of a nitrate by the formation of a precipitate consisting of an insoluble nitrate, and we must rely on tests of different character.

Tests for Nitrates

(i.) A nitrate when strongly heated in an ignition tube evolves oxygen, generally along with reddish-brown fumes of oxides of nitrogen.

Potassium nitrate evolves oxygen only; ammonium nitrate evolves colourless nitrous oxide N_2O, which, like oxygen, is able to ignite a glowing splint of wood.

(ii.) A solid nitrate when gently heated in a test-tube with strong H_2SO_4, evolves vapours of HNO_3.

(iii.) A solid nitrate when gently heated with strong H_2SO_4 and a few small pieces of metallic copper, produces nitric oxide NO by the action of the nitric acid which is formed upon the copper. The nitric oxide mixing with the air in the test-tube gives reddish fumes of NO_2, best seen on looking down the length of the test-tube.

(iv.) A more delicate test for the presence of a nitrate is the following:—dissolve some of the substance to be tested in cold water in a test-tube, add a few small crystals of ferrous sulphate $FeSO_4$ and shake gently for a couple of minutes; pour off the liquid from the undissolved crystals into another test-tube, and to this add cautiously about an equal bulk of strong H_2SO_4, pouring it down the side of the test-tube so that it runs to the bottom and forms a layer under the lighter solution; if any nitrate is present a brown ring will be observed where the two layers of liquid mix.

The chemistry of this reaction is somewhat complex; nitric acid is formed, and this, acting upon some of the $FeSO_4$, produces nitric oxide; the nitric oxide dissolves in the unaltered portion of the solution of $FeSO_4$ and gives a dark brown colour to it.

SYSTEMATIC EXAMINATION OF AN UNKNOWN MIXTURE

The mixture is supposed to contain only oxides, carbonates, chlorides, sulphates, or nitrates of the following metals :—lead, copper, iron, zinc, calcium, potassium, and ammonium. The method to be used is the same, whatever number of these salts may be mixed together; it is divided into three parts :—A. Preliminary Examination. B. Examination for Bases. C. Examination for Acids.

A. Preliminary Examination

I. Note the appearance of the solid substance given for examination.

II. Examine its solubility in water and in dilute acids.

III. Heat some of the substance in an ignition tube, and note what happens. The following are some of the effects which may be observed, and the inferences to be drawn from them :—

Observation.	*Inference.*
(i.) The substance fuses, and becomes solid again on cooling.	Probably Ca or K present.
(ii.) A sublimate is formed.	Am present.
Confirmatory: Mix some of the dry substance with powdered lime and heat gently in a test-tube—NH_3 evolved.	Presence of Am confirmed.
(iii.) A gas is given off.	
(*a*) A gas in which a glowing chip burns brightly.	O from a nitrate or peroxide; or possibly N_2O from $AmNO_3$.
(*b*) Red nitrous fumes.	A nitrate present.

Observation.	*Inference.*
(iv.) The substance changes colour.	
(*a*) It is white when cold, yellow when hot.	ZnO.
(*b*) It is yellow when cold, brown when hot.	PbO.
(*c*) It is red when cold, dark brown when hot.	Fe_2O_3.

IV. Place some of the substance in a small hollow scooped in a piece of charcoal, and heat in the blowpipe flame :—

Observation.	*Inference.*
(i.) The charcoal burns brightly and rapidly.	Nitrate present.
(ii.) A white infusible mass is left, which glows brightly in the flame.	Zn or Ca present.
Confirmatory: Moisten the residue with several drops of a solution of cobalt nitrate, and heat again in the oxidising flame of the blowpipe—a green mass.	Zn present.
(iii.) Metallic globules are formed which, when removed from the charcoal, are easily flattened by pressure, and are soft enough to mark paper.	Pb present.
(iv.) The substance volatilises.	Am present.
(v.) The substance fuses and sinks into the charcoal.	K or Ca probably present.

V. Mix some of the substance with twice its bulk of dry Na_2CO_3 (if potassium oxalate or cyanide is added it will assist the reduction), and heat on charcoal in the reducing flame of the blowpipe :—

Observation.	*Inference.*
(i.) Metallic globules are formed which are malleable and make a mark on paper.	Pb present.
(ii.) The residue removed from the charcoal placed on a clean silver coin and moistened with water leaves a brown stain.	Sulphate present.

Observation.	*Inference.*
(iii.) The residue removed from the silver coin to a mortar, pressed with the pestle, and gently washed with a stream of water to remove the lighter particles :—	
(*a*) Red metallic scales left.	Cu present.
(*b*) Black particles left which are attracted by a magnet.	Fe present.
(iv.) An incrustation is formed on the charcoal around the place heated :—	
(*a*) Brown when hot, yellow when cold.	Pb present.
(*b*) Yellow when hot, white when cold.	Zn present.
Confirmatory: Moisten the incrustation with solution of cobalt nitrate, and heat again in the oxidising flame—the colour is changed to green.	Zn present.

VI. Dip a clean platinum wire (moistened with HCl) in some of the powdered substance, and hold in the Bunsen flame :—

Observation.	*Inference.*
(i.) The flame is coloured.	
(*a*) Livid blue.	Pb present.
(*b*) Bright blue with a green mantle.	Cu present.
(*c*) Orange red.	Ca present.
(*d*) Lilac, appearing red when examined through blue glass.	K present.

VII. Make a clear borax bead in a loop at the end of a clean platinum wire, take up with it a very small quantity of the powdered substance and heat the bead in the blowpipe flame :—

Observation.	*Inference.*
(*a*) The bead is greenish-blue after the oxidising flame, colourless or red and opaque after the reducing flame.	Cu present.
(*b*) The bead is colourless or yellow after the oxidising flame, green after the reducing flame.	Fe present.

B. EXAMINATION FOR BASES

Prepare a solution of the substance given, using water if this is able to effect complete solution; otherwise dilute HCl should be used, unless lead has been found in the preliminary examination, when dilute HNO_3 is to be taken.

Take half a test-tube full of the moderately strong solution, and with this work completely through the following examination:—

A. Add dilute HCl.

(i.) No precipitate.	Pb probably absent.

With *the same solution* pass on to B.

(ii.) A white precipitate.	Pb present.

Add more HCl until the amount of the precipitate does not increase; filter, and with *the clear filtrate* pass on to B. The precipitate is to be examined by Table I.

B. Add a little *fresh* solution of H_2S (which must smell of the gas), or pass a few bubbles of the gas through the liquid:—

(i.) There is no precipitate.	Absence of Pb and Cu; also of Fe as ferric salt.

Pass on with the same solution to C.

(ii.) There is a dark precipitate.	Pb or Cu present.

Pass H_2S gas through the liquid until it smells strongly; filter; with *the clear filtrate* pass on to C, and examine the precipitate by Table II.

(iii.) There is a nearly white precipitate in a very fine state of division.	Fe present as ferric salt, or possibly due to presence of a nitrate.

Pass on *with the same liquid* to C, without filtering at this point.

C. Boil the liquid until it no longer smells of H_2S; add several drops of strong HNO_3 or 5 cb.c. of the dilute acid, and boil again. Filter if the liquid is not clear (neglecting the precipitate, which is only sulphur), and add ammonia until the liquid smells of it distinctly after being well shaken:—

(i.) There is no precipitate.	Absence of Fe.

Pass on with *the same solution* to D.

(ii.) There is a reddish-brown flocculent precipitate.	Presence of Fe.

Filter; with *the clear filtrate* pass on to D, and examine the precipitate by Table III.

D. Add a little *fresh* solution of H_2S (which must smell of the gas), or pass a few bubbles of the gas through the liquid :—

(i.) There is no precipitate.	Zn absent.

Pass on with *the same solution* to E.

(ii.) There is a white (or nearly white) precipitate.	Zn present.

Pass H_2S gas through the liquid until it smells distinctly of the gas; filter; with *the clear filtrate* pass on to E, and examine the precipitate by Table IV.

E. Add Am_2CO_3 solution :—

(i.) There is no precipitate.	Ca absent.

Pass on with *the same solution* to F.

(ii.) There is a white precipitate.	Ca present.

Add the Am_2CO_3 solution in excess; filter; with *the clear filtrate* pass on to F, and examine the precipitate by Table V.

F. Evaporate to dryness the portion of the liquid placed in the porcelain crucible, and ignite the residue *as strongly as possible* until no more white fumes of ammonium salts are given off. Then allow to cool. Add $\frac{1}{6}$ test-tube full of water to dissolve the residue, pour the solution back into the test-tube through a small clean filter, and add $NaHC_4H_4O_6$ (Sodium Hydrogen Tartrate); shake vigorously and put aside for ten minutes (occasionally shaking vigorously) :—

(i.) There is no precipitate.	Absence of K.
(ii.) There is a white crystalline pp. Examine the precipitate by Table VI.	Presence of K.

G. Place some of the original substance in a test-tube, and add a little NaOH solution; heat cautiously and smell the mouth of the test-tube:—

(i.) There is no smell of NH_3.	Absence of Am.
(ii.) There is a smell of NH_3.	Presence of Am.

TABLE I

EXAMINATION OF THE PRECIPITATE FORMED ON ADDITION OF HCl

The precipitate is Lead Chloride $PbCl_2$; the confirmation of this depends on the solubility of $PbCl_2$ in hot water.

Wash the pp. on the filter paper with a little cold water; place the funnel in a clean test-tube and pour on to the pp. on the filter half a test-tube full of boiling water; divide the hot liquid which runs through into two halves:—

(i.) Cool this under the tap; small white crystals of $PbCl_2$ separate from the liquid as it becomes cold.

(ii.) To this portion add a little dilute H_2SO_4—a white precipitate of $PbSO_4$ is formed.

TABLE II

EXAMINATION OF THE PRECIPITATE FORMED ON ADDITION OF H_2S

The precipitate may contain Lead Sulphide PbS, and Copper Sulphide CuS; their separation depends on the solubility of CuS in hot dilute H_2SO_4, by which PbS is not dissolved.

Wash the pp. well, best with hot water; then place the funnel in a clean test-tube, pierce the tip of the filter paper, and pour half a test-tube full of dilute H_2SO_4 on to the filter, letting it wash the precipitate into the test-tube underneath. Boil the pp. with the sulphuric acid, and filter:—

The *Residue*, if any, is PbS more or less completely converted into $PbSO_4$.

Pour a few drops of Am_2S over it—if the substance is blackened the presence of Pb is shown.

The *Filtrate* may contain Cu as sulphate.

Add ammonia to it in excess—a blue liquid shows the presence of Cu.

TABLE III

EXAMINATION OF THE PRECIPITATE FORMED ON ADDITION OF AMMONIA

The precipitate is Ferric Hydrate $Fe(OH)_3$; to confirm this dissolve it with a little dilute HCl poured on to the filter, and add Potassium Ferrocyanide $K_4Fe(CN)_6$ to the solution —a dark-blue precipitate of Prussian Blue shows the presence of Fe.

In order to ascertain whether the Iron is present in the substance examined as a ferrous or as a ferric salt, dissolve a little of the original substance in water or dilute acid and add to it some Potassium Thiocyanate KCNS—a deep red coloration shows the presence of a *ferric* salt, while the absence of such a coloration, when iron has been proved to be present, shows that the metal is there as a *ferrous* salt.

TABLE IV

EXAMINATION OF THE PRECIPITATE FORMED ON ADDITION OF H_2S TO THE ALKALINE SOLUTION

The precipitate is Zinc Sulphide ZnS; to confirm this dissolve the pp. in a little cold dilute HCl, boil the solution to expel H_2S and add NaOH by small quantities—a white precipitate which dissolves in excess of the sodium hydrate solution confirms the presence of Zn.

TABLE V

EXAMINATION OF THE PRECIPITATE FORMED ON ADDITION OF Am_2CO_3

The precipitate is Calcium Carbonate $CaCO_3$; to confirm this the pp. is washed with water and the funnel then placed in a clean test-tube; a third of a test-tube full of dilute acetic acid is then poured on to the pp. on the filter paper; this dissolves the pp. and the solution runs through into the test-tube.

Divide this solution into two halves :—

(i.) To one half add Ammonium Oxalate—a white pp.

(ii.) Test the other half by the flame test—reddish-yellow flame.

Presence of Ca confirmed.

TABLE VI

EXAMINATION OF THE PRECIPITATE FORMED ON ADDITION OF NaHT

The precipitate is Potassium Hydrogen Tartrate KHT ; to confirm this wash the pp. with a few drops of cold water, then take up some of the pp. on a clean platinum wire and hold it in the Bunsen flame.

A lilac flame-coloration, which appears red through blue glass, confirms the presence of K.

C. EXAMINATION FOR ACIDS

A. PRELIMINARY.

(i.) A *Nitrate*, if present, will have been already indicated by the tests in the Preliminary Examination III. (iii.) and IV. (i.).

(ii.) A *Sulphate* will have been indicated by the test in the Preliminary Examination V. (ii.).

(iii.) Put some of the dry salt in a test-tube and add dilute HNO_3—an effervescence indicates a *Carbonate.*

Confirmatory :—Pour the gas evolved during the effervescence into another test-tube half full of lime water, then shake—a turbidity in the lime water confirms CO_2 from a *Carbonate.*

(iv.) If a Carbonate has not been found—put some of the dry substance in a test-tube and add concentrated H_2SO_4—an effervescence and the evolution of white acid fumes indicate a *Chloride.*

Confirmatory :—(*a*) Test the fumes with blue litmus paper: they turn it red.

(*b*) Mix some of the dry substance with MnO_2; place the mixture in a test-tube with some concentrated H_2SO_4 and heat gently—*Chlorine* evolved (recognised by its smell, colour, and power of bleaching litmus paper)—*Chloride* confirmed.

B. WET TESTS FOR THE ACIDS :—

Prepare a solution from the substance by boiling it with water and a quantity of solid Na_2CO_3 equal to about twice the weight of substance taken; filter the hot liquid if any precipitate is formed, and divide the clear filtrate into three portions :—

(i.) Add HCl until there is no more effervescence, then $BaCl_2$—a white precipitate, which is not dissolved by more HCl, shows the presence of a *Sulphate.*

(ii.) Add HNO_3 until there is no more effervescence, then $AgNO_3$—a white curdy precipitate, which is not dissolved by more HNO_3 but dissolves when Ammonia is added, shows the presence of a *Chloride.*

(iii.) Add dilute H_2SO_4 until there is no more effervescence, then a few small crystals of $FeSO_4$ and shake gently for two or three minutes; pour off the liquid from the undissolved crystals into another test-tube, and add concentrated H_2SO_4, pouring it carefully down the side of the test-tube, so that it runs to the bottom and forms a heavy layer underneath the lighter solution—a brown ring where the two liquids meet shows the presence of a *Nitrate.*

C. CONFIRMATORY TESTS FOR THE ACIDS :—

(i.) *Carbonate.*—If the substance is soluble in water, add $BaCl_2$ to the solution—a white precipitate soluble with effervescence in dilute HCl confirms the presence of a Carbonate.

(ii.) *Chloride.*—Prepare a fairly strong solution of the original substance and add Lead Nitrate $Pb(NO_3)_2$ or Lead Acetate—a white precipitate, soluble in a sufficient quantity of boiling water and crystallising in white needles from this solution as it cools, confirms the presence of a Chloride.

(iii.) *Sulphate.*—To a solution of the substance in water or dilute HNO_3 add Lead Nitrate $Pb(NO_3)_2$ or Lead Acetate—a white precipitate, insoluble in boiling water, confirms the presence of a *Sulphate.*

(iv.) *Nitrate.*—(*a*) Heat a little of the substance with strong H_2SO_4 and one or two small pieces of copper—the evolution of reddish-brown fumes confirms the presence of a *Nitrate.*

(*b*) To a solution of the substance made acid with dilute H_2SO_4, add a little indigo solution and heat slightly—if the colour of the indigo is bleached from blue to pale yellow the presence of a *Nitrate* is confirmed.

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