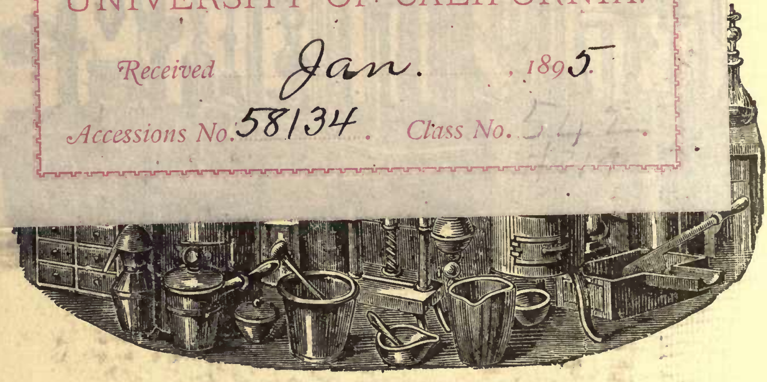
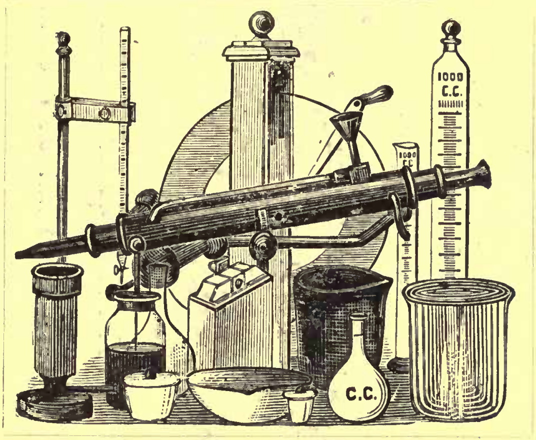
# GAS ENGINEER'S
# LABORATORY HANDBOOK

HORNBY

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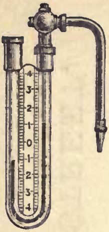

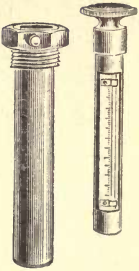


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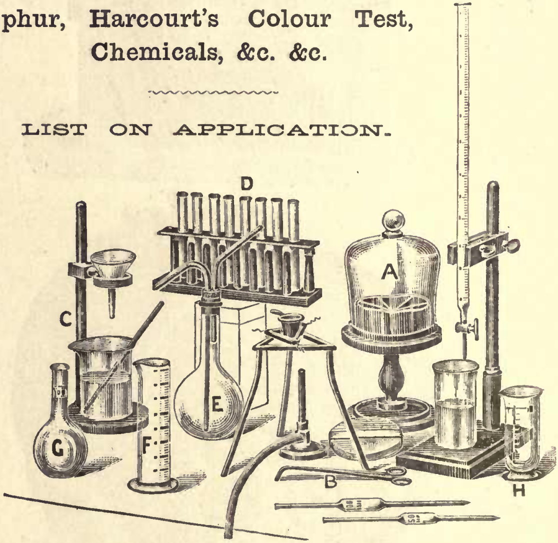
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THE

# GAS ENGINEER'S
# LABORATORY HANDBOOK

BY

JOHN HORNBY, F.I.C.

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London:
E. & F. N. SPON, 125 STRAND
New York:
SPON & CHAMBERLAIN, 12 CORTLANDT STREET
1894

This little Work is Dedicated

TO MY CHIEF

JOHN METHVEN, Esq., A.M.I.C.E

AS A SLIGHT RECOGNITION

OF HIS UNIFORM COURTESY AND KINDNESS

# PREFACE.

The object of this volume, as its title implies, is to describe the various analytical operations which are required in Gasworks.

In order to make it useful to those who may not have the opportunity of access to other works on analysis, the general operations incidental to the carrying out of analytical operations are described in detail, and further, as a means of assisting those who are dependent on self study, the methods of analysing certain substances obtainable in a perfectly pure state, and consequently having an established composition, are minutely described; these examples are chosen, as being typical of the more commonly occurring analyses required in Gasworks, as they involve the same methods of manipulation. By comparing the results obtained in the analysis of these simple substances with the figures demanded by theory, the operator can satisfy himself as to his competency to perform the same operations when they occur in the estimations required in his daily work.

For example, the analysis of barium chloride serves as a type of the methods of manipulation necessary in the determination of sulphur in coal, coke, and gas, and in ascertaining the accuracy of the solutions employed in the Referees' Ammonia Test, &c.; the estimation of calcium serves as a type of the analysis of lime; and so on.

The subject matter has been arranged as follows:—

Part I. treats of various preliminary operations necessary in quantitative work, such as the use of the balance, the preparation of substances for analysis, the ordinary operations employed in analysis, &c.

Part II. describes the method of estimating gravimetrically certain pure substances which are typical of the more commonly occurring analyses required in Gasworks.

Part III. gives an account of various operations connected with volumetric analysis, a description of the vessels employed for measuring liquids, and the method of calibrating the same; some simple volumetric processes applicable to gas testing being afterwards described.

Part IV. describes the more complex determinations required in Gasworks, some being gravimetric, others volumetric, while some embrace both methods. These include complete analysis of coal, fresh and spent purifying material, (lime, oxide of iron and Weldon Mud), ammoniacal liquor, sulphate of ammonia, fire bricks and clay, tar, crude gas for $CO_2$, $SH_2$ and $NH_3$, and purified gas for $NH_3$ and sulphur compounds.

Part V. treats of technical gas analysis, analysis of coal gas, furnace gases, &c.

In the Appendix a variety of tables of use in gas testing will be found.

In a book of this description there is not much scope for originality, and my work has principally consisted in collecting together, and arranging in a convenient and easily accessible form, the scattered literature dealing with the various subjects treated on, and in explaining by means of numerous practical examples the chemical reactions and calculations upon which the various tests depend.

I am greatly indebted to the excellent text-book on 'Quantitative Analysis' by Prof. Clowes and Mr. Coleman, which is published by Messrs. J. & A. Churchill. Many of the engravings and descriptions of analytical methods and processes have been, by permission, copied from that treatise. Reference may with advantage be made to Messrs. Clowes and Coleman's work for additional methods, as well as for fuller details concerning many of the processes included in my text.

I have also derived considerable assistance from the treatise on 'Quantitative Analysis' by Prof. Thorpe, published by Messrs. Longmans & Co. This is also well suited for general reference. The description of Hempel's Gas Apparatus and its applications is taken, by permission of Messrs. Macmillan & Co., from Hempel's 'Gas Analysis,' translated by Dennis. The last mentioned work should be consulted for fuller details, and for a description of Gas Analysis generally.

In addition to the above, the following works, which have also been consulted, will give further information on some of the subjects treated on:—

Sutton's 'Volumetric Analysis.'
Allen's 'Organic Analysis.'
Streatfield's 'Organic Chemistry.'
Cohen's 'Organic Chemistry.'
Arnold's 'Ammonia and Ammonium Compounds.'
Lunge's 'Tar and Ammonia Distillation.'
Hartley's 'Ammonia Liquor Tests.'
Winkler and Lunge's 'Technical Gas Analysis.'
Lunge and Hurter's 'Alkali Maker's Pocket Book.'
'The Journal of the Society of Chemical Industry.'
'The Journal of Gaslighting.'

J. H.

GASWORKS, BECKTON: *July* 1893.

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# THE GAS ENGINEER'S LABORATORY HANDBOOK.

## PART I.

### SECTION I.

#### INTRODUCTORY—THE BALANCE—WEIGHTS AND WEIGHING.

The chemical work of a gasworks laboratory chiefly consists in the quantitative determination of various substances whose composition is already known. The gas engineer is therefore principally concerned with "quantitative" analysis.

By "quantitative" analysis is meant that branch of practical chemistry which treats of the methods by which are determined the relative amounts of the constituents of a body, "qualitative" analysis simply informing us of the nature of the constituents, and how they may be separated.

The methods of quantitative analysis are divided into two branches, gravimetric and volumetric analysis.

By gravimetric analysis is meant the estimation of a substance by converting its known constituents into forms or combinations which admit of the most exact determination of their weight, and of which the composition is already accurately known.

By volumetric analysis is meant the method of determining the quantity of a substance (in solution) by the use of a measured volume of a reagent of known strength.

### *The Balance.*

1. The first essential for the correct performance of gravimetric analyses is the balance, which for careful quantitative work should turn easily and quickly, without too much oscillation, to $\frac{1}{10}$ of a milligram, or $\frac{1}{500}$ or $\frac{1}{600}$ of a grain, when 50 or 60 grams, or 1000 grains, are placed on each scale-pan.

Fig. 1 shows one of the forms of this instrument. It consists of a perforated brass beam, suspended at the centre on a triangular piece of hardened steel or agate termed a "knife-edge," which oscillates on a polished plate of agate, fixed on a brass support. At each end of this beam, is fixed a similar knife-edge of steel or agate, on which rest two agate plates, imbedded in brass settings. From each of these a hook depends, to which the pans are attached by light wires or thin straps. Figs. 2 and 3 give enlarged views of these terminal knife-edges and planes. When the instrument is not in use, the beam rests upon a frame connected with a rod which descends through the pillar of the balance, and which can be raised or lowered by an eccentric. The same movement also lifts up the plates connected with the pans, so that the knife-edges and agate planes are only in contact when the balance is oscillating. A second eccentric, connected with two bent levers, raises or depresses a support under each of the pans, so that they may be kept stationary whilst their load is being placed on them, or their vibrations arrested before the beam is lowered down on the centre plate. In some balances, all these movements are so connected that a single turn of the eccentric first rapidly releases the supports of the pans, then gently lowers the beam upon the centre knife-edge, and finally drops the pan suspensions upon the terminal knife-edges. By these arrangements the durability of the instrument is greatly increased.

To the beam is attached a long pointer, moving in front of a graduated ivory scale fixed to the pillar of the balance. This renders visible the slightest oscillations of the beam.

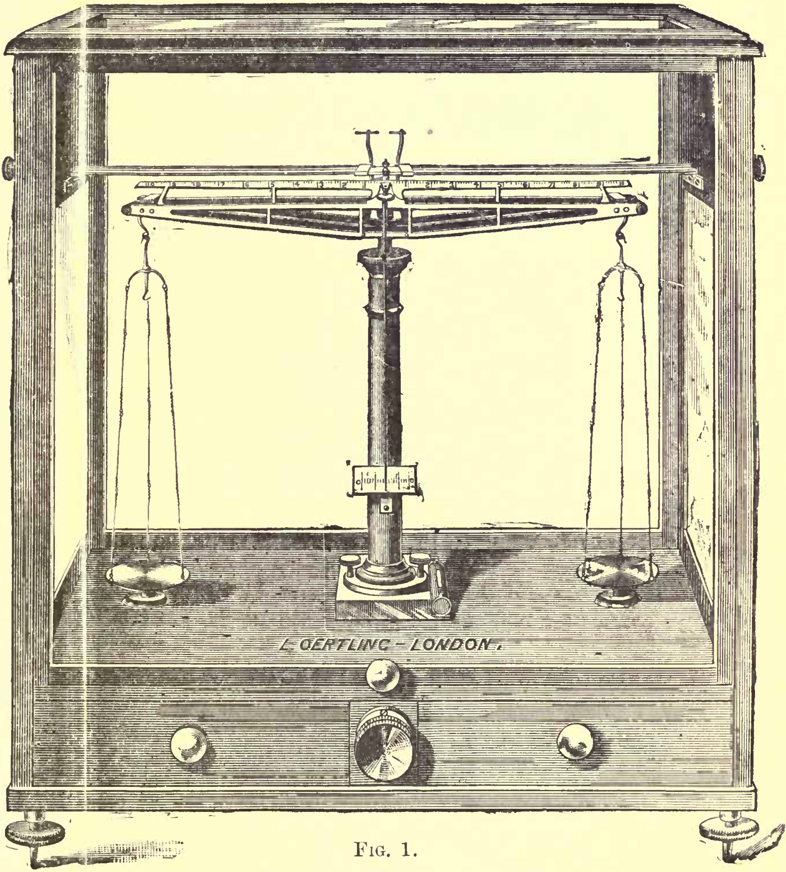


Fig. 1.

So long as the instrument is in equilibrium, the pointer hangs perfectly vertical, and opposite to the centre (zero) point of the scale, or it oscillates over an equal number of divisions on each side. The least preponderance on one side is indicated by a permanent deflection of the pointer from the zero, or by its vibrating through an unequal number of divisions on either side of the zero point. If from any cause the instrument itself is not in equilibrium, it may be adjusted by means of a little movable vane fixed on the top of the beam, by moving which, either to the right or to the left, the preponderance may be counterpoised. In some balances, in which the pans are suspended by a simpler method, this adjustment is effected by means of a little milled-head screw travelling along a thread, Fig. 3.

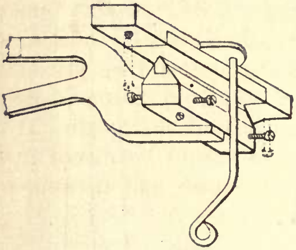


FIG. 2.

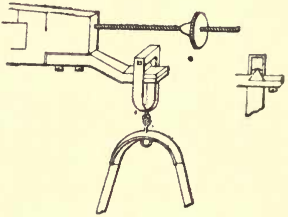


FIG. 3.

The accuracy of the balance depends (1) upon the equality of its arms, (2) upon the position of the point of support in relation to the centre of gravity of the instrument, from which also follows (3) that the points of suspension and support must be in the same plane, and (4) that the beam must be perfectly rigid.

The sensibility of the instrument depends (1) upon the freedom from friction of the knife-edges and planes, (2) upon the near approximation of the centre of gravity of the instrument to its point of support, and (3) upon the lightness of its beam. The relation of the centre of gravity to the point of support is, of course, adjusted as accurately as possible by the balance-maker, but by means of a small weight (termed a gravity bob) working on a thread, it may be altered at pleasure, and the stability of the balance thus regulated.

On account of the injurious effects of acid fumes and moisture, which rapidly impair the delicacy of a balance, the instrument is enclosed in a wooden case, having counterbalanced sliding glass sashes at the front and back. The case stops the entrance of dust, diminishes the rusting of the knife-edges, and prevents air-currents from acting upon the scale-pans during the process of weighing. The balance-case is supported on levelling screws, by which it can be adjusted to a perfectly horizontal position by means of a spirit-level.

The balance, if possible, should be kept from acid fumes, and, as a further precaution, a shallow vessel containing dried carbonate of potash or quicklime should be placed in the balance-case, being renewed when it becomes deliquescent by the absorption of moisture from the air. It is further necessary that the balance should be placed in a position free from extremes of temperature, and its support should be quite free from vibration.

### *The Weights.*

2. The weights generally employed in chemistry are those of the French or metric system, of which the gram is the unit; in the chemical operations incidental to gasworks, however, it is often necessary to use weights based on the English grain.

The metric system is in every respect the most convenient, for the following reasons:—(1) that the unit is extremely small; (2) that the different denominations in weight being related by multiples and submultiples of ten, they fit in with the system of numerical notation in general use, and consequently make chemical calculations more simple; and (3) that it bears a very simple relation to the measures of capacity and length.

For general use a set of weights ranging from 50 grams to a milligram on the metric system, and from 1000 grains to $\frac{1}{10}$ grain on the grain system, will be found convenient. They are generally preserved in a box, Fig. 4, the larger weights being placed in receptacles lined with velvet to prevent their being scratched, the smaller ones being also kept in separate compartments, covered by a plate of glass. The box is provided with a pair of small forceps with which the weights are always to be lifted, never on any account being touched by the fingers.

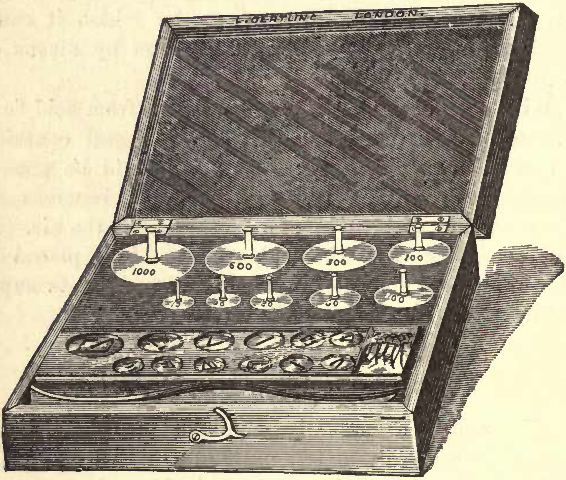


Fig. 4.

The gram weights down to one gram, and the grain weights down to ten grains, are generally made of brass, cylindrical in shape, and provided with handles for lifting by the forceps. The lower weights in both systems are usually made of platinum or aluminium foil, one corner of each being turned up for holding in the forceps.

3. Accompanying the weights are the riders. The rider

is a piece of fine wire bent as in Fig. 5. It is generally made either of gilt brass or of aluminium wire, and by reason of its form it is capable of "sitting" across the balance, and remaining on whatever part of the beam it may be placed. The object of the rider is to weigh the lowest fractions, such as milligrams and tenths of a milligram, and hundredths and thousandths of a grain. The rider in the metric system weighs one centigram, and in the grain system a tenth of a grain. The arm of the beam of the balance, from the centre to the terminal edge, is divided into ten equal parts, and each of these is (generally) further subdivided into five equal parts. The centigram rider, when placed just above the terminal knife-edge of the beam, weighs its normal weight of one centigram, or 10 milligrams; when placed on a point on the arm exactly in the middle of the two knife-edges it exerts only half this effect, that is to say, it is now equal to 5 milligrams; when placed at a fourth of the distance from the centre knife-edge it is equal to 2·5 milligrams; at three-quarters the distance 7·5 milligrams, and so on. The rider is moved from one division to another by means of an arm passing through the balance-case, and operated from the outside.

FIG. 5.

### *Testing the Balance and Weights.*

4. Before commencing a course of quantitative work, it is necessary to test the accuracy of the balance, which may be done by means of the following experiments:—

(*a*) The floor of the balance is first of all brought to the horizontal position, by the aid of the levelling screws and spirit-level; it is then accurately adjusted, if necessary, either by the regulating screws, or by means of pieces of tin foil, and a milligram weight placed in one of the scale-pans. A balance adapted for every-day work should turn very distinctly with this weight, whilst one adapted for delicate determinations should indicate the $\frac{1}{10}$ of a milligram with distinctness. It may be here pointed out, that the mere fact of the index needle pointing to zero on the scale, is not sufficient to prove that the balance is in equilibrium. The better plan is to observe the oscillations of the pointer, which can, if necessary, be caused to vibrate by moving the hand near one of the scale-pans, so as to create a slight current of air. The pointer should then traverse very nearly the same space on each side of the zero mark, travelling a lesser distance with each oscillation, and finally stop at the zero point.

(*b*) Both scale-pans are loaded with the maximum weight the balance is constructed to carry. The instrument is then accurately adjusted, and a milligram added to the weight in one of the scale-pans. This should cause the balance to oscillate to about the same extent as in (*a*), but in the majority of cases it is somewhat less.

(*c*) The balance is accurately adjusted (in the event of it being necessary to bring the scale-pans into equilibrium by adding tin foil to one of them, this tin foil must be left on the scale-pan during the experiment); both pans are then equally loaded with, say, 50 grams each, the balance, if necessary, being again adjusted (by adding small weights); the load on the two pans is then reversed, so as to transfer that of the right-hand pan to the left-hand, and *vice versâ*. If the balance has perfectly equal arms it should maintain its absolute equilibrium.

(*d*) The balance is again accurately adjusted; it is then arrested, and afterwards set in motion again, the same operation being repeated several times. A good balance should always resume its original equilibrium; faulty construction of the knife-edges will cause differences to appear.

(*e*) The speed of oscillation of the index pointer should be observed, for the slower the needle oscillates, the greater is the sensitiveness of the balance to small differences of weight, and *vice versâ*. It should not oscillate too slowly however, or it will cause the process of weighing to become wearisome.

The oscillations are slower the nearer the centre of gravity of the beam approaches the axis of its suspension.

5. The weights should be tested in the following manner to see if they agree among themselves.

One pan of the balance is loaded with a 1-gram weight, and then counterbalanced with small pieces of brass, and, finally, tin foil. The weight is then removed, and replaced by another 1-gram weight, any difference being noted; the remaining gram weight is then treated in the same manner. The 2-gram weight is then weighed against two single 1-gram weights, the 5-gram weight against three single gram weights, and the 2-gram weight, and so on. In comparing the smaller weights among themselves in this manner, they should not show the least difference on a balance turning with $\frac{1}{10}$ milligram.

### *The Process of Weighing.*

6. There are two methods for determining the weight of a substance—the direct method, and the method of substitution.

When weighing a substance by the direct method, the substance is placed upon one scale-pan, preferably the left, and the weight required to counterpoise it upon the other. Owing to this method being the more expeditious of the two, it is the one commonly employed. If, however, the balance is not in perfect equilibrium, or the arms of the beam are of unequal length, the *true* weight is not obtained by this method. This is of no consequence in ordinary analytical operations, since, in any one series of weighings, it is the relative weights only of the different substances which are commonly required, and these will be correctly obtained, provided the different substances be placed always upon the same scale-pan, and the condition of the balance does not undergo any alteration between the successive weighings of the series.

7. In the process of weighing by substitution, both the relative and absolute weights of substances are obtained. In this method, the substance is counterpoised by weights in the usual way; it is then removed from the scale-pan, weights being substituted in its place exactly balancing those already placed in the opposite scale-pan. The weights substituted for the substance will then represent the true weight of the substance. Operating in this way, it would not make any difference if the arms of the balance were not of equal length, or if the equilibrium of the instrument had not previously been adjusted.

8. The volume of the body weighed is almost invariably different from that of the weights which counterpoise it, consequently different volumes of air are displaced by the substance and the weight; it follows from this, that a body when counterpoised by weights in air would not be in equilibrium with the same weights in vacuo. The magnitude of the error from this cause is proportional to the difference between the volume of the weights and of the body weighed. If the volume of the body is greater than that of the weight, its weight, as indicated by the balance, is too small; and conversely, when the volume is less than that of the weights, the indicated weight is too great. In the generality of cases the error thus introduced is too small to be taken cognizance of, but in excessively accurate determinations it is necessary to take it into account. The absolute weight of a substance in vacuo can be obtained, if necessary, as follows. The weight of the volume of air displaced by the weights, is subtracted from the weight of the volume of air displaced by the substance, and the weight thus obtained is added to the weight on the scale-pan.

### *Directions for Weighing.*

9. The following directions describe the method of conducting the operation of weighing.

In the first place, see that the scale-pans and floor of the balance-case are quite clean, and free from dust.

Next, see that the rider hangs upon the projecting arm of the brass rod, and not upon the beam of the balance.

Then see if the instrument is in equilibrium, by gently lowering the supports, and setting the beam swinging. If the beam does not commence to oscillate on being released, start it, by gently waving the hand over one of the scale-pans; the pointer should then oscillate through equal spaces on each side of the zero of the scale; if this is not the case, bring it into equilibrium by moving the brass vane, or altering the terminal screws. If, however, it is simply required to obtain the weight of a body by difference, this adjustment is not necessary. Thus, supposing, for instance, we wish to weigh out a certain quantity of powdered coal for analysis. It may be put into a weighing bottle, Fig. 6, and the weight of bottle and coal together determined. About as much of the coal as is required (say 1 gram) is then emptied out and the bottle again weighed; the difference between the first and second weighings gives the weight of coal taken.

FIG. 6.

The substance whose weight is required, should invariably be placed on the left-hand scale-pan, the weights being placed by the forceps on the right-hand pan. The weights should be placed on the pan in a systematic manner, and not taken at random from the box. The better plan is to start with a weight which is a little too heavy, and then to try the effect of lower weights of the same denomination, in succession, until the balance assumes a state of equilibrium, or the weights are a little too small; in the latter case, the weights of the next lower denomination should be tried in the same way. For example, supposing we wish to determine the weight of a platinum crucible, which was ultimately found to weigh 16·715 grams. We place on the weight pan the 10-gram weight and release the beam, this proves too little; we then add another 10-gram weight, this proves too much; we substitute the 5-gram weight for the 10-gram, the weight is again too little; we add the 2-gram weight, this is again too much; we substitute one of the 1-gram pieces for the 2-gram piece, the weight is too little; we add the 0·5 gram, still too little; also the 0·2 gram, still too little, although it will now be noticed that the rate of the vibration of the beam becomes much slower, indicating that the balance is rapidly approaching equilibrium. We have found that 16·7 grams is not quite sufficient to balance the crucible, we therefore add 0·1 gram, too much (the pointer swings with increased energy in the opposite direction); we substitute 0·05 for the 0·1 gram, still too much; we then try the 0·02, the pointer now vibrates much more slowly, but still indicates that the weight is too great; we substitute 0·01 for the 0·02 gram, the pointer swings with the same slowness, but shows an equal deflection to the opposite side of the zero; we add 0·005 gram, the pointer now makes excursions of equal amplitude, that is, the balance is in equilibrium.

The position of the pointer at the end of the vibrations, should be read off several times in succession, in order to make sure that the balance is in equilibrium; the beam is then arrested, and the aggregate value of the weights needed determined, both from the vacant spaces in the box, and from the denominations marked on the weights themselves. This method of double reading should always be employed, so as to prevent false entries.

It should be particularly noted, that whenever an exchange of weights is necessary, the motion of the beam must be arrested. Under no circumstances must anything be added to, or taken away from the pans when the balance is in motion. By not attending to this precaution, the knife-edges would soon become worn, and the balance would consequently be inaccurate. The balance-case should be closed while the rider is being used, and the final observation of equilibrium must be always conducted in the closed case.

In weighing out substances for analysis, they should not be placed direct on the scale-pan, but weighed in vessels of glass, porcelain, or platinum. Powders may be weighed out from a stoppered weighing bottle, the difference in weight between the first and second weighings representing the amount of powder weighed out. Hygroscopic substances may be weighed in watch-glasses with ground edges, and held together by a brass clip. Hygroscopic, efflorescent and volatile substances—in fact, all substances which gain or lose weight on exposure to air, should be weighed in closed vessels.

A substance should never be weighed when warm. By placing a warm object on the balance-pan, the indications of the instrument are greatly affected. The ascending air current produced by the heat of the warm substance, acts against the pan containing the substance, and also against the beam above it, and the substance appears to weigh less than it ought to weigh. The warm air, after a time, also affects the portion of the beam against which it impinges, and by increasing its length disturbs the equality in the arms. Further, all substances condense upon their surface a certain amount of air and moisture, the weight of which depends upon the temperature: this also is an error in the same direction, and causes a body when warm to weigh less than when cold.

Apart from the question of temperature, a vessel, say a platinum crucible, when rubbed with a dry cloth and weighed immediately afterwards, always weighs sensibly less than after half-an-hour's exposure to the air of the balance-case, owing to the condensation of the air, with its accompanying aqueous vapour, upon its surface. It is advisable, therefore, to allow the crucible, if newly wiped, to remain in the balance-case some little time before weighing.

## SECTION II.

SAMPLING—MECHANICAL DIVISION—DRYING AND DESICCATION—SOLUTION AND EVAPORATION—PRECIPITATION—FILTRATION AND TREATMENT OF PRECIPITATES.

### *Sampling.*

10. The first operation in the conduct of an analysis is that of sampling, to which too much attention cannot be paid, as on the sample being a true representative of the whole bulk, depends the value of the analysis. For instance, in taking a sample of oxide of iron from a heap, if we were to simply take a sample from the outer portion, this would not give a fair indication of the bulk; by reason of that part of the heap being acted upon by the atmosphere, it would probably be found to be much drier as compared with the interior portion, and thus, at any rate as far as the amount of moisture was concerned, the sample would not represent the average of the heap.

The proper mode of procedure in the case above cited, is to dig well into the heap, and taking small portions from every part, mix these well together, afterwards selecting still smaller portions from this for the final sample; these latter portions being also turned over and well mixed. The sample should be immediately transferred to a tightly stoppered bottle, and labelled with the description and date of sampling.

In sampling coal or cannel, all pieces should be broken up into lumps not larger than walnuts, and well turned over in the manner just described.

### *Mechanical Division.*

11. The next operation is that of mechanical division. In order to prepare a substance for analysis, that is to say, to make it in a suitable condition for the action of solvents,

&c., it is generally necessary to have it in a minute state of subdivision, as this will create abundant points of contact for the action of the solvent. Substances are obtained in this fine state by being pounded or triturated. The operation of pounding is generally performed in mortars, the essential factor in the operation being, that the material of which the mortar is composed, is harder than the substance it is wished to reduce to powder, otherwise the substance may get contaminated with the material composing the mortar. For the ordinary salts, and substances not possessing any very considerable degree of hardness, a porcelain, glass, or Wedgwood-ware mortar may be used, but for minerals, and siliceous materials such as fire-bricks, an agate mortar is indispensable. In pounding coal, an iron mortar will be found useful.

In the operation of pulverisation, as in most others, much time and trouble may be saved by a systematic method of working, and an equally great loss incurred by careless manipulation. Some substances fall into small fragments when simply touched by the pestle, and are easily reduced to fine powder by a grinding motion, while others require a great deal of pounding, and necessitate care in order to avoid dispersion of the fragments. This may be prevented by striking with the pestle straight down, carefully avoiding lateral blows, as such blows chiefly cause the particles to fly about. After the substance has been tolerably reduced, it is more advantageous to substitute a movement between that of a blow and a grind, for the directly downward motion of the pestle. This is effected by grasping the handle firmly in the hand, the striking portion of the pestle pointing somewhat inwards, and then striking downwards, drawing the pestle towards the operator's body. When by this means a certain degree of fineness has been obtained, it is advisable to entirely substitute trituration for blows, the pestle then travelling in turn, over the whole of the lower portion of the inside of the mortar.

Where the matter adheres to the sides of the mortar, it must be scraped down towards the centre with a spatula. It is necessary to have only a moderate amount in the mortar at one time, as otherwise the fragments are protected by each other from the action of the pestle. The operation of pounding may be considerably assisted by that of sifting. For this purpose, the sets of sieves sold by dealers in chemical apparatus will be found useful. The following device may also be employed for the same purpose. A piece of clean and dry muslin is tightly tied round the top of a small beaker. On this is lightly poured some of the powdered substance; on gently tapping the muslin with a glass rod a portion falls through, the remainder, which is too large for the pores of the muslin, is returned to the mortar, and again pounded until it is likewise fine enough to pass through.

Impurities may be removed from porcelain and Wedgwood-ware mortars by rubbing in them a little sand saturated with a strong acid or alkali. It is seldom that any dirt is found to resist the action of nitric or sulphuric acids, or caustic potash.

### *Desiccation.*

12. Before an analysis can be proceeded with, it is necessary that the substance under examination is free from all unessential constituents. The most frequent of these is moisture, by which is meant, the moisture in excess of that which is necessary to the constitution of the body. In drying a substance, therefore, the aim generally is, to get rid of the adherent moisture without interfering with the chemically combined water, or with any other constituent. The methods of effecting the operation in each particular case, will depend upon the temperature to which the substance can be exposed without being decomposed. Substances containing water of crystallisation may frequently have their adherent moisture removed by pressing repeatedly between

folds of filter paper. Another easy but rather slow method of desiccation, is to expose the substance in a closed vessel, called a "desiccator," over a pan containing calcic chloride, strong sulphuric acid, or other hygroscopic body. A partial vacuum in the desiccator accelerates the operation. Many substances may be freed from water at a temperature of 100° C. (212° F.). A thin layer of the substance (powdered if solid), spread on a watch-glass, is exposed to a gentle current of air in a steam-jacketed oven, known as a water oven,

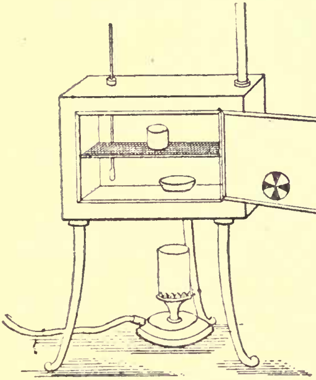


FIG. 7.

Fig. 7. The apparatus is made of copper, and is doubly-cased throughout with the exception of the door.

The method of using it is to fill it to about three-fourths of its height with water; it is then heated by means of a Bunsen burner, with the effect that when the water boils, the upper part of the hollow casing becomes filled with steam, and the temperature inside the oven approaches 100° C. A gentle current of air at the same time passes through the oven. The distillation of water may frequently be combined with the heating of water ovens.

When a temperature higher than 100° C. is required for the drying of a substance, an air oven is employed. An air oven is almost identical in construction with a water oven, the main difference being, that heated air takes the place of boiling water and steam within the jacket. Since the bottom of the oven comes into direct contact with the flame, it follows that it is usually hotter than the air in the interior, consequently the substance must not rest directly on the bottom. A convenient stand may be made by turning down the wire ends of a pipe-clay triangle at right angles to the plane of the triangle. A thermometer passing through a cork in the top of the oven should be provided, so as to register the temperature of the interior. With a little experience any required temperature may easily be obtained by regulating the tap on the gas supply, and after the heat has once been regulated the temperature will remain constant for several hours. Regulators are also sold by dealers in chemical apparatus, by attaching which the temperature may be kept constant for any length of time.

Many substances after drying readily take up moisture again, consequently some means must be adopted to prevent them from absorbing moisture from the air while cooling, or waiting to be weighed. Dried apparatus also which is left for weighing, should be preserved from the deposition of water upon its surface. The usual method of preventing the access of atmospheric moisture is to enclose them in an air-tight vessel, the air in which is kept dry by exposure to a hygroscopic substance, sulphuric acid or calcic chloride being the reagent commonly used. One form of this instrument is shown in Fig. 8. It consists of a broad glass jar contracted in the middle. The upper portion contains the substance to be weighed, the lower portion the desiccating agent.

A circular piece of perforated zinc forms a bottom to the upper portion, and on this a bent pipe-clay triangle or other support is laid. The body to be cooled and weighed is placed on this support. The upper rim of the desiccator is ground, and a flat lid, also ground, fits truly on to it, so that when the upper rim is lightly greased the flat lid fits air-tight. By this device, an air-tight chamber is obtained, in which a body may cool in dry air, and be kept from contact with moisture for an indefinite period.

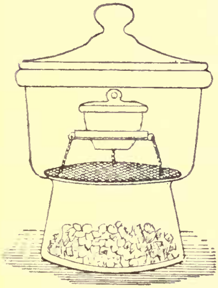


FIG. 8.

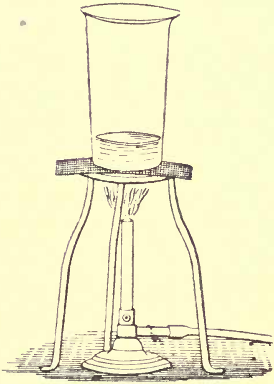


FIG. 9.

### *Solution of Solids.*

13. It is usually necessary before analysing a solid substance to have it in a state of solution, and in order to dissolve it the substance should be in a fine state of subdivision, being powdered, if necessary, as described in (11). If the substance is soluble in water, a convenient method of dissolving it is shown in Fig. 9. The substance is put into a beaker containing distilled water, and the beaker is then placed on a piece of wire gauze on an iron tripod over a Bunsen burner. Should "bumping" ensue, the contents of the beaker may be stirred occasionally with a glass rod. In order to guard against loss by spurting, the beaker is covered with a clock-glass.

If the substance is likely to effervesce whilst dissolving, as is sometimes the case, particularly on adding an acid to carbonates, the following methods may be employed:—(1) The substance is placed in a conical flask, the solvent being added through a small funnel which is left in the mouth of the flask, closing it during the process of solution; by this arrangement loss by spurting is prevented, but free passage of the gas given off is allowed. (2) Another method is to place the substance in a round flask inclined at an angle of about 45°. In this case also loss by spurting is prevented, as the drops thrown up during effervescence will be prevented from escaping by striking the inside of the flask, at the same time the gas will have a free exit.

It is necessary to exercise care in the selection of vessels intended to be used for purposes of solution or evaporation, so that they will not be acted upon by the solvent.

### *Evaporation.*

14. The process of evaporation is employed either to concentrate a solution, or to drive off the whole of the liquid by the aid of heat. It is usually conducted in basins made of porcelain, platinum, nickel, or silver, although sometimes a beaker or crucible is employed. Liquids are usually evaporated over boiling water in vessels known as water-baths; the heating agent in this case being steam. A simple form consists of a cylindrical copper vessel, rather more than half filled with water, and heated by a Bunsen burner. The upper portion of the vessel can be fitted with round rings of flat sheet copper, the diameters of the rings gradually diminishing in size. The top of the vessel may thus be made to fit and support vessels of different sizes. A more elaborate water-bath is shown in Fig. 10. The top of this arrangement is provided with holes of different sizes, so as to fit vessels of varying dimensions. These holes when not required for use are covered with lids. Should a vessel

employed for purposes of evaporation require to be afterwards weighed, it should rest on a glass ring, and not come into direct contact with the copper edge of the water-bath as above described, as such contact is likely to stain it. The upper part of a beaker, provided it is of the proper diameter, makes a very good support.

A broken or cracked beaker may easily be converted into such a support, by cutting round it with a diamond, or leading a crack round it about an inch below its upper edge. A water-bath of a simple form may be easily improvised by partially filling a beaker with water and heating it over a Bunsen burner. Should the water in the beaker be inclined to bump while boiling, the bumping may be prevented by placing a few small pieces of paper into the water.

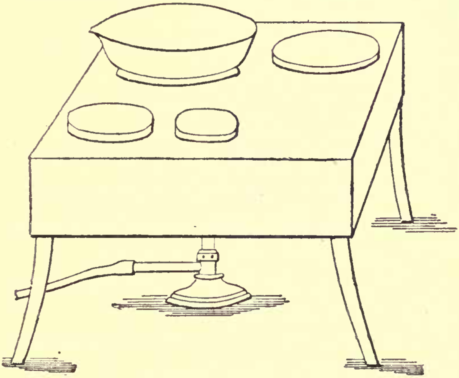


FIG. 10.

When a higher temperature than 100° C. is required in order to evaporate a liquid, one of the following methods may be employed. The vessel may be placed in a sand-bath, which is simply a shallow, saucer-shaped vessel of thin sheet iron filled with fine dry sand, heated by a Bunsen burner. By immersing the vessel to a depth of an inch or so in the sand, a uniform heat is imparted to the bottom of the vessel; or the vessel may be heated by direct contact with a naked flame, the rose head being placed on the Bunsen during the operation. Another method is to support the vessel on a piece of wire gauze, or on a sheet-iron plate. The latter plan possesses the advantage that the rate of evaporation may be modified by moving the vessel, say a beaker, to a greater or lesser distance from the part of the plate directly in contact with the flame. Where effervescence is likely to occur, it is advisable to use an evaporating dish, covering it with a large inverted funnel.

When conducting an evaporation it is advisable to observe the following precautions:—

(1) To prevent the liquid from actually boiling, or there will be a risk of loss by spurting.

(2) When conducting an evaporation over a naked flame or sand-bath, towards the end of the operation, the vessel should, if possible, be transferred to a water-bath, as a liquid, even when heated below its boiling point, is likely to spurt when it is in a thick and pasty state, by the separation of solid matter.

(3) Sometimes it happens that the liquid creeps up the side of the vessel during the process of evaporation. This particularly applies to solutions containing salts of ammonia. In the event of this occurring, the incrustation forming near the edge of the vessel should be detached, and pushed down by the aid of a glass rod.

### *Precipitation.*

15. The operation of precipitation is frequently made use of in quantitative analysis, and is employed when it is required to convert a substance in solution into an insoluble form, in which condition it may be collected and weighed. The operation is usually performed in glass beakers, on account of the facility with which the contents of the same may be transferred either to the filter, or to the vessels in

which they are to be weighed. In the event of having to deal with strongly alkaline liquids, or where the latter have to be heated for some time, porcelain basins are more suitable. Precipitation is usually effected with hot solutions, and but rarely in the cold. If the solution is boiling, the beaker containing it is covered with a clock-glass, so as to avoid any loss by spurting. The separation of the precipitate from the liquid in which it is formed, is effected either by decantation or by filtration, or by a combination of both these processes. As a general rule, where the liquid has to be filtered, it will be found advantageous to allow it to stand at rest for some hours after adding the precipitating agent, known as the "precipitant," for the reasons (1) that the complete precipitation of the whole of the substance in solution only takes place after some time; and (2) that in some cases, if the liquid is poured on the filter immediately after precipitation, there is a risk of its passing through the pores of the filter-paper. Some precipitates form very rapidly, and being of a flocculent character may be filtered off at once. The hydrates of iron and alumina are of this type. In all cases of precipitation, the addition of a large excess of the precipitant should be avoided, as this means increased labour in washing; but the presence of a slight excess is necessary, in order to ensure the complete throwing down of the whole of the substance in solution. This is easily ascertained, by adding a drop of the precipitant to the solution filtered from the precipitate, which will cause a further precipitation, if sufficient had not been added in the first instance.

### *Filtration and Washing of Precipitates.*

16. A precipitate is separated from the liquid in which it has been thrown down, by passing the solution through a specially prepared filter-paper, procurable at the dealers in chemical apparatus. Filter-papers of the best quality suitable for quantitative work are treated with hydrochlorine

and hydrofluoric acid, and then thoroughly washed, so as to extract the greater part of the inorganic matter; consequently when a paper of this description is burnt, it leaves an almost inappreciable amount of ash. Filter-papers should permit of rapid filtration, but at the same time the pores should be sufficiently minute, to prevent the passage of even the most finely divided precipitates. Swedish filter-papers fulfil these conditions admirably. The filter when required for use is folded twice at right angles, Fig. 11, opened out into the form of a cone, Fig. 12, and placed in a glass funnel, whose sloping sides should open at an angle of about 60°, and merge into the neck at a definite angle, when it will be found to fit the form of the folded filter-paper, and consequently give the latter uniform support. When placed in the funnel, the filter-paper is moistened with distilled water from the wash-bottle, for the purpose of causing the fibres of the paper to expand, and thus diminishing the size of the pores, without at the same time clogging them with solid particles. If this preliminary moistening is omitted, and a solution mixed with a precipitate is poured into the dry filter, some of the finely divided particles of the precipitate are drawn into the pores by capillary attraction, and tend to prevent the passage of the clear solution through them. The filter should never be allowed to reach higher than the top of the funnel, as otherwise there would be a danger of the paper giving way owing to the weight of the liquid, besides the further danger of some of the solution

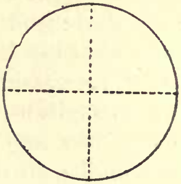


FIG. 11.

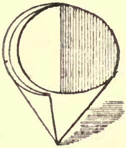


FIG. 12.

running down the outside of the funnel after passing through the projecting paper. It is even better that the funnel should project half-an-inch above the edge of the paper. The funnel is placed in a convenient stand, so that the edge of the stem (cut obliquely) touches the side of the vessel intended to receive the filtered solution, termed the filtrate. By allowing the liquid to fall down the side, all splashing, and consequent risk of loss of the filtrate is avoided. The solution to be filtered should never be poured directly into the funnel, but down a thin glass rod, Fig. 13, the stream of liquid being so directed as to fall against the side of the filter, and not into the apex, as that would endanger the bursting of the paper, and cause loss by splashing. A liquid retaining a precipitate in suspension, should never be poured higher than within half-an-inch of the margin of the filter. The rim of the vessel containing the liquid to be filtered should be slightly greased; this prevents the liquid running down the outside of the vessel. When not in use, the rod employed in pouring the liquid into the filter is placed in the vessel containing the precipitate, or, if it is

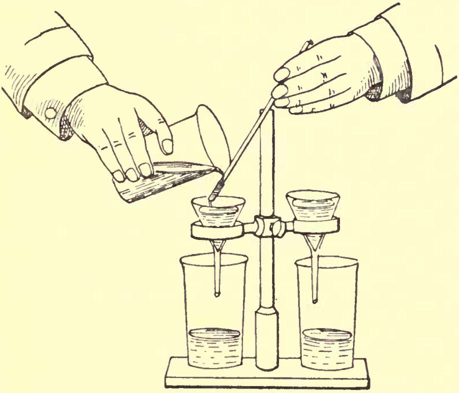


FIG. 13.

necessary that the latter should not be disturbed, in a little flask or beaker, which is afterwards rinsed out into the filter so soon as the whole of the precipitate has been transferred on to the filter. During the progress of the filtration, it is advisable to cover the various vessels with glass plates, so as to prevent dust from falling into them.

A special plate with a small hole at the side will be necessary for covering the beaker receiving the filtrate, so as to admit the stem of the funnel.

It occasionally may happen that some of the precipitate passes through with the filtered solution, barium sulphate and calcium oxalate, when freshly precipitated, being especially liable to do so. Should this occur, it may be necessary to pass the precipitate through the filter two or three times before it comes through quite clear. A clear filtrate may, however, be obtained in most cases by well boiling the mixture before filtering, which causes the finely divided particles of the precipitate to coalesce together. The presence of particular saline matters in solution also sometimes prevents a precipitate passing through; ammonium chloride, for example, exercises this property with barium sulphate.

As previously remarked, the operations of filtration and decantation are usually combined in the following manner. After decanting the clear liquid, a fresh quantity of distilled water, or other washing liquid, is poured upon the precipitate in the beaker, and the contents of the vessel agitated. The mixture is then allowed to subside until the liquid is clear again, and the clear liquid is poured through the same filter. These operations are then repeated several times. Finally the precipitate is transferred to the filter-paper by means of the spray from the wash-bottle, Fig. 14, the portions of the precipitate adhering to the beaker being removed by means of a glass rod capped with india-rubber tubing. Should it be found impossible to remove the last traces of the precipitate by means of the latter contrivance, the precipitate may usually be dissolved and re-precipitated. When the whole of the mixture has been poured on the filter, all the solution should be allowed to pass through before commencing to wash the precipitate on the filter. The object of washing, is to remove all soluble matters from the filter, and the operation is of great importance in analysis, as a precipitate imperfectly washed would weigh too high. For instance, in the determination of sulphuric acid by precipitation with an excess of barium chloride, if the excess of barium chloride is not removed by washing, we should be weighing, not the weight of barium sulphate alone, but a mixture of barium sulphate and chloride, which would, of course, give an erroneous result.

Precipitates are generally washed by means of the wash-bottle, shown in Fig. 14. This is simply a flask made of thin glass for the purpose of standing heat, fitted with a sound cork, through which two glass tubes pass. One, which is bent at an angle of 45°, has a piece of india-rubber tube attached to it, and enters the flask only a short distance; the other, which reaches nearly to the bottom of the flask, has its upper end drawn out to a fine point, and bent at a suitable angle for directing the stream of water. In order to hold the wash-bottle with convenience, a coil of thick string may be wrapped round its neck. On filling the wash-bottle with water and blowing through the india-rubber tube, a fine stream of water may be directed on any part of the filter.

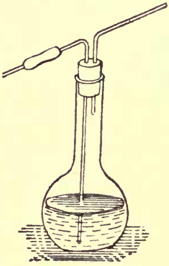


FIG. 14.

The precipitate remaining on the filter is now well washed (generally with hot distilled water, as hot water filters more rapidly than cold), the current of water being applied, first towards the upper part of the filter, and then directed gradually downwards. When the filter has in this manner been nearly filled up with water, the whole should be allowed to run through before adding any more; the operation is then repeated until the precipitate is sufficiently washed, which is known by testing some of the final washings. If the precipitate, while standing in the filter, cakes together into lumps, these must be broken up by directing upon them a strong current of water from the wash-bottle, as otherwise the water would not penetrate them, and some of the soluble matter would escape removal.

### *Accelerated Filtration.*

17. The process of washing the precipitate as described above is frequently very tedious, hence various expedients have been devised in order to accelerate the process.

One of the simplest means of attaining this result is to attach a bent tube to the funnel, as shown in Fig. 15. When the liquid descends and fills the longer limb of the tube, a considerable "pull or vacuum" is exerted on the contents of the funnel, consequently increasing the rate of filtration. The most effective method, however, for inducing rapid filtration is by means of the filter-pump, or other appliances which remove the atmospheric pressure from beneath the filter more or less completely, and yet leave that pressure in its entirety to act on the surface of the liquid in the funnel.

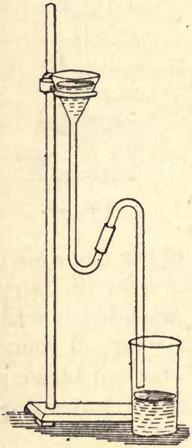


FIG. 15.

A handy form of filter-pump is that invented by Geissler, and shown in Fig. 16. It is made of glass. The most convenient place for fixing this apparatus is over the sink of the water supply. The upper vertical tube of the filter-pump should be attached, by means of a stout india-rubber tube, to a high-pressure water service, and in order to withstand the pressure of the water, the india-rubber tube should be firmly bound with strong copper wire to the water tap and filter-pump. The action of the pump is as follows. As shown in the drawing, there are three tubes fixed into the chamber in the upper portion of the apparatus. The upper vertical tube conveys the water from a high-pressure water service. This, in passing through the contracted end of the tube into the opening immediately below it, entangles air from the chamber, and carries it away through the lower vertical outlet pipe. Air is consequently pulled through the side horizontal tube, and a vessel placed in connection with this tube will consequently be gradually exhausted of its air. The side tube of the pump is connected with a tube from a small Woulffe's bottle. This tube passes through the cork of the bottle nearly to the bottom; the other tube of the bottle passes just through the cork in its neck. This tube is connected in its turn by means of india-rubber tubing, either with another Woulffe's bottle containing the funnel, or with the side tube of a filtering flask. The first Woulffe's bottle is employed in order to prevent any of the water from the water service from entering the Woulffe's bottle containing the filter, or filtering flask, when the water supply is suddenly checked. In the event of this occurring, the water from the filter-pump would be drawn back into the side tube by the partial vacuum created; but instead of entering the filtering flask it is arrested in the first Woulffe's bottle. In this way the water is prevented from mixing with the filtrate in the second Woulffe's bottle or filtering flask, and as soon as the filter-pump is re-started, the water in the Woulffe's bottle is rapidly sucked out.

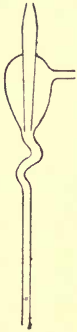


FIG. 16.

By reducing the pressure underneath the filter in the manner indicated, the pressure acting on the upper surface of the paper is frequently greater than the unsupported paper can bear without breaking; it is therefore necessary to support the apex of the filter-paper by a platinum cone, which is obtained of the desired shape in the following manner. A circular piece of writing paper, 10 or 12 centimetres in diameter, is folded in the shape of a filter, and placed in the funnel so as to fit accurately to the sides of the same, particularly near its apex. It is kept in position by a few drops of sealing-wax, and is saturated with oil by means of a feather, care being taken that no drops of the oil remain at the apex of the paper cone. A thin cream of plaster of Paris is then poured into the paper mould, and a small wooden handle is inserted into it just before the plaster solidifies. In a few hours the plaster cast will be dry enough to be removed from the funnel, together with the oiled paper. The outside of the same paper cone is now thoroughly oiled and inserted into a small capsule, or similar shaped vessel, of 4 or 5 centimetres in height, filled with a cream of plaster of Paris. As soon as the outer mould is dry, the plaster cone is removed, and the paper rubbed off it. In this way a solid cone fitting into a hollow cone is obtained, both of which have the angle of inclination of the funnel. A piece of platinum foil, of such a thickness that one square centimetre weighs 0·15 gram, is cut into the shape and size shown in Fig. 17; it is divided by a pair of scissors along the line *a b*, as far as the centre *a*. The foil is then held in the Bunsen flame for a few minutes to render it pliable, and placed against the plaster cone, so that the point *a* is at the end of the cone; the side *a*, *b*, *d*, is folded against the cone, and over this is folded the remainder, *a*, *b*, *c*, so that the foil also becomes a cone, the sides of which have exactly the same inclination as those of the plaster cast, and also of the funnel. The shape of the platinum cone may be completed by dropping it into the hollow mould, and pressing it down by

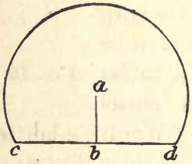


FIG. 17.

means of the plaster cone. If, after use, the cone should happen to get out of shape, the correct shape may at any time be obtained again, by simple pressure between the two cones. The platinum cone should not allow any light to pass through its apex. When properly made it will support a filter filled with liquid under the pressure of an atmosphere without the paper breaking. The small space between the folds of the foil is quite sufficient to allow of the passage of a rapid stream of water from the filter. The platinum cone is fitted for use in the following manner. A funnel is selected exactly fitting the cone, which is placed in the funnel, and then a dry filter-paper, folded in the ordinary manner, inserted. The filter-paper is pressed so that the cone and paper fit the sides of the funnel closely. The paper is then wetted by pouring in a little distilled water. The overlapping edges of the filter-paper are pressed down by the fingers, and any air-bubbles between the funnel and paper are also carefully removed by pressure. The funnel is then fitted, by a perforated india-rubber cork, either into the neck of a clean, strong, conical filtering flask, or into one tubulure of a Woulffe's bottle of suitable size; the side tube of the filtering flask, or the remaining tubulure of the Woulffe's bottle being placed in connection with the filter-pump. After making the connection between the pump and the vessel carrying the funnel, but before commencing filtration, it is advisable to fill the filter with distilled water and to start the pump. If any air is observed to be sucked down between the filter and funnel, this should be stopped by gently pressing the moistened edges of the paper against the funnel. In commencing filtration the pump should be started very gently, particularly if the precipitate is a fine one, as in that case particles of the precipitate may be at first easily drawn through the paper. To filter a precipitate by this apparatus, the supernatant liquid is cautiously poured on to the filter, the action of the pump is gently started, and as the liquid flows through into the filtering flask, fresh portions are added until the whole has been decanted. The precipitate is then transferred in the ordinary manner, and washed by the addition of water from an open mouthed vessel and not by a jet from a wash-bottle. The liquid in which the precipitate was originally formed, together with that necessary to transfer the precipitate to the filter, should be allowed to flow away completely before the operation of washing is commenced. As soon as the precipitate is drained, but before any channels begin to form in it, the filter should be filled up with distilled water, poured cautiously down the side of the funnel. When this lot of wash-water has drained away, the suction is continued until the precipitate is observed to shrink, when the filter is again filled up. This process is to be twice repeated, after which the precipitate may be drained almost dry by using the filter-pump a few minutes longer.

This method of filtration and washing is exceedingly rapid as compared with the old plan. A further advantage attends the use of the filter-pump, that is, the condition of the precipitate after filtration. Precipitates such as ferric oxide and alumina are left so dry that, without further drying, the precipitate wrapped in the filter-paper may be placed in the crucible over the Bunsen flame, and, after cautiously charring the paper, the whole may be ignited without risk of loss by particles being projected out of the crucible.

### *Drying of Precipitates.*

18. The next operation is that of drying the precipitate, which is effected as follows.

The water remaining in the stem of the funnel is first removed by means of a filter-paper, and the mouth of the funnel is then covered with a moistened filter; on drying, the paper adheres to the rim of the funnel, and in this way protects the precipitate from dust. The funnel is then placed in the water oven (12) and kept there until the paper and precipitate are quite dry. This method of drying the precipitate possesses the advantage that, when the precipitate is contracting, it detaches itself from the filter-paper, so that precipitates of a curdy or gelatinous nature, such as silver chloride, or the oxides of iron and chromium, may be almost completely shaken out of the funnel, direct into the crucible in which they are to be weighed. This easy separation of the dried precipitate from the paper is a material help to accuracy in obtaining the weight of the precipitate. Another method of drying, is to place the funnel in a tin cone, supported on a piece of wire gauze over a Bunsen burner, Fig. 18; but this method is open to the objection that it may cause charring of the paper, or fracture of the funnel, besides possessing the further disadvantage of causing the precipitate, by reason of the way it is heated, to adhere to the paper.

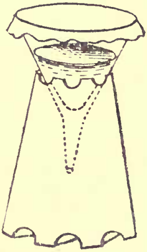


FIG. 18.

### *Ignition of the Precipitate.*

19. Since the precipitate is required to be in a perfectly dry state before it is in a fit condition for weighing, it is usually necessary to ignite it. The filter-paper itself is also burnt and converted into ash, and the weight of the resulting ash, which is usually extremely small, is subtracted from the total weight of the ignited precipitate and ash; it is therefore necessary to determine the average weight of the ash of the filter-papers employed, which is done as follows:—Six filter-papers are selected from different parts of a packet of filters; these are then carefully folded and burnt, one by one, to a perfectly white ash in a weighed platinum crucible, the crucible and ash being afterwards weighed. The weight of the ash thus obtained, divided by six, will give the amount of ash from one filter-paper. The vessels employed in the ignition of precipitates are crucibles

made of platinum, nickel, or porcelain. A platinum crucible is to be preferred whenever it is practicable to use it, as it is heated more rapidly and uniformly than one made of porcelain, and is better adapted to resist the action of a coal-gas flame than one made of nickel; but platinum cannot be used for the ignition of the chlorides of silver or lead, and some oxides, sulphides, and phosphides are liable to injure the metal also. The crucible is usually supported on a pipe-clay triangle, Fig. 19. The temperature to which a precipitate should be exposed, and the duration of the ignition, varies with the nature of the precipitate, but in all cases the heat should be applied gradually. This precaution is especially necessary when the substance is not perfectly dry, or is in the form of a very light powder, such as silicic acid.

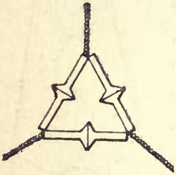


FIG. 19.

When heating precipitates which are capable of reduction, care must be exercised that the flame does not come into contact with the interior of the crucible, or possibly reduction of the substance may take place. A simple method of igniting precipitates requiring a comparatively low temperature, is to place the crucible upon a triangle, resting upon the chimney of a Wallace Argand burner minus the gauze top. When the gas as it comes from the ring at the bottom of the burner is lit, it causes a current of heated air to play against the sides of the crucible, and in this way the crucible may be uniformly heated to any temperature desired, up to a red heat. This method of heating is specially useful in the determination of calcium as $CaCO_3$. The ordinary method of ignition, however, is by means of a Bunsen burner, supplemented in some cases by the blow-pipe, the crucible being placed on a pipe-clay triangle, resting on the ring of an iron tripod, or retort-stand.

Precipitates may be ignited with, or apart from, the filter-paper, according as to whether the combustion of the filter-paper has any reducing action on the precipitate or not If the combustion of the filter-paper has no reducing action upon the precipitate, the latter may be ignited as follows. The filter and precipitate, first being thoroughly dried in the water oven, the filter-paper (which still contains the precipitate) is folded together, and the free edge turned down so as to enclose the precipitate in a small compass. The filter is then deposited in the crucible, and the lid placed on the latter so as to nearly cover it, leaving only a small space for the escape of the gases which are given off by the ignition and combustion of the filter-paper, Fig. 21. Heat is then applied, very moderately at the commencement. As soon as the flame due to the combustible gases ceases to appear, the lid may be removed, and the heat increased until the temperature required is obtained. When the filter-paper has been entirely converted into a white ash, the burner is taken away, and as soon as the crucible has cooled down a little, it is removed to the desiccator by means of a pair of clean crucible tongs. The lid of the crucible is then ignited until it is clean, placed upon the crucible, and the whole is weighed when cold.

It sometimes happens that the filter-paper is difficult to completely incinerate, black particles of carbon stubbornly remaining. In this event, the combustion may be accelerated by bringing the portions of unconsumed filter-paper into contact with the hottest portion of the crucible, by means of a stout platinum wire fused into a piece of glass tubing; or a small blow-pipe flame may be used, so as to produce a higher temperature. Another way of helping the combustion of the carbon, is to moisten the ash with a drop of a saturated solution of ammonium nitrate. Precipitates such as $Al_2O_3, 3H_2O$: or $Cr_2O_3, 3H_2O$; which have been washed and drained by means of the filter-pump, may be ignited without being previously dried in the water oven. In these cases, it is necessary to cover the crucible with its lid, until all the moisture and gases have been driven off, and to apply the heat very gradually, commencing with a small flame, placed at some distance below the crucible.

When the combustion of the filter-paper is liable to exert a reducing action on the precipitate, and also in cases in which it is desirable to keep the precipitate apart from the ash, the precipitate is ignited apart from the filter, in one of the following ways.

(*a*) The dry precipitate and paper are taken from the funnel, and the precipitate is carefully shaken out of the paper into the crucible standing on a piece of glazed paper, so as to catch any particles of precipitate which may fall outside the crucible. The glazed paper should be dark for light-coloured, and white for dark-coloured precipitates. The portions of the precipitate adhering to the filter are loosened by rubbing its sides together, care being taken that the surface of the paper is not destroyed by so doing, otherwise filaments of paper are liable to contaminate the precipitate, and may impair the accuracy of the result, by escaping being burnt in the subsequent ignition, or if burnt, may alter the composition of the precipitate. The precipitate, detached as above described, is then added to that already in the crucible. The filter-paper is then folded up as in Fig. 20, and afterwards rolled into a coil.

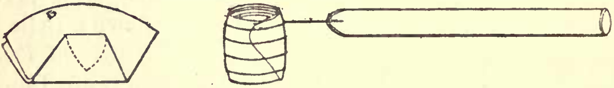


Fig. 20.

The paper should be folded in such a manner that the soiled half of the filter is in the centre; there is then less risk of loss from projection, or from the precipitate fusing to the platinum wire, which is wound round the paper in a spiral form. The platinum wire employed should be about 10 inches long and fairly stout, and, for convenience of handling, should be fused into a piece of glass tube.

The filter-paper is then ignited by means of a Bunsen burner, which is held in one hand, the paper being held in the other hand directly over the crucible standing on a piece of glazed paper. The paper is maintained in a glowing condition by being occasionally placed in the flame of the burner, until the carbon of the paper is entirely consumed. A slight tap of the platinum wire on the edge of the crucible will cause the ash to fall into the latter, when the ignition may be completed in the usual way by means of a Bunsen burner, at first gently with the lid on, and afterwards more strongly with the lid removed. The crucible is then placed in the desiccator and weighed when cold. It is then heated a second time and again weighed, to ascertain that its weight is constant.

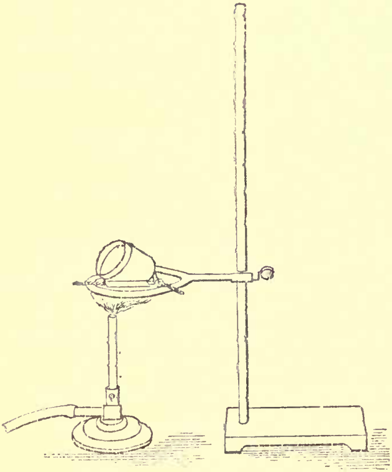


Fig. 21.

In every case before proceeding to use a crucible for the purpose of igniting a precipitate, it should first be heated, placed in the desiccator, and weighed, so as to obtain the weight of the empty crucible.

(*b*) Another method of igniting the filter-paper and precipitate, is to burn the paper on the lid, and the precipitate in the crucible, both being ultimately weighed together. The details of the method are as follows. The precipitate is shaken out into the crucible, the last traces being detached from the paper as completely as possible as in the previous method. The filter-paper is then cut up into small strips by means of a clean pair of scissors; these strips are then incinerated, one by one, on the inverted lid of the crucible, which is supported on a pipe-clay triangle. When all the strips have been burnt, the resultant ash is added to the precipitate in the crucible, and the ignition completed as already described.

As previously remarked, the intensity and duration of heat to which a precipitate should be exposed, depends on the nature and quantity of the precipitate, but as a general rule from five to ten minutes at a low red heat will be sufficient. A few words on the care of platinum vessels are necessary. Platinum crucibles, when red hot, should never be touched with brass tongs, or placed in brass rings, as these produce black stains on the metal. Should a platinum crucible become soiled, it may be cleaned by rubbing it with moist sea-sand, the particles of which, being rounded, will not scratch the metal. The nature of the sand should first be ascertained by examining a few grains of it through a magnifying glass; if they appear to be rounded, resembling pebbles, the sand is suitable, but if angular the sand should not be used. Should the stains not disappear on rubbing with sand, a little bisulphate of potassium may be fused in the crucible; this, when cold, is removed by boiling it with water, and the surface of the metal finally polished with sea-sand.

### *Collection of Precipitates on Weighed Filters.*

20. It occasionally happens that it is not possible to expel the moisture from a precipitate by ignition, before

weighing. It is therefore necessary, in such a case, to weigh the precipitate on the filter on which it has been deposited; consequently, we require to know the weight of the filter paper itself. The paper is folded in the usual manner, placed in a stoppered weighing tube, or between a pair of well-ground watch glasses with clip, Fig. 22, and heated in the water oven for an hour or so. The stoppered tube or watch-glasses containing the filter paper are then allowed to cool in the desiccator, and weighed when cold. The filter is then placed in the funnel, and the precipitate deposited on it in the usual manner, the tube or watch-glasses remaining in the balance case while this operation is proceeding. When ready for drying, the paper and precipitate are first of all dried in the funnel; the filter is then taken out of the same and placed in the weighing tube or between the watch-glasses, dried for some hours in the water oven, and repeatedly weighed until a constant weight is obtained.

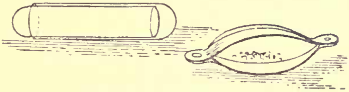


FIG. 22.

Another plan is to weigh two filters of equal size, A and B, against each other, and mark the difference in weight on B. The precipitate is collected on A, the filtrate and washings being allowed to pass through B; both are dried and weighed against each other, and the original difference in weight allowed for.

# PART II.

## SIMPLE GRAVIMETRIC DETERMINATIONS.

THE following simple gravimetric determinations are selected principally, with a view to their applicability to the estimations most frequently required in gasworks. In most of the examples, the substance selected for analysis is of known and definite composition. The accuracy of the result obtained can therefore be checked, by calculating from the formula of the substance, the percentage of the constituent present.

The substances recommended for analysis in these exercises, are generally soluble in water, but should a substance requiring estimation be found to be insoluble in water, it will be necessary then to ascertain, by experimenting on a small portion of the finely powdered substance, what are the most suitable solvents. These will usually be found to be either hydrochloric or nitric acids in the order named, or possibly a mixture of the two. If treatment with acids does not dissolve it, the substance may generally be decomposed by fusing it with about six times its weight of a mixture of sodium and potassium carbonates; it is then extracted with water, and the insoluble residue dissolved by treatment with acid.

21. In order to make the following exercises of any value, it is necessary that the substances chosen for the experiments should be quite pure; a few hints on the preparation of pure substances will therefore be of service.

The purity of a soluble and crystallisable chemical compound can usually be ensured by repeated crystallisation from a suitable medium. Owing to the fact that the impurities are left behind in the mother liquor by this mode of treatment, many commercial salts may be purified by one or two crystallisations from water. The following is the method of procedure in the case of some substances which are employed in the examples given for practice.

Potassium aluminium sulphate ($AlK(SO_4)_2,12H_2O$); copper sulphate ($CuSO_4,5H_2O$); barium chloride ($BaCl_2,2H_2O$); magnesium sulphate ($MgSO_4,7H_2O$).

The coarsely crushed commercial salt is dissolved in a beaker or evaporating basin, the contents of the vessel being heated and well stirred, until the solid no longer dissolves, so as to obtain a hot and nearly saturated solution of the solid. The hot liquid is then decanted from the excess of solid, into a crystallising dish. In the event, however, of any suspended solid matter remaining in the liquid, the latter must be passed through a filter-paper contained in a funnel, which is kept hot by means of a jacket of hot water. The apparatus employed to attain this result is shown in Fig. 23. It consists of a copper vessel, somewhat resembling a funnel in shape, and double walled. In communication with the interspace of the funnel-shaped portion of the apparatus is a sheet copper projection, *a*. The apparatus is provided with a handle for convenience of holding. The apparatus before being used is about half-filled with water. The water is then heated to boiling by placing a Bunsen burner beneath the projecting portion, *a*, which will be filled with water communicating with that in the jacket. In this way hot water will circulate throughout the copper jacket, and

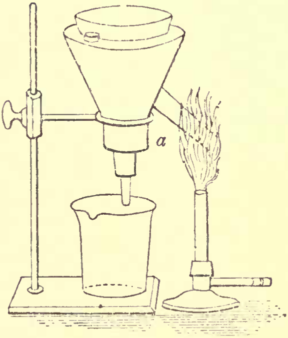


FIG. 23.

consequently, the funnel placed in it may be kept hot during filtration. After filtering, the clear hot solution (filtrate) is surrounded with a vessel containing cold water, so as to rapidly cool down the solution, and further the formation of small crystals. When the crystals have ceased to form, the supernatant liquid is poured off, and the crystals are placed upon porous tiles to drain. After draining for several hours, the crystals are broken up, and are finally dried by means of repeated pressure between folds of filter or blotting paper. They are then placed in a tight-fitting stoppered bottle.

22. Many chemicals may be prepared in a pure state by means of precipitation, and subsequent washing and drying.

The preparation of pure calcium carbonate is an example. Dissolve 100 grams of calcium chloride ($CaCl_2$) in 250 cc. of distilled water, heat the solution to boiling, filter if necessary, and then add a strong solution of pure ammonium carbonate $(NH_4)_2CO_3$, as long as any precipitate is thrown down. Pour the precipitate on to a filter-paper, and wash it with hot water until the latest washings cease to give a precipitate with a solution of nitrate of silver ($AgNO_3$). Then dry the precipitate in the water oven, detach it from the filter-paper, and preserve in a stoppered bottle.

23. The process of sublimation is sometimes employed for the purpose of separating volatile from non-volatile substances, the purification of such substances being in this way effected.

In order to purify a volatile substance, the sublimation may be effected by placing the substance in a porcelain crucible, and inverting over it a similar crucible. The lower crucible is then gently heated, when the substance will volatilise and condense in the upper one, which must be replaced by a cold one if it becomes too hot.

Another method is to place the substance into a porcelain dish, and condense its vapour by means of an inverted funnel, placed so as to cover the substance. Unless the substance which is to be sublimed is known to be quite dry, it is advisable to allow the first portion of its vapour to pass away, and carry off any moisture there may be in the substance with it.

The sublimation of iodine is an example of the first method of purification by means of sublimation.

Partly fill a porcelain crucible with a mixture of three parts of iodine and one part of iodide of potassium (KI), finely powdered, and well mixed together. This is then covered with a second crucible, and a gentle heat applied to the lower crucible. At the close of the operation the sublimed crystals are removed and stored in a clean dry bottle.

The sublimation of arsenious oxide ($As_2O_3$) is an example of the second method of purification by means of sublimation.

Place in a small porcelain dish a thin layer of $As_2O_3$. Then invert over it a clean glass funnel, and gently heat the dish. The sublimed $As_2O_3$ is detached from the funnel at the end of the operation and preserved for future use.

### ESTIMATION OF $SO_4$ IN A SULPHATE (SOLUBLE).

24. *The* $SO_4$ *is precipitated as barium sulphate and weighed in this form.*

The manipulation necessary in the determination of the above, finds frequent application in the analytical work required in gasworks.

The determination of the amount of sulphur in coal and coke, the sulphur compounds in purified gas, the testing of the solutions employed in the Referees' ammonia test, and in the testing of ammoniacal liquor and sulphate of ammonia, are typical instances.

The substance chosen for the estimation is copper sulphate ($CuSO_4,5H_2O$).

In order to obtain the substance in a state of purity, it is necessary to purify it by recrystallisation (21); but even this treatment is not always sufficient to purify the commercial salt (blue vitriol); as it not unfrequently contains ferrous sulphate, which cannot be removed even by repeated crystallisation, as the two sulphates tend to crystallise, together. By heating the solution, with a few drops of nitric acid, however, the ferrous salt is oxidised to ferric sulphate, and on concentrating the liquid, crystals of pure copper sulphate are easily obtained. In order to prepare a quantity of the pure salt, about 200 grams of clean, well-formed crystals of commercial blue vitriol are dissolved in about a quarter of a litre of hot water, a few drops of nitric acid are added, the solution is filtered if necessary, and boiled for half an hour; on cooling, the liquid deposits crystals of pure $CuSO_4$. After standing for a few hours the solution is poured off, and the mother liquor is drained as far as possible from the crystals. The crystals remaining are then broken up by means of a glass rod, and dried by pressure between folds of filter or blotting paper. It is advisable not to press the crystals with too great a force, as the sulphate may consequently become mixed with filaments of filter-paper, which would interfere with the accuracy of the after analytical operations. When the greater portion of the moisture has in this way been got rid of, the salt is wrapped in a fresh sheet of dry filtering paper, and the folds are placed under a heavy weight for an hour or two. When dry the salt is placed in a stoppered weighing tube. About one gram of the dried salt is now weighed out in the following manner. The stoppered weighing tube containing the $CuSO_4$ is carefully weighed, and about a gram of the salt is shaken out into a clean dry beaker of about 18 ounces capacity. On replacing the stopper and again weighing the tube, the loss of weight resulting, gives the exact amount taken for analysis. Care must of course be taken that all the salt shaken out of the tube is deposited in the beaker.

The salt is now dissolved in 30–40 cubic centimetres of distilled water, a few drops of hydrochloric acid are added, and the solution, covered with a clock-glass, is heated to boiling. The burner is then removed from underneath the beaker, and a saturated solution of barium chloride added drop by drop. In order to ascertain whether an excess of the precipitant has been added, the barium sulphate is left to settle down, and when the supernatant liquid is sufficiently clear, a drop of the barium chloride solution is poured down the side of the beaker. If an increased turbidity ensues, the liquid is again heated, and a further quantity of barium chloride solution added; the precipitate is once more allowed to settle, and the liquid again tested by cautiously adding barium chloride solution. When it is quite evident that the precipitation is complete, cover the beaker and set it aside in a warm place for the precipitate to settle. If it is attempted to filter the turbid liquid immediately, the finely divided precipitate will inevitably pass through the pores of the paper, but on standing, especially after precipitation from a hot solution slightly acidified with hydrochloric acid, the barium sulphate becomes denser, and assumes a granular form. When the precipitate has completely settled, add one more drop of barium chloride to be quite certain that the precipitation is complete, and proceed to collect the $BaSO_4$ on a filter; the supernatant liquid is first poured through, care being taken not to disturb the precipitate. The precipitate is then washed twice by decantation through the filter, and finally transferred to the latter by means of the wash-bottle with a fine jet, and a glass rod tipped with india-rubber tube. The precipitate is washed free from chloride, as shown by a portion of the latest washings not giving a precipitate with a solution of nitrate of silver.

When thoroughly washed, the filter and precipitate are dried in the water oven, and, when dry, the precipitate is ignited, apart from the filter, in the following manner. The precipitate is first detached as completely as possible from the filter, and placed in a platinum crucible. The filter is then folded up with the soiled surface in the middle, and bound round by a spiral of platinum-wire, and burnt to a white ash in the flame of a Bunsen burner. The ash is then added to the precipitate in the crucible, and the whole is ignited for about fifteen minutes at a dull red heat. It is then allowed to cool in the desiccator and weighed. The precipitate is ignited again for ten minutes, then allowed to cool, and weighed; this operation being repeated until the weight is constant.

From the weight of the $BaSO_4$ thus obtained, the percentage of $SO_4$ may be calculated in the manner described below.

*It is necessary to note that any* $BaSO_4$ *adhering to the filter paper will be reduced to* BaS. *If this is present in any appreciable quantity it will be necessary to reconvert it into* $BaSO_4$ *by the following procedure. The filter-ash which has been dropped into the crucible is moistened with two drops of dilute* HCl, *and one drop of dilute* $H_2SO_4$. *The contents of the crucible are then very gently heated until the excess of acid has been evaporated, great care being taken to avoid loss by spurting. The crucible and its contents are then strongly ignited.*

The following shows the method of entering the results, and making the necessary calculations.

*Example.*—Determination of the amount of sulphuric acid in copper sulphate, by precipitation as barium sulphate.

| | Grams. |
|---|---|
| Weight of tube + $CuSO_4$ (before) .. .. .. | 9·7612 |
| ,, ,, (after) .. .. .. .. | 8·6066 |
| Weight of $CuSO_4$ taken .. .. .. .. .. .. | 1·1546 |
| Weight of crucible + $BaSO_4$ + ash .. .. .. | 17·6024 |
| ,, alone .. .. .. .. .. .. .. | 16·5216 |
| | 1·0808 |
| Weight of filter ash .. .. .. .. .. .. .. .. | ·0022 |
| ,, $BaSO_4$ .. .. .. .. .. .. .. .. | 1·0786 |

When the weight of a precipitate has been ascertained, it is necessary to calculate that of the constituent the weight of which we wish to learn; this is done by the help of the atomic weights given in the Appendix.

In the present example, we first require to know the amount of $(SO_4)$ which is equivalent to 1·0786 grams, $BaSO_4$.

The molecular weight of $BaSO_4 = 233$ ($Ba = 137$, $S = 32$, $O = 64 = 233$), and this contains 96 parts by weight of $SO_4$ ($S = 32$, $O_4 = 64 = 96$).

Then, as $233 : 96 :: 1·0786 : x$. $x = 0·4444$ gram $SO_4$.

We thus have 0·4444 gram $SO_4$, from 1·1546 grams $CuSO_4,5H_2O$, so, to obtain the percentage, we say—

As $1·1546 : 100 :: 0·4444 : x$. $x = 38·49$ per cent.

That is, we have found 38·49 per cent. of $SO_4$ in the $CuSO_4$ $5H_2O$ operated on.

We have now to see if the analysis is correct or not, by checking it against the numbers demanded by theory.

The molecular weight of $CuSO_4,5H_2O = 249·5$ ($Cu = 63·5$, $S = 32$, $O_4 = 64$, $5H_2O = 90 = 249·5$), and this contains 96 parts by weight of $SO_4$, consequently.

As $249·5 : 100 :: 96 : x$. $x = 38·48$ per cent., hence the error in the estimation $= -0·01$.

A difference of 0·2 per cent. from the numbers demanded by theory, or from the result of a duplicate analysis, is the mean maximum error which is usually allowed in the case of simple quantitative estimations.

## ESTIMATION OF BARIUM IN A SOLUBLE COMPOUND.

25. Inasmuch as the estimations involving the same methods of manipulation as required in the previous example, are amongst those most frequently occurring in gasworks, it will be advisable to gain further experience in the

same, by estimating the amount of barium in a soluble salt, the operations being almost identical with those described in (24).

*The barium is precipitated as barium sulphate, and weighed in this form.*

Weigh out from a weighing bottle about 1 gram of pure recrystallised barium chloride ($BaCl_2,2H_2O$) into an 18-ounce beaker; dissolve the salt in about 100 cc. of distilled water to which a few drops of dilute hydrochloric acid have been added. Heat the solution to boiling, and add an excess of dilute sulphuric acid; keep up a boiling heat until the precipitate becomes compact and readily settles down. Allow the precipitate to settle down completely, decant the clear liquor through a No. 5 Swedish filter, wash the precipitate twice with hot water by decantation, filter, and finish the washing on the filter. The last washings must give no precipitate with $BaCl_2$ solution. Dry the precipitate in the water oven, and ignite it as described under the estimation of ($SO_4$). (24.)

From the weight of $BaSO_4$ thus obtained, the percentage of Ba may be calculated.

The following expresses the reaction:—

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

Consequently every 233 parts of $BaSO_4$ are equivalent to 137 parts of Ba.

## ESTIMATION OF ARSENIC AS ARSENIOUS SULPHIDE.

26. *This method is applicable to the determination of sulphuretted hydrogen in gas liquor or in crude coal gas.*

Weigh out accurately a little more than half a gram of pure resublimed arsenious oxide, place it in an 18-ounce flask, and add about 50 cc. of dilute hydrochloric acid. Heat the flask on a water bath until all the oxide is dissolved, being careful not to allow the temperature of the solution to attain 100° C., or some of the arsenic will be volatilised as chloride. A few drops of sulphurous acid solution are then added, in order to convert any $As_2O_5$ that may be present into $As_2O_3$, and the heating on the water bath is continued until all smell of $SO_2$ has ceased. The arsenic is then precipitated as $As_2S_3$, by passing $SH_2$ gas through the solution in the following manner. The flask containing the solution of $As_2O_3$ is provided with a doubly perforated india-rubber cork, through one of the holes in which a glass tube passes, bent at right angles, and reaching nearly to the bottom of the flask. A glass tube passes through the other hole also, one end of this tube terminating just below the bottom of the cork, the other end being slightly contracted in order to prevent air from diffusing into the flask. Sulphuretted hydrogen gas is then passed through the first tube into the liquid in a gentle stream, until the liquid is saturated with the gas. The flask is then left for some hours in a warm place, in order to ensure complete precipitation. In very accurate experiments, $CO_2$ gas is next passed through the liquid until the excess of $SH_2$ is removed. During the passage of the $CO_2$, the liquid is gradually heated to boiling; this makes the precipitate more dense, and consequently more easy to wash.

If care is taken that the precipitated $As_2S_3$ is kept covered, and is not much exposed to the air, the passage of $CO_2$ may be omitted. Filter through a double tared filter which has been dried at 100° C. (20), and wash the precipitate with hot water containing a little $H_2S$, until it is free from HCl. Test the filtrate and washings to see if all the As is precipitated, by saturating the liquid with $H_2S$ gas, and allowing it to stand. If any further precipitate is thrown down, add it to the main portion on the filter.

Dry the precipitate at 100° C. in the water oven until the weight is constant. Since the precipitate may contain a little free sulphur, it is necessary to treat it with carbon bisulphide in the same manner as described under cadmium (27). After this treatment, the precipitate is again dried at

100° C., and is finally weighed. From the weight of the $As_2S_3$ obtained, the percentage of As is calculated.

$$\text{As}\quad \overset{As_2S_3}{246} : \overset{As_2}{150} :: \text{weight of sulphide obtained} : \text{weight of arsenic,}$$

198 parts of $As_2O_3$ should yield 246 parts of $As_2S_3$.

## ESTIMATION OF CADMIUM, AS CADMIUM SULPHIDE (CdS).

27. *This method may be employed in the estimation of* $SH_2$ *in crude coal gas by means of cadmium chloride.*

Weigh out accurately about 1 gram of pure recrystallised cadmium sulphate ($CdSO_4,4H_2O$). Dissolve it in about 250 c.c. of distilled water, and add a few drops of dilute HCl. Pass a current of $SH_2$ into this solution, until it smells strongly of the gas. Filter off the precipitated cadmium sulphide, making sure that all the cadmium has been precipitated, by diluting a portion of the filtrate and passing $SH_2$ through it again. If no further precipitate is thrown down, collect on a tared filter (20) and wash the precipitate, first with diluted sulphuretted hydrogen water mixed with a little HCl, and finally with pure water. Dry the filter and precipitate in the water oven, and weigh. The precipitate sometimes contains free sulphur. In very accurate determinations, therefore, the dried precipitate should be repeatedly washed with carbon bisulphide, as long as any residue is observed on evaporating a few drops of the washings upon a clean watch-glass. The precipitate, after this treatment, is again dried and weighed, any difference between the two weighings being due to the removal of free sulphur. From the weight of CdS obtained the percentage of cadmium may be calculated.

$$\text{As}\quad \overset{CdS}{144} : \overset{Cd}{112} :: \text{weight of sulphide obtained} : \text{weight of cadmium.}$$

### Estimation of Calcium.

28. *This method is applicable to the determination of lime. The calcium is precipitated as calcium oxalate, and weighed either as carbonate or as oxide.*

Weigh out accurately into an 18-ounce beaker about 1 gram (not more) of pure calcium carbonate. Moisten it with distilled water, and dissolve it in dilute HCl. The beaker should be covered with a clock-glass, and the HCl added very cautiously, so as to prevent loss by the effervescence set up by the addition of the acid. The resulting solution is then diluted with distilled water to about 100 c.c., and the liquid heated nearly to boiling; a slight excess of solution of ammonia is then added, and finally a moderate excess of ammonium oxalate solution, after adding which the liquid is allowed to stand. As soon as the precipitate has completely subsided, pour off the supernatant liquid through a filter, being careful not to disturb the precipitate. Wash the precipitate in the beaker with hot water two or three times by decantation; and finally transfer the precipitate to the filter by means of a glass rod tipped with india-rubber tubing, and the jet of the wash-bottle. The washing of the precipitate on the filter is then continued, until the last portion of the wash-water no longer turns a solution of nitrate of silver milky. When that stage is reached, the filter and precipitate are dried in the water oven. The next operation is to convert the precipitate of calcium oxalate into calcium carbonate. The precipitate is transferred, as completely as possible, to a platinum crucible, and the filter-paper burnt in a platinum wire spiral (19*b*), the ash also being allowed to fall into the crucible. The crucible is then heated with the lid on, very gently at first, and finally to very faint redness for ten minutes. The bottom of the crucible must only appear of a faint red colour when shaded from direct light.

The crucible is then placed in the desiccator, and when cold weighed. During the process of ignition of the above precipitate, especially if it is at all over-heated, a portion of the calcium carbonate is often converted into calcium oxide (CaO); hence, after the first weighing, the contents of the crucible should be treated with a little strong solution of ammonium carbonate, which converts any CaO back again into $CaCO_3$. It is then placed again in the water oven to dry, heated to faint redness for a few minutes, allowed to cool, and weighed once more. These operations are all repeated, until the weight no longer increases by the treatment. From the weight of $CaCO_3$ finally obtained, the percentage of calcium may be calculated.

| | Mol. weight of $CaCO_3$ | | Atomic weight of calcium | | | | |
|---|---|---|---|---|---|---|---|
| As | 100 | : | 40 | :: | weight of $CaCO_3$ obtained | : | weight of calcium. |

The precipitate of calcium oxalate is, however, more readily converted into CaO than into $CaCO_3$, especially if it is small in amount, say not much more than 1 gram.

In order to convert the precipitate into CaO, the crucible and its contents are heated to bright redness for 15 minutes over the blow-pipe flame. The crucible is then allowed to cool, and weighed, and then heated again for 5 minutes in the same way, cooled, and weighed. These operations are then repeated, until the crucible ceases to lose weight. The weight of substance finally obtained when the weighings are steady, is that of the calcium oxide (CaO) plus the filter ash. From the weight of CaO thus obtained, the percentage of calcium may be calculated.

| | Mol. weight of CaO | | Atomic weight of Ca | | | | |
|---|---|---|---|---|---|---|---|
| As | 56 | : | 40 | :: | weight of CaO obtained | : | weight of calcium. |

## ESTIMATION OF IRON.

29. *This method of determination is applicable to the analysis of Bog Ore.*

*The iron is precipitated as ferric hydrate* ($Fe_2O_3, 3H_2O$). *This is then ignited and weighed as ferric oxide* ($Fe_2O_3$).

Weigh out accurately about 1·5 grams of pure ferrous ammonium sulphate $Fe(NH_4)_2 (SO_4)_2 6H_2O$ into an 18-ounce beaker, or preferably into a platinum or porcelain basin. Dissolve this in about 200 cubic centimetres of distilled water, containing a few drops of dilute sulphuric acid. Heat the solution with sufficient strong nitric acid to convert the iron, which originally was in the ferrous form, into the ferric state. This change may be known to be nearly effected when the further addition of a drop of strong nitric acid does not create any brown colour in the solution; but the change to the ferric condition should be proved to be complete by adding a drop of the dilute solution to a drop of a freshly made solution of potassium ferricyanide, placed on a white porcelain tile, when no blue colour should appear. While the solution is still hot, add an excess of solution of ammonium hydrate, and boil. The precipitate is washed once by decantation by boiling it with water, and is then transferred to a filter, the process of filtration being accelerated by the use of the filter pump.—(17) The washing is continued until the latest washings do not give any precipitate when boiled with a small quantity of $BaCl_2$ solution.

The precipitate is dried in the water-oven and transferred to a crucible. The filter is burnt apart from the precipitate, being added to the latter, which is then ignited at a bright red heat over a Bunsen burner, until a constant weight is obtained. From the weight of $Fe_2O_3$ obtained, the percentage of iron may be calculated.

*Of course, if the iron is already in the ferric condition it will not require the preliminary treatment with* $HNO_3$, *but may be precipitated at once with* AmHO *solution. It is necessary to note that the presence of certain non-volatile organic substances prevents the complete precipitation of iron from its solutions by* AmHO. *The organic substance should therefore be completely destroyed before precipitating with* AmHO, *by adding carbonate* *of soda and nitrate of potash to the solution, evaporating to dryness, and fusing the residue. (If the iron is in the solid state, the organic matter may be at once got rid of by ignition at a red heat.)*

*The iron is then dissolved out by heating with dilute* HCl, *or a mixture of* HCl *and* $HNO_3$, *and the filtered solution treated as above.*

$$\text{As}\ \overset{Fe_2O_3}{160} : \overset{Fe_2}{112} :: \text{weight of precipitate} : x = \text{weight of Fe.}$$

## Estimation of Alumina ($Al_2O_3$).

30. *The estimation of* $Al_2O_3$ *occurs in the analysis of Bog Ore and in fire-clay.*

*The* $Al_2O_3$ *is precipitated by* AmHO *as* $Al_2O_3, 3H_2O$. *This is converted into* $Al_2O_3$ *by ignition, and is weighed in this form.*

Weigh out accurately about a gram and a half of pure recrystallised potash alum $AlK(SO_4)_2 12H_2O$ into a platinum or porcelain dish. Dissolve the salt in about 150 c.c. of distilled water, add a solution of ammonium chloride in moderate quantity, and afterwards a slight excess of ammonium hydrate solution (AmHO). A white gelatinous precipitate of ($Al_2O_3, 3H_2O$) will form. Cover the vessel with a clock-glass, and bring the solution to the boiling point, continuing the boiling, until the liquid has only a faint smell of ammonia. Wash the precipitate by decantation and transfer it to a filter, employing the filter-pump to accelerate filtration. Continue washing the precipitate on the filter until it is free from dissolved sulphate. Dry in the water-oven, and ignite the precipitate with the filter-paper in a platinum crucible. The crucible should be heated gently at first, and gradually brought to a white heat over the blowpipe flame. It should then be cooled and weighed, the ignition being repeated until a constant weight is obtained.

From the weight of precipitate we can get the percentage of aluminium, but as a general rule aluminium is generally shown in analyses as combined with oxygen, in the form of alumina $Al_2O_3$.

### SEPARATION OF IRON AND ALUMINA.

31. Weigh out into a platinum basin about one gram of potash alum and half a gram of pure ferrous sulphate. Dissolve in a little distilled water, add hydrochloric acid and a few drops of nitric, and boil, in order to convert the iron into the ferric state. Then dilute to about 20 c.c. and add sufficient pure potash to produce a strong permanent precipitate. Now add excess of strong caustic potash, and keep at a boiling heat, constantly stirring with a platinum rod to prevent actual boiling, until the $Al_2O_3$ can be assumed to have passed into solution, and dilute, first as far as possible in the platinum basin, then by instalments, more largely, in a Berlin ware basin provided with a spout, and filter off the ferric hydrate ($Fe_2O_3,3H_2O$). Wash the precipitate thoroughly with hot water; the result is that substantially the iron and alumina are separated from each other. In general, however, what should be ferric oxide retains a more or less considerable portion of the alumina. It is necessary, therefore, to extract this alumina by redissolving the precipitate with hydrochloric acid, and repeating the treatment with potash. The crude ferric hydrate obtained cannot be ignited and weighed as it is, as it contains a certain amount of combined fixed alkali (KHO), which it carries down with it in the act of precipitation. It is requisite to dissolve it in hydrochloric acid, re-precipitate it by ammonia, and ignite it as in (29). From the alkaline liquors precipitate the $Al_2O_3$, by adding an excess of $NH_4Cl$, and heat the mixture until the $NH_3$ liberated is practically expelled, then treat the precipitate as in (30). For a check, determine the iron and alumina conjointly by precipitating with $NH_3$ and $NH_4Cl$.

## ESTIMATION OF MAGNESIUM.

32. Weigh out about one gram of pure, dry, crystallised magnesium sulphate, place it in a half-pint beaker, and dissolve it in about 50 c.c. of distilled water. Add enough solution of ammonium chloride to prevent the precipitation of any magnesia on adding ammonia (about one gram of solid $NH_4Cl$ will be sufficient), the ammonia being added in considerable excess. A solution of phosphate of soda is next added (about 1·5 of crystallised phosphate of soda dissolved in water) as long as it appears to produce a precipitate, and the solution is briskly stirred with a glass rod, but without rubbing the sides of the beaker; the glass rod may be left in the beaker. When the precipitate has subsided, a few drops more sodium phosphate are added to the clear liquid, in order to be quite sure that no further precipitate is produced. The beaker is now covered, and left for about twelve hours. The precipitate is then collected upon a filter, of about 4 inches in diameter before being folded, and is rinsed on the filter with distilled water containing one-eighth of its bulk of strong ammonia, the same solution being subsequently employed for washing the precipitate, the washing being continued until the washings, after being acidulated with nitric acid, give only a very slight turbidity with a solution of nitrate of silver.

The filter is then dried with the precipitate, and when dry, as much of the precipitate as possible is transferred to a platinum crucible, which is then gently heated (the cover being loosely placed on) and finally ignited at a red heat. In this way, the precipitate of phosphate of magnesium and ammonium ($MgNH_4PO_4$) is converted into magnesium pyrophosphate ($Mg_2P_2O_7$), which is allowed to cool, and weighed. It may then be emptied out of the crucible (preserving it for subsequent examination if the result should not prove correct) and the filter may be incinerated by igniting it, and allowing the ash to drop into the crucible containing the precipitate. When cool, the crucible is again weighed, when the total weight of ($Mg_2P_2O_7$) will be obtained. (The above method of incineration is recommended on account of the filter-paper in this case being very difficult to burn, from its retaining traces of phosphate, and it can scarcely be heated sufficiently to burn off the carbon on the top of the mass of precipitate.)

The amount of magnesium is calculated by the following proportion:—

| Mol. weight of magnesium pyrophosphate | | Two atomic weights of magnesium | | Weight of ignited precipitate | | Weight of magnesium found |
|---|---|---|---|---|---|---|
| 222·6 | : | 48·6 | : : | | : | *x*. |

### ESTIMATION OF SILICA IN SILICATES.

33. *This method of estimation is used in the analysis of fire-bricks and fire-clay.*

*The silica is converted into the insoluble form by evaporating the silicate to dryness with excess of acid. It is then rendered completely anhydrous by ignition, and weighed as silica* ($SiO_2$).

If a silicate is insoluble in acids it is first fused with six times its weight of fusion mixture ($Na_2CO_3 + K_2CO_3$) in a covered platinum crucible until all effervescence ceases; the fused mass is then separated from the crucible by boiling with hot water in a covered evaporating basin, HCl being added if required, and then treated as below.

If the silicate is soluble in water, or is capable of being decomposed by strong hydrochloric or nitric acids, it may be treated at once as below.

A suitable substance for an experimental determination is soluble glass, or sodium silicate, which is one of the soluble silicates, and consequently will not require the operation of fusion.

Weigh out accurately into a platinum basin about one gram of soluble glass in very fine powder, and moisten the powder with a little distilled water. Having covered the basin with a clock-glass, place it upon a water-bath, and gradually add strong HCl, at the same time keeping the contents of the basin constantly stirred up by the aid of a glass rod rounded at the end. When the powder is completely decomposed, which is known to be the case as soon as no gritty particles are felt with the glass rod, remove the clock-glass, and continue the heating until the liquid is evaporated to dryness. The mass should be continually stirred the whole time, any lumps which form being broken up by means of the glass rod. When the powder appears to be perfectly dry it may be removed to a sand-bath. It is then gently heated with a small Bunsen flame until, on placing a cold clock-glass on the basin for a few seconds, there is no appearance of any moisture on the clock-glass. All the silica will now be insoluble. When the contents of the dish are cold, moisten the powder with strong HCl, and warm it upon the water-bath; then add hot distilled water, stir the mixture well, allow the solid portion to settle, and decant the liquid through a filter. This treatment of the residue is repeated three times. The residue itself is then transferred to the filter, washed thoroughly with hot distilled water, and dried in the water-oven. The silica is next detached from the filter as completely as possible, and the filter is incinerated in a coil of platinum wire, and added to the precipitate. The crucible, covered with its lid, is then gently heated, care being taken that none of the fine, light silica is lost by being carried off with the escaping moisture; the best way of preventing this being to apply heat to the crucible very gradually, and only after some little time, to increase the temperature until the crucible attains a bright red heat, at which it must be kept for about a quarter of an hour. The crucible is then cooled in the desiccator, and when cold weighed in the usual manner, being re-heated and weighed, until a constant weight is obtained. The percentage of $SiO_2$ may then be calculated. In all determinations of silica, it is necessary to test the $SiO_2$ in order to see if it is pure. The ignited silica is heated with about 50 c.c. of a saturated solution of sodium carbonate for about 30 minutes. The silica, if pure, will then have completely dissolved. Should any residue remain, this is again treated with sodium carbonate solution, and if now there should still a residue remain, it should be washed, dried, and weighed. This weight, subtracted from the weight of the silica obtained in the first instance, will give the corrected weight of the silica. The dried residue is then fused with alkaline carbonates, the mass when cool is treated with HCl, and the $SiO_2$ is separated and estimated as above described.

The weight of $SiO_2$ thus found should be added to the corrected weight obtained above.

From the total weight of $SiO_2$, the percentage of $SiO_2$ may be calculated.

### ESTIMATION OF $CO_2$ IN A CARBONATE.

34. *Two methods of estimating the above are given. The first method is by weighing the* $CO_2$ *evolved on treating the carbonate with an acid; the second by finding the loss of weight which the carbonate suffers by the removal of* $CO_2$. *These methods may be employed in the testing of the fresh and spent lime met with in gasworks.*

(1) By direct weighing. The $CO_2$ is driven off on treating the carbonate with an acid, and then absorbed by means of soda lime in U tubes and weighed.

Pure calcium carbonate, or crystals of Iceland spar, may be employed in the estimation.

The apparatus used is shown in Fig. 24.

A conical flask *a*, having a capacity of from 100 to 150 cubic centimetres, is fitted with an india-rubber cork pierced with two holes. Through one of the holes a 50 c.c. pipette *b* passes, this being filled with dilute HCl for the decomposition of the carbonate before the experiment is started. The lower end of the pipette should descend to within about 1 inch from the bottom of the flask, and be drawn out to a fine point. To the other end of the pipette a piece of india-rubber tubing, about 8 or 9 inches long is attached, and this connects the pipette with the U tube *c* filled with soda lime. This U tube is open to the atmosphere at one end, and is intended to remove the $CO_2$ from the air, when it is required to aspirate the same through the apparatus. The india-rubber tube is provided with a screw pinch-cock, so as to open or close the connection with the U tube *c* at pleasure. Through the other hole in the cork of the flask a piece of glass tubing, bent at right angles, is inserted to the depth of about an inch. This serves to connect the outlet of the flask with the remaining portions of the train of apparatus, consisting of bulb and U tubes, connected by means of india-rubber tubing as shown. These are intended for drying, purifying, and absorbing the $CO_2$ gas given off in the flask *a*. The bulb tube *d* contains strong sulphuric acid, and its object is to free the gas from moisture; but it is also useful in denoting the speed at which the gas or air issues from the flask *a*. Liebig's potash bulbs answer

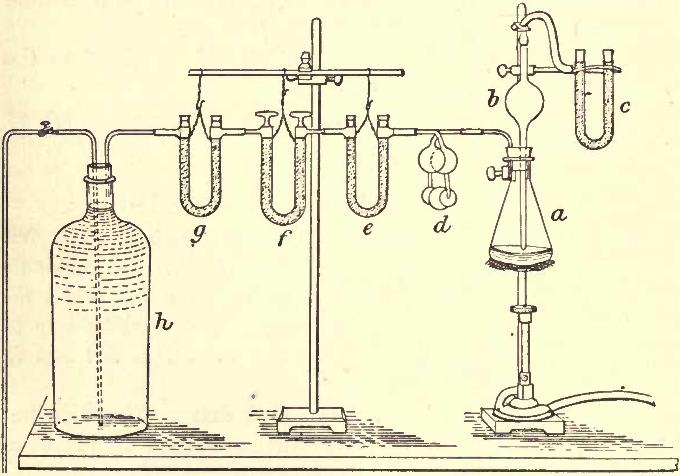


FIG. 24.

well for the purpose. The acid should about half fill the three bottom bulbs. The U tube *e* has the limb nearest to the bulb tube filled with fragments of calcium chloride, the other limb being filled with fragments of pumice impregnated with dehydrated sulphate of copper, in order to absorb the vapours of HCl given off during the heating of the flask *a*. The stoppered tube *f* is the one intended for the absorption of the $CO_2$ from the carbonate, and has the limb next to *e*, and half the other limb, filled with crushed soda-lime, the remaining portion of the limb being filled up with fragments of calcium chloride. During the process of absorbing $CO_2$ the soda-lime becomes heated, and in consequence moisture is evolved, this is taken up by the calcium chloride in the upper part of the limb, otherwise loss of weight would occur by reason of the escape of moisture. The remaining tube *g* is filled with fragments of calcium chloride, and is connected with a water aspirator *h*, which is employed to draw a current of air through the flask and tubes at the end of the experiment, so as to cause all the $CO_2$ to be sent forward to the absorption tube *f*.

The U tubes employed may be of two kinds, those not requiring to be weighed being of the form shown in Fig. 25. They are prepared for use as follows. In the first place they are washed perfectly clean with ordinary water, and finally rinsed with distilled water. They are then dried in the water-oven, and when quite dry they are ready for filling. Before filling them, however, it is necessary to see that the absorbent is in a proper condition, by crushing any large lumps and by rejecting any fine powder that may be present. This is necessary for the reason that as the absorbent takes up the gas by surface contact, the smaller the lumps, the larger is the absorbent surface presented to the current of the gas. On the other hand, if the ab-

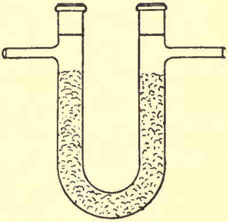


FIG. 25.

sorbent were in the state of fine powder, this would more or less completely fill up interstices, and in this way impede, or even entirely stop the passage of the gas. An absorbent may be prepared in a proper condition for use in the following manner. The substance is first coarsely powdered in a porcelain mortar, and the resultant powder is shaken upon a fine brass wire sieve, of about twenty strands to the inch, the sieve being perfectly clean and dry. The portion passing through the sieve is rejected; the remainder is then passed through a coarser sieve of only eight strands to the inch, so as to separate out the larger lumps. The smaller particles which pass through this sieve may then be used for filling the absorption tubes, and the larger lumps remaining behind are broken up again in the mortar, and again sifted as above. The U tube is then filled with the absorbent up to the level of the side tubes, and a loose plug of cotton-wool placed on the top, so as to prevent small particles of the absorbent from being carried through the side tubes by the issuing gas current. Two sound corks are then selected so as to tightly fit the open ends of the U tubes, and when fitted into the tubes, the top of the cork is cut off level with the top of the tube; finally the top of the cork is coated with melted sealing wax, so as to make it perfectly gas-tight.

When the absorbent is in the tube, it is most essential to protect it from contact with the atmosphere, and this may be effected by pushing over the side tubes short pieces of india-rubber tubing, closed with small pieces of glass rod. These plugs are only removed when the U tubes are being connected up for use, or while they are being weighed. Tubes *c* and *g* are filled in the manner above described.

The copper sulphate on pumice contained in the tube *e* is prepared in the following manner.

Lumps of pumice are broken up and sifted as just described, and the sifted particles are then heated with a moderately strong solution of copper sulphate. The excess of liquid is poured off, and the pumice, which is now thoroughly saturated with the solution, is heated in an air-oven to a temperature of 200 C., until dehydrated, as shown by the blue colour of the salt disappearing. It may then be used for partially filling the tube *e*.

For the absorption tube *f*, which contains soda-lime for the purpose of taking up the $CO_2$, as this tube is weighed, it will be more convenient to use the modern form of stoppered U tube. These stoppered tubes differ from those ordinarily employed, by having a hollow stopper, ground into the upper part of each limb. At the side of the hollow stopper, and on a level with the opening of the side tube, is a perforation, so that by simply turning the stopper round, communication between the U tube and the side tube may be opened or closed as required; and as the stoppers, when greased with a little resin cerate or vaseline, close the tube quite tightly, contact of the absorbent with the atmosphere may be prevented by simply turning the stopper; consequently it is not necessary to cover the side tubes, as in the ordinary form of U tube. A plug of cotton-wool is loosely inserted in these stoppers, so as to prevent small particles of the fragments from being carried out of the tube. The soda-lime for filling the tube *f* may either be purchased, or prepared in the following manner. Slake 200 grams of the best flare lime to a fine powder. Add 400 cubic centimetres of syrupy caustic soda solution. Make into a paste, dry quickly in a large copper dish protected from the access of $CO_2$, and granulate by constant stirring.

The tube is then filled with this material in the manner already described. The aspirator *h* may be improvised from a Winchester quart bottle in the following manner:—

A sound cork is selected to fit the neck of the bottle, and pierced with two holes. Into one of the holes a piece of glass tube, bent at right angles, is inserted, so as to just pass through the cork. Through the other hole, another glass tube bent at right angles passes, but in this case it passes to the bottom of the bottle. When in use, a piece of india-rubber tubing is attached to the longer glass tube, reaching below the level of the bottom of the bottle, in order that it may act as a syphon. At the bottom of the india-rubber tube there is a screw pinch-cock, in order to regulate the flow of the water. When required for use, the bottle is filled with water, and suction is applied to the syphon tube until it is also filled with water. As soon as this tube is filled, a continuous stream of water will continue to flow. After the suction is stopped air will replace the water in the bottle, and in this way a continuous current of air can be drawn through the train of apparatus when they are attached to the shorter tube in the bottle. The rate of aspiration is controlled by the screw pinch-cock. The next operation is to test the apparatus for soundness. The tubes and flask are connected up by means of short pieces of india-rubber tubing, the glass tubes "butting" against each other. To the end tube *g* a piece of glass tubing is attached, dipping into a beaker of water. Gentle suction is then applied at the outlet of the tube *c* until the water rises to about the height of 6 inches in the glass tube attached to *g*. The screw pinch-cock on *b* is then closed. If the level of the water in the glass tube does not alter during the space of ten minutes, we may safely conclude that the apparatus is air-tight, but if the level of the water in the tube falls the apparatus leaks somewhere.

The position of the leakage must be discovered and remedied by binding the leaky joints with fine copper wire, and, if necessary, replacing any defective india-rubber tubes with new ones. When all the joints have been made tight, the soda-lime absorption tube *f*, previously wiped clean and dry, is carefully weighed, and replaced in the train of apparatus. About 1 gram of calcium carbonate is then accurately weighed and placed in the flask *a*.

The pipette *b* is then filled by suction with dilute hydrochloric acid (1 : 3), the acid being retained in the pipette by the screw pinch-cock. The cork carrying the pipette is then inserted in the flask *a*, and is bound down to the neck of the flask, if necessary, by means of thin copper wire.

The aspirator and the U tube *c*, not being required until a later stage of the process, are not connected for the present.

The pinch-cock on the pipette is now slightly opened, so as to allow the acid to slowly fall on the $CaCO_3$ in the flask. Carbonic acid gas is given off, and drives forward the air in the flask through the train of apparatus. The bubbles of $CO_2$ should not pass through the bulbs containing sulphuric acid, at a greater speed than that at which the bubbles can be easily counted. When all the acid has passed out of the pipette into the flask, the pinch-cock on the pipette is closed, and as soon as gas has entirely ceased being evolved, the point of the pipette is gently pushed down beneath the surface of the liquid in the flask. The U tube *c*, and the aspirator *b*, are now connected to the rest of the apparatus, the pinch-cock on the pipette is opened, and the aspirator gently started. A small Bunsen burner is then placed under the flask *a*, so as to cause the contents of the flask to be in a state of incipient ebullition, in order to drive off all dissolved $CO_2$. The current of air drawn through by the aspirator replaces the $CO_2$ gas in the apparatus in front of the weighed soda-lime tube *f*, and carries it forward into the latter tube. The speed of the air passing through should be such that the bubbles can be counted as they pass through the drying bulb *d*, the speed being regulated by the pinch-cock on the syphon pipe leading from the aspirator. In order to expel the whole of the $CO_2$, a volume of air equal to about six times the capacity of the flask should be drawn through the apparatus. A guide as to whether the $CO_2$ has all been absorbed, may be found in the temperature of the soda-lime tube. At the commencement of the experiment it will be noticed that the soda-lime tube becomes very hot, owing to the heat generated by the chemical combination of the $CO_2$ and the soda lime; but as the absorption ceases, and air alone passes through, the tube becomes cool; hence, when the soda-lime tube attains its normal temperature again, the greater part of the $CO_2$ gas will have been absorbed. When sufficient air has been drawn through, the aspirator is shut off, and the stoppers of the soda-lime tube are turned so as to close the inlet tubes, the soda-lime tube is disconnected, and after standing for about half an hour is weighed. Any increase in weight over the weight at the commencement of the experiment is the amount of $CO_2$ given off from the calcium carbonate experimented on, and from these weights, the percentage of $CO_2$ in the $CaCO_3$ can be calculated.

(2) Estimation of $CO_2$ by difference. The $CO_2$ gas is expelled by treating the carbonate with an acid in a suitable apparatus, the loss of weight observed being the amount of $CO_2$ driven off. This method is more adapted for carbonates which are completely decomposed by $H_2SO_4$, since, when more volatile acids (HCl, &c.) are employed, the results are generally too high, owing to some of the acid being driven off by the heating at the latter stage of the experiment. The apparatus known as "Schrotters," Fig. 26, may be employed in the determination. It is made of thin blown glass, and its total weight should not exceed 50 grams. It consists of a flask *a*, in which the decomposition of the carbonate takes place.

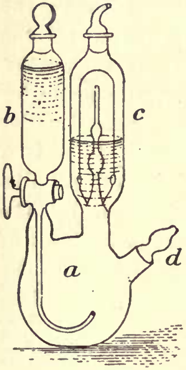


Fig. 26.

Communicating with the flask there are two compartments, *b*, and *c*, also an opening *d*, provided with a glass stopper, for the purpose of introducing the carbonate into the flask. The compartment *b* is filled with dilute acid, which is subsequently gradually allowed to fall into the flask *a* for the purpose of decomposing the carbonate by turning the stop-cock. The compartment *c* is half filled with strong sulphuric acid, for the purpose of drying the $CO_2$ gas before it leaves the apparatus. The gas passes up through the vertical inner tube, then down again, and out through the two holes at the bottom of the second tube. It then bubbles up through the strong sulphuric acid before escaping through the upper exit tube into the air. The experiment is made as follows. About 1 gram of the carbonate (say sodium carbonate) is accurately weighed and introduced into the flask *a*, by the opening *d*. The compartment *b* is then nearly filled with dilute sulphuric acid (1 : 5), and the compartment *c* is half filled with *strong* sulphuric acid. The whole apparatus is then accurately weighed. The dilute acid is now gradually allowed to fall into *a* by gently turning the stop-cock. As soon as the acid begins to act on the carbonate, bubbles of gas will be observed passing through the strong acid in *c*, and it is requisite to regulate the speed at which they pass by adjusting the rate of admission of the dilute acid, so that the separate bubbles can be easily counted. If the bubbles pass more rapidly than this, the gas will be imperfectly dried. When all the dilute acid has flowed into the flask, and bubbles of gas are no longer observed, the apparatus is placed upon a piece of wire gauze on an iron tripod, over a small Bunsen burner. At the same time, the aspirator employed in the previous experiment is attached by means of a piece of india-rubber tubing to the exit tube of *c*, and a gentle current of air is drawn through the flask. The liquid is then kept in a state of incipient ebullition, until a volume of air equal to about three times the capacity of the apparatus has been drawn through it. The flask is then allowed to cool, and when cold weighed. The loss of weight sustained gives the weight of $CO_2$ in the sodium carbonate operated on. As the results by this method are usually too high, it is not adapted for very accurate determinations. The error is due to the loss of water and acid during the heating and aspiration.

*Note.*—If sulphites or sulphides are present, in addition to a carbonate, as is sometimes the case with spent lime, their injurious influence is best obviated by adding to the substance a solution of yellow chromate of potash, in more than sufficient quantity to effect their oxidation.

# PART III.

## VOLUMETRIC ANALYSIS.

### *Introductory Remarks.*

The main principle upon which the system of volumetric analysis depends, is that of submitting the substance to be estimated to certain characteristic reactions, such reactions being produced by the employment of solutions of known strength; and from the volume of solution necessary to produce such a reaction, the weight of the substance is determined by the aid of the laws of chemical equivalence.

The following conditions are therefore essential to the successful carrying out of analyses on the volumetric system, as applied to liquids and solids.

(1) The provision of a solution of a suitable reagent, the chemical power of which is accurately known, called the "standard solution."

(2) The provision of a graduated vessel, from which portions of the standard solution may be exactly delivered, such vessel being known as a "burette."

(3) The reaction produced by the standard solution with any given substance must, either in itself or by an indicator, be such that its termination is strikingly apparent to the eye of the observer, and the quantity of the substance with which it has combined accurately determined thereby.

The following example of the estimation of the amount of chlorine in a substance, by means of a solution of nitrate of silver, will serve as an illustration of the method of volumetric analysis. The estimation depends upon the fact, that if we add to a solution of a chloride a solution of silver nitrate, in the presence of a solution of potassium chromate, as soon as the silver nitrate is added in excess, it produces a permanent dark red precipitate of silver chromate; consequently the appearance of a slight red coloration throughout the whole of the liquid, is an indication of the completion of the reaction.

The following equation shows what takes place:—

$$NaCl + AgNO_3 = AgCl + NaNO_3.$$

Consequently, if we have a solution of nitrate of silver, prepared by adding a definite weight of that substance to a known volume of water, on adding this solution to a solution of sodium chloride, until the reaction mentioned above takes place, every 170 parts of nitrate of silver required, indicates the presence of 35·5 parts of chlorine, or the equivalent weight of any chloride.

In an estimation of the amount of chlorine in a substance by this method, a solution of silver nitrate was employed which contained one-tenth of the molecular weight of silver nitrate, or 17·00 grams per litre (= 1,000 cubic centimetres); consequently, from the equation previously given, each c.c. of the solution was equivalent to 0·00355 gram of chlorine. 2·6223 grams of the substance were weighed out and dissolved, and required 27·5 c.c. of the silver solution for neutralisation.

Consequently the weight of chlorine in the substance $= 0{\cdot}00355 \times 27{\cdot}5 = 0{\cdot}097625$ gram, and the percentage weight of chlorine $\dfrac{0{\cdot}097625 \times 100}{2{\cdot}6223} = 3{\cdot}72.$

The operation of determining the amount of pure substance in any solution by means of standard solutions is termed "titration." The term is more applicable to the operation than testing or analysing, as these expressions may apply equally to qualitative and quantitative examinations, whereas titration can only relate to quantitative examinations.

Volumetric determinations may be broadly classified into three methods:—

(1) The determination of the substance is effected by saturation with another substance of opposite properties. This class embraces the analysis of acids and alkalies, and alkaline earths, and is the one which is the most frequently used in gasworks.

(2) The determination of the substance is effected, by a reducing or oxidising agent of known strength, the principle oxidising agents being potassium permanganate, potassium bichromate, and iodine, and the corresponding reducing agents, ferrous and stannous compounds, and sodic thiosulphate. This method of analysis is employed in the analysis of oxide of iron, and of Weldon Mud.

(3) The determination of the substance is effected by precipitating it in an insoluble and definite combination, as in the case of the determination of chlorine, previously described.

## NOTES ON THE MEASUREMENTS OF LIQUIDS AS APPLIED TO VOLUMETRIC ANALYSIS.

35. The metric system of weights and measures is the one usually adopted in systematic volumetric analysis, and the capacities of the different measuring vessels employed are based on the volume occupied by the weight of 1 gram of distilled water at a temperature of 15·5° C. = 60° F., this volume being called a cubic centimetre. A litre weighs 1000 grams, and has a capacity of 1000 cubic centimetres.

It is necessary to point out, however, that this is not the true, or normal cubic centimetre, as the latter contains 1 gram of distilled water at its greatest density, viz. 4° C., or 39° F., but as this is a temperature not often attainable in this country, it is better to adopt the temperature most easily attained, viz. 15·5° C., or 60° F. The true cubic centimetre (i.e. = 1 gram at 4° C., or 39° F.) contains only 0.998981 gram at 60° F.; but as the measuring vessels are used for ascertaining *relative*, and not absolute volumes of liquids, if all the vessels employed are graduated on the same basis no error can arise.

If necessary, the contents of a vessel can be reduced to absolute volume by means of a correction based on the expansion of water at different temperatures, as given in the Appendix.

It is very essential to see that the temperature is constant when graduating measuring vessels, since the volume of a liquid alters very considerably under different changes of temperature. Glass measuring vessels are always marked with the temperature at which the liquid is to be measured in them.

36. Measuring vessels are required either to measure or to deliver liquids, and the capacity of a glass vessel is indicated by a horizontal line scratched upon its surface, which shows its capacity to that particular level. Some vessels are intended both to measure and to deliver liquids. In this case it is necessary to have two marks; one of these is for the purpose of measuring the volume of a liquid, the other serves to deliver the same volume.

The surface of liquids contained in narrow tubes is always curved, by reason of the capillary attraction exerted by the sides of the tube, and in consequence, it is rather difficult to obtain a distinct level in the liquid to be measured. If, however, the lowest point of the curve or meniscus is caused to coincide with the graduation mark, a correct reading is always obtained.

While the volume of a liquid is being read off it is very essential that the measuring vessel is placed in a perfectly vertical position.

The accuracy of all measuring vessels should be carefully gauged or "calibrated," by weighing definite amounts of distilled water in them, at a temperature of 15·5° C.

The vessels usually required in volumetric analysis are measuring flasks, test mixers or measuring cylinders, pipettes, and burettes.

### *Measuring Flasks.*

37. A convenient series of measuring flasks is the following:—(1) 1000 c.c., (2) 500 c.c., (3) 250 c.c., and 100 c.c. The flasks should be fitted with well-ground glass stoppers, and the graduation marks of each flask should be near the middle of the neck, which should be narrow, in order to ensure accurate readings. The space between the marks and stopper allows the liquid to be more readily mixed by agitation. The flasks should be made thin enough to stand being heated without risk of fracture. Fig. 27 shows a convenient form of litre flask.

FIG. 27.

Measuring flasks are used both for measuring and for delivering liquids, consequently two marks are required. When the flask is filled to the lower mark it contains a certain volume, and when it is filled to the upper mark, the same volume can be poured out, or delivered from the flask.

Before a measuring flask is used, it is necessary to ascertain whether it contains or delivers exactly the volume marked upon it at the given temperature, this operation being known as "calibration."

In order to calibrate a flask for containing or measuring, it is first made quite clean and dry. It is then placed upon the pan of a sensitive balance, and gram weights equal in value to the number of cubic centimetres which the flask should contain, are placed on the pan beside the flask. The flask and weights are then exactly counterpoised by means of lead shot placed upon the other pan. The flask and weights are then removed. The flask is placed upon a level surface, and distilled water, at a temperature of 60° F., is poured into it, until the lower graduation mark on the neck is exactly coincident with the lowest point of the meniscus of the surface of the water. The neck of the flask above the mark is then carefully dried, and the flask (without the weights) is replaced upon the pan of the balance. If the balance is again in exact equilibrium, the flask is correctly graduated for measuring or containing. If the balance is not in equilibrium, then the flask may be regraduated correctly, or a correction may be applied each time the flask is used.

In order to regraduate the flask correctly, all that is necessary is to add to, or take away water from that already in the flask, until the balance is in equilibrium. The flask is then placed on a level surface, and a scratch made on the neck with a triangular file, or diamond, on a level with the lowest point of the meniscus. This will be the true level when *measuring* liquids.

In order to calibrate a flask for *delivering*, the flask is filled with distilled water to the mark on its neck. The water is then poured out, the flask inverted, and allowed to drain for fifteen seconds. The flask, with any remaining water adhering to it, is now placed upon one pan of the balance, the neck having been previously closed by the stopper, in order to prevent loss of weight by evaporation. Weights are then placed on the same scale-pan, and by the side of the flask, the value of the weights in grams being equal to the number of cubic centimetres which the flask is marked to deliver. The flask and weights are then counterpoised by means of shot placed upon the other pan of the balance. The flask and weights are now removed from the scale-pan, the flask placed on a level surface, and accurately filled to the *delivering* mark with distilled water. The stopper is then inserted, and the flask replaced on the balance. If the balance is still in equilibrium, the flask was correctly graduated in the first instance. If the balance is not in equilibrium, the flask must be regraduated in the manner described for graduating the flask for measuring. When the corrected volume is obtained, that quantity will be *delivered* by the flask when it is filled exactly to the delivering mark, and is allowed to drain for fifteen seconds after pouring out the liquid.

### *Test Mixers for Measuring Cylinders.*

38. Test mixers are tall cylindrical vessels, standing on a broad base, and provided with stoppers, as shown in Fig. 28. They are used to prepare test acids, test alkalies, and similar solutions, by the dilution of strong solutions to others of a fixed strength. These vessels are usually graduated throughout the greater part of their length, the graduations varying from 1 to 10 c.c. each, according to the size of the vessel. A measuring cylinder may be calibrated by running distilled water into it from a previously calibrated burette.

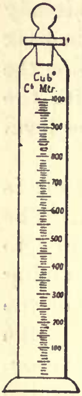
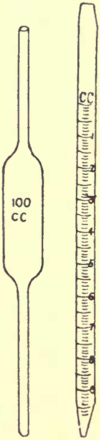


FIG. 28.

### *Pipettes.*

39. Pipettes are glass tubes for transferring specific quantities of liquid from vessel to vessel. They are of two kinds; one has a bulb, and one mark on the neck above the bulb, as shown in Fig. 29. This form is used for the accurate measurement and delivery of one specific quantity of a liquid, such as 5, 10, 20, 50, or 100 cubic centimetres. The other form is shown in Fig. 30, and consists of a narrow tube having a scale engraved upon it lengthwise, similar to the scale of a burette, and is used for delivering a varying quantity of a liquid.

Pipettes are chiefly used to measure with exactness any specified quantity of a solution which is to be subjected to volumetric analysis. The lower end of a pipette from which the solution flows out ought not to be more than $\frac{1}{25}$ inch in diameter. The upper end, which is closed by the finger when in use, should be narrowed by melting and thickening the glass, and then be cut square across, and ground or fused flat, so that the finger can press firmly upon it. In order to fill a pipette, it is held near the top by the thumb and middle finger of the right hand, and is then placed in the liquid it is wished to measure out. The mouth is then applied to the upper end, and the liquid gently sucked up into the pipette. During this operation, the progress of the rise of the liquor in the pipette should be carefully watched, and as soon as it is slightly higher than the top of the graduation, the instrument is removed from the mouth, and firmly closed with the top of the first finger. During the process of filling, it is necessary to see that the point of the pipette always remains below the surface of the liquid, otherwise air will enter the pipette, and may force the liquid up into the mouth, which, with corrosive liquids, might lead to serious consequences. An effectual means of preventing such an accident is to commence by dipping the point into the liquid, and holding it in the same by the left hand during the process of filling. The pipette having been filled in this way, it is held over the vessel from which it has been filled, the pressure of the finger on the top is relaxed, and the liquid allowed to flow slowly out, until the bottom of the meniscus of the liquid in the pipette exactly coincides with the graduation mark. The finger is once more firmly placed on the top of the pipette, which is then held over the vessel intended to receive its contents, and the latter is delivered into such vessel. There are several methods of delivering the contents of a pipette.



FIGS. 29, 30.

(1) The contents are allowed to run freely into the vessel, without permitting the point of the pipette to touch the sides of the vessel, and without blowing out the last drop from the pipette.

(2) After delivering the bulk of the liquid, the point of the pipette is made to touch the inside of the vessel, in order that capillary attraction may carry away the drop adhering to the point of the pipette.

(3) While holding the pipette as last described, the mouth is applied to the top of it, and the drop adhering to the point blown out.

The method generally adopted is to allow the liquid to flow out by its own momentum, and then to leave the pipette to drain for fifteen seconds with its point touching the inside of the glass vessel.

The correctness of a pipette is tested by filling it up to the mark with distilled water at 15·5° C., allowing the water to flow out as just described into a tared beaker and weighing.

### *The Burette.*

40. This instrument, a convenient form of which is shown in Fig. 31, is used for delivering an accurately measured quantity of any standard solution. It consists of a glass tube of equal diameter throughout its length, its upper end being slightly enlarged. The lower end is drawn out, and is closed, preferably by a glass stopcock; or the contracted end is connected by a short piece of india-rubber tubing with a small glass jet. The india-rubber tube is clamped with a brass pinchcock, which closes or opens the tube at will. The burette is graduated throughout the greater part of its length into cubic centimetres, and tenths of a cubic centimetre. Burettes usually contain 50 or 100 c.c. when filled to the zero or highest graduation mark.

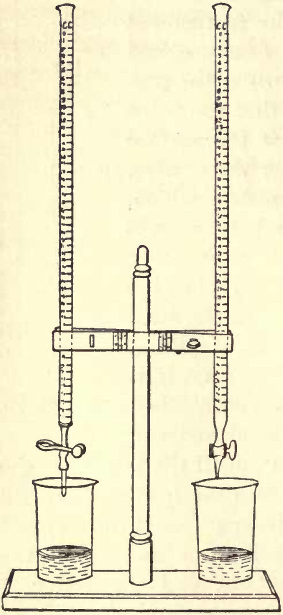


Fig. 31.

The burette is filled by closing the lower end, and pouring in the solution from the top, until it rises above the highest graduation; the stop-cock below is then opened, and the liquid is allowed to run out until the bottom of the meniscus just touches the graduation mark.

It is necessary to be careful when filling the burette, to see that no air-bubbles are left in the stop-cock, since, when the burette is in use, it is assumed, when the stop-cock is opened, that only liquid flows out, and the volume due to any air-bubble which might pass out, would be counted as so much liquid. The volume of liquid used in any determination is measured by the amount the liquid surface in the burette has been lowered. In order to facilitate exact readings, an Erdmann's float, Fig. 32, a little smaller in diameter than the burette, is often used. The float sinks with the liquid, and the reading on the burette, corresponding with the horizontal mark on the centre of the float, is the one taken.

When an Erdmann's float is not employed, the eye may be assisted in reading the graduations on the burette by using a small card, the lower half of which is blackened, the upper portion being left white.

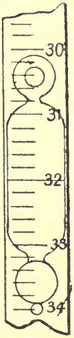


FIG. 32.

On holding the line of division between the black and white about an eighth of an inch below the surface of the liquid, and bringing the eye on a level with it, the meniscus can then be seen by transmitted light, bounded below by a sharply defined black line. A burette may be calibrated by filling it with distilled water at 15·5°C., the tube below being carefully emptied from air-bubbles. Successive quantities of water, in separate instalments of 5 c.c., are run off into a tared stoppered flask, which is then reweighed, and from which it is ascertained whether the weight in grams of water run out is exactly equal to the volume in c.c. as indicated by the burette.

If the reading of the burette is found to be incorrect, a table should be made out showing the true capacity up to each graduation, the error between any two successive weighings being equally distributed between the graduations included in the 5 c.c.

For example, in calibrating a burette from 0 to 10 c.c. the following were the weights of water obtained.

From 0 to 5 c.c. 4·9672 grams; and from 5 to 10 c.c. 4·9850 grams. The first c.c. division on the burette corresponds, therefore, to $\frac{4 \cdot 9672}{5} = 0 \cdot 99344$ c.c.; the second division corresponds to $0 \cdot 99344 \times 2 = 1 \cdot 98688$ c.c.; and so on for the first five divisions.

In the same way, the sixth division will contain $\frac{4 \cdot 985}{5} = 0 \cdot 99700$ c.c. Consequently the first six divisions of the burette will contain $4 \cdot 9672 + 0 \cdot 99700 = 5 \cdot 9642$ c.c., and seven divisions will contain $5 \cdot 9642 + 0 \cdot 9970 = 6 \cdot 9612$ c.c.

All measuring vessels should be in relative correspondence; that is, although they may not exactly contain the required quantity of water at standard temperature, they should relatively agree. For instance, two quarter-litres, when poured into the half-litre flask, should fill it to the graduation mark. The 50 c.c. pipette, when its contents are delivered into the half-litre flask ten times in succession, should also fill it to the graduation mark. 20 c.c. delivered into the burette from a 20 c.c. pipette, should increase the volume of liquid already in the burette by 20 c.c., and so on.

## NOTES ON THE PREPARATION OF SOLUTIONS.

### *Normal Solutions.*

41. The solutions employed in volumetric analysis are termed standard solutions.

The connecting link between measurement and weight, as applied to volumetric analysis, is that of the combining weight of the reagent used, or of some single fraction of the combining weight.

The following short table gives the combining weight of a few reagents, which are in common use in volumetric analysis.

| Name of reagent. | Symbol. | Molecular weight. | Combining weight or hydrogen equivalent. |
|---|---|---|---|
| Ammonia .. .. .. .. .. | $NH_3$ | 17·0 | 17·0 |
| Barium hydrate .. .. .. .. | $BaO, H_2O$ | 171·0 | 85·5 |
| „ carbonate .. .. .. .. | $BaCO_3$ | 197·0 | 98·5 |
| Calcium hydrate .. .. .. .. | $CaO, H_2O$ | 74·0 | 37·0 |
| „ oxide .. .. .. .. | $CaO$ | 56·0 | 28·0 |
| „ carbonate .. .. .. | $CaCO_3$ | 100·0 | 50·0 |
| Iodine .. .. .. .. .. .. | $I$ | 127·0 | 127·0 |
| Potassium hydrate .. .. .. | $KHO$ | 56·0 | 56·0 |
| Sodium hydrate .. .. .. .. | $NaHO$ | 40·0 | 40·0 |
| „ carbonate .. .. .. .. | $Na_2CO_3$ | 106·0 | 53·0 |
| Hydrochloric acid .. .. .. .. | $HCl$ | 36·5 | 36 5 |
| Nitric acid .. .. .. .. .. | $HNO_3$ | 63·0 | 63·0 |
| Oxalic acid (anhydrous) .. .. | $H_2C_2O_4$ | 90·0 | 45·0 |
| „ „ (crystallised) .. .. | $H_2C_2O_4, 2H_2O$ | 126·0 | 63·0 |
| Sulphuric acid .. .. .. .. | $H_2SO_4$ | 98·0 | 49·0 |

Standard solutions are made up in terms of the combining weight or hydrogen equivalent of the active reagent, as shown in the last column of the above table.

Solutions are termed "normal" (N) when one litre, at 15·5° C. = 60° F., contains the hydrogen equivalent of the active reagent, weighed in grams (H = 1).

42. Semi-normal, deci-normal, and centi-normal solutions are also used, being respectively $\frac{1}{2}$, $\frac{1}{10}$, or $\frac{1}{100}$, of the strength of a normal solution. They are usually designated $\frac{N}{2}$, $\frac{N}{10}$, and $\frac{N}{100}$ solutions.

It will be seen from the above table, that in the case of univalent substances, such as iodine, hydrochloric acid, sodium hydrate, &c., the equivalent and the atomic (or, in the case of salts, molecular), weights are the same; thus a normal solution of nitric acid must contain 63 grams of acid in a litre of the solution, and sodic hydrate 40 grams.

In the case of bivalent substances, such as barium hydrate, oxalic acid, sodium carbonate, sulphuric acid, the equivalent is one-half of the atomic (or, in the case of salts, molecular) weight; thus a normal solution of sulphuric acid would be made by adding 49 grams of pure strong acid to distilled water, cooling the mixture, and then diluting to 1 litre.

Also, in the case of trivalent substances, such as phosphoric acid, a normal solution of sodic phosphate would be made by weighing $\frac{358}{3} = 119 \cdot 3$ grams of the substance, dissolving in distilled water, and diluting to the measure of 1 litre.

43. In the preparations of solutions for volumetric analysis, the value of a reagent, as expressed by its equivalent hydrogen weight, must not always be regarded, but rather its particular reaction in any given analysis.

Thus, when using a solution of potassium permanganate ($KMnO_4$), as an oxidising agent, it is the available oxygen which has to be taken into account; and consequently, when preparing a normal solution, one-fifth of its molecular weight, $\frac{158}{5} = 31 \cdot 6$ grams, must be contained in the litre.

The great convenience of this equivalent system is, that the numbers used as coefficients for calculation in any analysis are familiar, and for technical purposes the plan allows the use of all solutions of systematic strength, and simply varies the amount of substance tested, according to its equivalent weight.

Thus, normal solutions of the following contain, per litre—

| | Grams. |
|---|---|
| Sulphuric acid | 49 |
| Hydrochloric acid | 36·5 |
| Nitric acid | 63 |
| Anhydrous carbonate of soda | 53 |
| Sodic hydrate | 40 |
| Ammonia | 17 |

100 c.c. of any of the normal acid solutions in the above list should exactly neutralise 100 c.c. of any of the normal alkalies, or the corresponding amount of the pure substance which the 100 c.c. contain. This system is especially useful in determining the amount of pure substance in commercial samples. For example, supposing we are buying commercial sulphuric acid, and wish to know the exact percentage of pure hydrated acid in it; 4·9 grams are weighed out, diluted with water, a little methyl-orange solution added, and normal alkali run in from a 100 c.c. burette until saturated; the number of c.c. of normal alkali used will show the percentage of real acid present. Supposing 62·6 c.c. are required = 62·6 per cent.

44. In order to obtain the percentage of pure substance in any commercial article, such as alkalies, acids, and various salts, by means of the normal solutions just described, the following method is adopted. With normal solutions, $\frac{1}{10}$ or $\frac{1}{20}$ (according to the atomicity) of the molecular weight in grams of the substance to be analysed, is weighed out for titration, and the number of c.c. required to produce the desired reaction will represent the percentage of the substance whose atomic weight has been used.

With decinormal solutions, $\frac{1}{100}$ or $\frac{1}{200}$ of the molecular weight in grams is taken, and the number of c.c. will, in a similar manner, represent the percentage. It may sometimes happen, however, that from the nature of the substance, or from the fact of its being in solution, that the above percentage method cannot be employed.

For instance, supposing we have a solution containing an unknown quantity of caustic soda, and we desire to know the strength of the same. We weigh, or measure out, a definite quantity of the solution, add a few drops of methyl-orange solution, and run in from a burette, normal acid until the solution is saturated, as shown by the change of colour. Supposing 38 c.c. of normal acid are required, then the molecular weight of sodic hydrate being 40: (Na = 23, H = 1, O = 16, = 40), 100 c.c. of normal acid will saturate 4·0 gram; therefore, as 100 c.c. are to 4·0 gram so are 38 c.c. to $x$, $\frac{4 \cdot 0 \times 38}{100} = 1 \cdot 52$ gram NaHO. The simplest way is to multiply the number of c.c. of test solution required in any particular determination by the $\frac{1}{1000}$ (or $\frac{2}{1000}$ in the case of a bivalent substance) of the molecular weight of the substance sought. This will at once give the amount of substance present.

45. Although solutions based on the above mentioned systematic principle, are those commonly adopted in analytical work, we require in addition, in the carrying out of some of the special determinations necessary in gas works, empirical solutions, based on the English system of weights and measures, and which are prepared to suit particular cases, not being based on any broad general principle. The solutions employed in the determination of the amount of ammonia in gas by the Referees' method, and in the commercial valuation of ammoniacal liquor, are cases in point. These are described in the Special Part.

46. In the preparation of standard solutions, it is necessary to bear in mind the fact, that saline substances on being dissolved in water, have a considerable effect upon the volume of the resulting liquid. The same thing occurs when mixing solutions of various salts or acids with each other.

In the case of strong solutions, the condensation in volume is, as a rule, very considerable, and therefore in preparing such solutions for volumetric analysis, or in diluting such solutions to a given volume, for the purpose of removing aliquot portions subsequently for examination, sufficient time must be afforded for the solutions to attain their constant volume at the standard temperature.

Table for the Systematic Analysis of Alkalies, Alkaline Earths, and Acids. (*Sutton.*)

| Substance. | Formulæ. | Atomic weight. | Quantity to be weighed so that 1 c.c. normal solution = 1 per cent. of substance. | Normal factor.* |
|---|---|---|---|---|
| | | | gram. | |
| Soda | $Na_2O$ | 62 | 3·1 | 0·031 |
| Sodic hydrate | $NaHO$ | 40 | 4·0 | 0·040 |
| ,, carbonate | $Na_2CO_3$ | 106 | 5·3 | 0·053 |
| ,, bicarbonate | $NaHCO_3$ | 84 | 8·4 | 0·084 |
| Potash | $K_2O$ | 94 | 4·7 | 0·047 |
| Potassic hydrate | $KHO$ | 56 | 5·6 | 0·056 |
| ,, carbonate | $K_2CO_3$ | 138 | 6·9 | 0·069 |
| ,, bicarbonate | $KHCO_3$ | 100 | 10·0 | 0·100 |
| Ammonia | $NH_3$ | 17 | 1·7 | 0·017 |
| ,, carbonate | $(NH_4)_2CO_3$ | 96 | 4·8 | 0·048 |
| Lime | $CaO$ | 56 | 2·8 | 0·028 |
| Calcic hydrate | $CaO,H_2O$ | 74 | 3·7 | 0·037 |
| ,, carbonate | $CaCO_3$ | 100 | 5·0 | 0·050 |
| Barium hydrate | $BaO,H_2O$ | 171 | 8·55 | 0·0855 |
| ,, carbonate | $BaCO_3$ | 197 | 9·85 | 0·0985 |
| Nitric acid | $HNO_3$ | 63 | 6·3 | 0·063 |
| Hydrochloric acid | $HCl$ | 36·5 | 3·65 | 0·0365 |
| Sulphuric acid | $H_2SO_4$ | 98 | 4·9 | 0·049 |
| Oxalic acid | $H_2C_2O_4,2H_2O$ | 126 | 6·3 | 0·063 |

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* This is the coefficient by which the number of c.c. of normal solution used in any analysis is to be multiplied, in order to obtain the amount of pure substance present in the material examined.

### *Storage of Standard Solutions.*

47. Standard solutions may be kept in any tightly stoppered bottle of a suitable size. A "Winchester quart" is a handy size for most purposes. The bottle should be clean and dry, and after the solution has been poured in the bottle should be immediately tightly stoppered, and labelled with the name, strength, and date when the solu-

tion was made. It is necessary that the bottle should be quite full, otherwise internal evaporation and condensation will cause drops of pure water to form on the upper part of the inside of the bottle.

When using the solution therefore, it is necessary to well shake the bottle before pouring out, so as to mix this water with the solution from which it originated.

This loss by evaporation may be lessened by storing the solutions in a cool place. Some solutions are affected by light, and in consequence should be kept in a dark cupboard, rather than upon open shelves where they would be exposed to the light.

## INDICATORS.

48. In many analyses it is necessary to add a substance which shall act as an indicator of the end of the process; such, for instance, is cochineal or methyl-orange in alkalimetry, potassium chromate in silver and chlorine estimations, and starch in determinations where iodine is employed. There are other processes, the final termination of which can only be determined by an indicator separate from the solution. An example is found in the estimation of iron by potassium bichromate, in which case a drop of the solution is brought into contact with another drop of a solution of ferricyanide of potassium, on a white tile; as soon as a blue colour ceases to appear when the two liquids are brought into contact, the end of the process is reached.

## INDICATORS USED IN THE VOLUMETRIC ESTIMATION OF ACIDS AND ALKALIES.

### *Cochineal Solution.*

49. This indicator possesses the advantage of not being affected in colour by $CO_2$. In order to prepare it, digest about 10 grams of powdered cochineal for several hours at a gentle heat in a litre of weak spirit, composed of 200 c.c.

of methylated spirit, with 800 c.c. of water. The clear liquid, when decanted off, is ready for use. Its normal colour is yellow, and this is changed to reddish-violet by alkalies. This reddish-violet solution of cochineal is changed to yellow again by mineral acids, but it is not so easily acted upon by weak organic acids. It should not be used in the presence of compounds of iron or aluminium, or of acetates.

It is well adapted for the estimation of ammonia in gas liquor (Will's Test), and in the analysis of ammonium sulphate.

### *Methyl-Orange, Tropæolin D, or Orange III.*

50. This indicator also is not affected by $CO_2$. Alkalies change its colour to yellow, and this changes to red on the addition of an acid. The solution does not change or decompose on keeping.

It is prepared by dissolving 1 gram of methyl-orange in powder (procurable from the operative chemist) in a small quantity of methylated spirit, and then making the solution up to 1 litre with methylated spirit diluted with its own volume of water.

Methyl-orange is employed in the estimation of ammonia in gas, and in the estimation of free ammonia in gas liquor.

### *Litmus Solution.*

51. This solution is used both for acids and alkalies. The solution is most sensitive when it is of a purple colour. The presence of an excess of acid, causes the litmus to assume a bright red tint; excess of alkali produces a pure blue tint.

In order to prepare a solution of this indicator, all the colouring matter is first extracted from solid litmus by repeatedly digesting it with hot water.

The solution thus obtained, is evaporated to a moderate bulk, and a slight excess of acetic acid added, in order to convert all carbonates present into acetates. The solution is then again evaporated over a water-bath, until it assumes a pasty condition, when an excess of methylated spirit is added. The spirit precipitates the blue colouring matter, a red colouring matter, together with the alkaline acetates remaining in solution. The precipitate is transferred to a filter, and washed with spirit. The pure colouring matter is then dissolved in warm water, and the solution, after being rendered purple by the addition of a little dilute nitric acid, is ready for use. Litmus solution must always be kept exposed to the air or it loses its colour, regaining it on exposure.

A convenient method of securing this exposure, is to have the cork which closes the bottle containing the litmus solution, made with several deep grooves. The great defect of litmus as an indicator is that the presence of free carbonic acid interferes with the production of the blue colour.

Accordingly, in titrating an acid solution by means of an alkaline carbonate, the litmus solution changes from blue to purple before the point of neutralisation by the acid is arrived at. This is caused by the liberated $CO_2$ acting upon the litmus. The $CO_2$ can be removed by boiling the liquid, and the neutral point is just passed, and the reaction is consequently complete, when the solution has assumed a bright red colour, which does not alter on boiling.

### *Phenol-phthalcin.*

52. This indicator, when in the form of an alcoholic solution, is without colour, but on adding a few drops to an alkaline solution the liquid changes to a beautiful red colour, which is destroyed by acids. Phenol-phthalein cannot be employed in the presence of $CO_2$, or of salts of ammonia.

Phenol-phthalein is employed in the estimation of $CO_2$ in coal gas (Sheard's method).

It is necessary to bear in mind that in judging the end of a re-action by means of an indicator, or of any final change of colour, it is requisite in all cases, that the one liquid should be run into the other in the same order. The liquids must not be reversed, or the results will fail to correspond. For instance, if cochineal is used as an indicator in standardising the acid, by running the acid into a standard solution of caustic soda, in all processes of estimating acid by the alkali, where cochineal is used as an indicator, the acid must be run into the alkali, and not the alkali into the acid.

## ANALYSIS BY SATURATION.

### (ALKALIMETRY AND ACIDIMETRY.)

### *Preparation of Normal Acid and Alkaline Solutions.*

53. In this particular branch of volumetric work, it is very essential to have at least one standard acid and alkali prepared with the greatest accuracy, and from which all others can be standardised.

Sulphuric acid is best adapted for preparing the normal acid, as it can easily be obtained pure. Normal sulphuric acid is not affected by boiling.

For the standard alkali, sodic carbonate is to be preferred, inasmuch as it can readily be obtained in a perfectly pure state, or can easily be made by igniting the pure bicarbonate.

The chief difficulty in times past with sodic carbonate has been, that, when using litmus as indicator, it was necessary to carry on the titration at a boiling heat, in order to get rid of the $CO_2$, which hindered the pure blue colour of the indicator, notwithstanding the alkali may have been in excess.

This difficulty is now got rid of by the employment of methyl-orange as indicator. But in case methyl-orange is not available, litmus may be made to give accurate results, if the saturation is carried on by rapidly boiling the liquid in a thin flask for a minute after each addition of acid, until the point is reached when one drop of acid in excess gives a pink red colour, which does not disappear on further boiling.

### *Normal Solution of Sodium Carbonate.*

(53 grams $Na_2CO_3$ per litre.)

54. This solution is prepared by dissolving pure $Na_2CO_3$ in distilled water, so as to make a solution containing 53 grams $Na_2CO_3$ per litre. It is consequently a semi-molecular solution.

If we dissolve $Na_2CO_3$ in water, the volume of the liquid contracts, therefore, we cannot obtain the correct weight per litre by simply dissolving the salt in the measured volume of water. After adding the salt to a quantity of water contained in the litre flask, the solution must be allowed to attain the normal temperature, and then sufficient distilled water added as will bring the volume up to the litre mark, the contents of the flask being well mixed.

The easiest way of obtaining pure $Na_2CO_3$, is to ignite the bicarbonate. It is necessary in the first place to see that the bicarbonate is pure, by testing it for chlorides and sulphates. This is effected by shaking a portion of the powder with distilled water in a stoppered bottle, and then testing the clear liquid. Should traces of these impurities be found, about 100 grams of the powder are shaken up with three or more separate quantities of distilled water, the salt being allowed to settle, and the supernatant liquid decanted off.

As soon as the washings are free from sulphates or chlorides, the salt is drained on a porous tile, and dried by pressing it between sheets of dry blotting or filtering paper. About 85 grams of the salt prepared as above are spread in a thin layer inside a weighed platinum or porcelain dish. The dish and its contents are then heated by means of a Bunsen burner to dull redness for about 10 minutes, care being taken that the salt does not fuse or frit.

The dish is now placed in the desiccator to cool, and when cold weighed; it is then heated, cooled, and weighed again, until the weight is constant. The weight of pure $Na_2CO_3$ is then obtained by subtracting the weight of the dish. The standard solution of $Na_2CO_3$ may now be prepared from the above by either of the two following methods.

(1) The volume of the normal solution which should contain the weight of $Na_2CO_3$ obtained as above is found by calculation. For example, if the sodium carbonate weighed 54·6 grams, it will require to be dissolved in

$$\frac{54\cdot6 \times 1000}{53} = 1030\cdot2 \text{ c.c.}$$

The salt is transferred without loss to a beaker, the dish being afterwards rinsed out with successive small quantities of distilled water into the same beaker. The salt is well stirred to assist solution, and when all has dissolved, the solution is poured into the litre flask, the beaker being also well rinsed out several times with small quantities of distilled water, and the rinsings added to the contents of the flask. The solution is now nearly made up to the mark on the flask with distilled water at a temperature of 60° F., the contents of the flask being well mixed by shaking. The stopper of the flask is then inserted, and the solution is again vigorously shaken to ensure thorough mixing. The contents of the flask are now made up to the exact volume by placing the flask on a level surface, and slowly pouring in water until the bottom of the meniscus just touches the litre mark on the flask. The additional volume of water required, amounting in the present example to 30·2 c.c., is then run into the flask from a burette, and the liquid is again thoroughly mixed by inverting the flask and giving it a rotary motion.

(2) Exactly 53 grams of $Na_2CO_3$, prepared as above, are weighed out and dissolved to a litre. The weight may be conveniently obtained by placing the dish on one scale-pan of the balance, and adjusting the weight on the other pan so that they are equal to the weight of the dish + 53; small portions of the salt are then removed with the point of a penknife or spatula until equilibrium occurs. The salt is then dissolved in a beaker as in (1), and on adding water to the litre mark a normal solution will be obtained.

### *Normal Sulphuric Acid.*

55. This solution is a semi-molecular one $\left(\frac{98}{2}\right)$ (sulphuric being a bibasic acid) and should contain 49 grams of pure $H_2SO_4$ per litre. It is prepared by adding a suitable quantity of pure strong oil of vitriol to distilled water contained in a thin glass vessel, then cooling the hot solution, and subsequently determining its strength by titrating a portion of it against the normal sodium carbonate solution. When the strength has been accurately determined, the amount of water required to dilute it, in order to reduce it to the proper strength, may be easily calculated.

Measure out 30 c.c. of pure, strong oil of vitriol, and run it into about 200 c.c. of distilled water contained in a thin glass flask. On adding the acid great heat will be given out; mix the solution well, and cool it by shaking it round in the flask, at the same time allowing a stream of water from a water tap to flow over the exterior. When cooled down to the normal temperature, the liquid in the flask is made up to exactly 1 litre with distilled water; it is then thoroughly mixed again, and a portion is titrated by normal sodium carbonate solution, as follows. A clean 20 c.c. pipette is partially filled with the normal sodium carbonate solution by suction; the liquid is well shaken round in the pipette, and is then allowed to run away into the sink, or other receptacle for waste. The pipette is again charged with the solution, but this time exactly to the 20 c.c. mark, by drawing up more than the required volume, rapidly placing the first finger on the top of the pipette, and slowly allowing the excess to flow out. The 20 c.c. of solution are then delivered into a clean beaker. Of course, in the event of the interior of the pipette being perfectly clean and dry at the commencement, there is no occasion for the preliminary rinsing with the solution, inasmuch as in this case there would not be any danger of the standard solution being diluted with the water adhering to the interior of the pipette.

A burette is next charged with the acid prepared as described, care being taken that the interior of the instrument is perfectly dry; if not, it should be first rinsed out with a portion of the solution, which is afterwards thrown away. It is also necessary to see that there are no air bubbles remaining in the tap at the bottom of the burette; should there be any, they may be got rid of by *rapidly* running a portion of the solution out of the burette, and then refilling the same. The solution in the beaker, previously diluted with distilled water, is placed underneath the burette, upon a white tile, or a clean filter paper, a small quantity of methyl-orange solution is added, and the acid run slowly in from the burette, the contents of the beaker at the same time being constantly stirred. The acid is run in until a single drop causes the golden yellow colour of the solution to suddenly change to a reddish-brown tint, shewing that the solution in the beaker has been exactly neutralised by the acid. The volume of acid required to effect the neutralisation is then read off on the burette, the result being checked by repeating the titration in exactly the same manner with a fresh quantity of the sodium carbonate solution. The two titrations should not differ by more than 0·1 c.c. If more than 20 c.c. are required, the acid is too weak; if less, too strong. It will generally be found that the acid is too strong, and will require diluting to the normal strength in the following manner. Supposing that the 20 c.c. of the alkaline solution required 18·6 of the acid solution to neutralise it; then each 18·6 c.c. of acid would require to have sufficient water added to it to make it up to 20 c.c., in order to produce a standard acid exactly equivalent to the standard alkali. Therefore, to make a litre (1000 cubic centimetres) of normal acid, 930 c.c. of the acid, prepared as above, are measured into a litre flask, and then made up to 1 litre with distilled water. After thoroughly mixing the acid diluted in this manner, it should again be titrated against the alkaline solution, as already described, in order to be certain of its accuracy.

It is necessary to bear in mind the fact that standard solutions, even when accurately prepared in the first instance, will gradually become more or less altered in strength after being stored for some time. A standard acid solution will therefore require to be tested occasionally, by titration against a freshly prepared standard sodium carbonate solution, as just described. In the event of the acid solution having altered in strength, it is not absolutely necessary (if the error is small) to bring it back to the correct standard. In place of doing this, a correction is made use of when working with the solution.

For instance, if 19·7 c.c. of acid are needed instead of 20, in order to neutralise 20 c.c. of a freshly prepared sodium carbonate solution, the number of c.c. of acid needed in any titration will require to be multiplied by $\frac{20}{19 \cdot 7} = 1 \cdot 015$, in order to find the number of c.c. which would have been needed if the acid had been of the correct strength. The "factor" required for this correction should be marked on the label of the bottle, together with the date.

The operation of determining the amount of acid in a solution in the above manner is termed "acidimetry."

### *Normal Hydrochloric Acid.*

(36·5 grams per litre.)

56. Pure strong hydrochloric acid is diluted with distilled water until it has a specific gravity of 1·10 at 60° F., as tested by a hydrometer. About 165 c.c. of this acid are diluted to 1 litre. Its exact strength is then determined by titration against normal sodium hydrate solution (58), and the solution is brought to the standard strength by dilution, as in the previous examples.

Hydrochloric acid is useful on account of its forming soluble compounds with the alkaline earths, but it has the disadvantage of volatilising at a boiling heat. The hydrochloric acid from which standard solutions are made, should be free from chlorine gas or metallic chlorides, and when evaporated in a platinum vessel should leave no residue.

### *Normal Oxalic Acid.*

(63·0 grams per litre.)

57. This solution is prepared by dissolving 63·0 grams of pure, not effloresced, crystallised oxalic acid in distilled water, and making up to 1 litre, the solution being afterwards checked with normal alkali. The solution is apt to deposit some of the acid at low temperatures, but keeps well if preserved from direct sunlight, and may be heated without volatilising the acid. It cannot be employed in alkalimetry when methyl-orange is used as the indicator.

This solution may be used in testing for caustic lime in the presence of a carbonate.

### *Normal Sodium Hydrate Solution.*

(40 grams per litre.)

58. Caustic soda suitable for the preparation of the above may be obtained from two sources, either by the action of metallic sodium on boiled and cooled distilled water, or from caustic soda purified by alcohol.

In order to obtain it from metallic sodium, about 25 grams of clean metallic sodium in small pieces are weighed out, and placed in distilled water contained in a platinum or silver dish. A clock glass is placed over the dish after the addition of each piece of sodium, in order to prevent loss of the NaHO by spurting, the chemical action set up by each separate piece of sodium being allowed to subside before a fresh piece is introduced.

In this way a strong solution of NaHO is obtained. If caustic soda purified by alcohol is used, then about 44 grams should be weighed out, and dissolved in distilled water. If either of these solutions are diluted until they have a specific gravity of about 1·05, then the solution will contain about 50 grams per litre. The water employed in diluting should be distilled, and recently boiled and cooled, so as to be free from $CO_2$. 20 c.c. of the solution thus prepared are then titrated against 20 c.c. of the normal acid (55), employing methyl-orange as indicator, and the solution is reduced to the exact normal strength, by diluting it with the necessary quantity of distilled water.

## ESTIMATION OF AMMONIA.

59. This determination comes under the head of alkalimetry. The principles upon which it is based apply to the determination of the ammonia in gas, and to the analysis of ammoniacal liquors and sulphate of ammonia. By alkalimetry is meant the method of determining the amount of alkali in a substance by saturation with a standard acid.

The quantity of free ammonia in a solution of ammonia and water may be determined by means of standard acid and methyl-orange solution. A definite quantity of the solution, say 10 c.c., is transferred to a small tared flask and weighed; in this way its absolute weight and specific gravity are obtained in one operation. If the 10 c.c. weighed 9·449 grams, its specific gravity would of course be 0·9449, water = 1.

The weighed quantity of the ammonia, is then diluted with 6 or 8 times its bulk of distilled water, and titrated direct with the standard acid.

Ammonia in combination may be determined by expelling it by means of caustic soda or lime, collecting the evolved ammonia in a known volume of standard acid, and determining the excess of acid by standard soda solution.

The following shows the method of determining the amount of ammonia in ferrous ammonium sulphate:—$Fe(NH_4)_2(SO_4)_2 6H_2O$.

The apparatus employed is shown in Fig. 33. It consists of a large flask A, having a capacity of about 30 fluid ounces, fitted with a cork pierced with two holes, into one of which is inserted a tube B about 4 inches long, and half an inch internal diameter, containing pieces of broken glass, and closed at the top with a cork, through which passes a small tube the diameter of a goose-quill, and about 2 inches long. Into the other hole is fixed a bent tube, C, one end of which passes nearly to the bottom of the flask A, the other being connected, by an india-rubber joint, to a similar tube passing through a cork adapted to a second flask E, of about 15 ounces capacity; a small funnel D is also passed through the cork so as to nearly reach to the bottom of the flask E. A sand bath F, supported on a tripod G, and a Bunsen burner H, completes the apparatus. There are various modifications of this apparatus employed, but the one described is about the most simple. Having connected the whole apparatus together, as shown, 100 cubic centimetres of normal sulphuric acid are poured, by means of a pipette, through the tube B into the flask A, so as to thoroughly moisten with acid the pieces of glass in the tube B. About 1 gram of the pure salt is accurately weighed out, all crystals showing a yellowish tinge being rejected. The quantity weighed out is dissolved in about 50 c.c. of distilled water, and the solution is poured into the flask E.

About 50 c.c. of a strong solution of sodium hydrate is then poured into E, the funnel tube rinsed out with a little distilled water, and the stop-cock closed. The burner is then lit beneath the sand-bath, and the contents of the flask E brought to incipient boiling. Ammonia will then be disengaged, and will be absorbed by the acid in the flask A, and in the tube B. The tube C should only just touch the acid in the flask A. When the ammonia ceases to be absorbed, the flame should be raised, and the contents of the flask E kept briskly boiling for ten minutes.

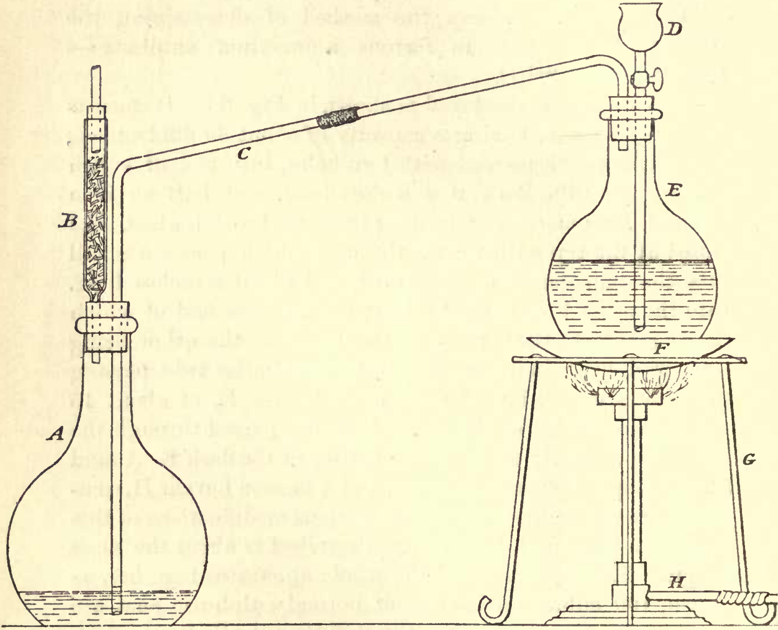

FIG. 33.

The apparatus is then disconnected, and the pieces of glass in B rinsed with a little distilled water into the flask A. The liquid in A is then made up to exactly 200 cubic centimetres, and divided into two portions of 100 c.c.

each. A 50 c.c. burette is then filled with normal soda solution, and one portion of the acid solution, coloured with a few drops of solution of cochineal, is titrated with the soda solution, the remaining portion being similarly treated. The two titrations should not differ by more than $\frac{1}{10}$ c.c.

*Example:*—1·3723 grams of crystallised ferrous ammonium sulphate were weighed out, and treated as above described, the ammonia evolved being absorbed by 100 c.c. of normal sulphuric acid. After absorption, the acid solution was made up to 200 c.c., and divided into two portions. 100 c.c. of this liquid (= 50 of the original solution) required 46·5 c.c. of normal soda solution to neutralise it, consequently, the whole of the solution would require $46 \cdot 5 \times 2 = 93$ c.c. of the soda solution. The number of c.c. neutralised by the ammonia = $100 - 93 = 7 \cdot 00$, and since each c.c. of acid corresponds to 0·018 gram of $NH_4$, the weight of $NH_4$ in the salt = $0 \cdot 018 \times 7 = 0 \cdot 126$ grams,

$$\therefore \text{ the percentage of } NH_4 = \frac{0 \cdot 126 \times 100}{1 \cdot 3723} = 9 \cdot 18,$$

which agrees with the percentage demanded by theory, as under.

The molecular weight of $Fe(NH_4)_2(SO_4)_2,6H_2O = 392$, and this contains 36 parts by weight of $NH_4$, consequently,

$$\text{As } 392 : 100 :: 36 : x. \quad x = 9 \cdot 18.$$

60. Another example of the process of alkalimetry as applied to the requirements of gasworks, is that of the analysis of lime.

In this case the lime, either in the form of carbonate or of caustic lime, is dissolved in an excess of standard hydrochloric acid, the volume of acid noted, and retitrated with a standard solution of caustic soda. The volume of caustic soda now required to neutralise the acid is deducted from the amount of acid originally added; the difference represents the amount of acid which has entered into combination with the lime.

Each c.c. of acid used represents 0·028 gram of caustic lime, or 0·037 gram of calcic hydrate, or 0·050 gram of $CaCO_3$. This method of determination is termed the "residual" method.

Full details are given under the heading "Analysis of Lime" in the Special Part.

## PROCESSES OF OXIDATION AND REDUCTION.

The principles involved in analytical processes based on the methods of oxidation and reduction are extremely simple; substances capable of absorbing oxygen are dissolved, and titrated with another substance of known oxidising power, as for example in the estimation of iron in ferrous compounds by means of permanganate of potash. The iron is eager to receive oxygen, and the permanganate is willing to give it up. During the process of absorption, the permanganate instantly loses its colour on being added to the iron solution, and the whole mixture is colourless, but as soon as the iron has taken up its complement of oxygen, or has been converted into the higher state of oxidation as a ferric salt, the rose colour of the permanganate solution no longer disappears, as there is no more oxidisable iron to operate on. The following equation expresses what has taken place in the above reaction:—

$$10FeO + 2MnKO_4 = 5Fe_2O_3 + 2MnO + K_2O.$$

The oxidising agents commonly employed are potassium permanganate, potassium bichromate, and iodine, the common reducing agents being sulphurous acid, sodic hyposulphite, stannous chloride and zinc.

A great many combinations may be arranged with this variety of materials, so as to make this system of analysis very comprehensive. The following, however, are the most typical, and serve to show the applicability of the system to the requirements of a gasworks.

(*a*) Permanganate of potash and ferrous salts, the rose colour of the permanganate acting as indicator (may be used in the analysis of Weldon Mud).

(*b*) Bichromate of potash and ferrous salts, the end of the reaction being shown by the cessation of the blue colour when brought in contact with a solution of ferricyanide of potassium as indicator (may be used in the estimation of iron oxides for purifying purposes, bog ore).

(*c*) Iodine with starch as indicator (may be used in the estimation of $SH_2$ in gas liquor).

## PREPARATION AND USE OF POTASSIUM PERMANGANATE SOLUTION.

61. The molecular weight of potassium permanganate ($Mn_2K_2O_8$) = 316·0, and as shown in the equation previously given, 1 equivalent, or 316 parts by weight of the salt, is equal to 10 equivalents, or 560 parts of iron. In order to prepare a decinormal solution of permanganate, therefore, 3·16 grams of the pure salt may be dissolved in a litre of water. 17·85 c.c. of this solution should then peroxidise 0·1 gram of iron; for 1000 c.c. being equal to 5·16 grams Fe, 17·85 c.c. are equal to 0·1 gram. The above method, theoretically, should give a solution of the exact strength, but practically we cannot calculate the strength of such a solution, i.e. the amount of oxygen that it is capable of yielding, beforehand, on account of its instability. It is absolutely necessary, therefore, to determine the power of the solution by direct experiment before using it. It is consequently not essential to make the solution strictly decinormal, as its power is accurately determined by experiment before use.

About 5 grams of the pure crystallised salt are dissolved in a small quantity of distilled water, and the resulting solution is diluted to 1 litre.

The solution should be kept in a dark blue Winchester quart bottle, and when not in use should be stored in a cool dark place, as the liquid, if exposed to light, becomes weaker owing to the decomposition of the salt; but a solution preserved in the above manner may be kept for a long period without experiencing much alteration.

The most accurate method of estimating the strength of the solution consists in determining the amount required to transform a known weight of iron from the condition of ferrous oxide to that of ferric oxide.

This is effected as follows:—About 1 gram of fine iron wire (flower wire), quite free from rust, is accurately weighed out into a 250 c.c. flask, and dissolved in about 100 c.c. of dilute sulphuric acid (1 part of acid to 6 of water). A small quantity of carbonate of soda is thrown into the liquid at the same time, in order that the air within the flask may be displaced by carbon dioxide.

The flask is provided with a cork and bent glass tube, furnished with a brass pinch-cock; the end of the tube dips beneath the surface of about 25 c.c. of water contained in a small flask, Fig. 34. Whilst the iron is dissolving, the pinch-cock closing the india-rubber connecting tube is taken off the latter, and slipped over the glass tube; by so doing there is a free communication between the two flasks. The solution of the iron may be accelerated by a gentle heat. The liquid is gradually brought to the boiling point, and kept briskly boiling for a minute or two, so as to expel the mixture of $CO_2$ and hydrogen; the india-rubber tube is then immediately closed, and the burner removed. In the course of a minute or so the pinch-cock is again opened, when the water from the small flask is driven over into the solution of iron. In proportion as it passes over, boiling distilled water is poured into the smaller flask, until the larger one is almost filled. The india-rubber tube is then once more closed, the flask and its contents allowed to cool, and the volume of the liquid made up to the containing mark; the stopper of the flask is inserted, and the liquid well shaken so as to cause it to be thoroughly mixed. Whilst the solution cools, a Mohr's burette fitted with a glass stop-cock (the permanganate solution gradually attacks india-rubber) is first rinsed out with, and then filled with the permanganate solution prepared as above. When cool, 50 c.c. of iron solution are taken out of the flask, and poured into about 200 c.c. of distilled water, contained in a beaker standing on a sheet of white paper. The permanganate solution is then added, drop by drop, to the liquid, with constant stirring, until the pink colour imparted to the solution remains. The permanganate is at first decomposed with great rapidity, but as the iron becomes oxidised the colour disappears more slowly, the rapidity of the change indicating the progress of the oxidation. The operation of standardising thus described should be repeated once or twice on successive portions of 50 c.c. of iron solution, and the mean of the results taken as representing the true value of the permanganate solution.

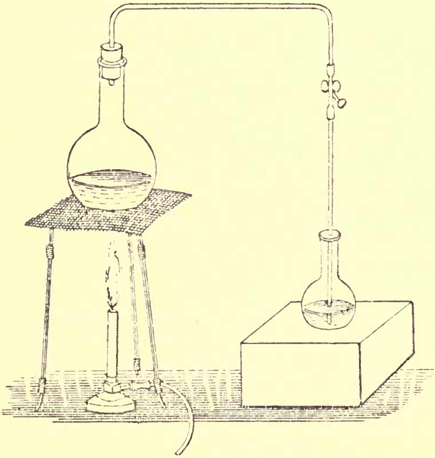

FIG. 34.

The reactions occurring in the foregoing are expressed by the following equations:—

$$10FeSO_4 + 2KMnO_4 + 8H_2SO_4$$
$$= 5Fe_2(SO_4)_3 + 2MnSO_4 + K_2SO_4 + 8H_2O,$$

or, shortly,

$$10FeO + Mn_2O_7 = 2MnO + 5Fe_2O_3.$$

As an illustration, let us assume that we have weighed out 1·10 grams of iron, and dissolved it in 250 c.c. of liquid; 50 c.c. of the solution, or a fifth part, would be equivalent to 0·2200 gram of iron. The iron employed, however, is not chemically pure, but if we assume that its impurities amount to 0·4 per cent. we shall not be far out. The amount of pure iron in the 50 c.c. is therefore

$$1 : 0·996 :: 0·2200 : x. \quad x = 0·21912.$$

We will further suppose that it has required 20 c.c. of the permanganate solution to produce a permanent coloration with the 50 c.c. of iron solution. Then, as 20 c.c. permanganate solution converts 0·21912 grams iron from protoxide to peroxide, 100 c.c. permanganate are equivalent to 1·09560 grams iron, or 1 c.c. to 0·010956 gram.

The strength of the permanganate solution may also be determined by means of pure ferrous sulphate, precipitated from its aqueous solution by means of alcohol. Ferrous sulphate so prepared keeps unchanged for very long periods.

Instead of $FeSO$ , the double sulphate of iron and ammonium $FeSO_4(NH_4)_2SO_4 + 6H_2O$ may be employed. This contains exactly one-seventh of its weight of iron: 0·7 gram of salt is equivalent to 1 gram of iron. The strength of the solution may also be determined by means of oxalic acid.

Of the several substances which may be used for titrating the permanganate solution metallic iron is the best, but whichever method is adopted, it is absolutely necessary that the solution to be titrated should contain free sulphuric acid. If there is a deficiency of free acid, the solution turns brown and eventually a precipitate collects. It is necessary to exercise discretion as to the acid employed for acidulation. Nitric acid cannot well be employed under any circumstances. Hydrochloric acid is liable to be oxidised and give off chlorine, unless it is very considerably diluted, and this change interferes with the accuracy of the result. Organic matter also reduces the permanganate, and destroys the accuracy of the determination.

62. Permanganate of potash may be employed in determining the amount of $MnO_2$ in the Weldon Mud employed in gas purification.

The method depends upon the fact than when $MnO_2$ is treated with an acid, it is decomposed and oxygen liberated, which combines with any substance present ready to receive it In the present instance the substance is a ferrous salt in an acid solution. The amount of permanganate required to oxidise a definite amount of the ferrous salt being known, on adding a weighed amount of the Mud to this solution, on again titrating with permanganate, it will be found that we do not require so much of that solution as we did in the first instance, due to the fact that the oxygen of the $MnO_2$ in the Weldon Mud, has supplied the place of the oxygen of the permanganate solution.

About 1·5 to 2 grams of "flower wire," perfectly free from rust, are accurately weighed out and dissolved in 100 c.c. of dilute $H_2SO_4$ (1 of acid to 4 of water) in the apparatus shown in Fig. 34. About the same weight of the finely powdered Mud is then added to the iron solution, and the liquid is gently heated until the Mud is dissolved. The solution is then boiled, the water allowed to flow back, and the contents of the flask made up to 250 c.c. When cold, the amount of residual ferrous sulphate is determined by the permanganate solution.

The following equation shows what takes place :—

$$2FeSO_4 + MnO_2 + 2H_2SO_4 = Fe_2(SO_4)_3 + MnSO + 2H_2O.$$

112 parts of iron correspond to 87·0 of $MnO_2$.

*Example* :—1·756 grams of flower wire were weighed out = 1·756 × 996 = 1·749 grams pure iron. This was dissolved in the requisite quantity of dilute sulphuric acid, and 2.12 grams Weldon Mud added to the iron solution. When cold, the solution required 86·2 of permanganate solution. 1 c.c. permanganate = 0·010956 Fe; consequently 0·010956 × 86·2 = 0·944407 Fe. 1·749 − 0·944407 = 0·8046 gram of iron, has been oxidised by the manganese dioxide.

Then as $112 : 87 :: 0{\cdot}8046 : x. \quad x = {\cdot}625,$

and $2{\cdot}12 : 0{\cdot}625 :: 100 : 29{\cdot}48.$

The Weldon Mud therefore contained 29·48 per cent. of manganese dioxide.

## USE OF POTASSIUM BICHROMATE SOLUTION.

63. When used as an oxidising agent in volumetric analysis, potassium bichromate always yields the whole of its oxygen to the hydrogen of the accompanying acid, a corresponding quantity of acidulous radical being set free, four-sevenths of this radical immediately combining with the potassium and chromium of the potassium bichromate, three-sevenths becoming available. Advantage is taken of this property in the estimation of iron (ferrous) salts. Thus, if a solution of a ferrous salt is acidified with a dilute acid (sulphuric) it is at once converted into a ferric salt on adding a solution of potassium bichromate in sufficient quantity, the reaction being as under.

$$6FeSO_4 + K_2Cr_2O_7 + 7H_2SO_4 = 3Fe_2(SO_4)_3 + Cr_2(SO_4)_3 + K_2SO_4 + 7H_2O.$$

From this it will be seen that one equivalent or 294·42 parts of potassium bichromate will convert 6 equivalents, or 336 parts of iron, from the ferrous into the ferric state. The final completion of the change of ferrous into ferric salt in the above reaction is ascertained by bringing a drop of the solution in contact with a drop of solution of freshly prepared potassium ferricyanide, when no blue coloration will be produced. So long as the faintest trace of ferrous sulphate remains, a drop of the liquid will give a blue colour with the solution of potassium ferricyanide.

The solution of bichromate can be standardised synthetically. $\frac{1}{60}$ of 294·42 grams = 4·907 grams of the pure dry reagent, dissolved in distilled water to 1 litre, should give a solution of which each c.c. peroxidises $\frac{1}{10}$ Fe = 0·0056 gram of iron.

The principal impurity in the salt to be avoided is sulphate. In order to test for it, dissolve 1 gram in 10 per cent. hydrochloric acid, and heat with a few drops of alcohol, to reduce the $CrO_3$ to $Cr_2Cl_6$. Dilute the dark-green solution, add chloride of barium, and allow to stand for twelve hours. Then decant off all except a few drops into another vessel, and dilute the slight residue with distilled water. The least quantity of sulphate of barium becomes visible.

In order to dehydrate the salt it is powdered, and after having been kept near its fusing point for about 10 minutes, it is fused at the lowest sufficient temperature. On cooling it breaks up into numerous little fragments, and thus spontaneously assumes a convenient form for being weighed out.

The titre, as calculated from the synthesis, ought to be perfectly correct; but unfortunately there is no method for proving the absence of surplus chromic acid in the salt, which, if present, would alter the strength of the solution. It is necessary, therefore, to test the strength of the solution by direct experiment. For this purpose a solution of ferrous sulphate of known strength is required. This is prepared from a weighed quantity either of metallic iron, or of ferrous ammonium sulphate.

In order to prepare the ferrous sulphate from metallic iron, about 1 gram of flower wire, free from rust, and in pieces of about an inch long, are accurately weighed out. Then fit up the apparatus shewn in Fig. 35. This consists of a 10-ounce round flask, provided with a perforated cork, through which passes a bent glass tube, one end of which passes just through the cork, while the other end dips below the surface of a little water contained in a small beaker. The flask is clamped in a slanting position upon a retort stand as shewn. The flask is now half filled with dilute sulphuric acid, a small piece of pure calc spar dropped in, and the cork and tube replaced. The calc spar will dissolve, giving off carbon dioxide, which will replace the air in the flask. When the effervescence has been kept up for some time, add the weighed wire, fit in the cork and tube, and arrange the apparatus as shewn in the figure. Heat the flask gently, and continue the heating until the wire is dissolved. While the iron is dissolving, some cold water free from dissolved oxygen may be prepared by boiling some distilled water for a few minutes, and then rapidly cooling it by immersing the flask in cold water. As soon as the iron wire is all dissolved, cool the solution, transfer it rapidly to a 250 c.c. flask, and rinse out the flask in which the iron was dissolved several times with the air-free water prepared as above, into the measuring flask, finally making up the solution to the 250 mark with the air-free water.

The titration is conducted as follows:—A 50 c.c. burette is filled with the solution of potassium bichromate. 25 c.c. of the iron solution are measured out into a 10-ounce flask, and diluted with about 50 c.c. of air-free water. A number of drops of a very dilute solution of a freshly prepared solution, of potassium ferricyanide are then placed on a white tile by means of a glass rod. (The solution of ferricyanide is prepared when required from a compact crystal of the salt, previously washed with water in order to remove any ferrocyanide. The solution does not keep long, especially if exposed to the action of light. Before use it must be tested with pure ferric chloride, with which it should give a brown colour, free from all shade of green.)

The solution of bichromate is now run from the burette into the iron solution, mixing well after each addition. As the solution of bichromate is being run in, a drop of the iron solution is taken out of the flask from time to time, by dipping a clean glass rod into the liquid, and bringing the drop adhering to the rod into contact with one of the drops of potassium ferricyanide solution on the tile. With the first few drops a deep blue coloration will be produced, but as the addition of the bichromate proceeds, the colour produced by the ferricyanide will gradually become fainter, and will finally disappear. When this stage is reached the volume of bichromate solution used is read off.

Two more similar titrations should then be made, the whole volume of bichromate solution required, as obtained from the first or preliminary titration, being run in at once within half a c.c., and the last portion being added gradually, drop by drop. The results of these separate titrations should not differ by more than the tenth of a cubic centimetre.

It is necessary to multiply the weight of iron contained in the solution employed in this titration by 0·996, in order to correct it for impurities present in the wire orginally used, and from the weight of pure iron thus found, the exact strength of the standard solution of bichromate may be calculated.

*Example*:—1·338 grams of flower wire were dissolved as above, and the solution made up to 250 c.c. 25 c.c. of this solution required 23·6 c.c. of the standard bichromate solution.

Therefore 236 c.c. of bichromate solution correspond to 1·338 grams of wire.

Consequently 1000 c.c. will correspond to $\frac{1 \cdot 338 \times 1000}{236}$ $= 5 \cdot 6695$ grams of wire, which are equal to $5 \cdot 6695 \times 0 \cdot 996$ $= 5 \cdot 6468$ grams of pure iron.

Each c.c. of the bichromate solution is therefore equivalent to 0·005646 gram of Fe.

In order to standardise the bichromate solution by means of ferrous ammonia sulphate, $Fe(NH_4)_2(SO_4)_2 6H_2O$, weigh out exactly seven grams of the substance, dissolve it in air-free water, and make up the solution to 250 c.c. as already described. The above weight of the salt will make a solution containing four grams of iron per litre, since the salt contains one-seventh its weight of iron.

The solution is titrated in exactly the same manner as the solution of iron wire just described, with the exception that it is necessary to add a little dilute sulphuric acid to the iron solution, before running in the bichromate.

The above method may be applied in the determination of the amount of iron contained in the oxide of iron employed in gas purification.

64. The following example shows the method of procedure in the case of hæmatite, which is an iron ore very rich in ferric oxide. Weigh out about 2·5 grams of hæmatite iron ore in the state of very fine powder. Place the ore into a beaker, and add hydrochloric acid diluted with its own volume of water. Cover the beaker with a clock-glass, and apply a gentle heat for about half-an-hour. Then add a *small* quantity of distilled water, allow any portion not dissolved to settle down, and decant the solution through a filter. Treat the residue again with a small quantity of dilute acid, and decant the liquid through the same filter as before. By these operations all the iron will generally be extracted from the ore, but it is necessary to see whether this is the case or not by testing a drop of the solution with a solution of potassium sulphocyanide. Should a decided red coloration ensue, it will be necessary to treat the ore a

third time with acid, and continue this treatment until no more iron is extracted. When this stage is arrived at, the residue is transferred to the filter, and washed free from acid, using as small a quantity of water as possible during the operation. The iron which is present in the solution in the ferric state is now converted into the ferrous state.

In order to effect this conversion, the solution and washings are transferred to the flask shown in Fig. 35. Should the whole of the liquid more than half fill the flask, it will be necessary first to evaporate it down to the bulk required. Strong hydrochloric acid is then added, and fragments of granulated zinc free from iron are dropped into the flask. The acid will dissolve the zinc, with the evolution of hydrogen. A portion of the nascent hydrogen thus evolved will act upon the ferric chloride and reduce it to ferrous chloride, the colour of the solution changing at the same time from yellow to pale green. After a time the action will become rather sluggish; it will then be necessary

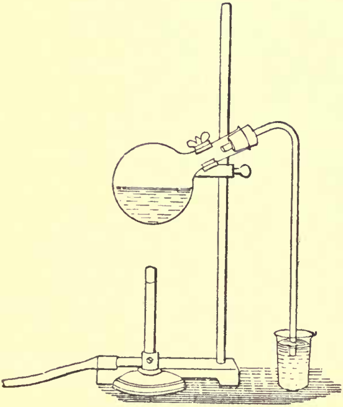

FIG. 35.

to gently heat the liquid in order to promote the action, continuing the heating until all the zinc is dissolved.

Before proceeding with the titration, it is necessary to see that the whole of the iron is converted into the ferrous state, by taking out a drop of the solution with a clean glass rod, and bringing it into contact with a drop of potassium sulphocyanide deposited on a white tile. No red colour, or only an exceedingly faint pink tint, should result. If a distinct red colour is produced, it will be necessary to add more zinc and hydrochloric acid, and the operation of reduction continued as above until all the zinc has dissolved. When the zinc has dissolved, the solution is then again tested with sulphocyanide, and the same operations repeated until all traces of the ferric salt has disappeared.

When the whole of the iron has been converted to the ferrous state, the solution is rapidly cooled out of contact with the air, and immediately made up to 250 c.c. with air-free water.

The titration is then at once effected as in (63), and completed as soon as possible.

The value of the bichromate solution being known, from the volume of that solution used, the quantity of iron present in the ore may be calculated, and from that the percentage.

## USE OF STANDARD IODINE SOLUTION.

65. Iodine dissolved in iodide of potassium may be used for numerous volumetric determinations, amongst others that of $H_2S$ in aqueous solution, and upon this, a method for determining the $H_2S$ in gas liquor is based. In the present example we require a decinormal solution of iodine, and a solution of starch.

The solution of iodine should contain $\frac{127}{10} = 12 \cdot 7$ grams of pure iodine per litre. 12·7 grams of pure resublimed iodine are accurately weighed out from a stoppered bottle into a litre flask. About 20 grams of pure potassium iodide are then added, together with about 250 c.c. of distilled water. The contents of the flask are well shaken until the iodine is completely dissolved, when the flask is filled up to the mark with distilled water.

Owing to the volatility of iodine, it is difficult to weigh out exactly the quantity requisite to make the standard solution. In order to get over this difficulty, about 13 grams may be weighed out by difference, this is then dissolved in the manner above described, and made up to the volume necessary to furnish a solution of decinormal strength. Thus, supposing 12·826 grams were weighed out, the volume of the iodine solution required would be $\frac{1000 \times 12 \cdot 826}{12 \cdot 7}$ = 1009·9 c.c. This solution must be kept in a well stoppered bottle, and in a cool place.

In order to prepare the starch solution, about 1 gram of starch free from acid is made up into a thin cream with a small quantity of cold water. This mixture is then poured into about 100 c.c. of boiling water contained in a porcelain dish, the boiling being kept up for a few minutes after adding the starch. The liquid is then allowed to stand until cold, when the clear solution is poured off, being then ready for use. Starch solution freshly prepared is much more sensitive to the action of iodine than that which has been kept for some time, consequently, wherever practicable, the solution should be prepared immediately before use.

When a dilute solution of sulphuretted hydrogen is brought into contact with free iodine the following reaction takes place.

$$H_2S + I_2 = 2HI + S.$$

In this determination it is necessary that the solution containing the $H_2S$ to be determined does not contain more than 0·04 per cent. of the gas, otherwise there is a possibility of the change not taking place entirely according to the above equation.

Measure out a portion of sulphuretted hydrogen water made by passing the gas into water, and titrate this with the decinormal iodine solution. This titration is made in order to give a rough idea of the quantity of $SH_2$ in the solution, should the latter contain more than 0·04 per cent. of the gas, the rest of the solution is measured out and diluted with air-free water to the requisite volume, and a measured volume of this diluted solution titrated with the iodine solution. This titration will indicate very nearly the proper amount of iodine solution required.

During the process of titration as conducted above, there is a liability of loss of gas by its escaping into the air, and also by the oxidising action of the same, and these tend to make the results rather low. In order to avoid this source of error the following method of procedure may be adopted. Introduce into a flask almost but not quite as much iodine solution as was required in the last titration, and add to this the quantity of diluted sulphuretted hydrogen solution employed in that particular titration. This amount will destroy the colour of the iodine solution. Now add a little starch solution, and carefully run in more iodine solution until a permanent blue colour is just obtained.

From the total volume of iodine solution used the amount of sulphuretted hydrogen can be calculated. Each c.c. $\frac{N}{10}$ iodine solution = 0·0017 gram $H_2S$.

# PART IV.

## SPECIAL ANALYSES REQUIRED IN GASWORKS.

### I. Proximate Analysis of Coal.

66. The proximate analysis of coal consists of the following determinations:—Moisture lost at some stated temperature, volatile matter, fixed carbon, ash, sulphur. The actual amounts of carbon, hydrogen, and nitrogen, are determined by means of elementary organic analysis.

#### *Estimation of Moisture.*

The actual temperature at which coal should be dried is rather a disputed point. It is known that coal does not lose all its moisture at 100° C., consequently some analysts recommend drying at a higher temperature than that of boiling water, but as 100° C. is a temperature easily obtained and kept constant, it is a convenient temperature to adopt as a standard. Powder about two grams of the coal very finely, weigh the same between two watch-glasses, and dry in the water-oven at a temperature of 100° C. Weigh every two hours. After drying for some time, it will be noticed that the coal increases in weight, consequently no constant weight will be obtained, the lowest weight observed should therefore be taken. The increase of weight, after drying for a period, has been attributed as being probably due to the slow oxidation of some matters in the coal (pyrites, &c.), or it may be caused by the absorption of gases into the pores of the coal which the expelled moisture left vacant.

### *Estimation of Volatile Matter and Coke.*

Take about two grams of finely powdered coal, and spread it in an even layer on the bottom of a thin platinum crucible. Weigh without the cover. Cover the crucible with its lid, and place it in an upright position on an iron ring, round which, sufficient platinum wire has been wound to prevent any contact between the iron and the platinum crucible. Then apply a powerful gas flame (to obtain comparable results, the length of the flame and the position of the crucible over it, should be the same in all experiments). Notice when the gases cease issuing from beneath the lid, continue the heating one minute longer, then take away the gas flame, place the crucible and cover for about five minutes in a desiccator to cool, and weigh it without the lid as soon as possible. The loss of weight observed represents the volatile matter which has been driven off by the heating.

### *Estimation of the Ash.*

Weigh out accurately about two grams of finely powdered coal or coke in a platinum boat, made by bending a piece of platinum foil over a glass rod. The boat should be capable of being easily pushed into a piece of combustion-tube of about one inch in diameter. The combustion tube and boat are then heated to redness in a gas-furnace; at the same time a gentle current of air should be drawn through the tube by means of an aspirator. Care should be taken that the temperature does not become high enough to soften the glass, or the boat and tube may be fused together. The apparatus is shown in Fig. 36.

FIG. 36.

As soon as the combustible matter is entirely consumed, allow the tube to cool, then withdraw the boat and weigh it. By this method of working, a beautifully clean ash can be obtained in a very short time. It is advisable to take a separate portion of coal for the determination of ash, and not to weigh a portion of the coke obtained in the determination of the volatile matter, as coke absorbs moisture very rapidly. The ash should be preserved for the further determination of the sulphuric acid which it contains.

### *Estimation of Sulphur.*

In determining the sulphur in coal, it is necessary to distinguish between the sulphur which goes over in the volatile matter, the sulphur remaining in the coke, but which can be converted into $SO_2$ by combustion, and the sulphur which is finally left in the ash combined as sulphate. For this purpose three determinations will be necessary.

(1) *Total Sulphur.*—This is best obtained by the method devised by Nakamura, which consists in heating the coal below a red heat in contact with alkaline carbonates, by which the coal, whether bituminous or not, rapidly undergoes without evolution of smoke, complete atmospheric oxidation, in a manner hardly to have been expected. The details of the method are as follows :—

Weigh out accurately about two grams of the *very finely powdered* coal, and mix it with four times its weight of dry fusion mixture (sodium and potassium carbonates mixed in their molecular proportions). The mixing is effected by adding the fusion mixture gradually to the coal, contained in a platinum crucible, and stirring constantly with a dry glass rod. The crucible is then loosely covered with its lid, and the mixture at first gently heated, so as not to volatilise the hydrocarbons, that is, so that no smell, or only a faint aromatic odour is given off. An Argand spirit lamp should be used as the source of heat, in order to avoid the possible absorption of sulphur from the flame of coal gas, which might occur if a Bunsen or other gas burner were used. Keep at a low temperature for some time, then slowly increase the heat without letting it reach that of visible redness, until the surface of the mass, which is at first of a dark grey colour, becomes only faintly grey. No smoke or odorous gases should escape during the whole of the oxidation. When the surface becomes only faintly grey, raise the temperature to a faint red heat, and keep it stationary for about forty to sixty minutes; at the end of this time the mass will have become almost white, owing to the complete combustion of the coal, but if iron be present in the coal the tint will be reddish. The crucible is now allowed to cool, and then placed in a beaker of distilled water, the beaker covered with a clock-glass, and gently heated for some time, when it will generally be found that the mass is wholly detached from the crucible, and the greater part dissolved. When that stage is reached, the crucible is carefully lifted out of the beaker with the crucible tongs, both tongs and crucible being well rinsed with distilled water from the wash-bottle into the beaker. Pure hydrochloric acid is now added to the contents of the beaker, in order to drive off the carbonic acid (due to the fusion mixture); the acid should be added very cautiously, and in small portions at a time, the clock-glass being immediately replaced after each addition, otherwise there is a risk of loss by spurting. When all the $CO_2$ appears to have been driven off, the solution should be raised to incipient ebullition, and maintained at that temperature for some time. It is then filtered, with the object of removing sand, &c., which need not be weighed. It is necessary to be careful not to lose any of the filtrate, and the filter-paper should be washed until it ceases to redden blue litmus-paper. The filtrate is then treated with a few drops of bromine, brought to the boiling point, an excess of solution of barium chloride added, and kept boiling for about ten minutes longer, the precipitated $BaSO_4$ is allowed to settle, filtered, washed free from acid and excess of $BaCl_2$, dried, ignited, and weighed, in the same manner as in the typical analysis of barium. From the weight of $BaSO_4$, the amount of sulphur in the coal operated on (233 parts $BaSO_4$ = 32 of S) is obtained, from which the percentage is easily calculated. To secure the complete oxidation of the coal by the above method, it is most important that it should be powdered as finely as possible; the best plan is to sift it through cotton cloth. Stirring the mixture during the oxidation, impedes the process instead of hastening it, as might have been expected, probably by closing the passages left by the unconsumed coal.

(2) *Volatile Sulphur in the Coke.*—The sulphur, which is converted into sulphur dioxide on combustion of the coke in air, is determined as follows:—

A quantity of coke, equivalent to a known weight of coal, is roasted in a platinum boat, in a combustion tube in a current of air, as in the determination of ash, Fig. 36 p. 114. The gaseous products of combustion are aspirated through 10 c.c. of standard solution of iodine placed in a bulbed U-tube. The following reaction then occurs:—$SO_2 + I_2 + 2H_2O = H_2SO_4 + 2HI$. The amount of free iodine remaining in solution is determined by standard solution of sodium thiosulphate, and the amount acted on by the $SO_2$ is obtained by difference. From this the weight of $SO_2$ and of S may be calculated.

(3) *Sulphur in the Ash.*—This can be done in most cases by boiling the ash with a little hydrochloric acid, filtering, and washing the residue. The sulphate is determined in the filtrate and washings as in (1), or the ash could be fused with alkaline carbonates.

67. The following are methods for ascertaining the amount of carbon, hydrogen, nitrogen and oxygen in coal. Organic bodies (of which coal is one) are always composed of hydrogen and carbon, generally in combination with oxygen, and often with nitrogen, the other elements, such as chlorine, sulphur, &c., occurring much less frequently.

When any organic substance containing carbon and hydrogen is burnt in air or in oxygen, these elements combine with the oxygen, and are converted into carbon dioxide and water respectively. Therefore, by making suitable arrangements, it is possible to collect and weigh the $CO_2$ and $H_2O$, and from the weight obtained, to deduce the amount of carbon and hydrogen in the substance operated on.

The operation of burning is termed making a "combustion," and is generally effected in a current of air or oxygen; but a common method is to heat the organic substance with some material which will readily part with its oxygen, such as oxide of copper or chromate of lead.

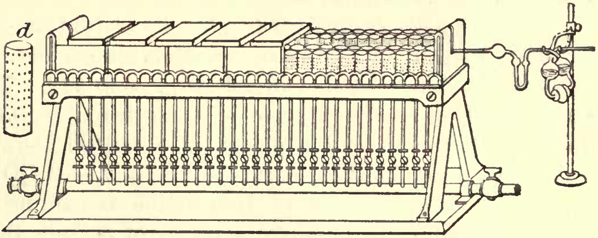

FIG. 37.

As combustions are always made in tubes (generally of glass) a furnace for heating the same is needed. There are a number of different forms of furnaces in use; the one shown in Fig. 37 is known as "Hoffman's." In this form of furnace the combustion of coal gas, supplied by a series of vertical gas jets, is effected by means of a number of perforated clay burners in which the gas is mixed with air. These burners are arranged so as to form a channel for the combustion tube, a system of stop-cocks serving to regulate the heat, or to confine it within any desired limit. The tube in which the combustion is made, known as the "combustion tube," should be of very hard glass, so as not to soften at a red heat, or, if it does so, not sufficiently to allow it to be blown out by the internal pressure of the gases. In order to make a combustion tube, procure a piece of good combustion tubing, about $\frac{1}{2}$ inch in diameter and 3 feet long, thoroughly clean it inside, by passing a piece of cotton wool attached to a string through it several times; next heat the tube strongly in the centre over the blow-pipe flame, turning it constantly round while doing so; then, when it is quite soft, separate the two halves in a direction at right angles to the length of the tube; let it cool, and when cold cut it in two at the narrowest part, and seal up the points in the blow-pipe so as to make two tubes of the form shown in Fig. 38, and finally round off the edges of the open end in the blow-pipe flame. A soft cork which fits the tube tightly, and pierced with one hole will also be required. The water is collected in a calcium chloride tube, Fig. 39. This tube is filled with granulated calcium chloride in small fragments (but no dust), a loosely fitting plug of cotton-wool being placed over the $CaCl_2$ at D, in order to prevent any small particles of $CaCl_2$ from being carried over. The open end of the tube is closed with a tight-fitting cork pierced with one hole, through which a piece of glass tube bent at right angles passes. This cork should be made quite gas-tight, by coating it with sealing-wax or melted paraffin. The apparatus should be provided with a loop of platinum wire, to enable it to be hung on the hook of the balance when weighing. When not in use, the ends of the tubes should be closed by small corks, made by cutting down the portions of cork left

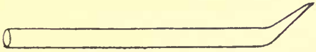

Fig. 38.

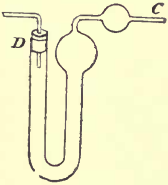

Fig. 39.

in the cork-borer when boring corks. The $CO_2$ is absorbed by the potash bulbs, shown in Fig. 40. The bulbs are filled, to the extent indicated by the dotted line in the figure, with strong potash solution, prepared by dissolving three parts of potash free from carbonate, in two parts of water. The bulbs are readily filled with this liquid contained in a porcelain dish, by dipping the end of the tube connected with the larger bulb beneath the surface of the potash solution, and gently aspirating at the other tube, until the required amount has been drawn in. The tube is then carefully dried, inside and out, with filter-paper, and the ends of the tube closed with small corks. A piece of platinum wire is twisted round the tubes where they touch, in the manner shown in the figure; this serves to suspend the bulbs from the hook of the balance-pan.

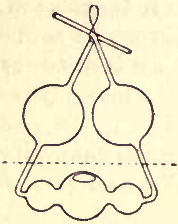

FIG. 40.

In order to make a combustion of a sample of coal, the combustion tube, Fig. 38, is made perfectly clean and quite dry. It is then filled for about 14 inches of its length with pure chromate of lead, which has previously been made hot. A sample of the coal is reduced to fine powder, and about five grains are weighed, and carefully dried, at a temperature of 240° F. About a fourth of the lead chromate in the combustion tube is then placed in a hot porcelain mortar, the coal is added, and the two are intimately mixed. The remainder of the chromate in the tube, with the exception of a couple of inches is then added, the whole well mixed, and quickly transferred back without loss to the combustion tube. The mortar is then rinsed with a little fresh hot chromate, which is also placed in the tube, which is then filled up to within 3 or 4 inches of its mouth with pure hot chromate. Some pure copper turnings are then pressed into the remaining space, so as to effect the decomposition of any oxides of nitrogen which may be formed during the combustion. The combustion tube is then rapped horizontally on the bench, so as to shake down the mass, in order to allow a free passage above by which the evolved gases can escape; it may then be placed in the furnace, the open end of the combustion tube projecting about an inch from the furnace. The calcium chloride tube, Fig. 39, previously weighed, is attached to the combustion tube by means of its cork, and to the end of this are connected, by a short piece of india-rubber tubing, the previously weighed potash bulbs, Fig. 40.

After making certain that all the connections are quite tight, the combustion is started, by lighting the gas burner nearest to the open end of the tube. As soon as the front of the tube attains a dull red heat, the next gas tap is opened, and the same operation gradually performed along the whole length of the tube, until the whole length attains a dull red heat. (It is necessary to effect the heating gradually, in order that the gases evolved may pass away slowly, as, if they were driven off too rapidly, a portion might escape absorption.)

As soon as bubbles cease to pass through the potash bulbs, the point of the combustion tube is broken off by means of a pair of pincers, and a current of dry atmospheric air, free from $CO_2$, is slowly drawn through the apparatus, by means of a suction tube connected to the end of the potash bulbs. In this way, the whole of the $CO_2$ and water vapour remaining in the apparatus is swept through, into the receptacles intended for their detention. The chloride of calcium tube and potash bulbs may now be disconnected, allowed to cool, and when cold re-weighed.

The gain in weight of the $CaCl_2$ tube is due to water, and of the potash bulbs to $CO_2$.

As an example, supposing that we had weighed out 5·5 grains of coal, and we found that the $CaCl_2$ tube had gained 3·2 grains in weight, and the potash bulbs 15·7 grains, then, in order to find out how much hydrogen the 3·2 grains of $H_2O$, are equivalent to, we should say—

Mol. weight of water    Mol. weight of hydrogen

As 18 : 2 :: 3·2 : $x$. $x$ = ·355;

and to obtain the percentage—

As 5·5 : 100 :: ·355 : $x$. $x$ = 6·46.

To obtain the amount of carbon the 15·7 grains of $CO_2$ are equal to, we should say—

Mol. weight of $CO_2$    Atomic weight of carbon

As 44 : 12 :: 15·7 : $x$. $x$ = 4·277,

and to obtain the percentage—

As 5·5 : 100 :: 4·277 : $x$. $x$ = 77·77.

In order to determine the amount of nitrogen, the latter element is converted into ammonia by a combustion with soda lime, in a similar combustion tube to the one employed in the determination of hydrogen and carbon, but it need not be so long. A convenient length is about 12 inches. About 9 inches of the length is filled with dry soda lime. A portion of this (about a third) is then shaken out into a porcelain mortar, and intimately mixed with about ten grains of finely powdered coal previously dried.

More soda lime is then shaken out (leaving an inch in the tube), mixed, and the whole replaced in the tube. The mortar is then rinsed with a little soda lime, which is added to the contents of the tube, which is now nearly filled with fresh soda lime. A plug of asbestos is placed in the mouth of the tube, the contents of which are then shaken down so as to leave a space for the passage of the gas; the tube is then placed in the combustion furnace.

The bulbs, Fig. 41, are now connected with the combustion tube by means of a perforated cork. The bulbs are filled with dilute sulphuric acid, made by adding 10 c.c. of normal sulphuric acid and diluting with distilled water. The combustion is then conducted as in the determination of carbon and hydrogen, but it may be effected more rapidly. When the evolution of gas has ceased, the bulbs are disconnected, and the contents separated from tarry matters by filtration.

The solution is then coloured with a few drops of cochineal solution, and titrated with a standard solution of caustic soda, of such a strength that 3 c.c. of the soda solution neutralise 1 c.c. of the acid solution. From the number of cubic centimeters of the soda solution required, the amount of ammonia may be determined as in (59).

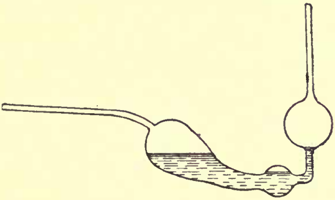

FIG. 41.

The amount of oxygen in an organic compound is always estimated by difference, that is, having ascertained the weight of all the other elements entering into the composition of the body under examination, the amount required to make up the 100 parts is put down as oxygen.

The above description is only intended to give a general idea of the method of performing an organic analysis, it is almost impossible to describe the various precautions necessary to be observed in order to make a reliable determination by this method: the only way of obtaining the requisite knowledge is to see an analysis actually performed by a skilful operator.

## *Specific Gravity.*

68. It is occasionally requisite to know the weight of a cubic foot of the coal, or the number of cubic feet corresponding to a ton. This is easily arrived at when the specific gravity of the coal is known. The specific gravity of a body expresses the relation which exists between the weights of equal bulks of one substance compared with another, chosen as the standard. For solids and liquids, the standard is distilled water at the temperature of 60° F.

In order to take the specific gravity of a sample of coal, a small piece weighing about an ounce is selected, and suspended by means of a horse-hair, or piece of fine silk, from the specific gravity pan of the balance. It is then accurately weighed, while hanging in the air. It is then immersed in a beaker of distilled water, as shown in Fig. 42, and again weighed. Before the weight in water is recorded, any adhering air bubbles must be removed from the surface of the coal. This is effected by removing the sample from the water after it has been immersed for a short time, and brushing the surface with a moistened camel's-hair brush. It can then be put back again into the water, and the weighing completed.

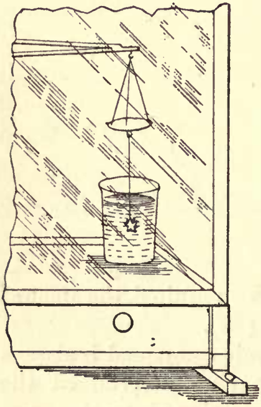

FIG. 42.

It is also desirable that the coal be sufficiently soaked; this is effected by immersing the sample, after attaching the horse-hair to it, in water in the flask of the filter pump, and exhausting the air within the apparatus as far as practicable.

The difference between the weight of the coal when weighed in air, and when weighed in water, is the weight of a bulk of water equal to the bulk of the coal immersed, hence the weight of the coal in relation to that of water; in other words its specific gravity, is found by dividing the weight in air by the loss of weight in water, as under.

| | Grains. |
|---|---|
| Weight of coal in air .. .. .. .. .. .. .. .. | 526·7 |
| „ „ water .. .. .. .. .. .. .. | 140·6 |
| Loss of weight in water, or difference representing the weight of an equal bulk of water | 386·1 |

Then $\frac{526 \cdot 7}{386 \cdot 1} = 1 \cdot 346$, the specific gravity of the coal.

The specific gravity being ascertained, the remaining data are easily calculated from the same. A cubic foot of water at 60° F., weighs exactly 1000 ounces, hence a cubic foot of coal having the specific gravity given above, will weigh 1364 ozs., or 85·25 lbs. avoirdupois. Having obtained this information, the measurement or space occupied by a ton of the same coal is arrived at as under—

$$\frac{2240}{85 \cdot 25} = 26 \cdot 28,$$

the measurement of a ton of the coal in cubic feet.

## II. Estimation of the Impurities in Crude Gas.

### *Sulphuretted Hydrogen and Carbonic Acid.*

69. These impurities may be determined with accuracy by the method devised by Mr. L. T. Wright, F.C.S., and described by him in a paper read before the Nottingham Section of the Society of Chemical Industry in the year 1885.

This method, which is founded upon the increase in weight of absorption tubes, admits of the use of a tolerably large quantity of gas, collected regularly during an interval of time sufficiently long to give an idea of the average composition of the gas supply to be tested. The reagent employed for the absorption of $SH_2$ in absorption tubes, may be described as an impure di- tri- ortho-phosphate, and may be prepared in the following manner. Solutions of 2 lbs. of hydrogen disodium phosphate in 1 gallon of water, and 2½ lbs. of cupric sulphate crystals in 1½ gallons of water are mixed with vigorous stirring, and the resulting bright blue precipitate washed by decantation, and then dried in a water bath to about 100° C. The material sometimes is light and powdery, sometimes hard, and then requires crushing in a mortar. Its action with $SH_2$ is very sharp.

The U-tubes used in these experiments are preferably of the modern form, with hollow round glass stoppers, which also serve as stop-cocks; the inlet and outlet connections being small pieces of glass tubing, fused into the sides of the U-tubes. A small hole in the stopper, corresponding to the hole in the side of the tube where the glass tubing is fixed on, makes the connection. A slight turn of the stopper opens or closes the tube. The tubes are weighed when charged with clean coal gas, which, on the same gas supply, varies but little in specific gravity.

The stoppers are lubricated with vaseline or resin cerate, and with this exception are perfectly clean. A small plug of cotton wool in each stopper, prevents the mechanical carrying forward of fine particles of the reagents by issuing gases.

Closed tubes charged with coal gas do not suffer any appreciable variations in weight during a period of two days. Mr. Wright found that such tubes, charged with cupric phosphate, gain in weight under the action of clean coal gas, but the increase soon reaches a limit, and the phosphate becomes "saturated." In order to attain this saturation point, it is necessary to pass 3 cubic feet of clean dry coal gas slowly through the tubes.

In order to charge the phosphate tubes, they are first cleaned and dried, great attention being paid to the ground portions of the tubes and stoppers. A plug of cotton wool is placed in the bend of the tube, then one leg of the same is charged with the cupric phosphate in a suitable state of subdivision, care being taken that the rough and fine portions of the powder are regularly distributed, for if all the rough portions are on one side and the fine on the other, a passage of the gas down the side where the coarse portions are takes place, and consequently the fine portions never come into action. The other leg is charged with powdered calcium chloride.

The ground portions of the tubes are wiped free from dust, and the stoppers, fitted with plugs of dry cotton wool in the hollow places, and lubricated with a little resin cerate, are put in position.

A current of dry coal gas to the extent of 3 cubic feet is passed through, and the tubes are then ready for weighing. A 6-inch U-tube charged in this manner will be capable of absorbing 20 grains of $SH_2$; and if, in the case of gas containing 10 grains of $SH_2$ per cubic foot, the experiments are made upon quantities of 0·5 cubic foot, each will serve for four analyses without being recharged. The calcium chloride will last much longer. The $CO_2$ is absorbed by soda lime, in a similar way, one half of a U-tube being filled with soda lime, and the other half with calcium chloride. Mr. Wright found it necessary to use the soda lime tubes in a moist condition, for when quite dry, soda lime has a much feebler absorptive power for $CO_2$. The requisite degree of moisture may be attained by exposing the soda lime to a moist atmosphere from twelve to eighteen hours. There are no inconveniences attending it in this condition. These soda lime tubes remain very constant in weight, when clean, pure, coal gas is passed through them, and therefore do not require saturating in the same manner as the phosphate tubes. A small quantity of dry, pure, coal gas should, however, be passed through, to expel air previous to their being weighed.

A 6-inch tube, charged with soda lime in the manner above described, serves for three analyses of 0·5 cubic foot each, of gas containing from 10 to 12 grains of $CO_2$ per cubic foot.

In cases where there is also ammonia in the gas to be tested, as in the inlet to the scrubbers, it will be requisite to effect its removal before the gas reaches the weighing tubes. This might be done by means of the drying cylinder containing calcic chloride, which effectually removes $NH_3$ from coal gas; but in order to maintain the neutrality of the calcium chloride, it is better to remove the $NH_3$ from the gas before drying, by passing the gas through a 12-inch U-tube filled with pieces of broken pumice saturated with syrupy phosphoric acid. It should be noted that in drawing off samples of coal gas for analysis, certain precautions are necessary, since vulcanised and iron tubing, when new, remove $SH_2$ from crude coal gas, so that it is advisable to have the services conducting the gas from the source to be tested to the absorption tubes as short as possible, and previously saturated with crude coal gas. It is further of importance to keep a tolerably quick current of gas through the iron pipe, in order to prevent the gas from becoming "stale." The iron service should be fitted with a four-way piece and three cocks. One of these serves to take the gas to the train of apparatus, another to be left open during the experiment, blowing gas away, and thus maintaining a rapid current in the iron pipe, while the third can be used for other purposes, such as the examination of gas for ammonia, &c. Before making an analysis it is necessary to blow away a little gas, in order to clear stale gas out of the iron service pipe and bring along a fresh supply. The complete apparatus can now be described.

The first piece in the train is the syrupy phosphoric acid tube, which can, however, be dispensed with in cases where the gas has been previously thoroughly washed free from ammonia. This tube is directly connected with the cock on the iron pipe by means of a small piece of "saturated" vulcanised tubing.

The outlet of this tube is in communication with a large drying cylinder, filled with small pieces of calcium chloride quite free from alkalinity. The outlet of the drying cylinder is provided with a T-piece, which carries on one arm a small piece of vulcanised tubing and a screw pinch-cock, for the purpose of blowing away a little gas before and during each experiment. The other arm of the T-piece is connected to the inlet of the cupric phosphate tube, the outlet of the latter being connected to the inlet of the soda lime tube. A gas meter connected to the outlet of the soda lime tube completes the apparatus. The meter most suitable for the purpose, is the test-meter employed in the Gas Referees' Sulphur Test. It is advisable to bring the pointer attached to the meter up to within a division or so of the zero mark by means of the key provided for that purpose, and then to blow gas through until the pointer exactly covers the mark: by operating in this manner, any error due to slackness in the gearing of the index is avoided. Before starting an experiment, gas should be blown away at the outlet of the drying cylinder, as well as at the cock on the iron service. When the tubes have been weighed full of clean coal gas, and all the pieces of the train joined tightly together, the stop-cocks are turned on, and the gas is allowed to traverse through the apparatus at a moderate rate, which is often more dependent on the pressure of the gas, than on the will of the operator. From 0·25 to 0·50 cubic feet per hour is a convenient rate, and one well within the limit at which the complete action of the reagents is obtained. The quantity of gas used for each experiment is a matter of choice, and will be determined by the amount of impurity in the gas to be examined. When the required quantity of gas has passed through the tubes, the inlet stopper of the phosphate tube, and the outlet stopper of the soda lime tube are turned off, and the two tubes are taken off together, and connected in their proper order to a supply of desiccated clean gas, in order to drive the gas in the phosphate tube through the soda lime tube. The stoppers can then be turned off, and the tubes weighed. Of course, before weighing the tubes, they must be wiped perfectly clean and dry, but as this operation causes them to be a little light, they should be left for about five or ten minutes in the balance case before being weighed. The results in grains, corrected for temperature and pressure, are best calculated to a cubic foot of clean coal gas at 60° F. and 30·0″ Bar. Since washed, but otherwise unpurified, coal gas contains about 0·6 grain of cyanogen per cubic foot, the results obtained by this method are slightly high. The absorption of cyanogen in the first tube is not complete—about one-half going forward into the soda lime tube. The error falls equally on each tube. Cyanogen may be estimated by passing a measured quantity of gas free from ammonia, but otherwise unpurified, through a U-tube filled with soda lime, and then making a combustion of the residue, as in an ordinary nitrogen determination. The tubes may be conveniently supported by suspending them from hooks on a light wooden framework or "gallows" by means of fine copper wire.

Having obtained the weights of $SH_2$ and $CO_2$ contained in a definite volume of gas, it is an easy matter to convert the same into volumes and from these data to arrive at the percentage by volume, in which form the results are generally expressed. The following examples will show the method of performing the necessary calculations:—

(1) For $SH_2$. Volume of gas passed, corrected to 60° F. and 30·0″ Bar. = 0·426 cubic feet.

| | Grains. |
|---|---|
| Weight of copper-phosphate tube after experiment | 1593·7 |
| " " " before " | 1590·6 |
| " $SH_2$ absorbed .. .. .. .. .. .. | 3·1 |

A cubic foot of $SH_2$ (dry) at 60° F. and 30·0″ Bar. may be taken as weighing 631·5 grains, consequently 1 grain will be equal to 0·0015835 of a cubic foot. Now in the example, as there are 3·1 grains of $SH_2$ in 0·426 cubic foot of gas, in 100 cubic feet there will be 727·7 grains; therefore 0·0015835 × 727·7 = 1·1523 cubic feet of $SH_2$ in 100 cubic feet of gas, or 1·1523 per cent. by volume.

(2) For $CO_2$. Volume of gas passed corrected to 60° F. and 30·0″ Bar. = 0·426 cubic feet.

| | Grains. |
|---|---|
| Weight of soda lime tube after experiment .. .. | 1763·4 |
| ,, ,, ,, before ,, .. .. | 1757·2 |
| ,, $CO_2$ absorbed .. .. .. .. .. .. | 6·2 |

A cubic foot of $CO_2$ (dry) may be taken as weighing 817·3 grains, consequently 1 grain will be equal to 0·0012235 of a cubic foot, and as there are 6·2 grains of $CO_2$ in ·426 cubic feet of gas experimented on, in 100 cubic feet there will be 1455·4 grains; therefore 0·0012235 × 1455·4 = 1·780 cubic feet of $CO_2$ in 100 cubic feet of gas, or 1·780 per cent. by volume.

It is necessary to notice that, as the gas is measured after the $H_2S$ and $CO_2$ are taken out, that the original volume will be greater than that actually recorded by the meter. In the above example it would be :—

| | | |
|---|---|---|
| Gas measured .. .. .. | 100 | cubic feet |
| $H_2S$ ,, .. .. .. | 1·152 | ,, |
| $CO_2$ ,, .. .. .. | 1·780 | ,, |
| | 102·932 | |

Consequently to get the true percentage it will be necessary to make the following calculations :—

For $H_2S$. As 102·932 : 100 : : 1·152 : 1·120 = 1·120 per cent.

For $CO_2$. As 102·932 : 100 : : 1·780 : 1·729 = 1·729 per cent.

70. *The carbonic acid in crude gas may also be estimated by a volumetric method devised by Mr. J. T. Sheard, F.C.S., and communicated by him to the Journal of Gas Lighting.*

The apparatus employed is shown in Fig. 43, and is a form of Volhard's absorption apparatus, as modified by Mr. Sheard; it may be said to consist of three parts, viz., the reservoir or body of the apparatus, the bulbs, and the scrubber, which is the long tube filled with pieces of broken glass or glass beads. The apparatus is made of blown glass, and has a total capacity of from 250 to 300 c.c. Not more, however, than about 40 c.c. of liquid, or the contents of rather more than one of the bulbs, can be employed in it for one estimation. When in use, the pressure of the gas forces the liquid out of the reservoir into the bulbs, and there bubbles through it.

The method of using the absorption tubes is to run into the reservoir, a known quantity of the absorbing liquid of known value, and then to cause a measured quantity of gas to bubble through it; the scrubber is afterwards rinsed with distilled water to wash down any portion of the absorbent that may have been mechanically carried into it by the gas, and the whole of the resultant liquid is titrated in the reservoir. The method adopted for the estimation of $CO_2$, consists in absorbing it in a solution of barium hydrate of known strength, the resultant liquid being afterwards titrated, in order to ascertain the amount of free hydrate remaining in solution, from which the amount of $CO_2$ absorbed is deduced. The indicator employed is phenol-phthalein, prepared by dissolving 0·5 gram of the substance in 1000 c.c. of 50 per cent. alcohol. In the presence of the hydrates of the alkalies and alkaline earths, this indicator gives a splendid purple-red colour, which is destroyed on neutralising the alkali by an acid, the end reaction being exceedingly sharp and distinct; carbonate of barium precipitated in a solution of the hydrate does not develop a red colour with phenol-phthalein, but remains neutral. It follows that the value of the solution of barium hydrate being known, a simple titration of the liquid after absorption of $CO_2$, will show the diminution in the amount of free hydrate present, and inferentially the amount of $CO_2$ absorbed. The solution of barium hydrate is made by dissolving the ordinary crystallised hydrate of baryta in water until the solution is saturated, and then syphoning off the clear liquid into a well-stoppered bottle. The strength of the clear liquid is then determined very accurately by titration with decinormal hydrochloric acid, and should be approximately equal in strength to that acid; i.e. if (say) much more than 10 c.c. of the acid are required to neutralise the same quantity of the baryta solution, a little water should be added to the latter to reduce its strength. It is necessary that the strength of the solution should be checked by titration from time to time, as, unless very carefully preserved from contact with the air, it gradually loses power through the absorption of $CO_2$. In addition to the gas absorption tubes, an aspirator, two 500 c.c. flasks, two 50 c.c. burettes (graduated into 1-10th c.c. and provided with glass stop-cocks), a supply of distilled water, and a decinormal solution of hydrochloric acid are required. Each cubic centimetre of this solution is equal to 0·0022 gram of $CO_2$. In order to make a test for $CO_2$ (the gas being previously freed from $H_2S$ by passing it through a small oxide purifier), a measured quantity—varying from 15 to 30 c.c. according to the amount of $CO_2$ expected to be met with in the gas—of the barium hydrate solution is run from a burette or pipette into the reservoir of one of the absorption tubes, and from 10 to 20 c.c., also accurately measured, into another tube. Should the smaller quantities mentioned be employed, an equal amount of distilled water is added, as the best results are obtained when the lower bulb of the first absorption tube is quite filled with liquid as the current of gas bubbles through it. About 30 c.c. is therefore the proper amount of liquid; if much more than this is employed, it is liable to be carried forward into the scrubber. The absorption tubes are placed in the stand, Fig. 43, and the apparatus connected up by means of india-rubber tubing and stoppers pierced by glass tubes, the inlet of the first tube being connected to the gas supply, and its outlet to the inlet of the second tube; the outlet of the latter being connected in turn, to the inlet of the aspirator. In connecting the gas-supply tube to the inlet of the first absorption tube, gas must first be allowed to blow away, and the india-rubber tube pushed over the bent glass tube while the gas is flowing, care being taken that the pressure of the gas supply is not so great as to force its way through the liquid in the bulbs without the aspirator being in operation. Assuming everything to be in readiness, the measuring flask is placed under the tap of the aspirator, and water allowed to flow, so as to draw the gas in bubbles through the liquid in the bulbs. It is necessary at this stage to see that the connections are quite gas-tight, which may be done by turning off the gas supply, and the tap of the

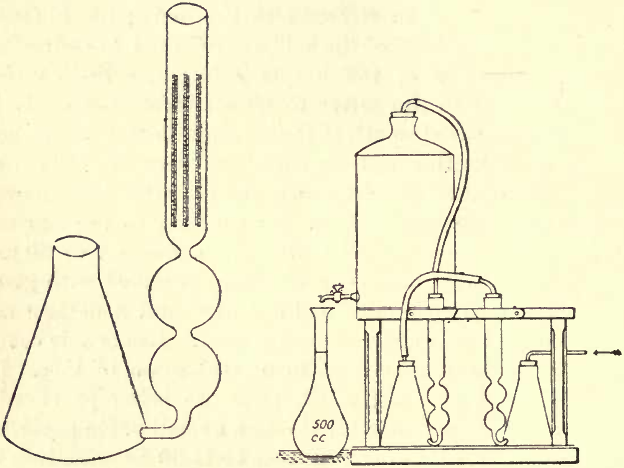

FIG. 43.

aspirator, and observing whether the level of the liquid in the bulbs remains stationary or not. If the connections are tight, the current of gas is continued until 500 c.c. have passed, as shown by the water that is run out of the aspirator. (Mr. Sheard found that the best speed of current was obtained when the flow of water from the aspirator is adjusted to the point at which it breaks from a succession of drops to a continuous stream.) The gas supply is then disconnected from the inlet to the first tube—which is done without stopping the flow of water, by slipping off the india-rubber gas-tube as the water in the flask passes the mark—a fresh flask is brought under the aspirator, and a further quantity of 500 c.c. of air drawn through the apparatus, in order to drive forward the gas remaining in the reservoirs of the two absorption tubes at the end of the first aspiration. The tubes are then disconnected, and a little water (free from $CO_2$) run through the scrubbers, in order to wash down any of the barium hydrate solution that may have been carried forward mechanically by the gas. A few drops of phenol-phthalein solution are now added, and the liquid is titrated with decinormal hydrochloric acid, the point of saturation being shown by the disappearance of the purple colour imparted to the liquid by the phenol-phthalein.

In titrating the carbonated liquid, the acid should be added a little at a time, and allowed to run down the sides of the reservoir, not dropped directly into the liquid, the liquid being gently agitated after each addition of acid. The difference between the quantity of free barium hydrate found at the close of the experiment, and that run into the tubes at the commencement of the same, shows the amount of $CO_2$ absorbed; and from this, the amount of the latter impurity existing in the gas may be calculated into grains per cubic foot, or percentage by volume.

*Example*:—Two gas absorption tubes were charged in the manner described, the first with 25 c.c. and the second with 18 c.c. of barium hydrate solution, 500 c.c. of gas followed by an equal quantity of air, being afterwards drawn through them.

At the end of the experiment, the scrubbers were rinsed with water, phenol-phthalein solution added, and the liquid in the tubes titrated with decinormal hydrochloric acid, 16·60 c.c. being required to neutralise that in the first tube, and 19·00 c.c. for the second. The titre of the barium hydrate solution used being 1 c.c. = 1·09 c.c. $\frac{N}{10}$ acid,

therefore,

| | First Tube. | Second Tube. |
|---|---|---|
| Equivalent of barium hydrate employed .. | 27·25 c.c. | 19·62 c.c. |
| $\frac{N}{10}$ acid required to neutralise the resultant liquid | 16·60 „ | 19·00 „ |
| Equivalent of $BaO,H_2O$ neutralised .. .. | 10·65 „ | 0·62 „ |

From these data, the percentage by volume, or grains, of $CO_2$ per 100 cubic feet of gas may be easily obtained, but in order to make it quite clear to those who are not used to such calculations, the process is described in detail.

(1) To obtain the percentage by volume.

The total equivalent of $BaO, H_2O$, neutralised as above, is $10{\cdot}65 + 0{\cdot}62 = 11{\cdot}27$ c.c., and as each c.c. is equal to 0·0022 gram $CO_2$, $11{\cdot}27 \times 0{\cdot}0022 = 0{\cdot}024794$ gram $CO_2$. Now the weight of 500 c.c. of $CO_2$ saturated with moisture = 0·914 gram, consequently—

$$\text{As } 0{\cdot}914 : 0{\cdot}024794 :: 500 : 13{\cdot}56,$$

that is, in the above experiment there were 13·56 c.c. of $CO_2$ in 500 c.c. of gas.

Therefore, in order to obtain the percentage we say—

$$\text{As } 500 : 100 :: 13{\cdot}56 : 2{\cdot}71 = 2{\cdot}71 \text{ per cent. by volume of } CO_2.$$

The above might be more shortly written thus:—

$$\frac{11{\cdot}27 \text{ c.c.} \times 0{\cdot}0022 \text{ gram} \times 100}{0{\cdot}914 \text{ gram}} = 2{\cdot}71 \text{ per cent. by volume,}$$

and more shortly still, by employing the factor 0·241 (obtained thus: $\frac{\cdot 0022 \text{ grm.} \times 100}{0\cdot 914 \text{ gram}} = 0\cdot 241$)

then 11·27 × 0·241 = 2·71 per cent. by volume of $CO_2$ as before.

(2) To obtain the weight in grains of $CO_2$ per cubic foot of gas.

As before, 11·27 × 0·0022 = 0·024794 gram $CO_2$, and as there are 15·432 grains in a gram, 15·432 × 0·024794 = 0·38262 grains $CO_2$ in 500 c.c. of gas. Now 1 cubic foot = 28,315 c.c., consequently—

As 500 : 28315 : : 0·38262 : 21·66 = 21·66 grains of $CO_2$ per cubic foot of gas.

This might be expressed more shortly thus:—

$$\frac{11\cdot 27 \text{ c.c.} \times 0\cdot 0022 \text{ gram} \times 15\cdot 432 \text{ grains} \times 28{,}315 \text{ c.c.}}{500 \text{ c.c.}}$$

= 21·66 grains $CO_2$ per cubic foot of gas, and from the figures $\frac{0\cdot 0022 \times 15\cdot 432 \times 28{,}315}{500}$ the factor 1·922 is obtained, which shortens the calculation materially thus: 1·92 × 11·27 = 21·66 grains $CO_2$ per cubic foot of gas as before.

*The amount of sulphuretted hydrogen in crude gas may also be estimated by means of an acid solution of cadmium chloride.*

71 The gas is slowly passed through a solution of the above contained in a couple of Woulffe's bottles, and is then measured by means of an aspirator or meter. After passing a quantity of gas at the rate of about half a cubic foot per hour, the bottles are disconnected, and the precipitated cadmium sulphide is washed on to a filter; the contents of the filter are then placed in a beaker with distilled water, and bromine added in excess so as to oxidise the CdS to $CdSO_4$. The

bromine is driven off by heat, and the resulting solution filtered and washed, the filtrate ($CdSO_4$) is then treated with an excess of $BaCl_2$ which precipitates $BaSO_4$. This is washed, dried, ignited and weighed in the usual manner.

233 parts $BaSO_4$ correspond to 32 of sulphur, and 32 parts of sulphur are equal to 34 of sulphuretted hydrogen, from which the quantity per cubic foot can readily be calculated.

The method of determining the amount of ammonia in crude gas is described in (92).

## III. TESTING PURIFIED GAS FOR SULPHURETTED HYDROGEN, AMMONIA, AND SULPHUR COMPOUNDS ACCORDING TO THE INSTRUCTIONS OF THE GAS REFEREES, &c.*

72. The testings for the above impurities extend over twenty hours of each day, and are made on 10 cubic feet of gas, which is tested successively for each of the following:—

(1) *Sulphuretted Hydrogen.*—In testing for this impurity, the gas, as it leaves the service-pipe, passes through an apparatus in which are suspended slips of bibulous paper impregnated with basic acetate of lead. The test paper from which these slips are cut, is prepared by moistening sheets of bibulous (filter or blotting) paper with a solution of one part of sugar of lead in eight or nine parts of water, and holding each sheet, while still damp, over the surface of a strong solution of ammonia for a few moments. As the paper dries, all free ammonia disappears.

It is hardly necessary to state, that if there be any $SH_2$ in the gas, the slips of lead paper will be discoloured.

(2) *Ammonia.*—The gas, on leaving the above mentioned apparatus, passes through a glass cylinder filled with glass beads, which have previously been moistened with a measured quantity of standard sulphuric acid; any ammonia in the gas

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* The full instructions of the Gas Referees as to the method of testing for the above impurities will be found in the Appendix.

is absorbed by the acid, and from the amount of acid neutralised, the amount of ammonia in the gas is determined.

(3) *Sulphur Compounds.*—The gas which has passed through the sulphuretted hydrogen and ammonia testing apparatus, passes next through a meter, by means of which the rate of flow can be adjusted to half a cubic foot per hour, the said meter being provided with an automatic arrangement for shutting off the gas when 10 cubic feet have passed through the same.

From the meter, the gas passes to the sulphur test, where it is burned in air impregnated with the vapour of ammonia, the resulting water vapour is condensed and collected, and contained therein will be found the sulphuric acid and other condensable products resulting from the combustion.

73. The operation of testing the gas for ammonia and sulphur, is effected in two stages. The first or preliminary stage, consists in arresting the ammonia by means of acid, and burning the gas as described; the second stage consists in finding out how much acid has entered into combination with the ammonia in the gas, and the weight of barium sulphate obtained from the condensed products of combustion on adding a solution of barium chloride.

74. In estimating the quantity of ammonia in purified gas, two solutions are required, one containing one grain of ammonia in 100 measures of the solution, and the other the equivalent amount of sulphuric acid in 25 measures of the solution, which will exactly combine with one grain of ammonia, that is to say, 25 measures of the acid solution should exactly neutralise 100 of the alkaline solution. In order to prepare these solutions we shall require various glass vessels graduated on the septem system. The following is a list of the same:—A decigallon mixer divided into 100 parts; pipettes to deliver septems; two burettes on stands, one for use with the acid and the other with the alkaline solution, each burette being graduated into 100 parts (septems), each part being further subdivided into fifths. (A septem is equal

to the one ten-thousandth part of a gallon, or to 7 grains weight of distilled water at 62° F., consequently a gallon contains 10,000 septems, and a decigallon 1000 septems).

75. The first operation is to prepare the sulphuric acid solution of such a strength, that 25 measures (septems) shall be equal to 1 grain of ammonia. Sulphuric acid combines with ammonia, in the proportion of one molecule of the acid to two of ammonia, as in the following equation;—

$$H_2SO_4 + 2NH_3 = (NH_4)_2\,SO_4,$$

and, consequently, in the proportion by weight of 98 to 34, therefore, as the Referees' calculations are worked out in grains, 2·8824 grains of acid are required to neutralise 1 grain of ammonia. Now the specific gravity of the strongest sulphuric acid obtainable is about 1·845, consequently a septem of such acid would weigh 12·915 grains.

The decigallon mixer contains 1000 septems, and, as the equivalent of one grain of ammonia requires to be contained in 25 septems of solution, the total contents of the mixer should contain forty times the equivalent of 1 grain of ammonia, or 115·2960 grains of acid, and as one septem of acid weighs 12·915 grains, theoretically we should require 8·93 septems of acid in the decigallon. Sulphuric acid of the specific gravity 1·845 is not generally obtainable however, owing to its absorbing moisture so readily; it is better, therefore, to add an excess of acid, as it is easier to reduce, than to increase the strength of the solution. If we add 9·4 septems of acid it is probable that we shall obtain a solution of about the right strength. Therefore, having filled the mixer with distilled water until the centre of the meniscus of the surface of the water, coincides with the top, or hundredth line of the measure, 9·4 septems of acid are added from one of the two burettes previously mentioned, which should always be reserved for the use of the acid solution. The best way of dealing with the strong sulphuric acid, is to fill the burette with it by means of a small lipped beaker, and having poured out the 9·4 septems into the mixer, empty the burette, and immediately wash it out with water until it ceases to redden blue litmus-paper, and afterwards turn it mouth downwards to drain. After adding the acid, the stopper of the mixer should be inserted, and the vessel well shaken for several minutes. It should then be allowed to stand in a cool place for some time, so as to attain the normal temperature, and allow time for contraction. When the vessel and its contents are quite cool, 50 septems are measured out into a beaker by means of a pipette, diluted with distilled water, neutralised with ammonia, made acid with hydrochloric acid, raised to the boiling point, the sulphuric acid precipitated by barium chloride, and the precipitate filtered, washed, dried, ignited and weighed as in (24). The net weight of barium sulphate produced should be 13·8 grains, which is arrived at in the following manner:—

Ninety-eight parts by weight of sulphuric acid, give 233 parts of barium sulphate as under:

$$\underset{98}{H_2SO_4} + \underset{208}{BaCl_2} = \underset{73}{2HCl} + \underset{233}{BaSO_4};$$

and as 25 septems of the acid solution contain 2·8824 grains of sulphuric acid, 50 septems will contain 5·7648 grains, consequently, if we make the following proportion, we obtain the theoretical quantity of barium sulphate which the 50 septems should give:—

$$\text{As } 98 : 5{\cdot}7648 :: 233 : 13{\cdot}76,$$

which may be expressed in round numbers as 13·8. Probably the weight obtained will be somewhat greater than this, due to the excess of strong acid taken; and, according to the Referees' Instructions, if the weight exceeds 13·9 grains, or falls below 13·7 grains, more water or sulphuric acid must be added, until the weight falls within these limits.

76. In order to determine the quantity of water which must be added to the solution in order to reduce the strength, if too strong, divide the number of septems of test-acid

contained in the mixer by 13·8, and multiply the quotient, by the difference between 13·8 grains, and the number of grains of sulphate obtained. The following is an example, the excess obtained being purposely exaggerated. On adding 9·4 septems of strong acid to a decigallon of water, the volume of the mixture will be 1006·9 septems (the decreased volume being due to the fact that when sulphuric acid combines with water contraction ensues). On taking out the 50 septems for the determination of the amount of $BaSO_4$, there will remain 1006·9 − 50 = 956·9 septems, which, being divided by 13·8, gives a quotient of 69·34. Now assuming the excess of sulphate to be 2·0 grains, then 69·34 × 2 = 138·68, as the number of septems of water necessary to be added.

This may be more clearly explained as under. 50 septems of dilute acid, if of correct strength, should give 13·8 grains $BaSO_4$; but if the $BaSO_4$ weighs 15·8 grains as in the example, we have as much acid present in the 50 septems as should be contained in 57·25 septems.

$$\frac{15{\cdot}8 \times 50}{13{\cdot}8} = 57{\cdot}25,$$

and consequently in 100 septems, we have as much acid as should be contained in 114·5 septems, therefore in the whole contents of the mixer we have as much acid present, as should be contained in 1095·6 septems.

$$\frac{956{\cdot}9 \times 114{\cdot}5}{100} = 1095{\cdot}6,$$

consequently, if we subtract 956·9 from 1095·6 we get 138·7 as the number of septems of water necessary to be added, which confirms the result in the preceding example.

After adding the necessary quantity of water, and well shaking up the contents of the mixer, on operating on another 50 septems of dilute acid, we ought to get very near to the 13·8 grains required, but in case the solution is still too strong, the additional quantity of water necessary to be added may be found by the before mentioned rule, due notice being taken of the volume of the solution remaining in the mixer after the abstraction of the portion taken out, and the water added, thus:—the 956·9 septems + 138·7 septems of water added, will be equal to about 1095·6 septems, and, after abstracting 50 septems for a second determination, there will remain 1045·6 septems to be divided by 13·8, in order to obtain a quotient to be multiplied by the excess weight of sulphate obtained.

77. In case it is wished to prepare a larger quantity of the acid at one time, "measure a gallon of distilled water into a clean earthenware jar or other suitable vessel. Add to this 94 septems of pure concentrated sulphuric acid and mix thoroughly." This solution should of course give the same weight of $BaSO_4$ as the one just described, viz. 13·8 grains, but assuming that the weight is greater.

"Add now to the diluted acid a measured quantity of water which is to be found by subtracting 13·8 from the weight of $BaSO_4$ obtained in the experiment and multiplying the difference by 726. If these operations have been accurately performed, a second precipitation and weighing of the $BaSO_4$ obtainable from 50 septems of the test-acid, will give nearly the correct number of 13·8 grains."

The only item requiring explanation in the above, is that relating to multiplying by 726. A gallon being equal to 10,000 septems, on adding 94 septems of strong acid to that quantity of water, we obtain 10,069 septems (the decrease as in the previous case being due to contraction) and on taking out 50 septems from this, we have remaining 10,019 septems, which, divided by 13·8, will give as a quotient the number 726, as in the case when preparing a decigallon of the solution, on dividing 956·9 by 13·8 we obtained the quotient 69·34.

78. Having obtained the sulphuric acid solution of the correct strength we now proceed to prepare the ammonia solution of equivalent strength for use with the same. The

strongest solution of ammonia obtainable, has usually a specific gravity of about 0·88, and contains about 2 grains of absolute ammonia ($NH_3$) per septem, consequently on adding 5 septems to the decigallon, we ought to obtain a solution of nearly the correct strength. This quantity of strong ammonia is added to a decigallon of distilled water, the stopper of the mixer inserted, and the contents well shaken; trials are afterwards made, in the manner described below, to see if 100 septems of the ammonia solution just neutralise 25 septems of the standard acid; if not, more water or ammonia must be added, as the case may be, until 100 septems will so neutralise 25 septems of the acid solution.

Having placed the acid burette perfectly upright in its stand, charge it with the acid solution, and pour from it exactly 25 septems into a clean dry beaker; the contents of the beaker are then largely diluted with distilled water, and coloured with a few drops of methyl orange solution, which will turn the solution pink. The burette reserved for the alkaline solution is then filled with the dilute ammonia solution, and the beaker containing the acid, is placed beneath it, upon a white tile or clean filter-paper.

The stop-cock of the burette is then opened, and the ammonia solution allowed to gradually fall into the acid solution, which is kept constantly stirred by means of a thin glass rod with rounded ends. The colour of the acid solution will gradually change from pink, to a golden yellow or sherry colour, and when this change is effected by the last drop or two of the solution, we may assume that the latter is correct, otherwise the strength will have to be increased or diluted, by adding strong ammonia or distilled water, as the case may be, until the last drop does so change the colour.

79. Should it be considered necessary to make a larger quantity of the ammonia solution, "measure a gallon of distilled water into a clean glass or earthenware vessel and add to it 50 septems of strong ammonia (sp. gr. 0·88)." This should give a solution of nearly the correct strength,

which must be tested and adjusted, if necessary, in the manner just described.

80. Both the acid and ammonia solutions should be kept tightly stoppered and in a cool place: this particularly applies to the ammonia solution, which is very apt to become weak owing to the volatility of ammonia.

## *Ammonia Apparatus.*

81. The glass cylinder filled with glass beads in which the acid is placed, is shown in Fig. 44, immediately in front of the meter; it has a movable nose-piece at one end, and a glass stop-cock at the other. It is supported in a perfectly horizontal position upon a stand.

The meter employed indicates the hourly rate of consumption by the observation of one minute, and in addition registers up to 10 cubic feet, when the flow of gas is capable of being automatically shut off.

## *The Sulphur Test Apparatus.*

82. Connected to the outlet of the meter is the sulphur test, Fig. 44, which, in the Referees' form of apparatus, consists of a small Bunsen burner with a steatite top, which is mounted on a short cylindrical stand A, perforated with holes for the admission of air, and having on its upper surface a deep circular channel to receive the wide end of a glass trumpet-shaped tube B. On the top of the stand, between the narrow stem of the burner and the surrounding trumpet tube B, pieces of commercial sesqui-carbonate of ammonia, weighing in all about two ounces, are placed. The products of the combustion of the gas, and of the gradual volatilisation of the sesqui-carbonate of ammonia, go upwards through the trumpet tube, into a vertical glass vessel C, which is filled with glass marbles, in order to break up the current of the ascending gases and promote condensation. From the top of the cylinder a long glass chimney-pipe D

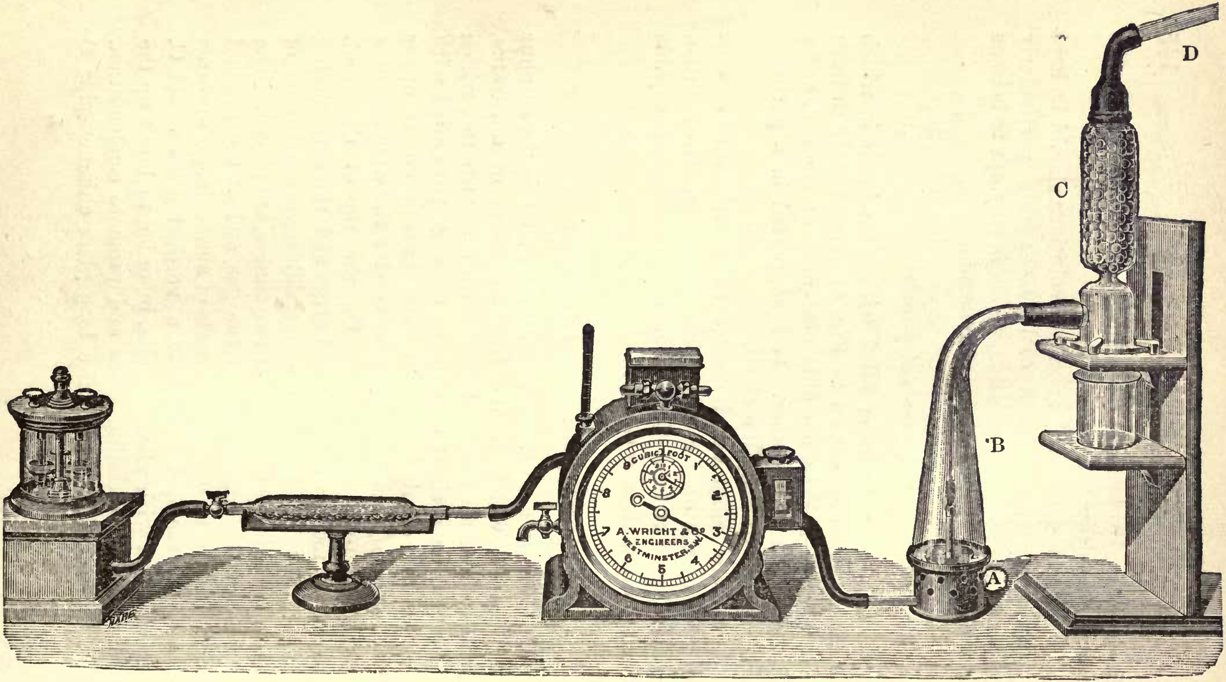
D
C
B
A
CUBIC FOOT
1
2
3
4
5
6
7
8
9
A. WRIGHT & Co
ENGINEERS
WESTMINSTER, S.W.

FIG. 44.

proceeds; this serves to effect some further condensation, as well as to regulate the draught, and affords a means of exit for the uncondensable gases. In the bottom of the cylinder a small glass tube is fixed; through this, the liquid formed during the testing, drops into a beaker placed underneath. The Gas Referees give various precautions to be observed in selecting and setting up the apparatus, which will be found in the Appendix, but in addition to those precautions, it is necessary to be careful not to place the apparatus where it is exposed to very low temperatures, or the results obtained will be inaccurate, owing to the fact that there will not be sufficient heat to thoroughly volatilise the sesqui-carbonate of ammonia. In the event of there being no chance of avoiding placing the apparatus in such a situation, then it will be necessary to enclose the trumpet tube in a casing. This may be made of tin, care being taken not to interfere with the air supply, otherwise there will be imperfect combustion.

### *Fitting up of the Apparatus.*

83. The whole train of apparatus is shown in Fig. 44.

The bench or table upon which the instruments are placed must be level and firm, and not be liable to vibrations.

The sulphuretted hydrogen test is fixed upon a stand at one end of the bench, a metal gas service pipe is connected to its inlet, and its outlet is connected by a short piece of red or black, but not grey, india-rubber tube to the ammonia cylinder. The whole train is then connected up as shown, by short pieces of the same kind of india-rubber tube.

84. In order to make a test we proceed as follows:—Suspend slips of lead paper in the sulphuretted hydrogen test. Remove the glass cylinder from its stand, taking out the ground glass nose-piece and closing the glass stop-cock. Remove also the glass chimney tube from above the sulphur test burner. Charge the ammonia cylinder from a burette or pipette, with 50 septems of the test acid, which is greatly in

excess of any quantity of ammonia likely to be found in the gas, unless the gas contains more than about 6 grains of ammonia in 100 cubic feet. In such case, it will be necessary to employ two cylinders in line. The whole interior surface of the cylinder should be wetted with the acid, as well as the glass beads; this is effected by holding the cylinder in a perfectly horizontal position, and gently rotating it for a minute or so. The glass cylinder is then replaced on its stand, and reconnected with the gas supply and meter. Place about 2 ounces of fresh sesqui-carbonate of ammonia around the sulphur test burner. The index pointer of the meter is then brought to zero, the gas supply turned on, the sulphur test burner lit, the trumpet tube replaced, and connected with the vertical glass cylinder or condenser, the chimney tube being afterwards placed on top of same. Before leaving the apparatus, see that it is burning at the proper rate of half a cubic foot per hour.

### *Determination of the Ammonia.*

85. At the end of each period of testing, the glass stopcock of the ammonia cylinder is closed, the nose-piece removed, and the cylinder fixed in a vertical position in a suitable support; a clean beaker is then placed beneath, and the contents of the cylinder allowed to drain into the beaker, taking care to avoid any loss by spurting. The cylinder is then well washed out with distilled water, the washings being collected in the beaker containing the acid drained from the cylinder; the washing should be continued until the liquid ceases to turn blue litmus paper red. The contents of the beaker are then well stirred round by means of a thin glass rod, half the contents (accurately measured) being put aside for analysis, and the remaining half transferred to a well-stoppered bottle, for future reference if required.

The burette used for the alkaline solution is then filled with the standard ammonia solution, and the liquid to be tested, having been diluted with distilled water and coloured with a few drops of methyl-orange solution, is placed on a white surface beneath the burette. The alkaline solution is now added, by small portions at a time, until the colour changes, showing that the acid is neutralised, when it will be found (unless there is no ammonia whatever in the gas) that the acid solution will require less than the 100 septems of alkali which it originally needed in order to neutralise it, the difference, being due to the quantity of ammonia in the gas, which has been absorbed by the acid.

The quantity of standard alkali remaining in the burette after the neutralisation of the acid is noted, and from this, the quantity of ammonia in the gas is found as per the Referees' Instructions (which, however, apply to 10 cubic feet of gas only).

86. " To find the number of grains of ammonia in 100 cubic feet of the gas, multiply by two the number of septems of test alkali remaining in the burette, and move the decimal point one place to the left."

As previously stated, the rule for finding the amount of ammonia, given above, is only applicable when 10 cubic feet of gas have passed through the cylinder, but, by means of the following method, the amount may be calculated on any quantity of gas. In order to understand the method it is necessary to note, that the burette containing the alkaline solution is divided into 100 parts, and the whole is equal to one grain of ammonia, consequently each division is equal to one-hundredth of a grain of ammonia. Therefore if we note the number of divisions remaining after the acid has been neutralised, we obtain the number of hundredths of a grain of ammonia corresponding to half the acid employed, and on multiplying this result by two (because the acid was halved) we obtain the number of hundredths of a grain of ammonia in the quantity of gas operated on, and from which the quantity per 100 cubic feet can easily be calculated. As an example, suppose after filling the cylinder with 50 septems of test acid, and passing 12·6 cubic feet of gas through the same, we found that it took 86 divisions of the ammonia solution to neutralise 25 (or 50 ÷ 2) divisions of the acid solution in place of 100 as it originally did, then $100 - 86 = 14$ divisions remain, and as each division is equal to one-hundredth of a grain of ammonia, 14 divisions are equal to 14 hundredths of a grain, then $0{\cdot}14 \times 2 = 0{\cdot}28$ grain of ammonia in the gas which has passed through the cylinder, and $\frac{0{\cdot}28 \times 100}{12{\cdot}6} = 2{\cdot}22$ grains per 100 cubic feet.

### *Determination of the amount of Sulphur.*

87. The following is the method of analysing the liquor produced in the sulphur test apparatus, so as to obtain the amount of sulphur. At the expiration of the test, the contents of the beaker containing the products of combustion, are placed in a graduated glass measure, and the trumpet tube, glass cylinder, and chimney tube are washed well out with distilled water, and the washings added to the liquid in the measure. This is thoroughly well mixed, and half the quantity poured into a beaker for analysis, the remainder being placed in a well-stoppered bottle for future reference if required. The contents of the beaker are then acidified with hydrochloric acid (preferably charged with free bromine to secure complete oxidation of the products in the condensed liquid). The beaker is now covered with a clock-glass, its contents raised to the boiling point, and an excess of a saturated solution of barium chloride added, the precipitated $BaSO_4$ is allowed to settle, and then filtered, washed, dried, ignited and weighed as in (24); the weight obtained multiplied by 11 and divided by 4 = the grains of sulphur per 100 cubic feet of gas.

88. This rule is based on the following:—233 parts by weight of $BaSO_4$ correspond to 32 parts of sulphur, and as grain weights are employed, 233 grains of $BaSO_4$, correspond

to 32 grains of sulphur, consequently one grain of $BaSO_4$ corresponds to ·13734 of sulphur, then, as half the liquor only was used, and exactly 10 feet of gas is assumed to have been burnt, $\cdot 13734 \times 2 \times 10 = 2\cdot 7468$, which is equivalent to multiplying by 11 and dividing by 4, $\frac{11}{4} = 2\cdot 75$.

As previously stated, the Referees' rule for calculating the amount of sulphur per 100 cubic feet, only applies to the products resulting from the combustion of exactly 5 cubic feet of gas (half the quantity obtained in the combustion of 10 cubic feet), consequently, if any other quantity of liquid than that due to 5 cubic feet of gas be operated on, the weight of sulphur in that quantity of gas will be found by multiplying the weight of sulphate obtained by ·13734, and then finding by proportion the number of grains per 100 cubic feet.

It may be of interest to state, that if the gas burnt amounts to 2·12 cubic feet, or if the portion of the condensed liquid taken, corresponds to 2·12 cubic feet, then the $BaSO_4$ weighed in centigrams, gives at once the grains of sulphur per 100 cubic feet without any calculation.

89. It is necessary in every experiment to note the temperature and pressure, and if necessary to make a correction for the same. When working to the Referees' Instructions on 10 cubic feet of gas, the number of grains of sulphur obtained as described above "is to be corrected for the variations of the temperature and atmospheric pressure," and "the readings of the barometer and thermometer are to be taken for the day on which the testing commenced, and also the day on which it closed, and the mean of the two is to be used.

"This correction may be made most simply, and with sufficient accuracy in the following manner:—

"When the Tabular Number (Appendix) is between 955–965, 966–975, 976–985, 986–995, increase the number of grains of sulphur by $\frac{4}{100}$ths, $\frac{3}{100}$ths, $\frac{2}{100}$ths, $\frac{1}{100}$th of the weight found.

"When the Tabular Number is between 996–1005 no correction need be made.

"When the Tabular Number is between 1006–1015, 1016–1025, 1026–1035, diminish the number of grains of sulphur by $\frac{1}{100}$th, $\frac{2}{100}$ths, $\frac{3}{100}$ths."

The following is an example:—

| | Grains. |
|---|---|
| Grains of $BaSO_4$ from half the liquid produced in the combustion of 10 cubic feet of gas | 5·6 |
| Multiplied by 11 and divided by 4 = grains of sulphur in 100 cubic feet of gas (uncorrected) or | 15·4 |
| Barometer mean (30·2)″ | |
| Thermometer „ 76° | |
| Tabular number 964 | |
| Add $\frac{4}{100}$ths of 15·4 grains = .. .. .. .. .. | ·61 |
| Grains of sulphur in 100 cubic feet of gas (corrected) | 16·01 |

When operating on quantities of gas other than 10 cubic feet, the gas is corrected by multiplying direct by the tabular number, and then making a proportion for the amount of sulphur per 100 cubic feet.

90. The Referees' sulphur test depends upon the following chemical reactions:—Among other substances, coal gas contains carbon, hydrogen, and sulphur, which, in the process of burning, combine with the oxygen of the air to form water, carbonic acid, and sulphurous acid ($S + O_2 = SO_2$), but this latter is speedily oxidised, more or less completely into sulphuric acid ($SO_3$). Ammonia being present, the acids unite with it, forming ammonium carbonate and sulphate, which are carried down in solution, by the condensed water resulting from the combustion.

Barium chloride combines with both ammonium carbonate and ammonium sulphate, the acid radicals of those salts uniting with the barium of the $BaCl_2$, to form barium carbonate and barium sulphate, while the chlorine of the $BaCl_2$ unites with the ammonium ($NH_4$) of the ammonium carbonate and sulphate, to form soluble ammonium chloride.

From the foregoing it will be seen that if to the solution, as obtained from the sulphur test, we were at once to add a solution of $BaCl_2$, we should get a precipitate of both barium carbonate and barium sulphate, so, in order to prevent this, the solution is acidified with HCl and boiled, which has the effect of driving off all the carbonic acid, and completely unites the $NH_4$, which at first was in solution as a carbonate with the hydrogen acid, so as to form the other soluble salt $NH_4Cl$. Then on adding the $BaCl_2$, we obtain the insoluble $BaSO_4$.

91. *If desired, the amount of sulphur in the gas can be arrived at by means of the liquor produced from the combustion of the gas in the Referees' apparatus, without going through the processes of precipitation and filtering, in the following manner :—*

The liquid obtained from the combustion in the Referees' sulphur test of a specified quantity of gas, is evaporated in a small porcelain basin whose weight has been accurately determined, and when the contents are nearly dry, the whole is transferred to an air-oven, and the complete drying effected at a temperature of about 240° F. The basin and its contents are then allowed to cool, and subsequently weighed on the balance ; after noting the weight, the basin is replaced in the air-oven for a few minutes, and then cooled and re-weighed, in order to make sure that all adhering moisture has been expelled : this operation should be repeated until the weight is constant. The increase in weight over the weight of the empty basin is due to sulphate of ammonia, with an infinitesimal quantity of tarry matter, and from this, the amount of sulphur may be obtained as under. Supposing that 24·6 grains have been obtained,

| | Equivalent weight of $(NH_4)_2SO_4$ | | Equivalent weight of Sulphur. | | | | |
|---|---|---|---|---|---|---|---|
| As | 132 | : | 32 | :: | 24·6 | : | $x$. |

$$x = 5·97 \text{ grains.}$$

### *Estimation of Ammonia in Crude Gas.*

92. The amount of ammonia in crude gas is estimated in exactly the same manner as the amount in purified gas, but by means of a stronger acid solution, as crude gas generally contains from 200 to 300 grains per 100 cubic feet.

A convenient strength of acid is 10 times that of the Referees' solution, and it is made by adding 10 times the quantity of acid which is employed in making the Referees' solution previously described, to the decigallon of distilled water; we should then obtain a solution of 10 times the strength. It must, however, be tested in the same manner as the Referees' solution was tested, but in place of estimating the amount of $BaSO_4$ in 50 septems, it is better to take, say, 10 septems, and multiply the result obtained by 5, which should equal 10 times 13·8 grains. The reason for taking 10 septems is that the 50 septems would give such a heavy precipitate as to make it unwieldy to manipulate. The ordinary ammonia solution, 100 septems of which is equal to one grain of ammonia, will serve for titrating.

93. The following is the method of making an experiment:—An ammonia cylinder is charged with 50 septems of the acid solution, and 5 feet of gas passed through, any tar that may be in the gas, being kept back by cotton wool placed in a calcium chloride tube. The cylinder and contents are washed out until free from acid, and the washings, after being well mixed, are placed in a graduated measure, which may conveniently be 20 ounces. If the washings do not amount to that volume, they must be diluted with distilled water to that extent. A twentieth part, or 1 ounce of the solution, after being largely diluted with distilled water, is now titrated with the ammonia solution as previously described, and, after neutralisation, the alkali remaining in the burette noted. Supposing this to be 60 septems, then, as each septem represents 0·01 grain of $NH_3$, the 60 septems would represent 0·60 grains of ammonia;

but as only a twentieth part of the liquor received was taken for analysis, this result must be multiplied by 20, which would give 12 grains of ammonia in the 5 cubic feet of gas, and therefore $\frac{12 \times 100}{5} = 240$ grains in 100 cubic feet.

### *Harcourt's Colour Test.*

94. The Referees' method for the estimation of sulphur compounds, although giving accurate results, is not adapted where rapidity is an essential, as, say, when it is required to know the condition of the sulphide vessels for the elimination of the sulphur compounds at any particular time; in this case the colour test devised by Mr. Vernon Harcourt, F.R.S., will be found more suitable.

The test is based on the fact that when coal gas containing bisulphide of carbon is passed over heated platinised pumice, the bisulphide is more or less decomposed into sulphuretted hydrogen and methane, and the sulphuretted hydrogen being led into a solution of a lead salt, the intensity of colour imparted to the lead solution from a given quantity of gas, as compared with a standard tint corresponding to a definite quantity of lead sulphide, at once enables us to determine the amount of $CS_2$ in the gas under examination. The general arrangement of the test is shown in Fig. 45.

95. The glass bulb, which is filled with platinised pumice, is to be so adjusted that it may be about an inch above the burner, and in the middle of the cylinder. In order to use the apparatus, first turn on the upper stop-cock, thus sending gas through the bulb at the rate of about half a cubic foot per hour, as may be judged by lighting the gas for an instant at the end of the horizontal arm, when a flame about an inch long should be produced. Raise the cylinder, which will be supported by the pressure of the wires, light the burner, and turn down the flame until it forms

a blue non-luminous ring. Lower the cylinder, and place the small fire-clay pieces upon it round the neck of the bulb.

96. A testing may be made five minutes after the burner is lit, except when the apparatus is used for the first time, when the gas should be allowed to flow through the bulb for a quarter of an hour or a little longer, and any number of testings may be made consecutively, so long as the heat is maintained. The manner of conducting a test is as follows:—

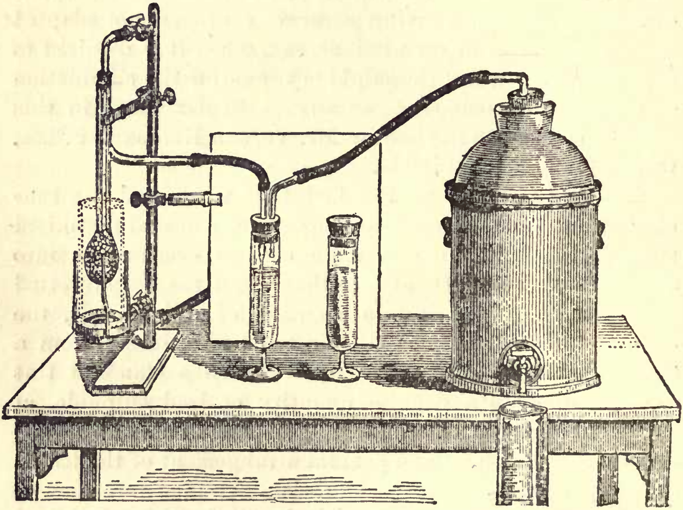

Fig. 45.

Lay a piece of white paper on the table by the side of the burner, and fix a piece of cardboard upright in the brass clip provided for that purpose—the cardboard serves as a background against which to observe the colour of the contents of the test glasses, and should receive a side light, and be as clear as possible from shadows. Place the "standard" glass, "day light" or "gas light" according to circumstances, in its receptacle, and dilute some of the concentrated lead syrup with about twenty times its volume of distilled water, and fill one of the test glasses provided, up to the mark scratched on same, with a portion of the liquid thus prepared.

Insert the india-rubber plug with capillary and elbow tubes, and connect up, as in Fig. 45, with the bulb and aspirator, placing the standard glass and test glass side by side. The capillary tube should descend very nearly to the bottom of the glass, but must not press on the bottom, or it will probably be broken. At starting, the aspirator should be full of water and the measuring cylinder empty. Turn the tap of the aspirator gradually, a stream of bubbles will rise through the lead solution. Turn off the tap for a minute, and observe the liquid at the bottom of the capillary tube. If it gradually rises, the india-rubber connections are not tight, and must be made so before proceeding further. It is necessary to avoid pressing the plugs into the test glass, or the aspirator, while they are connected, which would drive up the lead solution into the inlet tube.

97. When the connections are air-tight, allow the water to run into the measuring cylinder in a slender stream until the lead solution has become as dark as the standard. As the ascending bubbles interfere with the observation of the tint, it is best to turn off the tap when the colour seems almost deep enough; compare the two, turn on the tap if necessary, for a few moments; then compare again, and so on until the colour of the two liquids is the same. The volume of water which the measuring cylinder now contains, is equal to the volume of gas which has passed through the lead solution. This volume of gas contained a quantity of sulphur as carbon bisulphide, which, as lead sulphide, has coloured the liquid in the test glass to the same depth as the standard tint. The standard has been made such, that to impart this tint to the solution of liquid contained in the glass, 0·0187 grains of lead sulphide must be present, containing 0·0025 grains of sulphur. Hence, supposing the

measuring cylinder, each division of which corresponds to $\frac{1}{2000}$th of a cubic foot, to have been filled to the thirtieth division, $\frac{30}{2000}$ths of a cubic foot of gas contained 0·0025 grains of sulphur. From these figures the number of grains of sulphur existing as bisulphide of carbon in 100 cubic feet of gas can easily be calculated.

98. The following table gives the relation between (V) the divisions of the measuring cylinder filled with water, and (S) the grains of sulphur existing as bisulphide of carbon in 100 cubic feet of gas. Since gas contains, besides carbon bisulphide, some other sulphur compounds, which are not decomposed by the action of heat into sulphuretted hydrogen, and which contain sulphur amounting on an average to seven or eight grains per 100 cubic feet of gas, it is necessary to add this quantity to that found by the colour test if it is wished to know approximately the total amount of sulphur in the gas.

$$S = \frac{500}{V}$$

| V. | S. | V. | S. | V. | S. | V. | S. | V. | S. |
|---|---|---|---|---|---|---|---|---|---|
| 10 | 50·0 | 29 | 17·2 | 48 | 10·4 | 67 | 7·5 | 86 | 5·8 |
| 11 | 45·4 | 30 | 16·7 | 49 | 10·2 | 68 | 7·4 | 87 | 5·7 |
| 12 | 41·7 | 31 | 16·1 | 50 | 10·0 | 69 | 7·2 | 88 | 5·7 |
| 13 | 38·5 | 32 | 15·6 | 51 | 9·8 | 70 | 7·1 | 89 | 5·6 |
| 14 | 35·7 | 33 | 15·1 | 52 | 9·6 | 71 | 7·0 | 90 | 5·6 |
| 15 | 33·3 | 34 | 14·7 | 53 | 9·4 | 72 | 6·9 | 91 | 5·5 |
| 16 | 31·3 | 35 | 14·3 | 54 | 9·2 | 73 | 6·9 | 92 | 5·4 |
| 17 | 29·4 | 36 | 13·9 | 55 | 9·1 | 74 | 6·8 | 93 | 5·4 |
| 18 | 27·8 | 37 | 13·5 | 56 | 8·9 | 75 | 6·7 | 94 | 5·3 |
| 19 | 26·3 | 38 | 13·2 | 57 | 8·8 | 76 | 6·6 | 95 | 5·3 |
| 20 | 25·0 | 39 | 12·8 | 58 | 8·6 | 77 | 6·5 | 96 | 5·2 |
| 21 | 23·8 | 40 | 12·5 | 59 | 8·5 | 78 | 6·4 | 97 | 5·2 |
| 22 | 22·7 | 41 | 12·2 | 60 | 8·3 | 79 | 6·3 | 98 | 5·1 |
| 23 | 21·7 | 42 | 11·9 | 61 | 8·2 | 80 | 6·2 | 99 | 5·1 |
| 24 | 20·8 | 43 | 11·6 | 62 | 8·1 | 81 | 6·2 | 100 | 5·0 |
| 25 | 20·0 | 44 | 11·4 | 63 | 7·9 | 82 | 6·1 | 150 | 3·3 |
| 26 | 19·2 | 45 | 11·1 | 64 | 7·8 | 83 | 6·0 | | |
| 27 | 18·5 | 46 | 10·9 | 65 | 7·7 | 84 | 6·0 | | |
| 28 | 17·9 | 47 | 10·6 | 66 | 7·6 | 85 | 5·9 | | |

99. For another experiment, the test glass is to be disconnected and recharged; the water in the measuring cylinder being poured back into the aspirator. The colour of the standard is unaffected by exposure to light, but deepens if the liquid is warmed, resuming its original shade as the liquid cools. If, therefore, the glass containing the standard has been kept in a warm place, it should be allowed to cool before testing.

The lead solution used in the experiments becomes colourless after being exposed to the light for a few hours, and may therefore be used over and over again for twenty times or more, provided that it does not absorb carbonic acid from the air. The best mode of working is to have two well-corked bottles, into one of which the coloured liquid is emptied, while the test glass is recharged from the other.

It is hardly necessary to remark, that in the event of making an experiment for $CS_2$ on crude gas containing $SH_2$, the latter impurity should previously be removed by passing it through oxide of iron.

## IV. Analysis of Ammoniacal Liquor.

100. Gas liquor may be said to consist of a solution of the following ammonium salts.

*Volatile Salts :—*
Ammonium carbonate (mono- sesqui- bi-).
„ sulphide $(NH_4)_2S$.
„ hydrosulphide $NH_4HS$.
„ cyanide.
„ acetate.

*Fixed Salts :—*
Ammonium sulphate.
„ sulphite.
„ thiosulphate (hyposulphite).
„ thiocarbonate.
„ chloride.
„ sulphocyanide (thiocyanate).
„ ferrocyanide.

Also the salts of organic bases, especially those of the pyridine series, phenols, and other matters of a tarry character.

The term "free" as employed here, is not to be taken in its ordinary sense, but rather that the compounds of ammonia with such acids as carbonic and sulphydric, can be removed from their solutions by simply boiling them for a sufficiently long period, or by acting on them with dilute acid in the cold: the ammoniacal salts called "fixed" are not removed from their solutions by boiling, or by treatment with acid, so it is necessary to heat such solutions in the presence of a caustic alkali or lime, which sets the ammonia free from its combination with the acids.

Free ammonia in the ordinary sense of the word means a solution of caustic ammonia.

101. The chief and most often recurring of the volatile salts are the sulphide and the carbonate; of the fixed salts the chloride and the sulphocyanate.

The percentage of the total ammonia combined as fixed salt varies greatly in different samples of liquor.

The occurrence of these different ammonium salts and the proportion in which the ammonia is combined with each acid depend on (1) the description of coal distilled, (2) the temperature at which it is distilled, (3) the temperature to which the liquor has been subjected, and whether it has been exposed to the air or not, and (4) the point in the condensing or scrubbing plant from which the sample is taken, and the general treatment to which the gas is subjected during the operation of washing and scrubbing. For example if a coal is distilled which contains an appreciable quantity of sodium chloride (common salt), hydrochloric acid is given off, and we therefore find a large percentage of fixed ammonium chloride in the liquor. On the high or low heats of the retorts depends in a great measure the larger or smaller percentage of fixed ammonium sulphocyanate in the liquor. Exposing liquor to a high temperature, or to the air at ordinary temperatures, causes it to lose ammonia, thereby converting its ammonium sulphide $(NH_4)_2S$ as well as ammonium monocarbonate, $(NH_4)_2CO_3$ respectively, into ammonium hydrosulphide $NH_4HS$, and ammonium bicarbonate $NH_4HCO_3$. It also has the effect of oxidising its ammonium sulphide to ammonium thiosulphate, and in some very slight degree to ammonium sulphate.

102. *The value of ammoniacal liquor is commercially expressed by the number of ounces by weight of mono-hydrated sulphuric acid*, $H_2SO_4$, *which is required to saturate one gallon. For instance, if a liquor is said to be of eight-ounce strength, it means that a gallon of the liquor would require eight ounces of pure, strong, sulphuric acid to neutralise it.*

The crudest method by which the strength of liquor is determined, is based on the assumption that the quantity of acid required to saturate the liquor, bears a certain relation to the specific gravity of the latter. In this method a No. 1 Twaddell's hydrometer is employed, having a scale divided and numbered from zero at the top to 25 at the lower part of its stem. A glass jar of a suitable size having been filled with liquor, which should be at the temperature of 60° F., the hydrometer is immersed, and the division on the scale to which it sinks in the liquor noted. Each degree upon the scale is supposed to indicate the presence of as much $NH_3$ in one gallon of the liquor as will require two ounces by weight of pure strong sulphuric acid to neutralise it. For instance, if the hydrometer sinks to the division 5 on the scale, the liquor is said to be of ten-ounce strength.

This method of determining the strength of ammoniacal liquor is not at all an accurate one, and is only suitable for general guidance in the working of the scrubbing or washing plant in gasworks; it should never be used when the actual amount of ammonia in the liquor is required, for, supposing a liquor to contain an excess of caustic ammonia, its strength would be under-estimated, owing to ammonia being lighter than water, and thus reducing the specific gravity of the liquor; on the other hand, if a liquor had been repeatedly passed through the scrubbers, and thus become charged with an excess of carbonic acid, it would probably be over-estimated, as $CO_2$ would raise the specific gravity.

103. Another method of estimating the value of liquor is to find by an experiment the number of ounces of pure strong sulphuric acid which are required to neutralise the ammonia in a gallon of liquor, but as it would not be practicable to operate on so large a quantity as one gallon, or to employ strong sulphuric acid, the following modification is employed:—

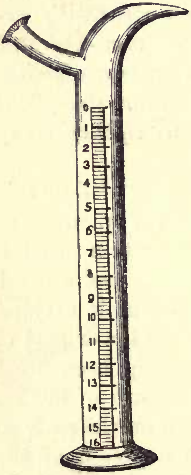

FIG. 46.

A graduated glass vessel called an alkalimeter, Fig. 46, is provided, and this is graduated into 16 parts.

A solution of sulphuric acid is made by adding one pound of pure sulphuric acid (sp. gr. 1845) to distilled water, and making up the solution to one gallon at a temperature of 60° F. The specific gravity of this solution should be 1064·4. The testing is conducted as follows:—

The alkalimeter is first rinsed out with a portion of liquor, and is afterwards charged with liquor to the top of the scale; this is then poured carefully out into a beaker or porcelain basin. The instrument is then well rinsed out with distilled water, and the rinsings added to the liquor in the basin. In order to guard against loss whilst pouring, it is advisable to grease the edge of the beak of the alkalimeter. A small quantity of the test acid is now poured into the alkalimeter and the instrument well rinsed out with the same; this is then poured away; the alkalimeter is next charged with the acid to the top of the scale. Having largely diluted the contents of the beaker with distilled water, and coloured the solution with a few drops of methyl orange indicator, which will give it a yellow tint, the contents of the alkalimeter are carefully poured into the liquor.

This operation should be performed very gradually and by small portions at a time, the contents of the beaker being well stirred between each addition of the acid, so as to allow of the disengagement of the carbonic acid and sulphuretted hydrogen in the liquor. When all the liquor has been neutralised, the solution will change to a pinkish tint, and on reading off the number of divisions of acid required for neutralisation, the ounce strength is obtained. Supposing it required nine divisions, then the liquor would be of nine-ounce strength. By this method the same results are obtained as if we had added strong acid to a gallon of liquor, for the alkalimeter being divided into sixteen parts, each part represents such a proportion of acid solution as would contain one ounce of the strong acid, while its total contents represent one gallon. It is first used to measure the liquor, and afterwards to measure the acid solution, and under these conditions it does not signify, within certain limits, what its true capacity may be.

This method of determining the strength of ammoniacal liquor is also unreliable, as it does not take any cognisance of the fixed ammonia. The only reliable method of obtaining the value of a sample of liquor, is to heat it with an excess of potash or soda, which will liberate the whole of the ammonia from its combination with acids; the evolved ammonia is led into a solution of sulphuric acid, which is afterwards titrated with an alkaline solution of equal strength, and from the amount of acid neutralised by the ammonia, the amount of the latter is determined as in (59).

104. The solution of sulphuric acid is of the same strength as that already mentioned, and is commonly known as ten per cent. acid. It is prepared by pouring about three pints of distilled water into a half-gallon graduated measure, and adding to this, half a pound (4·34 ounces by measure) of pure

concentrated sulphuric acid (sp. gr. 1845). The solution is well mixed by stirring with a glass rod, the vessel is covered, and the mixture allowed to cool down to the temperature of 60° F. When this temperature is attained, the additional quantity of water necessary to bring the volume to exactly half a gallon is added, and the solution again well mixed. Its specific gravity may now be tested by means of a delicate hydrometer, and if more than half a degree above or below 1064·4, it will be necessary to add small quantities of water or of acid, until the correct specific gravity is closely approached.

The following table, which is due to Mr. L. T. Wright, will be found useful when there is a difficulty in obtaining the temperature of 60° F.; it shows what the specific gravity of the acid should be at different temperatures below and above 60° F., in order to correspond to the specific gravity 1064·4 at 60° F.

| Temp. F. | Specific Gravity. | Temp. F. | Specific Gravity. | Temp. F. | Specific Gravity. | Temp. F. | Specific Gravity. |
|---|---|---|---|---|---|---|---|
| 35 | 1068·85 | 54 | 1065·64 | 61 | 1064·19 | 68 | 1062·72 |
| 40 | 1068·10 | 55 | 1065·45 | 62 | 1063·98 | 69 | 1062·51 |
| 45 | 1067·30 | 56 | 1065·24 | 63 | 1063·77 | 70 | 1062·30 |
| 50 | 1066·40 | 57 | 1065·03 | 64 | 1063·56 | 75 | 1061·20 |
| 51 | 1066·21 | 58 | 1064·82 | 65 | 1063·35 | 80 | 1060·05 |
| 52 | 1066·02 | 59 | 1064·61 | 66 | 1063·14 | 85 | 1058·95 |
| 53 | 1065·83 | 60 | 1064·40 | 67 | 1062·93 | 90 | 1057·80 |

105. The strength of the solution may also be checked gravimetrically or volumetrically.

In order to check it by the gravimetric method, exactly one ounce by measure of the solution is poured into a beaker, and having been largely diluted with distilled water, neutralised by ammonia, and afterwards rendered acid by hydrochloric acid, it is raised to the boiling point, and an excess of solution of barium chloride added. The precipitate of $BaSO_4$ obtained, is treated in the usual manner, and should weigh, after deducting the filter ash, 104·017 grains, which is arrived at as under.

Half a gallon = 80 fluid ounces, and ½ lb. of acid (3500 grains troy) are contained in ½ gallon of the solution, consequently each ounce by measure of the acid solution contains 43·75 grains of sulphuric acid, consequently $\frac{43{\cdot}75 \times 233}{98} = 104{\cdot}017$ grains of $BaSO_4$, which is equal to 43·75 grains of sulphuric acid. If the weight of $BaSO_4$ obtained differs from 104·017 grains, the solution must be made stronger or weaker, as the case may be, until the correct weight is obtained.

106. The solution may be checked volumetrically by neutralising chemically pure $Na_2CO_3$, using methyl orange as indicator. It is essential that the $Na_2CO_3$ be pure; this is ascertained by dissolving about 80 grains of it in distilled water, when a clear colourless solution should be obtained, and testing for the presence of sulphates or chlorides. To do this, add an excess of $HNO_3$ in order to expel all $CO_2$, and to one half of the acidified solution add a few drops of pure $BaCl_2$ solution, and to the other half a few drops of $AgNO_3$ solution. The solutions should remain clear in both cases. Should there be any difficulty in procuring pure $Na_2CO_3$ it may be obtained in the following way:—Shake up a quantity of the best commercial sodium bicarbonate ($NaHCO_3$) with successive small quantities of distilled water, until the washings when tested for Cl or $H_2SO_4$ in the manner described above, give negative results. The $NaHCO_3$ so treated, is then dissolved in boiling water, the solution filtered, evaporated over a Bunsen burner almost to dryness, stirring constantly as the mass becomes pasty, and finally to complete dryness on the water-bath. The salt is then roughly powdered and preserved in a stoppered bottle.

To standardise the acid solution, take about 200 grains of the $NaHCO_3$ and heat it carefully in a platinum crucible over a small Bunsen flame for about 20 minutes, stirring constantly so as not to fuse the $Na_2CO_3$, until all moisture is expelled; place the crucible in the desiccator to cool, and when cold, weigh out 47·32 grains on a watch-glass, this being the quantity which will be neutralised by 1 fluid ounce of the standard acid if it is correctly made up.

The weight 47·32 is thus obtained: 106 parts by weight of $Na_2CO_3$, are neutralised by 98 parts of $H_2SO_4$, and as 1 fluid ounce of the solution contains, as previously stated, 43·75 grains of $H_2SO_4$, this would require 47·32 grains of $Na_2CO_3$ for neutralisation: $\frac{43{\cdot}75 \times 106}{98} = 47{\cdot}32$.

The 47·32 grains of $Na_2CO_3$ are washed off the watch-glass, by means of distilled water from the wash bottle, into a deep white porcelain dish, a few drops of methyl orange solution added, and the standard acid run in from a graduated burette, the solution being constantly stirred while this is effected, until a drop of the acid suddenly changes the colour of the solution from a golden yellow to a reddish-brown tint. A second weighed quantity of $Na_2CO_3$ is then treated in the same way, in order to check the result obtained with the first quantity weighed out. Should the acid solution prove either too weak or too strong, the calculated quantity of water must be added, and the strength of the solution again tested with $Na_2CO_3$.

When the acid solution has been obtained of the correct strength we may compare it with the alkaline (soda) solution, which should exactly match it.

107. The latter solution is made by dissolving "stick" caustic soda, "not by alcohol," in water, filtering the solution through asbestos, and diluting until it has a specific gravity of about 1086 at 62° F.; this will about match the acid solution; in order to ascertain whether it does so, a burette having a capacity of 2 fluid ounces, and divided into 32 parts is provided; this is charged in the ordinary way with

2 fluid ounces of the caustic soda solution. Exactly 1 fluid ounce of the acid solution is then measured out by means of a pipette into a beaker, diluted with distilled water, coloured with a few drops of tincture of cochineal solution, and placed on a white tile or filter paper, beneath the burette containing the soda solution. The latter solution is then slowly run in to the acid (which is kept constantly stirred) until the colour suddenly changes to purple. If more or less than 1 ounce of the soda solution be required to effect this change, small additional quantities of caustic soda or of water must be added to the remainder of the ½ gallon, and fresh experiments made until the correct strength is attained. The solution must then be immediately bottled, tightly corked, and kept in a cool place.

108. The apparatus employed is the same as was described in the analysis of ferrous ammonium sulphate, and is shown in Fig. 33.

Having connected the whole apparatus together as shown, into the flask A place, by means of a pipette, 2 fluid ounces of the test acid; this should be poured through the tube B, in order to thoroughly moisten with acid the pieces of broken glass contained therein. Then pour into the flask E, one measured ounce of the ammoniacal liquor, closing the stop-cock of the funnel immediately after so doing; a little distilled water should be added to the measure which contained the liquor, and the rinsings added to the contents of the flask E. One ounce of caustic soda solution, made by dissolving some "stick soda" in the proportion of 1 ounce of soda to 9 ounces of distilled water, is also added to the contents of the flask E. The Bunsen burner beneath the sand-bath is then lit, and the contents of the flask E are brought to the boiling point, and maintained in that condition for about 30 minutes, by which time all the ammonia contained in the liquor will have passed over into the flask A. The stop-cock on the funnel is then opened, the Bunsen burner turned off, and the apparatus allowed to cool. The point of the tube in the flask A must not touch the surface of the acid during the experiment. The apparatus is now disconnected, and the pieces of glass in B rinsed with a little distilled water into the flask A. The whole contents of the flask A are then transferred without loss to a beaker, and coloured with a few drops of cochineal solution.

109. The burette having been charged with the standard solution of soda (107) the beaker is placed underneath, and the soda solution run in, until the colour of the acid solution changes to purple, when the division at which the solution now stands in the burette must be noted. The strength of the liquor under examination is indicated by the quantity of soda solution remaining in the burette, which represents the quantity of ammonia which has been given off from the liquor and absorbed by the acid in the flask A.

For example, if 12 divisions of soda solution be left in the burette, the liquor contains sufficient ammonia in 1 gallon to neutralise 12 ozs. of pure strong sulphuric acid. The reason of this is obvious. In the flask A, 2 ounces of acid were placed, and the burette contained originally 2 ozs. of soda solution, which exactly matched the 2 ozs. of acid. In the flask E 1 oz. of the liquor was placed, consequently the volumes of the two standard solutions are exactly double the volume of the liquor operated on. Now if the liquor had been exactly sixteen-ounce strength, its ammonia would have neutralised just 1 fluid ounce, or half the acid employed; the remaining half would then have been neutralised by half the soda in the burette, or by 16 divisions, and therefore 16 parts would be left and indicate the strength of the liquor. The excess of acid is employed in order to ensure that no ammonia escapes.

110. The following are useful notes in connection with this subject:—

Each ounce of acid ($H_2 SO_4$) combines with 0·347 ounce of ammonia, forming neutral ammonium sulphate ($NH_2 SO_4$). In order to find the quantity of sulphate of ammonium capable of being produced from any liquor, the ounce strength of which has been determined by distillation in the manner described, multiply the ounce strength by 1·347, and by the number of gallons. If the strength of the liquor is given in terms of the percentage of ammonia, multiply the total percentage ounces by 4·61, then by 1·347, and finally by the number of gallons. The product of the whole will be the total weight in ounces of sulphate. In order to calculate direct into pounds per gallon, multiply the ounce strength by ·841 in place of 1·347, moving the decimal point one place to the left, thus, liquor ten-ounce strength 10 × 841 = 8·410, moving the decimal point = ·8410 pounds sulphate per gallon.

111. The value of a liquor may also be determined in the same apparatus as described above, using the ordinary systematic normal solutions: 10 c.c. of the liquor are distilled with caustic soda, and the evolved ammonia led into 20 c.c. of normal acid.

The acid is then titrated with normal caustic soda solution. As an example, 8 c.c. of normal soda solution were required to neutralise the 20 c.c. of acid, then the quantity of ammonia is 12 × 0·017 × 10 = 2·04 per cent.

112. In order to be able to tell whether the scrubbers are doing their proper amount of work in taking out the impurities $CO_2$ and $SH_2$ from the gas, it is desirable to make periodical analyses of the liquor for the amounts of these gases. The following are methods for determining the same:—

## *Estimation of Sulphuretted Hydrogen in Gas Liquor.*

Weigh out 100 grains of the gas liquor into a light glass flask, and wash the contents of the flask into a beaker; then add an excess of solution of arsenite of soda, and afterwards hydrochloric acid to acid reaction, and allow the precipitate to settle. This, which is $As_2S_3$, should be collected on a weighed filter, and treated in exactly the same manner as in the case of $As_2O_3$ previously given (26). Every 246 parts of $As_2S_3$ obtained correspond to 102 parts of $H_2S$, and from this the percentage of $H_2S$, by weight, in the sample is arrived at. The cubic inches per gallon may be obtained from the percentage by weight in the following manner :—

First, take, by a hydrometer, the specific gravity of the liquor, from which the weight in grains of one gallon of the liquor is obtained. Thus, supposing the gravity of the liquor to be 1030, we say—

| Sp. gr. $H_2O$ | | Sp. gr. liquor. | | Weight gallon $H_2O$ in grains. | | Weight gallon liquor in grains. |
|---|---|---|---|---|---|---|
| As 1000 | : | 1030 | : : | 70,000 | : | 72,100. |

We next require to find the grains of $H_2S$ in one gallon of the liquor, and, supposing the percentage by weight was found to be 0·58, we say—

| Weight in grains of liquor analysed. | | Percentage of $H_2S$. | | Weight of gallon of liquor in grains. | | Weight of $H_2S$ in a gallon of liquor in grains. |
|---|---|---|---|---|---|---|
| As 100 | : | 0·58 | : : | 72·100 | : | 418·1. |

We next require to find the cubic inches of $H_2S$, corresponding to 418·1 grains; and to do this we say—

| Weight of one cubic inch of $H_2S$ | | One cubic inch | | Weight of $H_2S$ in a gallon | | Cubic inches per gallon |
|---|---|---|---|---|---|---|
| As 0·365 | : | 1 | : : | 418·1 | : | 1145. |

113. A method of determining volumetrically the amount of $H_2S$ in a sample of gas liquor is also given.

It is based on the fact that when a solution of iodine is brought into contact with sulphuretted hydrogen, the following reaction takes place :—

$$H_2S + I_2 = 2HI + S.$$

The first operation is to prepare a standard solution of iodine, which is performed as follows :—

Dissolve 149·4 grains of pure iodine with 300 grains of potassium iodide. This is then diluted with distilled water until it exactly measures 2 decigallons, when 100 septems of the solution will equal 1 grain of $H_2S$.

The following is the method of making an experiment:—

Weigh out, into a small flask, 100 grains of the gas liquor, and wash this out with distilled water into a large beaker; dilute this solution with distilled water until it measures about a pint; add some starch paste, and afterwards acetic acid to acid reaction. Then, having placed the beaker on a white filter-paper, run in the iodine solution until the contents of the beaker turns a blue colour (showing that all the $H_2S$ has been neutralised), when the number of divisions required should be noted, each septem representing $\frac{1}{100}$th of a grain of $H_2S$.

The following are the reasons for the above operations:

According to the equations previously given, an equivalent of $H_2S$ requires two equivalents of iodine, or 34 grains of $H_2S$ would require 254 grains of iodine, consequently 1 grain of $H_2S$ would require 7·47 grains of iodine.

As the burette employed holds 100 septems, and as a decigallon equals 2000 septems, in the present instance it is made to contain the equivalent of 20 grains of $H_2S$, as 1 grain equals 7·47 of iodine.

As 100 grains of gas liquor were taken, and as the whole contents of the burette equals 1 grain, each division equals the hundredth of a per cent., by weight, of $H_2S$. Supposing, for instance, that a sample of the liquor required 54 divisions of the iodine solution, this would be equal to 0·54 per cent. by weight.

### *Estimation of Carbonic Acid in Gas Liquor.*

114. Weigh out, into a small flask, 100 grains of the liquor, then transfer to a large beaker, dilute largely with distilled water, and then add an excess of solution of barium chloride. The solution is then gently warmed for the space of half an hour on the water-oven, and afterwards filtered and

washed. The precipitate should then be ignited at a gentle heat apart from the paper of the filter, the ash of which should subsequently be added. Every 197 parts of $BaCO_3$ obtained equal 44 parts of carbonic acid.

The precipitate, however, contains, not only the $BaCO_3$ but also any sulphuric acid present. In order, therefore, to obtain perfectly accurate results, it will be necessary, after weighing the precipitate of $BaCO_3$, to dissolve it in dilute hydrochloric acid, any residue of $BaSO_4$ which remains undissolved being collected on a filter, washed, dried, ignited and weighed in the usual manner, the weight obtained being deducted from the original weight of the $BaCO_3$, and a calculation of the amount of $CO_2$ made on the corrected weight.

The following is the method of obtaining the cubic inches per gallon from the percentage by weight:—

Supposing the specific gravity of the liquor to be 1,030, and the percentage by weight 3·06.

First obtain the weight of a gallon of the liquor in grains —as given in the sulphuretted hydrogen example = 72,100; then find the grains of $CO_2$ in a gallon of liquor, thus—

| Grains of liquor analysed | | Percentage of $CO_2$ | | Gallon of liquor in grains | | Grains of $CO_2$ per gallon |
|---|---|---|---|---|---|---|
| As 100 | : | 3·06 | : : | 72,100 | : | 2206. |

Then, to find the number of cubic inches equal to 2206 grains, we say—

| Weight of one cubic inch $CO_2$ | | One cubic inch | | Weight of $CO_2$ in a gallon in grains | | Cubic inches per gallon |
|---|---|---|---|---|---|---|
| As 0·472 | : | 1 | : : | 2206 | : | 4673. |

115. The following method has been proposed by S. Dyson for a complete analysis of gas liquor.

(*a*) *Determination of total Ammonia.*—25 c.c. of the liquor are boiled with magnesia, and the evolved $NH_3$ collected in 50 c.c. of standard sulphuric acid, which is afterwards titrated back with standard soda. Suppose this showed 2·045 per cent. $NH_3$. (When employing magnesia it must not be forgotten that its action is much less energetic and slower than that of caustic soda or lime.)

(*b*) *Determination of total Sulphur.*—25 c.c. of the liquor are allowed to fall, drop by drop, from a burette into water acidified with hydrochloric acid and containing an excess of bromine. The excess of bromine is evaporated off, the solution filtered from the precipitate formed (which appears to mainly consist of tribromo-phenol) and the sulphur precipitated as barium sulphate. This yields *e.g.* 0·3915 per cent. S.

(*c*) *Determination of Sulphides.*—Zinc sulphate and ammonium chloride are added to 25 c.c. liquor; the precipitate is filtered off and well washed. The filter-paper is perforated, and small portions of the precipitate are gradually washed through into water acidulated with hydrochloric acid, and containing an excess of bromine. When the whole of the precipitate has been washed through, the solution is heated to expel the excess of bromine, filtered, and barium chloride added to the filtrate. Suppose this showed 0·190 per cent. S = 0·303 $NH_4HS$.

(*d*) *Determination of Carbonates.*—An ammoniacal solution of calcium chloride is added to 25 c.c. liquor.

The precipitated $CaCO_3$ is filtered off, dissolved in 50 c.c. standard hydrochloric acid, and the solution titrated back with standard soda. Suppose this showed 1·795 per cent. $CO_2$ = 3·916 $(NH_4)_2CO_3$.

(*e*) *Determination of Chlorides.*—50 c.c. are evaporated to dryness in the water-bath. Water is added, and the undissolved tarry matters filtered off. The filtrate is mixed with a solution of copper sulphate and ferrous sulphate in about equal proportions (in order to remove thiocyanates) and the liquid again filtered.

Nitric acid and silver nitrate are added to the filtrate and the solution is boiled. The precipitate is allowed to settle and the supernatant liquid poured through a filter. Before finally filtering off the silver chloride, it is digested several times with hot nitric acid, to dissolve out the silver sulphide resulting from the decomposition of the silver thiosulphate. The silver chloride is then filtered off and weighed. Suppose this showed 0·944 per cent. $Cl = 14·23\ NH_4Cl$.

(*f*) *Determination of Thiocyanates.*—This is not quite easy, and is best done in the following way:—50 c.c. of the gas liquor are evaporated to complete dryness, and the residue is heated to 100° C. for three or four hours, in order to make the precipitate of thiocyanate, to be obtained later on, less finely divided. The residue is then digested with strong alcohol, rinsed on to a filter and washed with alcohol. The alcoholic filtrate is evaporated to dryness, water is added, and the insoluble organic matter filtered off.

A solution of ammonia thiocyanate is thus obtained, tolerably free from other ammoniacal salts (especially thiosulphate) and from organic matter. This solution is precipitated by the addition of copper sulphate and sulphurous acid (which is preferable to ferrous sulphate as a reducing agent) it is gently warmed (not boiled !), and, after settling, the cuprous thiocyanate, $CuCNS$, is filtered off. It is then washed into a flask, dissolved in nitric acid, and the liquid boiled for a considerable time. The copper is then precipitated as oxide by caustic soda; the weight of $CuO \times 0·96 =$ the equivalent amount of $NH_4CNS$. Suppose we obtain 0·180 $NH_4CNS$.

(*g*) *Determination of Sulphates.*—250 c.c. are evaporated to dryness. Water is added; the organic substances filtered off, and the solution boiled with HCl. A little zinc oxide is added, the liquid filtered, and the sulphates precipitated with barium chloride. Suppose this showed 0·019 per cent. $(NH_4)_2SO_4$.

(*h*) *Determination of Thiosulphates.*—This cannot be done by any direct method, but it can be arrived at by subtracting, from the amount of total sulphur, that existing as sulphides, thiocyanates and sulphates.

In the example before us—

| | | |
|---|---|---|
| Total sulphur ... .. .. .. .. .. .. .. .. | | 0·3915 |
| Sulphur in sulphides .. .. .. .. .. | 0·1900 | |
| „ thiocyanates.. .. .. .. .. | 0·0757 | |
| „ sulphates .. .. .. .. .. | 0·0046 | |
| | | 0·2703 |
| Sulphur in ammonium thiosulphate = 0·280 per cent $(NH_4)_2S_2O_3$ .. .. | | 0·1212 |

(*i*) *Ferrocyanides.*—The residue obtained by evaporating 250 c.c. of the liquor to dryness is dissolved in water, the solution filtered, and ferric chloride added to the filtrate. The precipitate of Prussian blue is filtered off, washed, and decomposed with caustic soda. The ferric hydroxide thus obtained is, after filtering and washing, dissolved in dilute sulphuric acid, reduced, and the solution titrated with centinormal potassium permanganate. The Fe × 5·07 equals the amount of $(NH_4)_4FeCy_6$. Suppose we have found 0·0415 per cent. of this compound.

The result will therefore be that one litre of the sample of gas liquor analysed contains in grams—

| | | | $NH_3$ |
|---|---|---|---|
| Total ammonia .. .. .. .. .. | 20·45 | | |
| Sulphur .. .. .. .. .. .. .. | 3·92 | | |
| Ammonium sulphide $NH_4HS$ .. .. .. | 3·03 | = | 1·01 |
| „ monocarbonate $(NH_4)_2CO_3$ .. | 39·16 | = | 13·87 |
| „ chloride $NH_4Cl$ .. .. .. .. | 14·23 | = | 4·52 |
| „ thiocyanate $NH_4CNS$ .. .. .. | 1·80 | = | 0·40 |
| „ sulphate $(NH_4)_2SO_4$ .. .. .. | 0·19 | = | 0·05 |
| „ thiosulphate $(NH_4)_2S_2O_3$ .. .. | 2·80 | = | 0·64 |
| „ ferrocyanide $(NH_4)_4FeCy_6$ .. .. | 0·41 | = | 0·10 |
| | | | 20·59 |

### *Analysis of Sulphate of Ammonia.*

116. The apparatus used in the estimation of ammoniacal liquor may be used for the analysis of $(NH_4)_2SO_4$. A carefully selected sample from the bulk of the salt is well ground up, and the whole of it passed through a sieve of about

8 meshes per linear inch, and a small portion taken from this. 10 grams of the latter are dissolved in 500 c.c. of water, and 50 c.c. of this solution are placed, together with an excess of caustic soda, into the flask E, and the ammonia evolved led into 20 c.c. of normal (55) acid contained in the flask A. At the end of the distillation, the acid is titrated back with normal caustic soda solution (58).

The following is an example :—

After going through the operations described above, it was found that the 20 c.c. of normal acid required 7 c.c. of normal caustic soda for neutralisation, then 13 c.c. of acid have been saturated by the $NH_3$ given off from the $(NH_4)_2SO_4$. As the 10 grams of $(NH_4)_2SO_4$ originally taken were dissolved to 500 c.c. and 50 c.c. used, it follows that the $NH_3$ given off from 1 gram of $(NH_4)_2SO_4$ amounts to $0 \cdot 017 \times 13 = 0 \cdot 221$ grams (1 c.c. normal acid corresponds to 0·017 grams ammonia). From these figures it follows that the salt contains 22·1 per cent. $NH_3$.

## V. Analysis of Lime.

117. Lime as employed in gasworks for purifying purposes, is usually obtained from some of the different varieties of limestone which are found in various parts of the country. The most suitable limestone for the production of a lime best adapted for purifying purposes, is the white chalk limestone, as this consists of almost pure carbonate of lime.

Some limestones contain notable quantities of carbonate of magnesia, and are then known as dolomites. The magnesium limestones of Yorkshire and Derbyshire are of this description.

On burning the limestone, carbonic acid is driven off, leaving caustic or quicklime, and it is in this condition that the lime is usually met with. The following equation shows what occurs :—$CaCO_3 = CaO + CO_2$.

Quick- or caustic lime (CaO) when brought into contact with water, enters into chemical combination with the latter, the combination being so energetic as to produce great heat. The resulting substance is a definite chemical compound, known as calcic hydrate, or slaked lime, the operation of adding water to the lime being known as slaking ($CaO + H_2O = CaO.H_2O$). A good idea of the value of a lime may be formed from the way it behaves on slaking; a good lime should slake easily, and form, when thus treated, a light uniform powder, free from lumps, which, on the addition of dilute hydrochloric acid, should not give any marked effervescence, indicating that all the $CO_2$ has been driven off in the operation of calcining the limestone.

When quicklime is exposed to the action of the atmosphere for a considerable length of time it slowly absorbs water therefrom, and falls to powder. In that condition it forms an exceedingly light dry powder, similar to calcined magnesia in appearance. Such lime which has "fallen," or become "air-slaked," as it is termed, is partially hydrated, its probable formula being, according to Prof. Wanklyn, $(CaO)_2H_2O$; lime in this condition is very caustic and corrosive. Lime is placed in the purifiers after being slaked.

118. The theoretical purifying value of perfectly pure hydrate of lime is as follows:—

Seventy-four parts by weight are capable of uniting with 44 parts of $CO_2$, or 34 parts of $H_2S$. Adopting a theoretical unit of 1 lb. of pure calcium hydrate, such amount is capable of uniting with 0·594 lb. of carbonic acid, equal to about 5 cubic feet, or to ·46 lb. of sulphuretted hydrogen equal to the same bulk. The actual value of any particular sample of lime will, of course, be less than the theoretical value, according to the amount of impurities which the lime contains.

119. The impurities existing in lime may be derived from the following sources:—

The chalk or limestone from which the lime is obtained may be impure, and therefore yield a correspondingly inferior lime on being calcined; the burning may not have been efficient, the consequence of which would be to leave an undue percentage of undecomposed carbonate in the lime; the lime may have been of good quality in the first instance, but may have become subsequently deteriorated by exposure to the weather (air-slaked).

120. In ascertaining the commercial value of a lime for purifying purposes, it is not generally necessary to make an exhaustive analysis with the idea of ascertaining the amount of the various impurities present, all that is required is to determine the amount of available lime (CaO). One method of arriving at the value of a sample of lime is to note the amount of water it takes up on slaking, as the weight of water taken up represents the quantity which has entered into chemical combination with the caustic lime, as previously explained.

121. A fair sample of the lime is taken, and from this between 6 and 7 grams are weighed out into a porcelain basin. Water is then added, in sufficient quantity to thoroughly slake the lime. The basin and its contents are then placed in an air-oven, and dried at a temperature of 250° F., and when quite dry, weighed. The total weight will have increased by the amount of water necessary to convert the caustic lime into hydrate, and the amount of such increase is, therefore, an indication of the purity or impurity of the sample. As an example, 6·84 grams of lime were weighed out into a basin, slaked, dried and weighed as above described; the gain of weight observed in the second weighing amounted to 2·05 grams = 30 per cent., consequently, as 18 parts of water absorbed, represent 56 parts of caustic lime, the percentage of the latter is obtained from the following equation:—

$$\text{As } 18 : 30 :: 56 : x.$$

$$x = 93{\cdot}33 \text{ per cent. of caustic lime.}$$

122. The amount of caustic lime alone, however, does not

always fairly represent the actual purifying value of any particular sample, for a portion of the real lime present may have already been converted into hydrate by the absorption of moisture from the atmosphere, and consequently, it would not be able to take up any more water. It is necessary, therefore, in order to ascertain the actual purifying value of any sample, to determine the amount of the whole of the lime present in an available condition. This is effected as follows :—

123. A portion of the slaked lime obtained as above, equal to a definite amount of the original lime, is placed in a beaker, and dissolved in an excess of a measured quantity of normal hydrochloric acid (56), should it be necessary to assist the solution by heat employing only a very moderate temperature. When dissolved, a little distilled water is added, and the solution coloured with a few drops of methyl orange solution. The liquid is then titrated with normal soda solution (58), until the pink colour of the solution changes to yellow. On deducting the number of c.c. of the soda solution which are required to neutralise the acid from the number of c.c. of acid added to the lime, the number of c.c. of acid which it has taken to combine with the lime, in accordance with the following equation, is arrived at :—

$$CaO,H_2O + 2HCl = CaCl_2 + 2H_2O,$$

and from this the percentage amount of lime can readily be calculated.

The following is an example on another sample of lime :— A portion of lime, weighing 5·462 grams, on being slaked, absorbed 1·44 grams of water = 26·36 per cent., which would correspond to 82 per cent. CaO. A fifth part of this slaked lime, weighing 1·380 grams, corresponding to 1·0924 grams of the original lime, was then dissolved in 50 c.c. normal hydrochloric acid. On titrating this solution with normal soda solution, it required 16·4 c.c. of the latter to neutralise it; therefore 50 − 16·4 = 33·6 c.c. of the acid were required to combine with the lime in the sample inferior lime on being calcined; the burning may not have been efficient, the consequence of which would be to leave an undue percentage of undecomposed carbonate in the lime; the lime may have been of good quality in the first instance, but may have become subsequently deteriorated by exposure to the weather (air-slaked).

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Therefore $0 \cdot 028 \times 33 \cdot 6 = 0 \cdot 9408$ gram of lime present, consequently $\frac{0 \cdot 9408 \times 100}{1 \cdot 0924} = 86 \cdot 1$ per cent. of lime in all forms, caustic, hydrated and carbonated. If there is much carbonate of lime present, shown by the lime effervescing strongly on the addition of the acid, it will be necessary to estimate the amount as in (34).

Should there be much difference between the result of the slaking and of the acid test as just described, the lime is either not sufficiently burnt, and will therefore contain a quantity of useless carbonate, which is shown by the effervescence just spoken of, or else it has been exposed to the air for some time, and become air-slaked to some extent.

124. The following is another method of determining the value of a sample of lime:—

For the amount of free CaO, weigh 100 grams of an average sample, slake it completely, put the resulting milk of lime into a half-litre flask, fill up to the containing mark, and shake well. Then take out 100 c.c., run it into a half-litre flask, fill up with distilled water, mix well, and employ 25 c.c. of the contents, equal to 1 gram of quicklime (CaO), for the test. Titrate with normal oxalic acid (57) and phenolphthalein as an indicator. The colour is changed when all free lime has been saturated and before the $CaCO_3$ is acted on. For $CO_2$, the CaO and $CaCO_3$ are titrated together by dissolving in an excess of normal HCl (56) and titrating back with normal soda (58). By deducting the CaO first obtained, the quantity of $CaCO_3$ is arrived at. For very accurate estimations the $CO_2$ is expelled by HCl, and absorbed in soda lime as in (34).

## VI. ANALYSIS OF LIMESTONE.

125. As mentioned under the analysis of lime, the lime employed in gasworks is usually met with as quick, or caustic lime, but as it may sometimes be requisite to know

the composition of the chalk or limestone from which the lime is obtained, the following method may be employed. The substance selected for an example is dolomite or magnesium limestone, which contains both calcium and magnesium carbonates. Limestone also contains moisture, silicious matter, iron and aluminium, and sometimes manganese and the alkali metals. Since the composition of limestone varies, in order to make quite certain what constituents are present, an exhaustive qualitative examination should precede the quantitative analysis. In the present example a homogeneous piece of dolomite should be selected, about 15 grams finely powdered, and the powder placed at once in a stoppered weighing bottle.

(*a*) *Estimation of Moisture.*—Dolomite as a rule does not contain much moisture. In order to ascertain whether any moisture is present, and at the same time to estimate its amount, 1·5 to 2 grams are accurately weighed out in a pair of watch-glasses provided with a brass clip, Fig. 22; the clip and upper watch-glass are removed, and the lower glass containing the powder is dried in the air-oven for an hour at a temperature of about 200° C. The substance is then removed from the air-oven, immediately covered with the upper watch-glass, the clip replaced, and the glasses allowed to cool in the desiccator. When cold the glasses are weighed. Should any loss of weight have taken place, the substance is dried again for half an hour in the same way, and weighed once more. These several processes are repeated, until the weight remains constant in two successive trials. The loss of weight then observed will give the amount of moisture present.

(*b*) *Estimation of* $CO_2$.—Transfer the dried dolomite from the last operation to the flask, Fig. 24. Decompose it with hydrochloric acid, and estimate the $CO_2$ gas evolved exactly as is described in (34). Care should be taken that the soda lime absorption tube *f* contains sufficient of the reagent to completely absorb all the $CO_2$ gas evolved; preferably a afterwards precipitated in the filtrate as hydrated peroxide of manganese.

In order to precipitate the iron and aluminium as basic acetates, the acid filtrate from the silica determination, is considerably diluted with water, and a solution of ammonium carbonate is gradually added, with constant stirring, until the precipitate which first forms is with difficulty redissolved. The colour of the solution will now have become a deep brown. The ammonium carbonate solution is continued to be added very slowly, until a very slight precipitate forms, which just redissolves on stirring. This is readily seen to be the case when the liquid becomes slightly opalescent.

A moderate quantity of ammonium acetate solution is now added, and the liquid boiled for a short time. The liquid is filtered while hot, and the precipitate twice washed with boiling water. Any manganese which has been precipitated with the Fe and Al is removed by redissolving the precipitate in hydrochloric acid, adding ammonium carbonate as before, and reprecipitating the Fe and Al with ammonium acetate solution as just described. The precipitate will now contain the iron and aluminium as basic acetates, the manganese remaining in solution.

Filter and wash the precipitate, without stopping, with hot water; add the first portion of the washings to the two filtrates, and treat the liquid, which should be perfectly colourless, as follows. Add bromine water to the solution until it acquires a permanent yellow colour, then add excess of ammonium hydrate solution, and warm. The manganese will be precipitated as hydrated peroxide. Filter this off, wash and ignite it strongly, and weigh as $Mn_3O_4$].

(*f*) *Estimation of Calcium.* — Collect the ammoniacal filtrates and washings from the iron and aluminium precipitate (*d*), heat the liquid to boiling, and then add a moderate excess of ammonium oxalate solution. Allow the precipitate to settle sufficiently long, then filter, and wash the precipitated calcium oxalate two or three times. Redissolve the precipitate in hot dilute hydrochloric acid, and repeat the precipitation by adding a slight excess of ammonium hydrate solution, together with a few drops of ammonium oxalate solution. Filter off, wash, dry and ignite the precipitate, and weigh it as carbonate, and finally as oxide, as in (28). The reprecipitation of the calcium oxalate is necessary, in order to completely separate the magnesium which is at first thrown down with the calcium.

(*g*) *Estimation of Magnesium.* — The precipitates and washings from the precipitation of the calcium are poured into a porcelain dish of about six inches diameter, and the liquid evaporated down to dryness, care being taken that it does not creep over the edge of the dish. When all the liquid has been driven off, place the dish on a sand-bath or on a piece of wire gauze, and heat it as long as ammoniacal fumes are driven off. It is necessary to remove these ammoniacal salts on account of their preventing the complete precipitation of magnesium. The residue in the dish is treated with a little strong hydrochloric acid, water added, and if necessary filtered. A moderate excess of ammonium hydrate solution is added to the clear liquid, and then an excess of sodium hydrogen phosphate solution. The liquid is allowed to stand for a few hours, or vigorously shaken in a stoppered bottle, the precipitate is filtered off, and washed with dilute ammonium hydrate solution; it is then ignited, and the Mg weighed as $Mg_2P_2O_7$ (32).

(*h*) *Estimation of the Alkali Metals.*—These are not usually determined in a limestone; they can, however, be estimated if necessary, by dissolving 5 grams of the dolomite in hydrochloric acid, and adding an excess of milk of lime, prepared by stirring up fresh and pure slaked lime into a cream with water. The milk of lime precipitates the whole of the metals present, except the alkali metals. The excess of lime is then removed by adding ammonium hydrate and ammonium oxalate solutions in excess, the precipitated calcium oxalate is filtered off, the filtrate is acidified with

HCl, and evaporated to dryness in a weighed platinum or porcelain dish.

The chlorides of sodium and potassium thus obtained, are heated for a short time on the sand-bath, and, after cooling in the desiccator, are finally weighed. A little calcium is frequently found in this residue, it is therefore always advisable to dissolve it in water containing a few drops of dilute HCl, and then to add a few drops of ammonium hydrate and of ammonium oxalate solution. If a precipitate forms, filter, evaporate the filtrate, and ignite and weigh the residue. The residue is then dissolved in water, a few drops of HCl added, and then an excess of platinic chloride solution; the liquid is evaporated on the water-bath until a semi-solid crystalline mass is obtained. This is then treated with alcohol, in which the potasssium salt $K_2PtHCl_6$ is insoluble. The further treatment is the same as in the analysis of fire-clay (131*e*).

In addition to the constituents enumerated above, limestone may also contain phosphate, sulphate and chloride. The phosphate may be estimated by dissolving from 3 to 5 grams of the dolomite in nitric acid; most of the acid is evaporated off, the liquid diluted, the residue filtered off, and the $(PO_4)$ in the filtrate precipitated with a solution of ammonia molybdate, and the phosphate estimated as $Mg_2P_2O_7$.

The sulphate may be estimated by dissolving 5 grams of the dolomite in dilute hydrochloric acid, filtering off the residue, and precipitating the $SO_3$ as $BaSO_4$.

The chloride may be estimated by dissolving 5 grams of the dolomite in nitric acid, filtering off the residue, and precipitating and weighing as AgCl.

126. The following is a rapid method for the analysis of a limestone:—

Dissolve 1 gram in HCl, filter the residue, wash, dry and ignite. In the presence of appreciable quantities of organic matter, weigh the filter after drying at 100° C. and ignite afterwards. The difference is taken as organic matter.

For the amount of lime, dissolve 1 gram in 25 c.c. normal HCl (56), and titrate with normal alkali (58). Deduct the latter from 25, and multiply the remainder by 2·8 to find the percentage of CaO, or by 5 to find the percentage of $CaCO_3$.

In this case MgO is calculated as CaO; if necessary the MgO or $MgCO_3$ obtained may be deducted. Magnesia is estimated by dissolving two grams of the limestone in HCl, precipitating the CaO by $NH_3$ and ammonium oxalate, filtering, and precipitating by sodium phosphate. The precipitate is allowed to stand for 24 hours, filtered, washed with dilute ammonia, dried, ignited and weighed as magnesium pyrophosphate, one part of which = 0·36036 MgO.

## VII. Analysis of Oxide of Iron (Bog Ore).

127. The principal information required in the analysis of oxide of iron for purification purposes is the amount of moisture and ferric oxide, and this is obtained as follows:—

(*a*) *Gravimetrically.*—Obtain a fair sample from the bulk, weigh out about six grams into a tared watch-glass, and dry in the water-oven at a temperature of 100° C. until the weight is constant; this will give the amount of water held in loose combination; the residue is then transferred to a platinum crucible, and ignited at a moderate red heat. The weight lost on ignition is due to the water of hydration and organic matter.

The residue in the crucible is then washed into a beaker, and digested with strong pure hydrochloric acid, a small quantity of pure nitric acid being subsequently added. The digestion should be continued until the undissolved residue has lost its red colour, showing that all the ferric oxide has passed into solution. The contents of the beaker are then diluted with distilled water and filtered, with the object of getting rid of the silica, sand, &c.; the filter containing the silica is washed with distilled water until quite free from acid. The filtrate is then heated, and a slight excess of ammonia added, which will precipitate the ferric oxide and alumina. The mixed precipitate is then filtered, and afterwards dissolved by pouring moderately concentrated hydrochloric acid on the filter, finally washing the latter with a small quantity of distilled water, so as to remove any traces of iron solution remaining on the filter-paper. The solution thus obtained is then brought to the boiling point, and an excess of solution of pure caustic potash added. This will precipitate the ferric oxide, which is then washed, dried, ignited and weighed as $Fe_2O_3$ (29); the alumina in solution can be estimated by adding a slight excess of strong HCl, and then a very slight excess of ammonium hydrate. The precipitate is then filtered off, washed, dried, ignited and weighed as $Al_2O_3$ (30).

(*b*) *Volumetrically.*—The amount of ferric oxide in bog ore may also be determined, volumetrically, by means of permanganate or bichromate of potash.

The solutions employed should be about decinormal strength. If permanganate is the standard solution used then the iron should be dissolved in sulphuric acid, and reduced to the ferrous state by means of zinc, but as most ores dissolve rather sluggishly in sulphuric acid, it is preferable to dissolve in hydrochloric acid, and reduce with ammonic bisulphite, employing bichromate as the standard oxidising solution.

The following is the method of making a determination:—Weigh out about 1·5 grams of the finely powdered ore, and dry in the water-oven at 212° F.; the loss is the amount of water loosely combined. The residue is then gently ignited; the loss on ignition is the combined water and organic matter, peat, &c. The ore is then digested with moderately strong hydrochloric acid for some time until all the iron is extracted; the solution is then diluted and filtered, and, to the filtrate, ammonia is added until a faint precipitate of ferric oxide appears. This is redissolved with a few drops of HCl and some strong solution of bisulphite of ammonia, added in the proportion of about 1 c.c. for 0·1 gram of ore, or 0·05 gram Fe. The mixture is well stirred, boiling water added, then acidified with dilute sulphuric acid, and boiled for half an hour; it is then ready for titrating.

Of course, when reduced to the ferrous state, the solution must be preserved out of contact with the air by the employment of the flask, Fig. 35.

As an example, 1·5 grams of bog ore were weighed out dried, ignited, dissolved and reduced as described. The solution then required 67 c.c. $\frac{N}{10}$ bichromate solution (63), which multiplied by $0·0056 = 0·3752$ gram Fe $= ·536$; $Fe_2O_3 = 35·7$ per cent.

## VIII. ANALYSIS OF SPENT OXIDE OF IRON.

### *Method of Determining the Amount of Sulphur in Spent Oxide of Iron.*

128. This method is based on the fact that free sulphur is dissolved by bisulphide of carbon, and on evaporating off the bisulphide, the sulphur is left behind in the solid form, in which state it may be weighed.

The apparatus employed, was designed by Mr. A. Stephenson of the Gas Purification Co., and is shown in Fig. 47.

Weigh 100 grams of spent oxide, dry at 212° F. in the water-oven, and weigh at intervals until a constant weight is obtained, which will give the amount of moisture. Place the dried material in the drawn out test-tube A, on a layer of about 2 inches of cotton wool. It is necessary to exercise a little care in placing the cotton wool in the test-tube; if rammed too tightly it will prevent the solution of sulphur filtering through; if too loosely it will permit some of the oxide to pass. Bisulphide of carbon is blown from the bottle B into the test-tube A on top of the spent oxide.

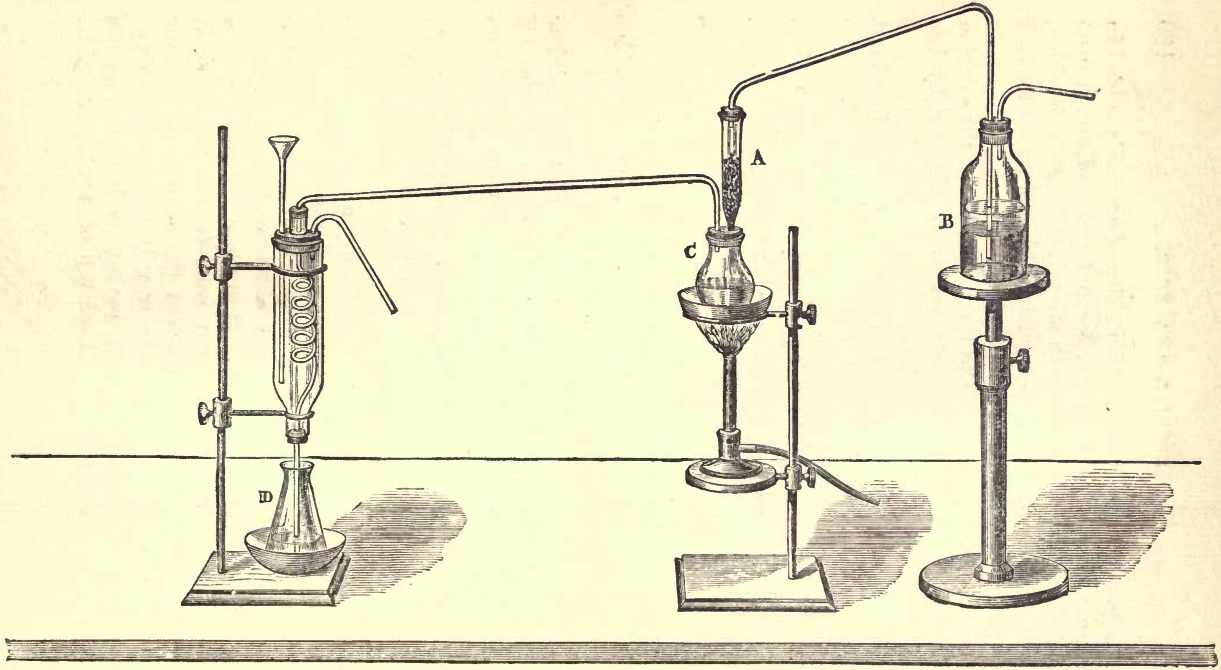

FIG. 47.

The $CS_2$ gradually percolates through the material, dissolving out the sulphur in its course, the solution finding its way by gravitation into the small flask C (previously weighed when clean and dry), which is placed in a copper water-bath. The Bunsen burner is then lighted, and the application of heat soon vaporises the bisulphide of carbon from the flask C. The vapour travels through the connecting tube into the condenser, and is recovered in the receiver D, under water, ready for further use. The sulphur remains behind in the flask C.

The oxide is treated with the bisulphide until the whole of the sulphur is dissolved out; three or four times the bulk of the oxide is about the proportion of $CS_2$ necessary to effect this.

When all the sulphur has been dissolved out, the flask C is placed in the water-oven to drive off the last remaining traces of $CS_2$, and weighed at intervals, until the weight is constant. The weight of the flask and sulphur, minus the weight of the empty flask, will give the weight of sulphur, and this will be the percentage amount on the "wet" sample.

It is necessary to remember that $CS_2$ is very inflammable, and in the gaseous state, when mixed with air in certain proportions, is explosive. The bisulphide in the holder should be covered with water.

The amount of free sulphur in spent Weldon Mud and lime may also be determined by the above method.

### *Estimation of Sulphocyanides in Spent Oxide.*

129. Fifty grams of the spent oxide, after freeing from sulphur, are extracted with warm water, and the solution partly evaporated on a water-bath, acidified with sulphurous acid, and precipitated with an excess of copper sulphate solution. The white precipitate is collected on a weighed filter, dried and weighed. 121·5 grams of copper salt correspond to 76 grams of ammonium sulphocyanide. If no chloride, cyanide, or ferrocyanide is present this estimation

may be expeditiously made with decinormal silver solution. 10 c.c. of this are taken and mixed with a few drops of nitric acid and ferrous ammonium sulphate solution, and titrated with the solution of sulphocyanide diluted to a known volume, till a red coloration is produced. Suppose that the aqueous extract of 50 grams spent oxide has been diluted to 500 c.c., and that 20 c.c. of the solution are required for the titration of 10 c.c. decinormal silver solution, then the whole liquid contains $\frac{10 \times 0 \cdot 0076 \times 500}{20} = 1 \cdot 9$ grams, and the spent oxide consequently contains $2 \times 1 \cdot 9 = 3 \cdot 8$ per cent. of ammonium sulphocyanide.

130. Estimation of ferrocyanides in spent oxide.

The residue from the extraction of the sulphocyanide, corresponding to 50 grams spent oxide, is warmed for some time with an excess of caustic soda. There should not be too great an excess, and the whole should be well stirred. The mixture is then filtered, the residue washed with hot water, and the filtrate boiled with pure ammonium chloride or sulphate solution until no further smell of ammonia is perceptible. In this manner the $Al_2O_3$ dissolved by the NaHO is precipitated.

Its elimination is necessary, as it is precipitated, together with Prussian blue, even in slightly acid solutions; moreover, by its precipitation, dark tarry matters are likewise carried down, and the solution is thus partially clarified. After filtering off the $Al_2O_3$, the solution is made slightly acid with HCl, heated to boiling, and poured into a hot dilute solution of ferric chloride. The Prussian blue thus precipitated is allowed to settle, the dark red liquid poured off, and the precipitate well washed by decantation.

It is then poured on to a filter, and washed with hot water until the filtrate is free from iron, dried and ignited in a platinum crucible to which the air has access. It is thus converted into ferric oxide, which is weighed, and the quantity of ferrocyanide calculated from the weight found.

560 parts of ferric oxide correspond to 636 parts ferrocyanogen (Fe $(CN)_6$) and to 860 parts of Prussian blue.

Dr. Arnold, in his 'Ammonia and Ammonium Compounds,' describes a volumetric process which he is in the habit of using.

42·2 grams of pure dry potassium ferrocyanide ($K_4Fe(CN)_6 + 3H_2O = 422$) are dissolved in water and the solution diluted to one litre. A dilute, slightly acid, ferric chloride solution is placed in a burette and 10 c.c. of the ferrocyanide solution titrated with it. So long as an excess of ferrocyanide remains in solution, no Prussian blue is precipitated. If a drop of the dark blue liquid be placed on filter-paper, it spreads out to a uniform blue spot. As soon, however, as all ferrocyanide is converted into Prussian blue, the spots do not spread uniformly, but give a blue patch, in which the particles of precipitated Prussian blue are easily seen. The end of the titration may be accurately detected by employing filter-paper previously soaked in a solution of ammonium sulpho-cyanide. The slightest excess of iron is then recognised by the fact that the blue particles no longer appear on a white, but on a yellow or red ground.

The strength of the iron solution having thus been ascertained, it is diluted to correspond with the ferrocyanide solution, and again compared with the latter in the manner above described. With this iron solution, all liquids containing ferrocyanides can be titrated with fairly accurate results. The liquids must, however, be neutral and free from alumina. Let it be supposed that 20 c.c. of the neutralised caustic soda extract (diluted previously to 500 c.c.) require 30 c.c. of ferric chloride solution, then as 1000 c.c. ferric chloride solution corresponds to 21·2 ferrocyanogen $(Fe(CN)_6)$ the whole extract contains

$$\frac{\frac{21\cdot2 \times 10}{1000} \times 500}{20} = 5\cdot3 \text{ grams} = 2 \times 5\cdot3$$

= 10·6 per cent. of ferrocyanogen.

## IX. Analysis of Fire-Clay and Fire-Bricks.

131. Fire-clay is the name given to any clay which is capable of standing a high temperature without melting; such clays are said to be refractory.

A refractory fire-clay will contain nearly pure hydrated silicate of alumina.

The more alumina that there is in proportion to the silica, the more infusible will be the clay. The composition of different fire-clays necessarily varies however. They contain

From 59 to 96 per cent. silica ($SiO_2$)
„ 2 „ 36 „ alumina ($Al_2O_3$)
„ 2 „ 5 „ oxide of iron ($Fe_2O_3$),

and a very small percentage of lime, magnesia, potash and soda.

The fire-resisting properties of the clay depend chiefly upon the relative proportions of these constituents. If the oxide of iron or alkalies are present in large proportions, they act as a flux, and cause fusion, the clay is then no longer refractory.

The following is the method of analysis. A quantity of the substance (fire-clay or brick) is reduced to an impalpable powder in an agate mortar and placed in a stoppered weighing tube.

About 2 grams of the sample are dried in a platinum crucible or platinum dish at a temperature of 100° C. until the weight is constant; the loss in weight gives the moisture. In the case of clay it is then ignited, at first gently, and afterwards strongly, and for a tolerably long time; the loss in weight corresponds with the combined water, together with the organic and volatile constituents of the clay if such are present.

1·5 grams of the powdered sample are then weighed accurately out into a platinum crucible and about four times its weight of a fusion mixture added, consisting of sodium and potassium carbonates mixed in molecular proportions. The whole is intimately mixed by means of a smooth rounded glass rod. It will be found convenient to add the fusion mixture by small portions at a time, since in this way a more thorough mixture is obtained. The mixture should only half fill the crucible.

The lid is then placed on the crucible, and the latter gently heated over the Bunsen flame; the temperature is gradually increased, care being taken that no loss ensues by the frothing due to the evolution of $CO_2$. When the mass is fused, the crucible is transferred to the blow-pipe flame, and the whole is kept at a bright red heat until effervescence ceases and the fused mass becomes tranquil. The flame is then removed, and the crucible is allowed to cool just below redness, when it is placed on a cold surface, such as a clean block of iron, so as to assist it to cool rapidly. When cold, the crucible and its contents are placed in a deep evaporating dish or in a shallow beaker; this is covered with a clock-glass, and tolerably strong hydrochloric acid added to the contents, which should be gently agitated after each addition of the acid, and kept covered during the operation. When effervescence has ceased, and the crucible is free from all adherent solid, remove the crucible by means of the crucible tongs, carefully rinsing off any adhering liquid by means of the jet from the wash-bottle, into the main portion of the liquid. On treating the fused mass with HCl as above described, most of the $SiO_2$ will separate out as a gelatinous mass.

If any gritty particles are felt, on stirring the bottom of the vessel with a glass rod, the fusion is imperfect. This is generally due to the original substance not having been powdered sufficiently finely. In this event, it is usually more satisfactory to make a fresh fusion, taking care that no coarse particles are present in the portion of the sample for the fresh fusion.

(*a*) *Estimation of the Silica.*—The liquid containing the gelatinous silica is then transferred (if necessary) to an evaporating basin, preferably of platinum, and evaporated to dryness upon a water-bath. When the contents of the basin become pasty, they should be continually stirred with a rounded glass rod to prevent the formation of lumps. When all the liquid has been driven off, the contents of the dish should be in the state of a fine powder. In order to expel the last trace of HCl, the dish should now be placed upon a sand-bath, and heated with a small Bunsen flame until no moisture is deposited on a cold clock-glass when placed upon the dish for a few seconds. The dish is then allowed to cool, and its contents are moistened with strong HCl. It is then heated on a water-bath for about half an hour, a small quantity of hydrochloric acid being occasionally added with stirring. Hot distilled water is now added, and the silica is filtered off and is washed free from dissolved chlorides. The precipitate is ignited apart from the filter (19*b*), the precipitate being transferred to the platinum crucible cautiously, since it consists of a very light powder, which is easily blown away. The lid is placed on the crucible, and the latter heated, exceedingly gently at first, and the temperature raised very gradually, or the escaping steam will carry some of the fine light powder away with it. The crucible is finally raised to a full red heat over the Bunsen flame.

(*b*) *Estimation of* $Al_2O_3$ *and* $Fe_2O_3$.—The filtrate from the $SiO_2$ is mixed with $NH_4Cl$ solution and then with $NH_3$ in slight excess, the hydrates of iron and aluminium, of which the precipitate consists, being separated as under.

The precipitate is washed, and dissolved upon the filter in hot dilute hydrochloric acid, and the solution allowed to flow into a nickel or porcelain dish containing about 50 c.c. of *pure* strong KHO solution. Wash out the acid which remains adhering to the filter-paper with a small quantity of distilled water, and allow these washings to also run into the dish, and boil the contents of the latter for a few minutes. The iron will be precipitated as ferric hydrate, while the hydrate of aluminium will remain in solution.

The iron precipitate is filtered off, it is then again dissolved in HCl, and reprecipitated by ammonium hydrate, in order to free the ferric hydrate from potash. It is then washed and ignited apart from the filter, at a red heat, and weighed as $Fe_2O_3$. The solution of the aluminium hydrate in the potassium hydrate solution is treated with a slight excess of strong hydrochloric acid, and then with a very slight excess of ammonium hydrate. It is then filtered off, washed, dried, ignited, and weighed as $Al_2O_3$.

Another method of separation is, after weighing the mixed hydrates of Fe and Al, to dissolve them in $KHSO_4$ and about 5 c.c. $H_2SO_4$, add about 1 gram hyposulphite of soda, boil, and titrate the solution with $\frac{1}{100}$ normal bichromate of potash; this will give the amount of iron.

(*c*) *Estimation of the Calcium.*—If the volume of the filtrate from the iron and alumina precipitate is very large, evaporate it down to a convenient bulk, add a little ammonium hydrate if not already alkaline, and then a slight excess of ammonium oxalate. Allow the liquid to stand, filter off, ignite, and weigh the precipitate as oxide (28).

(*d*) *Estimation of the Magnesium.*—Evaporate the filtrate and washings from the calcium oxalate precipitate to dryness, ignite the residue, and treat it with a little strong HCl, add water and filter, if necessary. To the clear solution add ammonium hydrate in moderate excess, and then an excess of sodium hydrogen phosphate solution. Allow the liquid to stand for a few hours, or shake it vigorously in a stoppered bottle, filter off, wash the precipitate with dilute ammonium hydrate solution, then ignite it, and weigh the Mg as $Mg_2 P_2O_7$.

(*e*) *Estimation of the Alkali Metals.*—Since sodium and potassium carbonates have been employed in the fusion, the alkali metals cannot be estimated in the filtrate from the magnesium. A separate portion of the substance is accordingly used for their determination.

Lawrence Smith's method for the determination of the alkali metals will be found the most convenient. The following is the mode of procedure.

Weigh out accurately about 1·5 grams of the finely powdered substance into a platinum crucible, intimately mix this with a mixture of 1·5 grams of pure recrystallised ammonium chloride, and 9 grams of pure calcium carbonate. Then heat the crucible to bright redness for an hour over a good Bunsen or blow-pipe flame, or preferably as follows.

Place the platinum crucible in a clay crucible containing a little calcined magnesia or lime at the bottom and round the sides, and heat the clay crucible in a gas furnace, which is capable of maintaining it at a bright red heat. When the crucible has been heated for an hour, allow it to cool, and place the platinum crucible and its contents in hot water in a covered platinum or porcelain dish, and boil for a time.

This procedure will dissolve out the alkaline chlorides together with some calcium hydrate. Filter and mix the filtrate with ammonium hydrate and ammonium carbonate solutions in excess, and with a few drops of ammonium oxalate solution. Allow the liquid to stand, filter into a platinum or porcelain dish, evaporate the filtrate to dryness, and heat the residue just below redness, but sufficiently strongly to drive off the ammoniacal compounds.

Dissolve the residue in water; add a few drops of AmHO and of ammonium oxalate solution to precipitate any trace of calcium still in solution; filter, and evaporate the filtrate to redness, with a few drops of HCl, in a weighed dish. Gently ignite the residue and weigh, repeating the ignition until the weight is constant. The weight of the residue thus obtained gives the combined weight of the potassium and sodium chlorides.

The residue is then dissolved in water and the potassium chloride is precipitated by platinic chloride in the following manner. To the solution of the residue a few drops of HCl are added, then an excess of platinic chloride solution, the liquid being afterwards evaporated on the water-bath until a semi-solid crystalline mass is obtained.

The platinic chloride is seen to be in excess by the supernatant liquid being of an orange colour, after the liquid has been concentrated to a small bulk.

When it is certain that there is an excess of platinic chloride, we may then proceed according to *a* or *b* as below.

(*a*) Pour alcohol upon the mass; gently shake the liquid round in the dish so as to mix the contents of the same well together, allow the precipitate to settle completely and pour off the liquid through a tared filter. Repeat these operations twice, and finally transfer the undissolved double salt to the filter by means of a small wash-bottle filled with alcohol. Continue washing the precipitate upon the filter with alcohol until the washings are no longer coloured. Dry the filter and its contents at 100° C., and weigh as $2KClPtCl_4$.

(*b*) A rather quicker method of treating the precipitated double salt is to wash it with alcohol by decantation until the alcohol is no longer coloured, the alcohol being decantated through an untared filter-paper. Care must be taken that as little as possible of the precipitate is poured off with the alcohol. The double salt, freed from the excess of $PtCl_4$ is now washed into a platinum crucible, dried at 100° C. and weighed. The filter, which will contain a little of the double salt, is then incinerated, and the ash is dropped into the crucible and weighed. By deducting from this weight the weight of the filter ash, the approximate weight of platinum left by ignition is found; this is calculated into double salt and the weight is added to that of the double salt already found in the crucible. If the quantity of precipitate left on the filter is appreciable, the weight of KCl left in the filter ash, not being allowed for, will introduce an error. The filter in this case should be ignited in a separate crucible, the KCl washed out from the ash by hot water, and the dried residue weighed. The true weight of the platinum in the ash is thus ascertained, and is made use of as mentioned above.

The weight of the sodium chloride in the mixed chlorides of potassium and sodium is then ascertained by difference. The chlorides are finally calculated into oxides.

## X. Assay of Coal Tar.

132. The assay of coal tar is usually limited to a laboratory operation, in which the various fractions are collected as nearly as possible under the same conditions as pertain to the large scale, though the details will necessarily vary with circumstances. With practice, good results are obtained with as small a quantity as 10 oz. of the tar, the yield corresponding closely with those given on a large scale; but the chief value of such laboratory operations is for comparing different samples of tar.

The following is the method of making the assay :—250 c.c., or 10 oz., measure of the tar to be examined should be placed in a glass retort, of such a capacity that the tar only fills about one-third of it, the object of this being to prevent the distillate being spoilt in the event of there being much frothing during the distillation. The retort may be conveniently supported on a cup-shaped piece of wire gauze, placed in an aperture in a sheet-iron plate. Over the retort a dome is placed: this may be made by removing the bottom from a tin can or bottle, and cutting out a piece of the side to allow the neck of the retort to pass through. The object of this contrivance is to confine the heat, and prevent the distillate or heavy vapour from falling back: without some arrangement of this kind, a satisfactory assay of coal tar in a glass vessel is nearly impossible.

The products obtained by the distillation are : (1) ammoniacal liquor; (2) total light oils; (3) creosote oil; (4) anthracene oils; and (5) pitch.

In obtaining the above fractions the character of the distillate is quite sufficient to indicate the point at which the receiver should be changed.

It is not necessary to insert a thermometer, nor to connect the retort to any condensing arrangement.

The lamp being lighted underneath the retort (a powerful

Bunsen should be used, as towards the close of the experiment it is necessary to maintain the wire gauze at a red heat), the ammoniacal liquor and naphtha should be collected together in a graduated cylinder, which should be changed when a drop of the distillate collected in a test-tube of water begins to sink. In this fraction the ammoniacal liquor and the total "light oils" will be found: the expression "light oils" means oils lighter than water. After standing for some time to allow perfect separation of the ammoniacal liquor and light oils, the volume of each should be observed, and, if deemed necessary, the strength of the liquor ascertained in the usual way by distillation with caustic soda, and titration of the distillate. The quantity of light oils obtainable will not be sufficient to allow of any further fractionation for benzols, &c.

The next fraction of the distillate will consist of creosote oil. At the commencement it will contain much naphthalene, and will very probably solidify in white crystals on cooling; but later on a more fluid distillate will be obtained. At a still later stage, a drop of the distillate collected on a cold steel spatula will be found to deposit amorphous solid matter, of a yellow, or greenish-yellow colour. When this action takes place, the receiver should again be changed, and the fraction obtained measured. If required, this fraction, which is called "carbolic and creosote oils," may be assayed for carbolic acid and naphthalene, as described later on.

With modern London tars it is generally found that this fraction is semi-solid; it will therefore be necessary to measure it while quite warm.

The next fraction of the distillate will be found rich in anthracene, and very often condenses in the neck of the retort as a yellow, waxy substance, which may, however, be melted out by the local application of a small Bunsen flame. The collection of the anthracene oil is known to be complete when no more distillate can be obtained, and the pitch remaining in the retort intumesces and gives off heavy, yellow fumes.

The distilled fraction containing the anthracene oil should then be measured and cooled thoroughly, and the resultant pasty mass pressed between folds of blotting paper, weighed, and assayed for real anthracene by the anthraquinone test. The result should be calculated into crude anthracene of 30 per cent., a standard which is generally adopted by manufacturers.

When the distillation for anthracene oil is complete, the retort should be allowed to cool, and when almost cold, the body of it should be immersed in cold water. This operation produces a rapid surface-cooling and shrinking of the pitch from the glass, which may then be broken away and removed by gentle tapping, leaving the cake of pitch clean, and in a fit state for weighing.

The following figures by Mr. B. Nickels, F.I.C., show the result obtained by the assay of four representative samples of London tar. The apparent excess is due to the fact that the samples of tar experimented on were measured into the retort, while the resultant pitch was weighed:—

| | A. | B. | C. | D. |
|---|---|---|---|---|
| Ammoniacal liquor .. | 2·5 | 3·7 | 8·0 | 5·0 |
| Total light oils .. .. | 2·5 | 3·4 | 0·5 | 3·2 |
| Carbolic and creosote oils | 21·3 | 17·0 | 23·0 | 20·0 |
| | Solid. | Solid. | Solid. | Solid. |
| Anthracene oils .. .. | 17·0 | 17·0 | 13·0 | 13·0 |
| | Semi-solid. | Semi-solid. | Semi-solid. | Semi-solid. |
| Pitch (grams per 100 c.c.) | 59·4 | 60·0 | 58·0 | 62·0 |
| | 102·6 | 101·1 | 102·2 | 103·2 |
| Pressed anthracene .. .. | 4·0 | .. | 1·5 | .. |
| Containing real anthracene | 13·4 per cent. | | 25·68 per cent. | |
| = 30 per cent. crude anthracene in tar | 1·8 | 1·47 | 1·3 | 1·12 |

The following table shows the mean composition of the foregoing samples in juxtaposition with the general yield of English country tar:—

| | London. | Country. |
|---|---|---|
| Ammoniacal water .. .. .. | 4·5 per cent. | 4 per cent. |
| Total light oils .. .. .. | 2·4 ,, ,, | 3 ,, ,, |
| Carbolic and creosote oils .. | 20·3 ,, ,, | 22 ,, ,, |
| Anthracene oils .. .. .. | 15·0 ,, ,, | 4 ,, ,, |
| Pitch (grams per 100 c.c.) .. | 59·9 ,, ,, | 67 ,, ,, |
| | 102·1 | 100 |

In the above assay the total light oils were collected together, but in practice, on the large scale, the distillate is generally divided into two fractions, which are known as "first light oils" and "second light oils." When they are not collected separately, the fraction is known as "crude naphtha" or "light oil."

The crude naphtha is generally re-distilled without any previous chemical treatment, the resultant distillate being known as "once run naphtha" and "last runnings."

On the large scale, once run naphtha, before being re-distilled, is purified by treating it with sulphuric acid of sp. gr. 1·845. By this means the hydrocarbons of the ethylene and crotonylene series are removed, as well as some of the higher homologues of benzene. A further treatment with caustic soda eliminates phenols and other bodies having an acid character. The oil is afterwards washed with water and again distilled.

## XI. Fractional Distillation.

### *Rectification of Benzene.*

133. It is often possible to separate almost completely, by a simple distillation, two liquids occurring together in a mixture, when their boiling points lie widely apart. The more volatile liquid first passes over, the temperature suddenly rises, and the higher-boiling liquid distils. It is otherwise when we have a liquid consisting of a mixture of

bodies boiling very near each other, especially in the case of homologous compounds such as occur in petroleum and coal-tar naphtha; one distillation only effects a very imperfect separation; a portion of the less volatile liquid is carried over by the vapour of the more volatile substance, the temperature rising throughout the distillation. In order to effect separation of the several substances in a case of this kind, recourse is had to the method of fractional distillation. It will be evident that if the vapours arising from a mixture of boiling liquids are somewhat cooled before reaching the condenser proper, *the less volatile portion*, carried upwards by the vapour of the more volatile liquid, will be partially condensed. If this is permitted to flow back into the retort, only the lower boiling portion passes into the condenser, and the separation is consequently more rapid and effective.

In order to carry out this partial condensation during the process of distillation, several forms of apparatus have been devised.

A very cheap and effective apparatus is that of Hempel, which consists of a long glass tube containing glass beads, or pieces of broken glass, Fig. 48.

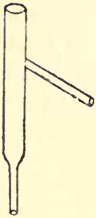

FIG. 48.

Fig. 49 shows the apparatus of Le Bel and Henninger for the same purpose; this form of apparatus has side tubes down which the condensed liquid flows. At the narrow parts of the tube *a*, *b*, *c*, *d*, are fixed small cups of wire gauze. Little pools of condensed liquid form in these cups, and this liquid may be said to wash the vapour passing upwards; in fact, a process of fractionating is carried on in these cups by the ascending vapours.

In carrying out a fractional distillation, the apparatus is arranged as in Fig. 50.

The flask is heated over wire gauze, or, in the case of a very volatile liquid, in a water-bath.

If wire gauze is used, the burner should be placed in a deep tin basin containing sand, in order to absorb the liquid in the event of the flask or retort cracking. The fractionating bulb or tube is fitted with a thermometer, the bulb of which is well below the exit tube. A number of clean dry flasks or bottles, fitted with corks and having labels attached, are required for recording the boiling point of the fraction.

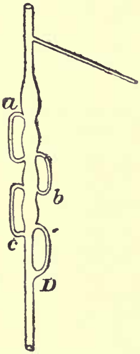

FIG. 49.

We will show the method of applying this principle in the rectification of a sample of benzene.

200 c.c. of the sample of benzene are introduced into an 8-oz. flask; a spiral of platinum wire or two or three pieces of broken pipe stems are added, in order that the liquid may boil without bumping. The flask is connected to the condenser by means of the fractionating apparatus, and heat so applied to the flask that the distillate comes over into the receiver in drops; it must not be allowed to come over in a continuous stream. The

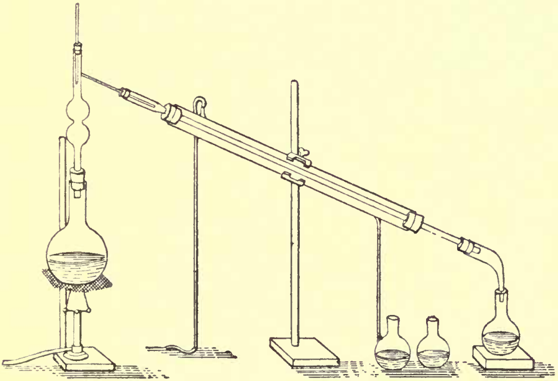

FIG. 50

indications of the thermometer must be carefully watched and noted, and the distillate between every five degrees collected in separate flasks.

These different fractions are then redistilled in order, adding the next to the residue of the previous one in the distilling flask. The portions boiling below 85° C. and above 105° being collected between every two or three degrees. The fractions below 85° and above 105° are redistilled as before and collected between every two degrees.

| A.<br>71·5–85° C. | B.<br>85–90°. | C.<br>90–95°. | D.<br>95–100°. | E.<br>100–105°. | F.<br>105–110°. | G.<br>110–115°. | Residue. |
|---|---|---|---|---|---|---|---|
| 19 c.c. | 53 c.c. | 26 c.c. | 15 c.c. | 13 c.c. | 17 c.c. | 21 c.c. | 33 c.c. |

| | A′.<br>below 79°. | B′.<br>79–81°. | C′.<br>81–85°. | D′.<br>85–105°. | E′.<br>105–108°. | F′.<br>108–110°. | Residue. |
|---|---|---|---|---|---|---|---|
| A | 5 c.c. | | | | | | |
| Added B | | 42 c.c. | (10 c.c.*) | | | | |
| Added C | | | (9 c.c.*) | | | | |
| Added D E | | | | 50 c.c. | | | |
| Added F | | | | | (11 c.c.*) | | |
| Added G | | | | | | 22 c.c. | 42 c.c. |
| * Refractioned C′ | | 12 c.c. | 7 c.c. | | | | |
| E′ | | | | | 6 c.c. | 5 c.c. | |
| | 5 c.c. | 54 c.c. | 7 c.c. | 50 c.c. | 6 c.c. | 27 c.c. | 42 c.c. |

The fraction 79-81° C., which is nearly pure benzene, can be further purified by freezing, pressing, and redistilling.

It will be found that by repeating the process the liquid is gradually separated into two large, fractions, consisting chiefly of benzene and toluene, and a number of smaller intermediate fractions.

The table by Dr. Cohen on page 206 gives the volume in c.c. and the boiling points of the fractions obtained by this method from 200 c.c. 50 per cent. benzene, each table denoting a complete series of fractionations.

## XII. Determination of the Specific Gravity of Gas.

134. This may be most conveniently performed by a method devised by Mr. Greville Williams, F.R.S., a description of which appeared in the Transactions of the Gas Institute for 1882. The following is taken from the above mentioned source:—

"The specific gravity of a gas may be defined as the comparative weights of equal volumes of air and the gas under the same conditions as to temperature and pressure. Whether the gas should be dried or not before its density be taken is a matter of no importance as regards the present paper. I, however, invariably weigh the gas and air in an absolutely dry state. It is quite unnecessary for me to describe the processes devised by Regnault, Bunsen, and others, as I have already done so very fully in Watt's 'Dictionary of Chemistry,' and in my 'Handbook of Chemical Manipulation.' . . . The sole object of this paper is to show that, by a simple modification of well-known processes, and without the use of an air pump, an accuracy may be obtained which leaves nothing to be desired for technical purposes; and the formula given for the calculations, which is of the utmost possible simplicity, does not introduce an error of any importance.

"If, however, perfect accuracy be desired, the corrections mentioned further on can easily be made. As with the method described by Banister,* only a globe with two stopcocks is used, which is represented in Fig. 51, one-quarter the actual size. Instead, however, of weighing the balloon, as Banister directs, against weights, I weigh against another balloon of as near as possible the same size, but a little lighter—a device very generally adopted in the determination of specific gravities of gases and vapours.

"The determination of the specific gravity of a gas is by no means a difficult operation; but, nevertheless, to obtain accurate results, attention must be paid to the minutest precautions, the neglect of any one being fatal to success. In the first place, the balance should be of the highest class; both planes and edges must be of agate or rock crystal; and it should distinctly indicate one deci-milligramme with the globe and its counterpoise hanging from the beam. If this be the case, the capacity of the globe need not be greater than 400 cubic centimetres.

"The usual methods employed involve exhaustion of the balloon by the air pump, either at each experiment, or, as with Letheby's, a preliminary exhaustion, which serves once for all. By the method here described, not only is no exhaustion required, but all corrections are avoided by the simple device of selecting a day when the barometer is steady, and working in a room where the temperature can be made the same when the globe is weighed with air as when it is weighed with gas. Both these conditions, I find, can be readily obtained in practice.

"The room in which I worked formed the centre one of a series of three, and had no doors; notwithstanding this, by means of a gas stove, I found it easy to regulate the temperature to 0·2 of a degree centigrade. If the room be too cold, the heat may be raised; if it be too hot, cooler air may be introduced by the ordinary means employed for ventilation. By working in this way, it will be seen that

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* 'Gas Manipulation,' Banister and Sugg, pp. 18, 19, 20.

the air and the gas may be weighed under the same conditions of temperature and pressure. It is proper, however, to remark that the air must be rendered absolutely free from carbonic anhydride and moisture. For this purpose it was drawn slowly through one bottle of potassium hydrate solution, two of sulphuric acid, six U-tubes of very active soda-lime, and four U-tubes of calcic chloride, before entering the globe. One U-tube of calcic chloride was placed, as a guard, after the globe.

"The drawing through of the air is effected by means of an aspirator, and the current is kept up until every trace of any gas previously experimented on is removed; this may be known by the balloon remaining constant in weight. When this is arrived at, and the temperature at which it is desired to make the experiment attained (and which should be as near as possible the average of the room), the tube attached to the aspirator is to be slipped off, and the tap of the globe on that side closed, and then the other tap. The balloon, having been carefully wiped with a clean chamois leather, or a soft silk handkerchief kept for that purpose, is to be hung on the balance by a stirrup of platinum wire, as represented in Fig. 51. The counterpoise, Fig. 52, is also to be hung on the balance by the glass hook. The counterpoise being somewhat lighter than the balloon, a very few grammes will be required to bring them into equilibrium.

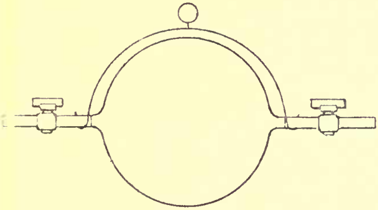

FIG. 51.

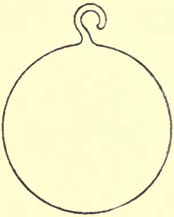

FIG. 52.

"Having adjusted the last deci-milligramme, by means of the rider apparatus, the balloon is to be left for five minutes, and the weight taken again; the last weight is to be called 'weight of balloon and air.' The balloon is then to be attached to a tube evolving the gas, which is to be passed slowly through six U-tubes of soda lime, to remove the last traces of carbonic acid, and four of calcic chloride, for one hour; a guard tube, as before, being employed at the exit. The tap next the gas supply is to be turned off first, the other immediately after; the balloon is then to be weighed with the same precautions as were taken with air. It is as well, in a first experiment, to send the gas a second time through the globe for another half hour; when, if it be found that the globe has not diminished in weight, it is, of course, certain that all the air has been expelled. The last weight is to be entered in the laboratory book as 'weight of balloon and gas.'

"From this time a specific gravity may be determined every hour without reweighing the balloon and air, as long as the barometer and thermometer remain the same. The weights of the balloon and air at various barometric pressures and temperatures should be tabulated, as when that pressure and temperature occur again, the previous determination of weight of air will render another one unnecessary.

"It is obvious that the temperature of the room in winter will be different to that in summer, but this will have no effect upon the result, as all that is necessary is that the temperature should be the same when both gas and air are weighed.

"Care must be taken that the calcic chloride and soda-lime are renewed at proper intervals.

"In order to subject the process to the severest possible test, I made some determinations of the specific gravity of hydrogen in the globe, Fig. 51, having a capacity of 443 cubic centimetres. The specific gravity of hydrogen being only 0·0693, it will be seen that an error of only 0·01 is very conspicuous. In four experiments, the following values were obtained :—

| No. | P. Grammes. | V. c.c. | T. C. | $n_t$ | D. | Theory. | Difference. |
|---|---|---|---|---|---|---|---|
| | | | Deg. | | | | |
| I. | 0·5038 | 443 | 16 | 0·001221 | 0·0685 | 0·06930 | −0·0008 |
| II. | 0·5045 | 443 | 14 | 0·001230 | 0·07412 | 0·06930 | +0·00482 |
| III. | 0·5020 | 443 | 16 | 0·001221 | 0·07192 | 0·06930 | +0·00262 |
| IV. | 0·5040 | 443 | 16 | 0·001221 | 0·0682 | 0·06930 | −0·0011 |

| Mean. | Theory. | Difference. |
|---|---|---|
| 0·0707 | 0·0693 | +0·0014 |

"If all the corrections are made, the mean density becomes a little higher, but even then the error is only 0·0126, which is well within the limits laid down at starting. Bunsen's standard experiments with his apparatus gave 0·079 ; difference 0·010.

"In the above table, P is the difference between the weight of the balloon and air, and the balloon and gas, in grammes. V is the capacity of the balloon in cubic centimetres ; T the temperature, centigrade ; $n_t$ the weight of one cubic centimetre of air at T ; D the experimental specific gravity. As care has been taken that the barometer shall not vary during the experiment, it does not enter into the formula, which for gases lighter than air is—

$$D = \frac{Vn_t - P}{Vn_t}.$$

"The value of $n_t$ may, when extreme exactitude is not required, be taken direct from the following table ; otherwise $n_t$ should be corrected for the elastic force of the atmosphere at the time of the experiment, as the table is calculated for a barometric pressure of 760 mm., or 29·922 inches.

"Having adjusted the last deci-milligramme, by means of the rider apparatus, the balloon is to be left for five minutes, and the weight taken again; the last weight is to be called 'weight of balloon and air.' The balloon is then to be attached to a tube evolving the gas, which is to be passed slowly through six U-tubes of soda lime, to remove the last traces of carbonic acid, and four of calcic chloride, for one hour; a guard tube, as before, being employed at the exit. The tap next the gas supply is to be turned off first, the other immediately after; the balloon is then to be weighed with the same precautions as were taken with air. It is as well, in a first experiment, to send the gas a second time through the globe for another half hour; when, if it be found that the globe has not diminished in weight, it is, of course, certain that all the air has been expelled. The last weight is to be entered in the laboratory book as 'weight of balloon and gas.'

"From this time a specific gravity may be determined every hour without reweighing the balloon and air, as long as the barometer and thermometer remain the same. The weights of the balloon and air at various barometric pressures and temperatures should be tabulated, as when that pressure and temperature occur again, the previous determination of weight of air will render another one unnecessary.

"It is obvious that the temperature of the room in winter will be different to that in summer, but this will have no effect upon the result, as all that is necessary is that the temperature should be the same when both gas and air are weighed.

"Care must be taken that the calcic chloride and soda-lime are renewed at proper intervals.

"In order to subject the process to the severest possible test, I made some determinations of the specific gravity of hydrogen in the globe, Fig. 51, having a capacity of 443 cubic centimetres. The specific gravity of hydrogen being only 0·0693, it will be seen that an error of only 0·01 is very conspicuous. In four experiments, the following values were obtained :—

| No. | P. Grammes. | V. c.c. | T. C. | $n_t$ | D. | Theory. | Difference. |
|---|---|---|---|---|---|---|---|
| | | | Deg. | | | | |
| I. | 0·5038 | 443 | 16 | 0·001221 | 0·0685 | 0·06930 | −0·0008 |
| II. | 0·5045 | 443 | 14 | 0·001230 | 0·07412 | 0·06930 | +0·00482 |
| III. | 0·5020 | 443 | 16 | 0·001221 | 0·07192 | 0·06930 | +0·00262 |
| IV. | 0·5040 | 443 | 16 | 0·001221 | 0·0682 | 0·06930 | −0·0011 |

| Mean. | Theory. | Difference. |
|---|---|---|
| 0·0707 | 0·0693 | +0·0014 |

"If all the corrections are made, the mean density becomes a little higher, but even then the error is only 0·0126, which is well within the limits laid down at starting. Bunsen's standard experiments with his apparatus gave 0·079; difference 0·010.

"In the above table, P is the difference between the weight of the balloon and air, and the balloon and gas, in grammes. V is the capacity of the balloon in cubic centimetres; T the temperature, centigrade; $n_t$ the weight of one cubic centimetre of air at T; D the experimental specific gravity. As care has been taken that the barometer shall not vary during the experiment, it does not enter into the formula, which for gases lighter than air is—

$$D = \frac{Vn_t - P}{Vn_t}.$$

"The value of $n_t$ may, when extreme exactitude is not required, be taken direct from the following table; otherwise $n_t$ should be corrected for the elastic force of the atmosphere at the time of the experiment, as the table is calculated for a barometric pressure of 760 mm., or 29·922 inches.

### TABLE FOR THE CALCULATION OF $n_t$.

| Temp. | | $n_t$ Grammes. | Temp. | | $n_t$ Grammes. | Temp. | | $n_t$ Grammes. |
|---|---|---|---|---|---|---|---|---|
| C. | F. | | C. | F. | | C. | F. | |
| Deg. | Deg. | | Deg. | Deg. | | Deg. | Deg. | |
| 1 | 33·8 | 0·001288 | 11 | 51·8 | 0·001243 | 21 | 69·8 | 0·001201 |
| 2 | 35·6 | 0·001284 | 12 | 53·6 | 0·001239 | 22 | 71·6 | 0·001197 |
| 3 | 37·4 | 0·001279 | 13 | 55·4 | 0·001234 | 23 | 73·4 | 0·001193 |
| 4 | 39·2 | 0·001275 | 14 | 57·2 | 0·001230 | 24 | 75·2 | 0·001189 |
| 5 | 41·0 | 0·001270 | 15 | 59·0 | 0·001225 | 25 | 77·0 | 0·001185 |
| 6 | 42·8 | 0·001266 | 16 | 60·8 | 0·001221 | 26 | 78·8 | 0·001181 |
| 7 | 44·6 | 0·001261 | 17 | 62·6 | 0·001217 | 27 | 80·6 | 0·001177 |
| 8 | 46·4 | 0·001257 | 18 | 64·4 | 0·001213 | 28 | 82·4 | 0·001173 |
| 9 | 48·2 | 0·001252 | 19 | 66·2 | 0·001209 | 29 | 84·2 | 0·001169 |
| 10 | 50·0 | 0·001248 | 20 | 68·0 | 0·001205 | 30 | 86·0 | 0·001165 |

"It is as well to repeat that the flow of a light gas should be continued until the balloon ceases to lose weight; and if the gas be heavier than air, until it ceases to gain in weight.

"The following numbers were obtained in the determination of the density of carbonic anhydride :—

| No. | P. Grammes. | V. c.c. | T. C | $n_t$. | D. | Theory. | Difference. |
|---|---|---|---|---|---|---|---|
| I. | 0·2833 | 443 | 17° | 0·001217 | 1·5254 | 1·5224 | + 0·0030 |
| II. | 0·2855 | 443 | 17° | 0·001217 | 1·5295 | 1·5224 | + 0·0071 |

| Mean. | Theory. | Difference. |
|---|---|---|
| 1·5274 | 1·5224 | + 0·0050 |

"The gas being heavier than air, the formula becomes

$$D = \frac{Vn_t + P}{Vn_t}.$$

"In conclusion, it may be said that, if the method above given be carefully carried out, the specific gravity of coal or any other gas, of which a cubic foot or two can be spared, may be readily and accurately determined, without the use of an air-pump."

# PART V.

## TECHNICAL GAS ANALYSIS.

### *Introduction.*

THE measurement of gases, and the quantitative analysis of gaseous mixtures, involve special methods and constitute a special branch of chemical analysis, frequently involving the use of special apparatus. Further, when dealing with the volume of a gas, special corrections and calculations are frequently required in order to arrive at comparative results. These corrections are necessitated by the very considerable effect which changes of temperature and pressure, and amount of moisture, exert upon the volume of a gas.

The most exact methods of gas analysis necessitate the use of mercury as the confining liquid, and also require a very considerable amount of manipulative skill on the part of the operator. They further involve the expenditure of a considerable amount of time, and in addition, require very expensive apparatus; consequently such methods are not adapted to the every-day requirements of a gasworks. For such requirements, something involving less time and care, even at the sacrifice of a slight degree of accuracy, is necessary. These conditions are complied with in a number of designs for apparatus for Technical Gas Analysis lately devised; amongst them, those of Hempel and Bunte are specially adapted for use in gasworks.

### *The Measurement of Gases.*

135. The volume of a gas can be found directly or indirectly; it is estimated either volumetrically, by titration, or gravimetrically. The quantity found is in all cases expressed in per cent. by volume. The volume of a gas is influenced by pressure, temperature, and the tension of the liquid over which it is measured.

Gases are measured in their condition at the time at which the measurement is made, that is at the pressure of the air as indicated by the barometer, and at the temperature as indicated by the thermometer. As water, in the case of technical gas analysis, is used as the trapping liquid, the gas is always in a state of complete saturation with moisture.

From the above remarks it will, be seen that the conditions under which gases are measured may be very different, and may vary during the progress of an analysis even from one observation to another. Every such change, unless duly taken notice of, might introduce very considerable errors. It is consequently necessary, in many cases, to make a correction, which consists in reducing the volume of gas, which is measured under known but varying conditions, to the volume which it would measure at the normal barometric pressure of 760 millimetres, at the normal temperature of 0° C., and in the dry state. Gases, when reduced to these standard conditions, are said to be in their normal state.

136. The reduction of the volume of a gas to the normal state is made by the aid of a formula based on the following data.

(1) *Pressure.*—According to Boyle's law the volume of a gas is in inverse proportion to the pressure to which it is subjected.

Consequently, if

$v$ = the observed volume of the gas,
$V_p$ = the volume of the gas at 760 mm. pressure,
$p$ = the observed barometric pressure,

$$\text{Then } V_p = \frac{p \times v}{760}.$$

(2) *Temperature.*—A gas expands by one-273rd = 0·003665) of its volume, for each degree centigrade rise n its temperature. Therefore, if

$v$ = the observed volume of the gas,
$V_\circ$ = its volume when reduced to 0° C.,
$t$ = the observed temperature,

$$\text{then } V_\circ = \frac{273 \times v}{273 + t} = \frac{v}{1 + 0{\cdot}003665 \times t}.$$

(3) *Correction for Moisture.*—When a gas is saturated with moisture by contact with water, under the same conditions of temperature and pressure, it always contains the same quantity of moisture. This water vapour mingled with the gas exerts a certain pressure, which is commonly termed the tension of aqueous vapour, and increases with the temperature. It is usually expressed in millimetres of mercury, and has been accurately determined for each degree of temperature (see Table in Appendix). It is necessary to deduct the pressure due to the tension of aqueous vapour from the barometric pressure.

Thus if $w$ is the tension of aqueous vapour in a volume of gas under pressure $p$, the true pressure on the gas $= (p - w)$. The three preceding corrections are embraced in the following formulæ, in which V represents the volume of the gas corrected for pressure and temperature, and for moisture if necessary.

$$\text{For a dry gas } V = \frac{p \times 273 \times v}{760\,(273 + t)} = \frac{p \times v}{760\,(1 + 0{\cdot}00365t)}.$$

For a moist gas $V = \frac{(p - w) \times 273 \times v}{760\,(273 + t)}$

$$= \frac{(p - w) \times v}{760\,(1 + 003665\,t)}.$$

137. Only those volumes can be directly compared with one another, that have been reduced to equal pressure and temperature, the tension of the confining liquid being also allowed for. Parallel gas measurements can be carried on as under—

(1) Varying pressure, varying temperature, and varying volume.

(2) Constant pressure, constant temperature, and varying volume.

(3) Constant temperature, varying pressure, and constant volume.

(4) Constant pressure, varying temperature, and varying volume.

In the first case, the gas volumes found must be reduced to like temperature and pressure. In the second and third, the resulting volumes can be directly compared since density and pressure are directly proportional.

The reductions of the volumes of gases to the normal state, may be omitted in analyses which are performed rapidly, as material changes of pressure and temperature are not likely to be then experienced; also in cases in which only approximately correct results are required.

### *Calibration of Measuring Tubes.*

138. The graduations of measuring tubes for gases represent either absolute cubic centimetres, and fractions of cubic centimetres capacity, or millimetres length on the tube.

In the latter case, it will be necessary to determine the value of the graduations in terms of cubic centimetres.

Since glass tubes always vary in diameter in different parts throughout their length, it follows that equal lengths on different parts of a graduated tube do not correspond to equal capacities; it is necessary, therefore, to calibrate a measuring tube throughout its length. The two liquids commonly used for calibrating tubes are mercury and water.

### *Calibration of the Hempel Burette.*

139. In this form of tube the gas is measured over water, consequently it is advisable to calibrate it by means of that liquid.

The measuring tube, Fig. 53, is disconnected from the pressure tube, and has then attached to its lower end, by means of a piece of india-rubber tubing, a glass tube about four inches long, with the end drawn out into a fine jet, and having a stop-cock in the centre. The stop-cock and tube must be firmly attached to the measuring tube by tightly binding the india-rubber tubing with thin copper wire, the glass ends butting against one another within the joint. The jet should be bent in a downward direction, so as to deliver the water drawn off, into a vessel placed beneath it. The measuring tube and stop-cock tube are completely filled with distilled water at the temperature of the room, which should be carefully noted. All air-bubbles must be excluded.

Successive portions of water, of 5 c.c. each, are then drawn off into a stoppered weighing bottle and weighed, the weight of water corresponding to each reading on the tube, being noted, and the bottle carefully dried between each weighing. The weight of water is then calculated into absolute volume, by applying the correction for the expansion of water given in the Table in the Appendix.

For example, if the first 5 c.c. weighed 4·92 grams at 17° C., its absolute volume would be $4{\cdot}92 \times 1{\cdot}00101 = 4{\cdot}925$ c.c.

When the top of the tube is being calibrated, it is

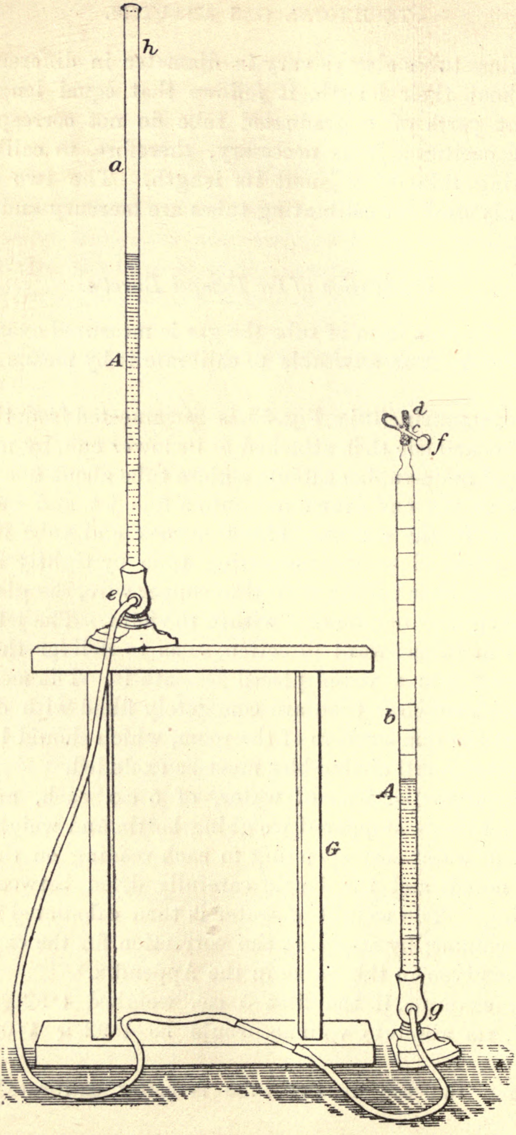

FIG. 53.

advisable to draw off smaller volumes of water than 5 c.c., if the measuring tube is to be used for small volumes of gas.

In all measurements in which water and similar liquids are employed, it is necessary that a certain time elapse for the liquid to drain down the sides of the measuring-tube before taking a reading. The time will vary from about 30 seconds to five minutes according to the liquid employed. On an average, however, about two minutes will be found sufficient.

## Gas Analysis by means of Hempel's Gas Apparatus.

140. The absorption of a gaseous constituent is frequently not carried out in the measuring tube itself, but in a separate vessel which serves for holding the absorbing liquid, and for bringing the gas into contact with it after being measured. When the absorption has been completed, the remaining gas is again carried over into the measuring tube, and its volume is read off. The volume of the gas absorbed follows from the difference of the two readings. This process admits of thoroughly utilising the absorbent, and dispenses with washing out the measuring tube after each estimation.

Hempel's apparatus is based upon the above principle, and consists of two portions; the burette, or measuring vessel, and the pipette or absorption vessel.

141. The gas burette is shown in Fig. 53.

It consists of two glass tubes, *a* and *b*, which are cemented into slots in semicircular weighted wooden feet, and are connected by about a yard of thin india-rubber tubing. To facilitate the cleaning of the burette, this india-rubber tube is divided in the centre, and the two ends are joined by a piece of glass tubing. Inside the feet, the tubes *a* and *b* are bent at right angles, and conically drawn out. The projecting ends are of about 4 millimetres external diameter, and are corrugated, to enable the india-rubber connecting tube to be tightly attached by means of thin copper wire. The measuring tube *b*, terminates at the top in a thick-walled capillary tube *c*, of from ½ to 1 millimetre internal diameter, and about 3 centimetres long. Over this a short piece of new black india-rubber tubing is firmly fastened on by means of thin copper wire. The india-rubber tube is closed by means of a strong brass pinch-cock which is placed close to the end of the capillary tube. The pinch-cock is always taken off from the india-rubber tube after using, as this helps to keep the latter in good condition. Notwithstanding the fact that readings cannot be made under the rubber tube, and that the pinch-cock cannot always be put on above the tube in exactly the same position, no practical error arises, since the glass tube *c* is so small in diameter. Hempel found that the differences in volume are much less than $\frac{1}{10}$ c.c., a variation which, in determinations not made over mercury, may be entirely disregarded. The graduated measuring tube *b*, contains 100 c.c., the lowest mark being slightly above the wooden foot. The cubic centimetres are divided into fifths, and the numbers run both up and down. The tube *a*, which may be called the level or pressure tube, is not graduated, and is somewhat widened at the upper end *h*, to facilitate the pouring in of liquids.

### *Manipulation of the Gas Burette.*

142. Fill the tubes *a* and *b* with water, taking care to drive all air out of the connecting rubber tube by suitably raising or lowering the tubes; then join the measuring tube to the gas supply by means of a glass or rubber tube containing water (this connecting tube can be easily filled with water by raising the pressure tube). To fill the burette with the gas to be examined, grasp the tube *a* in the left hand, close the rubber tube at *e* by pressing it between the little finger and the palm of the hand, and pour out the water in *a*.

Place the pressure tube on the floor, and open the pinch-cock *f*. The water will now flow into the pressure tube and the gas will be drawn into the measuring tube. When the latter is filled with the gas, close the pinch-cock *f*, disconnect *b* from the gas supply, and, after the liquid has run down the walls of the burette, take up the tubes by their wooden feet, and by raising or lowering, bring the water in the tubes to the same level. The gas is now under the pressure of the atmosphere, and its volume is read off. To measure off exactly 100 c.c., bring rather more than 100 c.c. of the gas into the burette, close the latter with its pinch-cock, and let the water run down. Now compress the gas to less than 100 c.c. by raising the pressure tube, close the rubber tube at *g* with the thumb and first finger of the left hand, set the pressure tube on the table, and raising the burette in the right hand to the level of the eye, carefully open the rubber tube, and let the water run back until the meniscus stands at the 100 c.c. mark. Keeping the rubber tube still compressed, open the pinch-cock for a moment. The excess of gas will escape, and there remains in the burette exactly 100 c.c. of gas under atmospheric pressure.

143. The next portion of Hempel's apparatus is the absorption pipette, which is used in connection with the burette or measuring tube. By using a series of these pipettes, a gaseous mixture can be submitted to the successive action of suitable absorbent reagents, and the proportions of the constituent gases thus ascertained.

The "simple absorption pipette" is shown in Fig. 54. It consists of two large bulbs *c* and *d*, joined together as shown, and of a thick-walled glass tube *a*, bent as in the figure, called the capillary tube. The bulb *c* holds about 150 c.c., and the bulb *d* about 100 c.c., so that when 100 c.c. of gas is brought into *c* sufficient space for the absorbing liquid will remain. To protect the pipette from being broken and to facilitate manipulation it is screwed on to a wooden stand. A short piece of india-rubber tubing is fastened by means of thin copper wire to the free end of the capillary tube.

### *Manipulation of the Absorption Pipette.*

147. To analyse a gas with the apparatus described, the burette is filled with distilled water which has been previously saturated with the gas to be analysed.

If simple pipettes are used, these are so filled with the absorbent that the bulb *d* remains empty. The absorbent also must be saturated, by shaking with the gases which are but slightly soluble in it. The saturation of liquids is most conveniently effected in a flask half filled with the liquid, a rapid stream of gas being led through the liquid, and the flask vigorously shaken. If the reagents in the absorption pipettes are at the temperature of the room, as can easily be ascertained by placing a thermometer at *k*, Fig. 56, the analysis is started by aspirating the gas into the measuring tube as already described. It will be found convenient to take exactly 100 c.c. so that the results may be read off directly in percentages.

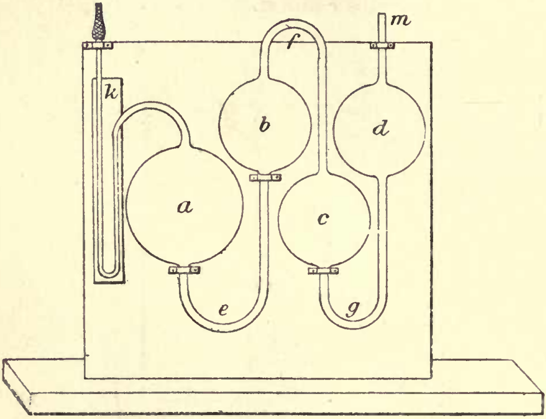

FIG. 56.

The apparatus is then arranged as in Fig. 57. The pipette is placed on the wooden stand G, and is connected with the burette by the capillary tube F, which is a piece of

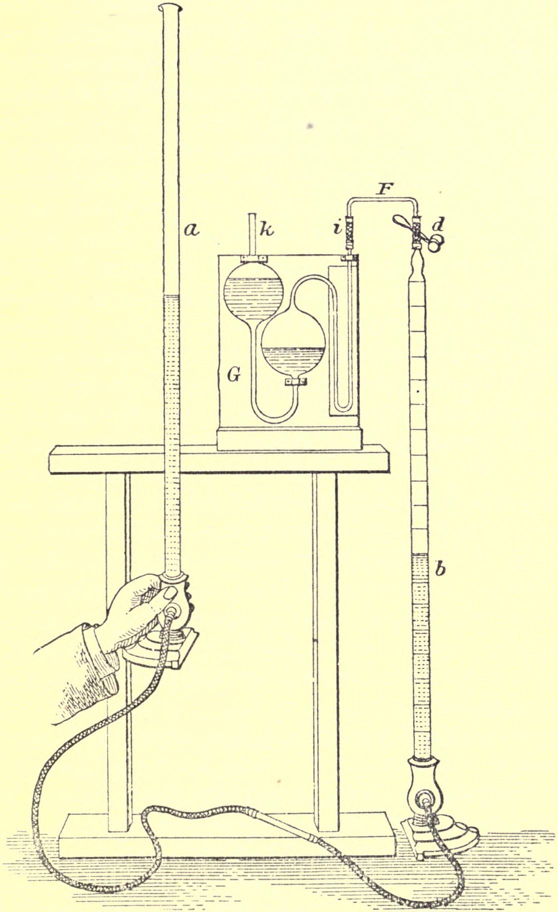

FIG. 57.

thermometer tubing of about 0·5 millimetre internal diameter. To avoid the enclosing of air bubbles, the rubber tube *d* is first filled with water by means of a capillary funnel, and the capillary F is then introduced; F is thus completely filled with water. The rubber tube *i* of the pipette is squeezed between the thumb and the first finger of the right hand, and while thus compressed and free from air, the capillary connecting tube is inserted.

Upon raising the pressure tube *a*, and opening the pinch-cock, the gas passes through the connecting tube into the absorption pipette. Any small air bubbles which may have been enclosed when F was inserted into *i*, are, at the beginning, separated from the gas by the water in F. If these bubbles do not take up more than 5 to 10 mm. space in the capillary tube of the pipette they may be neglected, since the error arising therefrom is only about 0·03 cubic centimetre. If the bubbles are larger, although with a little dexterity this may always be avoided, the gas is brought back into the burette by lowering the pressure tube and the operation is repeated. When the gas has passed over into the pipette, about ½ c.c. of water is allowed to follow, this water serving to rinse the capillary tube, and to free it sufficiently from the absorbing liquid which it previously contained. The gas is now enclosed between two columns of liquid, the absorbent on the one side, and the water in the capillary on the other. The burette having been closed by the pinch-cock, the pipette is disconnected and shaken, and the absorption of the gas thus effected.

The burette and pipette are then reconnected, the pressure tube is placed on the floor, and the gas is brought back into the burette, care being taken that the absorbing liquid does not pass farther than the connecting capillary F. The pinch-cock is closed, the pipette removed, and the reading of the remaining volume is made as before described.

The manipulation of the pipettes filled with solid absorbents is simpler still, for in this case no shaking is necessary, because of the large amount of surface contact, between the solid and the gas.

148. A separate pipette is used for each absorbent. After using, the pipettes are closed at *i* with a piece of glass rod, and at *k* with a small cork.

149. In order to obtain good results with the above apparatus, it is necessary to watch that the apparatus and reagents do not change in temperature during the progress of an analysis. A rise of temperature of 1° would cause an error of 0·3 per cent. in a total volume of 100 c.c.

It is also of importance that the confining liquids be allowed to flow down from the walls of the burette in exactly the same manner after each absorption, otherwise an error may be caused by the adhesion of more or less liquid to the glass of the instrument. In the case of gases which are confined over water, readings which are made one minute after the gas has been shaken with water in the burette, differ by several tenths of a c.c. from readings made five minutes later. Distilled water will run down completely in five minutes.

150. The method of analysing the more common gases which have to be dealt with in gasworks by means of Hempel's apparatus, will now be described. The following is a list of the same:—Oxygen, hydrogen, carbonic oxide, carbonic anhydride, marsh gas, olefiant gas.

### *Oxygen.*

151. This gas is but slightly soluble in water, and is determined either by combustion with an excess of hydrogen, or by absorption.

In the combustion with hydrogen, $\frac{2}{3}$ of the volume burned consist of hydrogen, and $\frac{1}{3}$ of oxygen. The volume of oxygen present is therefore found by dividing by 3 the decrease in volume resulting from the combustion. When oxygen is mixed with combustible gases, it is necessary to determine it by absorption.

The best absorbents for oxygen are a strongly alkaline (potash) solution of pyrogallic acid, or phosphorus.

152. The solution of potassium pyrogallate is made by mixing together, either directly, or in the absorption pipette, 5 grams of pyrogallic acid dissolved in 15 c.c. of water, and 120 grams of KHO dissolved in 80 c.c. of water.

It is necessary to note that KHO purified with alcohol should not be used, since this preparation even after strong ignition may cause erroneous results in the analysis.

The absorptions should not be carried on at a temperature under 15° C., for it has been observed that the potassium pyrogallate used for absorption is very much less active at a temperature under 7° C.

According to Hempel, at a temperature of 15° C., or higher, the last trace of oxygen can be removed with certainty in the space of three minutes, by shaking with the solution of potassium pyrogallate, while at lower temperatures the absorption is not complete after six minutes.

153. In order to prepare phosphorus in the necessary stick form for use in oxygen determinations, it is melted under water, in a test-tube placed in a water-bath. Enough phosphorus is used to form a column about six cm. high. A slightly conical glass tube of two to three mm. internal diameter is then dipped into the molten phosphorus, the upper end of the tube is closed with the finger, and the tube is lifted out, and dipped immediately into a tall beaker full of water. A peculiar movement takes place in the phosphorus enclosed in the tube at the moment when it solidifies, and since the phosphorus undergoes a marked decrease of volume when it becomes solid, the stick usually falls out of the tube upon gentle tapping; if it sticks, it can easily be pushed out with a wire.

Phosphorus sticks, as thus prepared, are used in the absorption pipette, Fig. 55. The cylindrical part is filled as full as possible with the sticks, the remaining space being filled with distilled water. In order to make an absorption with this reagent, the gas is driven over into the pipette, thereby displacing the water, and coming into contact with the moist sticks of phosphorus. A bright glow is visible when the reaction proceeds normally; the phosphorus burns to phosphoric acid, phosphorous acid, &c., at the expense of the oxygen.

After three minutes at the longest the absorption is complete. The end of the absorption is sharply shown by the disappearance of the glow, when the pipette is in a dark room.

Since the different products resulting from the oxidation of phosphorus are all soluble in water, the surface of the sticks of phosphorus is kept fresh by the action of the con-ining water alone, if that be renewed from time to time. And, further, since these oxidation products, as solid and liquid substances, have a very small tension, no error is caused by the white cloud which may be present in the gas residue after the absorption. The phosphorus can be used for a very large number of analyses, if it is protected from the action of the light. To do this, the cylindrical part of the pipette is covered with a small box, or the whole pipette is covered, when not in use, by a box of wood or cardboard which is impervious to the light.

154. Unfortunately this neat and accurate method is not universally applicable; the following are the conditions under which it can be used. The percentage of oxygen in the gas must not be more than 50; and the gas must be free from ammonia, olefiant gas, and other hydrocarbons. Consequently in gaseous mixtures such as coal gas, which contain olefiant gas, the phosphorus method would not be applicable unless the hydrocarbons were first removed.

### *Hydrogen.*

155. Hydrogen can be determined with great accuracy by burning with oxygen. The oxygen may be used mixed with nitrogen (atmospheric air), or pure oxygen made in

retorts blown from a glass tube, as proposed by Bunsen, may be employed. These retorts are half filled with dried and powdered chlorate of potash, and the end of the delivery tube is heated and bent in an upward direction. On heating the bulb of this improvised retort, the air is first driven out by a rapid evolution of oxygen, and the gas is then led directly into the endiometer, care being taken that the volume of oxygen does not amount to more than three or four times that of the hydrogen to be determined.

The quantity of hydrogen present is two-thirds of the volume disappearing in the combustion. If the mixture contains absorbable constituents also, these are first absorbed, and the residual gas is then used for the analysis.

156. When nitrogen is present, a considerable error may be caused by not avoiding, in the combustion, the temperature at which nitric acid is formed. It is consequently always necessary to calculate, after the experiment, the proportion of nitrogen to the oxyhydrogen gas burned. If this was less than six to one, the analysis must be repeated, with the addition of so much air that this proportion, or a still greater amount of nitrogen will be present.

If, on the other hand, the proportion of hydrogen to incombustible gas is very small, such an amount of electrolytic oxyhydrogen gas is added that complete combustion will result. The oxyhydrogen gas disappears completely on the combustion, and hence need not be exactly measured.

157. An accurately measured amount of pure hydrogen, mixed with an excess of air, may be used instead of the oxyhydrogen gas. The contraction resulting from the hydrogen added must then be allowed for. The combustion is made either in the explosion pipette, Fig. 58, by ignition with an electric spark, or in a glass tube filled with palladium black, or palladium sponge. The advantage of the combusttion with palladium is, that in a mixture of hydrogen, marsh gas, and nitrogen, the hydrogen alone may be burned; this is known as fractional combustion.

158. The arrangement of the apparatus for carrying out the reaction (fractional combustion) is shown in Fig. 59. The gas burette *A* and the gas pipette *B* are joined together by means of the capillary tubes *E*, and the tube *H*. This tube *H* is of about four mm. internal diameter and 20 cm. total length, and it contains four grams of palladium sponge. The gas pipette upon the stand *G* is filled with water, and its only use is so as to render it possible to repeatedly pass the gas through the palladium tube.

159. To determine the amount of hydrogen present in a mixture of hydrogen, nitrogen and marsh gas, from which, so far as possible, the absorbable constituents have already been removed, measure the gas in the burette, join it in the manner described to the pipette *B*, which is filled with water nearly to *i*, place the tube *H* in a large beaker containing warm water of from 90° to 100° C., and, after opening the pinch-cock *d*, drive the gas three times backward and forward through the palladium, by raising and lowering

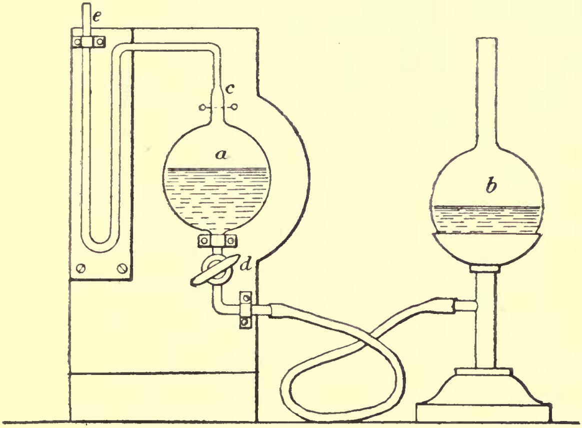

Fig. 58.

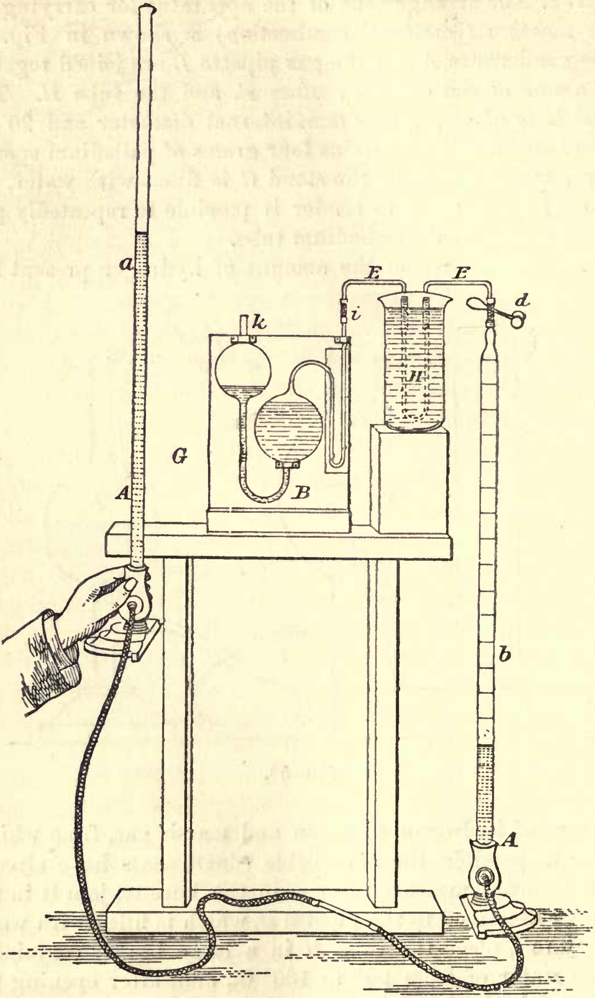

Fig. 59.

the tube *a*. Then replace the hot water with water of the temperature of the room, and lead the gas residue twice backwards and forwards through the tube, in order to completely cool the gas. It is in this manner possible to absorb with certainty every particle of hydrogen. Upon drawing the gas so far back into the measuring tube that the water in the pipette again stands near *i*, the difference between the two measurements made before and after the absorption, corresponds to the hydrogen plus the amount of oxygen in the air enclosed in the U-tube, when the apparatus was put together. This air volume, and therewith its oxygen contents, may be determined with sufficient exactness, once for all, by closing, with a piece of rubber tubing and glass rod, one side of the tube filled with palladium, cooling the tube to about 9° C. by placing it in cool water, and then, after connecting it by a capillary tube with a gas burette completely filled with water, warming it to 100° C. by placing it in boiling water. The expansion of the enclosed air volume corresponds to a difference of temperature of 91°, i.e., to a third of the enclosed volume of gas.

160. The palladium is regenerated after the reaction by first leading air over it, whereby it becomes quite hot, removing any drops of moisture which may collect, so that the palladium may easily be shaken out of the tube in the form of a dry powder, and then superficially oxidising the metal by heating it on the lid of a platinum crucible.

161. Before using the palladium, it should be heated, in portions of about one gram at a time, nearly to redness upon the cover of a platinum crucible, so that it is covered with a larger quantity of palladium oxide than would be formed by merely leading air over it.

The warm water in which the tube stands serves in the beginning to give the gases the temperature necessary to start the combustion, and at a later stage it prevents the temperature inside the palladium tube being raised too high by the reaction.

### *Carbon Monoxide, or Carbonic Oxide* (CO).

162. For absorbing CO, either an ammoniacal, or a hydrochloric acid solution of cuprous chloride is used.

163. The ammoniacal solution of cuprous chloride is prepared by dissolving 10·3 grams of copper oxide in 100 to 200 c.c. of concentrated common hydrochloric acid, and then allowing the solution to stand in a flask of suitable size, filled as full as possible with copper wire or copper wire gauze, until the cupric chloride is reduced to cuprous chloride, and the solution is completely colourless. The clear hydrochloric acid solution thus prepared is poured into a large glass beaker containing 1½ to 2 litres of water, in order to precipitate the cuprous chloride formed. After the precipitate has settled, the dilute HCl is poured off as completely as possible. The cuprous chloride is then washed into a 250 c.c. flask with about 100 to 150 c.c. of distilled water, and ammonia is led into the solution, which is still slightly acid, until the liquid assumes a pale blue colour. Since the tension of very concentrated ammonia solutions renders the absorption difficult, no more ammonia than is necessary should be added. While the ammonia is being led in, it is advisable to protect the contents of the flask from the oxidising influence of the air. This may be done by fitting the flask containing the cuprous chloride to be dissolved with a cork pierced with two holes, through one opening of which passes the delivery tube from the ammonia flask, while through the other hole is inserted a bent glass tube which dips into a little mercury. If a flask with a funnel tube is used for the evolution of $NH_3$, hydrogen may first be led through this tube, and the apparatus be thus completely freed from air. For the evolution of ammonia, about 200 c.c. of a concentrated ammonia solution of 0·9 sp. gr. is used.

The solution of cuprous chloride thus prepared is diluted with water to 200 c.c., and since the HCl is not completely washed out, there is, of course, some ammonium chloride present. 100 c.c. of this solution contain 7·3 grams of cuprous chloride; it is impracticable to use more dilute solutions of cuprous chloride.

164. For the preparation of the hydrochloric acid solution of cuprous chloride, 86 grams of copper scale are mixed with 17 grams of copper powder, prepared by reducing copper oxide with hydrogen, and this mixture is slowly brought into 1086 grams of hydrochloric acid of 1·124 sp. gr.; while the mixture is being added the vessel containing the HCl should be constantly shaken. A spiral of copper wire reaching from the bottom to the neck of the bottle is then placed in the solution, and the bottle is closed with an india-rubber cork. The solution is dark at first, but it becomes completely colourless on standing. On contact with the air it assumes a dark brown colour, due to the formation of some cupric chloride. In the analysis of a mixture containing a number of gases, if CO and H are present, the latter being determined by combustion with palladium after the elimination of CO, it will be necessary to use the ammoniacal cuprous chloride. If the amount of CO alone is to be ascertained the hydrochloric acid solution may be used. These cuprous chloride solutions are used in the double pipette, Fig. 56.

Solutions of cuprous chloride have not much tension, so that this may be disregarded in analyses which are not required to be strictly accurate. In exact determinations, however, the gases which have been in contact with the reagent must be freed from the gaseous HCl or from the $NH_3$. This can be effected in the burette itself, or in a pipette filled with distilled water.

## *Carbon Dioxide* ($CO_2$).

165. To absorb $CO_2$ a solution of caustic potash is used, consisting of one part of commercial caustic potash in two parts of water.

This solution is put into the simple pipette for solid and liquid reagents, Fig. 55, the cylindrical part being first closely filled with very short rolls of iron wire gauze. The gauze has a mesh of 1 to 2 mm. and the rolls are from 1 to 2 cm. long and about 5 mm. thick. When the percentage of $CO_2$ is not too high, it can be completely absorbed by simply passing the gas once into the pipette.

### *Ethylene* ($C_2H_4$).

166. Is either absorbed by fuming sulphuric acid, or by bromine water.

It is advisable to use sulphuric acid so concentrated that when the temperature is slightly lowered, crystals of pyrosulphuric acid will separate. The acid is used in a simple pipette provided with the three bulbs, Fig. 60. The small bulb is filled at the time of blowing with glass beads, which serve to give to the sulphuric acid the largest possible surface. With this arrangement, the complete absorption of the heavy hydrocarbons, and of ethylene in particular, is effected by passing the gas into the pipette once only. In this reaction some $SO_2$ is usually formed; and, further, the vapour of fuming sulphuric acid has a very high tension, so that the gas residue, before being measured, must be freed from the acid vapours in the caustic potash pipette, passing the gas once into the latter pipette being sufficient.

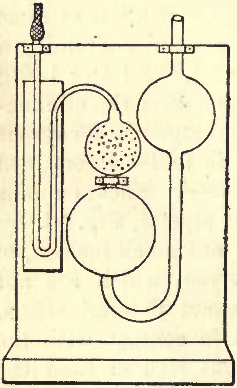

FIG. 60.

167. To avoid the rubber connections between the pipette and burette being attacked by the fuming sulphuric acid, the apparatus is put together in such a manner

that the acid does not quite fill the capillary tube of the pipette, and the connecting capillary is allowed to remain empty; the short rubber tube of the burette is also freed from liquid by means of a narrow tipped suction pipette, any reagent remaining in the rubber tube being first washed out by water with the same pipette.

If care be taken that the sulphuric acid is stopped, after the absorption, at the same point in the capillary at which it stood when the burette and pipette were first put together, then the small volume of air contained in the empty capillary tubes in the beginning causes, of course, no error in the determination of the heavy hydrocarbons or other gases with the exception of nitrogen. In the nitrogen determination allowance may be made for this air volume, but as each c.c. of the empty capillary corresponds to only 0·008 c.c., this value is below the limit of the usual unavoidable experimental errors.

After the absorption, the rubber tube is taken off from the pipette, and the capillary and the larger tube are closed air-tight by small pieces of glass rod, which are pushed over narrow rubber rings placed upon the glass tubes.

168. Bromine is also a good absorbent for ethylene. It is used in a similar pipette to the one just described. It is not necessary to completely fill the pipette with bromine; it will be quite sufficient if a few c.c. of bromine lie under water in the pipette. There is thus formed a saturated solution of bromine in water which absorbs the ethylene.

### *Marsh Gas or Methane* ($CH_4$).

169. There is no absorbent for marsh gas, which is always determined by combustion. One volume of methane unites with two volumes of oxygen, and one volume of $CO_2$ is formed.

To avoid the burning of nitrogen in the explosion, 100 volumes of incombustible gas are taken for from 25 to 37 volumes of the mixture of methane and oxygen (Bunsen).

### *Nitrogen.*

170. This gas is but slightly soluble in water.

No method of directly determining nitrogen is known. The residue of gas mixtures which cannot be directly determined is hence calculated as nitrogen. It follows from this, that all the errors of the preceding determinations fall upon the nitrogen, and the more complicated the gas mixture the more inexact are the results.

171. Hempel's apparatus may be used for determining the following.

(1) *Estimation of* $CO_2$ *in Air or in Furnace Gases.*—A simple absorption pipette, Fig. 54, is employed, containing a solution of KHO of sp. gr. 1·20.

(2) *Estimation of Oxygen in Atmospheric Air.*—A compound absorption pipette, Fig. 56, filled with a strong solution of potassium pyrogallate is employed.

(3) *Estimation of* $CO_2$, $O_2$, *and* $N_2$ *in a Furnace Gas.*—The gases are absorbed in the following order by the reagent mentioned.

(*a*) $CO_2$ by KHO solution.

(*b*) $O_2$ by alkaline pyrogallate solution.

(*c*) Measure the residual nitrogen.

(4) *Estimation of* $CO_2$, $O_2$, CO, *and* $N_2$ *in a Furnace Gas.*—Absorb the gases in the following order by the reagents mentioned.

(*a*) $CO_2$ by KHO solution.

(*b*) $O_2$ by alkaline pyrogallate solution.

(*c*) CO by a saturated solution of cuprous chloride in hydrochloric acid.

(*d*) Measure the residual nitrogen.

The following is a description of the method of making an analysis of coal gas by means of Hempel's apparatus.

172. A quantity of water for use in the measuring tube is first saturated with the gas, by taking a flask half full of

water, leading a rapid stream of gas through the liquid, and vigorously shaking the flask.

The hydrocarbon vapours are first absorbed with alcohol, then $CO_2$ with KHO, then the heavy hydrocarbons with fuming sulphuric acid, then oxygen with phosphorus, and lastly CO with ammoniacal cuprous chloride solution. The residue, which consists of marsh gas, hydrogen, and nitrogen, is measured, and is then led back into the cuprous chloride pipette, and a portion is taken for the explosion analysis. With ordinary illuminating gas 12 c.c. of the residue suffice for the explosion.

These 12 c.c. are measured off in the gas burette and enough air is drawn in to bring the mixture to about 100 c.c. In all these measurements, the running down of the liquid must be carefully waited for, because the amount of gas taken is so small, that any errors that may be made are greatly multiplied. The gas mixture is now burned in the explosion pipette. The gas is then transferred to the measuring tube and the total contraction is measured. Then the $CO_2$ is absorbed with KHO, and finally the oxygen in excess is absorbed with phosphorus. The last determination is made merely to be sure that a sufficient excess of oxygen was present in the combustion.

173. The following is an illustration. 100 c.c. of illuminating gas measured off. The hydrocarbon vapours are first absorbed in the following manner. Two explosion pipettes, Fig. 58, are used, containing 1 c.c. of alcohol and water respectively. These explosion pipettes are first filled completely with mercury, and the alcohol and water are run in at the capillary tube from burettes. Both liquids must be saturated with the illuminating gas before being used for the absorption. To do this about 50 c.c. of gas is passed into each pipette, and the pipettes are shaken for several minutes. The gas is then driven out and the pipettes are ready for use.

174. The measuring tube containing the 100 c.c. of gas is joined, by a fine-bore capillary tube, to the pipette containing

the 1 c.c. of alcohol, the gas is driven over, the pipette is closed by a pinch-cock and disconnected, and shaken for three minutes. The gas is then drawn back into the burette, and passed into the pipette containing the 1 c.c. of water, and is there shaken for three minutes. The gas is then drawn back into the measuring tube and measured $= 0 \cdot 7$ c.c. or per cent. hydrocarbon vapours. Passed into KHO pipette and drawn directly back into the measuring tube, measured after three minutes (time allowed for the running down). Measurement gave $4 \cdot 1$ c.c.; hence there was present $4 \cdot 1 - 0 \cdot 7 = 3 \cdot 4$ c.c. or per cent. $CO_2$.

175. Burette now connected by means of a dry piece of rubber tube and a dry capillary with the pipette containing fuming sulphuric acid. Gas driven over and drawn back at once into the measuring tube. Gas now passed again into KHO pipette, and after being drawn back into the measuring tube and allowed to stand three minutes again measured. The measurement gave $8 \cdot 4$ c.c.; hence there were $8 \cdot 4 - 4 \cdot 1 = 4 \cdot 3$ c.c., or $4 \cdot 3$ per cent. of heavy hydrocarbons present.

176. The gas now passed into phosphorus pipette, Fig. 55, and allowed to stand for three minutes, then drawn back into burette and measured at the end of three minutes. Reading gave $8 \cdot 4$ c.c., hence no oxygen was present.

177. The gas was then passed into the pipette containing ammoniacal cuprous chloride, Fig. 56, which had been repeatedly used, and was shaken therein for two minutes. It was then drawn back into the burette and transferred at once to a second pipette containing ammoniacal cuprous chloride which had been used but little, and it was here shaken for three minutes. Drawn back into the burette and measured after three minutes; the reading was 18 c.c. hence there was 18 c.c. $- 8 \cdot 4 = 9 \cdot 6$ c.c. or per cent. of CO present.

178. The remaining 82 c.c. of gas were then passed back into the cuprous chloride pipette, and the pipette was closed with an ordinary pinch-cock.

The water in the burette is poured out, the burette washed with hydrochloric acid and then with distilled water, and then filled with water which is saturated not with illuminating gas but with air. 12 to 15 c.c. of the gas residue are now measured off into the burette. In this case 13·2 c.c. were taken.

179. So much air is then drawn in that the total volume of the gas residue taken and the air amounts to about 100 c.c. In this case it was 99·6 c.c.

This mixture is now brought into an explosion pipette, Fig. 58, filled with mercury, care being taken that the capillary remains full of water. The rubber connecting piece is closed by a strong pinch-cock, and a piece of glass rod is slipped into the end of the india-rubber tube. The pipette is then vigorously shaken, the glass stop-cock is closed, the pipette is connected with the poles of an induction coil and the mixture is exploded. The glass stop-cock is at once opened and the remaining gas is transferred without delay to the measuring tube, and after three minutes measured. The result here was 78 c.c.

The total contraction was therefore $99\cdot6 - 78 = 21\cdot6$ c.c.

180. The gas remaining from the combustion is now passed into the KHO pipette, drawn directly back into the burette, and after three minutes measured. The reading was 73·2 c.c. Hence by the combustion $78 - 73\cdot2 = 4\cdot8$ c.c. of $CO_2$ were formed.

181. Although this gave all the data necessary for the calculation of the analysis, the remaining gas was nevertheless passed into the phosphorus pipette in order to be sure that an excess of oxygen was present in the combustion, or in other words that the gas was completely burned. The measurement gave 70·2 c.c.

Hence there were $73\cdot2 - 70\cdot2 = 3$ c.c. of oxygen in excess.

182. In the combustion of the marsh gas its own volume of $CO_2$ is formed, so that in the 13·2 c.c. of the gas residue taken for the explosion there were 4·8 c.c. of marsh gas.

The marsh gas in the total gas residue of 82 c.c. is found by the proportion—

$$13{\cdot}2 : 82 :: 4{\cdot}8 : x.$$

$$x = 29{\cdot}8 \text{ per cent. marsh.}$$

Since marsh gas in burning unites with twice its volume of oxygen, the contraction which has resulted from the combustion of the hydrogen is found by subtracting twice the volume of the $CO_2$ found from the total contraction.

$21{\cdot}6 - (2 \times 4{\cdot}8) = 12$ c.c. contraction due to the burning of hydrogen.

183. One volume of hydrogen unites in burning with one-half its volume of oxygen; hence the volume of the hydrogen is found by multiplying $12 \times \frac{2}{3}$. Thus the 13·2 c.c. of the gas residue taken for the explosion contained 8 c.c. of hydrogen. The total amount of hydrogen is given by the proportion—

$$13{\cdot}2 : 82 :: 8 : x.$$

$$x = 49{\cdot}6 \text{ per cent. hydrogen.}$$

184. The nitrogen is found by subtracting the sum of all the other constituents from 100. This gives 2·6 per cent.

Hence the illuminating gas contained—

| | | |
|---|---|---|
| 0·7 | per cent. | hydrocarbon vapours |
| 3·4 | „ | $CO_2$ |
| 4·3 | „ | heavy hydrocarbons |
| 0·0 | „ | oxygen |
| 9·6 | „ | CO |
| 29·8 | „ | $CH_4$ |
| 49·6 | „ | $H_2$ |
| 2·6 | „ | $N_2$ |
| 100·0 | | |

185. The determination of hydrogen may be made more exactly by absorption with palladium. The following analysis is given to illustrate the calculation of the analysis when the hydrogen is fractionally burned.

The direct absorption gave—

0·6 per cent. hydrocarbon vapours
3·4 „ $CO_2$
4·4 „ $C_2H_4$
0·3 „ Oxygen
10·1 „ CO

186. The residue of hydrogen, methane and nitrogen, amounted to 81·2 c.c. This was transferred to a pipette and 40·5 c.c. were measured off in a burette for the fractional combustion of the hydrogen. To this was added air (in this case 58·7 c.c.), and the mixture was passed into a pipette filled with water. Then more air was measured off in the burette and transferred to the pipette, so that the total amount of air added would, without doubt, be sufficient for the combustion of the hydrogen.

In this second measurement 16·1 c.c. of air were taken, so that the total amount of gas taken for the fractional combustion was 40·5 + 58·7 + 16·1 = 115·3 c.c. The gases were vigorously shaken in the pipette to thoroughly mix them, and were then fractionally burned by leading them over 0·5 gram of palladium black. [Palladium black is made by reducing palladious chloride with alcohol in a strongly alkaline solution, the same method being used in preparing platinum black with platinum chloride. The palladium black is placed in a small U-tube, Fig. 59, plunged into water at a temperature of 100° C., and the gas and air is passed slowly through the tube two or three times. The tube at the time must be connected with an ordinary absorption pipette filled with water, or else with the KHO pipette, which in this case, of course, simply acts as a kind of receiver.] The volume after the combustion was 81 c.c., hence the contraction was—

$$115{\cdot}3 - 81 = 34{\cdot}3 \text{ c.c.,}$$

corresponding to 22·9 c.c. of hydrogen, the total amount of hydrogen being found by the proportion—

$$\text{As} \quad 40{\cdot}5 : 81{\cdot}2 :: 22{\cdot}9 : x.$$

$$x = 45{\cdot}9 \text{ per cent. hydrogen.}$$

187. To determine the marsh gas, 19·9 c.c. of the residue of hydrogen, marsh gas and nitrogen were taken, and, together with 110 c.c. of air, were transferred to the explosion pipette. The gases were well mixed and were then exploded, freed from $CO_2$ in the KHO pipette, and measured.

There remained 90·5 c.c. The contraction was—

$$110 + 19\cdot 9 \quad 90\cdot 5 = 39\cdot 4 \text{ c.c.}$$

188. From the determination of the hydrogen by the fractional combustion, we know that in 19 c.c. of the residue the hydrogen would cause a contraction of 16·9 c.c. ($40\cdot 5 : 19\cdot 9 :: 34\cdot 3 : x.\quad x = 16\cdot 9$), hence the contraction due to the methane is equal to $39\cdot 4 - 16\cdot 9 = 22\cdot 5$ c.c., and the volume of the methane itself is 7·5 c.c.

The percentage of methane is—

$$19\cdot 9 : 81\cdot 2 :: 7\cdot 5 : x.$$

$$x = 30\cdot 6 \text{ per cent. methane.}$$

189. The nitrogen, determined by difference as before, is 4·7 per cent., so that the composition of the gas was as follows:—

| | | |
|---|---|---|
| 0·6 | per cent. | hydrocarbon vapours |
| 3·4 | „ | $CO_2$ |
| 4·4 | „ | $C_2H_4$ |
| 0·3 | „ | $O_2$ |
| 10·1 | „ | CO |
| 45·9 | „ | $H_2$ |
| 30·6 | „ | $CH_4$ |
| 4·7 | „ | $N_2$ |
| 100·0 | | |

## GAS ANALYSIS BY MEANS OF BÜNTE'S APPARATUS.

### *Analysis of Furnace Gases.*

190. These may be conveniently determined by means of the apparatus devised by Dr. Bünte, late of the gasworks, Munich. The following is a description of the apparatus and method of using it:—

A and B, Fig. 61, are two burettes fitted with three-way cocks, each graduated in fifths of a c.c., and capable of holding 110 c.c.; T, a one-gallon tubulated bottle, serving as a water reservoir, and F an aspirator. The burette is filled by opening the stop-cocks, *b* and *a*, and allowing water from the bottle, T, to enter the burette until it nearly fills the funnel, *t*. The stop-cock should then be closed and the india-rubber tube detached from the bottom of the burette. The longitudinal bore of the stop-cock, *a*, is now connected with the tube supplying the gas to be examined, and the gas aspirated by running the water out of the burette by means of the stop-cock, *b*. Rather more than 100 c.c., say, 105 c.c., of gas should be allowed to enter the burette, and the exact adjustment to the zero mark then made as follows:—By means of the bottle, T, sufficient water should be forced into the burette to compress the gas to about 95 c.c.; then *b* is closed, the bottle, T, is detached, and by cautiously turning the tap, *b*, the water is run out again exactly to the zero mark. The gas is still under a plus pressure, and now that pressure has to be established at which every reading off has to take place with this apparatus. For this purpose the funnel, *t*, should be filled with water up to the mark, when, on opening the tap, *a*, for an instant the excess of gas will escape through the water. The burette now contains exactly 100 c.c. of gas at the pressure of the atmosphere, plus the pressure of the column of water standing in the funnel, *t*.

### *Determination of Carbonic Acid.*

191. The tube, *r*, of the aspirator, F, is connected to the bottom of the burette, suction applied at S, stop-cock, *b*, opened, and as much water as can be aspirated allowed to run out. The stop-cock should then be closed, the tube removed, and the end of the burette dipped into a strong solution of caustic potash. On opening the stop-cock, *b*, a quantity of

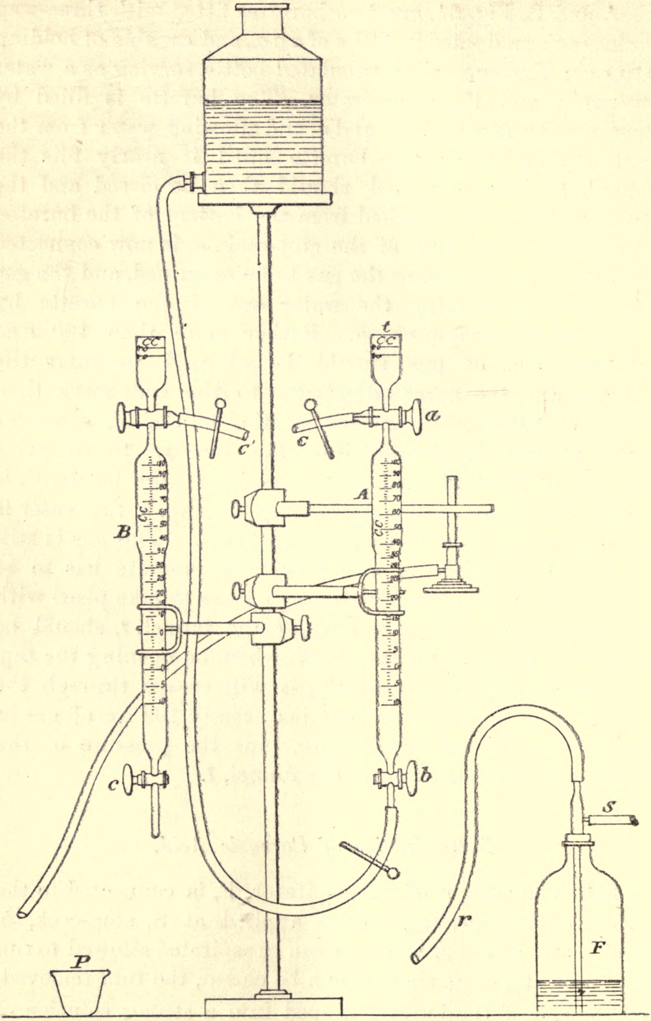

FIG. 61.

the solution will enter the burette. (Care should be taken that the bottom of the burette is always immersed in the solution, or air would enter and so spoil the experiment.) When the solution has entered the burette, the stop-cock, *b*, should be closed, the burette taken from its support, and the hand of the operator being firmly placed on *t*, the contents of the burette should be vigorously shaken up with the solution, when, if the solution of potash employed has been strong enough, it will be found that the whole of the $CO_2$ has been absorbed; therefore, on opening the stop-cock, *a*, and allowing water to flow down until the normal atmospheric pressure is reached (which is shown by the water ceasing to flow), and filling the funnel up to mark, on reading off the amount absorbed by the caustic potash = percentage of $CO_2$.

### *Determination of Oxygen.*

192. The caustic potash solution should be drawn off by means of the aspirator, and an alkaline solution of pyrogallic acid applied in the same manner as in the estimation of $CO_2$; if oxygen is present, the solution immediately turns black. The gas should be well shaken up with the solution, but in this case the absorbing liquid is not so active as the potash. It will thus be necessary to see if the gas will take up any more of the solution. Therefore, having replaced the burette in its support, the solution should again be applied to the burette, whereupon some of the liquid will enter in the place of the absorbed gas. The burette should then be well shaken, and the solution applied again, when, if the liquid remains at the same level as it previously stood at, the absorption is complete; otherwise, the process must be continued until there is no further absorption. After the reduction of the contents of the burette to normal pressure, the difference between the volume now observed and that at the commencement, after the abstraction of the $CO_2$, will give the percentage of O.

### *Determination of Carbonic Oxide.*

193. If oxygen has been proved to be present, carbonic oxide will most likely be absent, unless the gases have been brought together at a temperature insufficient to promote their combination. Carbonic oxide and hydrogen are most conveniently estimated by combustion, as under.

The burette should first be cleared from the solution of alkaline pyrogallate, by opening the cocks, *a* and *b*, and allowing a stream of water to run through the burette until clean, taking the precaution to always keep the funnel, *t*, sealed. It is now necessary to mix the gases with an excess of air, but as the burette would not hold sufficient to combine with the whole of the gases generally present, it is requisite to expel a portion and work on, say, half the volume. For instance, if 100 c.c. were originally taken, and there were found 10 per cent of $CO_2$, on driving out gas until it measured 45 c.c. we would get the equivalent of half the volume originally present. The air is admitted by placing the cock, *a*, in communication with the burette, opening the pinch-cock at *c*, and allowing water to flow out at *b*. This should be continued until the water level is two or three divisions below zero, when the cock, *b*, and the pinch-cock should be closed, the contents of the burette brought to the normal pressure, and the reading of the burette taken, the contents of the burette having been previously well shaken up so as to mix the gases with the air. Connection should then be made between the burettes, A and B, uniting the two at the india-rubber tubes, C and C′, by means of a piece of glass combustion tubing containing a small coil of palladium wire. The burette, B, should previously have been filled with water in the usual manner. The palladium wire in the combustion tube should be brought to a red heat by means of a Bunsen burner, and the gas in the burette is caused to pass from A over the heated wire into B by opening *b* and *c*, connecting the water-supply, T, with bottom of burette, A. When all the gas from A has passed over (shown by the burette being full of water) the operation is reversed, the gas being again collected in A. It is then allowed to stand until the normal temperature is attained, adjusted for pressure, and the volume then read off, and this reading kept for reference. A solution of caustic potash should then be applied in the manner described for the estimation of $CO_2$, and the diminution in volume also noted.

194. In order to work out the calculations, it will be necessary to notice what action takes place during the combustion. The gases to be dealt with are hydrogen, carbonic oxide and nitrogen, mixed with an excess of air; by passing over the red hot palladium wire, the oxygen of the air combines with the carbonic oxide CO, to form carbonic anhydride, $CO_2$; ($CO + O = CO_2$) and with the hydrogen to form water, $H_2O$; ($H_2 + O = H_2O$) the nitrogen of course not being affected. Now, supposing that after igniting the gases and treating the residue with caustic potash, a diminution in volume of 10 c.c. was observed, this would be equal to 10 c.c. of $CO_2$, for each volume of CO produces an equal volume of $CO_2$, and, as only half the original volume taken was treated, on multiplying the result by two, the true percentage of $CO_2$ will be obtained. Supposing, also, that after ignition, but before treatment with KHO, there was a diminution in the volume of 10 c.c., this would be due partly to the combination of the hydrogen with the oxygen, and partly to the combination of the carbonic oxide with the oxygen. The CO would require half its volume, or 5 c.c. of oxygen; therefore, on deducting 5 c.c. from the 10 c c. due to combustion, we get 5 c.c. as a result of the combination of the hydrogen with the oxygen. Hydrogen combines with oxygen to form water, in the proportion of two volumes to one; consequently, on multiplying 5 by $\frac{2}{3}$ we get 3·3 c.c. as the number of c.c. of hydrogen, and this multiplied by 2, gives 6·6 as the percentage of hydrogen. Nitrogen is calculated by difference.

195. In order to effect the estimation of CO by absorption, it is necessary to remove every trace of the pyrogallate solution by the use of the funnel and the suction bottle. This done, a concentrated solution of cuprous chloride in HCl is applied to the bottom of the burette in the usual manner. When absorption is complete the $Cu_2Cl_2$ is drawn off, the tube washed, and treated with a solution of KHO, for the purpose of absorbing any HCl vapour which may have been liberated in the reaction. After bringing to correct pressure, the reading shows the percentage of CO.

196. The gas may be conveniently taken off by tightly inserting a wrought iron tube, or preferably an inner platinum tube encased by an outer tube of wrought iron, into the furnace or flue, and then aspirating into a suitable vessel, taking every precaution to prevent any air mixing with the gases, by aspirating a sufficient quantity. The sample may then be transferred to the laboratory for analysis.

# APPENDIX.

## *GAS REFEREES' INSTRUCTIONS.*

### As to the Times and Mode of Testing for Purity.

THE testings for purity shall extend over twenty hours of each day, and shall be made upon ten cubic feet of gas, which shall be tested successively for each of the following impurities :—

### I. *Sulphuretted Hydrogen.*

The gas shall be passed, as it leaves the service-pipe, through an apparatus in which are suspended slips of bibulous paper, impregnated with basic acetate of lead.

The test-paper from which these slips are cut is to be prepared from time to time by moistening sheets of bibulous paper with a solution of 1 part of sugar of lead in 8 or 9 parts of water, and holding each sheet while still damp over the surface of a strong solution of ammonia for a few moments. As the paper dries all free ammonia escapes.

If any discoloration of the slip of test-paper is found to have taken place, this is to be held conclusive as to the presence of sulphuretted hydrogen in the gas. Fresh test-slips are to be placed in the apparatus every day.

In the event of any impurity being discovered, one of the test-slips shall be placed in a stoppered bottle, and kept in the dark at the testing place ; the remaining slips shall be forwarded with the daily report.

### II. *Ammonia.*

The gas which has been tested for sulphuretted hydrogen shall pass next through an apparatus consisting of a glass cylinder filled with glass beads, which have been moistened with a measured quantity of standard sulphuric acid. A set of burettes, properly graduated, is provided.

The maximum amount of ammonia allowed is 4 grains per 100 cubic feet of gas; and the testings shall be made so as to show the exact amount of ammonia in the gas.

Two test-solutions are to be used—one consisting of dilute sulphuric acid, of such strength that 25 measures (septems) will neutralise 1 grain of ammonia; the other, a weak solution of ammonia, 100 measures of which contain 1 grain of ammonia.

The correctness of the result to be obtained depends upon the fulfilment of two conditions:—

1. The preparation of test-solutions having the proper strength.
2. The accurate performance of the operation of testing.

To prepare the test-solutions the following processes may be used by the gas-examiner:—

Measure a gallon of distilled water into a clean earthenware jar or other suitable vessel. Add to this 94 septems of pure concentrated sulphuric acid, and mix thoroughly. Take exactly 50 septems of the liquid and precipitate it with barium chloride in the manner prescribed for the sulphur test. The weight of barium sulphate which the test acid should yield is 13·8 grains. The weight obtained with the dilute acid prepared as above will be somewhat greater, unless the sulphuric acid used had a specific gravity below 1·84. Add now to the diluted acid a measured quantity of water, which is to be found by subtracting 13·8 from the weight of barium sulphate obtained in the experiment, and multiplying the difference by 726. The resulting number is the number of septems of water to be added.

If these operations have been accurately performed, a second precipitation and weighing of the barium sulphate obtainable from 50 septems of the test acid will give nearly the correct number of 13·8 grains. If the weight exceeds 13·9 grains, or falls below 13·7 grains, more water or sulphuric acid must be added, and fresh trials made, until the weight falls within these limits. The test acid thus prepared should be transferred at once to stoppered bottles which have been well drained, and are duly labelled.

To prepare the standard solution of ammonia, measure out as before a gallon of distilled water, and mix with it 50 septems of strong solution of ammonia (sp. gr. 0·88). Try whether 100 septems of the test alkali thus prepared will neutralise 25 of the test acid, proceeding according to the directions given subsequently as to the mode of testing.

If the acid is just neutralised by the last few drops, the test alkali is of the required strength; but if not, small additional quantities of water or of strong ammonia solution, must be added, and fresh trials made, until the proper strength has been attained. The bottles in which the solution is stored should be filled nearly full, and well stoppered.

The mode of testing is as follows:—Take 50 septems of the test-acid (which is greatly in excess of any quantity of ammonia likely to be found in the gas), and pour it into the glass cylinder, so as to well wet the whole interior surface, and also the glass beads. Connect one terminal tube of the cylinder with the gas supply, and the other with the meter, and make the gas pass at the rate of about half a cubic foot per hour. Any ammonia that is in the gas will be arrested by the sulphuric acid, and a portion of the acid (varying with the quantity of ammonia in the gas) will be neutralised thereby. At the end of each period of testing, wash out the glass cylinder and its contents with distilled water, and collect the washings in a glass vessel. Transfer one-half of this liquid to a separate glass vessel, and add a quantity of a neutral solution of hæmatoxylin or litmus just sufficient to colour the liquid. Then pour into the burette 100 septems of the test alkali, and gradually drop this solution into the measured quantity of the washings collected, stirring constantly. As soon as the colour changes (indicating that the whole of the sulphuric acid has been neutralised), read off the quantity of liquid remaining in the burette. To find the number of grains of ammonia in 100 cubic feet of the gas, multiply the number of septems of test alkali remaining in the burette by 2, and move the decimal point one place to the left.

The remaining half of the liquid is to be preserved in a bottle, duly labelled, for a week.

### III. *Sulphur Compounds other than Sulphuretted Hydrogen.*

The gas which has been tested for sulphuretted hydrogen and ammonia shall pass next through a meter, by means of which the rate of flow can be adjusted to half a cubic foot per hour, and which is provided with a self-acting movement for shutting off the gas when ten cubic feet have passed.

The testing shall be made in a room where no gas is burnt other than that which is being tested for sulphur and ammonia.

The apparatus to be employed is represented in Fig. 62, and is of the following description:—The gas is burnt in a small Bunsen burner with steatite top, which is mounted on a short cylindrical stand, perforated with holes for the admission of air, and having on its upper surface a deep circular channel to receive the wide end of a glass trumpet tube. On the top of the stand, between the narrow stem of the burner and the surrounding glass trumpet tube, are to be placed pieces of commercial sesqui-carbonate of ammonia, weighing in all about two ounces.

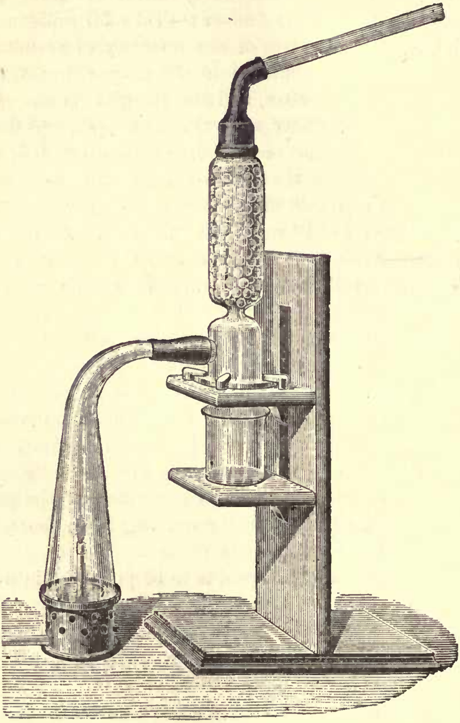

FIG. 62.

The products, both of the combustion of the gas and of the gradual volatilisation of the ammonia salt, go upwards through the trumpet tube into a vertical glass cylinder, packed with balls of glass, to break up the current and promote condensation. From the top of the cylinder there proceeds a long glass pipe or chimney, serving to effect some further condensation, as well as to regulate the draught, and afford an exit for the uncondensable gases. In the bottom of the cylinder is fixed a small glass tube, through which the liquid (formed during the testing) drops into a beaker placed beneath.

The following cautions are to be observed in selecting and setting up the apparatus :—

See that the inlet pipe fits gas-tight into the burner, and that the holes in the circular stand are clear. If the burner gives a luminous flame, remove the top-piece, and having hammered down gently the nozzle of soft metal, perforate it afresh, making as small a hole as will give passage to half a cubic foot of gas per hour at a convenient pressure.

See that the tubulure of the condenser has an internal diameter of not less than ¾ inch, and that its outside is smooth and of the same size as the small end of the trumpet tube.

See that the short piece of india-rubber pipe fits tightly both to the trumpet tube and to the tubulure of the condenser.

The small tube at the bottom of the condenser should have its lower end contracted, so that when in use it may be closed by a drop of water.

The india-rubber pipe at the lower end of the chimney tube should fit into, and not simply rest upon the mouth of the condenser, and the upper extremity of this tube may with advantage be given a downward curvature.

At the end of each period of testing, the cylinder and trumpet tube are to be well washed out with distilled water. Fresh pieces of sesqui-carbonate of ammonia are to be used each day.

The gas examiner shall then proceed as follows :—

The liquid in the beaker and the water used in washing out the apparatus shall be put into the same vessel, well mixed, and measured. One-half of the liquid so obtained is to be set aside, and preserved for a week, properly labelled, in case it should be desirable to verify the correctness of the testing.

The remaining half of the liquid is to be put into a flask, or beaker, covered with a large watch-glass—treated with hydrochloric acid, sufficient in quantity to leave an excess of acid in the solution—and then raised to the boiling point. An excess of a solution of barium chloride is now to be added, and the boiling continued for five minutes.

The vessel and its contents are to be allowed to stand till the barium sulphate settles at the bottom of the vessel, after which the clear liquid is to be as far as possible poured off through a paper filter. The remaining liquid and barium sulphate are then to be poured on to the filter, and the latter well washed with hot distilled water. [In order to ascertain whether every trace of barium chloride and ammonium chloride has been removed, a small quantity of the washings from the filter should be placed in a test-tube, and a drop of solution of silver nitrate added. Should the liquid, instead of remaining perfectly clear, become cloudy, the washing must be continued until, on repeating the test, no cloudiness is produced.] Dry the filter with its contents, and transfer it into a weighed platinum crucible. Heat the crucible over a lamp, increasing the temperature gradually, from the point at which the paper begins to char, up to bright redness. When no black particles remain, allow the crucible to cool; place it when nearly cold in a desiccator over strong sulphuric acid, and again weigh it. The difference between the first and second weighings of the crucible will give the number of grains of barium sulphate. Multiply this number by 11 and divide by 4; the result is the number of grains of sulphur in 100 cubic feet of the gas.

This number is to be corrected for the variations of temperature and atmospheric pressure in the manner indicated under the head of "Illuminating Power," with this difference, that the readings of the barometer and thermometer are to be taken for the day on which the testing commenced, and also the day on which it closed; and the mean of the two is to be used.

This correction may be made most simply, and with sufficient accuracy, in the following manner:—

When the tabular number is between 955–965, 966–975, 976–985, 986–995, increase the number of grains of sulphur by $\frac{4}{100}$ths, $\frac{3}{100}$ths, $\frac{2}{100}$ths, $\frac{1}{100}$th.

When the tabular number is between 996–1005, no correction need be made.

When the tabular number is between 1006–1015, 1016–1025, 1026–1035, diminish the number of grains of sulphur by $\frac{1}{100}$th, $\frac{2}{100}$ths, $\frac{3}{100}$ths.

*Example.*

| | |
|---|---|
| Grains of barium sulphate from 5 cubic feet of gas .. | 4·3 |
| Multiply by 11, and divide by 4 .. .. .. .. .. | 11 |
| | 4)47·3 |
| Grains of sulphur in 100 cubic feet of gas (uncorrected) | 11·82 |
| Add 11·8 × $\frac{2}{100}$ = .. .. .. .. .. .. .. | ·24 |
| Grains of sulphur in 100 cubic feet of gas (corrected) .. | 12·07 |

| | |
|---|---|
| Barometer (mean) .. .. .. .. .. | 29·4 |
| Thermometer (mean) .. .. .. .. | 58 |
| Tabular number .. .. .. .. .. | 985 |

Result .. .. .. 12·1 grs.

### As to the Maximum Amount of Impurity in each form with which the Gas shall be allowed to be charged.

*Sulphuretted Hydrogen.*—By the Acts of Parliament all gas supplied must be wholly free from this impurity.

*Ammonia.*—The maximum amount of this impurity shall be 4 grains per 100 cubic feet.

*Sulphur Compounds other than Sulphuretted Hydrogen.*—The maximum amount of sulphur with which gas shall be allowed to be charged shall be 17 * grains of sulphur in every 100 cubic feet of gas.

### Meters.

The meters used for measuring the gas consumed in making the various testings, having been certified by the Referees, shall, at periods of not less than seven days, be proved by the Gas Examiners by means of the Referees' Cubic Foot Measure—a description of which apparatus, with directions how to use it, is given below. Should a meter show any variation, water must be added or withdrawn until the meter is correct. Every testing place shall have the above-mentioned

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* The amount allowed in the winter is 22 grains.

apparatus, so that the Gas Examiner may employ it whenever he thinks necessary.

No meter other than a wet one shall be used in testing the gas under these instructions.

### Gas Referees' Cubic Foot Measure.

This instrument is a vessel of a cylindrical form with rounded ends, made of hardened tin about one quarter of an inch thick, fitted at each end with a narrow glass tube—the joints being made sound with india-rubber packing. The instrument stands in a vertical position firmly fixed to a strong plank.

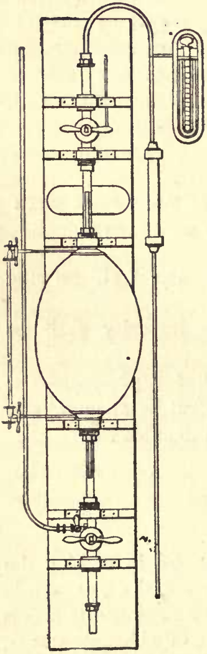

Fig. 63.

At the top of the instrument is a three-way cock, marked on the head of the key with a T, each arm of which shows the direction of a way through the plug; and attached to the side of this cock there is a small pipe, which serves to admit gas when the measure is to be charged. When the T is in its ordinary position communication is closed between the measure and the tube leading from it to the meters, but is open to the gas supply; but with the ⊣ in this position (i.e. with the stem pointing to the *opposite* side of the cock to that connected with the gas supply) the measure is open to the meters and shut to the gas.

At the bottom of the instrument there are three cocks, one large and two small ones. One of the small ones, when opened, admits water into the measure; the other small one is used for verifying the adjustment of the measure, by discharging the water into a standard measure. The large cock, when opened, allows the water to run off.

The cock at the top of the measure is continued by a tin pipe, which rises above the level of the water in the cistern, and then returns downwards, passing to the two meters.

Affixed round the glass tubes fitted to the upper and lower part of the measure there are narrow strips of paper, which indicate the exact measure of one cubic foot.

The instrument should be in communication with a tank of water in the same room.

To verify the meters employed in ascertaining the consumption of gas in the photometers, or those employed in the sulphur and ammonia tests, the mode of procedure is as follows:—

Starting with the bottle full of water, turn the three-way cock at the top of the instrument so as to place the measure in communication with the gas supply from the main, and open the large cock at the bottom of the apparatus. When the bottle is filled with gas to below the water-line, close the large cock, and turn the upper three-way cock so as to close the inlet for gas, while opening the way to the meters. Next turn the cock of the meter which is to be tested so as to cut off the ordinary gas supply and to place the meter in communication with the cubic foot measure, taking care that all the other meters are closed to the cubic foot measure. See that the tap is open which allows gas to pass from the meter through the governor to the burner. Then proceed gently to open the small lever cock and allow water to flow into the measure, sufficient to fill the lower glass tube up to the water-line. Note the exact position of the index hands. Now turn on the lever cock, watching the pressure-gauge attached to the measure, until gas is passing through the meter at about the normal rate of five cubic feet an hour. When the measure is nearly full, stand by it to check the rate of flow, and finally shut the water off as it reaches the upper water-line. Read the meter.

Should the meter have completed more than the prescribed number of revolutions when the measure is full, then some water must be removed from the meter; if the contrary is the case, then water must be added to the meter. The testing is then to be repeated until the meter is found to register correctly.

The dial of the photometer meters is divided into 50 divisions; and as each revolution indicates one-twelfth of a foot each division consequently represents the $\frac{1}{600}$th part of a cubic foot; and therefore 6 of these divisions represent $\frac{1}{100}$th part of a cubic foot.

The dial of the sulphur meters is divided into 100 parts; and as each complete revolution indicates one foot, each division consequently represents 1 per cent. of the volume sent through. For accurate measurement it is essential that the temperature of the gas in the measure and that in the meter should be the same. If the temperature of the water with which the measure has been filled is different from that of the meter, a sufficient time must be allowed to elapse after the measure has been filled with gas for the measure to have gained the temperature of the surrounding air. The measure and the meters must be so placed that the temperature of the air which surrounds them is the same.

The difference of about an ounce in the quantity of water in the meters when they are charged nearly to the water level will make them register 1 per cent. either fast or slow.

When the temperature of the cubic foot measure is higher than the temperature at the outlet of the meter, the meter (if correct) will register a smaller quantity than has actually passed to it from the measure; when the temperature of the measure is lower than that of the meter, the quantity registered ought to be greater. To find the volume which the meter ought to register when such a difference of temperature exists, divide the tabular number corresponding to the barometric pressure and the temperature of the cubic foot measure, by the tabular number corresponding to the barometric pressure and the temperature of the meter (see page 261).

## Table to facilitate the Correction of the Volume of Gas at different Temperatures and under different Atmospheric Pressures.

| Bar. | Ther. 40° | 42° | 44° | 46° | 48° | 50° | 52° | 54° | 56° | 58° | 60° | 62° | 64° | 66° | 68° | 70° | 72° | 74° | 76° | 78° | 80° | 82° | 84° |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 28·0 | ·979 | ·974 | ·970 | ·965 | ·960 | ·956 | ·951 | ·946 | ·942 | ·937 | ·932 | ·927 | ·922 | ·917 | ·912 | ·907 | ·902 | ·897 | ·892 | ·887 | ·881 | ·875 | ·870 |
| 28·1 | ·983 | ·978 | ·973 | ·969 | ·964 | ·959 | ·955 | ·951 | ·945 | ·941 | ·936 | ·930 | ·926 | ·921 | ·916 | ·911 | ·905 | ·900 | ·895 | ·890 | ·884 | ·879 | ·873 |
| 28·2 | ·986 | ·981 | ·977 | ·972 | ·967 | ·963 | ·958 | ·953 | ·949 | ·944 | ·939 | ·934 | ·929 | ·924 | ·919 | ·914 | ·909 | ·904 | ·898 | ·893 | ·887 | ·882 | ·876 |
| 28·3 | ·990 | ·985 | ·980 | ·976 | ·971 | ·966 | ·961 | ·957 | ·952 | ·947 | ·942 | ·937 | ·932 | ·928 | ·922 | ·917 | ·912 | ·907 | ·902 | ·896 | ·891 | ·885 | ·880 |
| 28·4 | ·993 | ·988 | ·984 | ·979 | ·974 | ·970 | ·965 | ·960 | ·955 | ·951 | ·946 | ·941 | ·936 | ·931 | ·926 | ·921 | ·915 | ·910 | ·905 | ·900 | ·894 | ·888 | ·883 |
| 28·5 | ·997 | ·992 | ·987 | ·983 | ·978 | ·973 | ·968 | ·964 | ·959 | ·954 | ·949 | ·944 | ·939 | ·934 | ·929 | ·924 | ·919 | ·914 | ·908 | ·903 | ·897 | ·892 | ·886 |
| 28·6 | 1·001 | ·995 | ·991 | ·986 | ·981 | ·977 | ·972 | ·967 | ·962 | ·958 | ·953 | ·947 | ·943 | ·938 | ·932 | ·927 | ·922 | ·917 | ·912 | ·906 | ·901 | ·895 | ·889 |
| 28·7 | 1·004 | ·999 | ·994 | ·990 | ·985 | ·980 | ·975 | ·970 | ·966 | ·961 | ·956 | ·951 | ·946 | ·941 | ·936 | ·931 | ·925 | ·920 | ·915 | ·909 | ·904 | ·898 | ·893 |
| 28·8 | 1·007 | 1·003 | ·998 | ·993 | ·988 | ·984 | ·979 | ·974 | ·969 | ·964 | ·959 | ·954 | ·949 | ·944 | ·939 | ·934 | ·929 | ·924 | ·918 | ·913 | ·907 | ·901 | ·896 |
| 28·9 | 1·011 | 1·006 | 1·001 | ·997 | ·992 | ·987 | ·982 | ·977 | ·973 | ·968 | ·963 | ·958 | ·953 | ·948 | ·942 | ·937 | ·932 | ·927 | ·921 | ·916 | ·910 | ·905 | ·899 |
| 29·0 | 1·014 | 1·010 | 1·005 | 1·000 | ·995 | ·990 | ·986 | ·981 | ·976 | ·971 | ·966 | ·961 | ·956 | ·951 | ·946 | ·941 | ·935 | ·930 | ·925 | ·919 | ·914 | ·908 | ·903 |
| 29·1 | 1·018 | 1·013 | 1·008 | 1·004 | ·999 | ·994 | ·989 | ·984 | ·979 | ·975 | ·969 | ·964 | ·959 | ·954 | ·949 | ·944 | ·939 | ·933 | ·928 | ·923 | ·917 | ·911 | ·906 |
| 29·2 | 1·021 | 1·017 | 1·012 | 1·007 | 1·002 | ·997 | ·992 | ·988 | ·982 | ·978 | ·973 | ·968 | ·963 | ·958 | ·952 | ·947 | ·942 | ·937 | ·931 | ·926 | ·920 | ·914 | ·909 |
| 29·3 | 1·025 | 1·020 | 1·015 | 1·011 | 1·006 | 1·001 | ·996 | ·991 | ·986 | ·981 | ·976 | ·971 | ·966 | ·961 | ·956 | ·950 | ·945 | ·940 | ·935 | ·929 | ·923 | ·918 | ·912 |
| 29·4 | 1·028 | 1·024 | 1·019 | 1·014 | 1·009 | 1·004 | ·999 | ·995 | ·990 | ·985 | ·980 | ·975 | ·969 | ·964 | ·959 | ·954 | ·949 | ·943 | ·938 | ·932 | ·927 | ·921 | ·915 |
| 29·5 | 1·032 | 1·027 | 1·022 | 1·018 | 1·013 | 1·008 | 1·003 | ·998 | ·993 | ·988 | ·983 | ·978 | ·973 | ·968 | ·962 | ·957 | ·952 | ·947 | ·941 | ·936 | ·930 | ·924 | ·919 |
| 29·6 | 1·036 | 1·031 | 1·026 | 1·021 | 1·016 | 1·011 | 1·006 | 1·001 | ·996 | ·992 | ·986 | ·981 | ·976 | ·971 | ·966 | ·960 | ·955 | ·950 | ·944 | ·939 | ·933 | ·927 | ·922 |
| 29·7 | 1·039 | 1·034 | 1·029 | 1·025 | 1·019 | 1·015 | 1·010 | 1·005 | 1·000 | ·995 | ·990 | ·985 | ·980 | ·974 | ·969 | ·964 | ·959 | ·953 | ·948 | ·942 | ·937 | ·931 | ·925 |
| 29·8 | 1·043 | 1·038 | 1·033 | 1·028 | 1·023 | 1·018 | 1.013 | 1·008 | 1·003 | ·998 | ·993 | ·988 | ·983 | ·978 | ·972 | ·967 | ·962 | ·957 | ·951 | ·946 | ·940 | ·934 | ·928 |
| 29·9 | 1·046 | 1·041 | 1·036 | 1·031 | 1·026 | 1·022 | 1·017 | 1·012 | 1·007 | 1·002 | ·997 | ·991 | ·986 | ·981 | ·976 | ·970 | ·965 | ·960 | ·954 | ·949 | ·943 | ·937 | ·932 |
| 30·0 | 1·050 | 1·045 | 1·040 | 1·035 | 1·030 | 1·025 | 1·020 | 1·015 | 1·010 | 1·005 | 1·000 | ·995 | ·990 | ·985 | ·979 | ·974 | ·968 | ·963 | ·958 | ·952 | ·946 | ·941 | ·935 |
| 30·1 | 1·053 | 1·048 | 1·043 | 1·038 | 1·033 | 1·029 | 1·024 | 1·019 | 1·014 | 1·009 | 1·003 | ·998 | ·993 | ·988 | ·983 | ·977 | ·972 | ·966 | ·961 | ·955 | ·950 | ·944 | ·938 |
| 30·2 | 1·057 | 1·052 | 1·047 | 1·042 | 1·037 | 1·032 | 1·027 | 1·022 | 1·017 | 1·012 | 1·007 | 1·002 | ·996 | ·991 | ·986 | ·980 | ·975 | ·970 | ·964 | ·959 | ·953 | ·947 | ·941 |
| 30·3 | 1·060 | 1·055 | 1·050 | 1·045 | 1·040 | 1·036 | 1·030 | 1·025 | 1·020 | 1·015 | 1·010 | 1·005 | 1·000 | ·995 | ·989 | ·984 | ·978 | ·973 | ·968 | ·962 | ·956 | ·950 | ·945 |
| 30·4 | 1·064 | 1·059 | 1·054 | 1·049 | 1·044 | 1·039 | 1·034 | 1·029 | 1·024 | 1·019 | 1·014 | 1·008 | 1·003 | ·998 | ·993 | ·987 | ·982 | ·976 | ·971 | ·965 | ·959 | ·954 | ·948 |
| 30·5 | 1·067 | 1·062 | 1·057 | 1·052 | 1·047 | 1·042 | 1·037 | 1·032 | 1·027 | 1·022 | 1·017 | 1·012 | 1·006 | 1·001 | ·996 | ·990 | ·985 | ·980 | ·974 | ·969 | ·963 | ·957 | ·951 |
| 30·6 | 1·071 | 1·066 | 1·061 | 1·056 | 1·051 | 1·046 | 1·041 | 1·036 | 1·031 | 1·026 | 1·020 | 1·015 | 1·010 | 1·005 | ·999 | ·994 | ·988 | ·983 | ·977 | ·972 | ·966 | ·960 | ·954 |
| 30·7 | 1·074 | 1·069 | 1·064 | 1·059 | 1·054 | 1·049 | 1·044 | 1·039 | 1·034 | 1·029 | 1·024 | 1·018 | 1·013 | 1·008 | 1·003 | ·997 | ·992 | ·986 | ·981 | ·975 | ·969 | ·963 | ·957 |
| 30·8 | 1·078 | 1·073 | 1·068 | 1·063 | 1·058 | 1·053 | 1·048 | 1·043 | 1·037 | 1·032 | 1·027 | 1·022 | 1·017 | 1·011 | 1·006 | 1·000 | .995 | ·990 | ·984 | ·978 | ·972 | ·967 | ·961 |
| 30·9 | 1·081 | 1·076 | 1·071 | 1·066 | 1·061 | 1·056 | 1·051 | 1·046 | 1·041 | 1·036 | 1·031 | 1·025 | 1·020 | 1·015 | 1·009 | 1·004 | ·998 | ·993 | ·987 | ·982 | ·976 | ·970 | ·964 |
| 31·0 | 1·085 | 1·080 | 1.075 | 1·070 | 1·065 | 1·060 | 1·055 | 1·049 | 1·044 | 1·039 | 1·034 | 1·029 | 1·023 | 1·018 | 1·013 | 1·007 | 1·002 | ·996 | ·991 | ·985 | ·979 | ·973 | ·967 |

⁂ The numbers in the above table have been re-calculated from the formula $n = \frac{17{\cdot}64\,(h - a)}{460 + t}$, where $h$ is the height of the barometer in inches, $t$ the temperature on the Fahrenheit scale, and $a$ the tension of aqueous vapour at $t^\circ$. If $v$ is any volume at $t^\circ$ and $h$ inches pressure, and V the corresponding volume at 60° and 30 inches pressure, $V = v\,n$.

## ATOMIC WEIGHTS, EQUIVALENT WEIGHTS, AND VALENCY OF ELEMENTS.

| | Symbol and Valency. | Atomic Weight. | Equivalent Weight. | | Symbol and Valency. | Atomic Weight. | Equivalent Weight. |
|---|---|---|---|---|---|---|---|
| Aluminium | $\mathrm{Al^{iv}}$ | 27·5 | 13·75 | Molybdenum | $\mathrm{Mo^{vi}}$ | 96 | 48 |
| Antimony | $\mathrm{Sb^{iii\ v}}$ | 122 | 122 | Nickel | $\mathrm{Ni^{ii}}$ | 59 | 29·5 |
| Arsenic | $\mathrm{As^{iii\ v}}$ | 75 | 75 | Niobium | $\mathrm{Nb^{v}}$ | 94 | 47 |
| Barium | $\mathrm{Ba^{ii}}$ | 137 | 68·5 | Nitrogen | $\mathrm{N^{iii\ v}}$ | 14 | 14 |
| Beryllium | $\mathrm{Be^{ii}}$ | 9·4 | 4·7 | Osmium | $\mathrm{Os^{iv\ vi}}$ | 199 | 99·5 |
| Bismuth | $\mathrm{Bi^{iii\ v}}$ | 208 | 208 | Oxygen | $\mathrm{O^{ii}}$ | 16 | 8 |
| Boron | $\mathrm{Bo^{iii}}$ | 11 | 11 | Palladium | $\mathrm{Pd^{ii\ iv\ vi}}$ | 106 | 53 |
| Bromine | $\mathrm{Br^{i\ vii}}$ | 80 | 80 | Phosphorus | $\mathrm{P^{iii\ v}}$ | 31 | 31 |
| Cadmium | $\mathrm{Cd^{ii}}$ | 112 | 56 | Platinum | $\mathrm{Pt^{ii\ iv\ vi}}$ | 197·18 | 98·59 |
| Caesium | $\mathrm{Cs^{i}}$ | 133 | 133 | Potassium | $\mathrm{K^{i}}$ | 39 | 39 |
| Calcium | $\mathrm{Ca^{ii}}$ | 40 | 20 | Rhodium | $\mathrm{Rh^{ii\ iv\ vi}}$ | 104 | 52 |
| Carbon | $\mathrm{C^{iv\ ii}}$ | 12 | 6 | Rubidium | $\mathrm{Rb^{i}}$ | 85 | 85 |
| Cerium | $\mathrm{Ce^{iv}}$ | 137 | 68·5 | Ruthenium | $\mathrm{Ru^{iv\ viii}}$ | 104 | 52 |
| Chlorine | $\mathrm{Cl^{i\ vii}}$ | 35·5 | 35·5 | Selenium | $\mathrm{Se^{ii\ iv\ vi}}$ | 79 | 39·5 |
| Chromium | $\mathrm{Cr^{ii\ iv\ vi}}$ | 52·5 | 26·25 | Silver | $\mathrm{Ag^{i}}$ | 108 | 108 |
| Cobalt | $\mathrm{Co^{ii\ vi}}$ | 59 | 29·5 | Silicium | $\mathrm{Si^{iv}}$ | 28 | 14 |
| Copper | $\mathrm{Cu^{ii}}$ | 63·5 | 31·75 | Sodium | $\mathrm{Na^{i}}$ | 23 | 23 |
| Didymium | $\mathrm{Di^{iv}}$ | 144 | 72 | Strontium | $\mathrm{Sr^{ii}}$ | 87·5 | 43·75 |
| Erbium | $\mathrm{Er^{iii}}$ | 170·6 | 85·3 | Sulphur | $\mathrm{S^{ii\ iv\ vi}}$ | 32 | 16 |
| Fluorine | $\mathrm{F^{i}}$ | 19 | 19 | Tantalium | $\mathrm{Ta^{v}}$ | 182 | 182 |
| Gold | $\mathrm{Au^{iii}}$ | 197 | 197 | Tellurium | $\mathrm{Te^{ii\ iv\ vi}}$ | 125 | 62·5 |
| Hydrogen | $\mathrm{H^{i}}$ | 1 | 1 | Thallium | $\mathrm{Tl^{i\ iii}}$ | 204 | 204 |
| Indium | $\mathrm{In^{iii}}$ | 113 | 56·5 | Thorium | $\mathrm{Th^{iv}}$ | 231·5 | 231·5 |
| Iodine | $\mathrm{I^{i\ vii}}$ | 127 | 127 | Tin | $\mathrm{Sn^{iv}}$ | 118 | 59 |
| Iridium | $\mathrm{Ir^{iv\ vi}}$ | 193 | 96·5 | Titanium | $\mathrm{Ti^{iv}}$ | 48 | 24 |
| Iron | $\mathrm{Fe^{ii\ iv\ vi}}$ | 56 | 28 | Uranium | $\mathrm{Ur^{vi}}$ | 240 | 240 |
| Lanthanum | $\mathrm{La^{iv}}$ | 139 | 69·5 | Vanadium | $\mathrm{V^{v}}$ | 51 | 25·5 |
| Lead | $\mathrm{Pb^{ii\ iv}}$ | 207 | 103·5 | Wolframium | $\mathrm{W^{vi}}$ | 184 | 92 |
| Lithium | $\mathrm{Li^{i}}$ | 7 | 7 | Yttrium | $\mathrm{Y^{iii}}$ | 88 | 44 |
| Magnesium | $\mathrm{Mg^{ii}}$ | 24 | 12 | Zinc | $\mathrm{Zn^{ii}}$ | 65 | 32·5 |
| Manganium | $\mathrm{Mn^{ii\ vii}}$ | 55 | 27·5 | Zirconium | $\mathrm{Zr^{iv}}$ | 90 | 45·0 |
| Mercury | $\mathrm{Hg^{ii}}$ | 200 | 100 | | | | |

## Factors for calculating Gravimetric Analyses.

| Substance Weighed. | Substance to be determined. | Factor. |
|---|---|---|
| Ammonium: | | |
| Ammonium chloride, $NH_4Cl$ | Ammonia, $NH_3$ | 0·31776 |
| Ammonium platinum chloride, $(NH_4)_2PtCl_6$ | Ammonia, $NH_3$ | 0·07620 |
| | Nitrogen, N | 0·06276 |
| Arsenic: | | |
| Arsenic tersulphide, $As_2S_3$ | Arsenic, As | 0·60975 |
| „ „ „ | Arsenious oxide. $As_2O_3$ | 0·80488 |
| „ „ „ | Arsenic anhydride, $As_2O_5$ | 0·93496 |
| Barium: | | |
| Barium sulphate, $BaSO_4$ | Barium oxide, BaO | 0·65665 |
| „ carbonate, $BaCO_3$ | „ „ „ | 0·77655 |
| Calcium: | | |
| Calcium sulphate, $CaSO_4$ | Calcium oxide, CaO | 0·41176 |
| „ carbonate, $CaCO_3$ | „ „ „ | 0·56000 |
| Carbon: | | |
| Carbonic anhydride, $CO_2$ | Carbon, C | 0·27273 |
| Calcium carbonate, $CaCO_3$ | Carbonic anhydride | 0·44000 |
| Barium Carbonate, $BaCO_3$ | „ „ | 0·22335 |
| Chlorine: | | |
| Silver Chloride, AgCl | Chlorine, Cl | 0·24739 |
| „ „ „ | Hydrochloric acid, HCl | 0·25435 |
| „ „ „ | Sodium chloride, NaCl | 0·40767 |
| Iron: | | |
| Ferric Oxide, $Fe_2O_3$ | Iron, Fe | 0·7000 |
| „ „ „ | Ferrous oxide, FeO | 0·9000 |
| Hydrogen: | | |
| Water, $H_2O$ | Hydrogen, H | 0·11111 |
| Magnesium: | | |
| Magnesium pyrophosphate, $Mg_2P_2O_7$ | Magnesium, Mg | 0·2160 |
| Sulphur: | | |
| Barium sulphate, $BaSO_4$ | Sulphur, S | ·13734 |
| „ „ | Sulphuric anhydride, $SO_3$ | 0·3433 |
| „ „ | Sulphurous anhydride, $SO_2$ | 0·2746 |

These factors are employed in the following manner:—The weight of a precipitate having been obtained, on multiplying this weight by the factor given, the weight of the substance to be determined is arrived at. Thus, on multiplying any weight of ferric oxide by the factor 0·7 the equivalent weight of iron (Fe) will be obtained; or any weight of $BaSO_4$, on being multiplied by ·13734, will give the equivalent weight of sulphur.

## SOLUBILITY OF DIFFERENT SALTS.

*Remark.*—The solubility is given in parts of the anhydrous salt dissolved by 100 parts of water.

| 100 Water Dissolve. | Cold. | Boiling. |
|---|---|---|
| Alum, ammonia | 9 | 422 |
| „ potash | 9·5 | 357 |
| Aluminium sulphate | 33 | 89 |
| Ammonium oxalate | 4·5 | 40·8 |
| „ nitrate | 199 | .. |
| „ sulphate | 66 | 100 |
| Barium chloride | 35 | 60 |
| „ hydrate | 5 | 10 |
| „ nitrate | 8 | 35 |
| Boric acid | 2 | 21 |
| Bromine | 3 | .. |
| Calcium carbonate | 0·0036 | .. |
| „ chloride | 400 | .. |
| „ hydrate | 0·128 | 0·079 |
| „ nitrate | 400 | .. |
| „ sulphate | 0·23 | 0·21 |
| Copper acetate | 7 | 19·8 |
| „ nitrate | 127 | .. |
| „ sulphate | 21 | 75 |
| Iron protosulphate | 20 | 178 |
| Lead acetate | 46 | 71 |
| „ chloride | 3 | 5 |
| „ nitrate | 48 | 139 |
| „ sulphate | ·008 | .. |
| Magnesium oxide | ·002 | ·002 |
| „ carbonate | ·02 | .. |
| „ chloride | 200 | 400 |
| Manganous chloride | 62 | 123 |
| Oxalic acid | 11·5 | 100 |
| Potassium hydrate | 200 | .. |
| „ chromate (neutral) | 48 | .. |
| „ bichromate | 10 | 102 |
| „ oxalate (acid) | 2·5 | 10 |
| „ sulphite | 100 | .. |
| „ hyposulphite | hygroscopic | .. |
| „ bitartrate | 0·4 | 10·5 |
| „ tartrate (neutral) | 133 | 296 |
| „ cyanide | 122 | .. |
| „ ferrocyanide | 28 | 91 |
| „ ferricyanide | 40 | 82 |
| „ iodide | 141 | 221 |

### SOLUBILITY OF DIFFERENT SALTS—*continued.*

| 100 Water Dissolve. | Cold. | Boiling. |
|---|---|---|
| Sodium acetate (borax) | 35 | 150 |
| ,, borate | 4 | 55 |
| ,, hydrate | 61 | .. |
| ,, hyposulphite | 50 | more than 200 |
| ,, phosphate | 12 | .. |
| ,, sulphite | 25 | 100 |
| Strontium hydrate | 1·6 | 34·8 |
| ,, nitrate | 20 | 113 |
| ,, chloride | 53 | 102 |
| Tartaric acid | 76 | 200 |
| Tin (stannous) chloride | 270 | .. |
| Zinc chloride | 300 | .. |
| ,, sulphate | 50 | 95 |

### BOILING POINTS.

| | Centigrade. | Fahrenheit. |
|---|---|---|
| | ° | ° |
| Alcohol, absolute | 78 | 172·4 |
| Ammonia, anhydrous | −38·5 | −37·3 |
| ,, nitrate, satur. solution | 164 | 327 |
| Barium chloride, satur. solution | 104·4 | 220 |
| Bisulphide of carbon | 47·0 | 116·6 |
| Benzol | 80·4 | 177 |
| Bromine | 63·0 | 145·4 |
| Calcium chloride, satur. solution | 179·5 | 355·1 |
| ,, ,, 66 per cent. solution | 156 | 312·8 |
| ,, ,, 33 ,, ,, | 128 | 262·4 |
| ,, nitrate, satur. solution | 152 | 305·6 |
| Carbonic acid | −78 | −108 |
| Ether | 35 | 95 |
| Hydrochloric acid, 20·2 per cent. HCl | 110 | 230 |
| Iodine above | 200 | 392 |
| Methylic alcohol | 60 | 140 |
| Mercury | 357 | 674·6 |
| Naphthalene | 217 | 422·6 |
| Nitric acid, most concentrated | 86 | 186·8 |
| ,, ,, specific gravity 1·42 | 121 | 249·8 |
| Nitrous anhydride | −2 | 28·4 |
| ,, oxide | −88 | −126 |
| Nitrogen dioxide | 28 | 82·4 |

## BOILING POINTS—*continued.*

| | Centigrade. | Fahrenheit. |
|---|---|---|
| | ° | ° |
| Potassium chloride, satur. solution | 110 | 230 |
| „ chlorate „ „ | 105 | 221 |
| „ acetate „ „ | 169·4 | 336·9 |
| „ carbonate „ „ | 135 | 275 |
| „ nitrate „ „ | 118 | 244·4 |
| Sodium chloride | 108·4 | 227·1 |
| „ acetate | 124·4 | 255·9 |
| „ carbonate | 106 | 222·8 |
| „ phosphate | 106·6 | 223·8 |
| „ nitrate | 122 | 251·6 |
| Sulphur | 448 | 838 |
| Sulphuric acid | 326 | 618·8 |
| „ anhydride α | 15 | 59 |
| „ „ β | 50 | 122 |
| Sulphurous anhydride | −10 | 14 |
| Turpentine, spirits of | 160 | 320 |

## FUSING POINTS.

| | Centigrade. | Fahrenheit. |
|---|---|---|
| | ° | ° |
| Aluminium | 700 | 1292 |
| Antimony | 432 | 809 |
| Asphalt | 100 | 212 |
| Bismuth | 260 | 500 |
| Boric acid | 186 | 367 |
| Brass | 900 | 1652 |
| Bromine | −22 | −7·6 |
| Bronze | 900 | 1652 |
| Cadmium | 316 | 600 |
| Cobalt | 1500 | 2732 |
| Colophonium | 135 | 275 |
| Copper | 1100 | 2012 |
| Cupric chloride | 498 | 928 |
| Cuprous chloride | 434 | 813 |
| Fat, oxen | 40 | 104 |
| „ sheep | 42 | 107·6 |
| „ pig | 27 | 80·6 |
| Fluorspar | 902 | 1655 |
| Glass | 1200 | 2192 |
| Glass containing lead | 1000 | 1832 |

Fusing Points—*continued.*

| | Centigrade. | Fahrenheit. |
|---|---|---|
| | ° | ° |
| Gold | 1075 | 1967 |
| Iron, cast, white | 1075 | 1967 |
| „ „ grey | 1275 | 2327 |
| „ wrought | 1550 | 2822 |
| Iodine | 113 | 235·4 |
| Lead | 326 | 618 |
| „ oxide | 954 | 1749 |
| „ chloride | 498 | 928 |
| Magnesium | 500 | 932 |
| Mercury | −39 | −38·2 |
| Mercuric chloride | 293 | 560 |
| Naphthalene | 79 | 174·2 |
| Nickel | 1500 | 2732 |
| Palm oil | 29 | 84·2 |
| Paraffin | 45–60 | 113–140 |
| Platinum | 1775 | 3227 |
| Pitch (coal tar) | 150–200 | 300–400 |
| Phosphorus | 44 | 111·2 |
| Potassium chlorate | 359 | 678 |
| „ iodide | 634 | 1173 |
| „ carbonate | 834 | 1533 |
| „ nitrate | 329 | 624 |
| Stearic acid | 70 | 158 |
| Steel | 1375 | 2507 |
| Silver, metallic | 960 | 1760 |
| „ chloride | 451 | 843·8 |
| „ nitrate | 217 | 422 |
| Strontium chloride | 825 | 1517 |
| Selenium | 217 | 422 |
| Sodium chloride | 772 | 1421 |
| „ sulphate | 861 | 1581 |
| „ nitrate | 316 | 600 |
| „ chlorate | 302 | 575 |
| „ carbonate | 814 | 1497 |
| Spermaceti | 45–50 | 113–122 |
| Thallium | 290 | 554 |
| Tin | 230 | 446 |
| Wax, bee's | 62–70 | 143–158 |
| Zinc | 412 | 773 |

## SPECIFIC GRAVITY OF NITRIC ACID AT 60° F. (15° C.), CALCULATED FROM KOLB'S RESULTS. (*Lunge and Hurter.*)

| Degrees Twaddell. | Specific Gravity. | Percentage by Weight. | | Grammes per Litre. | | Degrees Twaddell. | Specific Gravity. | Percentage by Weight. | | Grammes per Litre. | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | $HNO_3$ | $N_2O_5$ | $HNO_3$ | $N_2O_5$ | | | $HNO_3$ | $N_2O_5$ | $HNO_3$ | $N_2O_5$ |
| 1 | 1·005 | ·88 | ·75 | 8·8 | 7·5 | 41 | 1·205 | 33·02 | 28·30 | 397·9 | 341·1 |
| 2 | 1·010 | 1·75 | 1·50 | 17·6 | 15·1 | 42 | 1·210 | 33·79 | 28·96 | 408·8 | 350·4 |
| 3 | 1·015 | 2·62 | 2·25 | 26·6 | 22·8 | 43 | 1·215 | 34·55 | 29·61 | 419·8 | 359·9 |
| 4 | 1·020 | 3·50 | 3·00 | 35·7 | 30·6 | 44 | 1·220 | 35·32 | 30·27 | 430·9 | 369·3 |
| 5 | 1·025 | 4·37 | 3·75 | 44·8 | 38·4 | 45 | 1·225 | 36·08 | 30·93 | 442·0 | 378·9 |
| 6 | 1·030 | 5·25 | 4·50 | 54·0 | 46·3 | 46 | 1·230 | 36·85 | 31·58 | 453·2 | 388·5 |
| 7 | 1·035 | 6·12 | 5·25 | 63·3 | 54·2 | 47 | 1·235 | 37·61 | 32·24 | 464·5 | 398·1 |
| 8 | 1·040 | 6·95 | 5·95 | 72·3 | 62·0 | 48 | 1·240 | 38·38 | 32·90 | 475·9 | 407·9 |
| 9 | 1·045 | 7·77 | 6·66 | 81·2 | 69·6 | 49 | 1·245 | 39·15 | 33·55 | 487·4 | 417·8 |
| 10 | 1·050 | 8·59 | 7·36 | 90·2 | 77·3 | 50 | 1·250 | 39·91 | 34·21 | 498·8 | 427·6 |
| 11 | 1·055 | 9·41 | 8·07 | 99·3 | 85·1 | 51 | 1·255 | 40·68 | 34·86 | 510·5 | 437·6 |
| 12 | 1·060 | 10·23 | 8·77 | 108·4 | 97·3 | 52 | 1·260 | 41·44 | 35·52 | 522·1 | 447·6 |
| 13 | 1·065 | 11·06 | 9·48 | 117·8 | 101·0 | 53 | 1·265 | 42·21 | 36·18 | 532·8 | 456·6 |
| 14 | 1·070 | 11·88 | 10·18 | 127·1 | 108·9 | 54 | 1·270 | 42·97 | 36·83 | 545·7 | 467·8 |
| 15 | 1·075 | 12·70 | 10·89 | 136·5 | 117·0 | 55 | 1·275 | 43·74 | 37·49 | 557·7 | 478 0 |
| 16 | 1·080 | 13·52 | 11·59 | 146·0 | 125·1 | 56 | 1·280 | 44·50 | 38·15 | 569·6 | 488·2 |
| 17 | 1·085 | 14·34 | 12·29 | 155·6 | 133·4 | 57 | 1·285 | 45·27 | 38·80 | 581·7 | 498·6 |
| 18 | 1·090 | 15·16 | 12 99 | 165·2 | 141·6 | 58 | 1·290 | 46·04 | 39·46 | 593·9 | 509·1 |
| 19 | 1·095 | 15·98 | 13·70 | 175·0 | 150·0 | 59 | 1·295 | 46·80 | 40·11 | 606·0 | 519·5 |
| 20 | 1·100 | 16·80 | 14·40 | 184·8 | 158·4 | 60 | 1·300 | 47·57 | 40·77 | 618·4 | 530·1 |
| 21 | 1·105 | 17·57 | 15·06 | 194·1 | 166·4 | 61 | 1·305 | 48·33 | 41·43 | 630·7 | 540·6 |
| 22 | 1·110 | 18·35 | 15·72 | 203·7 | 174·6 | 62 | 1·310 | 49·10 | 42·08 | 643·2 | 551·3 |
| 23 | 1·115 | 19·12 | 16·39 | 213·2 | 182·7 | 63 | 1·315 | 49·86 | 42·74 | 655·6 | 562·0 |
| 24 | 1·120 | 19·89 | 17.05 | 222·7 | 190·9 | 64 | 1·320 | 50·63 | 43·40 | 668·3 | 572·8 |
| 25 | 1·125 | 20·67 | 17·71 | 232·5 | 199·3 | 65 | 1·325 | 51·40 | 44·06 | 681·0 | 583·8 |
| 26 | 1·130 | 21·44 | 18·38 | 242·3 | 207·7 | 66 | 1·330 | 52·24 | 44·78 | 694·8 | 595·6 |
| 27 | 1·135 | 22·21 | 19·04 | 252·0 | 216·0 | 67 | 1·335 | 53·09 | 45·51 | 708·7 | 607·5 |
| 28 | 1·140 | 22·98 | 19·70 | 262·0 | 224·6 | 68 | 1·340 | 53·94 | 46·24 | 722·8 | 619·6 |
| 29 | 1·145 | 23·76 | 20·36 | 272·0 | 233·2 | 69 | 1·345 | 54·79 | 46·96 | 736·9 | 631·6 |
| 30 | 1·150 | 24·53 | 21·03 | 282·1 | 241·8 | 70 | 1·350 | 55·64 | 47·69 | 751·1 | 643·8 |
| 31 | 1·155 | 25·30 | 21·69 | 292·2 | 250·4 | 71 | 1·355 | 56·53 | 48·45 | 766·0 | 656·6 |
| 32 | 1·160 | 26·08 | 22·35 | 302·5 | 259·3 | 72 | 1·360 | 57·42 | 49·22 | 780·9 | 669·4 |
| 33 | 1·165 | 26·85 | 23·01 | 312·8 | 268·1 | 73 | 1·365 | 58·31 | 49·98 | 795·9 | 682·2 |
| 34 | 1·170 | 27·62 | 23·68 | 323·1 | 276·9 | 74 | 1·370 | 59·21 | 50·75 | 811·2 | 695·3 |
| 35 | 1·175 | 28·40 | 24·34 | 333·7 | 286·0 | 75 | 1·375 | 60·10 | 51·51 | 826·4 | 708·4 |
| 36 | 1·180 | 29·17 | 25·00 | 344·2 | 295·0 | 76 | 1·380 | 61·00 | 52·29 | 841·8 | 721·6 |
| 37 | 1·185 | 29·94 | 25·66 | 354·8 | 304·1 | 77 | 1·385 | 61·97 | 53·12 | 858·3 | 735·8 |
| 38 | 1·190 | 30·71 | 26·33 | 365·4 | 313·2 | 78 | 1·390 | 62·95 | 53·96 | 875·0 | 750·0 |
| 39 | 1·195 | 31·49 | 26·99 | 376·3 | 322·6 | 79 | 1·395 | 63·92 | 54·79 | 891·7 | 764·2 |
| 40 | 1·200 | 32·26 | 27·65 | 387·1 | 331·8 | 80 | 1·400 | 64·90 | 55·66 | 908·6 | 778·8 |

## Specific Gravity of Nitric Acid at 60 F. (15° C.)—*continued.*

| Degrees Twaddell. | Specific Gravity. | Percentage by Weight. | | Grammes per Litre. | | Degrees Twaddell. | Specific Gravity. | Percentage by Weight. | | Grammes per Litre. | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | $HNO_3$ | $N_2O_5$ | $HNO_3$ | $N_2O_5$ | | | $HNO_3$ | $N_2O_5$ | $HNO_3$ | $N_2O_5$ |
| 81 | 1·405 | 66·01 | 56·61 | 927·4 | 794·9 | 94 | 1·470 | 82·90 | 71·05 | 1219 | 1045 |
| 82 | 1·410 | 67·12 | 57·56 | 946·4 | 811·2 | 95 | 1·475 | 84·28 | 72·24 | 1243 | 1066 |
| 83 | 1·415 | 68·23 | 58·51 | 965·4 | 827·4 | 96 | 1·480 | 85·66 | 73·43 | 1268 | 1087 |
| 84 | 1·420 | 69·34 | 59·46 | 984·6 | 844·0 | 97 | 1·485 | 87·05 | 74·61 | 1293 | 1108 |
| 85 | 1·425 | 70·45 | 60·39 | 1004 | 860·6 | 98 | 1·490 | 88·43 | 75·80 | 1318 | 1130 |
| 86 | 1·430 | 71·83 | 61·57 | 1027 | 880·4 | 99 | 1·495 | 89·82 | 76·98 | 1343 | 1151 |
| 87 | 1·435 | 73·21 | 62·76 | 1050 | 900·0 | 100 | 1·500 | 91·20 | 78·18 | 1368 | 1173 |
| 88 | 1·440 | 74·59 | 63·94 | 1074 | 920·6 | 101 | 1·505 | 92·66 | 79·43 | 1394 | 1195 |
| 89 | 1·445 | 75·98 | 65·13 | 1097 | 940·4 | 102 | 1·510 | 94·13 | 80·69 | 1421 | 1218 |
| 90 | 1·450 | 77·36 | 66·31 | 1122 | 961·8 | 103 | 1·515 | 95·59 | 81·94 | 1448 | 1241 |
| 91 | 1·455 | 78·75 | 67·50 | 1146 | 982·4 | 104 | 1·520 | 97·06 | 83·20 | 1475 | 1264 |
| 92 | 1·460 | 80·13 | 68·68 | 1170 | 1003 | 105 | 1·525 | 98·53 | 84·46 | 1502 | 1287 |
| 93 | 1·465 | 81·52 | 69·87 | 1195 | 1824 | 106 | 1·530 | 100·00 | 85·71 | 1530 | 1311 |

## Percentage of Pure HCl Grammes per Litre 60° F. (15° C.), calculated from Kolb's Results. (*Lunge and Hurter, Alkali Maker's Pocket Book.*)

| Degrees Twaddell. | Specific Gravity. | Percentage of HCl. | Grammes per Litre. | Degrees Twaddell. | Specific Gravity. | Percentage of HCl. | Grammes per Litre. |
|---|---|---|---|---|---|---|---|
| 1 | 1·005 | 1·12 | 11·32 | 21 | 1·105 | 21·06 | 232·68 |
| 2 | 1·010 | 2·12 | 21·45 | 22 | 1·110 | 22·06 | 244·80 |
| 3 | 1·015 | 3·12 | 31·67 | 23 | 1·115 | 23·05 | 257·02 |
| 4 | 1·020 | 4·11 | 41·99 | 24 | 1·120 | 24·05 | 269·34 |
| 5 | 1·025 | 5·11 | 52·41 | 25 | 1·125 | 25·05 | 281·76 |
| 6 | 1·030 | 6·11 | 62·93 | 26 | 1·130 | 26·04 | 294·28 |
| 7 | 1·035 | 7·10 | 73·55 | 27 | 1·135 | 27·04 | 306·90 |
| 8 | 1·040 | 8·10 | 84·27 | 28 | 1·140 | 28·04 | 319·62 |
| 9 | 1·045 | 9·10 | 95·09 | 29 | 1·145 | 29·03 | 332·44 |
| 10 | 1·050 | 10·09 | 106·01 | 30 | 1·150 | 30·03 | 345·36 |
| 11 | 1·055 | 11·09 | 117·02 | 31 | 1·155 | 31·03 | 358·34 |
| 12 | 1·060 | 12·09 | 128·14 | 32 | 1·160 | 32·02 | 371·44 |
| 13 | 1·065 | 13·08 | 139·36 | 33 | 1·165 | 33·02 | 384·64 |
| 14 | 1·070 | 14·08 | 150·68 | 34 | 1·170 | 34·02 | 397·94 |
| 15 | 1·075 | 15·08 | 162·10 | 35 | 1·175 | 35·01 | 411·34 |
| 16 | 1·080 | 16·07 | 173·62 | 36 | 1·180 | 36·01 | 424·84 |
| 17 | 1·085 | 17·07 | 185·24 | 37 | 1·185 | 37·01 | 438·44 |
| 18 | 1·090 | 18·07 | 196·96 | 38 | 1·190 | 38·01 | 452·14 |
| 19 | 1·095 | 19·07 | 208·78 | 39 | 1·195 | 39·00 | 466·00 |
| 20 | 1·100 | 20·06 | 220·70 | 40 | 1·200 | 40·00 | 479·84 |

## Table of the Percentages of real Sulphuric Acid ($SO_4H_2$) corresponding to various Specific Gravities of Aqueous Sulphuric Acid.

Bineau; Otto. Temp. 15°.

| Specific Gravity. | Per cent. | Specific Gravity. | Per cent. | Specific Gravity. | Per cent. |
|---|---|---|---|---|---|
| 1·8426 | 100 | 1·568 | 66 | 1·2476 | 33 |
| 1·842 | 99 | 1·557 | 65 | 1·239 | 32 |
| 1·8406 | 98 | 1·545 | 64 | 1·231 | 31 |
| 1·840 | 97 | 1·534 | 63 | 1·223 | 30 |
| 1·8384 | 96 | 1·523 | 62 | 1·215 | 29 |
| 1·8376 | 95 | 1·512 | 61 | 1·2066 | 28 |
| 1·8356 | 94 | 1·501 | 60 | 1·198 | 27 |
| 1·834 | 93 | 1·490 | 59 | 1·190 | 26 |
| 1·831 | 92 | 1·480 | 58 | 1·182 | 25 |
| 1·827 | 91 | 1·469 | 57 | 1·174 | 24 |
| 1·822 | 90 | 1·4586 | 56 | 1·167 | 23 |
| 1·816 | 89 | 1·448 | 55 | 1·159 | 22 |
| 1·809 | 88 | 1·438 | 54 | 1·1516 | 21 |
| 1·802 | 87 | 1·428 | 53 | 1·144 | 20 |
| 1·794 | 86 | 1·418 | 52 | 1·136 | 19 |
| 1·786 | 85 | 1·408 | 51 | 1·129 | 18 |
| 1·777 | 84 | 1·398 | 50 | 1·121 | 17 |
| 1·767 | 83 | 1·3886 | 49 | 1·1136 | 16 |
| 1·756 | 82 | 1·379 | 48 | 1·106 | 15 |
| 1·745 | 81 | 1·370 | 47 | 1·098 | 14 |
| 1·734 | 80 | 1·361 | 46 | 1·091 | 13 |
| 1·722 | 79 | 1·351 | 45 | 1·083 | 12 |
| 1·710 | 78 | 1·342 | 44 | 1·0756 | 11 |
| 1·698 | 77 | 1·333 | 43 | 1·068 | 10 |
| 1·686 | 76 | 1·324 | 42 | 1·061 | 9 |
| 1·675 | 75 | 1·315 | 41 | 1·0536 | 8 |
| 1·663 | 74 | 1·306 | 40 | 1·0464 | 7 |
| 1·651 | 73 | 1·2976 | 39 | 1·039 | 6 |
| 1·639 | 72 | 1·289 | 38 | 1·032 | 5 |
| 1·627 | 71 | 1·281 | 37 | 1·0256 | 4 |
| 1·615 | 70 | 1·272 | 36 | 1·019 | 3 |
| 1·604 | 69 | 1·264 | 35 | 1·013 | 2 |
| 1·592 | 68 | 1·256 | 34 | 1·0064 | 1 |
| 1·580 | 67 | | | | |

## Table of Specific Gravity and Percentage of Caustic Potash in Aqueous Solution.

| Sp. gr. at 15° C. | Percentage of KOH. | Sp. gr. at 15° C. | Percentage of KOH. | Sp. gr. at 15° C. | Percentage of KOH. | Sp. gr. at 15° C. | Percentage of KOH. |
|---|---|---|---|---|---|---|---|
| 1·009 | 1 | 1·166 | 19 | 1·374 | 37 | 1·590 | 54 |
| 1·017 | 2 | 1·177 | 20 | 1·387 | 38 | 1·604 | 55 |
| 1·025 | 3 | 1·188 | 21 | 1·400 | 39 | 1·618 | 56 |
| 1·033 | 4 | 1·198 | 22 | 1·412 | 40 | 1·630 | 57 |
| 1·041 | 5 | 1·209 | 23 | 1·425 | 41 | 1·642 | 58 |
| 1·049 | 6 | 1·220 | 24 | 1·438 | 42 | 1·655 | 59 |
| 1·058 | 7 | 1·230 | 25 | 1·450 | 43 | 1·667 | 60 |
| 1·065 | 8 | 1·241 | 26 | 1·462 | 44 | 1·681 | 61 |
| 1·074 | 9 | 1·252 | 27 | 1·475 | 45 | 1·695 | 62 |
| 1·083 | 10 | 1·264 | 28 | 1·488 | 46 | 1·705 | 63 |
| 1·092 | 11 | 1·276 | 29 | 1·499 | 47 | 1·718 | 64 |
| 1·101 | 12 | 1·288 | 30 | 1·511 | 48 | 1·729 | 65 |
| 1·110 | 13 | 1·300 | 31 | 1·525 | 49 | 1·740 | 66 |
| 1·119 | 14 | 1·311 | 32 | 1·539 | 50 | 1·754 | 67 |
| 1·128 | 15 | 1·324 | 33 | 1·552 | 51 | 1·768 | 68 |
| 1·137 | 16 | 1·336 | 34 | 1·565 | 52 | 1·780 | 69 |
| 1·146 | 17 | 1·349 | 35 | 1·578 | 53 | 1·790 | 70 |
| 1·155 | 18 | 1·361 | 36 | | | | |

## Table of Specific Gravity and Percentage of Soda ($Na_2O$) in Aqueous Solution.

Tünnermann.

| Sp. gr. | Per cent. | Sp. gr. | Per cent. | Sp. gr. | Per cent. | Sp. gr. | Per cent. |
|---|---|---|---|---|---|---|---|
| 1·4285 | 30·220 | 1·3198 | 22·363 | 1·2392 | 15·110 | 1·1042 | 7·253 |
| 1·4193 | 29·616 | 1·3143 | 21·894 | 1·2280 | 14·500 | 1·0948 | 6·648 |
| 1·4101 | 29·011 | 1·3125 | 21·758 | 1·2178 | 13·901 | 1·0855 | 6·044 |
| 1·4011 | 28·407 | 1·3053 | 21·154 | 1·2058 | 13·297 | 1·0764 | 5·440 |
| 1·3923 | 27·802 | 1·2982 | 20·550 | 1·1948 | 12·692 | 1·0675 | 4·835 |
| 1·3836 | 27·200 | 1·2912 | 19·945 | 1·1841 | 12·088 | 1·0587 | 4·231 |
| 1·3751 | 26·594 | 1·2843 | 19·341 | 1·1734 | 11·484 | 1·0500 | 3·626 |
| 1·3668 | 25·989 | 1·2775 | 18·730 | 1·1630 | 10·879 | 1·0414 | 3·022 |
| 1·3586 | 25·385 | 1·2708 | 18·132 | 1·1528 | 10·275 | 1·0330 | 2·418 |
| 1·3505 | 24·780 | 1·2642 | 17·528 | 1·1428 | 9·670 | 1·0246 | 1·813 |
| 1·3426 | 24·176 | 1·2578 | 16·923 | 1·1330 | 9·066 | 1·0163 | 1·209 |
| 1·3349 | 23·572 | 1·2515 | 16·379 | 1·1233 | 8·462 | 1·0081 | 0·604 |
| 1·3273 | 22·967 | 1·2453 | 15·714 | 1·1137 | 7·857 | 1·0040 | 0·302 |

### Specific Gravity of Caustic Ammonia containing different Quantities of $NH_3$, Temperature 14° C. (57° F.). (*Carius.*)

| Specific Gravity. | Percentage $NH_3$ | Specific Gravity. | Percentage $NH_3$ | Specific Gravity. | Percentage $NH_3$ |
|---|---|---|---|---|---|
| 0·8844 | 36·0 | 0·9021 | 28·2 | 0·9239 | 20·4 |
| 0·8848 | 35·8 | 0·9026 | 28·0 | 0·9245 | 20·2 |
| 0·8852 | 35·6 | 0·9031 | 27·8 | 0·9251 | 20·0 |
| 0·8856 | 35·4 | 0·9036 | 27·6 | 0·9257 | 19·8 |
| 0·8860 | 35·2 | 0·9041 | 27·4 | 0·9264 | 19·6 |
| 0·8864 | 35·0 | 0·9047 | 27·2 | 0·9271 | 19·4 |
| 0·8868 | 34·8 | 0·9052 | 27·0 | 0·9277 | 19·2 |
| 0·8872 | 34·6 | 0·9057 | 26·8 | 0·9283 | 19·0 |
| 0·8877 | 34·4 | 0·9063 | 26·6 | 0·9289 | 18·8 |
| 0·8881 | 34·2 | 0·9068 | 26·4 | 0·9296 | 18·6 |
| 0·8885 | 34·0 | 0·9073 | 26·2 | 0·9302 | 18·4 |
| 0·8889 | 33·8 | 0·9078 | 26·0 | 0·9308 | 18·2 |
| 0·8894 | 33·6 | 0·9083 | 25·8 | 0·9314 | 18·0 |
| 0·8898 | 33·4 | 0·9089 | 25·6 | 0·9321 | 17·8 |
| 0·8903 | 33·2 | 0·9094 | 25·4 | 0·9327 | 17·6 |
| 0·8907 | 33·0 | 0·9100 | 25·2 | 0·9333 | 17·4 |
| 0·8911 | 32·8 | 0·9106 | 25·0 | 0·9340 | 17·2 |
| 0·8916 | 32·6 | 0·9111 | 24·8 | 0·9347 | 17·0 |
| 0·8920 | 32·4 | 0·9116 | 24·6 | 0·9353 | 16·8 |
| 0·8925 | 32·2 | 0·9122 | 24·4 | 0·9360 | 16·6 |
| 0·8929 | 32·0 | 0·9127 | 24·2 | 0·9366 | 16·4 |
| 0·8934 | 31·8 | 0·9133 | 24·0 | 0·9373 | 16·2 |
| 0·8938 | 31·6 | 0·9139 | 23·8 | 0·9380 | 16·0 |
| 0·8943 | 31·4 | 0·9145 | 23·6 | 0·9386 | 15·8 |
| 0·8948 | 31·2 | 0·9150 | 23·4 | 0·9393 | 15·6 |
| 0·8953 | 31·0 | 0·9156 | 23·2 | 0·9400 | 15·4 |
| 0·8957 | 30·8 | 0·9162 | 23·0 | 0·9407 | 15·2 |
| 0·8962 | 30·6 | 0·9168 | 22·8 | 0·9414 | 15·0 |
| 0·8967 | 30·4 | 0·9174 | 22·6 | 0·9420 | 14·8 |
| 0·8971 | 30·2 | 0·9180 | 22·4 | 0·9427 | 14·6 |
| 0·8976 | 30·0 | 0·9185 | 22·2 | 0·9434 | 14·4 |
| 0·8981 | 29·8 | 0·9191 | 22·0 | 0·9441 | 14·2 |
| 0·8986 | 29·6 | 0·9197 | 21·8 | 0·9449 | 14·0 |
| 0·8991 | 29·4 | 0·9203 | 21·6 | 0·9456 | 13·8 |
| 0·8996 | 29·2 | 0·9209 | 21·4 | 0·9463 | 13·6 |
| 0·9001 | 29·0 | 0·9215 | 21·2 | 0·9470 | 13·4 |
| 0·9006 | 28·8 | 0·9221 | 21·0 | 0·9477 | 13·2 |
| 0·9011 | 28·6 | 0·9227 | 20·8 | 0·9484 | 13·0 |
| 0·9016 | 28·4 | 0·9233 | 20·6 | 0·9491 | 12·0 |

## SPECIFIC GRAVITY OF CAUSTIC AMMONIA—*continued.*

| Specific Gravity. | Percentage $NH_3$ | Specific Gravity. | Percentage $NH_3$ | Specific Gravity. | Percentage $NH_3$ |
|---|---|---|---|---|---|
| 0·9498 | 12·6 | 0·9654 | 8·4 | 0·9823 | 4·2 |
| 0·9505 | 12·4 | 0·9662 | 8·2 | 0·9831 | 4·0 |
| 0·9512 | 12·2 | 0·9670 | 8·0 | 0·9839 | 3·8 |
| 0·9520 | 12·0 | 0·9677 | 7·8 | 0·9847 | 3·6 |
| 0·9527 | 11·8 | 0·9685 | 7·6 | 0·9855 | 3·4 |
| 0·9534 | 11·6 | 0·9693 | 7·4 | 0·9863 | 3·2 |
| 0·9542 | 11·4 | 0·9701 | 7·2 | 0·9873 | 3·0 |
| 0·9549 | 11·2 | 0·9709 | 7·0 | 0·9882 | 2·8 |
| 0·9556 | 11·0 | 0·9717 | 6·8 | 0·9890 | 2·6 |
| 0·9563 | 10·8 | 0·9725 | 6·6 | 0·9899 | 2·4 |
| 0·9571 | 10·6 | 0·9733 | 6·4 | 0·9907 | 2·2 |
| 0·9578 | 10·4 | 0·9741 | 6·2 | 0·9915 | 2·0 |
| 0·9586 | 10·2 | 0·9749 | 6·0 | 0·9924 | 1·8 |
| 0·9593 | 10·0 | 0·9757 | 5·8 | 0·9932 | 1·6 |
| 0·9601 | 9·8 | 0·9765 | 5·6 | 0·9941 | 1·4 |
| 0·9608 | 9·6 | 0·9773 | 5·4 | 0·9950 | 1·2 |
| 0·9616 | 9·4 | 0·9781 | 5·2 | 0·9959 | 1·0 |
| 0·9623 | 9·2 | 0·9790 | 5·0 | 0·9967 | 0·8 |
| 0·9631 | 9·0 | 0·9799 | 4·8 | 0·9975 | 0·6 |
| 0·9639 | 8·8 | 0·9807 | 4·6 | 0·9983 | 0·4 |
| 0·9647 | 8·6 | 0·9815 | 4·4 | 0·9991 | 0·2 |

## SOLUBILITY OF AMMONIA IN WATER BY WEIGHT. 1 GRAMME WATER DISSOLVES AT 760 MM. PRESSURE. (*Roscoe and Dittmar.*)

| Temperature Centigrade. | Grammes $NH_3$ Absorbed. | Temperature Centigrade. | Grammes $NH_3$ Absorbed. | Temperature Centigrade. | Grammes $NH_3$ Absorbed. |
|---|---|---|---|---|---|
| ° | | ° | | ° | |
| 0 | 0·875 | 20 | 0·526 | 40 | 0·307 |
| 2 | 0·833 | 22 | 0·499 | 42 | 0·290 |
| 4 | 0·792 | 24 | 0·474 | 44 | 0·275 |
| 6 | 0·751 | 26 | 0·449 | 46 | 0·259 |
| 8 | 0·713 | 28 | 0·426 | 48 | 0·244 |
| 10 | 0·679 | 30 | 0·403 | 50 | 0·229 |
| 12 | 0·645 | 32 | 0·382 | 52 | 0·214 |
| 14 | 0·612 | 34 | 0·362 | 54 | 0·200 |
| 16 | 0·582 | 36 | 0·343 | 56 | 0·185 |
| 18 | 0·554 | 38 | 0·324 | | |

## CONVERSION OF DEGREES ON THE CENTIGRADE THERMOMETER INTO DEGREES OF FAHRENHEIT'S SCALE.

| Cent. | Fahr. | Cent. | Fahr. | Cent. | Fahr. | Cent. | Fahr. |
|---|---|---|---|---|---|---|---|
| ° | ° | ° | ° | ° | ° | ° | ° |
| − 50 | − 58·0 | − 6 | 21·2 | 38 | 100·4 | 82 | 179·6 |
| − 49 | − 56·2 | − 5 | 23·0 | 39 | 102·2 | 83 | 181·4 |
| − 48 | − 54·4 | − 4 | 24·8 | 40 | 104·0 | 84 | 183·2 |
| − 47 | − 52·6 | − 3 | 26·6 | 41 | 105·8 | 85 | 185·0 |
| − 46 | − 50·8 | − 2 | 28·4 | 42 | 107·6 | 86 | 186·8 |
| − 45 | − 49·0 | − 1 | 30·2 | 43 | 109·4 | 87 | 188·6 |
| − 44 | − 47·2 | 0 | 32·0 | 44 | 111·2 | 88 | 190·4 |
| − 43 | − 45·4 | + 1 | 33·8 | 45 | 113·0 | 89 | 192·2 |
| − 42 | − 43·6 | 2 | 35·6 | 46 | 114·8 | 90 | 194·0 |
| − 41 | − 41·8 | 3 | 37·4 | 47 | 116·6 | 91 | 195·8 |
| − 40 | − 40·0 | 4 | 39·2 | 48 | 118·4 | 92 | 197·6 |
| − 39 | − 38·2 | 5 | 41·0 | 49 | 120·2 | 93 | 199·4 |
| − 38 | − 36·4 | 6 | 42·8 | 50 | 122·0 | 94 | 201·2 |
| − 37 | − 34·6 | 7 | 44·6 | 51 | 123·8 | 95 | 203·0 |
| − 36 | − 32·8 | 8 | 46·4 | 52 | 125·6 | 96 | 204·8 |
| − 35 | − 30·0 | 9 | 48·2 | 53 | 127·4 | 97 | 206·6 |
| − 34 | − 29·2 | 10 | 50·0 | 54 | 129·2 | 98 | 208·4 |
| − 33 | − 27·4 | 11 | 51·8 | 55 | 131·0 | 99 | 210·2 |
| − 32 | − 25·6 | 12 | 53·6 | 56 | 132·8 | 100 | 212·0 |
| − 31 | − 23·8 | 13 | 55·4 | 57 | 134·6 | 101 | 213·8 |
| − 30 | − 22·0 | 14 | 57·2 | 58 | 136·4 | 102 | 215·6 |
| − 29 | − 20·2 | 15 | 59·0 | 59 | 138·2 | 103 | 217·4 |
| − 28 | − 18·4 | 16 | 60·8 | 60 | 140·0 | 104 | 219·2 |
| − 27 | − 16·6 | 17 | 62·6 | 61 | 141·8 | 105 | 221·0 |
| − 26 | − 14·8 | 18 | 64·4 | 62 | 143·6 | 106 | 222·8 |
| − 25 | − 13·0 | 19 | 66·2 | 63 | 145·4 | 107 | 224·6 |
| − 24 | − 11·2 | 20 | 68·0 | 64 | 147·2 | 108 | 226·4 |
| − 23 | − 9·4 | 21 | 69·8 | 65 | 149·0 | 109 | 228·2 |
| − 22 | − 7·6 | 22 | 71·6 | 66 | 150·8 | 110 | 230·0 |
| − 21 | − 5·8 | 23 | 73·4 | 67 | 152·6 | 111 | 231·8 |
| − 20 | − 4·0 | 24 | 75·2 | 68 | 154·4 | 112 | 233·6 |
| − 19 | − 2·2 | 25 | 77·0 | 69 | 156·2 | 113 | 235·4 |
| − 18 | − 0·4 | 26 | 78·8 | 70 | 158·0 | 114 | 237·2 |
| − 17 | + 1·4 | 27 | 80·6 | 71 | 159·8 | 115 | 239·0 |
| − 16 | 3·2 | 28 | 82·4 | 72 | 161·6 | 116 | 240·8 |
| − 15 | 5·0 | 29 | 84·2 | 73 | 163·4 | 117 | 242·6 |
| − 14 | 6·8 | 30 | 86·0 | 74 | 165·2 | 118 | 244·4 |
| − 13 | 8·6 | 31 | 87·8 | 75 | 167·0 | 119 | 246·2 |
| − 12 | 10·4 | 32 | 89·6 | 76 | 168·8 | 120 | 248·0 |
| − 11 | 12·2 | 33 | 91·4 | 77 | 170·6 | 121 | 249·8 |
| − 10 | 14·0 | 34 | 93·2 | 78 | 172·4 | 122 | 251·6 |
| − 9 | 15·8 | 35 | 95·0 | 79 | 174·2 | 123 | 253·4 |
| − 8 | 17·6 | 36 | 95·8 | 80 | 176·0 | 124 | 255·2 |
| − 7 | 19·4 | 37 | 98·6 | 81 | 177·8 | 125 | 257·0 |

## Conversion of Degrees, etc.—*continued.*

| Cent. | Fahr. | Cent. | Fahr. | Cent. | Fahr. | Cent. | Fahr. |
|---|---|---|---|---|---|---|---|
| ° | ° | ° | ° | ° | ° | ° | ° |
| 126 | 258·8 | 175 | 347·0 | 224 | 435·2 | 273 | 523·4 |
| 127 | 260·6 | 176 | 348·8 | 225 | 437·0 | 274 | 525·2 |
| 128 | 262·4 | 177 | 350·6 | 226 | 438·8 | 275 | 527·0 |
| 129 | 264·2 | 178 | 352·4 | 227 | 440·6 | 276 | 528·8 |
| 130 | 266·0 | 179 | 354·2 | 228 | 442·4 | 277 | 530·6 |
| 131 | 267·8 | 180 | 356·0 | 229 | 444·2 | 278 | 532·4 |
| 132 | 269·6 | 181 | 357·8 | 230 | 446·0 | 279 | 534·2 |
| 133 | 271·4 | 182 | 359·6 | 231 | 447·8 | 280 | 536·0 |
| 134 | 273·2 | 183 | 361·4 | 232 | 449·6 | 281 | 537·8 |
| 135 | 275·0 | 184 | 363·2 | 233 | 451·4 | 282 | 539·6 |
| 136 | 276·8 | 185 | 365·0 | 234 | 453·2 | 283 | 541·4 |
| 137 | 278·6 | 186 | 366·8 | 235 | 455·0 | 284 | 543·2 |
| 138 | 280·4 | 187 | 368·6 | 236 | 456·8 | 285 | 545·0 |
| 139 | 282·2 | 188 | 370·4 | 237 | 458·6 | 286 | 546·8 |
| 140 | 284·0 | 189 | 372·2 | 238 | 460·4 | 287 | 548·6 |
| 141 | 285·8 | 190 | 374·0 | 239 | 462·2 | 288 | 550·4 |
| 142 | 287·6 | 191 | 375·8 | 240 | 464·0 | 289 | 552·2 |
| 143 | 289·4 | 192 | 377·6 | 241 | 465·8 | 290 | 554·0 |
| 144 | 291·2 | 193 | 379·4 | 242 | 467·6 | 291 | 555·8 |
| 145 | 293·0 | 194 | 381·2 | 243 | 469·4 | 292 | 557·6 |
| 146 | 294·8 | 195 | 383·0 | 244 | 471·2 | 293 | 559·4 |
| 147 | 296·6 | 196 | 384·8 | 245 | 473·0 | 294 | 561·2 |
| 148 | 298·4 | 197 | 386·6 | 246 | 474·8 | 295 | 563·0 |
| 149 | 300·2 | 198 | 388·4 | 247 | 476·6 | 296 | 564·8 |
| 150 | 302·0 | 199 | 390·2 | 248 | 478·4 | 297 | 566·6 |
| 151 | 303·8 | 200 | 392·0 | 249 | 480·2 | 298 | 568·4 |
| 152 | 305·6 | 201 | 393·8 | 250 | 482·0 | 299 | 570·2 |
| 153 | 307·4 | 202 | 395·6 | 251 | 483·8 | 300 | 572·0 |
| 154 | 309·2 | 203 | 397·4 | 252 | 485·6 | 301 | 573·8 |
| 155 | 311·0 | 204 | 399·2 | 253 | 487·4 | 302 | 575·6 |
| 156 | 312·8 | 205 | 401·0 | 254 | 489·2 | 303 | 577·4 |
| 157 | 314·6 | 206 | 402·8 | 255 | 491·0 | 304 | 579·2 |
| 158 | 316·4 | 207 | 404·6 | 256 | 492·8 | 305 | 581·0 |
| 159 | 318·2 | 208 | 406·4 | 257 | 494·6 | 306 | 582·8 |
| 160 | 320·0 | 209 | 408·2 | 258 | 496·4 | 307 | 584·6 |
| 161 | 321·8 | 210 | 410·0 | 259 | 498·2 | 308 | 586·4 |
| 162 | 323·6 | 211 | 411·8 | 260 | 500·0 | 309 | 588·2 |
| 163 | 325·4 | 212 | 413·6 | 261 | 501·8 | 310 | 590·0 |
| 164 | 327·2 | 213 | 415·4 | 262 | 503·6 | 311 | 591·8 |
| 165 | 329·0 | 214 | 417·2 | 263 | 505·4 | 312 | 593·6 |
| 166 | 330·8 | 215 | 419·0 | 264 | 507·2 | 313 | 595·4 |
| 167 | 332·6 | 216 | 420·8 | 265 | 509·0 | 314 | 597·2 |
| 168 | 334·4 | 217 | 422·6 | 266 | 510·8 | 315 | 599·0 |
| 169 | 336·2 | 218 | 424·4 | 267 | 512·6 | 316 | 600·8 |
| 170 | 338·0 | 219 | 426·2 | 268 | 514·4 | 317 | 602·6 |
| 171 | 339·8 | 220 | 428·0 | 269 | 516·2 | 318 | 604·4 |
| 172 | 341·6 | 221 | 429·8 | 270 | 518·0 | 319 | 606·2 |
| 173 | 343·4 | 222 | 431·6 | 271 | 519·8 | 320 | 608·0 |
| 174 | 345·2 | 223 | 433·4 | 272 | 521·6 |  |  |

## SOLUBILITY OF SULPHUR IN CARBON BISULPHIDE AT 15° C. (59° F.).

| Specific Gravity. | Per cent. S. | Specific Gravity. | Per cent. S. | Specific Gravity. | Per cent. S. | Specific Gravity. | Per cent. S. | Specific Gravity. | Per cent. S. |
|---|---|---|---|---|---|---|---|---|---|
| 1·271 | 0 | 1·296 | 6·0 | 1·321 | 12·1 | 1·346 | 18·1 | 1·371 | 25·6 |
| 1·272 | 0·2 | 1·297 | 6·3 | 1·322 | 12·3 | 1·347 | 18·4 | 1·372 | 26·0 |
| 1·273 | 0·4 | 1·298 | 6·5 | 1·323 | 12·6 | 1·348 | 18·6 | 1·373 | 26·5 |
| 1·274 | 0·6 | 1·299 | 6·7 | 1·324 | 12·8 | 1·349 | 18·9 | 1·374 | 26·9 |
| 1·275 | 0·9 | 1·300 | 7·0 | 1·325 | 13·1 | 1·350 | 19·0 | 1·375 | 27·4 |
| 1·276 | 1·2 | 1·301 | 7·2 | 1·326 | 13·3 | 1·351 | 19·3 | 1·376 | 28·1 |
| 1·277 | 1·4 | 1·302 | 7·5 | 1·327 | 13·5 | 1·352 | 19·6 | 1·377 | 28·5 |
| 1·278 | 1·6 | 1·303 | 7·8 | 1·328 | 13·8 | 1·353 | 19·9 | 1·378 | 29·0 |
| 1·279 | 1·9 | 1·304 | 8·0 | 1·329 | 14·0 | 1·354 | 20·1 | 1·379 | 29·7 |
| 1·280 | 2·1 | 1·305 | 8·2 | 1·330 | 14·2 | 1·355 | 20·4 | 1·380 | 30·2 |
| 1·281 | 2·4 | 1·306 | 8·5 | 1·331 | 14·5 | 1·356 | 20·6 | 1·381 | 30·8 |
| 1·282 | 2·6 | 1·307 | 8·7 | 1·332 | 14·7 | 1·357 | 21·0 | 1·382 | 31·4 |
| 1·283 | 2·9 | 1·308 | 8·9 | 1·333 | 15·0 | 1·358 | 21·2 | 1·383 | 31·9 |
| 1·284 | 3·1 | 1·309 | 9·2 | 1·334 | 15·2 | 1·359 | 21·5 | 1·384 | 32·6 |
| 1·285 | 3·4 | 1·310 | 9·4 | 1·335 | 15·4 | 1·360 | 21·8 | 1·385 | 33·2 |
| 1·286 | 3·6 | 1·311 | 9·7 | 1·336 | 15·6 | 1·361 | 22·1 | 1·386 | 33·8 |
| 1·287 | 3·9 | 1·312 | 9·9 | 1·337 | 15·9 | 1·362 | 22·3 | 1·387 | 34·5 |
| 1·288 | 4·1 | 1·313 | 10·2 | 1·338 | 16·1 | 1·363 | 22·7 | 1·388 | 35·2 |
| 1·289 | 4·4 | 1·314 | 10·4 | 1·339 | 16·4 | 1·364 | 23·0 | 1·389 | 36·1 |
| 1·290 | 4·6 | 1·315 | 10·6 | 1·340 | 16·6 | 1·365 | 23·2 | 1·390 | 36·7 |
| 1·291 | 4·8 | 1·316 | 10·9 | 1·341 | 16·9 | 1·366 | 23·6 | 1·391 | 37·2 |
| 1·292 | 5·0 | 1·317 | 11·1 | 1·342 | 17·1 | 1·367 | 24·0 | | |
| 1·293 | 5·3 | 1·318 | 11·3 | 1·343 | 17·4 | 1·368 | 24·3 | | |
| 1·294 | 5·6 | 1·319 | 11·6 | 1·344 | 17·6 | 1·369 | 24·8 | Saturated. | |
| 1·295 | 5·8 | 1·320 | 11·8 | 1·345 | 17·9 | 1·370 | 25·1 | | |

## WEIGHTS AND MEASURES.

The corresponding values of the French and English weights and measures are here given. The use of the French or decimal system is strongly recommended by its extreme simplicity. The smaller denominations are obtained by taking a tenth, hundredth, thousandth, &c., of the unit chosen; they are designated by the Latin prefixes deci-, centi-, milli-, &c. The higher denominations are 10 times, 100 times, 1000, &c., times the unit, and are named by the Greek prefixes deca-, hecto-, kilo-, &c. Examples of this will be found in the Tables given below.

The starting-point of the French system is the "metre" (= 39·37 inches); this is the "unit of length." The "unit of measure" is the "litre," which is 1 cubic decimetre. The "unit of weight" is the gram, which is the weight of 1 cubic centimetre of distilled water at 4° C.

The chief conveniences arising from the use of this system are:—

1. That all the different denominations can be written as one; since they are either multiples by ten, or are decimal fractions, of the unit. Thus 5 decagrams, 3 grams, 4 decigrams, 8 milligrams would be written 53·408 grams.

2. That since 1 cubic centimetre of water at 4° C. weighs 1 gram, we may obtain the *weight* of water to be used from the *measure*, by simply converting the measure into cubic centimetres; the number thus obtained will represent at once the corresponding weight of water in grams. Of course this conversion is strictly accurate only when the water is measured at 4° C. But for ordinary purposes the error introduced, when the water is at the temperature of the air, is too small to be of any importance in the preparation of solutions.

The weights and measures most frequently used for chemical purposes are the gram, the millimetre (mm.), the litre, and the cubic centimetre (c.c.) which is $\frac{1}{1000}$ of a litre.

### English Weights and Measures.

*Apothecaries Weight.*

| lb. | | oz. | | drms. | | scruples. | | grains |
|---|---|---|---|---|---|---|---|---|
| 1 | = | 12 | = | 96 | = | 288 | = | 5760 |
| | | 1 | = | 8 | = | 24 | = | 480 |
| | | | | 1 | = | 3 | = | 60 |
| | | | | | | 1 | = | 20 |

*Avoirdupois Weight.*

| lb. | | oz | | drms. | | grains. |
|---|---|---|---|---|---|---|
| 1 | = | 16 | = | 256 | = | 7000 |
| | | 1 | = | 16 | = | 437·5 |
| | | | | 1 | = | 27·343 |

*Imperial Measure.*

| gallon. | | pints. | | fluid oz. | | fluid drms. |
|---|---|---|---|---|---|---|
| 1 | = | 8 | = | 160 | = | 1280 |
| | | 1 | = | 20 | = | 160 |
| | | | | 1 | = | 8 |

1 gallon = 70,000 grains of water at 16°·7 C.
1 fluid ounce = $\frac{1}{20}$ pint = 437·5 „ „ „
1 gallon = 277·280 cubic inches.
1 fluid ounce = 1·733 „ „

### French Weights and Measures.

*Measures of Length.*

| | | metre. | | inches. | | mile. | fur. | yds. | ft. | inches. |
|---|---|---|---|---|---|---|---|---|---|---|
| Millimetre | = | 0·001 | = | ·03937 | = | .. | .. | .. | .. | ·03937 |
| Centimetre | = | 0·01 | = | ·39371 | = | .. | .. | .. | .. | ·39371 |
| Decimetre | = | 0·1 | = | 3·93708 | = | .. | .. | .. | .. | 3·9371 |
| Metre | = | 1·0 | = | 39·37079 | = | .. | .. | .. | 3 | 3·371 |
| Decametre | = | 10·0 | = | 393·70790 | = | .. | .. | 10 | 2 | 9·7 |
| Hectometre | = | 100·0 | = | 3937·07900 | = | .. | .. | 109 | 1 | 1 |
| Kilometre | = | 1000·0 | = | 39370·79000 | = | .. | 4 | 213 | 4 | 10·2 |
| Myriometre | = | 10000·0 | = | 393707·90000 | = | 6 | 1 | 156 | 0 | 6 |

1 inch = ·0254 metre = 2·5399 centimetres.
1 foot = ·3048 „
1 sq. inch = 6·4514 sq. centimetres.

## French Weights and Measures—*continued.*

### *Measures of Capacity.*

1 litre = 1 cubic decimetre.

| | litre. | cubic inches. | pints. |
|---|---|---|---|
| Millimetre, or Cubic Centimetre (c.c.) .. = | ·001 = | ·06103 = | 0·00176 |
| Centilitre .. .. .. = | ·01 = | ·61027 = | 0·01761 |
| Decilitre .. .. .. = | ·1 = | 6·1027 = | 0·17608 |
| Litre .. .. .. .. = | 1·0 = | 61·027 = | 1·76077 |
| Decalitre .. .. .. = | 10·0 = | 610·27 = | 17·60773 |
| Hectolitre .. .. .. = | 100·0 = | 6102·7 = | 176·07734 |
| Kilolitre .. .. .. = | 1000·0 = | 61027·0 = | 1760·77341 |
| Myriolitre .. .. .. = | 10000·0 = | 610270·0 = | 17607·73414 |

1 cubic inch = ·01639 litre.
1 cubic foot = 28·31531 litres.
1 gallon = 4·54336 „

### *Measures of Weight.*

1 gram = the weight of 1 cubic centimetre (c.c.) of water at 4° C.

| | grams. | grains. | Avoirdupois. lbs. | ozs. | drms. |
|---|---|---|---|---|---|
| Milligram .. .. = | ·001 = | 0·01543 | | | |
| Centigram .. .. = | ·01 = | 0·15432 | | | |
| Decigram .. .. = | ·1 = | 1·54323 | | | |
| Gram .. .. .. = | 1·0 = | 15·43235 | | | |
| Decagram .. .. = | 10·0 = | 154·32349 = | 0 | 0 | 5·65 |
| Hectogram .. .. = | 100·0 = | 1543·23488 = | 0 | 3 | 8·5 |
| Kilogram .. .. = | 1000·0 = | 15432·34880 = | 2 | 3 | 5 |
| Myriogram .. .. = | 10000·0 = | 154323·48800 = | 22 | 1 | 2 |

1 grain = 0·0649 gram.
1 oz. (Troy) = 31·1035 grams.
1 lb. (Avoirdupois) = 453·593 „

| Grms. | Grains. | Deci-grms. | Grains. | Centi-grammes. | Grains. | Milli-grammes. | Grains. |
|---|---|---|---|---|---|---|---|
| 1 | 15·4323 | 1 | 1·5432 | 1 | ·1543 | 1 | ·0154 |
| 2 | 30·8646 | 2 | 3·0864 | 2 | ·3086 | 2 | ·0308 |
| 3 | 46·2969 | 3 | 4·6297 | 3 | ·4630 | 3 | ·0463 |
| 4 | 61·7292 | 4 | 6·1730 | 4 | ·6173 | 4 | ·0617 |
| 5 | 77·1615 | 5 | 7·7161 | 5 | ·7716 | 5 | ·0771 |
| 6 | 92·5938 | 6 | 9·2593 | 6 | ·9260 | 6 | ·0926 |
| 7 | 108·0261 | 7 | 10·8026 | 7 | 1·0802 | 7 | ·1080 |
| 8 | 123·4584 | 8 | 12·3458 | 8 | 1·2345 | 8 | ·1234 |
| 9 | 138·8907 | 9 | 13·8890 | 9 | 1·3889 | 9 | ·1389 |

## Relationship between the Metrical System and English Weights and Measures.

A cubic foot of water measures 6¼ gallons, and weighs exactly 1000 ounces; therefore the same relationship exists between the cubic foot and the avoirdupois ounce as between the litre and the gramme. The usual tables giving the weight of a litre of a gas in terms of the gram, may therefore be read as English tables giving the weight of a cubic foot in terms of the avoirdupois ounce.

## Useful Memoranda.

Weight of 1 c.c. of dry hydrogen at 0° C. and 760 mm. = 0·0000896 gram.
Weight of 1 c.c. of dry air at 0° C. and 760 mm. = 0·0012937 gram.
Specific gravity of hydrogen, air as unit = 0·0693.
Specific gravity of air, hydrogen as unit = 14·43.
Weight of 1 c.c. of mercury at 0° C. = 13·596 grams.
Coefficient of expansion for gases = $\frac{1}{273}$ = 0·003665.
Average percentage of oxygen in air, by volume = 20·96.

## Percentage by Volume, corresponding to the Weight in Grains of $CO_2$ per cubic foot of Gas, at 60° Fahr. and 30 in. Bar.

| Percentage by Volume. | Grains of $CO_2$ per cubic foot. | Percentage by Volume. | Grains of $CO_2$ per cubic foot. | Percentage by Volume. | Grains of $CO_2$ per cubic foot. |
|---|---|---|---|---|---|
| 0·1 | 0·817 | 1·1 | 8·987 | 2·1 | 17·157 |
| 0·2 | 1·634 | 1·2 | 9·804 | 2·2 | 17·974 |
| 0·3 | 2·451 | 1·3 | 10·621 | 2·3 | 18·791 |
| 0·4 | 3·268 | 1·4 | 11·438 | 2·4 | 19·608 |
| 0·5 | 4·085 | 1·5 | 12·255 | 2·5 | 20·425 |
| 0·6 | 4·902 | 1·6 | 13·072 | 2·6 | 21·242 |
| 0·7 | 5·719 | 1·7 | 13·889 | 2·7 | 22·059 |
| 0·8 | 6·536 | 1·8 | 14·706 | 2·8 | 22·876 |
| 0·9 | 7·353 | 1·9 | 15·523 | 2·9 | 23·693 |
| 1·0 | 8·170 | 2·0 | 16·340 | 3·0 | 24·510 |

### Percentage by Volume, corresponding to the Weight in Grains of $NH_3$ per cubic foot of Gas, at 60° Fahr. and 30 in. Bar.

| Percentage by Volume. | Grains of $NH_3$ per cubic foot. | Percentage by Volume. | Grains of $NH_3$ per cubic foot. | Percentage by Volume. | Grains of $NH_3$ per cubic foot. |
|---|---|---|---|---|---|
| 0·1 | 0·315 | 1·1 | 3·471 | 2·1 | 6·627 |
| 0·2 | 0·631 | 1·2 | 3·787 | 2·2 | 6·943 |
| 0·3 | 0·946 | 1·3 | 4·102 | 2·3 | 7·258 |
| 0·4 | 1·262 | 1·4 | 4·418 | 2·4 | 7·574 |
| 0·5 | 1·578 | 1·5 | 4·734 | 2·5 | 7·890 |
| 0·6 | 1·893 | 1·6 | 5·049 | 2·6 | 8·205 |
| 0·7 | 2·209 | 1·7 | 5·365 | 2·7 | 8·521 |
| 0·8 | 2·524 | 1·8 | 5·680 | 2·8 | 8·836 |
| 0·9 | 2·840 | 1·9 | 5·996 | 2·9 | 9·152 |
| 1·0 | 3·156 | 2·0 | 6·312 | 3·0 | 9·468 |

### Solubility of Gases in Water (by Volume) at a pressure of 760 mm. = 29·92 in. (*Bunsen.*)

| 1 Vol. of Water dissolves at °C. | Ammonia. | Atmospheric Air. | Carbon Dioxide. | Carbon Monoxide. | Hydrogen. | Nitrogen. | Oxygen. | Sulphuretted Hydrogen. |
|---|---|---|---|---|---|---|---|---|
| ° | | | | | | | | |
| 0 | 1049·6 | 0·02471 | 1·7967 | 0·03287 | 0·0193 | 0·02035 | 0·04114 | 4·3706 |
| 1 | 1020·8 | 0·02406 | 1·7207 | 0·03207 | 0·0193 | 0·01981 | 0·04007 | 4·2874 |
| 2 | 993·3 | 0·02345 | 1·6481 | 0·03131 | 0·0193 | 0·01932 | 0·03907 | 4·2053 |
| 3 | 967·0 | 0·02287 | 1·5787 | 0·03057 | 0·0193 | 0·01884 | 0·03810 | 4·1243 |
| 4 | 941·9 | 0·02237 | 1·5126 | 0·02987 | 0·0193 | 0·01838 | 0·03717 | 4·0442 |
| 5 | 917·9 | 0·02179 | 1·4497 | 0·02920 | 0·0193 | 0·01794 | 0·03628 | 3·9652 |
| 6 | 895·0 | 0·02128 | 1·3901 | 0·02857 | 0·0193 | 0·01752 | 0·03554 | 3·8872 |
| 7 | 873·1 | 0·02080 | 1·3339 | 0·02796 | 0·0193 | 0·01713 | 0·03465 | 3·8103 |
| 8 | 852·1 | 0·02034 | 1·2809 | 0·02739 | 0·0193 | 0·01675 | 0·03389 | 3·7345 |
| 9 | 832·0 | 0·01992 | 1·2311 | 0·02686 | 0·0193 | 0·01640 | 0·03317 | 3·6596 |
| 10 | 812·8 | 0·01953 | 1·1847 | 0·02635 | 0·0193 | 0·01607 | 0·03250 | 3·5858 |
| 11 | 794·3 | 0·01916 | 1·1416 | 0·02588 | 0·0193 | 0·01577 | 0·03189 | 3·5132 |
| 12 | 776·6 | 0·01882 | 1·1018 | 0·02544 | 0·0193 | 0·01549 | 0·03133 | 3·4415 |
| 13 | 759·6 | 0·01851 | 1·0653 | 0·02504 | 0·0193 | 0·01523 | 0·03082 | 3·3708 |
| 14 | 743·1 | 0·01822 | 1·0321 | 0·02466 | 0·0193 | 0·01500 | 0·03034 | 3·3012 |
| 15 | 727·2 | 0·01795 | 1·0020 | 0·02432 | 0·0193 | 0·01478 | 0·02989 | 3·2326 |
| 16 | 711·8 | 0·01771 | 0·9753 | 0·02402 | 0·0193 | 0·01458 | 0·02949 | 3·1651 |
| 17 | 696·9 | 0·01750 | 0·9519 | 0·02374 | 0·0193 | 0·01441 | 0·02914 | 3·0986 |
| 18 | 682·3 | 0·01732 | 0·9318 | 0·02350 | 0·0193 | 0·01426 | 0·02884 | 3·0331 |
| 19 | 668·0 | 0·01717 | 0·9150 | 0·02329 | 0·0193 | 0·01423 | 0·02858 | 2·9687 |
| 20 | 654·0 | 0·01704 | 0·9014 | 0·02312 | 0·0193 | 0·01403 | 0·02838 | 2·9053 |

## Volume and Density of Water at different Temperatures.

(*Mean results of the observations of Kopp, Pierre, Despretz, Hagen, Matthiessen, Weidner, Kremers, and Rossetti.*)

| Temp.<br>° | Sp. gr. of Water (at 0° = 1). | Volume of Water (at 0° = 1). | Sp. gr. of Water (at 4° = 1). | Volume of Water (at 4° = 1). |
|---|---|---|---|---|
| 0 | 1·000000 | 1·000000 | ·999871 | 1·000129 |
| 1 | 1·000057 | 0·999943 | ·999928 | 1·000072 |
| 2 | 1·000098 | ·999902 | ·999969 | 1·000031 |
| 3 | 1·000120 | ·999880 | ·999991 | 1·000009 |
| 4 | 1·000129 | ·999871 | 1·000000 | 1·000000 |
| 5 | 1·000119 | ·999881 | 0·999990 | 1·000010 |
| 6 | 1·000099 | ·999901 | ·999970 | 1·000030 |
| 7 | 1·000062 | ·999938 | ·999933 | 1·000067 |
| 8 | 1·000015 | ·999985 | ·999886 | 1·000114 |
| 9 | 0·999953 | 1·000047 | ·999824 | 1·000176 |
| 10 | ·999876 | 1·000124 | ·999747 | 1·000253 |
| 11 | ·999784 | 1·000216 | ·999655 | 1·000345 |
| 12 | ·999678 | 1·000322 | ·999549 | 1·000451 |
| 13 | ·999559 | 1·000441 | ·999430 | 1·000570 |
| 14 | ·999429 | 1·000572 | ·999299 | 1·000701 |
| 15 | ·999289 | 1·000712 | ·999160 | 1·000841 |
| 16 | ·999131 | 1·000870 | ·999002 | 1·000999 |
| 17 | ·998970 | 1·001031 | ·998841 | 1·001160 |
| 18 | ·998782 | 1·001219 | ·998654 | 1·001348 |
| 19 | ·998588 | 1·001413 | ·998460 | 1·001542 |
| 20 | ·998388 | 1·001615 | ·998259 | 1·001744 |
| 21 | ·998176 | 1·001828 | ·998047 | 1·001957 |
| 22 | ·997953 | 1·002049 | ·997826 | 1·002177 |
| 23 | ·997730 | 1·002276 | ·997601 | 1·002405 |
| 24 | ·997495 | 1·002511 | ·997367 | 1·002641 |
| 25 | ·997249 | 1·002759 | ·997120 | 1·002888 |
| 26 | ·996994 | 1·003014 | ·996866 | 1·003144 |
| 27 | ·996732 | 1·003278 | ·996603 | 1·003408 |
| 28 | ·996460 | 1·003553 | ·996331 | 1·003682 |
| 29 | ·996179 | 1·003835 | ·996051 | 1·003965 |
| 30 | ·995894 | 1·004123 | ·995765 | 1·004253 |
| 35 | ·99431 | 1·00572 | ·99418 | 1·00586 |
| 40 | ·99248 | 1·00757 | ·99235 | 1·00770 |
| 50 | ·98833 | 1·01181 | ·98820 | 1·01195 |
| 60 | ·98351 | 1·01677 | ·98338 | 1·01691 |
| 70 | ·97807 | 1·02243 | ·97794 | 1·02256 |
| 80 | ·97206 | 1·02874 | ·97194 | 1·02887 |
| 90 | ·96568 | 1·03554 | ·96556 | 1·03567 |
| 100 | ·95878 | 1·04300 | ·95865 | 1·04312 |

## COMPARISON OF TWADDELL'S HYDROMETER WITH SPECIFIC GRAVITY.

| Twaddell. | Specific Gravity. | Twaddell. | Specific Gravity. | Twaddell. | Specific Gravity. | Twaddell. | Specific Gravity. | Twaddell. | Specific Gravity. | Twaddell. | Specific Gravity. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 0 | 1·000 | 25 | 1·125 | 52·6 | 1·263 | 81 | 1·405 | 110 | 1·550 | 141 | 1·705 |
| 1 | 1·005 | 26 | 1·130 | 53 | 1·265 | 82 | 1·410 | 111 | 1·555 | 142 | 1·710 |
| 1·4 | 1·007 | 26·8 | 1·134 | 54 | 1·270 | 83 | 1·415 | 112 | 1·560 | 143 | 1·715 |
| 2 | 1·010 | 27 | 1·135 | 54·8 | 1·274 | 84 | 1·420 | 112·6 | 1·563 | 144 | 1·720 |
| 2·8 | 1·014 | 28 | 1·140 | 55 | 1·275 | 84·8 | 1·424 | 113 | 1·565 | 145 | 1·725 |
| 3 | 1·015 | 28·4 | 1·142 | 56 | 1·280 | 85 | 1·425 | 114 | 1·570 | 146 | 1·730 |
| 4 | 1·020 | 29 | 1·145 | 57 | 1·285 | 86 | 1·430 | 115 | 1·575 | 146·4 | 1·732 |
| 4·4 | 1·022 | 30 | 1·150 | 58 | 1·290 | 87 | 1·435 | 116 | 1·580 | 147 | 1·735 |
| 5 | 1·025 | 30·4 | 1·152 | 59 | 1·295 | 87·6 | 1·438 | 117 | 1·585 | 148 | 1·740 |
| 5·8 | 1·029 | 31 | 1·155 | 59·4 | 1·297 | 88 | 1·440 | 118 | 1·590 | 149 | 1·745 |
| 6 | 1·030 | 32 | 1·160 | 60 | 1·300 | 89 | 1·445 | 119 | 1·595 | 150 | 1·750 |
| 7 | 1·035 | 32·4 | 1·162 | 61 | 1·305 | 90 | 1·450 | 119·4 | 1·597 | 150·6 | 1·753 |
| 7·4 | 1·037 | 33 | 1·165 | 61·6 | 1·308 | 90·6 | 1·453 | 120 | 1·600 | 151 | 1·755 |
| 8 | 1·040 | 34 | 1·170 | 62 | 1·310 | 91 | 1·455 | 121 | 1·605 | 152 | 1·760 |
| 9 | 1·045 | 34·2 | 1·171 | 63 | 1·315 | 92 | 1·460 | 122 | 1·610 | 153 | 1·765 |
| 10 | 1·050 | 35 | 1·175 | 64 | 1·320 | 93 | 1·465 | 123 | 1·615 | 154 | 1·770 |
| 10·2 | 1·052 | 36 | 1·180 | 65 | 1·325 | 93·6 | 1·468 | 124 | 1·620 | 155 | 1·775 |
| 11 | 1·055 | 37 | 1·185 | 66 | 1·330 | 94 | 1·470 | 125 | 1·625 | 156 | 1·780 |
| 12 | 1·060 | 38 | 1·190 | 66·4 | 1·332 | 95 | 1·475 | 126 | 1·630 | 157 | 1·785 |
| 13 | 1·065 | 39 | 1·195 | 67 | 1·335 | 96 | 1·480 | 127 | 1·635 | 158 | 1·790 |
| 13·4 | 1·067 | 40 | 1·200 | 68 | 1·340 | 96·6 | 1·483 | 128 | 1·640 | 159 | 1·795 |
| 14 | 1·070 | 41 | 1·205 | 69 | 1·345 | 97 | 1·485 | 129 | 1·645 | 160 | 1·800 |
| 15 | 1·075 | 42 | 1·210 | 70 | 1·350 | 98 | 1·490 | 130 | 1·650 | 161 | 1·805 |
| 16 | 1·080 | 43 | 1·215 | 71 | 1·355 | 99 | 1·495 | 130·4 | 1·652 | 162 | 1·810 |
| 16·6 | 1·083 | 44 | 1·220 | 71·4 | 1·357 | 99·6 | 1·498 | 131 | 1·655 | 163 | 1·815 |
| 17 | 1·085 | 45 | 1·225 | 72 | 1·360 | 100 | 1·500 | 132 | 1·660 | 164 | 1·820 |
| 18 | 1·090 | 46 | 1·230 | 73 | 1·365 | 101 | 1·505 | 133 | 1·665 | 165 | 1·825 |
| 18·2 | 1·091 | 46·2 | 1·231 | 74 | 1·370 | 102 | 1·510 | 134 | 1·670 | 166 | 1·830 |
| 19 | 1·095 | 47 | 1·235 | 75 | 1·375 | 103 | 1·515 | 134·2 | 1·671 | 167 | 1·835 |
| 20 | 1·100 | 48 | 1·240 | 76 | 1·380 | 104 | 1·520 | 135 | 1·675 | 168 | 1·840 |
| 21 | 1·105 | 48·2 | 1·241 | 76·6 | 1·383 | 105 | 1·525 | 136 | 1·680 | 168·4 | 1·842 |
| 21·6 | 1·108 | 49 | 1·245 | 77 | 1·385 | 106 | 1·530 | 137 | 1·685 | 169 | 1·845 |
| 22 | 1·110 | 50 | 1·250 | 78 | 1·390 | 107 | 1·535 | 138 | 1·690 | 170 | 1·850 |
| 23 | 1·115 | 50·4 | 1·252 | 79 | 1·395 | 108 | 1·540 | 138·2 | 1·691 | 171 | 1·855 |
| 23·2 | 1·116 | 51 | 1·255 | 79·4 | 1·397 | 109 | 1·545 | 139 | 1·695 | 172 | 1·860 |
| 24 | 1·120 | 52 | 1·260 | 80 | 1·400 | 109·2 | 1·546 | 140 | 1·700 | 173 | 1·865 |

To convert degrees Twaddell into specific gravity (water = 1·000) multiply the number of degrees by 5, and add 1·000 to the product. To reduce specific gravity (water = 1·000) to degrees Twaddell: deduct 1·000, and divide the remainder by 5.

## BOILING POINTS OF WATER AT DIFFERENT BAROMETRICAL PRESSURES.

| Boiling Point F. | Barometer Inches. | Boiling Point F. | Barometer Inches. | Boiling Point F. | Barometer Inches. |
|---|---|---|---|---|---|
| ° | | ° | | ° | |
| 184 | 16·676 | 195 | 21·124 | 206 | 26·529 |
| 185 | 17·047 | 196 | 21·576 | 207 | 27·068 |
| 186 | 17·421 | 197 | 22·030 | 208 | 27·614 |
| 187 | 17·803 | 198 | 22·498 | 209 | 28·183 |
| 188 | 18·196 | 199 | 22·965 | 210 | 28·744 |
| 189 | 18·593 | 200 | 23·454 | 211 | 29·331 |
| 190 | 18·992 | 201 | 23·937 | 212 | 29·922 |
| 191 | 19·407 | 202 | 24·441 | 213 | 30·516 |
| 192 | 19·822 | 203 | 25·014 | 214 | 31·120 |
| 193 | 20·254 | 204 | 25·468 | 215 | 31·730 |
| 194 | 20·687 | 205 | 25·992 | 216 | 32·350 |

## TEMPERATURE OF STEAM AT HIGH PRESSURES.

| Pressure in Atmospheres of 30 inches of Mercury. | Temp. Fahr. | Pressure in Atmospheres of 30 inches of Mercury. | Temp. Fahr. | Pressure in Atmospheres of 30 inches of Mercury. | Temp. Fahr. |
|---|---|---|---|---|---|
| | ° | | ° | | ° |
| 1 | 212·0 | 8 | 339·4 | 15 | 390·0 |
| 2 | 249·5 | 9 | 348·4 | 16 | 395·4 |
| 3 | 273·3 | 10 | 356·5 | 17 | 400·8 |
| 4 | 291·2 | 11 | 364·3 | 18 | 405·9 |
| 5 | 306·0 | 12 | 371·1 | 19 | 410·7 |
| 6 | 318·2 | 13 | 377·8 | 20 | 415·4 |
| 7 | 329·5 | 14 | 384·0 | | |

## GRAINS OF SULPHUR (CORRECTED) IN 100 CUBIC FEET OF GAS, CALCULATED FROM BARIUM SULPHATE OBTAINED BY GAS REFEREES' METHOD. (*Sugg.*)

| Grains of Barium Sulphate. | Tabular Number. | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | 1060 | 1040 | 1020 | 1000 | 980 | 960 | 940 | 920 |
| | Aerorthometer Number. | | | | | | | |
| | 943 | 962 | 980 | 1000 | 1020 | 1042 | 1064 | 1087 |
| 1 | 2·59 | 2·64 | 2·70 | 2·75 | 2·81 | 2·86 | 2·93 | 2·99 |
| ·2 | 3·11 | 3·17 | 3·24 | 3·30 | 3·37 | 3·44 | 3·51 | 3·59 |
| ·4 | 3·63 | 3·70 | 3·77 | 3·85 | 3·93 | 4·01 | 4·10 | 4·18 |
| ·6 | 4·15 | 4·23 | 4·31 | 4·40 | 4·49 | 4·58 | 4·68 | 4·78 |
| ·8 | 4·67 | 4·76 | 4·85 | 4·95 | 5·05 | 5·16 | 5·27 | 5·38 |
| 2 | 5·19 | 5·29 | 5·39 | 5·50 | 5·61 | 5·73 | 5·85 | 5·98 |
| ·2 | 5·71 | 5·82 | 5·93 | 6·05 | 6·17 | 6·30 | 6·44 | 6·58 |
| ·4 | 6·23 | 6·35 | 6·47 | 6·60 | 6·73 | 6·88 | 7·02 | 7·17 |
| ·6 | 6·74 | 6·87 | 7·01 | 7·15 | 7·30 | 7·45 | 7·61 | 7·77 |
| ·8 | 7·26 | 7·40 | 7·55 | 7·70 | 7·86 | 8·02 | 8·19 | 8·37 |
| 3 | 7·78 | 7·93 | 8·09 | 8·25 | 8·42 | 8·59 | 8·78 | 8·97 |
| ·2 | 8·30 | 8·46 | 8·63 | 8·80 | 8·98 | 9·16 | 9.36 | 9·57 |
| ·4 | 8·81 | 8·98 | 9·17 | 9·35 | 9·54 | 9·73 | 9·95 | 10·16 |
| ·6 | 9·33 | 9·51 | 9·71 | 9·90 | 10·10 | 10·30 | 10·53 | 10·76 |
| ·8 | 9·85 | 10·04 | 10·24 | 10·45 | 10·66 | 10·88 | 11·12 | 11·36 |
| 4 | 10·38 | 10·58 | 10·78 | 11·00 | 11·22 | 11·46 | 11·70 | 11·96 |
| ·2 | 10·90 | 11·11 | 11·32 | 11·55 | 11·78 | 12·03 | 12·28 | 12·56 |
| ·4 | 11·41 | 11·63 | 11·86 | 12·10 | 12·31 | 12·60 | 12·87 | 13·16 |
| ·6 | 11·93 | 12·16 | 12·40 | 12·65 | 12·90 | 13·17 | 13·45 | 13·75 |
| ·8 | 12·45 | 12·69 | 12·94 | 13·20 | 13·46 | 13·74 | 14·04 | 14·35 |
| 5 | 12·97 | 13·22 | 13·48 | 13·75 | 14·03 | 14·32 | 14·63 | 14·95 |
| ·2 | 13·49 | 13·74 | 14·02 | 14·30 | 14·59 | 14·89 | 15·21 | 15·54 |
| ·4 | 14·00 | 14·27 | 14·56 | 14·85 | 15·15 | 15·46 | 15·80 | 16·14 |
| ·6 | 14·52 | 14·80 | 15·10 | 15·40 | 15·71 | 16·03 | 16·38 | 16·74 |
| ·8 | 15·04 | 15·33 | 15·64 | 15·95 | 16·27 | 16·60 | 16·97 | 17·33 |
| 6 | 15·56 | 15·86 | 16·18 | 16·50 | 16·84 | 17·18 | 17·55 | 17·93 |
| ·2 | 16·07 | 16·38 | 16·72 | 17·05 | 17·40 | 17·76 | 18·14 | 18·53 |
| ·4 | 16·59 | 16·91 | 17·25 | 17·60 | 17·97 | 18·33 | 18·72 | 19·12 |
| ·6 | 17·11 | 17·44 | 17·79 | 18·15 | 18·53 | 18·90 | 19·31 | 19·72 |
| ·8 | 17·63 | 17·97 | 18·33 | 18·70 | 19·09 | 19·47 | 19·89 | 20·32 |
| 7 | 18·16 | 18·51 | 18·87 | 19·25 | 19·64 | 20·05 | 20·48 | 20·92 |
| ·2 | 18·67 | 19·04 | 19·41 | 19·80 | 20·20 | 20·62 | 21·06 | 21·52 |
| ·4 | 19·19 | 19·57 | 19·95 | 20·35 | 20·76 | 21·19 | 21·65 | 22·12 |
| ·6 | 19·71 | 20·10 | 20·49 | 20·90 | 21·32 | 21·77 | 22·23 | 22·72 |
| ·8 | 20·23 | 20·63 | 21·03 | 21·45 | 21·88 | 22·34 | 22·82 | 23·31 |

Grains of Sulphur, etc.—*continued.*

| Grains of Barium Sulphate. | Tabular Number. | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | 1060 | 1040 | 1020 | 1000 | 980 | 960 | 940 | 920 |
| | Aerorthometer Number. | | | | | | | |
| | 943 | 962 | 980 | 1000 | 1020 | 1042 | 1064 | 1087 |
| 8 | 20·75 | 21·15 | 21·57 | 22·00 | 22·45 | 22·91 | 23·40 | 23·91 |
| ·2 | 21·27 | 21·68 | 22·11 | 22·55 | 23·01 | 23·49 | 23·99 | 24·51 |
| ·4 | 21·79 | 22·21 | 22·65 | 23·10 | 23·57 | 24·06 | 24·57 | 25·11 |
| ·6 | 22·31 | 22·74 | 23·19 | 23·65 | 24·13 | 24·63 | 25·16 | 25·71 |
| ·8 | 22·83 | 23·27 | 23·72 | 24·20 | 24·69 | 25·20 | 25·74 | 26·30 |
| 9 | 23·35 | 23·80 | 24·26 | 24·75 | 25·25 | 25·78 | 26·33 | 26·90 |
| ·2 | 23·87 | 24·33 | 24·80 | 25·30 | 25·82 | 26·35 | 26·91 | 27·50 |
| ·4 | 24·38 | 24·85 | 25·34 | 25·85 | 26·37 | 26·92 | 27·50 | 28·10 |
| ·6 | 24·90 | 25·38 | 25·88 | 26·40 | 26·94 | 27·50 | 28·08 | 28·70 |
| ·8 | 25·42 | 25·91 | 26·42 | 26·95 | 27·50 | 28·07 | 28·67 | 29·29 |
| 10 | 25·94 | 26·44 | 26·96 | 27·50 | 28·06 | 28·64 | 29·25 | 29·89 |
| ·2 | 26·46 | 26·97 | 27·50 | 28·05 | 28·62 | 29·21 | 29·84 | 30·49 |
| ·4 | 26·98 | 27·50 | 28·04 | 28·60 | 29·18 | 29·79 | 30·42 | 31·09 |
| ·6 | 27·50 | 28·03 | 28·58 | 29·15 | 29·74 | 30·36 | 31·01 | 31·68 |
| ·8 | 28·02 | 28·56 | 29·12 | 29·70 | 30·30 | 30·93 | 31·59 | 32·28 |
| 11 | 28·53 | 29·08 | 29·66 | 30·25 | 30·87 | 31·50 | 32·18 | 32·88 |
| ·2 | 29·05 | 29·61 | 30·19 | 30·80 | 31·43 | 32·08 | 32·76 | 33·48 |
| ·4 | 29·57 | 30·14 | 30·73 | 31·35 | 31·99 | 32·65 | 33·35 | 34·08 |
| ·6 | 30·09 | 30·67 | 31·27 | 31·90 | 32·55 | 33·22 | 33·93 | 34·67 |
| ·8 | 30·61 | 31·20 | 31·81 | 32·45 | 33·11 | 33·80 | 34·52 | 35·27 |
| 12 | 31·13 | 31·73 | 32·35 | 33·00 | 33·67 | 34·37 | 35·10 | 35·87 |
| ·2 | 31·65 | 32·26 | 32·89 | 33·55 | 34·23 | 34·94 | 35·69 | 36·47 |
| ·4 | 32·17 | 32·79 | 33·43 | 34·10 | 34·79 | 35·52 | 36·27 | 37·07 |
| ·6 | 32·69 | 33·32 | 33·97 | 34·65 | 35·35 | 36·09 | 36·86 | 37·66 |
| ·8 | 33·20 | 33·85 | 34·51 | 35·20 | 35·91 | 36·66 | 37·44 | 38·26 |
| 13 | 33·72 | 34·37 | 35·05 | 35·75 | 36·48 | 37·23 | 38·03 | 38·86 |
| ·2 | 34·24 | 34·90 | 35·59 | 36·30 | 37·04 | 37·81 | 38·61 | 39·46 |
| ·4 | 34·76 | 35·43 | 36·13 | 36·85 | 37·60 | 38·38 | 39·20 | 40·06 |
| ·6 | 35·28 | 35·96 | 36·66 | 37·40 | 38·16 | 38·95 | 39·78 | 40·65 |
| ·8 | 35·80 | 36·49 | 37·20 | 37·95 | 38·72 | 39·53 | 40·37 | 41·25 |
| 14 | 36·32 | 37·02 | 37·74 | 38·50 | 39·28 | 40·10 | 40·95 | 41·85 |
| ·2 | 36·84 | 37·55 | 38·28 | 39·05 | 39·84 | 40·67 | 41·54 | 42·45 |
| ·4 | 37·35 | 38·07 | 38·82 | 39·60 | 40·40 | 41·25 | 42·12 | 43·05 |
| ·6 | 37·87 | 38·60 | 39·36 | 40·15 | 40·96 | 41·82 | 42·71 | 43·64 |
| ·8 | 38·39 | 39·13 | 39·90 | 40·70 | 41·52 | 42·39 | 43·29 | 44·24 |
| 15 | 38·91 | 39·66 | 40·44 | 41·25 | 42·09 | 42·96 | 43·88 | 44·84 |

## TENSION OF AQUEOUS VAPOUR IN INCHES OF MERCURY FROM 1° TO 100° F.

| Temperature Fahrenheit. | Inches of Mercury. | Temperature Fahrenheit. | Inches of Mercury. | Temperature Fahrenheit. | Inches of Mercury. |
|---|---|---|---|---|---|
| 1° | ·046 | 36° | ·212 | 71° | ·759 |
| 2 | ·048 | 37 | ·220 | 72 | ·785 |
| 3 | ·050 | 38 | ·229 | 73 | ·812 |
| 4 | ·052 | 39 | ·238 | 74 | ·840 |
| 5 | ·054 | 40 | ·247 | 75 | ·868 |
| 6 | ·057 | 41 | ·257 | 76 | ·897 |
| 7 | ·060 | 42 | ·267 | 77 | ·927 |
| 8 | ·062 | 43 | ·277 | 78 | ·958 |
| 9 | ·065 | 44 | ·288 | 79 | ·990 |
| 10 | ·068 | 45 | ·299 | 80 | 1·023 |
| 11 | ·071 | 46 | ·311 | 81 | 1·057 |
| 12 | ·074 | 47 | ·323 | 82 | 1·092 |
| 13 | ·078 | 48 | ·335 | 83 | 1·128 |
| 14 | ·082 | 49 | ·348 | 84 | 1·165 |
| 15 | ·086 | 50 | ·361 | 85 | 1·203 |
| 16 | ·090 | 51 | ·374 | 86 | 1·242 |
| 17 | ·094 | 52 | ·388 | 87 | 1·282 |
| 18 | ·098 | 53 | ·403 | 88 | 1·323 |
| 19 | ·103 | 54 | ·418 | 89 | 1·366 |
| 20 | ·108 | 55 | ·433 | 90 | 1·401 |
| 21 | ·113 | 56 | ·449 | 91 | 1·455 |
| 22 | ·118 | 57 | ·465 | 92 | 1·501 |
| 23 | ·123 | 58 | ·482 | 93 | 1·548 |
| 24 | ·129 | 59 | ·500 | 94 | 1·596 |
| 25 | ·135 | 60 | ·518 | 95 | 1·646 |
| 26 | ·141 | 61 | ·537 | 96 | 1·697 |
| 27 | ·147 | 62 | ·556 | 97 | 1·751 |
| 28 | ·153 | 63 | ·576 | 98 | 1·806 |
| 29 | ·160 | 64 | ·596 | 99 | 1·862 |
| 30 | ·167 | 65 | ·617 | 100 | 1·918 |
| 31 | ·174 | 66 | ·639 | | |
| 32 | ·181 | 67 | ·661 | | |
| 33 | ·188 | 68 | ·685 | | |
| 34 | ·196 | 69 | ·708 | | |
| 35 | ·204 | 70 | ·733 | | |

## TENSION OF AQUEOUS VAPOUR FOR EACH TENTH OF A DEGREE CENTIGRADE FROM 0° TO 30° C. (*Regnault.*)

| Temp. C. | Tension in mm. of Mercury. | Temp. C. | Tension in mm. of Mercury. | Temp. C. | Tension in mm. of Mercury. | Temp. C. | Tension in mm. of Mercury. |
|---|---|---|---|---|---|---|---|
| 0·0 | 4·6 | 3·0 | 5·7 | 6·0 | 7·0 | 9·0 | 8·6 |
| ·1 | 4·6 | ·1 | 5·7 | ·1 | 7·0 | ·1 | 8·6 |
| ·2 | 4·7 | ·2 | 5·8 | ·2 | 7·1 | ·2 | 8·7 |
| ·3 | 4·7 | ·3 | 5·8 | ·3 | 7·1 | ·3 | 8·7 |
| ·4 | 4·7 | ·4 | 5·8 | ·4 | 7·2 | ·4 | 8·8 |
| ·5 | 4·8 | ·5 | 5·9 | ·5 | 7·2 | ·5 | 8·9 |
| ·6 | 4·8 | ·6 | 5·9 | ·6 | 7·3 | ·6 | 8·9 |
| ·7 | 4·8 | ·7 | 6·0 | ·7 | 7·3 | ·7 | 9·0 |
| ·8 | 4·9 | ·8 | 6·0 | ·8 | 7·4 | ·8 | 9·0 |
| ·9 | 4·9 | ·9 | 6·1 | ·9 | 7·4 | ·9 | 9·1 |
| ·0 | 4·9 | 4·0 | 6·1 | 7·0 | 7·5 | 10·0 | 9·2 |
| ·1 | 5·0 | ·1 | 6·1 | ·1 | 7·5 | ·1 | 9·2 |
| ·2 | 5·0 | ·2 | 6·2 | ·2 | 7·6 | ·2 | 9·3 |
| ·3 | 5·0 | ·3 | 6·2 | ·3 | 7·6 | ·3 | 9·3 |
| ·4 | 5·1 | ·4 | 6·3 | ·4 | 7·7 | ·4 | 9·4 |
| ·5 | 5·1 | ·5 | 6·3 | ·5 | 7·8 | ·5 | 9·5 |
| ·6 | 5·2 | ·6 | 6·4 | ·6 | 7·8 | ·6 | 9·5 |
| ·7 | 5·2 | ·7 | 6·4 | ·7 | 7·9 | ·7 | 9·6 |
| ·8 | 5·2 | ·8 | 6·4 | ·8 | 7·9 | ·8 | 9·7 |
| ·9 | 5·3 | ·9 | 6·5 | ·9 | 8·0 | ·9 | 9·7 |
| 2·0 | 5·3 | 5·0 | 6·5 | 8·0 | 8·0 | 11·0 | 9·8 |
| ·1 | 5·3 | ·1 | 6·6 | ·1 | 8·1 | ·1 | 9·9 |
| ·2 | 5·4 | ·2 | 6·6 | ·2 | 8·1 | ·2 | 9·9 |
| ·3 | 5·4 | ·3 | 6·7 | ·3 | 8·2 | ·3 | 10·0 |
| ·4 | 5·5 | ·4 | 6·7 | ·4 | 8·2 | ·4 | 10·1 |
| ·5 | 5·5 | ·5 | 6·8 | ·5 | 8·3 | ·5 | 10·1 |
| ·6 | 5·5 | ·6 | 6·8 | ·6 | 8·3 | ·6 | 10·2 |
| 7 | 5·6 | ·7 | 6·9 | ·7 | 8·4 | ·7 | 10·3 |
| ·8 | 5·6 | ·8 | 6·9 | ·8 | 8·5 | ·8 | 10·3 |
| ·9 | 5·6 | ·9 | 7·0 | ·9 | 8·5 | ·9 | 10·4 |

TENSION OF AQUEOUS VAPOUR—*continued.*

| Temp. C. | Tension in mm. of Mercury. | Temp. C. | Tension in mm. of Mercury. | Temp. C. | Tension in mm. of Mercury. | Temp. C. | Tension in mm. of Mercury. |
|---|---|---|---|---|---|---|---|
| 12·0 | 10·5 | 15·0 | 12·7 | 18·0 | 15·4 | 21·0 | 18·5 |
| ·1 | 10·5 | ·1 | 12·8 | ·1 | 15·5 | ·1 | 18·6 |
| ·2 | 10·6 | ·2 | 12·9 | ·2 | 15·6 | ·2 | 18·7 |
| ·3 | 10·7 | ·3 | 12·9 | ·3 | 15·7 | ·3 | 18·8 |
| ·4 | 10·7 | ·4 | 13·0 | ·4 | 15·7 | ·4 | 19·0 |
| ·5 | 10·8 | ·5 | 13·1 | ·5 | 15·8 | ·5 | 19·1 |
| ·6 | 10·9 | ·6 | 13·2 | ·6 | 15·9 | ·6 | 19·2 |
| ·7 | 10·9 | ·7 | 13·3 | ·7 | 16·0 | ·7 | 19·3 |
| ·8 | 11·0 | ·8 | 13·4 | ·8 | 16·1 | ·8 | 19·4 |
| ·9 | 11·1 | ·9 | 13·5 | ·9 | 16·2 | ·9 | 19·5 |
| 13·0 | 11·2 | 16·0 | 13·5 | 19·0 | 16·3 | 22·0 | 19·7 |
| ·1 | 11·2 | ·1 | 13·6 | ·1 | 16·4 | ·1 | 19·8 |
| ·2 | 11·3 | ·2 | 13·7 | ·2 | 16·6 | ·2 | 19·9 |
| ·3 | 11·4 | ·3 | 13·8 | ·3 | 16·7 | ·3 | 20·0 |
| ·4 | 11·5 | ·4 | 13·9 | ·4 | 16·8 | ·4 | 20·1 |
| ·5 | 11·5 | ·5 | 14·0 | ·5 | 16·9 | ·5 | 20·3 |
| ·6 | 11·6 | ·6 | 14·1 | ·6 | 17·0 | ·6 | 20·4 |
| ·7 | 11·7 | ·7 | 14·2 | ·7 | 17·1 | ·7 | 20·5 |
| ·8 | 11·8 | ·8 | 14·2 | ·8 | 17·2 | ·8 | 20·6 |
| ·9 | 11·8 | ·9 | 14·3 | ·9 | 17·3 | ·9 | 20·8 |
| 14·0 | 11·9 | 17·0 | 14·4 | 20·0 | 17·4 | 23·0 | 20·9 |
| ·1 | 12·0 | ·1 | 14·5 | ·1 | 17·5 | ·1 | 21·0 |
| ·2 | 12·1 | ·2 | 14·6 | ·2 | 17·6 | ·2 | 21·1 |
| ·3 | 12·1 | ·3 | 14·7 | ·3 | 17·7 | ·3 | 21·3 |
| ·4 | 12·2 | ·4 | 14·8 | ·4 | 17·8 | ·4 | 21·4 |
| ·5 | 12·3 | ·5 | 14·9 | ·5 | 17·9 | ·5 | 21·5 |
| ·6 | 12·4 | ·6 | 15·0 | ·6 | 18·0 | ·6 | 21·7 |
| ·7 | 12·5 | ·7 | 15·1 | ·7 | 18·2 | ·7 | 21·8 |
| ·8 | 12·5 | ·8 | 15·2 | ·8 | 18·3 | ·8 | 21·9 |
| ·9 | 12·6 | ·9 | 15·3 | ·9 | 18·4 | ·9 | 22·1 |

## TENSION OF AQUEOUS VAPOUR, ETC.—*continued.*

| Temp. C. | Tension in mm. of Mercury. | Temp. C. | Tension in mm. of Mercury. | Temp. C. | Tension in mm. of Mercury. |
|---|---|---|---|---|---|
| 24·0 | 22·2 | 26·0 | 25·0 | 28·0 | 28·1 |
| ·1 | 22·3 | ·1 | 25·1 | ·1 | 28·3 |
| ·2 | 22·5 | ·2 | 25·3 | ·2 | 28·4 |
| ·3 | 22·6 | ·3 | 25·4 | ·3 | 28·6 |
| ·4 | 22·7 | ·4 | 25·6 | ·4 | 28·8 |
| ·5 | 22·9 | ·5 | 25·7 | ·5 | 28·9 |
| ·6 | 23·0 | ·6 | 25·9 | ·6 | 29·1 |
| ·7 | 23·1 | ·7 | 26·0 | ·7 | 29·3 |
| ·8 | 23·3 | ·8 | 26·2 | ·8 | 29·4 |
| ·9 | 23·4 | ·9 | 26·4 | ·9 | 29·6 |
| 25·0 | 23·5 | 27·0 | 26·5 | 29·0 | 29·8 |
| ·1 | 23·7 | ·1 | 26·7 | ·1 | 30·0 |
| ·2 | 23·8 | ·2 | 26·8 | ·2 | 30·1 |
| ·3 | 24·0 | ·3 | 27·0 | ·3 | 30·3 |
| ·4 | 24·1 | ·4 | 27·1 | ·4 | 30·5 |
| ·5 | 24·3 | ·5 | 27·3 | ·5 | 30·7 |
| ·6 | 24·4 | ·6 | 27·5 | ·6 | 30·8 |
| ·7 | 24·6 | ·7 | 27·6 | ·7 | 31·0 |
| ·8 | 24·7 | ·8 | 27·8 | ·8 | 31·2 |
| ·9 | 24·8 | ·9 | 27·9 | ·9 | 31·4 |

## SPECIFIC GRAVITY OF VARIOUS GASES.

| Name of Gas. | Molecular Weight. | Experimental Specific Gravity. Air = 1. | Weight of 1 cubic foot in grains at 60° F. and 30·0° Bar. | Number of cubic feet equal to 1 lb. |
|---|---|---|---|---|
| Hydrogen, $H_2$ .. .. .. | 2 | 0·06926 | 37·15 | 188·42 |
| Marsh gas, $CH_4$ .. .. .. | 16 | 0·558 | 297·20 | 23·55 |
| Ammonia, $NH_3$ .. .. .. | 17 | 0·597 | 315·77 | 22·17 |
| Carbonic oxide, CO .. .. | 28 | 0·9678 | 520·10 | 13·46 |
| Olefiant gas, $C_2H_4$ .. .. | 28 | 0·971 | 520·10 | 13·46 |
| Nitrogen, $N_2$ .. .. .. .. | 28 | 0·97137 | 520·10 | 13·46 |
| Air .. .. .. .. .. .. | .. | 1·000 | 535·96 | 13·03 |
| Oxygen, $O_2$ .. .. .. .. | 32 | 1·10563 | 594·40 | 11·77 |
| Sulphuretted hydrogen, $SH_2$ | 34 | 1·1912 | 631·54 | 11·09 |
| Carbonic acid, $CO_2$ .. .. | 44 | 1·529 | 817·30 | 8·56 |
| Water vapour, $H_2O$ .. .. | 18 | 0·615 | 334·35 | 20·93 |
| Bisulphide of carbon, $CS_2$ .. | 76 | 2·640 | 1411·70 | 4·95 |

In order to obtain the weight of a cubic foot of any gas whose molecular formula is known, calculate the molecular weight, and multiply the weight of 1 cubic foot of hydrogen (37·15 grains) by half the molecular weight of the gas; this will give the weight in grains of 1 cubic foot of the gas.

For example, the molecular weight of marsh gas = 16, divide this by 2 = 8, which multiplied by 37·15 = 297·20 grains, as in the table.

When it is not requisite to be strictly accurate, a cubic foot of hydrogen may be taken as weighing 37 grains; this is a number easily remembered, and by means of the same the weights of different gases are easily calculated.

The same rules also apply to the metric system of weights and measures, the standard in this case being the weight of a litre of hydrogen at 0° C. and 760 mm.; this weighs ·08958 gram.

## PRODUCTS OF THE DESTRUCTIVE DISTILLATION OF COAL. (*Mills.*)

| Name. | Formula. | Boiling Point. | Melting Point. |
|---|---|---|---|
| | | ° C. | ° C. |
| Hydrogen | $H_2$ | .. | .. |
| Methylic hydride | $CH_4$ | .. | .. |
| Hexylic ,, | $C_6H_{14}$ | 68 | .. |
| Octylic ,, | $C_8H_{18}$ | 119 | .. |
| Decylic ,, | $C_{10}H_{22}$ | 171 | .. |
| Paraffin | $C_nH_{2n} + 2$ | 400 (?) | .. |
| Ethylene | $C_2H_4$ | − 102·5 | .. |
| Tritylene | $C_3H_6$ | .. | .. |
| Tetrylene | $C_4H_8$ | − 5 | .. |
| Pentylene | $C_5H_{10}$ | 31 | .. |
| Hexylene | $C_6H_{12}$ | 71 | .. |
| Heptylene | $C_7H_{14}$ | 97 | .. |
| Acetylene | $C_2H_2$ | .. | .. |
| Crotonylene | $C_4H_6$ | 25 | .. |
| Terene | $C_5H_8$ | .. | .. |
| Hexoylene | $C_6H_{10}$ | 80 | .. |
| Styrolene | $C_8H_8$ | 145 | .. |
| Thiophene | $C_4H_4S$ | 84 | .. |
| Thiotoluene | $C_5H_6S$ | 113 | .. |
| Thioxene | $C_6H_8S$ | 137 | .. |
| Benzene | $C_6H_6$ | 80 | 5·17 |
| Parabenzene* | $C_6H_6$ | 97 | .. |
| Toluene | $C_7H_8$ | 111 | .. |
| Orthoxylene | $C_8H_{10}$ | 143 | .. |
| Paroxylene | $C_8H_{10}$ | 137 | .. |
| Metaxylene | $C_8H_{10}$ | 137 | .. |
| Cumene | $C_9H_{14}$ | 166 | .. |
| Mesitylene | $C_9H_{12}$ | 163 | .. |
| Cymene | $C_{10}H_{14}$ | 166 | .. |
| Terpene | $C_{10}H_{16}$ | 171 | .. |
| Naphthalin | $C_{10}H_8$ | 218 | 80 |
| Methylnaphthalin | $C_{11}H_{10}$ | 242 | − 18 |
| Naphthalin hydride | $C_{10}H_{10}$ | .. | 210 |
| Naphthols, α, β | $C_{10}H_8O$ | 279, 288 | 94. 33 |
| Phenyl | $C_{12}H_{10}$ | 254 | 70 |
| Acenaphthene | $C_{12}H_{10}$ | 285 | 100 |
| Fluorene | $C_{13}H_{10}$ | 295 | 113 |
| Phenanthracene | $C_{14}H_{10}$ | 340 | 99 |
| Anthracene | $C_{14}H_{10}$ | 360 | 213 |
| Fluoranthrene | $C_{15}H_{10}$ | .. | 109 |
| Pyrene | $C_{16}H_{10}$ | .. | 148 |
| Anthracene hydride | $C_{14}H_{12}$ | 305 | 106 |
| Methylanthracene | $C_{15}H_{12}$ | .. | 200 |
| Chrysene | $C_{18}H_{12}$ | .. | 249 |

* Dipropargyl.

PRODUCTS OF THE DESTRUCTIVE DISTILLATION OF COAL—*continued.*

| Name. | Formula. | Boiling Point. | Melting Point. |
|---|---|---|---|
| | | ° C. | ° C. |
| Retene | $C_{18}H_{18}$ | 350 | 99 |
| Picene (?) | $C_{22}H_{14}$ | 519 | 338 |
| Water | $H_2O$ | 100 | .. |
| Hydric sulphide | $H_2S$ | .. | – 85 |
| ,, cyanide | $HCN$ | 26 | – 18 |
| ,, sulphocyanide | $HCNS$ | .. | .. |
| Carbonic oxide | $CO$ | – 193 | .. |
| ,, dioxide | $CO_2$ | – 78 | .. |
| ,, disulphide | $CS_2$ | 47 | – 110 |
| Sulphuric dioxide | $SO_2$ | – 10 | .. |
| Hydric acetate | $C_2H_4O_2$ | 120 | 15 |
| Acetonitril | $C_2H_3N$ | 77 | .. |
| Ethylic alcohol (?) | $C_2H_6O$ | 78 | – 130·5 |
| Phenol | $C_6H_6O$ | 182 | 42 |
| Hydric benzoate | $C_7H_6O_2$ | 250 | 120 |
| Kreasols, *o. m. p.* | $C_7H_8O$ | 118, 201, 199 | 31 (?), 36 |
| Pyrokreasols, *α*, *β*, *γ* | $C_{28}H_{26}O_2$ | .. | 104, 124, 195 |
| Phlorol | $C_8H_{10}O$ | 219 | .. |
| Ammonia | $NH_3$ | – 33 | – 70 (?) |
| Butylamine | $C_4H_{11}N$ | 75·5 | .. |
| Aniline | $C_6H_7N$ | 182 | – 8 |
| Cespitine | $C_5H_{13}N$ | 96 | .. |
| Pyridine | $C_5H_5N$ | 115 | .. |
| Picoline | $C_6H_7N$ | 134 | .. |
| Lutidine | $C_7H_9N$ | 157 | .. |
| Collidine | $C_8H_{11}N$ | 170 | .. |
| Parvoline | $C_9H_{13}N$ | 188 | .. |
| Coridine | $C_{10}H_{15}N$ | 211 | .. |
| Rubidine | $C_{11}H_{17}N$ | 230 | .. |
| Viridine | $C_{12}H_{19}N$ | 251 | .. |
| Acridine | $C_{13}H_9N$ | 360 | 107 |
| Carbazol | $C_{12}H_9N$ | 355 | 238 |
| Phenylnaphthylcarbazol | $C_{16}H_{11}N$ | .. | 330 |
| Leucoline | $C_9H_7N$ | 220, 238 | .. |
| Lepidine | $C_{10}H_9N$ | 254, 268 | .. |
| Iridoline | .. | .. | .. |
| Cryptidine | $C_{11}H_{11}N$ | 272 | .. |
| Tetracoline | $C_{12}H_{13}N$ | 292 | .. |
| Pentacoline | $C_{13}H_{15}N$ | 312 | .. |
| Hexacoline | $C_{14}H_{17}N$ | 327 | .. |
| Heptacoline | $C_{15}H_{19}N$ | 347 | .. |
| Octacoline | $C_{16}H_{21}N$ | 362 | .. |
| Pyrrhol | $C_4H_5N$ | 133 | .. |
| Carbon (hydrogenated) | $C_n$ | .. | .. |
| Sulphur | $S_2$ | 400 | 115 |
| Nitrogen | $N_2$ | – 193 | – 213 (?) |

## SPECIFIC GRAVITIES OF SOME COAL TAR COMPOUNDS.

(*Mills.*)

| Compound. | Specific Gravity. | | |
|---|---|---|---|
| | At 0° C. | At 15° C. | At 18° C. |
| Benzene .. .. .. .. | ·8991 | ·8846 | — |
| Toluene .. .. .. .. | ·8841 | ·8702 | — |
| Xylene (mixed isomers) .. | ·877 | — | — |
| Naphthalin .. .. .. .. | — | — | 1·158 |
| Carbonic disulphide .. .. | — | 1·272 | — |
| Phenol .. .. .. .. | — | — | 1·065 |
| Aniline .. .. .. .. | 1·036 | — | — |

## CHANGES OF VOLUME WHEN GASES ARE BURNT IN OXYGEN.

| Name of Gas. | Molecular Formula | Combustion requires | | Combustion yields | | Volume of Gas. | | | Contraction. | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Combustible gas. | Oxygen. | Aqueous Vapour (Condensed). | $CO_2$. | Before Combustion. | After Combustion. | After combustion and absorption of $CO_2$. | After Combustion. | Ratio of Combustible Gas to contraction. | After Combustion and absorption of $CO_2$. | Ratio of Combustible Gas to Contraction. |
| | | vols. | vols. | vols. | vols. | vols. | vols. | vols. | vols. | | vols. | |
| Acetylene | $C_2H_2$ | 2 | 5 | 2 | 4 | 7 | 4 | 0 | 3 | $\frac{2}{3}$ | 7 | $\frac{2}{7}$ |
| Benzene.. | $C_6H_6$ | 2 | 15 | 6 | 12 | 17 | 12 | 0 | 5 | $\frac{2}{5}$ | 17 | $\frac{2}{17}$ |
| Butylene | $C_4H_8$ | 2 | 12 | 8 | 8 | 14 | 8 | 0 | 6 | $\frac{1}{3}$ | 14 | $\frac{1}{7}$ |
| Carbonic Oxide .. | CO | 2 | 1 | — | 2 | 3 | 2 | 0 | 1 | 2 | 3 | $\frac{2}{3}$ |
| Ethane .. | $C_2H_6$ | 2 | 7 | 6 | 4 | 9 | 4 | 0 | 5 | $\frac{2}{5}$ | 9 | $\frac{2}{9}$ |
| Ethylene | $C_2H_4$ | 2 | 6 | 4 | 4 | 8 | 4 | 0 | 4 | $\frac{1}{2}$ | 8 | $\frac{1}{4}$ |
| Hydrogen | $H_2$ | 2 | 1 | 2 | — | 3 | 0 | — | 3 | $\frac{2}{3}$ | — | — |
| Methane | $CH_4$ | 2 | 4 | 4 | 2 | 6 | 2 | 0 | 4 | $\frac{1}{2}$ | 6 | $\frac{1}{3}$ |
| Propylene | $C_3H_6$ | 2 | 9 | 6 | 6 | 11 | 6 | 0 | 5 | $\frac{2}{5}$ | 11 | $\frac{2}{11}$ |

## SPECIFIC HEATS. (*Regnault.*)

### (*a*) *Solids and Liquids.*

Water = 1·0000.

| | | | |
|---|---|---|---|
| Antimony | ·0508 | Platinum | ·0324 |
| Bismuth | ·0308 | Phosphorus | ·1187 |
| Brass | ·0939 | Sulphur | ·2026 |
| Bricks | ·189–·241 | Silver | ·0570 |
| Carbon | ·2411 | Steel (Hard) | ·1175 |
| Copper | ·0951 | Steel (Soft) | ·1165 |
| Glass | ·1937 | Tin | ·0562 |
| Gold | ·0324 | Zinc | ·0956 |
| Iron (Cast) | ·1298 | Alcohol | ·7000 |
| Iron (Wrought) | ·1138 | Mercury | ·0333 |
| Lead | ·0314 | Sulphuric Acid | ·3350 |

### (*b*) *Gases and Vapours.*

| | Air = 1·000 at Constant Pressure. | Water = 1·0000. | |
|---|---|---|---|
| | | Constant Volume. | Constant Pressure. |
| Atmospheric Air | 1·00000 | 0·1687 | 0·2377 |
| Alcohol Vapour | 1·8986 | 0·3200 | 0·4513 |
| Carbonic Acid | 0·9104 | 0·1535 | 0·2164 |
| Carbonic Oxide | 1·0793 | 0·1758 | 0·2479 |
| Ether Vapour | 2·0235 | 0·3411 | 0·4810 |
| Hydrogen | 14·3231 | 2·4146 | 3·4046 |
| Nitrogen | 1·0265 | 0·1730 | 0·2440 |
| Oxygen | 0·9180 | 0·1548 | 0·2182 |
| Water Vapour | 1·9794 | 0·3337 | 0·4750 |

## HEATS OF COMBUSTION WITH OXYGEN.

| Name of Substance. | | British Thermal Units. | Pounds of Water at 212° F., evaporated per lb. of Substance. |
|---|---|---|---|
| Hydrogen .. .. .. .. .. .. | | 61,500 | 63·66 |
| Benzene .. .. .. .. .. .. .. | | 18,600 | 19·25 |
| Carbon bisulphide .. .. .. .. .. | | 6,152 | 6·37 |
| Carbon burning to $CO_2$ .. .. .. .. | | 12,906 | 13·36 |
| " " CO .. .. .. .. | | 2,495 | 2·58 |
| CO " $CO_2$ .. .. .. .. | | 4,478 | 4·63 |
| Charcoal (wood) .. .. .. .. .. | | 12,455 | 12·90 |
| Coal, anthracite .. .. .. .. .. | | 15,600 | 16·14 |
| " bituminous best .. .. .. .. | | 15,504 | 16·05 |
| Coke from ditto .. .. .. .. | | 14,375 | 14·88 |
| Coal, average quality .. .. .. .. | | 13,600 | 14·08 |
| Coke from ditto .. .. .. .. | | 12,800 | 13·25 |
| Olefiant gas .. .. .. .. .. .. | | 21,500 | 22·25 |
| Marsh " .. .. .. .. .. .. | | 24,020 | 24·86 |
| Petroleum.. .. .. .. .. .. .. | | 20,272 | 20·98 |
| Propylene .. .. .. .. .. .. | | 21,200 | 21·94 |
| Sulphur .. .. .. .. .. .. .. | | 4,102 | 4·25 |
| Wood, dry .. .. .. .. .. .. | | 7,824 | 8·10 |
| | cub. ft. per lb. | | |
| Coal gas (17 candles) .. | 32·227 | 21,696 | 22·46 |
| Water gas .. .. .. | 22·862 | 6,649 | 6·88 |
| Producer gas .. .. .. | 14·369 | 1,897 | 1·96 |
| Producer water gas .. | 13·478 | ,983 | 1·02 |

The British standard unit of heat (thermal unit) is the amount of heat required to raise the temperature of 1 lb. avoirdupois of water 1° Fahrenheit. The French standard unit of heat (calorie) is the amount of heat required to raise the temperature of 1 kilogram of water 1° Centigrade.

## WEIGHT OF 1000 CUBIC FEET OF GAS OF DIFFERENT SPECIFIC GRAVITIES AT 60° FAHR., AND 30 INCHES BAR.

(*Newbigging.*)

| Specific Gravity Air = 1·000. | Weight per 1000 cubic feet. lbs. | Specific Gravity Air = 1·000. | Weight per 1000 cubic feet. lbs. |
|---|---|---|---|
| ·380 | 29·146 | ·515 | 39·500 |
| ·385 | 29·529 | ·520 | 39·884 |
| ·390 | 29·913 | ·525 | 40·267 |
| ·395 | 30·296 | ·530 | 40·651 |
| ·400 | 30·680 | ·535 | 41·034 |
| ·405 | 31·063 | ·540 | 41·418 |
| ·410 | 31·447 | ·545 | 41·801 |
| ·415 | 31·830 | ·550 | 42·185 |
| ·420 | 32·214 | ·555 | 42·568 |
| ·425 | 32·597 | ·560 | 42·952 |
| ·430 | 32·981 | ·565 | 43·335 |
| ·435 | 33·364 | ·570 | 43·719 |
| ·440 | 33·748 | ·575 | 44·102 |
| ·445 | 34·131 | ·580 | 44·486 |
| ·450 | 34·515 | ·585 | 44·870 |
| ·455 | 34·898 | ·590 | 45·253 |
| ·460 | 35·282 | ·595 | 45·636 |
| ·465 | 35·665 | ·600 | 46·020 |
| ·470 | 36·049 | ·605 | 46·403 |
| ·475 | 36·432 | ·610 | 46·787 |
| ·480 | 36·816 | ·615 | 47·170 |
| ·485 | 37·200 | ·620 | 47·554 |
| ·490 | 37·583 | ·625 | 47·937 |
| ·495 | 37·966 | ·630 | 48·321 |
| ·500 | 38·350 | ·635 | 48·704 |
| ·505 | 38·733 | ·640 | 49·088 |
| ·510 | 39·117 | ·645 | 49·471 |

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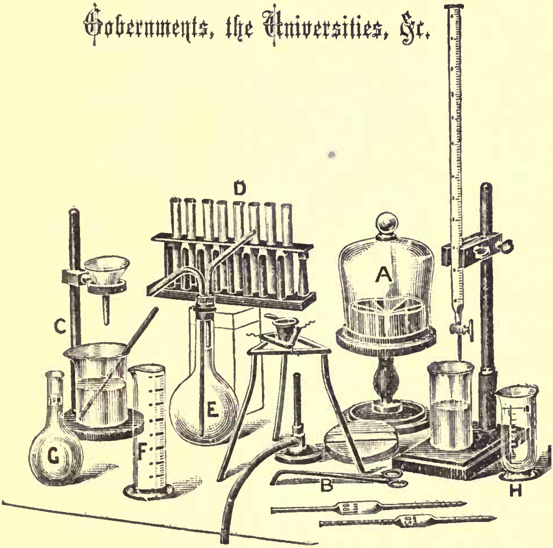
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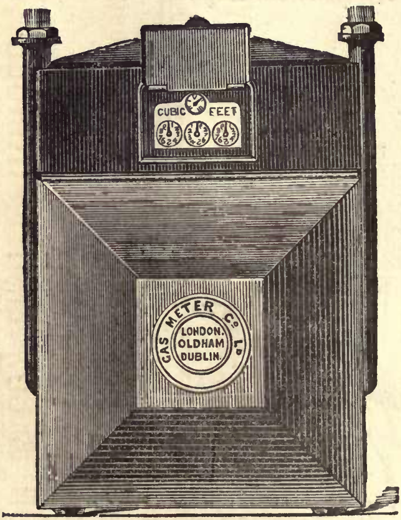
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