A handbook of chemical manipulation

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Section XV. Crucibles and Operations at High Temperatures

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SECTION XV.

CRUCIBLES AND OPERATIONS AT HIGH TEMPERATURES.

264. Crucibles are generally used for experiments which require very high temperatures, and are made of many different materials, according to the purposes for which they are intended. Those of clay are commonly used in laboratories, and are convenient if the operations are not of great delicacy, and the material to be heated is not liable to injury from the contact of the matter of which the vessel is composed. For the fusion and reduction of metals, and the preparation of many chemical compounds, they are invaluable, and it becomes important therefore to be acquainted with the characters of the various kinds in general use, in order to enable us to make a selection of the best adapted for any operation which may present itself.

265. *Cornish crucibles* are very refractory, and if treated well, may be used for the same kind of operation several times in succession without cracking, especially if they are recharged before cooling; they are well adapted for the fusion of alloys, melting and reduction of gold and silver, fusion of chromate of lead, ignition of oxide of copper, &c.; and, moreover, they have the convenience of covers, which is not the case with Hessian crucibles, which in many respects have much resemblance in properties; the latter, however, are triangular, while the Cornish are round.

266. Ordinary English crucibles may be obtained of several shapes and sizes, according to the purpose for which they are to be used; figs. 144, 145, and 146 give an idea of their general appearance. It will be seen that they are made both triangular and round, besides the egg-shaped vessel, fig. 147, generally known as a "skittle-pot."

For ordinary fusions the triangular crucible is convenient, and affords considerable facilities for pouring the melted metal from the angles into small moulds or other positions. They all have covers. The skittle-pots are particularly convenient in

Fig. 144.

Fig. 145.

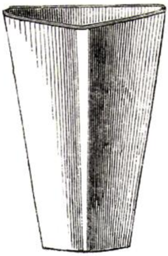


Fig. 146.

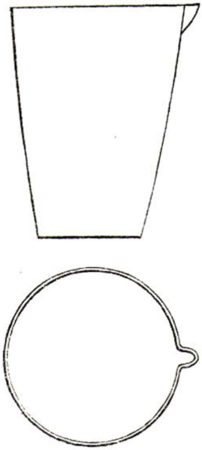


Fig. 147.

some operations, from the facility with which reduced and melted globules descend and form a button at the bottom; they are, moreover, useful for charring animal and vegetable substances without access of air, and in some metallic fusions. It must not, however, be forgotten that most kinds of English crucibles, except the Cornish and London, are not only fusible at very high temperatures, but are peculiarly liable to crack if suddenly heated or cooled, and should therefore be warmed through before being placed in the furnace; and when the operation for which they have been used is concluded, they should, immediately after the contents are poured out, be placed among the hot ashes to cool slowly. It is seldom, however, that they can be used twice, unless it is for the same operation; and when this is the case, it is usually necessary to recharge them before the temperature has much fallen, and even then they often crack in the fire on the second heating; I have on occasions, however, fused some hundred-weights of chloride of calcium with only a few large crucibles, by taking the precaution not only to refill them before the temperature had much fallen, but also to warm the charge before inserting it.

267. *Hessian crucibles.*—These are sold in nests, and in material have much resemblance to the Cornish; they are, however, triangular, and are more neatly made. They withstand a very high temperature, and if warmed before insertion in the fire, are not very liable to crack*; they have the inconvenience of not being provided with covers; this may be obviated by using an English cover, or by inverting one of the same kind a size smaller inside the larger one.

268. *Blue pots.*—These crucibles, which resemble fig. 146 in appearance, are made from a mixture of plumbago and clay, and are admirably adapted for many metallurgical and technical operations, but their composition renders their use in chemical experiments very limited; this is unfortunate, as they are not only capable of resisting very high temperatures, but are also less liable to crack than most others. They are much softer than ordinary crucibles, and may easily be sawn and drilled so as to make small chauffers and furnaces, which are extremely neat in appearance, and retain heat a long time, but are much less convenient than the small iron furnace, fig. 19, in practice, from the fact that after being used a few times they invariably crack, and then require to be bound round with wire to enable them to be used; they may be obtained of all sizes, from an ounce to 2 or 3 gallons capacity.

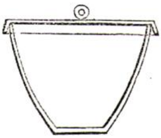
269. *Porcelain crucibles.*—These beautiful vessels are now made in Germany and France of the most exquisitely white thin and hard porcelain; they are to be procured of all shapes and sizes, and have become absolutely indispensable in the laboratory. They do not crack when heated, and are but little acted on by

* Mitchell, 'Manual of Practical Assaying,' states that "the Hessian pots are worst of all; they do not stand the least change of temperature without certain fracture, so that they require to be very carefully used." This does not accord with my experience, for I have used the same Hessian crucible for igniting oxide of copper to distinct redness (preparatory to use in organic analysis) a dozen or more times in succession without a crack occurring, and without taking any special precautions.

even the most energetic chemical reagents; they therefore communicate no impurity to the substances which are being examined in them. For some operations they supersede platinum, particularly in the ignition of precipitates of the more reducible metals, which would infallibly destroy a platinum crucible. Their beautiful whiteness frequently makes them eligible instead of test-glasses, the action of coloured reagents being very often equally well observed in them as by ordinary transmitted light. They do not retain colouring-matters, and are not porous. Their covers are also excellently adapted for many cases of testing.

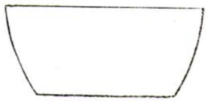
270. The crucible of the shape represented in the margin may be procured of any size, from a few drops capacity to about half a pint, and is very well adapted for the ignition of precipitates in analysis, especially where the use of a platinum vessel is inadmissible.

Fig. 148.



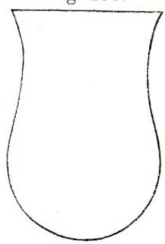
271. The capsule, fig. 149, is also adapted for ignitions and fusions, but is more appropriate to evaporations and crystallizations on the small scale, especially those in organic research.

Fig. 149.



272. The porcelain digester, as it is termed (fig. 150), would more properly have been described in the section on Solution. It is an admirably useful vessel in many experiments, but is not, I think, a very general favourite. I know of no vessel better adapted for slow evaporations and other operations on the sand-bath; and I have had some glass ones made, which are excellent for many purposes, and are particularly convenient from the facility with which they may be cleaned.

Fig. 150.



273. Small porcelain capsules of the shapes seen in figs. 151, 152 and 153, but of larger size, may be obtained of extreme thinness

and lightness, and are better for the ignition of chloride, bromide, &c. of silver than any other vessels with which I am acquainted.

Fig. 151.

Fig. 152.

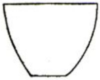
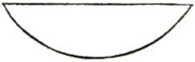


Fig. 153.

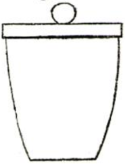


I have often had them in almost every-day use for months for the ignition of silver and platinum salts, &c., and have found them neither vary in weight nor crack. Those which are unglazed on the bottom part of the outside, may very conveniently have their weights marked upon them with a black-lead pencil, which will remain legible for some little time; and ink, although destroyed by the heat, leaves a red mark, caused by the iron in it, which may be read even after several ignitions.

It must be remembered that porcelain capsules, from their small conducting power, require a more powerful heat to raise their contents to the same temperature than vessels of platinum.

274. *Platinum crucibles.*—Perhaps no single invention has more contributed to the development of accurate analysis than the discovery of the method of constructing platinum vessels; their uses are almost infinite, and the platinum crucible has become one of the most indispensable of all the analyst's instruments. They are chiefly employed in the ignition of precipitates for analysis, and in the fusion of silicates with carbonated alkalies to render them soluble, a preliminary step to their analysis. The shapes of the vessels that may be used for these purposes are numerous, but the most ordinary form of the platinum crucible is that represented in fig. 154. The lids of some are, however, constructed in such a manner that they may, when separated from the crucible, be used as a capsule for ignitions and evaporations of various kinds, as in fig. 155.

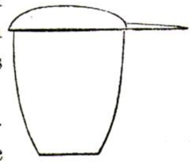
Fig. 154.



275. Spoons made of the same form as those alluded to in the section on Blowpipe Apparatus, but much larger, are also very valuable for many operations. Platinum

capsules, of the form of figs. 156 and 157, are also extremely convenient. Platinum crucibles are absolutely unacted on by carbonated alkalies at a high temperature, but this is not the case when caustic alkalies are substituted: the vessel is then liable to be seriously damaged.

Fig. 155.



276. In the ignition of platinum salts of organic bases, in order to establish their atomic weights, &c., it is advisable to employ porcelain capsules, for by

Fig. 156.

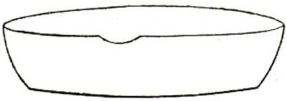
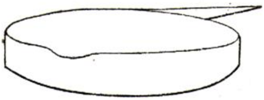


Fig. 157.



their use it is possible to obtain results of equal precision, without injury to the valuable platinum crucible; for when this operation is performed in it, the spongy platinum formed adheres with tenacity, and cannot be entirely removed. It is also of great importance to observe that no metallic oxides of easy reduction are to be heated with filters, or in any other manner that can possibly introduce free carbon, as the metal will unite with the platinum at a high temperature, and the capsule will probably be rendered useless.

277. It is convenient to have counterpoises made of lead or other metal for all the platinum vessels, so that the increase may at once give the weight of the precipitate without calculation. See also *ante*, pp. 71 and 72.

Platinum capsules, &c. may be heated in many ways, which will be seen by inspection of the articles on Lamps and Furnaces, where the methods best adapted for the purpose are detailed.

278. When used in furnaces, they must be protected by being placed within another crucible of clay, as otherwise they would be infallibly destroyed by the slag, and also by the vapours containing sulphur, which invariably exist in furnaces. It

has been said that crucibles, &c. are never to be weighed in a heated state, but it frequently happens that a substance is to be estimated which rapidly increases in weight even in a covered crucible, for their lids seldom fit very accurately after they have been much used. In these cases it is usual to place the hot crucible or a wire triangle over a vessel of sulphuric acid under a bell-jar, which mode of proceeding effectually prevents all access of moisture, and also retards the cooling; but where it is desired to proceed rapidly, any delay like this is a source of some annoyance; I have therefore been in the habit of placing the hot crucible upon a clean steel anvil which, from its size and conducting power, renders it perfectly cold in a space of time so short that few substances would absorb moisture during the operation; where, however, this is not practicable the first method must be adopted (§ 84.)

Where silicates have been fused in platinum vessels with carbonated alkalies, it is usual to extract their contents with hydrochloric acid, in order to bring them into a state fit for analysis; but it must be remembered that if the silicate contained certain peroxides, chlorine will be liberated during the solution of the fused mass in the acid, which will act upon the crucible, and not only injure it, but introduce platinum into the analysis. Under such circumstances, the crucible must be boiled with water until the contents are sufficiently softened to allow of removal, and when the whole has been removed, and transferred to a beaker, hydrochloric acid may be poured on and digested with it until the required result is obtained.

It must not be forgotten that when an acid is added to the contents of the crucible after a fusion with alkaline carbonates, a violent effervescence will take place unless it is added by small portions, and even then danger is incurred of loss of substance. During the effervescence, it is proper therefore to place a capsule over the beaker, which will prevent the escape of the particles of liquid thrown up, as they will strike upon the convex surface, and trickling down, will fall back; after the effervescence has ceased, however, the bottom of the capsule must be washed by a stream of water from the washing-bottle (§ 200), the liquid being allowed to drop into the beaker with the rest.

279. Gold crucibles are exceedingly valuable in many operations, from the admirable way in which they bear the action of alkalies; but they are comparatively fusible, and from their value, of course require proportionate care.

280. Silver crucibles are much used for fusions with caustic alkalies, as they are far less acted on by them than platinum; but it must be remembered that they are dissolved by nitric acid, even when very dilute, are very fusible, and in general are more easily destroyed than platinum or gold crucibles.

281. Iron crucibles are sometimes to be obtained, and for some purposes are extremely convenient; but their use is limited, and they do not therefore demand any special notice. The same observation applies to those made of copper; it may, however, be remarked, that if Wöhler's new process for preparing pure potash from the nitrate, is found practically convenient, it will cause them to be more extensively employed than they have been hitherto.

282. *General remarks on crucible operations.*—Crucibles at high temperatures are handled by means of tongs of various shapes and sizes, according to the weight and nature of the vessels to be lifted. When large and heavy, they are to be lifted out of the fire with strong ring-tongs. The latter are almost indispensable in these operations, because if straight ones were used they would break a piece out of the edge, and perhaps be the cause of serious accidents.

283. Small platinum and porcelain crucibles are handled by means of the tongs represented in the figs. 158 and 159.

It is very important to see that the anvil or other place upon which the platinum crucible is placed to cool, is free from dirt or fragments of readily fusible metals; as, if the former adhered to the crucible, an error would be introduced into the weighings, and if the latter, the vessel would stand a great chance of being destroyed.

284. Most persons commencing experiments at high temperatures, raise the heat too much; it is well, therefore, for the

student to make himself familiar with the nature of the various terms used to express roughly the intensities of the tempera-

Fig. 158.

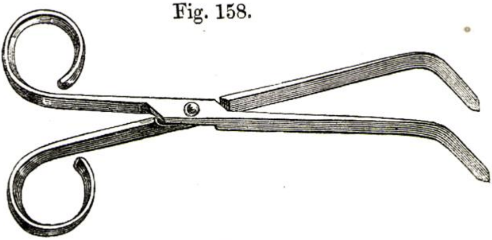
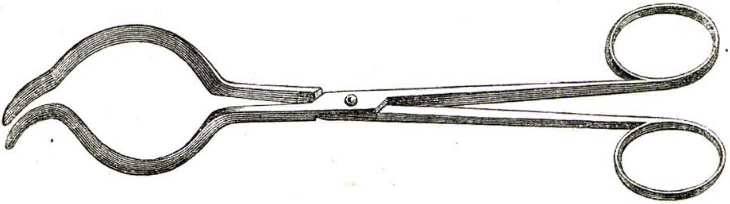


Fig. 159.



tures of furnaces. The expressions alluded to are generally known as dull red, cherry-red, yellow, bright yellow, white, and blue heat*; these distinctions are generally sufficient to enable the experimenter to apply the proper degree to his crucible in the present state of the science. Undoubtedly the time is approaching when terms so loose as these will be quite inadequate to express the gradations of temperature adapted to refined processes, but for the present they suffice, it being a very rare occurrence to resort to the indications of the pyrometer, except for the purpose of ascertaining the fusing-points of metals and other substances which require a very high temperature for fusion.

285. Charcoal crucibles were much in vogue amongst the older chemists for experiments of reduction; they fell, however, into disuse, until the employment of them was revived by M. Deville.

* This term, *blue* heat, is employed by M. Sainte-Claire Deville as indicative of the highest temperature obtainable in furnaces; it was also used long ago by Faraday.

Two ways of making them are adopted: the first and more ancient, consists in taking a piece of fine hard charcoal and modelling it to fit the inside of a clay crucible; a hole is then bored in the charcoal sufficiently large to contain the subject of experiment, and a stopper is made to fit it; this is also cut out of another piece of charcoal. When used, the substance is placed in the aperture, and the stopper inserted; the whole is then placed inside the clay crucible, and the latter has a cover luted to it, a small hole being left for escape of gas. The next method is much simpler, and is perhaps more generally useful: a quantity of charcoal is powdered to a moderate degree of fineness, and after being moistened with weak glue or gum-water, is pressed into a crucible of refractory clay; a hole is then made in the charcoal paste by pressing in it the small end of a pestle; it is dried at a very gentle heat, and is then ready for use*.

It is well known that in ordinary furnaces the maximum heat is an inch or two above the bars; it is usual therefore to support the crucible to be heated, upon some substance equally able to stand the temperature to be employed. A portion of an old crucible, or a fragment of brick, is generally used. The fire must be built round the crucible with great care, so that when it falls, as the combustion proceeds, the position of the vessel may not be altered, or its safety, and that of its contents, endangered. It is not always convenient to warm the crucible previous to the operation in a separate furnace; it is therefore a good plan to commence building up the fire round the crucible-support with a few pieces of ignited charcoal, and then continue it with cold coke in small pieces. For an ordinary-sized furnace, where the space between the crucible and the walls all round is about $7\frac{1}{2}$ inches, the fragments should not much exceed the size of a large walnut; by this means the combustion will proceed so gradually that the crucible will run but small danger of fracture during the heating. When the whole of the fuel has acquired a full red heat, if it be gently touched with a rod, it will sink in

* For another kind of carbon crucible, see Appendix, on the "Production of High Temperatures."

consequence of the charcoal on which it rested having been consumed; more fuel must now be added, and the furnace, if its construction will admit of it, should be filled almost to the top, or at least as nearly so as is practicable, without affecting the free egress of the air and smoke through the flue. In almost all ordinary cases, the operation will be concluded without the addition of any more fuel; but where the experiment requires the protracted application of a very intense heat, more will be required, and it is very necessary to be able to supply it from an adjacent stove, kept burning merely to supply the one where the fusion, &c. is proceeding, as, if cold fuel be added, the temperature of the furnace will fall considerably, the process will be proportionably retarded, and, if it come in contact with the intensely ignited crucible, a fracture will probably result. For these reasons, it is far better to keep on adding red-hot coke by small portions as the fuel sinks, so that the crucible shall always remain covered to the depth of 4 or 5 inches with the red-hot fuel. In protracted experiments, a certain quantity of slag or clinker falls to the grating and closes it up, thereby materially retarding the operation by preventing free ingress of the air; if this be neglected for a short time, it accumulates so much that it will be dangerous to attempt its removal during a fusion, as it would be extremely difficult to do so without disturbing the crucible, and thereby running a risk of losing its contents. For the same reason, if any considerable number of experiments in crucibles at very high temperatures have to be performed, it is proper to take every care to provide the best coke, and to see that the furnace is properly freed from clinkers every time it is to be used. The tendency of some metals and alloys to absorb silicium at these temperatures, must not be overlooked in operations where such an action may exert an injurious tendency.

Sometimes even the best crucibles will open and discharge their contents into the fire during an ignition, especially where fluxes are present; it is therefore good policy to make a point of having the crucible well luted before exposure to the fire.

To prepare this luting, I have always found the following mode of procedure answer extremely well:—Sift some good Stourbridge clay through a tolerably fine sieve (about twelve holes to the inch), and let it be thoroughly beaten in a mortar with some fresh horse-dung to a smooth paste; pass a cloth slightly wet over the crucible so as to damp it, and apply the luting with a knife or spatula; let it be neatly and evenly applied, to the depth of a quarter of an inch, then let the crucible be placed in a moderately warm situation until thoroughly dry, and when this is the case, any cracks which have appeared are to be made good, and after being dried the second time the vessel is ready for use. The same mode of proceeding answers very well for stoneware retorts and tubes.

286. Very frequent operations in laboratories of research are the ignition of oxide of copper, and the fusion of chromate of lead; when these are performed, extreme care is requisite to prevent fragments of coke or coal, or even the smoke of the furnace, finding their way into the crucible. It is best to place the crucible containing the material on the top of the furnace until quite hot, and during this preliminary heating to make up the fire with coke, so that it may be quite clear and free from smoke; the rings (§ 29) may then be removed until an aperture is obtained rather less than the largest part of the crucible, which may then be dropped into the aperture, it being supported by its widest part; it will soon become very hot, and in a fit state to be inserted in the fire, which should be so managed that the fuel does not extend to within an inch of the top of the crucible. If oxide of copper which has been used is being ignited, after treatment with a little nitric acid to reoxidize the reduced metal, it may be lifted out of the fire and stirred occasionally until the whole has acquired a dull red heat, and the nitrate of copper become perfectly decomposed. The fusion of the chromate of lead is accelerated by stirring now and then, as otherwise the bottom part fuses and descends, sometimes leaving the rest above it in the cooler part of the crucible.

287. If proper care be taken, earthen crucibles may be used

for operations which are generally supposed to be necessarily performed in more valuable vessels. If properly freed from iron and other impurities, chloride of calcium may be prepared of the purest white in ordinary English crucibles.

288. Porcelain basins are very well adapted for some fusions, as for instance the white chloride of zinc; the furnaces, figs. 1 and 17, are convenient for this purpose; one or two of the rings being removed, the basin containing a dense solution of the purified chloride may be supported upon it, care being taken to exclude dust and dirt: this method is far more efficacious and economical than the use of gas, especially where large quantities have to be made.

289. In ignitions in porcelain and platinum crucibles over gas- and spirit-lamps, much care is required to prevent loss of the material. Some substances decrepitate violently when exposed to heat; when this occurs, the lid of the crucible should be kept on. The decrepitation, however, may sometimes be prevented by finely pulverizing the matter to be ignited.

290. In the ignition of the platinum and gold salts of the organic alkaloids, it often happens that the metal volatilizes with the vapours, although the temperature had been kept below redness until the organic matters were destroyed. It is necessary, at such times, to cover the salt with pure dry carbonate of soda; and, after the ignition, to wash the latter away on a filter; the whole is then to be re-ignited and the filter-ash deducted from the weight of the metal obtained. Where the salt of silver, gold or platinum is difficult to burn, which very seldom happens, it is sometimes admissible to drop a small fragment of solid and extremely pure nitrate of ammonia into the crucible, to facilitate combustion of the carbon; but great care is requisite in its use, to avoid a deflagration violent enough to project any of the metal. For other precautions necessary to be observed in operations of this class, see §§ 90, 218, & 276.

291. In every case of ignition to remove carbon, care must be taken to allow air to enter the crucible, and this may be done in several ways, the most eligible of which have been already described (§ 86).

292. The method of cooling a platinum crucible with rapidity has been more than once mentioned; if, however, a porcelain vessel were exposed to the same treatment, its fracture would be inevitable; when it is desired, therefore, to cool it as quickly as possible without danger of fracture, it may be placed upon a piece of wire-gauze, supported upon a capsule or other convenient support; or, if its contents are hygroscopic, upon a triangle over a surface of sulphuric acid.

293. In connexion with the furnace operations with crucibles at high temperatures, there are a few precautions which must not be neglected. If, while a substance is in a state of fusion, it is desired to add more, it must be carefully ascertained that no moisture is contained in it, as violent and dangerous explosions, accompanied with projection of the fused matter, may be the result. It is often convenient to remove one of the rings (§ 29) and lay a plate of talc upon the aperture, to enable the operator to view the interior of the furnace and, in some circumstances, of the crucible also.

294. Where it is necessary to melt a more with a less fusible substance, as, for example, in the preparation of brass, the less fusible metal is invariably to be melted first, then the other to be added, and the whole well stirred together. If, in the example given, the zinc were first fused, and the copper added, almost the whole of the former metal would be oxidized or volatilized before the temperature had become sufficiently high to fuse the copper.

295. It is a very common operation in laboratories to have to reduce the silver, gold, and platinum from the constantly accumulating stock of precipitates and filter-papers. It is, however, much better in the case of silver to keep the papers separate and heat them by themselves, as if the mixture of solution and papers be placed in a basin to be dried down, there is sometimes a formation of some explosive compound, apparently derived from the mutual action of nitric acid, nitrate of silver, and the organic matter of the paper, which may be the cause of loss by projection. In the case of the platinum, the whole mixture when dry may be mixed in a red-hot crucible with nitre, and when the

organic matter is destroyed, the platinum may with ease be separated from the residue. With the gold and silver residues, the dried mass may be mixed with soda and borax, and be exposed to a brilliant yellow heat for half an hour, at the end of which time the silver or gold, as the case may be, will be obtained in a button.

296. Mr. How has shown that much trouble may be caused by the silver and platinum washings being inadvertently mixed, as the platinum dissolves in nitric acid when fused with silver. If, however, the nitric solution containing the two metals be evaporated to dryness and fused, water will separate the nitrate of silver, the platinum remaining behind insoluble; if any platinum still adheres to the silver, the latter must be precipitated by hydrochloric acid.

297. It is becoming a common operation in the analysis of inorganic compounds to effect reductions by hydrogen gas in porcelain crucibles; this may be performed in the manner shown in the engraving, fig. 160, where the tube, *a a*, conveying dry hydrogen, passes through a hole in a platinum lid adapted to a porcelain crucible, and descends a short distance into the latter, which may be supported upon a wire triangle over a lamp. It is sometimes possible to obtain porcelain covers with a tube of the same material, made in one piece, as seen in section, fig. 161, but they are rather difficult to procure.

Fig. 160.

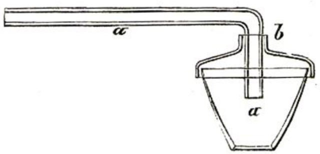
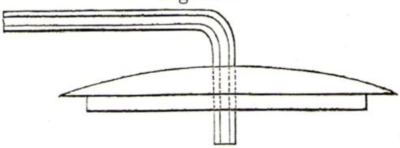


Fig. 161.



298. *Operations at high temperatures in tubes.*—In the chemistry of the present day tubes are much used as a means of exposure of substances to high temperatures, frequently with a gas of some

kind passing over it. The process of organic analysis in a current of oxygen may be instanced as an example. From the large surface which is by this means of operating exposed to the action of gaseous matter, a very advantageous mode of experimenting is the result, and, in fact, some of the most beautiful and accurate experiments in modern chemistry are thus performed. The material of which tubes are made for this purpose is generally glass, as it is easily worked to different shapes to suit the various purposes to which it may be applied. Earthen tubes are worse than useless for most experiments of this class, and those made of porcelain, although not porous like those last named, are so extremely liable to crack, that it is far better wherever possible to use glass, the only drawback being its fusibility; the hard Bohemian glass (of which the combustion-tubes for organic analysis are made) is, however, capable of withstanding a full red heat when supported as in a Liebig's furnace, and is therefore sufficiently refractory for all ordinary purposes.

299. Where the use of a very high temperature is unavoidable, gun-barrels may very often be made use of, and it will be shown farther on that they are well adapted for nitrogen determinations by Peligot's modification of the method of Varrentrapp and Will, where, although the heat required is not very great, it is seldom possible to use the same glass tube twice; and as in many laboratories it is common in the analysis of nitrogenized manures, soils, &c. to make three or four combustions a-day, the saving of tubes becomes worthy of attention. A good gun-barrel may be used for nearly a hundred analyses before being destroyed. A piece of iron gas-pipe may often be employed instead of a gun-barrel, and for some kinds of reduction experiments answers extremely well. It is often advisable to protect the glass tube by an outer one of copper, which may be constructed of a ribbon of the metal the same length as the tube, or a little shorter, and rather more than three times its diameter; after being heated to dull redness and allowed to cool to anneal and render it easy to work, it may be rolled round the tube with the hands, a small

piece of iron or copper wire being twisted rather tightly round either end to keep it firmly fixed.

300. It is necessary to be aware of the high conducting power of an exterior tube of copper when applied in this manner, as, if not known and guarded against, it would sometimes prejudicially affect the results. When required merely as a support, a narrow strip of metal may be made to lie upon the vertical supports throughout the whole length of the combustion-furnace, the tube resting upon it being thus prevented from curvature when softened by the high temperature.

301. Figure 7 on page 14 represents the general mode of procedure in experiments where a gas is to be passed over a substance at a red heat, as, for instance, in the reduction of ferric oxide or oxidized copper turnings by hydrogen. The piece of combustion-tubing, *a*, rests upon a series of vertical supports intended to prevent flexure. The gas evolved from an apparatus not seen in the figure, but which will be described further on, passes through the tube *b* into the bottle *c*, filled to about one-third its height with concentrated sulphuric acid, intended to absorb the moisture with which the gas is saturated; from thence it passes through *d* into the combustion-tube, which latter is closed at either end by corks, *e*, *e*, fitted with tubes, the posterior one being attached by means of a tube of caoutchouc to the tube *d*, evolving the dry gas. It is necessary to allow the gas to stream rapidly through the apparatus for some time before putting the red-hot charcoal in the furnace, in order that the atmospheric air may be perfectly expelled, as otherwise a violent explosion will occur, and of course the fragments flying about are liable to cause serious injury to the operator. It is always necessary to continue the stream of gas until no more water is produced, as may be proved by none condensing upon a cold glass plate held close to the exit-tube, or, in general, by steam ceasing to make its appearance. In all cases it is better to continue the flow of gas until the apparatus is quite cold, when the tube should be closely corked at either end, until the contents are wanted for use.

302. In experiments where a very intense heat is required,

it is necessary to lute the tube, no matter what may be the material of which it is composed; for if of iron, although the temperature may be far short of the point of fusion, the metal is rapidly oxidized and destroyed when exposed to a current of air at a red heat; the same applies to copper, and even platinum is injured by the sulphurous vapours to be found to a greater or less extent in all furnaces. In the case of the latter metal, it is essential to lute the tube to protect it from slag and the fusible impurities abounding in fires at strong heats.

As instances of some interesting furnace-tube experiments, may be mentioned the decomposition of water by metallic iron, the formation of the same by passing hydrogen over reducible metallic oxides, the formation of the beautiful sesquichloride of chromium by passing chlorine gas over a mixture containing oxide of chromium and charcoal, Berzelius's experiments on the separation of antimony, arsenic and sulphur from minerals, by passing chlorine over them at high temperatures, the conversion of toluidine into aniline, Captain Reynold's experiments on the production of propylene from valerianic acid, the formation of the anhydrous chloride of aluminium, the decomposition of some oily bases, with production of others having the power of giving coloured reactions, &c.

It appears from some experiments, that bodies standing high in homologous series may, by passing through tubes heated to moderately high temperatures, be reduced with deposition of carbon and evolution of gaseous hydrocarbons into members lower down in the scale; it need scarcely be said that a careful investigation of these decompositions is absolutely certain to be rewarded by valuable discoveries.