# A handbook of chemical manipulation

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**London, 1857**

Section XXII. Gas Manipulation

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## GAS MANIPULATION.

451. Experiments with gases are of everyday occurrence in all laboratories, many processes of research being dependent upon the operator's knowledge of their peculiarities, and familiarity with the methods of working with them. It will be convenient to divide the subject into five parts, namely, 1. Preparation of gases. 2. Storing of them. 3. Estimation of them: *a*, by volume; *b*, by weight. 4. Analysis of gaseous mixtures.

### Preparation of Gases.

452. *a*, Sulphuretted hydrogen; *b*, carbonic acid; *c*, hydrogen; *d*, oxygen; *e*, chlorine; *f*, sulphurous acid; *g*, cyanogen and chloride of cyanogen; *h*, nitrous acid; *i*, muriatic acid.

453. *a. Sulphuretted hydrogen.*—This is by far the most commonly employed of the gases in analytical research; its uses are numerous, but the property which, of all others, renders it indispensable to the analyst, is the exceedingly characteristic nature of its behaviour with solutions of the metals.

454. In fact, the latter may be divided into two great groups, by the manner in which their acid solutions react with it, some being precipitated and others not. Among the former are found antimony, arsenic, bismuth, cadmium, copper, gold, iridium, lead, mercury, palladium, platinum, rhodium, silver, tellurium, and tin. Moreover, its reactions are in some instances so marked, that a shrewd guess may be made as to the identical metal. It is also invaluable for the precision with which certain metals may be isolated from complex organic mixtures, even when existing in them in very minute quantity.

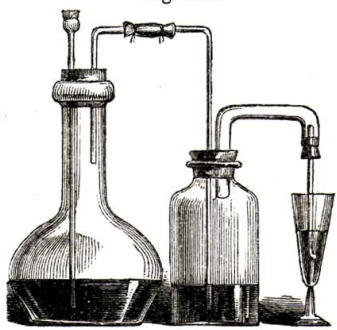
455. It is also much used as a deoxidating agent, many solutions of the higher oxides of metals being reduced to the protosalts with precipitation of sulphur. Its deoxidating properties are also turned to valuable account in organic research.

Iodine and bromine in contact with water may be converted by it into the corresponding hydracids. A minute consideration of these points belongs, however, to works on general chemistry.

456. Sulphuretted hydrogen may be prepared from either the sulphide of iron or that of antimony.

457. *From sulphide of iron.*—This is generally the more convenient of the two methods of preparation, as it does not involve the application of heat. The material from which it is obtained is the protosulphuret of iron, which, for the purpose, ought to be in small fragments: fig. 218 represents the apparatus most usually employed for this purpose.

Fig. 218.

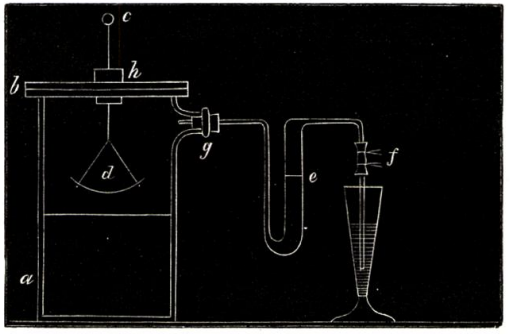


The metallic sulphide is contained in the large flask provided with a thistle-funnel, and a tube by which the gas is conveyed into the washing-bottle. It will be seen that the tube leading from the generating-flask is divided and connected with a caoutchouc tube, to allow of freedom of motion in the apparatus. The washing-bottle is used to retain any sulphuret of iron, or solution of the sulphate or other impurities carried over mechanically by the current of gas. To effect this washing, the tube evolving the gas just dips under the surface of the water in the washing-bottle. It is not advisable for it to be immersed deeper than this, as such a procedure of course creates a pressure in the flask, causing the fluid in it to ascend in the pipe of the thistle-funnel, and, if allowed to penetrate deeper into the water in the washing-bottle, this pressure, added to that in the vessel containing the fluid to be acted on, may prove inconvenient. By increasing the length of the thistle-funnel the gas may be passed a considerable depth under the surface of any fluid, it being always remembered that the height to which the fluid in the generating-flask will rise in the tube of the funnel, is equal to the depth

which the tube penetrates under the surface of the water in the washing-bottle, plus that which it dips in the liquid being acted on.

458. The apparatus, fig. 219, is for enabling a current of sulphuretted hydrogen to be obtained at any time, and yet to prevent

Fig. 219.




the sulphuret of iron from being dissolved after the required amount of gas has been obtained. It moreover prevents the escape of gas into the apartment after the experiment is finished. It was first described by the late Mr. Kemp of Edinburgh. A glass vessel, *a*, has a lateral opening, *g*, to which is attached the washing-apparatus, *e*. A plate-glass circle, *b*, fits air-tight upon *a*, the two surfaces being adapted to each other by grinding. The plate has an aperture drilled in it to admit a cork, *h*, which allows the wire, *e*, to slide up or down. The basin, *d*, is attached to the wire, and is intended to support the sulphuret of iron. It is made of lead or porcelain, and is perforated. The washing-apparatus, *e*, is attached to *g* by a cork. The form in the engraving may be modified in many ways to suit the operator's convenience. A piece of caoutchouc tubing, *f*, enables a clean exit-tube to be attached when required. The vessel, *a*, may be filled to about a third of its capacity with dilute sulphuric acid. To obtain a current of gas, the basin, *d*, is depressed until it just touches the liquid. If the quantity of gas required is very small,

*d* is to be raised again immediately, as the acid adhering to the fragments will be sufficient.

459. The fluids on which the action of the gas is to be tried must always be tolerably dilute, and, if it is intended to separate the metals contained in it into groups in the usual manner, should be distinctly, and even pretty strongly acid; a great excess, however, being avoided, as it involves the introduction of an inconveniently large quantity of ammoniacal salt when the fluid has to be rendered neutral or alkaline in a subsequent stage of the analysis. The production of a precipitate is not invariably owing to the presence of a metal capable of being thrown down by the gas, as when sulphuretted hydrogen is passed into solutions of salts of some of the higher oxides, pure sulphur is thrown down as a milk-white powder; an acid liquid containing persalts of iron giving a copious precipitate of sulphur, according to the equation

$$Fe^2O^3 + HS = 2FeO + HO + S.$$

460. It is necessary to perform operations with this gas in places where there is good ventilation, in consequence of its deleterious nature, and also to prevent it from acting upon the other substances contained in the laboratory. All salts of lead will be blackened if even a minute quantity of the gas escapes near them. Baryta salts are (from the almost invariable presence of a trace of lead in them) generally discoloured by it, as also metallic apparatus.

461. Where there is much testing with sulphuretted hydrogen, it is a good plan to use water saturated with the gas, instead of the latter itself, and, in fact, in all cases where it is not necessary to expose the fluid under examination to the action of a continued stream.

462. To obtain a knowledge of the appearances caused by the action of a stream of this gas upon various metallic solutions is of great importance to the analyst, as it frequently enables a pretty accurate guess to be made at the nature of the substances present. For this purpose it is well to practice upon solutions of known composition, until the reactions are familiar.

463. A blue colour is formed by sulphuretted hydrogen in solutions containing vanadium. The precipitates formed in solutions of copper and lead, and perhaps many other metallic salts, vary greatly in colour with the concentration and degree of acidity of the fluid, and with the mixtures present. If, for example, sulphuretted hydrogen is passed into a solution containing lead and arsenic acid, the first precipitate will be black; and if this is filtered off, or allowed to settle, a yellow one results. In this case, the phenomena are caused by the ease with which the lead is precipitated, the sulphuret of that metal being formed immediately, the yellow compound of arsenic and sulphur, corresponding to arsenic acid, being formed with greater difficulty; it is usual, therefore, to reduce the arsenic to the state of arsenious acid before precipitation.

464. A metal may be thrown down by this gas with quite different appearances, according to the state of oxidation in which it exists in the solution; protosalts of tin, for example, giving a dark-brown precipitate, while the persalts are thrown down of a bright yellow colour.

465. A peculiar appearance is caused when the gas under consideration is passed into a solution of corrosive sublimate. It may be mentioned, that at first, when the gas is in small quantity, a white compound of mercuric sulphide with the chloride is formed; and as the precipitates pass through various shades of colour, from white through yellow and red to black, it would appear that several compounds momentarily exist, but are all rapidly decomposed, the ultimate product being mercuric sulphide of a black colour until sublimed, when vermilion is obtained.

466. When insoluble salts are to be decomposed by sulphuretted hydrogen, as very frequently occurs, more especially in organic chemistry, it is necessary to keep stirring up the precipitate, in order that it may be thoroughly exposed to the action of the gas, as, if allowed to remain at the bottom of the beaker, or other vessel in which the operation is performed, the decomposition would be very incomplete even after a considerable time.

467. It is sometimes required to prepare hydriodic acid by passage of this gas into a mixture of water and iodine; agitation is particularly essential in this instance, from the way in which the iodine becomes enveloped with the precipitated sulphur, masses accumulating which are troublesome to get rid of when once allowed to form.

468. It will be seen on reference to the engraving in p. 280, that the large tube proceeding from the washing-bottle has a small tube fitted to it with a cork; this is in order that when it is wished to test solutions of different kinds one after another, it should not be required to wait until the tube last used is cleansed from the adhering metallic sulphide before proceeding to the next experiment; a few of these tubes may always be kept in reserve. It is sometimes more convenient to attach them to one of vulcanized caoutchouc.

469. An alcoholic solution of sulphuretted hydrogen is much used, conjointly with ammonia, in some branches of organic chemistry, for the purpose of reducing the nitro-compounds of certain hydrocarbons, with the intention of converting them into organic bases; the following equation, showing the manner in which this decomposition takes place in the case of the formation of aniline from nitrobenzole, may be taken as expressing the general nature of the process, even where the hydrocarbons are of a different constitution:—

$$C^{12}\left\{\begin{matrix} H^5 \\ \\ NO^4 \end{matrix}\right. + 6HS = \left.\begin{matrix} C^{12}\,H^5 \\ H \\ H \end{matrix}\right\} N + 4HO + 6S$$

The amount of sulphur deposited being directly in the ratio of the quantity of base formed.

470. *b. Carbonic acid.*—Carbonic acid, although of far less general applicability in research, is, nevertheless, often required to be prepared in the laboratory. It is usually obtained by acting on fragments of marble with hydrochloric acid. It is much better to use the marble in fragments than in powder, as in the latter case the gas is evolved too tumultuously, while in the former it is procured in a steady and easily manageable stream, well

adapted for acting on solutions or other matter exposed to its influence. It is improper to use black marble in experiments where a tolerably pure gas is required, as it evolves sulphuretted hydrogen at the same time as the carbonic acid. The apparatus seen in fig. 218, p. 280, is adapted to the preparation of this gas. The operation is precisely the same as in the case of sulphuretted hydrogen, the only difference being that carbonate of lime in the state of marble fragments is substituted for the ferrous sulphide, and hydrochloric acid for the sulphuric.

471. It is easy in all cases where a gas does not require heat in its preparation, to connect two ordinary glass bottles with glass tubes and corks, so as to form a very efficient apparatus for the purpose; the method of putting it together is obvious from fig. 218. In this, and, in fact, in all cases where gases are prepared and passed into fluids under any pressure, it is necessary to take considerable care in the selection of corks, in order to avoid the necessity of making good the joints with luting. Nevertheless, when large quantities of the gas are required, it is impossible to avoid the use of luting unless an expensive apparatus is employed.

A very effectual arrangement for the preparation of carbonic acid, sulphuretted hydrogen, or pure hydrogen on the large scale, consists of a large stone jar closed with a bung, through which pass two tubes, namely, a large one of glass, having a glass funnel fitted to it with a cork, and a piece of tin gas-pipe passing under the surface of water contained in a washing-bottle, from whence it is conveyed into the liquid to be acted on.

472. When the fluid which is to be submitted to the action of the gas is of a nature to receive injury from contact with a metallic pipe, it is very easy to adapt a piece of glass tube to the metallic one. In this arrangement it is necessary to have recourse to luting, and the best for the purpose is the almond- and linseed-meal mixture previously mentioned.

473. Carbonic acid is, it has been said, less frequently used than sulphuretted hydrogen in research, but, nevertheless, it is indispensable in many operations. It is used to convert the

ordinary carbonate of potash into the bisalt, and to precipitate lime and baryta from alkaline solutions. As produced by the combustion of coke, it has been employed to precipitate soluble salts of lead with it, to form white-lead. It is not unfrequently used as a non-oxidizing atmosphere in some chemical operations, such as the distillation of fluids easily decomposed by contact with air. Where the chloride of calcium in the spongy state, used in organic analysis, is alkaline, it is usual to pass a gentle stream of carbonic acid through the chloride-of-calcium-tube for about half an hour, in order that none of the carbonic acid gas produced during the combustion of the organic substance should be absorbed during its passage before reaching the potash-bulbs. It is necessary, of course, to remove the remaining carbonic acid from the chloride-of-calcium-tube before connecting it with the combustion-tube, &c.; this is best done by gentle suction with the lips, applied by means of a suction-tube, or by simply applying them to the tube itself, of course avoiding to wet the outside of it, or, if such should happen, it must be carefully wiped off preparatory to weighing.

474. In operations where lime, &c. is to be precipitated by a current of carbonic acid from any solution, the property of this gas to dissolve the precipitate again when in sufficient excess must not be forgotten. Where it is suspected that such an error has been committed, it may be corrected by boiling the solution for a short time, by which means the excess of carbonic acid is expelled, the earthy carbonate being again precipitated.

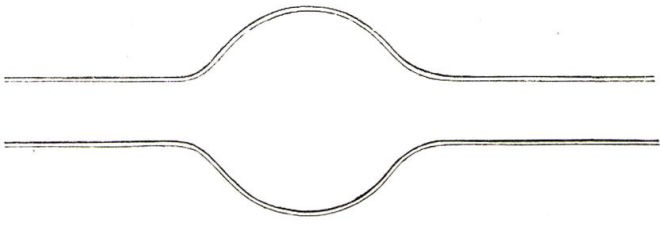
475. *c. Hydrogen.*—In the preparation of this gas, the same system of apparatus given for sulphuretted hydrogen and carbonic acid may be employed; the substances by which the water is decomposed to furnish the gas, with the assistance of sulphuric or muriatic acids, being iron or zinc. The manipulation required demands no specific notice. There are, however, some particular cases in which precautions must be taken. If, for example, it is required to reduce a metallic oxide with hydrogen, it is necessary to dry the gas before passing it into the tube in which the reduction is performed. In the instance of the preparation of the metallic

copper used in organic analysis to reduce the oxides of nitrogen, and sometimes for other purposes, it is merely necessary to substitute a flask of concentrated sulphuric acid for the washing-bottle in fig. 218; see also p. 187.

476. The uses of hydrogen in analysis have been much increased lately by the researches of MM. Rivot and St.-Claire Deville, and it is probable that they will be still further extended. It is important in experiments where the reducing property of hydrogen is to be made use of on mixtures of a heavy with a comparatively light substance, not to have too rapid a current of the gas, as it is liable to carry away mechanically a portion of the less weighty matter.

477. When the loss of weight consequent upon the passage of hydrogen at a red heat over a mixture of a reducible and non-reducible metallic oxide is to be ascertained, it is unnecessary, where the mixture has been obtained by precipitation, to employ the whole of the precipitate. If the weight of the latter is accurately known, a portion is introduced into the bulb of the reduction-tube, figs. 129 and 220, which is weighed before and

Fig. 220.



after its introduction. By this means the loss of weight consequent upon the passage of the gas over a known portion of the mixture may be calculated upon the whole quantity, and as accurate a result will be obtained as if the entire amount had been introduced. In fact a more exact experiment is generally made thus, because where a considerable quantity of substance is treated in the manner alluded to, a portion is liable to escape the action of the gas from its being covered by the superin-

cumbent mass, and in all cases the larger the quantity worked upon, the more troublesome the reduction becomes to effect completely.

478. The use of hydrogen in distillation to prevent oxidation has been previously alluded to, § 363, p. 231.

479. In taking the densities of the vapours of substances liable to oxidation at elevated temperatures, results are obtained greatly varying from the numbers required by theory. To obviate this, the flask in which the density is to be determined, has its neck inserted into a vessel through which a stream of hydrogen gas is passed for some hours; by this means the diffusive property of the gas enables us to get rid of the whole of the oxygen from the globe, and thus avoid the source of error caused by the oxidation of the vapour.

480. When it is required to obtain hydrogen gas absolutely pure, it is sometimes procured by the electrolysis of water; the apparatus required for this purpose will be found in its proper place.

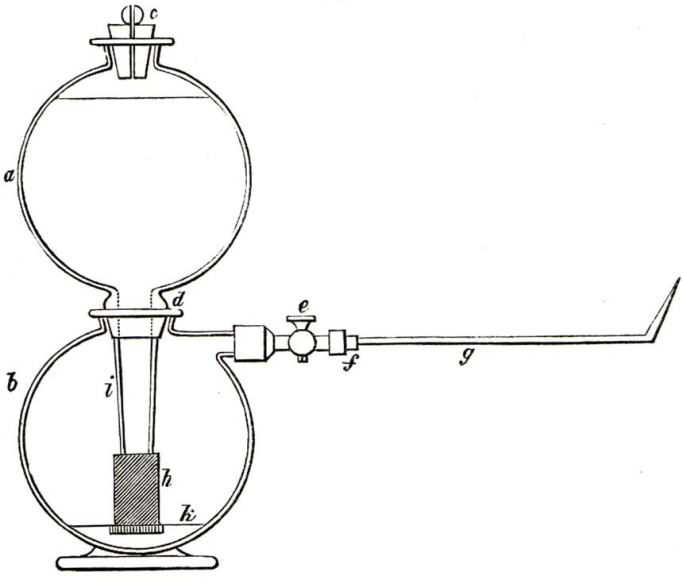
A very pure hydrogen may be procured on passing the gas evolved by the action of dilute sulphuric acid on zinc, first through water, then through a solution of nitrate of silver; after this it bubbles up through a tolerably concentrated solution of caustic potash; and lastly, it is dried by sulphuric acid. A considerable time must be allowed to elapse before the gas is collected, as the quantity of air in so complex an apparatus as that required for its complete purification is considerable; and the stream must be tolerably rapid, and well sustained, to ensure the removal of the last traces.

481. Hydrogen is frequently prepared by means of an apparatus on the principle of that seen in fig. 221, and known as Dœbereiner's lamp. The chief advantage in its use is, that it allows of a supply being obtained at any moment, and with the addition of a small brass cup containing spongy platinum, constitutes an instantaneous light. It can also be used very conveniently as a Marsh's apparatus.

The instrument consists principally of two vessels, *a* and *b*:

*a* is a globe with a long neck, and provided at the bottom with a

Fig. 221.



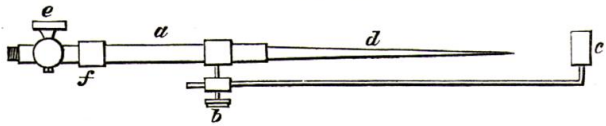

flange, to enable it to support a cylinder of zinc, *h**; it has stopper at *c*, which is, however, perforated. It fits tightly into *b*, by means of a thick portion ground conical to fit accurately into the neck of the lower vessel at *d*. The lower portion of the lamp has a pipe projecting from it, furnished with a tap, *e*, which is so made as to allow of the other pieces of apparatus, presently to be described, being attached. The mode of action of the instrument is very simple. Dilute sulphuric acid, sufficient to fill the lower vessel when the zinc is in its place, is poured in, and the upper portion is inserted, care being taken that it fits properly at *d*; the acid meeting with the cylinder of zinc held round the tube by the flange, generates hydrogen with tolerable rapidity; *e* being closed, the gas is unable to escape, and

* It is preferable to have a moveable cork flange to support the zinc.

causes sufficient pressure to force the liquid up the tube, *i*, into *a*; the level of the liquid in *b* is now at *k*, and the acid being no longer in contact with the zinc, all evolution of gas ceases; and by opening the stopcock, *e*, the pressure of the column of fluid causes the gas to escape with rapidity. If it is desired to use the lamp as a Marsh's apparatus, the fluid suspected to contain arsenic is introduced into *a*, and allowed to descend into the lower compartment, so that the hydrogen may act on the arsenic at the moment of its formation. A piece of hard glass tube, free from lead, drawn out and bent upwards, as in figure 221, is attached at *f*, by means of a perforated cork, and as soon as the air is expelled (by allowing the liquid to descend twice, and again making it rise into *a*, by means of the stopcock, *e*), the gas is inflamed at the point of the tube, and a cold porcelain plate is depressed upon it until the presence or absence of arsenic is determined.

The process is sometimes modified, by heating the tube, *g*, in the centre by means of a spirit-lamp, when the arseniuretted hydrogen is decomposed with deposition of the metal. When it is intended to use the instrument as an instantaneous-light apparatus, the appliances seen in fig. 222 are attached. It con-

Fig. 222.

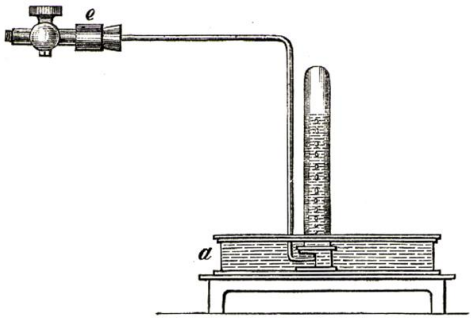



sists, essentially, of a brass tube, *a*, upon which slides stiffly a small piece of tube, carrying a rod, to which is attached a nut, perforated to allow a wire to slide through it, which is capable of being fixed in its position by a screw with a milled head, *b*. This wire has a small brass cup, *c*, fastened to its extremity, the concave part of which is directed towards the fine orifice of the nozzle, *d*; this cup contains a little spongy platinum. If the tap, *e*, be turned when the vessel, *b* (fig. 221), is full of gas (the apparatus in fig. 222 being substituted for *f g*, fig.

221), the hydrogen escaping by the aperture of *d*, plays upon the platinum, and being by the peculiar structure or condition of the metal brought into intimate contact with condensed oxygen, its union takes place with formation of water, and the action is sufficiently energetic to cause the ignition of the platinum, which in its turn inflames the excess of hydrogen. This takes some time to describe, but the effect, as the name of the lamp implies, is, when all things are in good order, instantaneous.

482. If, as not unfrequently happens, it is wished to collect the hydrogen over mercury or water, the arrangement seen in fig. 223 is adopted. It merely consists of a glass delivery-tube,

Fig. 223.




which may be attached by a cork to the stopcock, *e*, instead of the tube *g* in fig. 221.

483. *Oxygen.*—The uses of oxygen in research are very limited, and, with the exception of its power of converting carbon into carbonic acid at a red heat, I do not remember any application that requires particular manipulation, except in experiments for the lecture-table, and in eudiometrical processes, to be alluded to in another section.

The method of applying it to the purpose first mentioned, will be described under the head of Organic Manipulation. For all purposes where a good quality of gas is required, it is prepared

by heating chlorate of potash mixed with about one quarter of its weight of powdered oxide of copper or manganese. This addition enables the gas to be obtained at a lower temperature than would otherwise be necessary. In heating the mixture either in flasks or large test-tubes, care should be taken to apply the heat very gradually, or a fracture is almost sure to take place. A charcoal fire is, for this reason, better than a lamp, as the temperature is more diffused and gradual. The delivery-pipe must not be immersed too deeply in the water of the pneumatic trough, or the pressure will cause the tube to blow out, in consequence of the softening of the glass caused by the high temperature. Many other points in the manipulation connected with this gas will be alluded to under other heads.

484. *Chlorine.*—This gas is used in a multitude of cases, each requiring a different system of apparatus, or a modification peculiar to the circumstances of the experiment in hand. Great care must be taken to avoid breathing it, as its effect upon the lungs, even when much diluted with air, is very distressing; should such an accident occur, most rapid relief is obtained by inhaling the vapour of hot water, the face being placed upon a jug so as to permit of the steam being easily breathed: a very little ammonia added to the water greatly increases its efficacy. Another reason for preventing its escape into the laboratory, is the great injury done by it to all metallic instruments. Where a continued stream of the gas is required to be passed for hours through a solution, as in Rose's excellent process for separating nickel and cobalt, the exit of the gas must be into a chimney, or some other place where no inconvenience can arise from it.

If an exceedingly large quantity of gas is to be passed for hours through or over a substance, as in acting on large quantities of naphthaline in Laurent's process for preparing the bichloride, or in making considerable quantities of pentachloride of phosphorus, &c., the oxide of manganese in very fine powder should be placed in a six-gallon stone bottle, provided with a cork, through which two tubes pass. One of these is merely for the exit of the gas, the other is a safety-funnel, by which the

hydrochloric acid is to be added. The stone bottle is to be placed in a water-bath, all the joints of the tubes being made with rather long pieces of vulcanized tubing, in order that the various parts of the apparatus may be susceptible of considerable movement without deranging the rest. The chlorine first passes into an empty bottle kept cold in order to allow the greater part of the moisture to deposit, and then two flasks of concentrated sulphuric acid, one after the other, to ensure perfect dryness. The gas then, by means of a tolerably wide tube, enters the flask in which the naphthaline, phosphorus, or other matter is contained, care being taken that if at any stage of the operation the substance acted on thickens under the influence of the gas, the tube by which it enters does not quite touch the matter acted on. Another tube from the flask is allowed to pass out of the window, to get rid of the excess of chlorine and other gases and vapours generally evolved under such circumstances. If it is suspected that volatile products of decomposition are evolved, as not unfrequently happens in operations of this class, the gases before passing out of the window may be made to traverse a bulbed tube immersed in a good freezing mixture. This will generally be found to retain any products that may be formed. The method above described must of course be modified to meet the varying circumstances of the experiment.

485. *Sulphurous acid.*—Where a considerable quantity is required, the iron pot with the tube screwed on, fig. 198, is perhaps the most convenient apparatus. In this case it is evolved from a mixture of bruised charcoal and concentrated sulphuric acid. The pasty mass is gently heated, the gas being copiously evolved. The mixture is almost without action upon iron. It is also sometimes procured by the action of sulphuric acid upon mercury or copper, but this process is only adopted upon the small scale, where a pure product is required. As a very effective condensation is necessary, where the gas is to be condensed by itself, the methods of effecting condensation described in §§ 352 and 383, may be employed. It is proper

to have the exit-pipe immersed a short distance under the surface of mercury, so as to assist the condensation by slight pressure. The product must be kept either in thin stoppered flasks, which are to be retained at as low a temperature as possible, or, which is preferable, sealed tubes. It is seldom used in this condition, except, perhaps, for demonstrating the spheroidal state of water, by the conversion of it into ice in a red-hot crucible, as in Boutigny's remarkable experiment. Sulphurous acid very much diluted, as prepared by passing the gas produced by the mutual action of charcoal and sulphuric acid into water, is used to reduce arsenic acid to arsenious acid, previous to precipitating the fluid by a stream of sulphuretted hydrogen.

486. On the large scale, sulphite of soda is prepared by passing the gas, produced as last mentioned, into a strong solution of carbonate of soda. The sulphite of soda, thus prepared, is one of the ingredients used in making hyposulphite of soda according to Berzelius's method. A strong solution of sulphide of sodium, procured by boiling caustic soda with excess of flowers of sulphur, has the sulphite of soda added to it until the dark colour of the sulphide disappears; a little more of the latter is then added, until the fluid assumes a pale but distinct straw tint. On evaporation, crystals of hyposulphite of soda, slightly yellow, are obtained, which by recrystallization may be procured colourless and very pure. The bisulphites of soda and ammonia are invaluable in organic research, from the powerful tendency which they evince to combine with the aldehydes, and some other bodies.

487. *Cyanogen.*—This gas has lately been used with great success as a reagent, more especially by Hofmann, in his extremely beautiful researches on aniline, an investigation which, from the completeness with which all the analogies which the base alluded to presents with ammonia, have been followed out, has probably never been surpassed.

The gas is evolved in a test-tube by the action of heat upon cyanide of mercury, and is conveyed into another, where any aqueous or mercurial vapour is condensed, the pure gas passing

into a third, where it exerts its action upon the fluid submitted to it. For details connected with the action of cyanogen and the chloride of that radical upon organic bases, the reader is referred to the original papers of Hofmann in the Quarterly Journal of the Chemical Society of London.

488. *Nitrous acid.*—The action of this gas upon oils and other matters has been studied of late to a sufficient extent to make its mention here not improper. If nitric acid moderately diluted is placed in a flask with a bent tube to convey the produced gas into the solution to be acted on, and a few pieces of starch are added, and the whole is gently warmed, a mixture of nitrous and hyponitric acids is given off with extreme regularity; and by removing the flask from the source of heat, may be passed through the oil or other matter to be examined in a slow steady stream, excellently adapted for producing the intended effect.

489. *Muriatic acid gas.*—When it is desired to submit any fluid to the action of muriatic acid, as in the preparation of the ethers, the production of the muriate of camphene, &c., several ways may be adopted. The gas may be procured by merely heating hydrochloric acid, and passing the evolved gas first through sulphuric acid and then into the fluid, or by adding concentrated sulphuric gently through a safety-tube into strong muriatic acid, in a flask provided with a bent tube, to convey the gas to the place where it is to exert its action: or the action of sulphuric acid upon common salt may be resorted to as being the more economical method of procedure. In most cases, no matter in what way the gas is procured, the matter to be acted on should be kept very cold, to prevent the vapour from escaping before it has had time to produce the desired reaction.

490. There are other gases often required in the laboratory for purposes either of demonstration or research, but the methods of manipulation in the following pages will be found amply sufficient to indicate the mode of procedure to be adopted, and the apparatus to be made use of.

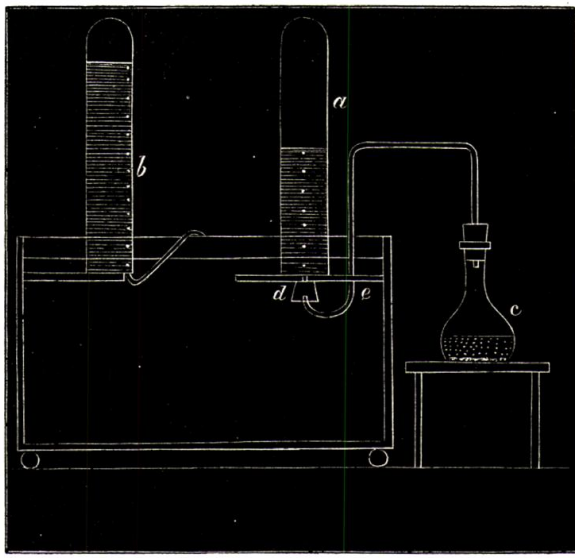
491. *Collection and retention of gas.*—The way in which chemists at the present day collect elastic fluids is pretty much the

same as it was in the time of Priestley, the differences being only in the arrangement of the apparatus, not in the principle.

The method depends upon the displacement of water or mercury by the gas as it is evolved from any of the instruments either to be alluded to or those already given. If a bottle be immersed in a vessel of water, so that it becomes filled, and it is then raised, bottom upwards, out of the liquid, the latter will not escape so long as the mouth of the vessel is under the water; the same thing happens with mercury. The older chemists were in the habit of suspending the jars to be filled with gas by means of strings, in the pneumatic trough as it is termed, but the method shown in fig. 224 is that now generally adopted.

It will be seen that two jars stand upon the shelves of the

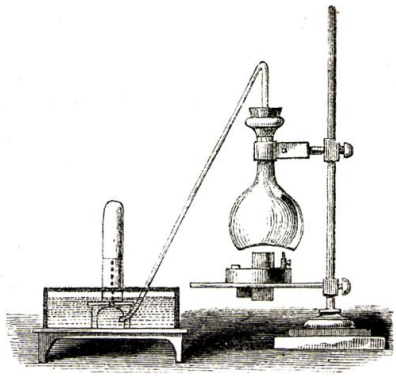
Fig. 224.




trough, in both of which the water has been partially displaced, that in *a* by the gas evolved from the flask, *c*, the tube of which passes through a slit cut in the shelf at *e*.

492. When an inverted jar of water has the end of a pipe delivering gas passed under it, the water is displaced, descending into the trough, the level of the fluid in which of course rises, the gas supplying the place in the jar. At *d* is a small funnel placed immediately beneath the aperture in the shelf, by which the gas ascends. Sometimes, instead of this arrangement, the jars are so placed upon a plain shelf that they project over it sufficiently to allow of the delivery-tube being placed under it, as at *b*, in the figure. Another method is to have notches cut in the shelf to permit the tube to pass under the jar without any danger of the latter falling, as may take place where they are allowed to project over the shelf. But by far the most convenient and portable method is that seen in fig. 225, where what is termed a bee-hive shelf is used. The last-named piece of apparatus consists of a vessel shaped like a bee-hive, only flat at the top, so as to support a gas-jar. An aperture in the side permits the passage of a delivery-tube. The use of this little contrivance (the invention, it is believed, of Mr. J. J. Griffin) almost dispenses with the use of the cumbrous pneumatic trough, any basin or water-tight box answering the purpose. The water should be poured in until it covers the shelf for about an inch, when the jar, full of water and inverted, may be placed upon it, and supported, if necessary, by a ring of the retort-stand, or by Gahn's cylinder-holder, described at p. 161.

Fig. 225.



493. Pneumatic troughs of large size adapted for class illustration and lecturing, are frequently made of japanned tinned-iron or wood. Their construction and use will be obvious from what

has already been said. Where it is necessary to fill several jars with gas one after another, they may all be arranged in a row upon the shelf of the large trough, and the delivery-tube may be brought in succession under each until it has become full of the gas. In this latter case, however, as the quantity of water displaced gradually accumulates, the trough soon becomes filled with water to an inconvenient height, and may easily overflow. The undue rise of water is to be carefully avoided, because the jars, when full of gas, are easily upset as the water rises, their buoyancy being considerable. It is best to have a siphon, with its longer leg closed with a cork or tap, hanging over the side of the trough, so that any quantity of water required may be drawn off. Where very small troughs, such as that seen in use with the beehive shelf in fig. 225, are employed, it is of course impossible to fill the gas-jars by immersion in the troughs, and then inverting them; in this case one of two methods may be employed, depending upon the calibre of the jars. If they are long and narrow, as in figs. 226 and 232, they should be ground at the opening, and then, being inverted, may be filled up with water, and a ground-glass plate being placed over the aperture, they may be restored to their proper position as used in the trough, and the mouth being placed under water, the glass plate may be removed, and the jar put in its place on the shelf. Where, on the other hand, the jars are too large to be closed with a plate, they must be filled with water at a cistern or some other convenient vessel; and a small tin-tray or an evaporating-basin, or even, if nothing else be at hand, a saucer or small cheese-plate, should be slipped under the jar beneath the surface of the water, where the small quantity of fluid in the plate will be quite sufficient to retain the water in the jar on lifting it out. In the same manner a jar full of gas may be taken from the trough and put aside until wanted.

494. The jars used at the pneumatic trough vary in size and capacity according to the purposes for which they may be required.

Fig. 226 shows a form of gas-jar of very frequent use in the laboratory in various researches, especially at the mercurial trough. It is made very thick in the glass, to enable it to bear the great

weight of the metal; it is also ground at the bottom, to facilitate the removal of the jar and contents by closing it with a glass plate, in the manner described further on.

Fig. 227 is chiefly used in experiments involving the transference of a gas to a globe or bladder, by the method the details of which will be found further on.

Fig. 228 represents a form of jar much used in demonstrative

Fig. 226. Fig. 227.

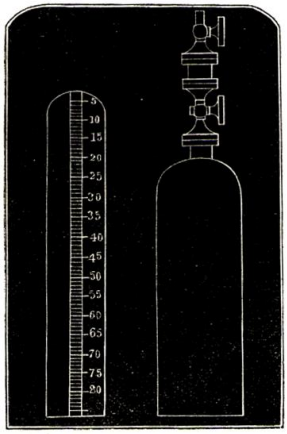
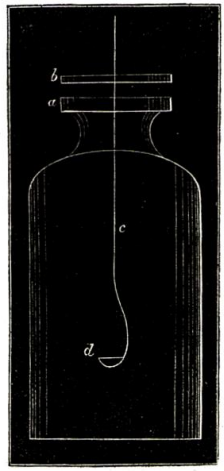


Fig. 228.




experiments; it consists of a wide-mouthed bottle open above and below, the outside of the neck being ground smooth at *a*, to admit of its being accurately closed by a glass plate, or by the air-tight collar, *b*, through which passes a wire, having a small spoon, *d*, at the lower end. To use this jar for deflagrations, as, for example, to show the combustion of sulphur in oxygen, the mouth is closed with a glass plate, slightly greased to ensure complete adhesion, and the bottle being filled with water, is to be inverted in the pneumatic trough, and the gas passed in until full. When this is the case, the collar and wire being at hand, the matter to be burnt is placed in the spoon and ignited, and the plate of

glass being quickly removed, the spoon, with its lighted charge, is to be quickly and steadily inserted, the collar, *b*, resting on *a*. Fig. 229 is a form of jar equally adapted to lecture experiments, and, when graduated, to researches. It is, however, not used where ignitions are to be performed in gases, except for the combustion of antimony or copper in chlorine, where the gas is inserted by displacement instead of over the water-trough, as described at p. 302, and in this case the round top is very inconvenient, and the jar seen in fig. 231 is almost always substituted. Fig. 230 is used in lecture experiments, to show the combustion of phosphorus in air or oxygen gas. In using this apparatus, the globe previously filled at the trough with gas, where oxygen is used, is, after the phosphorus has been ignited, steadily placed over the deflagrating spoon, *a*, supported on the stand, *b*. Figs. 231 and 232 show two forms of plain gas-jars much used in experiments at the lecture-table; the first is well adapted for showing the effect of oxygen upon the gas produced by the action

Fig. 229.

Fig. 230.

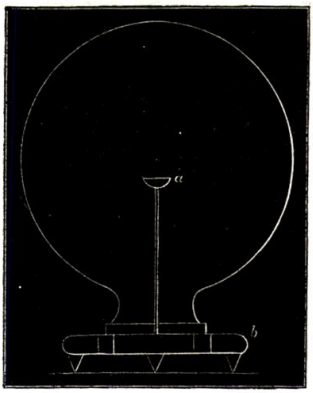
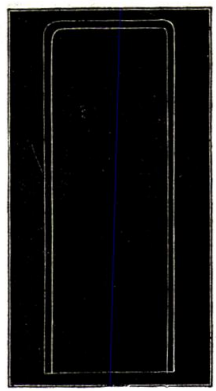


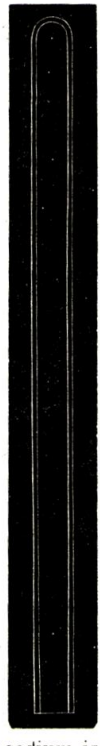

Fig. 231.



of nitric acid upon copper, the binoxide of nitrogen being converted

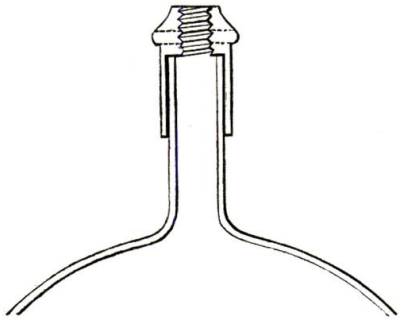
into the red fumes of hyponitric acid. It is equally well adapted, from its flat top, for experiments with chlorine, carbonic acid, &c., where it is filled by displacing the air with the heavy gas. The tall tube, fig. 232, is useful for showing the decomposition of water by sodium. The vessel, the mouth of which is ground, so as to enable it to be closed with a glass plate, is filled with water, by pouring the latter in from a jug; the plate being then placed over the mouth, it is inverted in the pneumatic trough, and the plate is removed. If, now, a pellet of sodium is wrapped in paper and dexterously inserted under the edge, it ascends to the top, and as soon as the fluid has penetrated the paper, the decomposition of the water takes place, with evolution of hydrogen gas, the liquid becoming of course depressed; if, now, the plate is placed under the jar, and the latter being raised mouth upwards, the plate is removed, a light being at the same time brought close to the aperture, the inflammability of the gas will be shown. It is better in this experiment not to use sufficient sodium to expel all the water, as a little oxygen, or even atmospheric air, may be introduced, and, on the approach of the light, an explosion will take place, the experiment thus becoming more perceptible to those at some distance from the lecture-table. The reason that sodium is used, is because it is more manageable than potassium, from being not quite so rapidly oxidized by water, so that less heat being developed, there is little danger of fracturing the jar. The jar, fig. 227, has a cap cemented to it to allow of stopcocks or other pieces of apparatus being attached, to enable the gas to be transferred to bladders or other receptacles. A convenient form of cap is that given in fig. 233, where it appears attached to the neck of a glass balloon; the female screw allows of the stopcock of the bladder or gas-bag being attached or removed. To ascertain the tightness of a gas-jar with the stopcocks and cap attached,

Fig. 232.



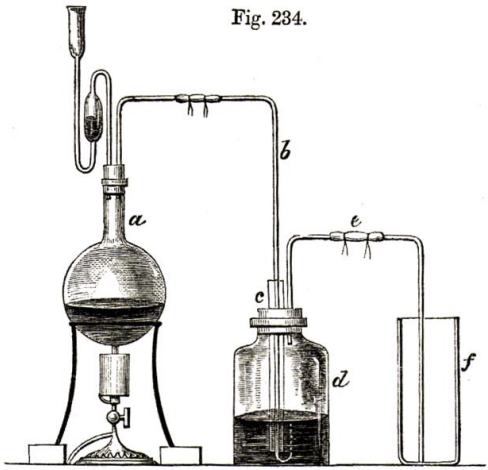
it is to be placed upon the shelf of the pneumatic trough, and, applying the mouth to the opening, air is to be drawn out so as to cause the water to stand several inches inside the jar, above the level of that in the trough. The stopcock is now to be turned off, and the height of the water is to be marked with a piece of gummed paper. If at the end of an hour or so the water has not fallen, the cap and its adjuncts are perfect.

Fig. 233.



495. Heavy gases, such as chlorine and carbonic acid, are frequently collected very conveniently by the method of displacement, the arrangement of the apparatus being evident from fig. 234,

Fig. 234.

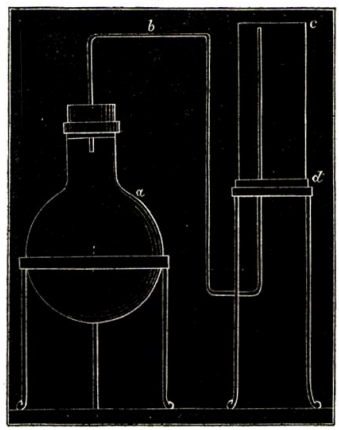



where *a* represents a flask in which the gas is prepared, which

passes through *b*, down the large tube *c*, into the washing-bottle, *d*. The washed gas passes from *d*, through the tube *e*, to the bottom of the cylinder, *f*, which is the same as figure 231 inverted. The heavy gas gradually accumulates in *f*, rising higher and higher until the atmospheric air is expelled; when this is the case, the mouth is covered with a glass plate ground to fit it, and the vessel is ready for the lecture-table. In the case of chlorine, it is easy to know when the cylinder is full from the colour of the gas, but with carbonic acid it is proper to continue the stream until an ignited splinter held close to the side at *f* is extinguished by the gas, which, having filled the vessel, flows over the sides owing to its high density, and then extinguishes a burning body as effectually as water.

496. Ammonia may be conveniently obtained in jars for the lecture-table by an inversion of the latter process as shown in fig. 235, where the ammoniacal gas liberated from the flask, *a*, is carried by the bent tube, *b*, to the top of the cylinder, *c*, until a piece of turmeric paper is turned red the instant it is held near the mouth of the cylinder, as at *d*.

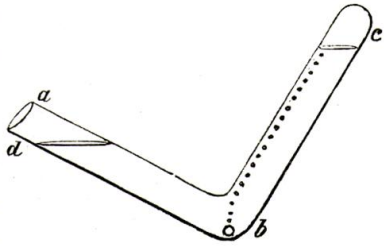
Fig. 235.




497. Where very small quantities of gases are being examined as to their solubility or any other physical or chemical character, it is sometimes convenient to dispense with the pneumatic trough altogether, and several contrivances have been adopted to meet this requirement. One of these, Kerr's tube-receiver, fig. 236, although less used than it deserves to be, will be described from its great convenience in many experiments of research where the quantities are very limited. It consists of a V-tube, so constructed that the lowest part, *b*, is not exactly in the centre, but rather towards

the side, *c*, so that if the apparatus be filled with the acid to *d*, and a fragment of carbonate of lime, for example, be dropped in, it will occupy the position seen in the figure, and the bubbles of gas will ascend in the limb, *b c*. If it is desired to replace the acid by water in order to examine the behaviour of the gas towards reagents, it may easily be effected by plunging the apparatus into water, keeping the end, *c*, uppermost, when the acid will flow out and its place be occupied by the water. The tube being filled with water, except that portion of *b c* occupied by gas, it is easy, by closing the end, *a*, with the thumb and depressing *c*, to obtain any quantity between the thumb and the surface of the water in the limb, *b a*, so as to try the effect of flame, &c. upon it. The method of performing these experiments will, however, be made more evident by consulting the description of Cooper's mercurial receiver.

Fig. 236.

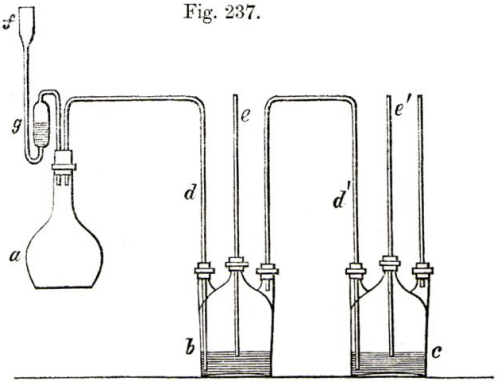



498. In most instances where a gas is to be collected for experiments to be made upon it, it is essential to allow all the air contained in the delivery-tubes, and in that portion of the generating flask above the materials, to be expelled before collecting the remainder; where this precaution is necessary, it is improper to allow the gas and air to stream from the end of the delivery-tube into the apartment, even in cases where no immediate inconvenience would result from such a procedure, because by this means it is impossible to ascertain with accuracy the point at which it is necessary to stop; but if, on the other hand, the first portions which come over are received at the pneumatic or mercurial trough in small tubes, it is easy to ascertain by appropriate tests when the air is sufficiently expelled to make it proper to receive the remainder in the regular manner. On other occasions, however, as, for example, in one of the older and least-used

methods of determining nitrogen, the whole of the gas is received from the commencement.

499. *Solution of gases.*—Where gases are to be dissolved in fluids, the simplest method of proceeding consists in passing the former into the latter contained in a test-glass; the manipulation necessary in a case of this kind having already been described, will not be repeated. But if, on the other hand, the gas is extremely soluble in the absorptive media employed, there is considerable tendency in the fluid to rush back into the generating bottle. As the ordinary routine of laboratory-work presents numerous examples of this kind, and as it is very necessary for the student to be familiar with all the precautions requisite to ensure freedom from accident, the operation will be described somewhat minutely. If, for example, it is desired to obtain a saturated solution of ammonia in water, by the reaction of slacked lime and chloride of ammonium, the materials are mixed in the flask, to which is

Fig. 237.




fitted a perforated cork; a tube passes from this into a little water contained in the first bottle, *b*, intended to wash the gas and retain any impurities mechanically carried over; from this another tube proceeds into *c*, which contains the water to be saturated, and thence, if necessary, into one or two more bottles. The

delivery-tubes proceed to the bottom of the water, and it is to be observed that when contraction takes place in *a* or *b*, which frequently happens, there is a tendency in the fluid to rush up the tubes, *d d'*, the fluid in *c* going back into *b*, and that in the latter into the flask, *a*; but this is prevented by the safety tubes, *e e'* and *f*; the two former dip only just below the surface of the water in the bottles, and therefore when contraction takes place, air enters by them and restores the equilibrium of pressure. But as there is no fluid in the flask for a tube to dip into, the Welter's tube, *f*, is used, a little water, or sometimes mercury occupying the bend. As mercury is nearly fourteen times denser than water, it is necessary that the delivery-tube, *d*, should be fourteen times longer than the distance that exists from the bottom of the curve in *f* to the surface, *g*, of the metal in the tube and arm of the safety-tube: if this is so managed, air will preferably enter by bubbling up through the mercury, than by forcing the column of water from *b* through *d* into *a**.

500. If, on the other hand, alcohol is to be saturated with ammoniacal gas, liquor ammoniæ may be gently heated in a retort, in contact with fragments of quartz, to facilitate escape of the gas, which, after passing through a U-tube filled with fragments of quicklime, may be passed into alcohol kept cold by immersing the flask containing it in water or otherwise. In this case it is unnecessary to employ a Welter's tube, because, if a glass tube passes through a cork in the tubulature of the retort and just dips into the fluid, air will enter by it, if any contraction takes place, and thereby prevent the recession of the alcohol.

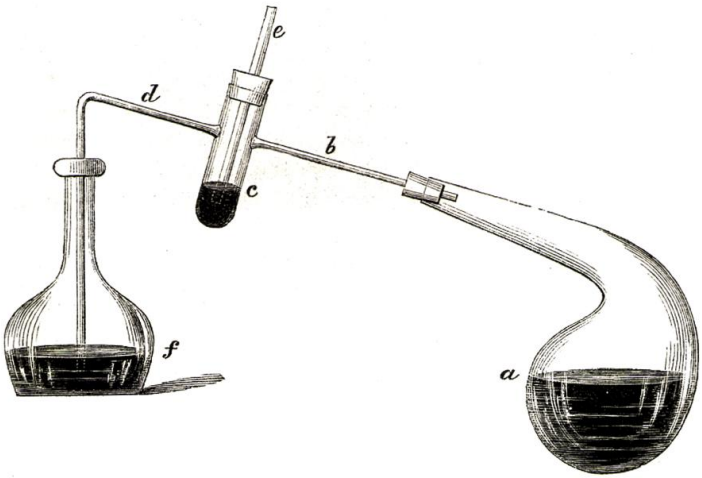
501. It has been said in the section on Distillation, that it is often required to add a portion of fluid to the contents of a retort during an operation, and this may be effected easily by a tube often used in experiments with gases. It also acts as a safety-tube. It is seen at several places in this volume, among others in fig. 237. When the fluid poured in at *f* rises above the bend of the tube over the bulb, it begins to flow over into the retort,

* A very simple method of constructing a Welter's safety-tube will be found in the section on Glass-working.

and thus any desired quantity may be added, and if contraction occurs, air enters through the liquid into the retort. The length of the column of fluid in the limb, *f*, must be sufficient to prevent any outward pressure from ejecting it and thus allowing escape of gas.

502. A very neat safety-tube, which is especially convenient in cases where the retort has no tubulature, is seen in fig. 238.

Fig. 238.




Muriatic acid gas is formed in *a* by the reaction of salt and sulphuric acid; the gas passes through *b* into a tube containing a little mercury, and thence through *d* into water contained in the bottle, *f*. The tube, *c*, is closed with a cork, through which passes a tube, *e*, dipping a short distance (about $\frac{1}{2}$ an inch) into the metal. On contraction taking place in *a*, air enters by *e* through the mercury and prevents the regurgitation of the acid in *f*. Another, but more expensive safety-tube, equally adapted with the last described to retorts which have no tubulature, is seen in fig. 239. One very important use of these safety-tubes is, that in the event of the exit of the apparatus becoming choked, the

retort is saved from bursting by the gas escaping in the direction of the arrows.

Fig. 239.

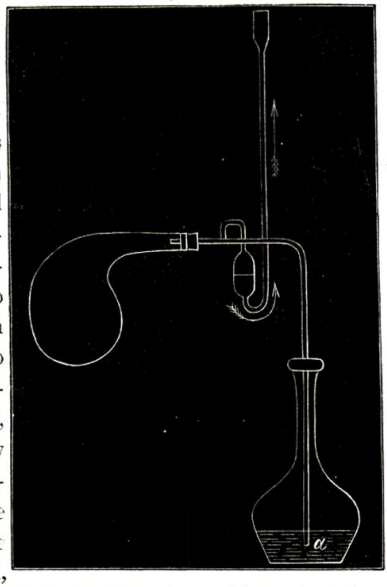


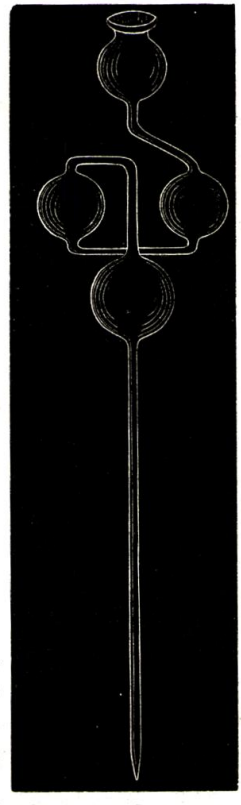

Fig. 240.

503. Two more forms of safety-tubes are seen in figs. 240 and 241. The former is constructed with glass valves, one of which opens inwards and the other outwards; they require, however, to be finished with extreme care to answer the intended purpose, and are certainly not more convenient than the common form. The other is intended, in the event of sudden condensation taking place in the retort, to prevent the fluid from finding its way in, and thus by mixing with the contents, spoil the result. It also has other and better uses, which will be seen by inspecting *d*, fig. 100.

504. A very convenient method of obtaining the most intimate contact of a gas with a fluid on which it is to exert a reaction, especially where the gas is valuable or the quantity at the operator's disposal is small, consists in passing it through the fluid contained in a potash-tube, as in fig. 242. The apparatus should be inclined by means of a cork, as is described in the chapter on the manipulation connected with organic analysis. This is particularly useful in experiments on the action of heat upon some organic bodies. On exposing the hydrochlorate of camphene to a high temperature in contact with lime, by passing it over frag-

ments of the latter in a long glass tube, both fluid and gaseous products are evolved. As they pass from the tube into a Woulfe's bottle protected by a screen of tin plate from the heat, and also immersed in cold water, the greater portion of the products obtained are condensed, only a few drops of an excessively volatile fluid being found in the U-tube cooled by a powerful freezing mixture, but a very considerable quantity of the gas is retained in the potash-tube filled with alcohol.

Fig. 241.



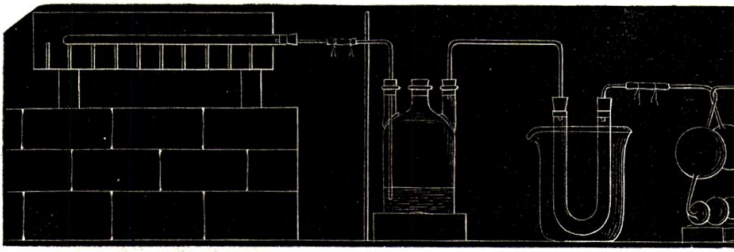
A similar method of condensing easily dissipated products should be adopted in submitting volatile acids to the action of the pile. Kolbe found that valyl (butyl) vapour was carried away with the gas formed by the electrolysis of valerianic acid, and I observed in electrolysing butyric acid the same to happen to a still greater extent in consequence of the superior volatility of propyl.

505. *The Gasometer.*—This is an indispensable piece of apparatus in all laboratories of research; and in consequence of the very general adoption of Hofmann's method of performing organic analysis, finds its way into places where otherwise its presence would not be required.

506. The gasometer, as modified for the last-named purpose, will be described in its place, under the head of Manipulation connected with Organic Analysis. The method of using the ordinary gasometer will be evident from the following description of fig. 243. The vessel, *a*, *b*, *c*, *d*, is a cylinder of copper or zinc, made air-tight, and surmounted by another much shorter one, *h*, *i*, *k*, *l*. These two are connected with each other by the tubes *e* and *f*, which also perform the office of supports

for the upper cylinder. The tube, *e*, descends to the bottom of the lower cylinder, and serves to allow the water in *h*, *i*, *k*, *l* to

Fig. 242.



descend to displace the gas which escapes by the cock, *b*, to the place where it is desired to be sent. The tube, *f*, on the other

Fig. 243.

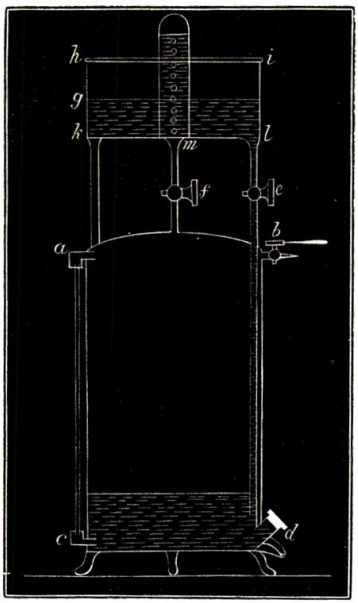
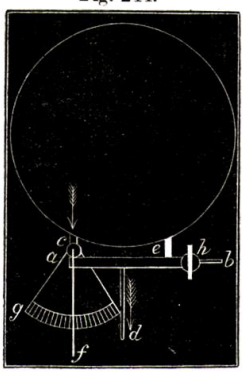



Fig. 244.




hand, only connects the upper with the lower cylinder, and does not descend into the latter; it is used to supply gas-jars, which for

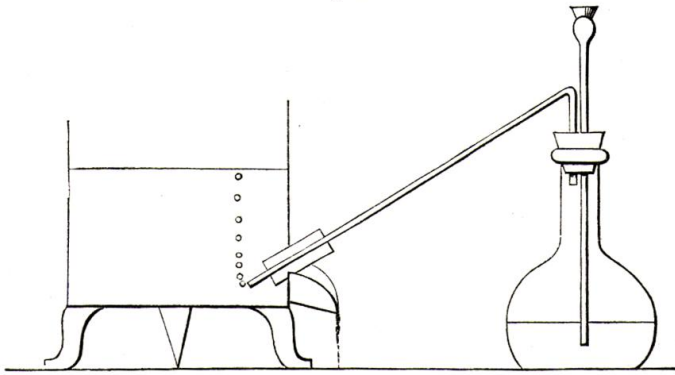
this purpose are filled with water at a cistern or other convenient place, conveyed to the water-tank, *h*, *i*, *k*, *l*, and placed immediately over the aperture, *m*. The glass tube, *a*, *c*, is used as a gauge to indicate the amount of gas, and for this purpose opens into the lower cylinder at the top and bottom, where it is fastened in with cement. In the figure, it is represented as standing out from the gas-holder; in general, however, it is better to have it sunk in, so as to incur less danger of fracture. The pipe with a cap, seen at *d*, is intended to introduce the gas from the apparatus where it is generated, and will be described further on.

To illustrate the method of using the instrument, let it be supposed that all the cocks are closed, and that the upper cylinder is half-filled with water. If, now, the tap, *e*, is turned so as to open a communication between the two, water will, it is true, descend for a short time; but *b* and *f* being closed, so that the air cannot escape, no result takes place, with the exception of a slight condensation, proportional to the height of the column of water. If, now, *f* is turned so as to open a passage between the vessels, bubbles of air will rise into the bell-jar placed to receive them. If *f* is shut off and *b* opened, the air will escape by the latter, and may by suitable connexions be carried to any desired spot. In some gasometers the part represented at *b* is of a somewhat more complex structure than appears by fig. 243. This part is seen in plan in fig. 244, as observed on looking downwards. The circle indicates the body of the gas-holder shown at *a*, *b*, *c*, *d*, in the previous engraving. In fig. 244, *a*, *b* represents a brass tube, connected by a rectangular bend, at *a*, with the body of the gasometer, and capable of being opened or closed against the passage of the gas by the stopcock, *c*. This latter has a lever, *f*, 7 or 8 inches long, attached to it, which moves over a small divided segment of a circle, *g*, enabling the operator not only to make smaller movements of the handle than could be done without it, but also to place it in exactly the same positions on several occasions, which would be impossible without some guide of this kind. It must be remembered, however, that the flow of gas will not always be of equal rapidity when

the handle is in the same position on the scale, unless the pressure in the chamber, *a*, *b*, *c*, *d* (fig. 243), is the same. The cross piece, fig. 244, *e*, parallel to *c*, is merely a support to assist in keeping the tube, *a*, *b*, immoveable. The aperture, *b*, which is capable of being closed with a stopcock, *h*, is intended to enable another gasometer, containing a different gas, to be connected with the first when necessary. The pipe, *d*, is the outlet for the gas, and is the part to which a flexible tube is generally attached, to direct the gas to its destination. The direction of the current is shown by arrows.

507. Fig. 245 is an enlarged view of *d* in fig. 243. It consists of a short wide tube, soldered into the side of the gaso-

Fig. 245.



meter, and so arranged that the lowest portion of the outer rim is higher than the highest portion of the inner rim. The tube is seen delivering gas into the gasometer, which has been filled with water by means of the upper cistern, until it escaped by *b*, fig. 243, the cap, *d*, being previously screwed on. The taps, *f* and *e*, being then closed, the cap, *d*, may be removed, and the gas-delivering tube inserted, when the water will escape by *d* as the vessel becomes filled with gas. When it is seen by the gauge that the lower chamber is filled with gas, or bubbles of the latter escape by *d*, fig. 243, the delivery-tube is withdrawn,

and the cap, *d*, screwed in its place: the further manipulations will be evident from what has been already said.

508. The operator, by the exercise of a little ingenuity, will easily be able to form a gas-holder out of a tin oil-can or even a glass carboy, for purposes which do not require any great amount of accuracy in workmanship.

In order to ascertain the tightness of the joints in the gasometer described, it is merely necessary to close the lower aperture, and pour water into the upper cylinder, at the same time opening *f*, *e* and *b*, and continuing the addition of water until, the gasometer being full, the water escapes at *b*; the cocks are then to be closed, and the cap, *d*, being removed, the apparatus is allowed to stand for some time, when if no leaks exist, the water will not escape; but if, on the contrary, the gauge indicates the descent of the water, some deficiency will be found either in the stopcocks or seams, which must be made good before using the apparatus.

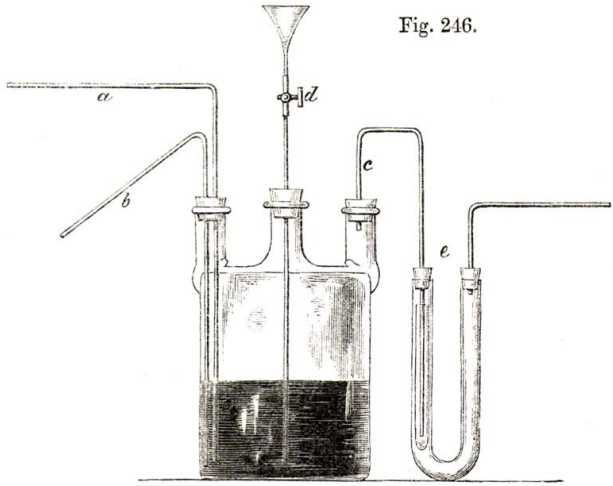
509. Where a very considerable pressure has to be met by the gas, as, for instance, in cases in which the delivery-tube dips deeply into mercury, the column of water in the gasometer may not be sufficient to overcome the resistance; to do away with this difficulty is easy: it is sufficient to fasten a long pipe with a funnel at the top, into the aperture at *l*, fig. 243, through which the water is to be introduced, and by increasing the length according to the amount of resistance, the gas may be conveyed into any required position.

510. The gas, as thus obtained, is saturated with moisture, and therefore in an unfit state to be used in some experiments. When it is suspected that the presence of water might prove injurious, it may be removed by passing the gas through a washing-bottle, or a Liebig's potash apparatus filled with sulphuric acid, or through a U-tube filled with fragments of pumice-stone moistened with sulphuric acid.

511. A very elegant, and at the same time convenient gasometer, particularly adapted for use in organic analysis, where it is desired to complete the combustion with a current of oxygen, is seen in fig. 246. It is constructed out of a large Woulfe's bottle, through the middle tubulature of which passes a tube to

which a stopcock, surmounted by a funnel, is cemented; this is intended to regulate the flow of the water which expels the gas. Another tubulature permits the passage of two tubes, one of which

Fig. 246.




passes to the bottom of the bottle and serves to admit the gas, the other, *b*, allows of the exit of the water as the oxygen enters. The gas as it escapes by *c*, on opening the stopcock, *d*, is dried by passing through the U-tube, *e*, in which is placed a small tube to retain water carried over mechanically, in order to prevent the too rapid moistening of the chloride of calcium contained in the other limb; it is necessary, when used in organic analyses, to pass the gas through a bottle containing sulphuric acid, so as to partially desiccate it before entering the chloride-of-calcium-tube, and also to allow of the rapidity of the flow of gas being made evident to the eye. In ordinary operations this may be dispensed with.

512. *Transference of gases at the pneumatic trough.*—To transfer a gas from one jar to another at the water-trough is an operation of ease or difficulty, according to the relative shapes and sizes of the vessels used. If it is merely required to transfer

from a plain to a graduated jar for the purpose of measurement, or to instance an equally common case, where it is required to add a certain quantity of one gas to a portion of another kind standing in a jar over water, and if the jars are tolerably large, it is necessary to advance that which is to receive the contents of the other about half its diameter over the shelf with the left-hand, while that which is to be *poured upwards* into it is to be steadily depressed with the right hand until its mouth is a short distance below the edge of the other. The jar in the right hand is then to be inclined in such a manner that it shall lean with its top away from the other, while the upper edge of its lower extremity projects a short distance under the bottom of that which is supported by the left hand. The decanting jar is then to have its highest portion steadily and slowly lowered, in such a manner that the contents flow upwards into the other in bubbles not too large, as in the latter case it is difficult to prevent them from escaping. It happens, somewhat unfortunately, that small tubes, which in experimental research are constantly used, are rather troublesome to transfer in this manner, as the ascending bubbles interfere with the descent of the water at the same time. This difficulty may be obviated by the use of funnels to direct the ascending gas, or by having the decanting tubes made with a lip. Or where it is merely desired to make gaseous mixtures of only approximative accuracy, as in lecture experiments, one of the lipped measures holding 3 or 4 ounces may be used.

513. Instances of transference of gases from the apparatus in which they were generated to the places where their action is to be exerted, have already been given.

514. When a gas standing over water is to be made to enter a bladder or gas-bag, a jar provided with a cap and stopcock must be used; a bladder is to be selected, closed with a brass piece in which a stopcock is inserted, capable of being joined to that in the gas-jar by means of the connectors described further on. Before allowing the gas to pass from the jar to the bladder, the latter must be moistened, and so squeezed that all the air is expelled; it should then have the connecting piece joined to it, and

the latter to the stopcock on the air-jar; the tap is now to be turned in such a manner as to open communication between the two; and the bell-jar being steadily and slowly depressed, the gas will be forced into the bladder; the taps may then be turned off, and the bladder, with its contents, be removed.

In all cases where a gas standing over water or mercury is to be made to enter a globe or bladder, care must be taken to stop the depression of the jar in time to prevent the fluid from being forced into the bladder.

To fill a jar, fitted with a stopcock, with water at the pneumatic trough, it is merely necessary to open the stopcock and depress the jar, exactly as in the last process, until all the air is expelled, and the water just reaches the brass-work of the cap, but no further; the tap is then to be turned off, and the jar raised full of water and placed on the shelf of the trough, ready to receive the gas from the delivery-tube.

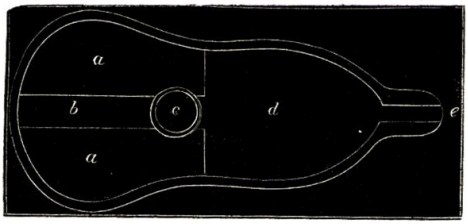
515. *Manipulation with gases over mercury.*—The chief difference in the modes of operating with jars at the mercurial and pneumatic trough, is caused by the great weight of the mercury. If a jar of moderate size, say holding 200 cubic centimetres, be filled with water from a jug or other convenient vessel, and its mouth is then covered with a glass plate, nothing can be easier than to invert the jar in the trough, and, by removing the plate, to leave the jar in a fit condition to receive the gas. But if mercury be used, the inexperienced operator will find that great care is required to keep the glass plate so tightly in its place as to prevent some of the metal escaping at the moment of inversion.

516. The operation may, however, be easily managed after a little practice, and taking care to press firmly with the two first fingers of the left hand upon the plate, while the right hand assists in raising up the other end of the jar. It must not be forgotten, that, from the great weight of the metal, a very slight blow will be sufficient to cause a fracture to a jar filled with it, and therefore it is necessary to have mercurial jars made stouter than those used at the water-trough. Whenever a jar has been filled with mercury, it is necessary to insert a glass rod, and with

it to bring to the surface any air-bubbles which may be seen between the metal and the glass, as they are liable to cause considerable errors in delicate experiments if allowed to remain.

517. The expense of mercury being considerable when required in large quantities, mercurial troughs are generally made as small as possible, and it is necessary therefore, in almost all cases, to fill the jar from some convenient vessel, and to transfer it to the trough. Stoneware mercurial troughs are in common use, and, if carefully used, very convenient; but it must not be forgotten that their hardness somewhat endangers the safety of any jars that may be allowed to strike the side with even a slight blow. The kind of porcelain trough in most common use is represented in figs. 247 and 248, where the first shows a plan, and the

Fig. 247.

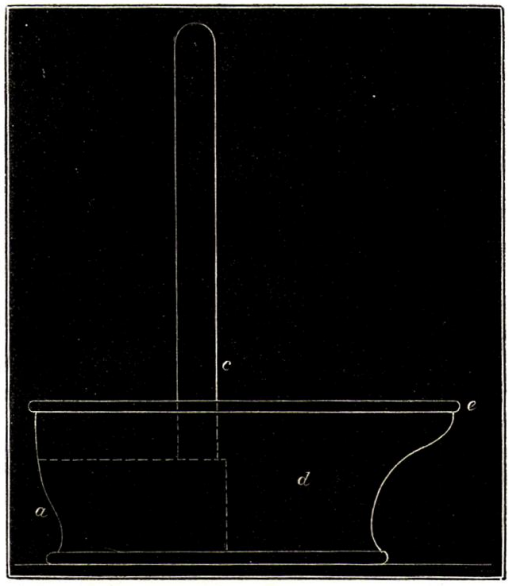



second, fig. 248, a section of it, with the position occupied by a jar. In fig. 247, *a a* represent two projections, one on each side of the interior of the trough, which serve to support the tube, *c*. The hollow space between these two solid portions shown at *b*, is intended to admit the delivery-tube; *d* shows the body of the trough, and *e*, a spout by which the mercury may be poured off when the instrument is done with. As the same letters refer to the same portions of fig. 248, no further description is necessary.

518. Mr. Griffin has constructed a stoneware trough for minute experiments, capable of being worked with only four pounds of mercury, and yet allowing tubes 6 inches in length, and nearly 1 inch in diameter, to be inverted, when full, with safety. It is seen in plan and section in figs. 249 and 250. The part at *a* is on the principle of the bee-hive shelf, which latter is a contri-

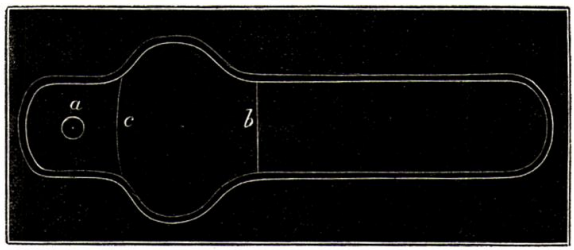
vance of the same person. Between *b* and *c* there is a cavity

Fig. 248.




capable of admitting a glass tube 2 inches long by $\frac{1}{2}$ an inch wide, to enable caustic potash or any other substance required to

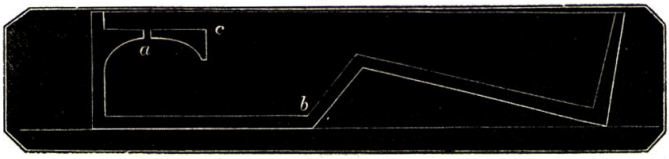
Fig. 249.




be passed into the gas-jar. There are also two recesses, to enable the thumb and finger to enter the trough, to manage the tube used to introduce the reagents alluded to.

519. It is essential to use these troughs upon a tray, in order

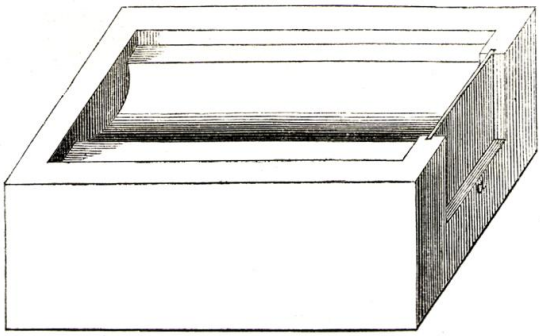
Fig. 250.




that the mercury, small portions of which will inevitably be spilled, may be gathered up with facility. As soon as the metal is done with, it should be poured away into an iron bottle kept for the purpose.

520. Fig. 251 represents Bunsen's wooden mercurial trough,

Fig. 251.

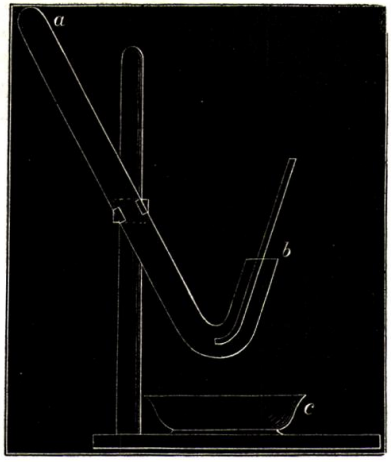



which is exceedingly well adapted for eudiometrical experiments. It is made of wood, and has a piece of plate-glass inserted at *a*, by slipping between two grooves. It does not require so much mercury as would appear from its size.

521. A most excellent substitute for a mercurial, and even for a water-trough in many experiments, is the instrument shown in fig. 252, and known as Cooper's mercurial receiver. It consists of a glass tube, *a b*, closed at one end, and bent upwards, as seen in the figure. It is filled with the metal by inverting and

pouring it in at *b*. On again inverting it cannot fall out, in consequence of the pressure of the atmosphere being unable to act upon the end, *a*. If, now, the delivery-tube, as seen in the engraving, is made to enter, and gas is sent in, the mercury descends and escapes by *b*; but the operation is concluded when the gas occupies from *a* to the bend of the tube. One of the great advantages of this contrivance, is the facility which it affords for making a great number of experiments upon a small quantity of gas; for if the thumb be placed upon the end, *b*, and the tube is so inclined as to permit a little of the gas to flow round the bend into the part *b*, so as to occupy a space of from ½ to 1 inch between the mercury and the thumb, it may be examined as to its chemical or physical characters, such as smell or inflammability. And by filling up the vacant space with mercury, another portion may be made to occupy the same position, and may also be examined, and so on until all the gas has been used. The basin, *c*, fig. 252, is placed to catch the mercury which falls on the admission of the gas.

Fig. 252.




522. It is necessary in all cases to guard against the great pressure exercised by mercury as compared with water, a pressure which is as their respective densities; and as mercury is 13·5 times heavier than an equal bulk of water, it is evident that a tube dipping 1 inch into mercury will exercise as great a pressure as if it dipped into 13·5 inches of water; this pressure must be provided against by increased security of all the joints of the apparatus; and for the same reason it is essential not to allow

the tube delivering a gas to dip to an unnecessary extent under the surface of the metal.

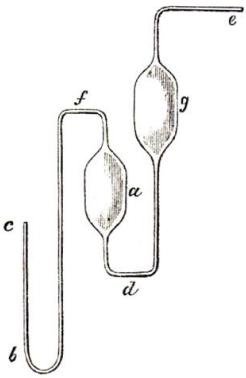
523. It is advisable in all cases where a gas is to be passed into a tube over mercury, to make it turn up at the end, in order to throw the gas up at once, and prevent that tendency to escape, by returning along the outside of the tube, which is sometimes perceived in experiments at the mercurial trough.

524. When a dish is placed under a jar of mercury in order to remove it to the mercurial trough, care must be taken that it is not too thin, for as more force is applied in lifting such a weight than when water is used, there is some danger of breaking the basin unless it is tolerably stout.

525. *Transference of gases.*—One of the most common operations in experiments upon gases, is to remove a portion from a quantity standing over water or mercury for the purpose of submitting it to further examination, such, for instance, as ascertaining its behaviour towards various reagents. Sometimes, however, it is desired to remove a portion of a gas standing over water to a tube at the mercurial trough, and yet it is necessary to avoid the presence of water in the latter vessel. The late Dr. Henry describes, in his 'Elements of Chemistry,' a transferrer which was invented by Cavendish:—"A tube, 8 or 10 inches long, and of very small diameter, is drawn out to a fine bore, and bent at the end, so as to resemble the italic letter *l*. The point is then immersed in quicksilver, which is drawn into the tube till it is filled by the action of the mouth. Placing the finger over the aperture at the straight end, the tube filled with quicksilver is next conveyed through the water, with the bent end uppermost, into an inverted jar of gas. When the finger is removed, the quicksilver falls from the tube into the trough, or into a cup placed to receive it, and the tube is filled with the gas. The whole of the quicksilver, however, must not be allowed to escape, but a column is to be left a few inches long, and kept in its place by the finger. The tube is to be removed from the water and dried by an assistant with a towel or with blotting-paper; the point of the bent tube is then to be introduced into the aperture

of the tube standing over quicksilver, and on withdrawing the finger from that aperture, which is now uppermost, the pressure of the column of quicksilver, added to the weight of the atmosphere, will force the gas from the bent tube into the one standing in the mercurial trough." But since the time when Cavendish contrived the transferrer last described, many instruments have been invented for the same purpose, some of which serve also to enable gases to be treated with reagents away from the measuring-tube. The annexed cut, fig. 253, represents one of the most convenient of them; it illustrates the shape most commonly employed, but several modifications are adopted in special cases of research. For the double purpose of transferring a gas and treating it with a reagent, the tube, *a*, is partly filled with mercury and partly with the liquid, to the action of which the gas is to be subjected. The tube, *b c f*, is also filled with the metal. The portion *b c* is introduced under, and then up into the jar containing the gas to be experimented upon, and the lips being applied at *e*, the mercury is drawn from *a* into *g*, and of course at the same time the gas passes through *c b* and *f* into *a*. As it is now essential that the tube, *b c*, should be closed, that is done, if the gas alluded to stands over mercury, by merely depressing the aperture, *c*, beneath the surface of the metal, and applying a slight suction to *e*.

Fig. 253.

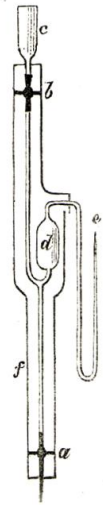



As soon as the action of the absorbent is completed, which may be facilitated by agitation, and it is desired to pass the gas into the measuring-jar, or into one where it may be intended to act upon it with some other reagent, the aperture, *c*, being again depressed below the mercury, the metal is to be drawn in by the same method as before, until it stands higher in *d g e*; the tongue for a second closes *e*, and then a moistened finger is dexterously

substituted for the tongue. To cause the passage of the gas into the jar, it is merely necessary to gently withdraw the finger from covering the orifice, when the pressure of the mercury in *g* will force the gas into the vessel.

A tranferring pipette, which is extremely easy to use, has been devised by Dr. W. A. Miller. It consists of a system of glass tubes, having at the centre a bulb, *d*, fig. 254, with a capacity of about a cubic inch. A funnel, *c*, holding enough mercury to fill the bulb and all the tubes, is joined to a steel stopcock*, *b*, which at its inferior extremity is cemented to a long tube communicating with the bulb. From the latter proceed two tubes, the lower of which descends and is cemented into a second steel stopcock, *a*, while the upper tube is bent four times at right angles, and terminates in a fine orifice, *e*. To use the instrument, the stopcock, *a*, is opened, and mercury poured in at *c* until it descends and fills the tube, *f*, escaping by *a*; the latter is then closed; the metal still running in gradually fills the bulb, *d*, and escapes by *e*; the stopcock, *b*, is then shut. If the gas to be transferred is in a tube over mercury or water contained in a tall cylinder, *e* must first be depressed beneath the surface of the fluid, and then raised inside the cylinder of gas; *a* is now to be opened so as to allow the mercury to escape, which will cause the gas to enter by *e* and fill *d*; the orifice, *e*, is then to be depressed beneath the metal, and a little allowed to enter, so as to close the aperture. After this, the pipette may be removed, any communication between the gas in *d* and the external air being shut off by the mercury in *e*. In order to transfer the gas to another tube over mercury or water, it is necessary to fill the funnel, *c*, with mercury, and introduce *e* into the tube as before. On opening *b*, the metal will enter *d*, dis-

Fig. 254.




* I have constructed this pipette with the stopcocks of vulcanized caoutchouc, on the principle represented in fig. 189, p. 221, and have found them to answer excellently.

placing the gas, which may then be measured, due attention being given to the pressure, temperature, &c., after which it may be subjected to any desired treatment. The whole apparatus is fastened to a board to prevent fracture.

526. The analysis of gases having now become a problem which the chemist has frequently to solve, it is necessary to describe the instruments which have been found to yield the most accurate results. These are undoubtedly those of Regnault and Messrs. Frankland and Ward. The latter gentlemen's apparatus is a modification of Regnault's, and possesses certain advantages over it in practice. For anything more than the descriptions following, the student is referred to Regnault's 'Elements of Chemistry,' an excellent translation of which has been made in America by Dr. Betton, edited by Messrs. Booth and Faber; and also to the paper of Messrs. Frankland and Ward in the 'Quarterly Journal of the Chemical Society.'

527. The apparatus of M. Regnault is seen by reference to figs. 255 and 256. The former represents a geometrical projection of the anterior surface, and fig. 256 gives a vertical section made through a plane perpendicular to this face.

528. "The apparatus is composed of two parts, which may be separated and united at pleasure; and while the first, or *the measurer*, serves to measure the gas under given conditions of temperature and moisture, in the second the gas is subjected to various absorbent reagents, on which account we shall call it the *absorption-tube*.

"The measurer is composed of a tube, $a\,b$, of 15 to 20 millimetres diameter internally, divided into millimetres, and terminating above by a curved capillary tube, $b\,e\,r'$, while the lower end is luted into a cast-iron piece, $p'\,q'$, having two tubulures, $a$, $i$, and a stopcock, R.

529. "To the second tubulure, $i$, is luted a straight tube, $i\,h$, open at both ends, of the same diameter as the tube, $a\,b$, and also divided into millimetres." The stopcock, R, is somewhat peculiar, and will be found fully described in a subsequent por-

tion of this volume *, where it is seen in the three principal positions in which the key may be turned. "A communication can

Fig. 255.

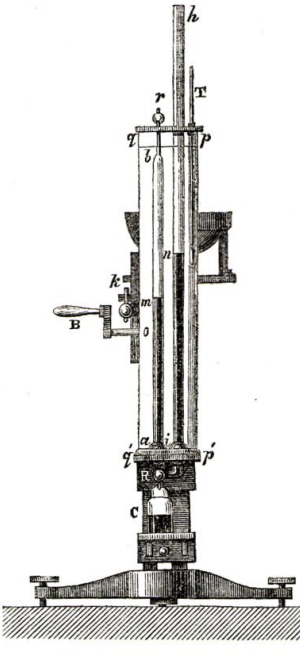
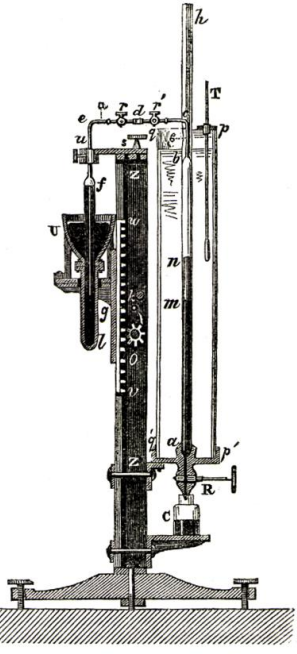



Fig. 256.




therefore be established at will between the tubes $a\,b$, $i\,h$, or one or other of these tubes only may be opened to the external air.

"The two vertical tubes and the cast-iron piece form a manometric apparatus contained in a glass cylinder, $p\,q\,p'\,q'$, filled with water, which is maintained at a constant temperature, marked by the thermometer, T, during the whole time of the analysis. The manometric apparatus is fixed on a cast-iron stand, Z Z', furnished with adjusting screws.

530. "The absorption-tube is composed of a bell-glass, $g\,f$, open

* On the analysis of certain gases by the method of ultimate organic analysis.

at the bottom, and terminated above by a curved capillary tube, *f e r*. The bell-glass dips into a small mercurial bath, U, of cast-iron, exactly represented in figure 257, while the basin, U, is fixed on a plate which can be raised at will along the vertical support, Z Z′, by means of the toothed rack, *v w*, which works with a toothed pinion, *o*, set in motion by the crank, B. The ratchet, *r*, arrests the toothed racks, and consequently keeps the basin, U, in any given position. A counterpoise affixed to the ratchet facilitates its working, and as it is turned to one side or the other, the ratchet is thrown in or out of gear with the pinion. The ends of the capillary tubes, which terminate the absorption-tube and measurer, are luted to two small steel stopcocks, *r*, *r*′, the ends of which exactly fit each other, and have the same shape as those represented in figures 258 and 259 in section*. Fig. 258 represents a section of stopcocks, *r r*′. It will be seen that the first tubulure is terminated by a plane surface, *a b*, and a projecting cone, *c*, while the second has also a plane surface, *a*′ *b*′, and a hollow cone, *c*′, which exactly fits the plane surface and projecting cone of the other. In order to close them hermetically, it is sufficient to press the two parts against each other by means of the clamp (fig. 260, and in section, fig. 261), which is to be tightened with screws, after having poured in a small quantity of melted caoutchouc.

Fig. 257.

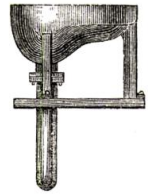


Fig. 258.

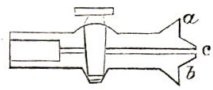



Fig. 259.

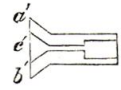



Fig. 260.

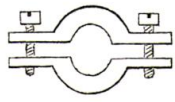


Fig. 261.

" The absorption-tube is maintained in a vertical position by means of pincers, *u*, lined with cork, which are easily

* This description is introduced for completeness here, although in Regnault it is placed in a different part of the work.

opened or closed when the tube is to be removed or replaced. The measurer, $a\,b$, is traversed at $b$ by two platinum wires opposite to each other, the ends of which approach to the distance of a few millimetres from the inside of the bell-glass, and of which the other ends are fastened with wax to the lower edge of the large cylinder. The electric spark is passed into the bell-glass by means of these wires, and the water in the cylinder is no obstacle if the spark be furnished by a Leyden jar.

531. "Let us suppose that in this apparatus a mixture of atmospheric air and carbonic acid is to be analysed.

532. "Through the tube, $i\,h$, the measurer, $a\,b$, is filled with mercury, until the latter escapes through the stopcock, $r$, which is then closed, and at the same time the absorption-tube, $g\,f$, is filled with mercury, to effect which the tube, $g\,f$, is detached from the pincers, $u$, and plunged into the bath, U, the stopcock, $r$, being opened; and the operator sucks with a glass tube furnished with a caoutchouc tubulure, the edge of which is applied to the plane part of the tubulure, $r$. When the mercury begins to escape, the stopcock, $r$, is closed.

"The gas to be analysed, which has been collected under a small bell-glass, is then introduced into the absorption-tube, and the extravasation is easily performed in the bath, U, on account of the shape given to the latter. The absorption-tube being then replaced by the pincers, $u$, the two tubulures, $r, r'$, are fitted to each other; then, elevating one end of the bath, U, and allowing the mercury of the measurer to flow from the other through the cock, R, and lastly, opening the stopcocks, $r, r'$, the gas is caused to pass from the absorption-tube into the measurer. When the mercury begins to rise in the capillary tube, $f\,e$, its escape through the stopcock, R, is slackened, so as to cause the mercury to rise very gently in the tube, $f\,e\,r$, and the cock, $r$, is closed when the mercurial column reaches a mark, $a$, on the horizontal leg, $e\,r$, at a small distance from the tubulure, $r$. The level of the mercury is then brought to a given division, $m$, of the tube, $a\,b$, and the difference in height of the two columns can immediately be read on the scale of the tube, $i\,h$. The water in the cylinder has been

several times agitated throughout by blowing air into it by means of a tube which descends to the bottom.

533. "Let $t$ be the temperature of the water, which is to be stationary during the analysis; $f$ the elastic force of the aqueous vapour saturated at this temperature; V the volume of the gas; H the height of the barometer; and lastly, $h$ the height of the mercury elevated: then will $H+h-f$ be the elastic force of the gas when supposed dry. The temperature of the water in the cylinder should be nearly that of the surrounding air, which does not vary sensibly during the short duration of the experiment; and it is unnecessary therefore to reduce to 32°, by calculation, the height of the barometer, and that of the mercury elevated in the manometric apparatus, $a\ b\ i\ h$. The gas collected in the measurer is, moreover, always saturated with moisture, because the sides of the tube, $a\ b$, are moistened with a small quantity of water; and this is constantly the same, since it is that which the mercury does not remove when the tube is filled with it.

534. "When this is done, the mercury is again allowed to flow through the stopcock, R, and the cock, $r$, is opened, in order to allow all the gas, as well as a column of mercury, to pass into the tube, $r\ c\ b$, after which the stopcock, $r'$, is closed. The absorption-tube is then detached, and a drop of a concentrated solution of potassa is passed up by means of a curved pipette, when the absorption-tube is again fitted to the measurer, and the bath, U, allowed to fall to its full extent; and then, after having poured a large quantity of mercury into the tube, $h\ i$, the stopcocks, $r$, $r'$, are successively opened. The gas thus passes from the measurer into the absorption-tube, and the small quantity of solution of potassa completely moistens the sides of the bell-glass. The cock, $r$, is closed when the mercury begins to fall in from the measurer into the vertical leg, $e\ f$, of the absorption-tube; and, after waiting for a few moments, in order to give time for the absorbing action of the potassa, the gas is passed from the absorption-tube back into the measurer, by causing the bath, U, to ascend, and the mercury to flow through

the cock, R. As soon as the alkaline solution begins to rise in the tube, $f\ e$, an inverse movement is caused by closing the stopcock, $r$; that is, the gas is again passed from the measurer into the absorption-tube, by lowering the bath, U, and again pouring mercury into the tube, $i\ h$. The intention of this operation is to again moisten the sides of the bell-glass, $f\ g$, with the solution of potassa, and subject the gas to the absorbing action of the new layer of potassa.

535. "If it be deemed necessary, these operations may be repeated several times; although, after the second, the whole of the carbonic acid is generally absorbed. The gas is then passed for the last time from the absorption-tube into the measurer, and the cock, $r$, is closed when the top of the alkaline column reaches the mark $a$. The level of the mercury in the tube, $a\ b$, being brought to $m$, the difference of height, $h'$, of the mercury in the two legs, $a\ b$ and $i\ h$, is measured, and the height, H', of the barometer is noted down. We shall suppose that the temperature of the water in the cylinder has not changed; if otherwise, it must be restored to the temperature, $t$, by the addition of hot or cold water.

536. "The elastic force of the gas, dry and deprived of carbonic acid, is therefore $(H' + h' - f)$; and consequently $(H + h - f) - (H' + h' - f) = H - H' + h - h'$ is the diminution of elastic force caused by the absorption of the carbonic acid; and

$$\frac{H - H' + h - h'}{H + h - f}$$

represents the proportion of carbonic acid in the gas when supposed dry.

537. "The proportion of oxygen which exists in the gas remaining must now be determined; for which purpose the absorption-tube is detached, and washed several times with water. It is dried first with tissue-paper, and then by bringing it into connexion with an air-pump; and, lastly, after having filled it with mercury, it is fitted to the measurer. The bath, U, being raised as high as possible, the mercury is allowed to run

through the stopcock, R; then opening carefully the cocks, $r$ and $r'$, the mercury of the absorption-tube is passed into the tube, $a\ r'$, of the measurer, taking care to close the cock, $r'$, when the extremity of the mercurial column reaches a second mark, ℭ, on the vertical leg, $b\ c$. The mercury in the measurer is again brought to the level, $m$, and the difference of level, $h''$, and the height, H'', of the barometer is ascertained. $H'' + h'' - f$ is therefore the elastic force of the dry gas, the quantity of which is somewhat smaller than in the measure made immediately after the absorption of the carbonic acid, because a small quantity (about $\frac{1}{3000}$) has been lost by detaching the absorption-tube from the measurer.

"This small loss does not affect the result of the analysis, because the gas is again measured.

538. "The absorption-tube being once more detached from the measurer, the hydrogen gas intended to burn the oxygen is now introduced into the latter, by arresting the ascending mercury at the mark ℭ. The mercury is again levelled to $m$, the difference of height, $h'''$, of the two columns of mercury measured, and the height, H''', of the barometer observed. $H''' + h''' - f$ is therefore the elastic force of the mixture of hydrogen and oxygen to be analysed. As sometimes required for the perfect admixture of the gases, combustion by the electric spark cannot be immediately effected. The gas must again be passed from the measurer into the absorption-tube, and a small quantity of mercury, which produces an agitation in the gas, allowed to flow through the tube, $c\ d\ e\ f$; and, lastly, the mixture is passed back into the measurer, this time allowing the mercury to entirely fill the tube, $r'\ c\ b$, in order that the whole volume of gas may be subjected to combustion.

539. "The electric spark is then applied, and after having established an excess of pressure in the measurer, $a\ b$, the stopcocks, $r$, $r'$, are carefully opened, in order to allow the mercurial column to retrograde into the tube, $b\ c\ r'$; and it is stopped when it reaches the mark ℭ. The elastic force of the gas re-

maining is again measured, after having levelled the mercury to $m$; and $H'''' + h'''' - f$ is then the elastic force. Consequently, $(H''' + h''' - f) - (H'''' + h'''' - f) = H''' - H'''' + h''' - h''''$ is the elastic force of the gaseous mixture which disappeared during the combustion; $\frac{1}{3}(H''' - H'''' + h'''')$ is the elastic force of the oxygen contained in the dry gas, of which the elastic force is $(H'' + h'' - f)$, and

$$\frac{1}{3}\,\frac{H''' - H'''' + h''' - h''''}{H'' + h'' - f}$$

is the proportion of oxygen contained in the gas when freed from carbonic acid; whence the proportion of oxygen in the original mixture may be easily deduced.

540. "The example chosen shows the mode of operating with the apparatus; the manipulations are of such a simple character, that the operator requires no assistant; and, lastly, the operation is so rapid, that less than three quarters of an hour is required for that just described; the greater portion of which time is consumed by the absorption of the carbonic acid and the cleansing of the bell-glass after the experiment. Air freed from carbonic acid can be analysed in less than twenty minutes."

541. The above will, it is hoped, sufficiently illustrate the nature and mode of using this admirable instrument, but for any other points which may require elucidation, the reader is referred to the work cited.

542. Messrs. Frankland and Ward, the first of whom has long been well known for his dexterity in the manipulation connected with gas analysis, while admitting the advantages which the apparatus just described possesses over even the very accurate method of Bunsen, the latter requiring the use of a room capable of being maintained at a standard temperature, take exception to M. Regnault's method of determining volumes by ascertaining the pressure of a constant volume greater or less than that of the atmosphere as expressed in millimetres of mercury, as having the disadvantage of expressing large variations in

bulk by small numbers, as compared with Bunsen's method. They illustrate the force of their remarks by analyses made on a mixture of air with a very small portion of carbonic acid, where they show that an error of observation equal to $\frac{1}{10}$th of a millimetre had an effect seven times greater with Regnault's method, than when that of Bunsen was adopted. They commence the description of their process, which is intended to combine the advantages of both methods, by stating the requisites of a perfect instrument; these are,—

543. "1. The determinations of the gaseous volumes should be made in a manner entirely independent of the pressure and temperature of the external atmosphere.

"2. Such determinations of volume should also be self-correcting, as regards the tension of aqueous vapour and the variations in the density of mercury.

"3. Each change of volume should be expressed by a numerical difference as large as possible.

"4. In order to avoid the inconvenience and loss of time occasioned by tedious calculations, references to tables, &c., the numerical expression of each volume actually read off should either be the true and corrected volume, or a number from which such volume can be at once obtained by the most simple arithmetical process.

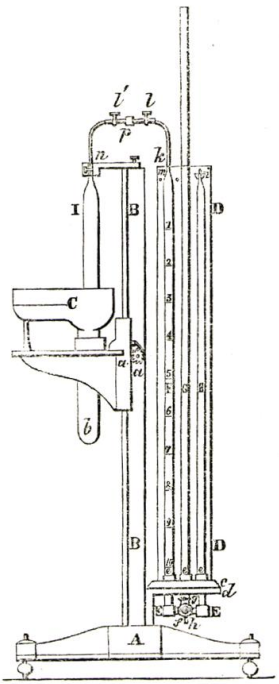
"The advantage of securing the first and second of these qualifications is sufficiently obvious; for whilst we can then carry on the operations of gaseous analysis without an apartment of nearly constant temperature set apart for the purpose, the accuracy of our results is also not liable to be affected by any errors that may have been made in the determination of the expansion of gases by heat, the tension of aqueous vapour, and the expansion coefficient of mercury.

"The third and fourth qualifications are also scarcely of inferior importance, the one for securing delicacy in the readings, and the other for economizing time, and enabling the operator to see the corrected results of his experiments during their progress, an advantage which can best be appreciated by

those who have had to make large numbers of consecutive gas analyses.

"In constructing an apparatus to combine the foregoing qualifications, we have taken as a model the ingenious and very convenient arrangement of MM. Regnault and Reiset, with the mechanical arrangement of which our apparatus, though different in principle, has much in common.

Fig. 262.




544. "Our instrument, which is represented by the accompanying figure, consists of the tripod A, furnished with the usual levelling screws, and carrying the vertical pillar, B B, to which is attached on the one side the moveable mercury-trough, C*, with its rack and pinion, *a a*, and on the other the glass cylinder, D D, with its contents. This cylinder is 36 inches long and 4 inches internal diameter; its lower extremity is firmly cemented into an iron collar, *c*, the under surface of which can be screwed perfectly water-tight upon the bracket plate, *d*, by the interposition of a vulcanized caoutchouc ring. The circular iron plate, *d*, is perforated with three apertures, into which the caps, *e e e*, are screwed, and which communicate below the plate with the T-piece, E E. This latter is furnished with a double-way cock, *f*, and a single-way cock, *g*, by means of which the tubes cemented into the sockets, *e e e*, can be made to communicate with each other, or with the exit-pipe, *h*, at pleasure.

* The mercury-trough, including its tubular well, *b*, may be conveniently constructed of gutta percha.

"F G H are three glass tubes, which are firmly cemented into the caps, *e e e*. F and H, which are only slightly shorter than the glass cylinder, are each from 15 to 20 millimetres internal diameter, and are selected of as nearly the same bore as possible, to avoid a difference of capillary action. The tube, G, is somewhat wider, and may be continued to any convenient height above the cylinder. H is accurately graduated with a millimetre scale, and is furnished at top with a small funnel, *i*, into the neck of which a glass stopper, about 2 millimetres diameter, is carefully ground. The tube, F, terminates at its upper extremity in the capillary tube, *k*, which is carefully cemented into the small steel stopcock, *l*. F has also fused into it, at *m*, two platinum wires, for the passage of the electric spark. After this tube has been firmly cemented into the cap, *e*, its internal volume is accurately divided into ten perfectly equal parts, which is effected without difficulty by first filling it with mercury from the supply-tube, G, up to its junction with the capillary attachment, and then allowing the mercury to run off through the nozzle, *h*, until the highest point of its convex surface stands at the division 10, previously made, so as exactly to coincide with the zero of the millimetre scale on H; the weight of the mercury thus run off is carefully determined, and the tube is again filled as before, and divided into ten equal parts, by allowing the mercury to run off in successive tenths of the entire weight, and marking the height of the convexity after each abstraction of metal. By using the proper precautions with regard to temperature, &c., an exceedingly accurate calibration can in this way be accomplished.

545. "The absorption-tube, I, is supported by the clamp, *n*, and connected with the capillary tube, *k*, by the stopcock and junction-piece, *l' p*, in exactly the same manner as in Regnault's apparatus.

546. "When the instrument is thus far complete, it is requisite to ascertain the height of each of the nine upper divisions on the tube above the lowest or tenth division. This is very accurately effected in a few minutes, by carefully levelling the instrument,

filling the tube, G, with mercury, opening the cock, *l*, and the stoppered funnel, *i*, and placing the cock, *f*, in such a position as to cause the tubes, F H, to communicate with the supply-tube, G. On now slightly turning the cock, *g*, the mercury will slowly rise in each of the tubes, F and H; when its convex surface exactly coincides with the ninth division on F, the influx of metal is stopped, and its height in H accurately observed; as the tenth division on F corresponds with the zero of the scale upon H, it is obvious that the number thus read off is the height of the ninth division above that zero-point. A similar observation for each of the other divisions upon F completes the instrument.

547. "Before using the apparatus, the large cylinder, D D, is filled with water, and the internal walls of the tubes, F and H, are once for all moistened with distilled water by the introduction of a few drops into each through the stopcock, *l*, and the stoppered funnel, *i*. The three tubes being then placed in communication with each other, mercury is poured into G, until it rises into the cup, *i*, the stopper of which is then firmly closed. When the mercury begins to flow from *l*, that cock is also closed. The tubes, F and H, are now apparently filled with mercury, but a minute and imperceptible film of air still exists between the metal and glass; this is effectually got rid of by connecting F and H with the exit-tube, *h*, and allowing the mercury to flow out until a vacuum of several inches in length has been produced in both tubes; on allowing the instrument to remain thus for an hour, the whole of the film of air above mentioned will diffuse itself into the vacuum, and will become visible as a minute bubble in each tube, on allowing the vacuum to be filled up from the supply-tube, G. These bubbles are, of course, easily expelled, on momentarily opening the cock, *l*, and the stopper, *i*, whilst G is full of mercury. The absorption-tube, I, being then filled with quicksilver, and attached to *l* by the screw-clamp, the instrument is ready for use.

548. "In localities where a constant supply of water from street mains can be had, the temperature of the water in the cylinder, D D, can be maintained perfectly constant, by allowing a con-

tinuous stream direct from the main to flow into the bottom of the cylinder, and make its exit near the top. By this arrangement, it has been proved by one of us, in an extensive series of experiments, that the temperature of a cylinder supplied from the Manchester high-pressure mains, does not vary more than 0°·02 Centigrade in twelve hours, a variation which scarcely requires correction in the most delicate experiment.

"In illustration of the manner of using our apparatus, we will take as an example an analysis of atmospheric air. A few cubic inches of air freed from carbonic acid having been introduced into the tube, I, it is transferred into F for measurement by opening the cocks, *l l'*, and placing the tube, F, in communication with the exit-pipe, *h*: the transference can be assisted, if needful, by elevating the trough, C. When the air, followed by a few drops of mercury, has passed completely into F, the cock, *l*, is shut, and *f* turned, so as to connect F and H with *h*. Mercury is allowed to flow out until a vacuum of 2 or 3 inches in length is formed in H, and the metal in F is just below one of the divisions; the cock, *f*, is then reversed, and mercury very gradually admitted from G, until the highest point in F exactly corresponds with one of the divisions upon that tube: we will assume it to be the sixth division. This adjustment of mercury, and the subsequent readings, can be very accurately made by means of a small horizontal telescope placed at a distance of about 6 feet from the cylinder, and sliding upon a vertical rod. The height of the mercury in H must now be accurately determined; and if, from the number thus read off, the height of the sixth division above the zero of the scale in H be deducted, the remainder will express the true volume of the gas; but in order to compare this with subsequent readings made at other divisions upon F, the number thus obtained, which evidently represents the pressure of the gas, is reduced to what it would have been had the gas been expanded to the tenth division of F. Bearing in mind that the pressure of a gas is inversely as its volume, this reduction is very simply effected by multiplying the number as above obtained by $\frac{6}{10}$ or 0·6; and in all cases any determination of pressure made at any division

upon F may be reduced to the pressure of the same volume when expanded to the tenth division, by the use of a fractional multiplier whose denominator is 10, and numerator the number of the division at which the determination is made.

549. "As the temperature is maintained constant during the entire analysis, no correction on that score has to be made; the atmospheric pressure being altogether excluded from exerting any influence upon the volumes or pressures, no barometrical observations are requisite; and as the tension of aqueous vapour in F is exactly balanced by that in H, the instrument is in this respect self-correcting.

550. "Hydrogen being then introduced in the same way as the original gas, and the volume determined anew, the electric spark is passed through the mixture by means of the platinum wires at *m*, and the determination of the contraction caused by the explosion terminates the analysis."

551. *Eudiometers.*—It would perhaps have been more in accordance with the ordinary modes of arrangement, to have introduced the more simple kinds of eudiometer first, and then to have proceeded to a description of the more complex; but it has been preferred to reverse this,—to give the more perfect methods the priority, and to make the others merely supplemental to them.

There are several instruments in use for the analysis of gases by the passage of the electric spark through mixtures, but before proceeding to notice these, it will be proper to call attention to a few precautions which are necessary to ensure success with all of them. The spark is obtained either from an electrophorus, the prime conductor of an electrical machine, or a Leyden jar*. In the case of the two former, it is essential that every facility should be given to enable the spark to pass as a bright distinct flash, and not in the feeble manner in which the electric fluid is evolved from points. It is necessary, therefore, where the source of electricity is not sufficiently powerful, that extra care should be taken to ensure roundness of the ends of the wires in the interior

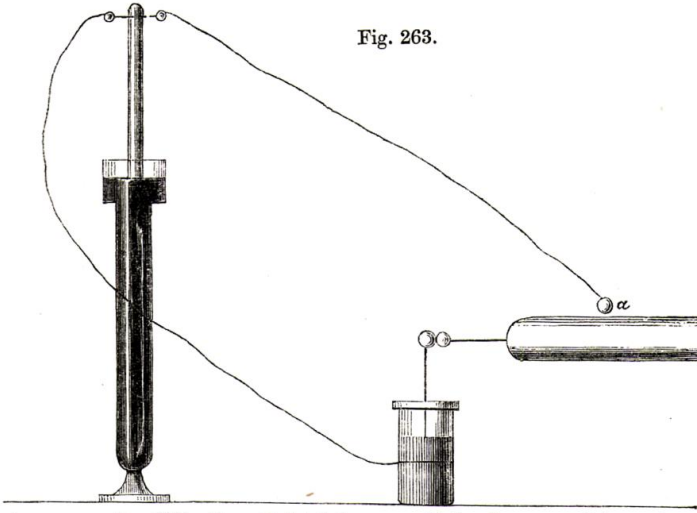
* In the Appendix another method will be described of obtaining the spark, even in cold, damp rooms.

of the eudiometer from which the spark is given off, and also that those on the exterior should have knobs, or if this is inconvenient, that the hooks should not be thin. Where a Leyden jar is used, these precautions are not so essential, as there is then much less difficulty in detonating the mixture.

552. It is moreover essential that the quantity of gas to be exploded should not be too large, as otherwise it will be difficult or impossible to prevent loss. It is not easy to give a general rule for the quantity of gaseous mixture that may be detonated with safety, because such great differences exist in the degrees with which expansion takes place on the passage of the spark; the operator must therefore be guided to a great extent by his judgment; but it is frequently proper not to explode more gas than is equal to a tenth of the internal measure of the instrument, although sometimes a third may be exploded without danger.

553. When the gas has been introduced, the exterior of the tube is to be well dried by friction with a warm cloth, as damp much

Fig. 263.




increases the difficulty of obtaining a sufficiently powerful spark.

The charge may be passed through the eudiometer in several

ways; that represented in fig. 263 is frequently convenient. One ball of the tube is attached to a copper wire in contact with the exterior of the jar, while the other ball is connected with another wire terminating in a brass ball, which is approached to within a $\frac{1}{4}$ or $\frac{1}{2}$ an inch of the conductor of an electrical machine, the knob of the conductor being in contact with the ball which communicates with the interior of the jar. The ball, *a*, is supported by a piece of glass tube at the proper distance from the conductor. When the preparations are complete, the electrical machine is worked, and as soon as the tension of the electricity becomes sufficiently powerful to overcome the resistance of the air between the ball, *a*, and the conductor, a bright spark will pass and cause inflammation of the gaseous mixture. The eudiometer figured above is of the common kind, but where it is feared that the expansion consequent on the explosion might project a portion of the contents of the vessel, the contrivance of M. Gay-Lussac, fig. 264, may be adopted. It will be seen that it is provided with a valve which descends as the mixture expands on passage of the spark, and thus closes the aperture; but on the contraction which takes place immediately afterwards, it ascends, and thus allows the mercury to enter and fill the vacuum thus produced.

Fig. 264.

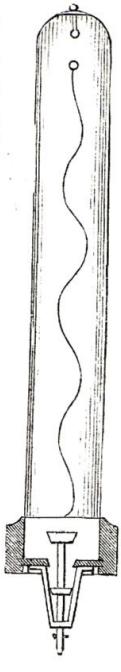
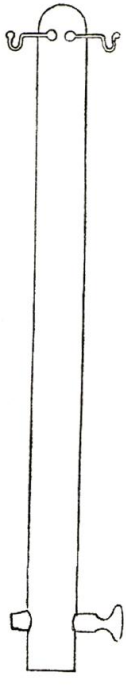


Fig. 265.



554. Mitscherlich uses a eudiometer similar to fig. 265. It is closed by the glass stopper, *a*, until after the explosion, when it is turned to allow entrance of the mercury.

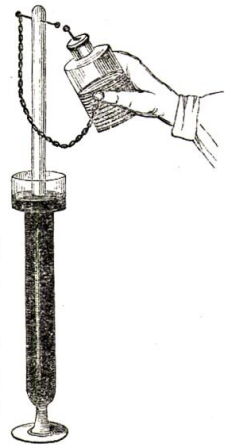
A much better way of preventing escape of gas at the moment

of explosion, is by pressing the lower end of the eudiometer against a piece of vulcanized caoutchouc, the latter being placed at the bottom of the mercurial trough. The caoutchouc should be moistened with solution of corrosive sublimate to remove the film of air which adheres to it, and may give rise to errors.

If the wires of the eudiometer are too thin to allow good sparks to pass, balls attached to wires may be fastened by twisting those on the exterior of the eudiometer.

555. The connexion between the interior and exterior of the jar should be always by wires in eudiometrical experiments, and not by a chain, as is frequently represented; in the latter case, there is sometimes a difficulty in obtaining perfect contact between all the links, especially when the jar is held by the hand while exploding. If inconvenience is occasioned by the rigidity of the wire, it may be divided into two or three pieces, and hooked together so as to form links, but their being so few in number prevents any danger of non-contact if they are clean and properly attached by small hooks.

Fig. 266.



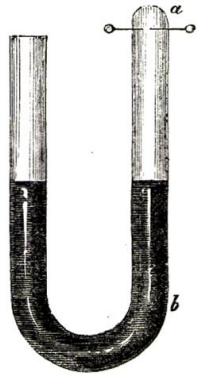
556. It is seldom that any difficulty is found in selecting a copper wire of sufficient pliability; if, however, this is the case, the wire may be bent into a spiral in one or two places so as to allow freedom of motion, as is sometimes adopted with the wires from the battery in electro-depositing. In the arrangement, fig. 266, the charge passes directly from the one coating of the jar to the other through the eudiometer, and does not affect the operator.

Dr. Ure's eudiometer was intended to render gas-analysis easy of performance by one person, but, from what has been previously said, it will be seen that with a little management almost all operations of eudiometry may be performed without the necess ty of an assistant. Nevertheless, it has other qualities which would render its omission improper; not the least of these is the com-

paratively small amount of mercury with which it may be worked. One important feature is the manner in which the elasticity of air is made to serve as a spring, and by this means to moderate the violence of the explosion.

557. The instrument, fig. 267, may be filled with mercury by inclining it a little on one side, and, the open end being immersed in the trough, the delivery-tube from the gasometer or apparatus evolving the necessary gas is to be placed beneath the aperture, or it is more generally convenient to introduce the gas by means of a small-lipped transfer-tube; by this means a measured quantity may be readily obtained in the instrument. The leg, *a b*, is graduated, but before estimating the volume of gas, it is necessary to carefully level the mercury in both limbs by a pipette, or otherwise. Before passing the spark, it must be observed that about 2 inches of the open leg are free from mercury, so that when held as in fig. 268, the portion included between the thumb and the surface of the metal may serve as the spring before alluded to. The end of the thumb must touch one of the balls, while with the other the spark is taken from the conductor or electrophorus-plate. On allowing the spark to pass, a slight sensation is perceived as if the finger was pressed outwards, and directly after, as soon as the diminution of volume has occurred, the reverse sensation is perceived, the thumb being drawn in by the pressure of the atmosphere; it must now be removed slightly to one side, so as to permit ingress of air, not too rapidly, and the metal will ascend in the closed limb; mercury is now to be poured into the other limb until it occupies exactly the same height in both legs, when the volume may be read off and the amount of contraction observed. When, from the reasons before detailed, a spark from

Fig. 267.



the prime conductor of a machine, or that of a good electrophorus, is insufficient to cause the inflammation of the gas, the arrangement must be modified so as to prevent the unpleasant effects of the shock.

558. The method seen in fig. 268 is then to be employed. It will now be seen that the thumb does not touch the knob nearest to it, but that the latter is in connexion with the outer coating of

Fig. 268.

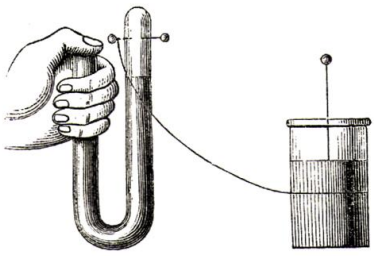
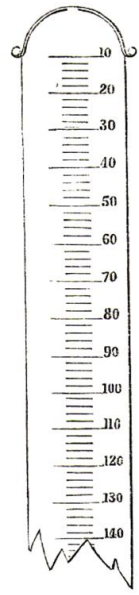


Fig. 269.




the jar. On bringing the other wire of the eudiometer in communication with the interior coating of the jar, by means of the knob of the latter the spark passes and inflames the mixture.

*Bunsen's Eudiometer.*—This is constructed of the form shown in fig. 269; it is from 60 to 70 centimetres long, and 19 millimetres in internal diameter, and $1\frac{1}{2}$ in thickness of glass. The wires, which are bent upwards and along the top, are 3 millimetres apart at their extremities. The graduations of the tube are arbi-

trary, the value of them being determined by experiment after the instrument is finished.

559. *Reading off volumes.*—In ascertaining the volume which a gas occupies in a jar over mercury or water, it is essential that the vessel should be perfectly upright. The plain surface of the mercury is to be considered as the line indicating the volume. The eye must be brought accurately to a level with this surface before reading off, for if it be either above or below, the error of observation becomes so great as to render the result of no value. A very simple but accurate method of estimating the height of a surface of mercury or water in a jar is by placing a mirror behind the jar while reading off, the eye being lowered until one-half of the pupil is seen by the mirror to cut the level of the fluid; the line on the eudiometer or gas-jar which crosses the reflexion of the centre of the pupil is to be taken as the true level. In reading off volumes over water, it is best to take the centre of the dark ring of fluid, formed by the attraction of the sides of the tube, as the true level.

The error of the meniscus of a tube may be determined thus:—A quantity of mercury is put into it, and the level of the convex surface read off; the convexity is then destroyed by the addition of a few drops of solution of corrosive sublimate and the height is again read off: the difference between the two readings is the error sought. When the mercury or water stands above the general level in the trough, the height must be carefully measured with a pair of compasses and the necessary correction made; of course in the case of mercury this correction has far greater influence upon the result than where water is the fluid used to confine the gas. In the former case, the height of the metal in the jar is to be deducted from that of the barometer in making the correction for pressure. Where differences of level are to be estimated with great precision, it is necessary to make use of a cathetometer.

560. But there is another source of error which must not be lost sight of, when an accurate result is to be obtained. When tubes are graduated by pouring equal quantities of mercury into

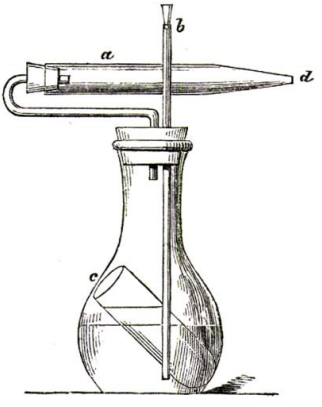
them, their position is exactly the reverse of that in which the volume of a gas is observed; it is necessary, therefore, when the volume of the gas has been ascertained by reading off over the surface of the metal by means of a mirror (§ 559), to add to the number so obtained double the error of meniscus of the tube previously ascertained.

It is a good plan to mark on each gas-jar its own error of meniscus with a writing diamond, in some part where it does not interfere with the graduations.

561. *Estimation of carbonic acid in carbonates by weight.*—Several ingenious pieces of apparatus have been invented for this purpose; most of them are extremely easy to construct, and, when used carefully, afford accurate results. For the estimation of the amount of available alkali in the pearl and soda ashes of commerce they answer well, but certainly do not surpass, if they even equal in accuracy, the process by means of a test acid, either by weighing or the volumetrical method.

562. The most simple arrangement is, perhaps, that of Parnell, seen in fig. 270, and, in careful hands, it yields equally accurate results with the more complex arrangement of Fresenius and Will; and has, moreover, the advantage of being less weighty, and therefore better adapted for delicate balances. It consists of a glass flask, fig. 270, of about two ounces capacity, fitted with a sound cork through which two tubes pass, one serving to connect the chloride-of-calcium-tube, *a*, while the other, *b*, will be described presently. A small test-tube, *c*, is so placed in the flask, and is of such a size, that it cannot fall down, but its contents may be made to flow out by inclining the apparatus to one side. To perform the experiment, a weighed

Fig. 270.



quantity of the carbonate is placed in the flask, and water added up to the level seen in the figure; the test-tube is then filled nearly to the top with concentrated sulphuric acid, and is carefully lowered into the flask; the cork with the tubes attached is then affixed, the aperture, *b*, being closed with a small cork. The little cylinders which are procured by perforating corks with Mohr's borers, are well adapted for this purpose. The whole apparatus is now carefully weighed; the flask is then to be inclined so as to allow some of the acid to flow out, and, when the effervescence has subsided, a little more, and so on until no more carbonic acid is evolved; the flask is now to be so inclined as to cause the whole of the acid to mingle with the aqueous fluid, and thus cause a considerable rise of temperature; this expels the carbonic acid from the liquid; but as an atmosphere of the latter gas fills the flask, it must be removed and replaced by air, as the difference in density of the two is very considerable. For this purpose, the cork, *b*, is removed and air is sucked out at *d*, until it no longer tastes of carbonic acid; the flask is then allowed to become perfectly cold, and the little cork being replaced, it is then reweighed: the difference in the two weighings is the amount of carbonic acid in the specimen. On drawing air for some time through the apparatus, it begins slowly to acquire weight, arising from the moisture in the atmosphere being absorbed by the chloride of calcium; and although the error introduced by this means is too minute to affect ordinary experiments, it must not be neglected where, from the quantity of material in the flask being limited, or other causes, a small difference has an important bearing on the result. In this latter case another chloride-of-calcium-tube is to be attached to the aperture, *b*, and the air must be drawn through by means of a suction-tube applied at *d*, as will be mentioned in describing the apparatus of Fresenius and Will.

563. The latter consists of two flasks, *a* and *b*, fig. 271: *a* has a capacity of two, and *b* two and a half fluid ounces of water; each is closed with a cork, through which pass two tubes; one of these, *c*, is a siphon, the short leg just passing through the cork in *b*, and the longer reaching nearly to the bottom of *a*. Another

tube, *d*, reaches to the bottom of *b*, and a third, *e*, enters *a*. A small cork stopper is fitted to *d* at the commencement of the analysis. This cork is obtained in the same manner as that used to close *b* in the Parnell's apparatus previously described, and is preferable to the piece of wax generally used for this purpose.

Fig. 271.

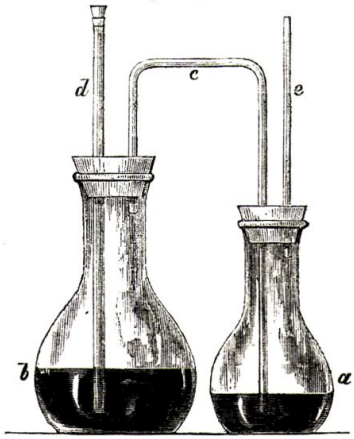



Fig. 272.


Fig. 273.

564. The carbonate to be examined is weighed out and projected into *b*, the capsule in which it has been ignited being rinsed with distilled water into the same, the rinsings, &c. filling the flask to about one-third its bulk. Enough strong sulphuric acid is then poured into *a* (without soiling the neck) to cover the exit of the tube, *c*, to about $\frac{3}{4}$ths of an inch. The aperture of *d* is then closed with its cork, and after weighing the apparatus, the suction-tube, fig. 272, is applied to *e*, and a little air is drawn out, which has the effect of creating a partial vacuum in *b*, the air escaping in bubbles through the acid in *a*. On removing the lips, the sulphuric acid recedes by the tube, *c*, into *b*, and a rapid effervescence instantly takes place, the carbonic acid escaping through *e*; but it becomes perfectly dried by its passage through the sulphuric acid; the mixture of the acid with the water in *b*

causes a rise of temperature, and when this falls, and the air contracts, more acid enters; and this is continued (assisted, if required, by suction, as before) until the carbonate is completely decomposed; when this is accomplished, more acid is forced to enter *b*, so as to cause sufficient rise of temperature to expel all the carbonic acid; the cork is now removed from *d*, and the chloride-of-calcium-tube, fig. 273, is attached by means of the perforated cork; the suction-tube being applied to *e*, air is drawn through until no more taste of carbonic acid is perceived; the two last-mentioned tubes are then removed, the cork is replaced in the aperture of *d*, and as soon as the flasks have become perfectly cold, they are wiped with a clean dry cloth, and weighed; the difference between the first and second weighings gives the amount of carbonic acid in the specimen. For the precautions necessary where hyposulphites, &c. are present, the student is referred to works on quantitative analysis. As showing the accuracy of the method, the two examples following may be quoted. They were made some years ago, to ascertain the purity of a sample of carbonate of potash, to be used for making a test-acid, and were not originally intended for publication.

66·11 carbonate of potash gave 21·01 carbonic acid.
54·26 ,, 17·31 ,,

or, per cent.,

| I. | II. | Mean. | Theory. |
|---|---|---|---|
| 31·78 | 31·90 | 31·84 | 31·88 |

565. A multitide of contrivances have been described for effecting the same result as that described above; it is, however, quite unnecessary to detail them, as none possess greater accuracy, and few are more easy to use.