A handbook of chemical manipulation

Williams, Charles Greville

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Section II. Furnaces

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SECTION II.

FURNACES.

27. It is not intended to notice more than a very small portion of the furnaces that have been invented by chemists at different times. Their number is immense; even the first chemists, the adepts, had furnaces of almost every pattern that fancy could suggest, or complicated, yet mostly useless, processes require.

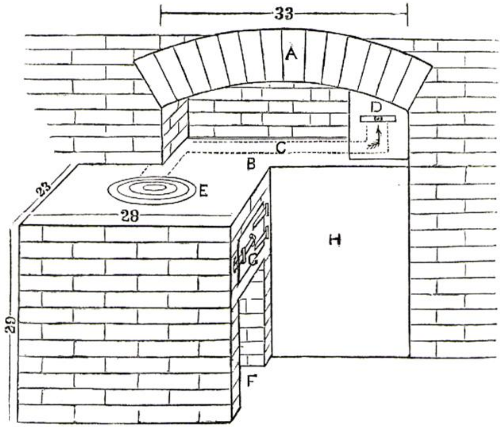
The Athanor was perhaps their masterpiece; it was intended to afford a steady heat, adapted for digestion or distillation, for a very long period, sometimes for many months. This was effected by a contrivance at once simple and ingenious: it consisted in having an air-tight tower, with a close-fitting lid at the back of the furnace, immediately connected with the fireplace; this held the charcoal in such a manner, that, as it burnt in the grate, it descended in the tower and replenished the fire.

28. The operations of modern chemistry do not generally require a long-continued heat to be applied to retorts or vessels for digestion; and where it occurs, gas is by far the most economical and convenient fuel. Lately, however, it has become necessary in some researches to expose substances in sealed tubes to 212° or higher for many hours, sometimes even for a week or more; and it is advisable in such cases, especially where regard is had to economy of time, to keep up the heat to the highest point, whatever that may be, during the whole period, instead of letting the temperature fall during the night. Gas, as being so easily regulated, will prove the most convenient source of heat in these experiments; where it is not to be had, considerable difficulty is generally found in constructing an arrangement which shall dispense with the operator's attention during the night. The manipulations connected with this subject will be found in the section on Pressure tube operations.

29. The first furnace which presents itself is that which is ordinarily in use in the laboratory, and which may be either on

Brande's or Luhme's principle. The former is generally used, and as it frequently happens that it has to be placed in a room

Fig. 1.



where there is a fireplace, and economy of space being usually of importance, a modification will be described which has been found extremely convenient in practice. The figures indicate the dimensions at the parts where they are attached: A is the arch of the fireplace; B the sand-bath under the arch; C the flue, indicated by the dotted lines; D the damper; E the rings by which the aperture over the fireplace is enlarged or contracted; F the ashpit, which may very conveniently have a door to regulate the draught; G the fire-door; H the hot closet, the roof consisting of an iron plate, between which and the top plate the flue runs between two courses of bricks. The upper plate, both outside and inside the arch, is in one piece. The rings fit easily into each other, in the manner indicated by the sections (figs. 2 and 3); if they fitted exactly, the expansion on heating would cause them to adhere. The space between each ring is somewhat exaggerated, for the sake of distinctness. They may be procured in

Fig. 2.

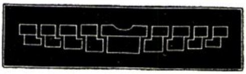
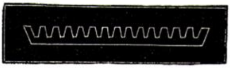


Fig. 3.

sets of the chemical instrument makers, or it is easy to have a wooden pattern made, and get them cast, in places where they are not procurable ready made. The price is very trifling. This furnace will give heat enough to melt several ounces of copper with ease, and by nearly closing the damper and opening the fire-door, a gentle heat fit for distillation or evaporation may be obtained. The fire-bars are loose, and fit into notches in iron bearings (fig. 4) built into the wall of the furnace at each end. By this means it is easy to replace a bar when destroyed. Several contrivances for distilling in different ways with this furnace will be described in the section on Distillation. The hot chamber is convenient for drying preparations, precipitates, &c. The temperature of course varies with the fire in the furnace, but from 100° to 150° may be taken as an average. It has an iron grating about half-way between the floor and the top, which is useful to support capsules, bottles, funnels, with their filters, &c. put in to dry.

Fig. 4.



It is advisable, in constructing this furnace, to have a slip of iron $1\frac{1}{2}$ inch high, placed in such a manner as to confine the sand to that portion of the iron plate which is beneath the arch. In order to obtain a sufficient draught, and yet not to prevent the possibility of using a hood, several contrivances are applicable.

In that given, the chimney to the fireplace is closed by slates about 30 inches from the top of the plate, and the furnace-flue is carried into another chimney running parallel with the first; by this means the pipe of the hood is enabled to play into the fireplace chimney without injuring the draught of the furnace.

The hood is not represented in the engraving; it projects completely over the furnace, its lower edge being about 3 feet 6 inches from the iron plate.

It will be seen that an arrangement of this kind has many advantages: it occupies little room, from the sand-bath being under the arch, while the light which comes laterally is quite sufficient for every purpose. Crucible operations are conveniently executed, from the great facility which the rings offer to the ingress and egress of the vessels; moreover, by placing a sheet of talc over the centre hole, after removal of the small stopper, the whole operation can be watched without distress to the face and eyes.

Evaporation is readily performed in Berlin or other porcelain dishes over the naked fire, on a ring of the proper size; or even, when it is wished to proceed more slowly, by placing the basin on the top of the rings, the stopper being in its place. In some sublimations, when considerable heat is required to be applied to flasks, two or three rings may be removed, and a sand-pot introduced of one of the shapes shown in figs. 5 and 6. They

Fig. 5.

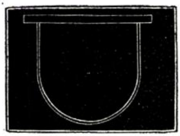
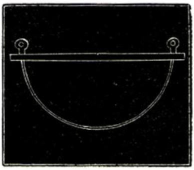


Fig. 6.

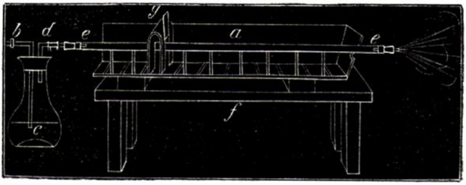


are made of cast iron, and may be obtained of the instrument makers at a very low price. In the section on Distillation, a method will be given for converting pots of this kind into retorts for destructive distillation, and for preparing sulphurous acid on a large scale.

Several evaporations may be conducted simultaneously at different parts of the plate and sand-bath, so that any temperature may with facility be commanded, from the melting point of copper or gold, and even, with care, cast iron, to the evaporation of an alcoholic solution, or, in the closet, the drying of the most delicate organic preparations.

There is one disadvantage which this furnace possesses, namely, that it offers no means for performing the numerous tube operations which it is so frequently necessary to resort to even in some of the least complex researches. In these cases, great use may generally be made of the sheet-iron combustion-furnace represented below.

Fig. 7.

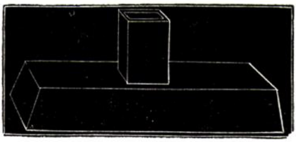


30. The cut represents the method of reducing copper turnings used in the ultimate analysis of organic substances containing nitrogen; *a* is the combustion-furnace, of sheet iron, seen in section. In figure 8, *a* is the front end, *b* a sectional view of the anterior extremity, showing the position of the supports, and *c* a screen to confine the fire to the parts required. The details of the method of using this instrument will be found in its proper place.

The length of this furnace, to be practically useful, should not be less than 30 inches by 4 inches high, and $3\frac{1}{2}$ broad at the bottom, increasing to 5 inches at top. Where considerable heat is required, as in the reduction of somewhat refractory substances, it is merely requisite to fan the ignited charcoal with a piece of cardboard; or a long parallelogram of sheet iron, ending in a chimney, as in fig. 9, may be employed. Instead of

Fig. 8.

Fig. 9.



the latter, a couple of the chimneys belonging to chauffers may be used, as they will raise the heat sufficiently for all the purposes to which the furnace is likely to be applied.

31. Where a very intense heat is required to be given to the tubes, one of Luhme's furnaces may be employed, which, from

its extreme adaptability to most chemical operations, will be described. It in many cases renders the brick furnace, shown in fig. 1, unnecessary. The first section, fig. 10, represents the body of the furnace, which is lined with fire-clay, and has apertures on each side for the passage of a tube. These holes are capable of being closed by the small doors shown open. Fig. 11 shows the body of the furnace with its fire- and ashpit-doors closed. The three projections seen at the top, are to allow of a large pan being placed over the fire without destroying the draught.

Fig. 10. Fig. 11. Fig. 12.

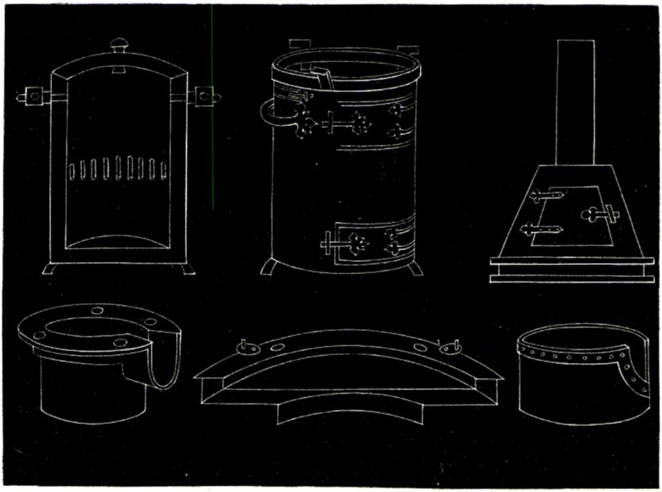
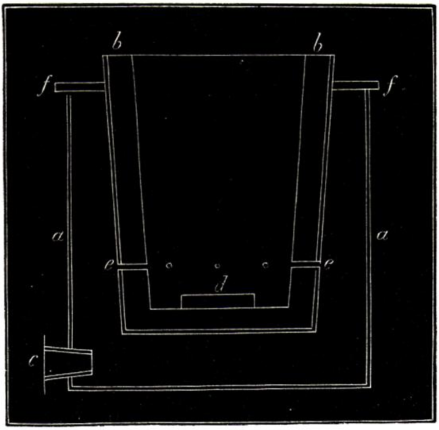


Fig. 13. Fig. 14. Fig. 15.

The chimney, fig. 12, which fits on over the body, has a door to allow of the introduction of fuel. Fig. 13 is a sand-bath adapted to distillations with retorts; it rests when in use on fig. 15; the broad depression in fig. 13, intended to allow of the passage of the neck of the retort, fitting into the corresponding notch in fig. 15. Four apertures on the top of fig. 13 allow of the escape of the hot air, so that the combustion is only slightly impeded by its use. Fig. 14 is a section of a broad shallow sand-bath, adapted to digestions and many other operations. When in use it rests on the top of fig. 11. The products of combustion pass between the double pieces of which it is composed, and escape by four apertures in the rim, two of which are seen in the engraving; they are capable of being closed by small moveable pieces of metal, so as to form, in connexion with the fire- and ashpit-doors, a perfect control over the temperature. The furnace just described, and which I have had in use for a long time, I have always been accustomed to feed with the London gas-coke. That kind which is obtained in Scotland, and sometimes elsewhere, which leaves an ash almost equal in bulk to the coke itself, is of course useless, and in such a difficulty, charcoal, although a very expensive fuel, must be used.

In most laboratories of research, such furnaces as the two just described will be sufficient for the performance of all the operations likely to be undertaken; but it nevertheless some-

Fig. 16.



times happens that experiments requiring a very intense heat are necessary; when such is the case, recourse must be had to a blast-furnace. The most convenient and powerful of these is that of Sefström. It consists of a thick sheet-iron cylinder, *a a* (fig. 16); on the top of this is a strong iron ring, *f f*, which is intended to support a second cylinder, *b b*, which drops into it; this has six tubes, $\frac{5}{8}$ths of an inch in diameter, fastened into it, which are intended to conduct the air from the vacant space. They are $2\frac{1}{4}$ inches long, and are, as is likewise the rest of the cylinder, covered with fire-clay. The tube *c*, which projects at the bottom of the outer case, is intended to admit the nozzle of a pair of powerful bellows. The proper composition for lining the surface is prepared by patiently mixing fresh Stourbridge clay with a little horse-dung, and adding about a sixth of a powder made by pulverizing old crucibles. The composition must not be applied too moist, and is to be allowed to dry spontaneously.

It is generally found on drying that cracks have been formed, which must be filled up with a little more of the composition. When *quite* dry, a gentle fire is made in the furnace, and gradually increased to the highest pitch by a vigorous application of the bellows; it is then allowed to cool, and the surface of the clay is usually found to be vitrified from the intense heat. The crucible is always supported in the furnace about 3 inches from the bottom. A piece of brick or an old crucible, as at *d*, fig. 16, resting on a thin stratum of sand, to prevent it from adhering to the lining, forms perhaps the best support.

Staffordshire coke should be used, from its small tendency to produce a fluid slag, which would fill up the tubes, and so prevent the ingress of the air. By means of this furnace may be obtained the most powerful heat which can be applied to crucibles in laboratories*. In fact, its power is greater than the resistance offered by the best vessels: it not unfrequently happens that a good Hessian crucible is completely fused; this is more likely to happen with common gas-coke than with the Staffordshire, as the slag produced from the furnace acts as an

* The improved apparatus of M. H. Sainte-Claire Deville, by means of which even platinum may be melted and volatilized, will be described in the Appendix.

energetic flux. This description of furnace was especially valuable at a time when, in mineral analyses, fusions with carbonate of baryta were often necessary.

The platinum crucibles are to be imbedded in magnesia contained in a good Hessian or London crucible, as, if exposed to the naked fire, they would be readily destroyed by the slag; even if they escaped this, they would soon be rendered rotten and useless by the sulphurous and other vapours which are invariably present in an active state in the atmosphere of a blast-furnace.

It is scarcely necessary to caution the chemist against the danger arising from the performance of an operation evolving such an intense heat and abundance of sparks, in an unprotected or unsafe place. It is advisable, where the full power of the furnace is required, to have a good fire at hand, in order to replenish with bright red-hot fuel, as great reduction of temperature would arise from feeding it with cold coke. In ordinary experiments this will not be necessary, as, from the intensity of the heat, operations performed with it are usually soon finished, and frequently without the necessity of adding any fresh fuel. The furnace will be found of service in the reduction of iron ores on the small scale; also in the formation of alloys of the more refractory metals.

32. It was intended to give an illustration of Aikin's blast-furnace, formed from large black-lead crucibles, but the superiority both in point of economy and durability of the one just mentioned will render such a description unnecessary, and enable us to devote the space to the consideration of some other convenient and more frequently used furnaces.

33. In establishments where fused potash, nitrate of silver, chloride of zinc, and similar preparations are made, the little furnace about to be described is, from its simplicity and cheapness, very useful; it may be used also for distillations, and small crucible experiments. It is, however, less applicable for distillations than other operations, because there is no fire-door; the fuel being introduced at the aperture in the plate, which is closed by rings, *c*, fig. 17, like the furnace first described. The

heat is regulated by a damper, *d*, in the usual manner. The chief peculiarities are in the arrangement of the grates and the shape of the fireplace; for it will be seen there are notches at different heights supporting square rods on which fire-bars may be placed, as at *b b b*, to adapt the furnace either for crucible operations, distillations or fusions, in pans on the rings. Those bars not in use, are to be removed and the holes to be closed by brick or iron stoppers; and, if a very intense heat be required, the interstices, if any, may be made good with a little Stourbridge clay. As the ends of the bars project from the furnace, they may be removed with a pair of tongs, and the fire is thus immediately extinguished. The bars are of the usual shape and lie side by side, the square piece at the ends preventing them from lying so close as to injure the draught. Fig. 18 gives a view of the bars as seen when looking down into the furnace, and also a side section. This arrangement, when playing into a tall chimney, forms an intensely powerful wind-furnace, and that at a very small cost, as it can be erected by any workman. It should be lined with fire-bricks. It will be seen that the fireplace is made slightly conical, so as to allow the easy descent of the fuel. The addition of a fire-door of course makes the furnace fit for distillation with retorts, &c. The top is formed by a plate of wrought iron pierced for the rings; or if cast, it should be made with a flange to fit them accurately. This furnace may

Fig. 17.

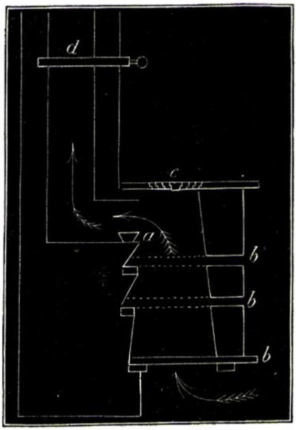
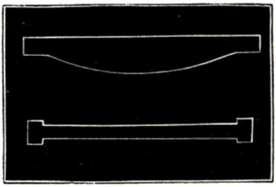


Fig. 18.



also be used for cupellations by placing the cupel on the exit of the hot air, as at *a*; and the progress of the experiment may be watched from the ring-hole by removing the stopper *c*. In this operation a piece of red-hot coke may be placed on the cupel, in such a manner as to concentrate the heat on the assay without preventing the access of the air.

It is necessary to observe, that the furnace, if intended for operations requiring a very powerful heat, should be constructed of a different size to what it would be if only used to evaporate and fuse the chemicals alluded to, or similar preparations. Even then, however, the same relative proportions may be preserved.

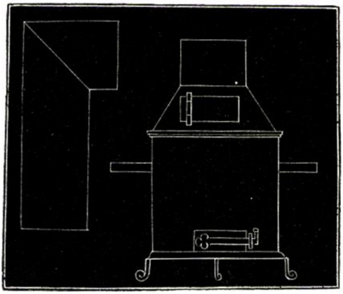
Considerable care and attention is requisite in using powerful furnaces, to obtain a temperature adapted to the operation. It may be taken as a rule, generally, that there is more danger of heating a crucible too highly than too little. Carelessness in this respect is often the cause of much disappointment and annoyance to persons unaccustomed to the necessary manipulations.

34. Where many consecutive crucible operations of the same kind are to be performed, as, for instance, in the preparation of a considerable quantity of a fused chemical product, much advantage is often gained by not permitting the crucible to cool. If, in making crude nitrite of potash by fusing nitre, we first melt the salt in an iron basin to expel the water, and then put it into a previously warmed common English crucible, and gradually raise the temperature to bright redness, we shall often find, on pouring out its contents, that the crucible is uninjured, and if immediately returned to the fire may be refilled with the dried salt, and the operation continued in the same manner until many pounds of the nitrite have been made. If, on the contrary, the damp nitre* be placed in a cold crucible, and the latter be at once introduced into the fire, it is sure to be destroyed, and, in all probability, the contents lost. It is therefore advisable to put the crucibles intended to be used, on the top of the furnace for an hour or so before they

* Commercial saltpetre, although apparently dry and containing no water of crystallization, always retains a considerable amount between the layers of the crystals.

are wanted, to ensure their complete dryness and to prevent too rapid change of temperature, on being put into the fire. When crucibles or stoneware retorts are required to be luted, as is the case when they are to be exposed to a long-continued or violent heat, a very convenient mixture for the purpose is composed of sifted Stourbridge clay well mixed with a little damp horse-dung to which a small quantity of a strong solution of borax has been added. It is to be evenly applied over the outer surface with a knife. The vessel should then be placed on a moderately warm part of the furnace until perfectly dry, and the cracks which will probably have formed must then be filled up and the drying be repeated. Further directions for the application of luting will be found in the section on Distillation at a high temperature.

Fig. 19.

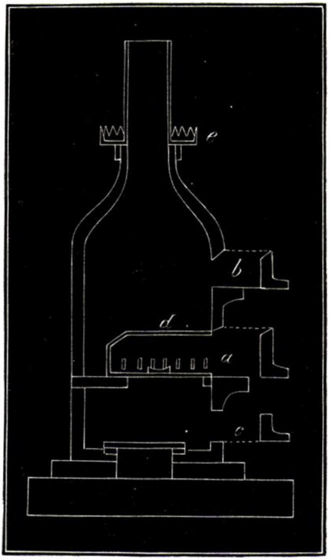


35. One of the most convenient and often used substitutes for the furnace is the sheet-iron chauffer, fig. 19. It is intended to burn charcoal, and, when used with the additional chimney, it gives a very good heat, quite sufficient for the decomposition of siliceous minerals by the improved processes of Lawrence Smith, and others. To adapt it for tube operations it is provided with holes opposite each other, which are capable of being closed by means of small pieces of sheet iron moving on a rivet. The grate is supported by three projecting pieces of sheet iron riveted on. The small door at the top is for the purpose of enabling fuel to be added without removing the chimney. By means of an iron triangle placed over the chauffer, after the chimney is removed, we at once adapt it to distillations and evaporations. The smallest dimensions practically convenient are 6 inches in diameter and the same in depth, the grate being placed immediately above the

door, which is $2\frac{1}{4}$ inches high by about $3\frac{1}{2}$ long. One of the advantages connected with it, is the facility it affords to operations requiring its presence on the tables or other parts of the laboratory. When placed upon any wooden substance it is supported by a tile, which prevents any danger from the heat.

Its use, to contain a supply of red-hot charcoal during the process of organic analysis in laboratories not possessing a complete arrangement for the purpose, will be alluded to in the section devoted to that subject. A furnace of this description is far more convenient than any substitute made from "blue pots," which are recommended in many chemical works. The latter invariably crack after one or two operations, are more troublesome to construct, much heavier, and, in fact, less eligible in every respect. Sometimes, however, the French clay-chauffers may, from their convenience of form and cheapness, be used with advantage; and the better class of them may be used a considerable time before any serious injury occurs. They should be bound with strips of iron, or wire, in several places, in order to keep them together when fissures make their appearance.

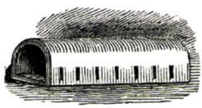
Fig. 20.



36. It has been said that the furnace represented in fig. 17 may be used for cupellations, but when an apparatus especially adapted to this process is desired, that depicted in fig. 20 is perhaps as convenient as any. It is made, preferably, of sheet iron lined with fire-clay. Those constructed entirely of an earthen material, are liable to the disadvantage alluded to in the case of the crucible-furnaces made from blue pots. It consists, essentially,

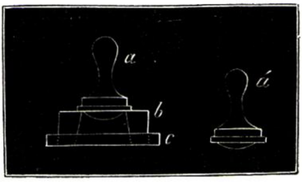
of a chamber either square or cylindrical, provided with three apertures: one, *a*, to admit the muffle, fig. 21; another, *b*, to introduce the fuel; and a third, *c*, to regulate the draught. The muffle, *d*, is seen to be supported at one end by the edge of the aperture in the furnace, and at the other by a projection opposite to it. The small gallery, *e*, placed round the chimney, is intended to support the newly-made cupels during the process of drying, sufficient heat for which is obtained partly by radiation and partly by conduction. The muffles are small earthen ovens pierced on the sides to enable a current of air to play on the assay, by which means the more oxidizable metals are converted into a state which enables them to be absorbed by the cupel.

Fig. 21.



The best fuel for a small assay-furnace is charcoal in moderate-sized pieces mixed with somewhat smaller fragments of coke or well-sifted cinders. The cupels are formed by mixing finely sifted bone-ashes with just sufficient water to enable the powder to adhere; it is then pressed into a cupel-mould, fig. 22, the superfluous mass being removed by a knife; and the stamper, *á*, being introduced, a smart blow is given by a mallet, and, on reversing the mould, a little pressure enables us to remove the cupel, which must be thoroughly dried before use. In fig. 22, in addition to the stamper, *a*, and the mould, *b*, a ring, *c*, is introduced; this is, however, only used where the unnecessary complication of having *b* made in two pieces, to facilitate the removal of the cupel, is adopted; the ordinary arrangement, which requires only the two parts *a* and *b*, is by far the best. The division of *c* into two pieces, and keeping them together by a ring during the time the cupel is being formed, appears to be an imitation of Plattner's mould for crucibles. The details of the process of cupellation are of course foreign to a book on manipulation, as they would afford sufficient

Fig. 22.



material for a treatise on that subject exclusively*; but we may mention, that it is founded on the non-oxidizability of gold and silver; if, therefore, we expose a mixture of these metals on a cupel, with the addition of a certain quantity of lead, to a current of air at a high temperature, the less valuable metals present are converted into oxides, which, being fluxed by the excess of fusible oxide of lead, become fluid, and are absorbed by the cupel. At the end of the operation, therefore, we have the pure metal (or a mixture, if both gold and silver be present) in the state of a globule on the cupel; it is then removed and weighed. If silver be present, the resulting button is again subjected to the operations of quartation and parting, the details of which are to be found in the work mentioned in the note, and also in most manuals of chemistry.

37. It must be remembered, that, in furnaces of the ordinary kind, the maximum heat is about $1\frac{1}{2}$ or 2 inches above the bars; the crucible should therefore be supported on a piece of brick, or another crucible inverted, by which means it will acquire much more heat than it would if put directly on the bars.

38. It is sometimes required to construct a furnace that shall heat an exceedingly large sand-bath; and as it is, of course, an object to accomplish this end with as small an expenditure of fuel as possible, the flue is made to wind backwards and forwards, so as to deprive the current of air and smoke of as much heat as possible; the bricks at the end of each turn are capable of being removed, so as to enable the flue to be cleaned without taking the plate off. A sand-bath of this description is extremely valuable in numerous operations, especially for sublimations which are to be performed at a gentle heat; such, for instance, as the manufacture of pyrogallic acid, much used of late years in photography. By the arrangement alluded to, all gradations of temperature, from a dull red to a gentle digesting heat, may be obtained; moreover, the flue is provided with a damper, which enables us to increase or lessen the fire at will.

* For information on these points, the reader is referred to Mitchell's 'Manual of Practical Assaying.'