A handbook of chemical manipulation

Williams, Charles Greville

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Section XII. Filtration and Washing of Precipitates

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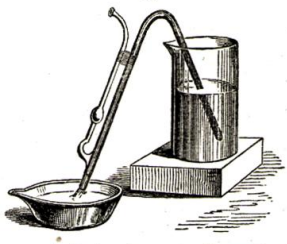
FILTRATION AND WASHING OF PRECIPITATES.

189. In analytical operations, it almost always happens that filtration immediately follows the formation of a precipitate, its object being to collect the substance together, to enable all soluble matters to be removed by washing; but sometimes it is considered preferable to wash the precipitate by the process called decantation, which consists in adding a considerable quantity of water to it in some convenient vessel (preferably a tall and narrow one), and, after thorough incorporation of the washing-fluid and the precipitate by stirring, it is allowed to settle until the whole of the solid matter has been deposited; the fluid is then poured off, and the operation repeated until the precipitate is sufficiently washed, the latter point being ascertained in all cases by testing. When the washing is completed, the precipitate is freed from water as much as possible by a siphon, or careful pouring off, and the rest is removed by a pipette, or fold of bibulous paper, after which the precipitate may generally be placed in a warm place to dry. It is convenient to transfer the wet precipitate to a capsule or evaporating basin, in which to complete the removal of the water and subsequent desiccation, as the capsule or dish may, without fear of injury, be placed upon a sand-bath to dry.

Fig. 105.



190. Where the water cannot well be poured off from a precipitate without danger of disturbing it, it is advisable to use a siphon to remove the liquid, as in fig. 105. The leg in the liquid has the end turned up, so that the precipitate is not disturbed by the current.

Methods of constructing very convenient siphons for this

and other purposes will be found in the section on Glass-working.

191. Filtration is, in fact, a species of sifting, being a process by means of which we separate more dense and coarse from finer and more subtile particles, by the use of a medium capable of letting the latter pass through its pores while the former is retained. It will easily be seen, therefore, that many substances are capable of performing a function of this kind under certain circumstances; and in practice several varieties of filters are employed, according to the numerous cases which have to be met: paper, calico, flannel, sand, glass, asbestos, tow, and many other media may be resorted to with advantage in chemical processes, where a solid is to be separated from a liquid; at present we shall only have to deal, at any length, with paper, that being almost exclusively used in experiments of research.

It is of the greatest importance to the chemist that his filter-papers should be of unexceptionable quality, yet, until within the last few years, it was a matter of extreme difficulty to obtain a paper even tolerably well adapted for the purpose; now, however, such an advance has been made in this respect, that excellent paper may be easily procured, already cut to the most convenient shape.

192. There are several properties which ought to be possessed by a good filtering-paper; in the first place, it ought to filter rapidly, and yet it should be sufficiently close in its texture to retain the finest powders; it must not become too rotten when wet, or it is liable to break by even the most careful pouring of a solution into it. It is highly desirable that it should contain no soluble salts, or several sources of error are introduced into experiments. It is, moreover, especially necessary that, if any soluble matters *are* present, they should not consist of sulphates or chlorides.

193. It is proper to caution the student against the fallacy of supposing, that because filters are of the same size they must necessarily be of the same weight. It has been recommended to collect precipitates upon a double filter, and when the washing is

completed, to dry the precipitate upon both filters, and in weighing to place the second filter upon the opposite scale of the balance as a counterpoise, the increase of weight of the one filter being supposed to indicate the weight of the precipitate; but this procedure involves more than one source of error: it is, however, merely necessary to instance one, which is, that the texture of paper is so irregular, that even when cut by an arming-press, as is generally the case with the circular filters found in commerce, it is extremely rare to find two of the same, or even of a tolerable approximation to the same weight.

194. By far the most convenient shape for a filter is that of a circle, as it then folds without any projecting parts, and exactly fits the funnels, provided they are constructed with the proper angle.

They should not, when burnt, yield more than $\frac{1}{460}$ to $\frac{1}{500}$ of their weight in ashes; and it is a good plan to burn a dozen of each of the sizes in use in the laboratory separately, and ascertain the weight of the ash; this amount, divided by 12, will give the average weight of the ash of one filter, which is therefore a constant quantity to be deducted from the weight of precipitates ignited with filters.

195. When it is desired to collect a precipitate upon a weighed filter, with the intention of ascertaining the weight of the former by the increase of the latter, it is important to ascertain whether the filtering-paper contains any soluble matters, as if so, the paper loses during washing, and thus vitiates the result unless precaution is taken to prevent it, by washing the filters intended to be used for some time with hot water; when, after being dried, they may be employed for the purpose alluded to, without much fear of error.

196. There are two methods of folding filters in use in the laboratory: the first is by far the more useful and convenient, and is performed with ease and rapidity; the second is, on the contrary, seldom used, and whenever necessary, may probably with much advantage have a calico-bag filter substituted for it.

If a square piece of paper be folded in half, diagonally, and then into quarters, it will form a triangular figure like that in

fig. 106, and if the corners are cut off in the direction of the curved line, a circle will be formed on opening out, the mode of

Fig. 106.

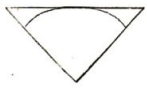


Fig. 107.

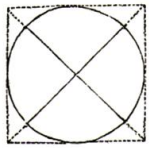
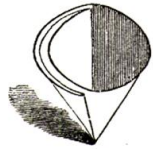


Fig. 108.



formation of which is seen by fig. 107, where the dotted lines represent the parts removed; upon opening the folded paper, so that three thicknesses come on one side and one on the other, a filter is obtained, as shown in fig. 108, which merely requires to be placed in a funnel supported by a retort-stand, or other convenient instrument, and is then, after being wetted, ready for the reception of the liquid to be filtered.

197. The ribbed filter, as it is called, was in considerable use among the older chemists and pharmaceutists, but is now very little resorted to, from its requiring some time to construct, and when made, being far from convenient, especially where the solid product of a filtration is the part required for use. It is obtained by taking a piece of paper, and after doubling it, folding it into halves, quarters and eighths, as in fig. 109, where it is seen that the creases are all on one side of the paper; the foldings are now repeated on the other side, each crease coming between the previous ones, and projecting in the reverse direction; if the paper be now opened out and then depressed in the centre, a filter resembling fig. 110 will be obtained, and after cutting off the spires, may be dropped into a funnel, and the liquid to be filtered poured in. The intention of having the ribs, is to afford passages for the air and liquid between the paper and the funnel, so as to allow of rapid filtration; the whole arrangement is seen from fig. 111, where a funnel con-

Fig. 109.

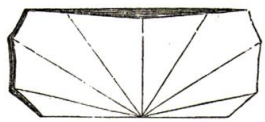


Fig. 110.

taining one of these filters is supported by a piece of wood perforated in the centre and placed over a beaker. In quantitative experiments the plain filter, single or double, is invariably used, the ribs and creases of the other rendering it almost impossible to remove a precipitate thoroughly from its surface. Where large numbers of filters are used, it is economical to purchase the paper in sheets, and cut it into circles for use in the laboratory, instead of buying the circular filters ready made. For this purpose an apparatus of great simplicity and convenience has been contrived by Mr. Stevenson, a philosophical instrument maker of Edinburgh. It consists of several discs of metal of the sizes of the filter to be cut; to the centre of each is capable of being attached a moveable rotating arm, carrying a knife which, by means of a very simple arrangement, may be adjusted to any size of filter. With a little practice, filters may be cut with great perfection by it.

Fig. 111.

198. When a filter and its contents, or a filter alone, have to be weighed after drying, it is necessary to do so in such a manner that they shall not be in contact with the air, for dry paper is so excessively hygroscopic that, under the circumstances mentioned, the filter would greatly increase in weight, even during the short time required for it to be on the pan of the balance. A very good method of weighing filters is between two watch-glasses ground at the surface and held together by a clip, fig. 61. In some cases a wide test-tube, like that represented in fig. 88, but fitted with a cork, may be used, or even a platinum crucible with a close-fitting lid is available under certain circumstances.

199. In most filtrations it is necessary to support the filtering-paper in a funnel; and the latter must invariably be of glass for all purposes connected with research. Funnels should be made to an angle of 60°, in order that the circular filters may accurately fit. It is, moreover, desirable for them to be ground on the top, to enable a glass plate to be used as a cover when required, either for the purpose of preventing the access of dust,

evaporation of spirit, or the absorption of carbonic acid in the filtration of alkaline solutions.

Filter-stands, retort-stands, perforated boards, &c., will be described in a section appropriated to Supports for Apparatus.

200. *Performance of the Operation.*—When a liquid is ready for filtration, and the funnel, supported by a retort-stand, or in some other convenient manner, has a filter placed in it, the latter is to be wetted by a little water, and the fluid and the precipitate are then carefully transferred to it, either by pouring down a rod (§ 164) or otherwise, and any particles of precipitate adhering to the vessel are to be washed into the filter by a stream from the washing-bottle, fig. 112. This instrument, the contrivance of Leopold Gmelin, has now become indispensable in all laboratories; its construction is extremely simple: a flask of any convenient size has two tubes adapted to it air-tight, and bent as shown in the figure; one of these reaches to the bottom of the flask, the other merely enters it; the vessel is filled with water, and when required to be used, the mouth is applied to the tube on the right-hand, and air being forced into the flask, the water issues in a jet from the left-hand tube, which ends in a fine orifice. It is convenient to have a small piece of vulcanized india-rubber tubing fastened to the end where the mouth is applied, as by its means much facility is gained in moving the bottle in various directions, the pressure not being required to be kept up all the time by the mouth, it being sufficient to blow into the flask; and then closing the caoutchouc tube by the teeth, a stream of water will issue for some seconds, while the bottle may be moved in every direction.

Fig. 112.

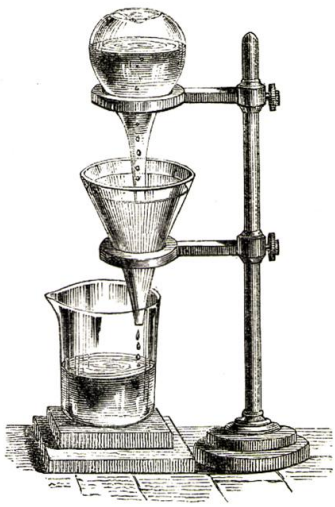
201. Before Gmelin's washing-bottle was introduced, the apparatus employed by Berzelius for effecting the same object was in general use; it consisted of a flask having a tube with a fine orifice adapted to it. On blowing into the tube and closing the aperture dexterously with the tongue while inverting it, a fine

and powerful stream of water can be directed on a precipitate in a manner admirably adapted for stirring it up thoroughly, and washing away any soluble matters.

Several washing-bottles are required in a laboratory, even if only one person is at work; one for cold and another for hot distilled water; the latter must have some arrangement for enabling it to be handled with ease when containing boiling water: a wire handle is very easy of construction. Bottles are also required for alcohol and ether; and in investigations where a number of precipitates or other substances have to be washed with any particular fluid, it is desirable to have a washing-bottle filled with it at hand.

202. It is proper in almost every case, especially if the operator has not considerable experience in analytical chemistry, to test the washing-water at certain intervals as it passes through, to ascertain when the operation is completed. Of course the test will vary in almost every case, and must be selected accordingly. The filtering-paper should never rise above the edge of the funnel; and even if, from inadvertence, such should be the case, especial care must be taken that the liquid is not permitted to rise above the level of the funnel; if, however, the operation in hand be an analysis, it is better that the paper should be thrown away and a fresh one taken.

Fig. 113.

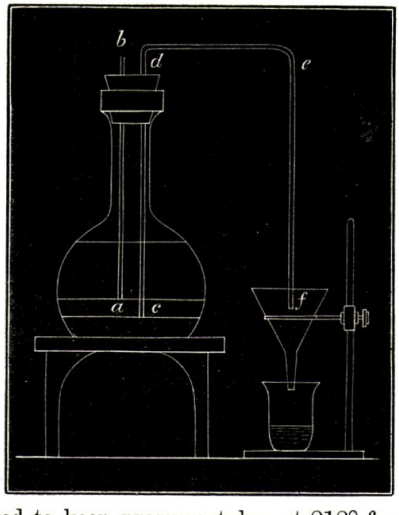


203. It so frequently happens that precipitates require prolonged washing, that many expedients have at different times been adopted to render the procedure independent of the presence of the chemist. The most simple method of effecting this is to invert a flask of distilled water over the funnel containing the precipitate to be washed, as in

fig. 113, in such a manner that the neck of the flask is below the water; in this position no water escapes from the flask until the liquid in the funnel falls below the neck; when this takes place, a bubble of air enters and an equivalent bulk of water escapes, and the washing proceeds slowly in this manner, until the whole of the liquid has passed through the precipitate.

204. Another method is by the use of a peculiar modification of the siphon, said to have been first adopted by Gay-Lussac; it is constructed as follows:—A bottle of convenient size is taken, and fitted with tubes in the manner represented by fig. 114, where it is seen that *a b* is merely a straight tube open at both ends, passing tight through a cork, which is perforated to allow the passage of the even-legged siphon, *c d e f*, the legs of which descend a little below the bottom of *a b*; the end, *f*, of the siphon is intended to be placed in the funnel, which is so arranged that when fullest the water should rise to the same height as *a*. It will be observed that the effective difference of the length of the limbs of the siphon is equal to the space between *c* and *a*; when the liquid rises to *a* the flow ceases; but directly it falls below, the funnel becomes replenished, and by this means a constant level is obtained. The arrangement is exceedingly useful where a fluid is required to be boiled for a long time, and it is desired to prevent loss by evaporation; as, for example, in the water-baths employed to keep pressure-tubes at 212° for many hours.

Fig. 114.



Nothing but its extreme fragility prevents the washing-tube of Berzelius from being much used in cases of protracted washing of precipitates. It is represented in fig. 115. It is fixed by a

Fig. 115.

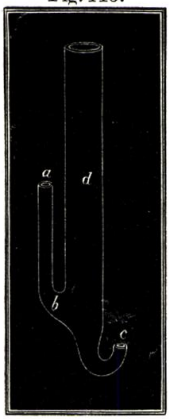
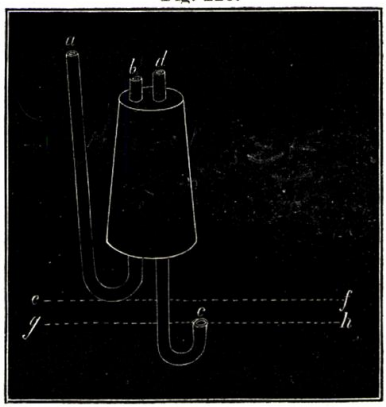
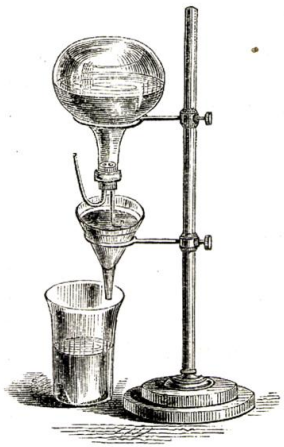


Fig. 116.



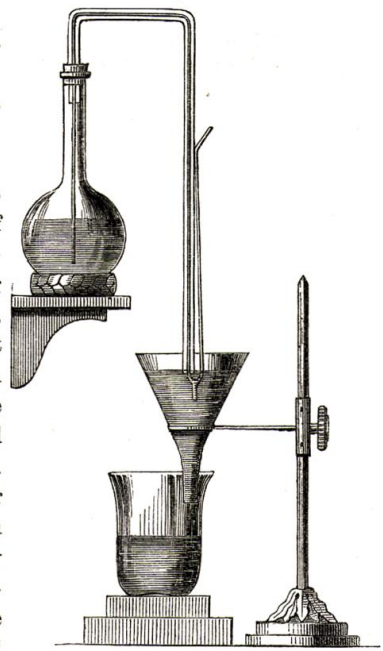
perforated cork into a flask supported as in fig. 117. The mode of action is somewhat peculiar: when inverted in a funnel, the point, *c*, is placed below the level of the liquid, and in consequence of its smallness, it exerts a somewhat retarding effect upon the egress of the water, so that when inverted in the air, only a very small quantity escapes; but when the point is immersed, the resistance is removed and air enters by *a* as the water escapes by *c*, and by this means a constant level is preserved. By arranging tubes in a cork in the manner shown in fig. 116, the same end may be attained, and the chief objection to the beautiful contrivance of Berzelius, viz. its fragility, is at once removed.

Fig. 117.



205. It should be observed that the downward tendency of the fluid in the last arrangement, equal to the column of water between *e f* and *g h*, is less than the resistance to the passage of air through the water in the tube, *a b*, which resistance is owing to its capillarity, and therefore, when the bottle is inverted, no liquid escapes after the first few drops; but this resistance is destroyed by even the small force exercised by the contact of the water in the funnel with that in the tube, *c*; and if the funnel be filled until the water just rises to the height *g h*, the water will flow out as it sinks in the funnel and maintain a constant level. The conducting power of a piece of wet paper or a finger applied to *c*, is sufficient to cause the water to flow. On constructing this arrangement, care must be taken to copy exactly the relative lengths and positions of the tubes. Another and very elegant method of obtaining continuous filtration, is that seen in fig. 118. An ordinary glass siphon, with suction-tube, has, previous to bending, another tube of larger calibre placed outside it; during the bending the two become attached, but nevertheless sufficient space remains between them for air to pass. The whole arrangement is fixed by a cork into a flask. On sucking, the water flows, air passing between the two tubes to supply the place of the water removed, but as soon as the water rises in the funnel

Fig. 118.



sufficiently high to close the opening of the wider tube, the air no longer is able to enter the flask and the flow ceases. As the water passing through the precipitate falls in the funnel below the opening of the wide tube, the flow again proceeds, and so on, until the whole of the water in the flask has been expended.

206. The following method of securing continuous filtration, is mentioned by Dr. R. D. Thomson as the contrivance of his pupil, Mr. Eustace C. Summers, and is probably convenient where extremely troublesome precipitates are being washed, or it is necessary for the operator to be absent from the laboratory for considerable intervals; under any other circumstances it would, from the space required for its use, cause more trouble than would be repaid by its superior effectiveness. Into the lower aperture of one of the stone bottles so much used as aspirators, a piece of tube is fitted by a cork passing at an angle in the same way that the opening is made in a gasometer to pass in the tube evolving gas. A glass tube, smaller than the one last mentioned, is bent into a siphon, the shorter leg of which is inserted inside the first glass tube; the longer leg dips into the funnel containing the precipitate; by this arrangement the water in the reservoir escapes by the siphon, air entering between the two tubes to supply its place; but when the liquid in the funnel rises so as to close the aperture of the siphon, the further egress of the water is prevented until the dropping of the water from the funnel has lowered the surface sufficiently to permit a further escape.

207. Where, as frequently happens, a liquid is being filtered, which, from its weight, corrosiveness, or other reasons, it is feared, will break the filter-paper, it is necessary to use a double one, which is merely one common filter placed inside the other in opposite directions, so that the triple side of one filter is placed against the single one of the other; by this means the filter becomes so much strengthened as to bear very considerable pressure without fracture, especially if the aperture of the funnel is not too large, a fault at all times to be avoided.

208. It is requisite, when making or folding filters, to carefully

insure against the presence of moisture on the hands, as in that case it is almost a matter of certainty that a hole will be found in the paper on pouring the solution into it.

Circular filters are so extremely convenient, that where they are not to be procured it is advisable to have some tin quadrants adapted to the size of the funnels, so that when a square piece of paper is folded into a filter it may be placed against the metal and the corners be removed, the piece of tin serving as a guide to the scissors.

209. When a filter has been wetted and is ready to have the solution poured into it, care must be taken that the fluid is not allowed to descend from too great a height, as not only does such a proceeding cause a risk of loss by splashing, but it endangers the safety of the filter, the mechanical force of a fall of water of a few inches being quite sufficient to break many of the less tough kinds of paper when wet.

210. It frequently happens when a precipitate is poured, together with its solution, into a filter, that the fluid runs turbid at first; it must then be returned to the funnel until it comes through perfectly brilliant, and this must never be judged of by the appearance of the liquid as it runs from the funnel, but by letting some run into a glass or test-tube, and holding it between the eye and the light.

211. Filtration is not merely applicable to the separation of solid from fluid particles, but also at times to the removal of one fluid from another; but this is only practicable where they are not miscible; if, for instance, a mixture of oil and water be poured into a thoroughly wet filter, the water will pass through to the exclusion of the oil. This mode of procedure is useful in researches on oily bases, as, where they have in the crude state been dissolved in an acid, any non-basic oil separates and rises to the surface, and if the two are thrown upon a wet filter, the acid solution of the bases passes through, leaving the other matter behind.

212. *Calico Filters.*—This description of filter is much used in manufacturing operations, and also in laboratories of research where large quantities are being worked upon. They are particularly useful where infusions or decoctions have to be filtered

from feculent matters. The best material for their construction, as far as my experience goes, is what is termed "yard-wide unbleached calico."

If large quantities of precipitates have to be filtered, or where careful collection is desirable, a very convenient and useful strainer is formed by stretching a piece of calico over a square wooden frame, fastening it on by means of a few tacks. A filter of this description affords peculiar facilities for the collection of every particle of the precipitate, whereas conical bags are somewhat troublesome to thoroughly empty without waste.

Flannel bags, from their great strength, are sometimes useful, but only in coarse operations: they are inadmissible for liquids containing free alkali.

213. Sand, glass and asbestos are adapted for concentrated acids and strong alkaline solutions; although not often used, they are sometimes extremely convenient in cases which otherwise would cause considerable difficulty. Sand and glass filters are constructed in the same manner, and therefore one description will serve for both. The throat of the funnel is first to be obstructed with rather large pieces of the substance used; on this are strewed smaller pieces, then coarse powder, and finally, fine powder. Care must be taken not to disturb the arrangement of the powders when pouring the liquid in, and it is generally necessary to pour it back two or three times before it becomes bright.

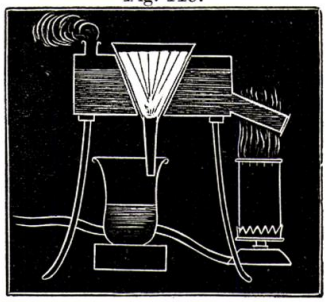
214. Asbestos filters are made by merely obstructing the throat of a funnel with clean asbestos, *lightly* packed, otherwise the liquid will pass with difficulty. In making filters with sand, glass or broken flint, it is sometimes convenient to use a little asbestos at first, instead of the larger pieces of the material. It is of importance to ascertain that the sand, &c. does not contain any substance capable of being acted on by the fluid filtering; and it is proper for this reason to wash it with hydrochloric acid, and afterwards to remove the latter by repeated edulcoration with water until the washings no longer precipitate nitrate of silver.

215. Tow is a rough medium of filtration, and is only applied

in the coarsest operations, such as the removal of mechanical impurities from oils, infusions, &c., and is seldom or never used in experiments of research, especially where any delicacy of manipulation is necessary.

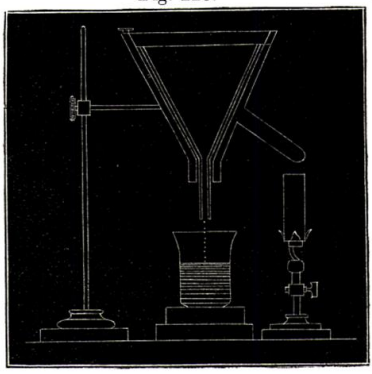
216. In the filtration of viscid fluids on the large scale, it is necessary to use pressure to force them through the pores of the medium, as, from their sluggish motion, they would, without this assistance, pass through so slowly, that in many cases the prolonged exposure to the air would cause injury, if not decomposition. The pressure is obtained in several ways; of these, two are most generally adopted, namely, atmospheric pressure obtained by the use of an air-pump, and the pressure of a column of fluid: these methods are seldom or never resorted to except in manufacturing laboratories.

Fig. 119.



217. It is a very common occurrence that a hot fluid containing a considerable quantity of matter dissolved, and which is deposited on cooling, has to be filtered; it is then required to keep the funnel, &c. hot during the whole time of filtration; this may be accomplished very conveniently by Plantamour's ebullition funnel, shown in figs. 119 and 120, the latter being the one more generally used.

Fig. 120.



It will be seen, that in the first instrument a chamber contain-

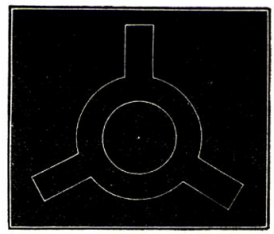
ing water has an opening which enables a funnel containing the fluid to be filtered at the high temperature to pass through, the fluid in the vessel being maintained in ebullition by means of a lamp placed beneath a projecting portion of the apparatus. The second is simply a double funnel, the interior of which contains water capable of being kept boiling by a portion projecting over a lamp-flame in the same manner as the first. These two instruments, or one of them, should find a place in every well-appointed laboratory. Where access is unattainable to an ebullition funnel, the apparatus for filtration must be placed within the hot-air closet, or, if very small, in the square water-bath, fig. 52.

218. Where volatile fluids have to be filtered, the funnel used should have its upper surface ground, and a glass plate (also ground) may be placed over it to prevent evaporation as far as possible.

Platinum salts of organic bases, no matter how finely crystallized, must, before burning to ascertain the quantity of platinum, or organic analysis, be finely pulverized; they are then generally found to be rather adhesive, and the particles to have a tendency to hang together; but if washed upon a filter with a mixture of alcohol and ether, the impurity causing this adhesion is removed, and when dry the powder of the platinum salt is found to be extremely mobile, and generally, on combustion, gives a nearer result than if the process is omitted*.

219. When the quantity of fluid to be filtered is extremely small, it is necessary to use proportionably small filters, and these are sometimes supported over a beaker or other convenient vessel by the porcelain three-legged instrument, fig. 121. In all quantitative operations the point of the funnel should touch the edge of the beaker or other vessel into which the filtrate is to

Fig. 121.



* Phil. Mag. October, 1854.

fall, in order to prevent the drops from striking the fluid with sufficient force to cause splashing.

220. It is sometimes required to filter caustic alkalies out of contact of the air of the laboratory in order to prevent absorption of carbonic acid, and this is easily managed by an arrangement either the same as, or on the principle of fig. 122, where, for every drop of liquid which falls, a corresponding portion of air is expelled from the lower and passes into the upper vessel, and so enables the filtration to proceed. The neck of the upper bottle is slightly choked by linen, asbestos, or some other convenient substance, care always being taken to select one unacted on by the fluid filtering.

Fig. 122.