A handbook of chemical manipulation

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Section XVIII. Distillation

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SECTION XVIII.

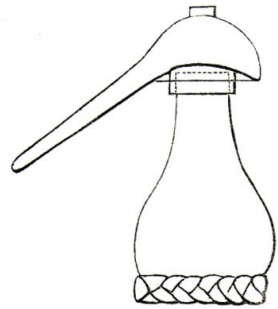
DISTILLATION.

327. It has been said that distillation differs from evaporation, inasmuch as in the former case it is the fluid which is required, while in the latter it is not regarded, but, on the contrary, every facility is given for its escape.

Distillation depends upon the fact, that all liquids when heated to a certain degree, called their boiling-point, become converted into vapour, which, by the application of cooling agencies, may again be condensed into the fluid state. It will readily be seen that, as liquids differ so greatly in the temperatures necessary for their vaporization, and also that, as vapours require such very different degrees of cold (if the expression may be allowed) for condensation, the methods of distillation must be exceedingly numerous to suit all cases; it would therefore be almost a hopeless task to enumerate every contrivance which has been adopted to meet the ever-varying cases which constantly occur in research. But as the subject is one of such great importance and every-day application, especial care will be taken to point out the more salient features in the manipulation.

328. One of the most ancient instruments for the purpose is the alembic, which may be considered the type of all distillatory apparatus, and although seldom used in modern chemistry, it must be confessed that it was admirably adapted to the processes in vogue among the alchemists, by whom it was chiefly used: it consisted of two portions, capable of being separated; the lower, known as the body, cucurbit, bolt head, belly, chamber, and by several other fanciful expressions,

Fig. 168.



was a gourd-shaped vessel (whence one of its names, cucurbit), well fitted for being cleansed, or to have solids or liquids introduced. The head, or capital, was also singularly perfect in shape for the intended uses; it had a species of channel all round to carry any liquid which condensed in it to the beak, by which it was conveyed to the receiver. Modifications of the alembic are still used, and will be pointed out in the proper place.

329. The simplest apparatus used by chemists for distillation, consists of the retort and receiver, fig. 90, where *a* is the retort having its neck inserted into a receiver, *b*. The retort rests on a triangle, *d*, supported on a small clay furnace, *c*. The receiver is supported by a tripod, *e*, of iron wire. Retorts seldom crack if heated over a charcoal fire in this manner, in consequence of the complete diffusion of the heat over the whole body of the vessel.

Retorts require care in their selection, as they are sometimes so imperfectly formed as to be scarcely adapted for the purpose intended. They should be selected light and free from specks and flaws, which are liable to cause them to crack on the application of heat. They should also be well made about the part where the neck is bent, the curve being as round as possible, and not like a large wrinkle, as is the case with those which are carelessly formed. With tubulated retorts, the tubulature should be directly over the body, so as to permit the entrance of a thermometer; no retort should be used in which the tubulature is formed so far forward, that when in its position a liquid or solid cannot be added without soiling the neck, as is the case with many that are sold. The form usually made in England is more globular and less deep than those imported from the Continent. I am inclined to prefer the latter form for general use. It is a convenient plan to draw out the necks of retorts, which may readily be done in the blowpipe-flame. By this contrivance, the neck may be introduced into receivers without the use of an adapter, an important point in many distillations.

It is constantly necessary during distillation to add liquid or

solid matters without stopping the process, hence one reason for the necessity of having the tubulature directly over the body of the retort. This addition requires considerable care, for several reasons; in the first place, the addition of solid matter to a boiling fluid, by forming a number of points for vapour to be evolved at, frequently causes the fluid to froth over and contaminate the liquid in the receiver, so that the process is retarded, as the fluid has to be returned to the retort, and the distillation repeated. The powder or other solid body must therefore be added very gradually. If the powder be heavy, it falls to the bottom and forms a cake, which, by preventing the proper distribution of the heat, and causing an undue rise of temperature in one spot, is almost sure to cause the fracture of the vessel; if possible, therefore, the distillation should be arrested and the powder introduced by small portions, and by a gentle motion of the retort in the hands is to be thoroughly disseminated throughout the liquid until dissolved; or if comparatively insoluble, a wire should be passed through the cork which closes the tubulature, and being bent into a handle on the outside, be kept in motion during the distillation, so as to prevent the accumulation of the powder at the bottom; and the heat is to be applied with greater care than usual, and less directly upon the bottom.

If, on the other hand, the matter to be added is in the liquid state, it is to be considered whether the addition will cause any chemical action, and if so, it is to be added with sufficient care to prevent the rising of the materials to such an extent as to cause the contents of the retort to boil over into the receiver. It is necessary that care should be taken in the addition of cold liquids to a glass vessel containing a fluid at a high temperature, as it is very liable to cause fracture. If introduced by a funnel, it must not be allowed to penetrate very far into the retort so as to cool the bottom part too much, but rather be added by small degrees, the funnel penetrating only a short distance below the surface; by this means sufficient heat is immediately communicated to the fluid added to prevent fracture of the vessel. Substances in hard lumps must not be added at all during the progress of the distil-

lation, but must be introduced while the retort is held, with the bottom only slightly depressed, so that the pieces added may slide gently down the neck, or through the tubulature, into the body of the retort without any concussion.

330. In distillations of liquids the habitudes of which are not thoroughly known, it is well to have the retort capable of holding twice the quantity of materials to be introduced, in order to prevent the probability of an overflow. In some processes, the contents of the distillatory apparatus are so prone to froth, that vessels of even three or four times the usual size (compared with the bulk of the contents) are insufficient to retain the fluid during the boiling. In the preparation of formic acid from starch or sugar, for instance, the contents of the still sometimes swell to thirty times their original volume, and much annoyance is experienced until the peculiarities of the operation are become familiar to the operator.

Next to the selection of proper retorts for the process about to be undertaken, the receiver best adapted for the purpose claims attention. In the great majority of cases, a common globular flask is all that is required; it is shown in fig. 169. Fig. 170 is a

Fig. 169.

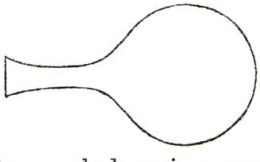


Fig. 170.

a long-necked receiver, very convenient in many instances, and from the distance the vapour has to travel before it can escape into the air, acts well as a condenser. They are also useful where Gay-Lussac's method of ascertaining the value of peroxide of manganese is used. Receivers, the same as figs. 169 and 170, with the addition of a tubulature, are also convenient in many distillations. Figs. 171 and 172 are more complex in shape, but are nevertheless necessary, not only in the preparation of many organic substances, such as those formed by the action of chlorine

or other gases upon alcohol or hydrocarbons, but also in a great number of other processes; and that shown in fig. 171 is often used in ordinary distillations, from the effective manner in which the globe acts as a condenser, and also as a valve or safety-tube; for if the end, *a*, is immersed in the liquid in a flask, and contraction takes place in the retort, the fluid rises in the long tube, and may, perhaps, partly fill the globe; but as soon as it has all passed in, air-bubbles rise up through the column of fluid and restore the balance of pressure. Where in a distillation a gas as well as a fluid is produced, the usual method of proceeding must be somewhat modified; the arrangement to be selected of course depending upon the nature of the gas and fluid formed.

Fig. 171.

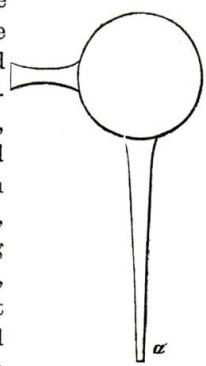
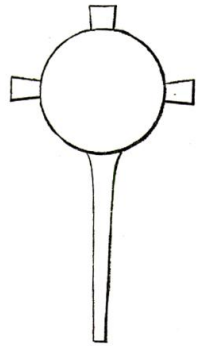


Fig. 172.



331. It is frequently observed, that, from various reasons depending upon the nature of the distillation, sudden contraction takes place, and if the neck of the retort dips into the distilled liquid, it will infallibly rush back, and the experiment will in all probability be destroyed. Accidents of this kind may be prevented by the use of the safety-tube, several modifications of which are shown in the section on Manipulation connected with researches on Gases.

332. *Substitutes for retorts.*—In many processes of distillation, retorts are by no means the most convenient kind of vessel in which to perform the operation, and it is possible to contrive a great many substitutes. Several that are not shown in the pages immediately following, will be seen by reference to the figures representing various processes involving distillation in the course of the work. The most obvious and commonly used substitute

for a retort and receiver, is constructed from two flasks connected by a tube fitted with corks.

Small distillations may sometimes be advantageously effected by means of test-tubes connected in the same manner. The tube delivering the vapour passes through a cork in the recipient, the aperture in which holds the tube tolerably tight, but allows the air to escape as it expands by the heat. The tube acting as the condenser may be kept immersed in cold water to facilitate condensation of the vapour.

333. Tube-retorts may be made of a great variety of forms, and are extremely valuable in experiments of research, from the facilities which they afford for working upon quantities of liquid so small that they would be lost in retorts or other distilling apparatus of the usual size; they are therefore much employed in investigations on the products of decomposition of organic bodies, particularly such as are only procurable with difficulty or at great expense. In experiments of demonstration they are equally useful, where the results are only to be observed by one or two persons; and combinations and decompositions may be observed in them as readily, and sometimes more so, than in working on much larger quantities.

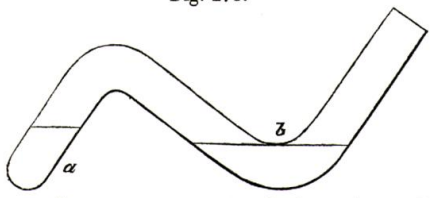
The student will do well to practise the construction of these vessels, which will be found extremely easy after a little experience. Faraday, whose dexterity as an operator in tube chemistry was proverbial before he devoted himself to electrical and magnetic investigations, was perhaps one of the first to publish the methods of using vessels constructed of tube for most of the operations in chemical research, and since that time their use has become greatly extended. And when the student is reminded that the magnificent and highly difficult investigation of cacodyle, by Bunsen, was almost exclusively worked out in tube apparatus, he will be able to form some idea of their value.

In the section on Glass-blowing, descriptions will be found of the various methods of bending, blowing, and cutting glass tubes, and of constructing retorts used in tube chemistry; also a few

general directions as to the best methods with which the author is acquainted of forming various articles of glass apparatus.

334. Figs. 173 to 177 represent a few of the most commonly used tube-retorts, but several others will be seen on referring to

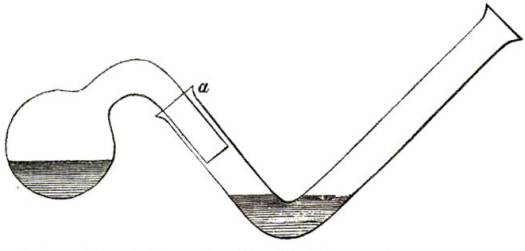
Fig. 173.



the engravings of apparatus scattered throughout the pages of this volume.

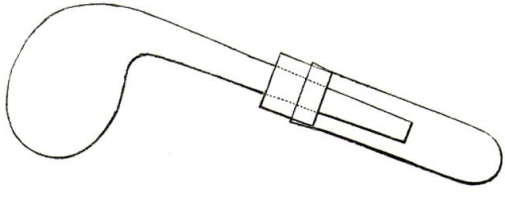
Fig. 173 shows a form of retort easily made, and sometimes

Fig. 174.



very useful. The fluid to be distilled is made to occupy the part *a*, and gentle heat is applied just sufficient to raise the vapour,

Fig. 175.



the retort being held vertically. The part *b* is kept cool by a piece of moistened rag, or any other convenient method, and this

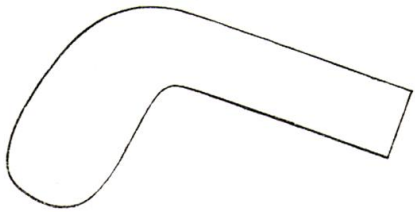
is continued until the portion of the substance introduced which contaminates the sides is washed down; the retort may then be placed as in the figure, and the distillation be proceeded with, the angle, *b*, still being kept cool; a retort and receiver are thus formed in one piece, and as the distillate may easily be removed by a pipette, the instrument is seen to be managed with ease, the only disadvantage being the loss of time in washing out the liquid to be distilled from *b*. Clarke's retort and receiver, fig. 174, are in some slight degree like the last; the chief differences are in the shape of the retort, and in the arrangement consisting of two pieces instead of one; the form of the receiver is the same, and it also resembles the first in being open. It is generally, however, made much larger, and intended to hold about two fluid ounces. The receiver must of course be kept cool; a short test-tube full of water may be inserted at the open end, to assist, by the coldness of the fluid, in effecting condensation. The neck of the retort fits with sufficient stiffness, at *a*, to render it merely necessary to support the receiver by the vertical clamp (§ 238) or otherwise. If the neck of the retort fits too loosely, it may be tightened by rolling a piece of paper round it.

335. The small retort and receiver, fig. 175, are easily constructed out of the fragments of tubing which are constantly accumulating in the laboratory: it is a useful arrangement where the quantity of fluid under examination is extremely small. The liquid may be introduced by immersing the point in the fluid to be inserted, and alternately heating and cooling until the object is effected. It is preferable, however, to make the neck sufficiently large to admit a small funnel drawn out of a piece of tubing. A small test-tube makes an excellent receiver, the neck of the retort being held steady by passing through a cork, which, however, does not fit so tightly as to prevent the escape of the expanded air on the application of heat.

Fig. 176 shows another way of constructing these little vessels, which is more especially useful where a sublimate is formed which would choke up the small orifice of that last described, or

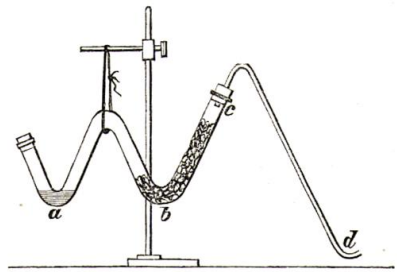
if it is desired to clean it out for a repetition of the experiment, or other purposes.

Fig. 176.



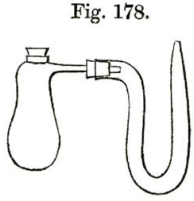
336. Fig. 177 shows an arrangement of the retort and receiver of a somewhat different kind to those previously described; it is used where it is necessary to pass the vapour of a substance over some solid matter to effect a change in the vapour previous to collecting it. There are many problems of this kind constantly occurring in the laboratory; if, for example, it is wished to dry a gas formed on applying heat to some substance in *a*, it may be conveniently effected in many instances by passing its vapour through the desiccating agent contained in fragments at *b c*; or oil of vitriol may in some cases be placed at *b*, in just sufficient quantity to close the bend of the tube, the end being corked; the gaseous products are conveyed away by the tube, *d*. Hydrobromic acid is sometimes prepared in this apparatus by having bromine at *a*, the vapour of which is passed through pieces of phosphorus at *b*, and fragments of glass wetted with water, from *b* to *c*; the acid gas is evolved by the tube, *d*, and may either be collected in the mercurial trough, or be absorbed by water.

Fig. 177.



337. The common alembic has been alluded to at p. 202; it was, however, omitted to be stated there, that it is well adapted for operations where a sublimate is formed simultaneously with a fluid, as the latter drains off through the channel previously described, and leaves the solid matter comparatively pure, at least as regards contamination with the liquid products of distillation. Small alembics blown at the lamp are sometimes employed; they are made in two pieces, the head being fitted to the body by grinding, and the former having a tubulature to allow of more fluid being introduced during the distillation. They are, however, too fragile and expensive for common use. In alembics, the chief condensation is in the head, its globular shape well fitting it for that purpose. It is perhaps strange that alembics are so little used, as, if made with the beak at a much less acute angle than as generally seen, so that they may be attached to a condenser, they are exceedingly convenient for many operations, from the facility with which they may be cleaned. This is particularly the case with those made of stoneware described further on.

Fig. 178.



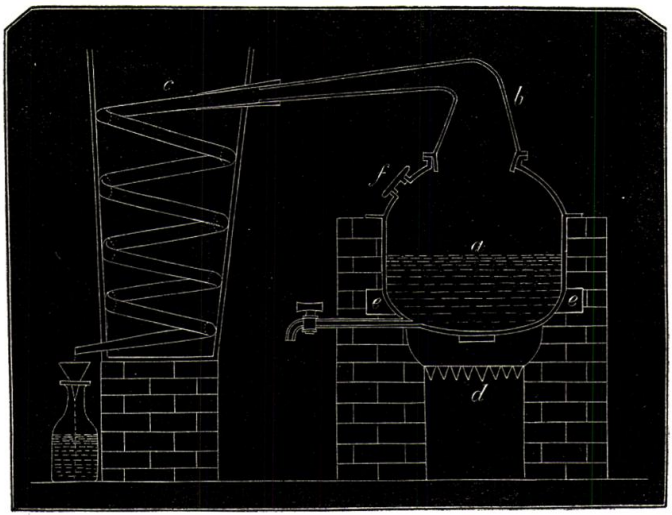
An extremely convenient mode of effecting distillations on the small scale, is by the use of a U-tube for a receiver, having one arm bent and bordered for the reception of a cork, into which the neck of the retort is inserted, fig. 178. The other end of the receiver has a small opening, to allow of the expansion of the vapour. A beaker containing a freezing mixture may be used, if necessary, to cool the condenser; in most cases cold water is sufficient, if changed when it becomes warm to the touch.

338. The ordinary still and worm to be found in almost all laboratories, is constantly in use for a great number of operations. It is frequently required for preparing distilled water; and if the head be removed, the boiler, if of a capacity of from twelve to twenty gallons or more, is very useful for preparing decoctions of plants, previous to an examination of their constituents, evaporation of large quantities of liquids, preparation of various

substances, &c. Many varieties of stills are now in use, and considerable ingenuity has been displayed in adapting them to the purposes for which they are required; it is seldom, however, that in a laboratory of research more than two stills are required, and these are generally of the simpler description, the more complex ones being almost exclusively used in technical operations, and a description of them does not, therefore, come within the scope of this work.

339. The still shown in fig. 179 consists of a stout copper boiler, *a*, inserted in brick-work, and furnished with a tap to run

Fig. 179.



off the liquid when necessary; *f* is the man-hole, which serves to introduce fresh quantities of fluid without removing the head. It is also sometimes used to insert a thermometer, when it is desired to ascertain the temperature, as in preparing formic acid from starch, and many other operations. The head is seen to be provided with a flange, which rests on the rim surrounding the top of the boiler; the tube leading from the head fits into the

enlarged end of the worm-pipe, and the points of junction here and where the head fits in to the boiler, are made good with a luting of almond- and linseed-meal. The worm-tub is kept well supplied with cold water during the distillation by a pipe leading to the bottom, the heated water by its diminished density floating on the top and flowing away by another pipe; neither of these are shown in the engraving. The flue is so constructed that it winds once or twice round the pot, as at *e e*, and finally escapes into the chimney; by this contrivance a great saving of fuel is effected. It is not advisable, as a general rule, to fill the boiler higher than is shown in the figure, especially where there is a tendency to froth. The fire-bars are seen at *d*; the supports on which they rest are not shown; they are the same as in fig. 4, p. 12.

340. It is proper where very volatile liquids, such as ether, are distilling, to place a piece of moist bladder over the exit-pipe of the worm and the funnel into which the liquid drops, to prevent evaporation.

Where it is wished to distil as rapidly as possible, the head should be kept hot by covering it with a cloth, which effectually prevents condensation there, and causes almost the whole of the vapour formed to pass at once into the worm.

341. The operation of distillation is in general one of great ease and simplicity, and few precautions are required; it is, however, necessary to avoid urging the fire too fiercely, as, if the steam is formed with too great rapidity, the head is liable to be blown off, there being considerable resistance offered to its escape by the worm. The distillate should never be perceptibly warm as it issues from the condenser; when such is the case, the fire must be slackened and the supply of cold water increased.

342. If spirituous fluids are being distilled, to increase the quantity of the more volatile ingredient in the distillate, the fire should be kept low, so that the fluid may issue very slowly; it is sometimes better that the liquid in the still should not boil in these operations, in order that the amount of water coming over with the spirit may be as little as possible. If a weak spirit

is to be concentrated, as in distilling off the alcohol or ether from the residues constantly accumulating in the laboratory, it is better to have a water-bath made to fit inside the still, the head fitting tolerably tight. The weak spirit is to be placed in the bath, the body of the still being filled to the usual height with water; the head is then luted on, and the water in the still made to boil; the fluid in the bath never reaches quite to 212°, and the ether or alcohol comes over readily; when this stops, the contents of the bath will be found to contain very little spirit and may be thrown away, except where some valuable substance is contained in it.

343. When a substance has been dissolved in alcohol or ether for the purpose of obtaining it in crystals, and the first crop has been separated, it is generally necessary to concentrate the fluid to obtain a second crop; the value of alcohol and ether renders it usual to perform this by distillation, in order that the spirit may be recovered and used again for similar purposes; as this is usually an operation performed upon very small quantities, it is best to distil from a flask placed upon a small water-bath, and attached by a glass tube to one of the condensers to be described further on.

Fluids containing mixtures of spirit and water are liable to boil with concussion, or, as it is usually termed, "bumping;" this may be obviated in several ways, most of which have for their object the production of surfaces containing points, in order to permit with ease the evolution of the vapour; pieces of wire, preferably platinum, fragments of glass or quartz, &c., may be used for this purpose: even fragments of cork or wood thrown into the retort to some extent assist the evolution of the steam.

It must be observed, however, that if the liquid in the retort commenced bumping before the platinum wire, &c. was introduced, it must be allowed to cool 20 or 30 degrees before attempting to retrieve the oversight, as, if fragments of metal, wood, cork, glass, &c. be thrown into a retort at or even a little below the boiling-point of the contents, it frequently happens that so large a quantity of vapour is suddenly formed that the fluid boils over and may do considerable injury, especially in the case of such inflammable liquids as alcohol and ether.

344. It is necessary, as a general rule, to note the quantity of fluid introduced into a still, because from the opacity of the vessel it is impossible to see when the contents are getting low, but if it is known how much is contained in it, and the quantity coming over is observed, no fear need be entertained of proceeding so far as to injure the bottom of the vessel.

345. In the distillation of vegetable matters with water, it is a good practice to place them upon a perforated false bottom raised a few inches above that of the still, in order to ensure them from burning. Where herbs are to be distilled to procure essential oil, it is proper to add some salt to the water, in order to raise its boiling-point a few degrees, as by this means the oil is procured with more facility, and, according to some distillers, in larger quantity.

346. One disadvantage of the ordinary worm-condenser is the difficulty with which it is cleaned, and one product is liable therefore to be contaminated with that which has been previously distilled. In all cases where a fluid has passed over which is not readily removed by running cold water through the worm, the latter should be closed by a cork at its lower end, and filled up with some solvent capable of removing the obnoxious matter.

The solvent to be used of course varies with the different matters to be removed; alcohol, wood-spirit, benzole, dilute solution of caustic potash, or acetic acid, are the substances most commonly resorted to for this purpose; where wood-spirit or benzole has been used, it is necessary afterwards to empty the worm-tube and blow steam through it until the odour is removed, before proceeding to distil. In fact, this operation alone will frequently remove impurities from the worm without the use of any solvent.

347. The difficulty of cleaning the worm has led to the invention of several modifications of it, the greater number of which are only employed in technical operations. One of them, however, may be mentioned here; it is extremely simple in principle, but requires considerable care in its manufacture to answer the purpose perfectly, as, if the workmanship is not good, it will be found difficult to prevent leakage. The ends of the worm (which is merely a zig-zag) project from the tub at each bend, and are connected together

by pieces of tube which slip on tightly, and are made good, if necessary, by a little luting. It is evident that if the joints are taken off, it is easy to pass a bottle-brush or other convenient cleanser down the tubes, so as to remove any dirt with facility.

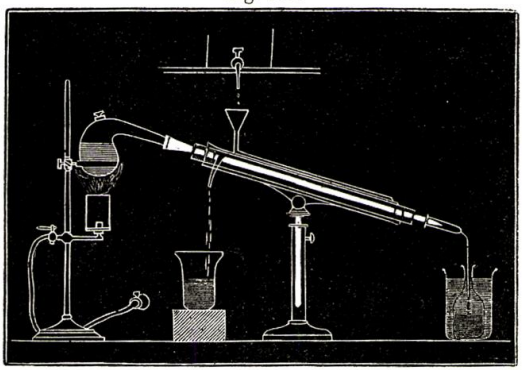
348. Retorts require somewhat more care in their use than stills, from the fragility of the material of which they are constructed; if used properly, however, it is seldom that they break during a distillation. The remarks upon the proper degree of stoutness in material given with regard to flasks, apply with equal force to retorts; they need not therefore be repeated. In general it is essential to apply the heat exactly under the centre of the bottom of glass vessels, as, if the sides become heated many degrees above the liquid, the comparatively cold fluid, if thrown upon it during the ebullition, is liable to cause fracture. There are, on the other hand, instances in which it is necessary to place the lamp a little on one side of the retort, so that the full heat may not play directly upon the bottom: some liquids which boil with violent concussions, although a considerable quantity of platinum may be in the retort, may be brought over quite quietly by this means. When a liquid is introduced into a retort and heat is first applied, it is usual to find water condense on the outside of the glass; it is advisable to remove this with a cloth. Care must be taken so to regulate the heat that the fluid may never be distilled too rapidly for the means of condensation, as, in the case of volatile liquids, considerable loss may be occasioned. If the fluid being distilled has a very high boiling-point, it is better to cover the dome of the retort with a hood of some kind to prevent condensation there, because if this be neglected the operation is much and unnecessarily prolonged, and with liquids decomposable by protracted ebullition, loss is incurred; where, however, the object is to separate a more from a less volatile fluid, this procedure is injurious to the success of the experiment, as the less volatile portion is retained by the condensing power of the dome, while the more volatile one passes over.

349. Where the substance being distilled is very volatile, or contains a difficultly condensable ingredient, and efficient means

of cooling are not to be had, the receiver may be attached to the retort by a well-fitting cork, through which, or the tubulature of the receiver, a small tube passes, and dips a short distance under mercury, or, in some cases, a fluid capable of absorbing the volatile product; the mercury allows any permanently elastic matter to escape, and so prevents disruption of the joints, while any fluid condensable with the aid of a slight pressure is retained. Where this precaution is adopted, it is necessary to ensure against the possibility of the fluid whose pressure is made use of, rushing back into the receiver. This may be easily effected by the use of one of the safety-tubes described further on.

It is advisable, as a general rule, to which there are, however, some exceptions, not to allow the beak of the retort or other distillatory vessel to dip under the surface of the distillate, as, in the event of the heat being suddenly lessened, contraction takes place and the liquid will in all probability rush back into the retort; this is almost sure to happen under the circumstances indicated, unless the neck of the retort is so large as to more than contain the distilled fluid, because, as soon as the fluid has all entered the neck, air-bubbles pass through it and equalize the pressure in the same manner as with the quilled receiver previously mentioned.

Fig. 180.



350. An excellent method of condensing in distillations with retorts is by the use of Liebig's condenser, fig. 180. It is seen in

other places in this work. A tube of tin is closed at each end by a cork through which a glass tube passes; the tin portion is to contain the cold water, which by means of the funnel enters at the lower end, and, after becoming heated, escapes by the upper aperture. Simplicity of form is one of the chief merits of this apparatus, enabling it to be cleaned with facility after any operation.

351. It frequently happens that the neck of the retort is too large to enter the recipient; one method of obviating this has been already given, p. 203; another is by the use of adapters, which are tubes, generally of glass, rather wide at one end and small at the other, the wider end to admit the retort-neck, the other to enter the receiver. The simpler shapes are easily made with the blowpipe and some large tubing, which, for this purpose, is preferably of flint-glass. Figs. 181 to 186 show several varieties of adapters fitted for different purposes.

Fig. 181.

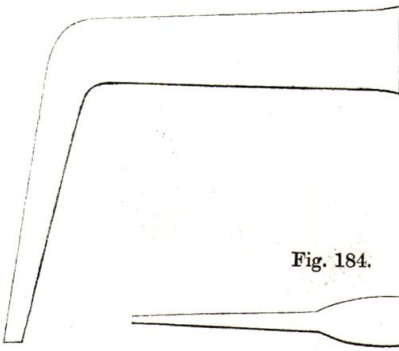


Fig. 182.

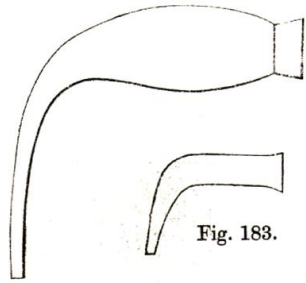


Fig. 183.

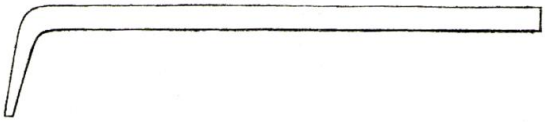
Fig. 184.

If it is merely desired to connect the exit of a Liebig's condenser with a bottle or flask intended to contain the distillate, figs. 181, 182, and 183 are the best; but, if the fluids being distilled do not require much refrigeration, and it is simply intended to connect the retort or other distilling apparatus with the receiver, figs. 184, 185, and 186 are convenient. It is

often an effectual method of condensation to arrange the apparatus

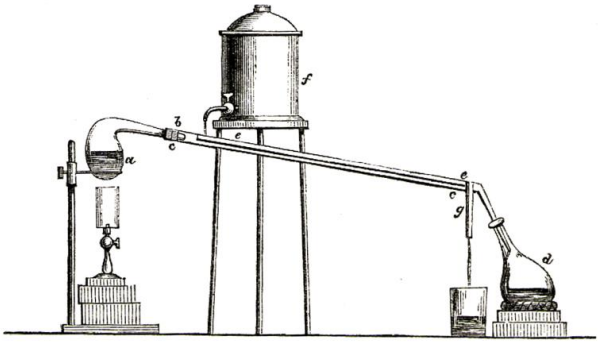
Fig. 185.

Fig. 186.



as in fig. 187, where it will be seen that a retort, *a*, is fitted at *b* with a cork into a long adapter, *c c*, similar to fig. 186; the bent

Fig. 187.



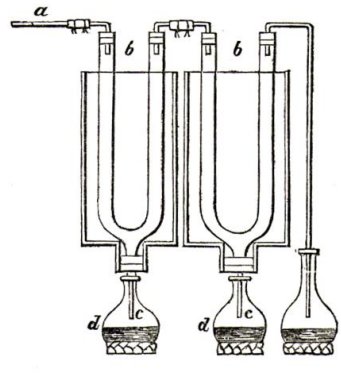
point enters a flask, *d*; at *e e*, a piece of bibulous paper is placed along the adapter and receives the water which falls from the tap of the water-bottle, *f*. At *g* another piece of paper is placed across the adapter, its ends hanging down, in order to conduct the water which has performed its office into the vessel beneath. It must be observed, that if the tube, *c c*, is placed too slanting, the paper, *g*, will be unable to prevent the water from passing *g* and entering the flask, *d*. In some cases it is impossible to prevent great obliquity of the conducting tube, *c c*, as in connecting with re-

ceivers those alembics the beaks of which have a "quick dip," as it is termed, or, in other words, make too acute an angle, with a vertical axis passing through the vessel, it is then necessary to pass a pierced but tightly-fitting cork over the conducting-tube, making it occupy the place of *g* in the above figure; the water will then be conducted by the cork into a vessel placed beneath it.

In distilling fluids which are not corrosive, it will be found exceedingly convenient to have some adapters made of tinned iron, somewhat of the shape of fig. 186. In order to prevent the condensing water passing into the recipient, a piece of tin, shaped like a heart, is pierced in its centre to permit it to be passed over the bend for a short distance. It is then soldered in its place, and all the water running along the condenser stops short at the impediment thus placed in its course, and runs down in a stream from the lower end into a pan placed to receive it.

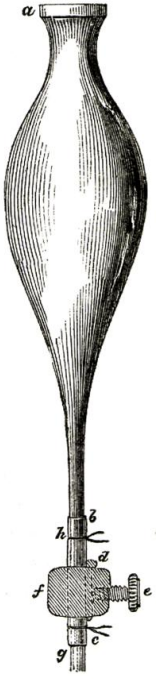
352. When the liquid to be distilled is very volatile, or if a mixture contains a very volatile ingredient, several expedients may be resorted to in order to effectually condense the whole of the vapour. Sometimes, after passing through a Liebig's condenser, it is conducted into a flask immersed in snow and salt, or some other convenient freezing mixture, or into the arrangement seen in fig. 188, where the tube, *a*, is supposed to come from a condenser, and enters a series of U-tubes, *b b*, immersed in a freezing mixture contained in the vessels perforated at the bottom, to allow the passage of the conducting tubes, *c c*, which are attached to the U-tubes, and terminate in the flasks, *d d*, which may likewise be placed in a freezing mixture.

Fig. 188.



This is, however, an expensive arrangement, and one that may generally have substituted for it a far simpler one constructed of flasks, and connected by glass tubes fitted into corks. Where no freezing mixtures are at hand, it is sometimes necessary to avail ourselves of the property of evaporating fluids to carry away a large amount of heat, which becomes latent as they are converted into vapour; for this purpose alcohol, ether, wood-spirit, and some other volatile fluids are employed; their use is, however, limited, and it is far better to avoid them if possible. Fused nitrate of ammonia, when dissolved in water, developes cold, and as it may be evaporated down and used again any number of times, it becomes a very useful auxiliary in many researches.

Fig. 189.



An extremely convenient mode of cooling in distillatory experiments on the small scale, is by means of the contrivance seen in fig. 189. It consists of an adapter, *a b*, having at its lower end, *b*, a piece of vulcanized india-rubber tube, *b c*, which passes through a square hole mortised in a block of wood, *f*. A small piece of hard wood, *d*, lies in the cavity, and, on turning the screw, *e*, is pressed against the tube, so as to collapse it to any required extent, and consequently regulate the flow of water. These compression stop-cocks are adapted to a multitude of purposes in the laboratory, and are very easily made by any person with the aid of the tools to be described further on. A piece of glass tubing, *g*, is tied at the lower end of the vulcanized-pipe, which latter is securely fastened by silk at *h* and *c*.

353. *Fractional distillation.*—It frequently happens in organic researches, that mixtures containing several substances of different boiling-points present themselves, and the only available means of separation sometimes consists in what is termed fractional

distillation. This process is not only one of great labour, but, unfortunately, it has little claim to accuracy as regards the *absolute* separation of bodies, it being, perhaps, almost impossible to procure one substance out of a mixture of homologous fluids in such a state that it can be said to contain no admixture of its associated compounds. This arises from the fact that the boiling-point of an organic fluid is dependent upon the relative number of the atoms of carbon and hydrogen present, the former element raising while the latter lowers the degree at which it is converted into vapour; it will therefore be seen, that on endeavouring to separate bodies by this means, where homologous groups are present, it is possible to obtain fluids having the theoretical boiling-point, and proportion of carbon and hydrogen in the hundred parts, and yet being admixtures of the fluids above and below in the homologous series, the deficiency in the carbon and hydrogen of those lower in the series being exactly counterbalanced by the excess in that portion present belonging to a higher position in the group. This mode of separating bodies is always, therefore, to be considered as by no means preventing the necessity for further modes of purification being adopted in all possible cases, and it is only with some neutral hydrocarbons that any great difficulty occurs in this respect.

354. Where only one substance is sought to be obtained from a mixture, and there is ample material to work upon, it is possible to obtain it in a state of *comparative* purity, although with considerable loss*, by distilling it several times in a retort with a thermometer in the tubulature, and each time rejecting all liquids boiling below or above the point known to be that theoretically belonging to the fluid under examination; by this means a large quantity of the mixture may be made in general to yield sufficient of the substance sought for an examination of its chief properties. It is far better, wherever practicable, to submit the liquid to a complete fractionation, which is performed by distilling it with a

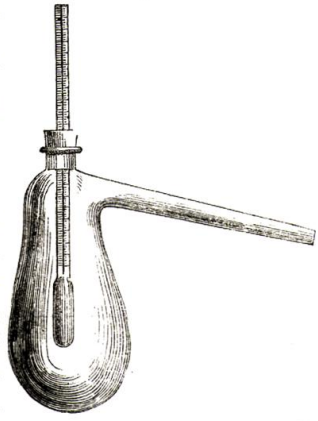
* This loss is not absolute, but as a considerable portion of the substance sought becomes by the process disseminated through a great mass of the other bodies, it may be said to be lost for all practical purposes.

thermometer in the tubulature, and changing the receiver at, say, every 10° Fahr., or 5° Centigrade; by this means fractions are obtained, which are to be again distilled into a fresh series of bottles in the same manner, which forms the second fractionation. It will be seen that where a fluid begins with a low boiling-point and rises very high towards the end, this process involves an immense amount of labour: if we suppose the fluid to commence ebullition at 200° Fahr. and rise to 400°, there will be twenty bottles required, the contents of each to be again distilled, and generally towards the beginning of the operation, each fraction, when redistilled, spreads itself over four or five bottles, requiring therefore constant attention; and when the liquids are completely fractionated fifteen times, as was the case in the investigation of the bases from Dippel's oil by Dr. Anderson, and in my own experiments on the naphtha from Boghead coal, 300 distillations become necessary as a minimum; in each of those researches, however, at least 1000 distillations were made, involving an amount of labour, patience and attention, which few who have not worked out such processes can imagine. By the method given, there are only two series of bottles required during the operation, one containing the fluids being distilled, and another receiving the products of the distillation. As each distillation reaches its close, a point is attained when it would be unsafe to proceed further, the quantity of liquid in the retort being too small; this should, in a careful operation, be very little, but as it is constantly occurring, it must not be wasted, and it is generally, perhaps, the best plan to draw it off with a siphon or pipette when the retort is nearly cold, and (as most fluids become coloured by ebullition) place it in the bottle corresponding with the second fraction above it of the series then being distilled, not the series being distilled *into*; by this means, the last distillates are always colourless, if the liquid does not come over coloured.

355. It is a disputed point whether the bulb of the thermometer should be immersed in the liquid of the retort during a fractional distillation, or in the vapour; but if we consider that the vapour represents the fluid distilling, which is always a

nearer approach to homogeneity than that in the body of the retort, which is always of a higher temperature, it will be admitted that the bulb should be as near the fluid as may be, but not in it, to indicate the boiling-point of the fluid distilling over. This does not apply to pure liquids: for then the bulb should be thoroughly covered with the boiling fluid. In fractional distillations no regard need be had to the influence of atmospheric pressure, as the fractions are all produced nearly under the same circumstances; this does not apply to ascertaining the boiling-points when the fluids are pure. For the influence of the barometric indications upon the result, see p. 55. Fractional distillations will generally be commenced with a large retort, but as the liquid becomes dispersed over a number of bottles, and consequently the quantity of fluid in each distillation is much lessened, a smaller retort must be used, and if the fluid is small in quantity and very valuable, that figured in the margin will be found extremely convenient, and as it is made from tubing at the glass-blower's lamp, it is light and not liable to crack. The thermometer is held vertically by the cork. Where it is merely intended to isolate a more from a less volatile fluid, an apparatus, the same as, or on the principle of, that employed by Wurtz to separate the butylic alcohol from the amylic and others of the same series in fusel oil, is of great assistance. The less volatile fluids are condensed in a bulb placed over the flask, while the more volatile pass over; by an arrangement of this kind much labour is saved. When liquids have been fractionated several times, portions are almost invariably obtained, boiling many degrees below the original point of ebullition, which fact has been mistaken by some for a breaking up of the liquid with production

Fig. 190.



of fluids of more simple constitution; but this is not the case, at least in the majority of instances, the lowering of the boiling-point simply arising from the successive removals of the more carburetted and consequently less volatile portions of the mixture. It is not intended to assert that breaking up never takes place, for it has been said by some chemists that certain fluid hydrocarbons, when distilled until their boiling-point has become nearly constant, and then kept for a considerable time, boil at a very different temperature than at the time of preparation, the differences in height of the barometer of course being allowed for. If, therefore, some hydrocarbons break up at normal temperatures, it is to be supposed that they would be far more liable to do so at the point required for distillation; the alleged phenomena, however, require confirmation. Mansfield, whose experience in the fractionation of fluid hydrocarbons from coal-naphtha gives weight to his opinion, denies that any breaking up takes place in those examined by him. It would be interesting, therefore, to expose fluids of this class, the boiling-point of which is constant, to a tolerably high temperature in sealed tubes, and then examine the fluid, as by this means the question might probably be decided. It is doubtless the case that when organic fluids, more especially those with somewhat high boiling-points, are distilled, decomposition takes place to a greater or less extent, as is made evident by the colour which is at last acquired by liquids which were perfectly limpid before the operation; the change, however, appears to depend upon a gradual oxidation of the hydrogen of the substance, water being formed and carbon eliminated, and differs entirely from a molecular breaking up. This is confirmed in many ways, amongst others by the fact that water becomes evident as the distillation proceeds, no matter how carefully exposure to the damp air has been avoided; and also, that when a current of dry hydrogen, carbonic acid, or nitrogen is substituted for air, the decomposition is prevented. It must be remembered when distilling in a current of gas, that if it is one less dense than air the boiling-point will be lowered, and *vice versâ*.

356. When volatile organic bases are fractionated, those with the very high boiling-points become decomposed with a formation

of pyrrol, the presence of this substance being shown by its characteristic reaction of dyeing red, slips of deal wood moistened with hydrochloric acid.

357. A method of separation of volatile organic acids of the series $C^n H^n O^4$, dependent upon an entirely different principle to the method last alluded to, has been proposed by Liebig, and has afforded excellent results in the hands of several chemists. The process consists of partial saturation and distillation, and is thus performed:—The mixture of, say butyric and valerianic acids, is divided into two portions, one of which has potash added to it until neutralized, the other is then added, and the mixture distilled; if, now, the quantity of alkali added is less than is required to neutralize the valerianic acid, nothing but valerianate of potash remains in the retort, a mixture of valerianic and butyric acids, containing much less valerianic acid than before, distilling over. If, on the other hand, the quantity of potash added is more than enough to neutralize the valerianic acid, the surplus will of course be saturated with butyric acid, and thus a mixture of valerianate and butyrate of potash remains in the retort, the former salt being in the greater quantity, while the distillate contains only butyric acid. When acetic acid is present, the reactions are somewhat different; but for details the reader is referred to the paper itself*.

358. *Special cases of distillation.*—There are many fluids which, either from peculiarities in their methods of boiling, or from their action upon vessels constructed of ordinary materials, require particular precautions in the operation of distillation. Only a few of these need be instanced as types of the general methods to be adopted.

One of the most dangerous and troublesome fluids to distil is sulphuric acid; its great causticity, and the suffocating nature of the vapours formed by its contact with carbonaceous matters at high temperatures, make the destruction of a retort containing it, while over the fire, a serious accident, and one which must be avoided by every means in our power. The manufacturers of

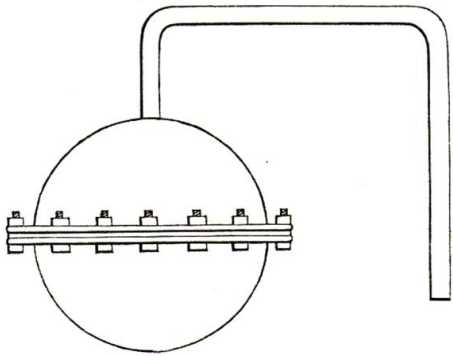
* Liebig's 'Annalen,' Sept. 1849, and Chem. Gaz. 1850, p. 24.

this important acid have been induced from this cause to use stills of platinum, which, although of such great expense in the first instance, are found much more economical in the end. Sulphuric acid possesses in an eminent degree the unpleasant property of concussive boiling previously alluded to, and it is necessary therefore to have a considerable quantity of fragments of glass or platinum in the retort, to facilitate the escape of vapour. It is, moreover, not only unnecessary but improper to apply any artificial refrigeration to the receiver, as the extremely hot acid coming into contact with the cooled vessel would almost infallibly cause its fracture; and, moreover, its boiling-point being so high, if we have a tube, 4 or 5 feet long, to connect the retort with the recipient, no fear need be entertained of the acid vapours escaping into the laboratory. It is perhaps scarcely necessary to caution the operator against allowing any organic matter to find its way into the acid to be distilled, as a decomposition takes place with formation of sulphurous acid, the presence of which may prove highly prejudicial in many operations in which the acid is likely to be used. With sulphuric acid, advantage is gained by preventing the heat from playing immediately upon the bottom of the retort; this has already been mentioned as a means of preventing concussive ebullition in other cases, and Berzelius applied the method to the distillation of sulphuric acid, by placing the bottom of the retort upon a truncated cone, which in its turn rested upon a hearth, and a charcoal fire was made round it, so as to apply the heat in such a manner that the boiling commenced from the sides instead of the bottom.

359. Hydrofluoric acid requires a special apparatus for its production, in consequence of its great tendency to combine with silica rendering it impossible to use glass vessels, as they would be destroyed in a very short time, and, what is far more serious, prevent the possibility of obtaining a pure product. Perhaps the most convenient apparatus for the production of hydrofluoric acid, where it is required to be frequently made, is that represented in fig. 191.

It consists of two hemispherical iron pots with broad flanges, which permit them to be laid mouth to mouth, and afford the space to pierce holes for the admission of the screws, the nuts of which are seen in the engraving. The pots are lined throughout with lead; in fact, two leaden vessels of the same shape as the

Fig. 191.

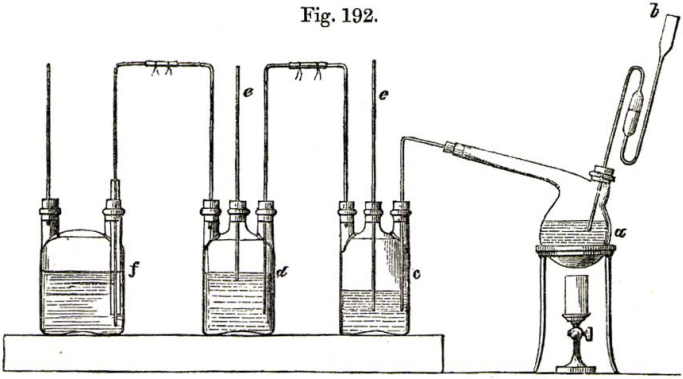


iron ones are constructed, which exactly fit inside them, and are also provided with flanges, as by means of the screws the two leaden surfaces are brought into such close contact that scarcely any leakage occurs, and what there is may be effectually stopped by a little plaster of Paris. A hole is made through both lead and iron at the top, to admit the leaden pipe, which is ground in quite tight. The chief advantages of this apparatus over those usually described, consist in the facility with which the sulphate of lime may be removed after the action, the economy with which the leaden lining can be replaced, and the protection afforded to it by the iron exterior; moreover, the iron pots are to be obtained with ease, as they merely consist of the larger sizes of sand-pots mentioned at the commencement of the work (p. 13).

360. There are several cases of distillation of frequent occurrence, in which a liquid has to be saturated with a gas evolved by the reaction of certain substances upon each other; the pre-

paration of hydrochloric acid may be taken as illustrating the general features of the operation, and showing the precautions necessary. The chloride of sodium is placed in the retort, *a*, and

Fig. 192.



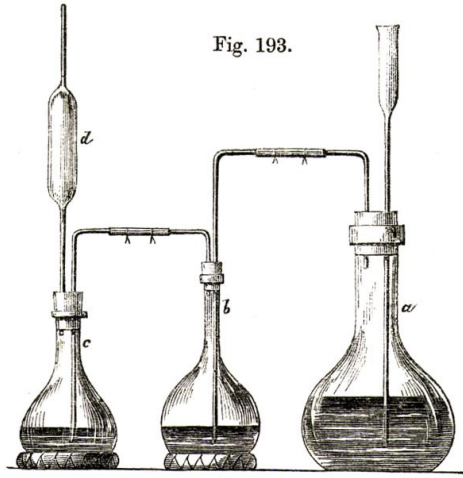
sulphuric acid is introduced very gradually by the funnel, *b*; the muriatic acid gas passes at first into the bottle, *c*, where any impurities mechanically carried over are deposited; it then flows into *d*, by a tube which dips under water; these bottles are seen to be provided with safety-tubes, *e e*, which, by admitting air, if any contraction occurs, prevent the contents from being forced back: the mode of action of these tubes, and the varieties of them in use, will be explained in the section on Gaseous Manipulation. In the same manner as the last, the gas, if any is unabsorbed by *d*, passes into *f*; at first the water in *c* and *d* absorbs all that comes over; but as it becomes saturated, it passes into the other bottle. As heat is developed during the absorption, it is proper to immerse the bottles in cold water. It is unnecessary to apply heat to the retort at the commencement of the operation, that developed by the reaction being sufficient; but as the flow of gas becomes slackened, a small lamp-flame may be placed beneath it and gradually increased. The bottle, *f*, is fitted up in a manner different from the others: the mode of action will be explained further on.

361. When mixtures are to be distilled which it is expected

might explode by a temperature of 212°, the contrivance shown at p. 45 may be used with advantage.

362. Mercury may easily be distilled in small quantities in plain glass retorts, which should not be too large; for if the dome of the retort is far above the mercury, it will be necessary to heat it to a degree that may endanger its safety, in order to drive the metal over. From the high temperature required, it is improper to use tubulated retorts, as if closed by a stopper they are liable to fracture, and corks are rapidly carbonized. The steady heat of a small charcoal fire is perhaps the most convenient for the purpose. Where larger quantities are to be distilled, an iron retort is preferable to any other, and the bottles in which the metal is imported, if fitted with a bent iron tube, answer the purpose extremely well. The addition of copper filings much facilitates the process, where it is desired to obtain as pure a product as possible. The exit-tube in either case must be plunged under water.

Fig. 193.



363. It has already been mentioned, at p. 225, that fluids distil at a lower temperature in a current of hydrogen than when

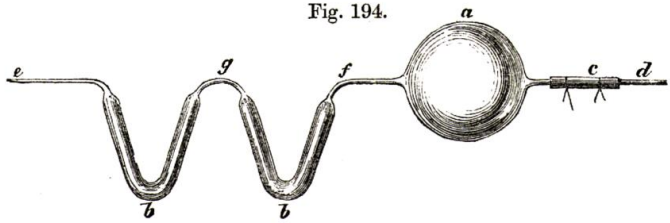
the operation is conducted in the usual manner. This method of operating has to be resorted to in several cases; in the first place, it is not unfrequently necessary to expel a more from a less volatile fluid in the purification of the latter, as, for instance, where it is desired to drive off ammonia from a fluid base having a comparatively high boiling-point; to effect this, the apparatus shown in fig. 193 may be used, where *a* represents a bottle fitted up with tubes for the preparation of hydrogen gas, which is dried by passing through sulphuric acid contained in *b*, from whence it streams through the basic liquid in *c*. Sometimes it is required to heat the latter, in which case the volatilized base is condensed in *d* and falls back into the flask, the dry hydrogen carrying away the ammonia and water present in the fluid. The operation is generally a somewhat protracted one. It is often required to distil fluids of no great stability in an atmosphere of some gas having little or no tendency to impart oxygen. The ebullition of fluids is often by this means considerably facilitated. Phosphite of ethyl, which boils at 191° Cent. (375°·8 Fahr.) in air, comes over at 188° C. (370°·4 Fahr.) in a current of hydrogen gas*. Where it is merely wished to prevent oxidation, or to facilitate the ebullition of fluids of no great stability, but not spontaneously inflammable, a current of dry hydrogen evolved from an apparatus similar to that shown in the last cut, passes into a retort the neck of which is drawn out and bent, so as to enter a U-tube, which, if necessary, may be immersed in a freezing mixture and have a conducting tube passing into a flask (in the manner of those in fig. 188), likewise kept cold. Sometimes the current of hydrogen carries away so large a quantity of the fluid that its loss would be serious; it is then necessary to connect the first with another, or even two or three more U-tubes or other condensers, according to circumstances.

364. If the fluid is spontaneously inflammable, as is the case with many organic compounds containing antimony, arsenic and other metals, it is necessary to have the joints made by fusion, and the apparatus is then preferably constructed from glass tubes.

* Railton, Chem. Soc. Quart. Journ. Oct. 1856.

Fig. 194 gives an idea of one of the simpler kinds of apparatus for the purpose. The substance is formed by the action of heat

Fig. 194.



upon the ingredients in *a*, and is condensed in the V-tubes, *b b*, the second of which is drawn out to a point at *e*. Before the reaction is commenced, a current of dry hydrogen is passed through the whole by means of the tube, *d*, connected with a gasometer or other source of the gas; *d* is connected in its turn with *a*, by a caoutchouc tube, *c*, containing a piece of glass rod, almost large enough to fill it. When all the air in the apparatus has been expelled by the hydrogen, the tube, *c*, is tightly tied, and *d* is removed. The bulb, *a*, is then heated until the substance has distilled over into the V-tubes, *b b*, which are kept very cold during the operation. When no more fluid is condensed in the receivers, a powerful blowpipe-flame is directed upon the parts, *f* and *g*, in succession, which are thus closed and removed until wanted for examination; finally, the point, *e*, is closed by fusion. From what has been said, it will easily be seen that the above is far from being a universal method, the method of manipulating requiring various modifications according to the nature of the substance operated on.

365. When it is necessary to observe the temperature at which a fluid distils in a current of hydrogen or other gas, it is sometimes proper to use a flask provided with three apertures: one for the tube delivering the gas, another for the exit of the distilled liquid, and a third, generally in the centre, for the thermometer. By this arrangement, however, a considerable surface of cork is exposed to the action of the heated vapour, and with fluids the boiling-point of which is above 300° Fahr., it soon

becomes brittle, and even rotten, so that it is difficult to keep it from leaking; but if the surface of the cork is comparatively small, this does not occur, or at least with the same rapidity. There are small three-necked Woulfe's bottles, blown from tube at the lamp, to be procured from the dealers in chemical apparatus, which, from their thinness, are well adapted for this purpose.

366. In some distillations it is advisable to allow the ingredients to digest for some time before applying the fire, particularly if fluids are to be dehydrated by contact of quicklime, chloride of calcium, or other substances used to remove water. The less active the matter intended for the purpose, the longer the digestion is required to be. In most instances, the object may be effectually attained by putting the ingredients together over night, and distilling in the morning.

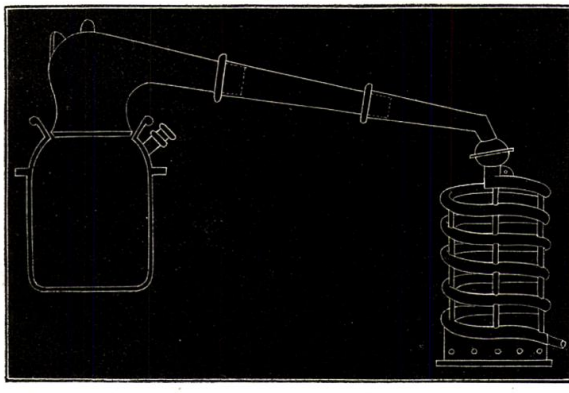
367. In making formic acid by the more common processes, the starch or sugar, oxide of manganese and water are introduced into the still, and a fire being put under it until the fluid has obtained a temperature of about 100° Fahr., or a little higher, the sulphuric acid is added by degrees, the fire being previously removed; an immense quantity of carbonic acid gas is evolved, and sometimes the action is so violent, that, although the fire has been taken out, the mixture froths over; in any case, it is better to allow the ingredients to digest for five or six hours before distillation, when, by careful regulation of the heat, the dilute acid may be brought over without any further danger of frothing. The formiate of lime produced in a subsequent stage of the process is best distilled with sulphuric acid, to obtain the strong acid, in the stone alembic, p. 239.

The formic acid, as thus obtained, invariably contains a certain amount of sulphurous acid, which may be rendered sufficiently fixed to allow of distilling over the formic acid in a state of comparative purity, by adding just enough of a solution of chromate of potash to convert the sulphurous into sulphuric acid. If this is done with moderate care, no fear need be entertained of lessening the product by decomposition of the formic acid.

The salt alluded to is moreover useful as a test of the presence of sulphurous in formic acid, as a very small portion converts the yellow colour of the chromate into a beautiful green, even in the cold, by reduction to the state of green oxide.

368. For a great many purposes, but more especially in the production of chemical reagents on a somewhat large scale, as is so frequently required in certain investigations, a stone still and worm are much employed, and will be found exceedingly useful. It is scarcely safe to use a sand-bath for them, although some withstand that method of applying heat very well. It is better therefore to use a chloride-of-calcium bath, the heat given by which is sufficient for almost all the purposes to which a stoneware still is likely to be applied. For the preparation of prussic and valerianic acids on the large scale, they are indispensable. The

Fig. 195.



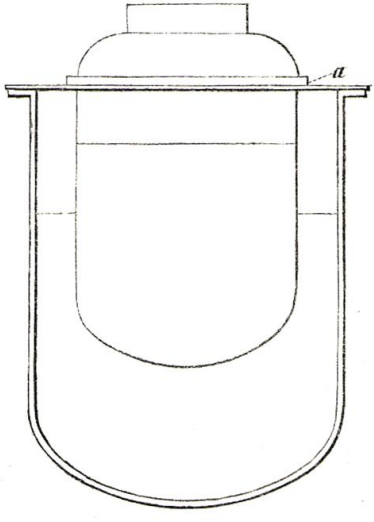
worm should be inserted into a wooden tub, the spout projecting at the bottom. This is the most easily fractured part of the apparatus, and requires especial care. It is a good plan, as a means of protection, to have two stout pieces of wood nailed to the tub, and standing out to the same extent as the exit of the worm, but not placed so close as to prevent easy access.

369. When these stills are in operation, it is necessary to cover the head with a cloth, to prevent condensation and con-

sequent loss of time and fuel. The juncture of the head with the body is closed by means of almond and linseed luting. It is absolutely essential that great care be taken to prevent the chloride-of-calcium bath becoming dry, or even too low, as in that case the addition of cold water would infallibly break the still; in fact, when water has to be added to keep up the height of the fluid, it should always be nearly if not quite boiling.

370. The best method of supporting the still in the bath is seen in fig. 196. A hole is punched out in a circular piece of strong sheet-iron, just large enough to permit the easy entrance of the still as far as the flange, *a*, which supports it in the bath. The iron pot is at least 6 inches larger all round than the still. A hole about 2 inches across is punched in the iron ring, to allow of the addition of hot water to replenish the bath. As the solution of chloride of calcium is liable to froth up and boil over at times, it is better not to allow it to fill the pot to a greater height than that indicated by the line in the figure.

Fig. 196.



371. The stone worms are so cheap, that it is better, where they are required frequently for any particular purpose, to keep them for that exclusively, especially if from its nature there is any difficulty in cleaning them out, their opacity throwing great difficulty in the way of ascertaining their freedom from dirt or impurities.

372. *Destructive distillation.*—The products of the destructive distillation of coal, and various animal and vegetable substances,

have furnished chemists with many of their most interesting products. The number of these already obtained would, to recount their history, require a volume; but a few will be mentioned, in order to show that a description of the apparatus required, and the methods of using them, are deserving of a place in this work.

373. Wood, by destructive distillation, yields, in addition to acetic acid, methylic alcohol, acetone, xylite, lignone, paraffine, kreosote, the beautiful pittacal, and the host of substances studied by Reichenbach, several others which are as yet imperfectly known. Small as the per-centage of nitrogen in ordinary wood is, the author found methylamine to accompany the ammonia formed by destructive distillation of the impure acetate of lime made on the large scale, from the acetic acid produced simultaneously with wood-spirit.

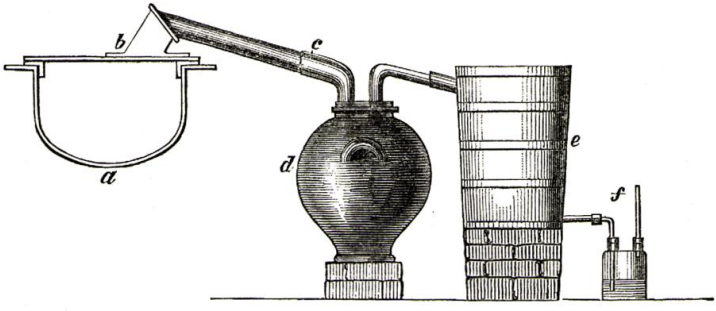
Coal, by destructive distillation, yields the whole series of fluid hydrocarbons homologous with the starting-point, benzole, many gaseous ones, being still far from well known, and numerous organic bases, viz. aniline, the pyridine and chinoline series, and the somewhat mysterious body, pyrrol, known by its characteristic reaction of staining fir-wood moistened with hydrochloric acid, bright red or crimson. Indigo yields aniline and other substances. Boghead coal, and probably Burmese naphtha, yield hydrocarbons of more than one class, one appearing to be identical with the alcoholic radicals.

374. These few substances mentioned (for they are few compared with the multitude inviting study) are sufficient to indicate that a wide field for investigation remains comparatively unexplored in the products of the destructive distillation of organic matter; and also that no apology is due for devoting some pages to a consideration of the apparatus best adapted for researches on the subject.

375. Where it is intended to distil large quantities, recourse will doubtless be had to the cast-iron retorts used at gas-works; but, on the small scale, the stout iron pots to be obtained at the iron-wharves, and shown in figs. 197 and 198, are very convenient.

The pot, *a*, in the latter figure, is provided with a very broad flange, supporting an iron cover made after the manner of a saucepan-lid, and perforated with an aperture of about 6 inches

Fig. 197.



diameter, to admit the head, which is to be riveted on. The exit-pipe is made large until it enters the first recipient, *d*, with which it is connected by the adapter, *c*. The vessel, *d*, may very well consist of a six-gallon stone jar, closed with a large bung pierced with two apertures. All the tar and other semi-solid and easily condensable matters having been deposited here, the more volatile products pass into the worm, *e*, which is kept constantly supplied with a stream of cold water having lumps of ice floating in it. All the fluid hydrocarbons, and most other matters, are completely condensed by this means; but if very volatile bases are present, they require further precautions; the exit-pipe, *f*, is therefore made to dip into dilute muriatic acid contained in a second bottle, which effectually retains all the basic products. The iron pot is set in brick-work, just in the way of an ordinary still, no particular precautions being necessary, except that of being able to remove the fire with rapidity in the event of the products coming over too rapidly. It is to be remembered that the great danger is not of having too little, but too much heat; and it is a good general rule, to bring over the products at the lowest possible temperature, the fire being increased as the evolution of volatile products slackens, this being easily

seen by watching the rapidity with which the bubbles of gas pass through *f*. Where the gas is very fœtid, as is the case when most substances containing a large per-centage of nitrogen are distilled, it may be passed through a rather thick solution, prepared by stirring up chloride of lime and water to a creamy consistence, or the gas may be ignited or passed by means of a long tube under the ash-pit of the furnace. The lid of the still is luted up with a mixture of finely-sifted fire-clay and fresh horse-dung beaten to a plastic mass in an iron mortar. To apply the luting in the most successful manner, the lid being inverted, the luting is to be placed all round, and the lid being then put in its place, is to be kept down by placing heavy weights on it.

376. In some operations, a neater but more expensive apparatus for distillation is made by drilling a series of holes round the rim of a carron pot, and fastening the lid down by screws and nuts, as seen in section, fig. 198. The exit-pipe is made of wrought iron, and is inserted into the lid by having a screw tapped on its end to fit a hollow screw made in the plate which covers the pot. This arrangement is excellently adapted for the preparation of sulphurous acid from bruised charcoal and sulphuric acid.

Fig. 198.

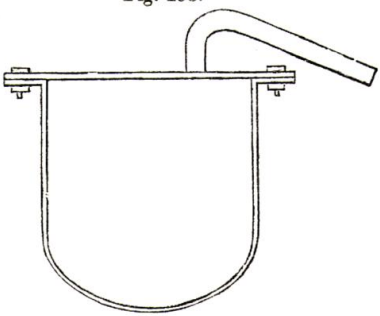
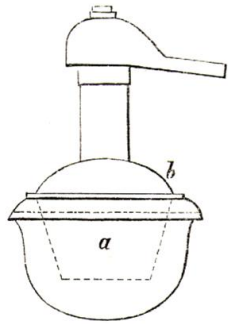


Fig. 199.



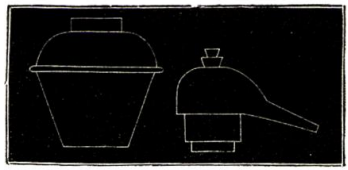
377. Where destructive distillation is to be performed on a much smaller scale, as in preparing aniline from indigo, chinoline from cinchonine or quinine, trimethylamine from narco-

tine, &c., the arrangement shown in fig. 199 may be used. It is constructed of stout sheet-iron, and has the head, belonging to a stoneware alembic similar to fig. 200, luted to it with the almond and linseed luting previously alluded to. By far the best way of heating these alembics is by means of a species of hot-air bath, formed of an iron pot similar to those used to melt lead in, but with the three feet removed, which may be done by filing them three parts through, and then giving a smart tap with the hammer. These pots are covered with a lid fitting somewhat tightly on; it has a hole in the top large enough to admit the alembic, which is supported by the flange which joins the body, *a*, to the dome, *b*. A small hole, about 1 inch in diameter, is left in the lid of the hot-air bath, to enable the operator to observe when it reaches a red heat. The pot is to be heated by placing it on the largest hole of the furnace, fig. 1, all the rings being removed.

The great advantage possessed by this arrangement, is the rapidity with which the temperature can be regulated as compared with a sand-bath. In the latter case, the sand takes a considerable time to acquire the requisite temperature, and when it has done so, it requires a still longer time to cool, whereas the pot can be heated to redness as rapidly or slowly as may be required, and may be cooled with equal facility.

The manner in which the alembic is surrounded prevents accession of currents of cold air, so that even when constructed of earthenware they may be raised to a red heat and cooled again a great number of times without fear of fracture. The stoneware alembic alluded to is shown in section in fig. 200. They are somewhat difficult to obtain, but their extreme convenience in many operations renders it well worth while to have them made of the potters, so many of whom are to be found in Lambeth, near London. The aperture in the top of the head is useful for

Fig. 200.



the introduction of fluids, &c. after the joints have been luted up, or during the progress of a distillation.

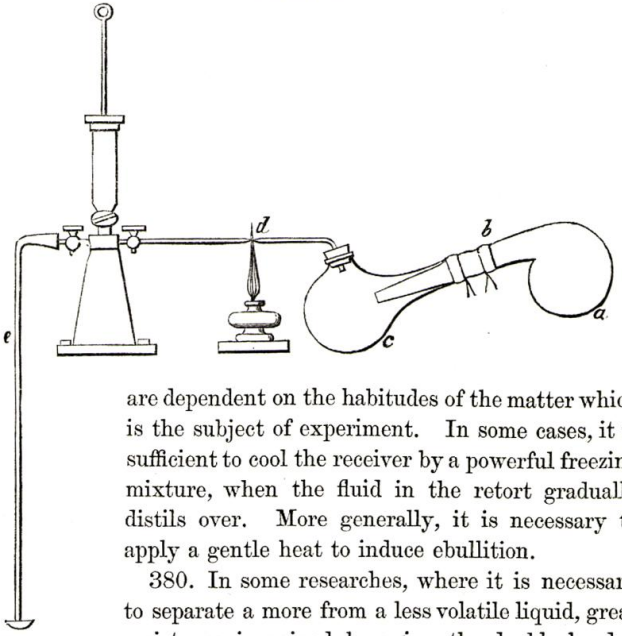
378. It has been said that, as a general rule, the lowest possible temperature should be used in destructive distillations where the object is to examine the products obtained; this rule holds good in the process for procuring aniline from indigo; the latter substance, being finely pulverized, is to be mixed with a very concentrated solution of potash, and the whole is to be evaporated with constant stirring, in an iron pot, until a faint odour of aniline is perceptible; directly this is observed, the mass is to be rapidly transferred to the iron alembic with the stone head, fig. 199, which is then to be inserted in the hot-air vessel, and the apparatus being placed on the aperture in the furnace-plate, and ample condensation being ensured, the fire is to be very gradually raised until no more aniline is procured.

It is important in the construction of these alembics to have the beak made at a much more obtuse angle with the body than is usually done (see also p. 219), as, if this is attended to, much greater ease is found in attaching them to condensers. The caprylic alcohol, which has become an object of interest lately, is readily and conveniently prepared from saponified castor-oil in an apparatus similar to fig. 199.

379. There are many occasions in organic and inorganic chemistry where it is desired to effect distillation under reduced pressure, and consequently at a lower temperature than if performed in the ordinary manner. This is easily managed by the use of the apparatus seen in fig. 201. A glass retort, *a*, has its neck inserted into a tubulated receiver, *c*, with which it is connected, air-tight, by the caoutchouc tube, *b*. The tubulature of the receiver is fitted with a cork and tube, *d*, which connect it with a small air-pump. The air is then exhausted as far as may be requisite, the degree being made evident by the rise of the mercury in the tube, *e*, which dips into a small basin of the metal; if, now, a lamp-flame be applied at *d*, the tube is soon closed by the pressure of the atmosphere, and the distillation may then be commenced.

In distillations of this kind there are several methods of proceeding, which will suggest themselves to the operator, as they

Fig. 201.



are dependent on the habitudes of the matter which is the subject of experiment. In some cases, it is sufficient to cool the receiver by a powerful freezing mixture, when the fluid in the retort gradually distils over. More generally, it is necessary to apply a gentle heat to induce ebullition.

380. In some researches, where it is necessary to separate a more from a less volatile liquid, great assistance is gained by using the double-headed stills shown in figs. 202 and 203. The method of using them consists in placing within the chamber, *b*, which surrounds the head, *c*, a fluid, the boiling point of which is higher than that of the more volatile product which it is wished to obtain, and below that of the liquid which is to remain behind in the still. If, for instance, it is desired to obtain benzole from coal-naphtha, it is sufficient to fill the chamber, *b*, with water, and proceed with the distillation in the ordinary manner; it is evident, that, as the temperature of the head can never exceed 212°, the hydrocarbons, which require a higher point for volatilization, will be condensed on arriving at that part of the head which is sur-

rounded with water, and return to the body *a*, but that the liquids which boil at or below 212° will pass the head and proceed to

Fig. 202.

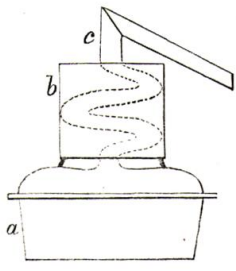
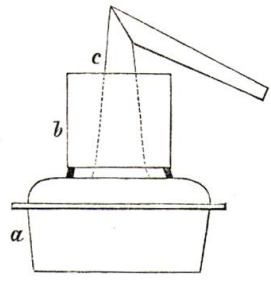


Fig. 203.



the receiver. Sometimes it is unnecessary to have a worm passing through the water, a rather considerable elongation of the head being sufficient to prevent the passage of fluids of high boiling-points, as in fig. 203.

381. If retorts or flasks are to be exposed to temperatures so high as to endanger their safety, or where fluids are to be operated on which, either from their inflammability or value, it is particularly essential to prevent being spilled if any accident should occur, we may employ vessels covered by the electrotype process with a thick coating of copper: the method of effecting this will be found in the section on Electrical Manipulations.

382. In operations on very limited quantities, where the use of ordinary retorts, or even the small ones described in the section on Fractional Distillation, would be impossible, recourse may be had to the apparatus shown in figs. 204 and 205; it consists of a very small tubulated retort made from a piece of large tubing; when the bulb has been blown on the end of it, and the neck bent as in fig. 204, the point of the blowpipe-flame is directed on the part *a*, fig. 205, and a piece of red-hot glass rod is placed against it and immediately withdrawn; it thus forms a thin tube, which may be bent as at *a b*. This construction enables us to introduce the fluid to be distilled without soiling the neck of the retort, an advantage not generally found in these instruments as constructed from

tubes; the fluid is made to enter by dipping the end, *b*, into it and applying suction to *c*, carefully avoiding the entrance of moisture: when a sufficient quantity has entered, the point, *b*, may be closed by the blowpipe. This method is especially useful in experiments where it is wished to operate upon known quantities.

Fig. 204.

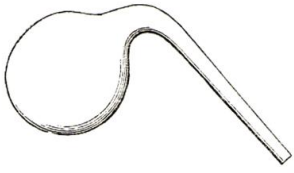
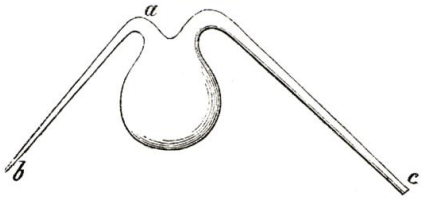
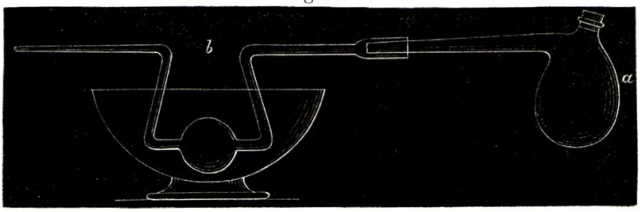


Fig. 205.



383. Where an extremely volatile fluid is one of the products of a distillation or reaction, the appliances shown in figs. 206 and 207 may be made use of with advantage. The retort, *a*, in which the materials are being heated, is connected with the bulb apparatus, *b*, immersed in a freezing mixture. The product may be transferred to the little tube apparatus, fig. 207, in which

Fig. 206.



it is to be preserved; it may be introduced by the method described in the section on Pressure-tube Operations (§ 304).

Fig. 207.

384. It not unfrequently happens that it is wished to submit a fluid to the action of a substance at a moderately high temperature (generally a little above the boiling-point of the liquid) for a considerable time, as, for example, in the preparation of some hydrocarbons by the action of sodium on iodine or bromine compounds. In this case it is convenient to use the apparatus shown in fig. 208. The flask, *a*, contains the substance to be cohobated, and, in the example given, a quantity of sodium; it is immersed in the copper tallow-bath, *c*, which is heated by the gas-lamp, *d*. The vapour which rises condenses in the long wide tube, *b*, which has a capillary opening at its upper end, and falls back into the sodium; by this means the whole of the iodine or bromine may be removed in an hour or two, and the tube, *b*, and its cork being removed, it is to be replaced by a bent tube connected with a receiver, and the pure hydrocarbon or other fluid distilled over.

Fig. 208.