PART I.

INTRODUCTORY.

§ 1. Metallurgy in its fullest sense treats of metals in their natural state, the mode of their extraction from the ore, and their application to various manufacturing purposes.

It is a chemical art requiring a knowledge of the laws of Chemistry, and a metallurgical training is not complete without some acquaintance with that special branch known as Analytical Chemistry, by which the amount of every individual constituent of a compound may be determined. In fact, Metallurgy is the application of Chemistry, Physics and Mechanics to the treatment of metalliferous materials with a view to the extraction of the metals and their conversion to workable forms.

To certain groups of metals different modes of treatment are applicable, which vary from each other as much as the metals do themselves. All these methods rest on general principles, and in many cases the same reagents, the same apparatus, and the same processes are employed.

§ 2. The term metal has long been applied to a certain number of the chemical elements which have well-defined physical characters in common, by which they may be readily recognised. Gold and silver were formerly regarded as the types, on account of their high specific gravity, brilliant lustre, and superior conductivity for heat and electricity. But these properties are by no means distinctive of metals, as there are many bodies, not classified among the metals, which have some of these physical characteristics.

Of the metallic elements there are about seventeen which may be called "useful" metals, viz.: silver, aluminium, arsenic, gold, bismuth, cobalt, copper, iron, mercury, manganese, nickel, lead, platinum, tin, antimony, zinc, and magnesium.

§ 3. Mercury, Caesium, and Gallium are the only metals liquid at the ordinary temperature, all the rest being solid and opaque except in extremely thin sheets. Copper is red, gold is yellow, the others white or greyish white.

The behaviour of the metals under the influence of heat is very varied. Tin, antimony, bismuth, lead, and zinc melt below a red heat. Gold, silver, and copper require a bright red. Iron, manganese, nickel, and cobalt, an intense white; while platinum will only melt in the oxy-hydrogen flame or by the agency of an electric current. With the exception of arsenic,* all are capable of becoming liquid when heated under ordinary conditions, and at high temperatures volatilised. Arsenic, antimony, mercury, zinc, and cadmium are readily volatile. There appears to be no relation between volatility and fusibility, for zinc and antimony require a much higher temperature than tin to fuse them, yet tin requires a strong heat to volatilise it.

§ 4. One of the most distinctive features of a metal is an internal mobility, in virtue of which its shape may be altered by pressure without disruption of the mass. This property is possessed by metals in varying degrees so that the "malleability" or power of being extended by pressure without cracking, and "ductility," or the capability of

* Professor Mallet and other observers have proved that arsenic may be liquefied by heat when under pressure.

being permanently elongated by a tensile stress combined with lateral pressure, which allows most metals to be drawn into wire, are by no means equal in extent: nor is the order of their malleability the same as for ductility, for the former depends on the softness and tenacity, while the latter is much more dependent on tenacity. By tenacity is understood the strength with which metals resist an attempt to pull their particles asunder by the action of a tensile stress. The tenacity is generally diminished as the temperature is increased, while, within certain limits, the reverse is the case with regard to malleability and ductility. Some metals have a very feeble tenacity and are then said to be brittle. When a metal resists rupture by a bending or twisting force it is said to be "tough." "Elasticity" is the power a body possesses of resuming its original form after the removal of an external force which has produced a change in that form. The point at which the elasticity and the applied stress counterbalance each other is termed the limit of elasticity. "Sonorousness" is an attribute of the harder metals, and is very marked in some of their alloys. "Hardness" is the resistance offered by the molecules of a substance to their separation by the penetrating action of another body, and like all other physical properties is considerably modified by the presence of impurities, so that in many cases softness is a test of purity. All malleable metals become hardened by pressure, and require occasional annealing during the process of manufacture. The fractured surface of metals is often characteristic, being spoken of as fibrous, crystalline, granular, silky, columnar, conchoidal, etc. Crystalline structure is often accompanied by brittleness, and fibrous structure by high tenacity.

§ 5. With regard to conducting power for heat and electricity the metals occupy the first place, silver standing at the head of the list. No physical property is more affected by even traces of impurities than conductivity; it is also diminished by a rise in temperature. The capacity of metals for heat extends over a wide range; iron, for example, being more than $\frac{1}{10}$th that of water, while lead is less than $\frac{1}{30}$th. For the most part metals are heavier than water, platinum occupying the highest position among the useful metals, being $21\frac{1}{2}$ times heavier than an equal bulk of water. The specific weight determines the value of a metal for many purposes, as gold, for instance, the specific gravity of which being $19\frac{1}{2}$, greatly increases its convenience as a circulating medium, and aluminium is suitable for making small weights on account of its lightness.

The metallic elements are usually basic in character when united with oxygen, but this property is only relative, as an oxide which is basic in one compound may become acid when allied with a stronger base.

§ 6. By uniting two or more metals in various proportions an almost infinite variety of combinations may be obtained, possessing to a greater or less extent the properties of their constituents. The effect of this union is generally to increase the hardness, lower the melting point, alter the specific gravity, and otherwise modify the character of the components. Many metals are capable of uniting with others in definite proportions forming true chemical compounds; but as such compounds can be dissolved by the molten metals, they may with few exceptions be mixed in almost all proportions. The similarity in basic character of the metals would lead one to suppose that such combinations would be held together by feeble degrees of affinity, which would make it difficult to isolate them from their solvent metals. That there is in many cases some kind of affinity is evidenced by the heat produced when some metals are alloyed together. Thus, aluminium and copper, platinum and tin, arsenic and antimony, bismuth and lead, gold and just melted

tin evolve heat when they unite; on the other hand, lead and tin absorb heat in their union. But there is no great alteration of properties which is the general distinctive feature of chemical combination, the change being chiefly limited to variations in colour, malleability, tenacity, etc., while the product is always decidedly metallic. In some cases a small amount of a malleable metal added to another highly malleable one, will produce a brittle alloy, such as lead in gold. It is possible that two metals may combine when melted and separate on cooling. Matthiessen considers it probable that the condition of metals in alloys when in the liquid state is one of the following:—1. A solution of one metal in another; 2. Chemical combination; 3. Mechanical mixture; 4. A solution or mixture of two or all of the foregoing; and 5. One or more of the metals in the alloy may be present in an allotropic form.* Alloys are usually formed by melting the metals together, but they may also be prepared by strongly compressing together the constituent metals previously reduced to a fine state of division.

a. The action of acids and other solvents on alloys varies with the relative quantities of the metals present. Silver when alloyed with much gold is not affected by nitric acid, but when the silver is in excess, it may be entirely dissolved. Platinum, which when alone is insoluble in nitric acid or sulphuric acid, is readily dissolved when united with much silver.

b. The separation of the constituents of some alloys may be brought about by raising them to the temperature at which the most fusible melts, which then "liquates" out. In this way silver is removed from copper by means of lead; tin from iron and arsenic; mercury from gold, etc.

* See author's work on "Mixed Metals," p. 43. Macmillan & Co, Ltd.

LABORATORY APPLIANCES.

§ 7. A Metallurgical Laboratory requires as much space as is usually occupied by a Chemical Laboratory, and, in

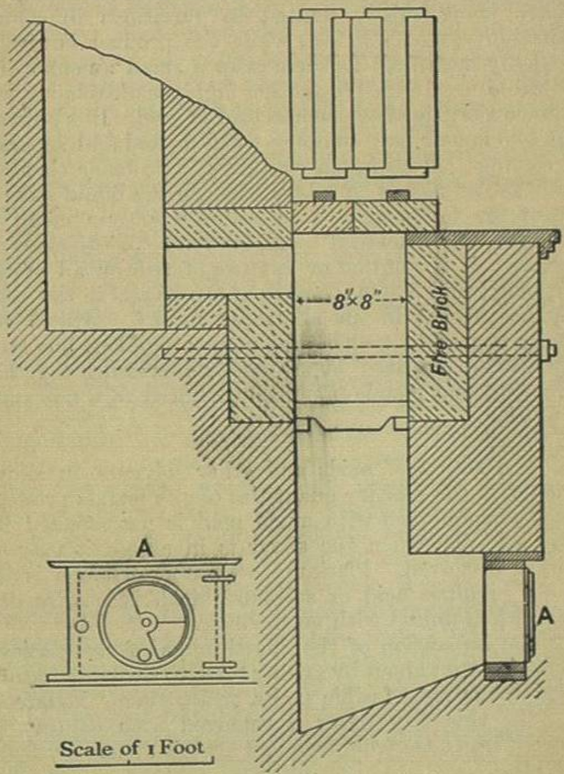


Fig. 1.

addition, necessary room for furnaces and muffles, proper

space being allowed for students to work at the fires, this being generally the busiest and most crowded part. The basement of a building is the most convenient, so that a good height of chimney may be obtained. The floor should be of brick or stone near the furnaces; all other parts may be boarded, or, if a brick floor extends all over the laboratory, it is advisable to have wooden stages in front of the work benches, so as to avoid damp, which is an usual accompaniment of basement rooms.

§ 8. The work benches should be fitted with drawers and cupboards, and with shelves above for reagents and fluxes. The tops of the benches require to be made of hard wood such as teak, or American walnut, to admit of the various hammering and pounding operations required in treating the products of the crucible, and the preparation of the tests for the furnace.

§ 9. The proper construction of the furnaces is a matter of the first importance, as a slight difference in the arrangement of the flues will considerably affect the draught, and prevent that high temperature requisite in melting such metals as iron, steel and nickel. The small wind furnaces used at the Royal School of Mines, and recommended by the Science and Art Department, are 1 foot deep and 8 inches square (Fig. 1). The draught hole is 6 inches by 3 inches, and the ashpit should have

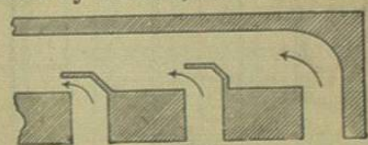


Fig. 2.

a capacity at least equal to that of the furnace. The bars are 9 inches long, $1\frac{1}{4}$ inches wide at top, and gradually tapering to the bottom. These are rather

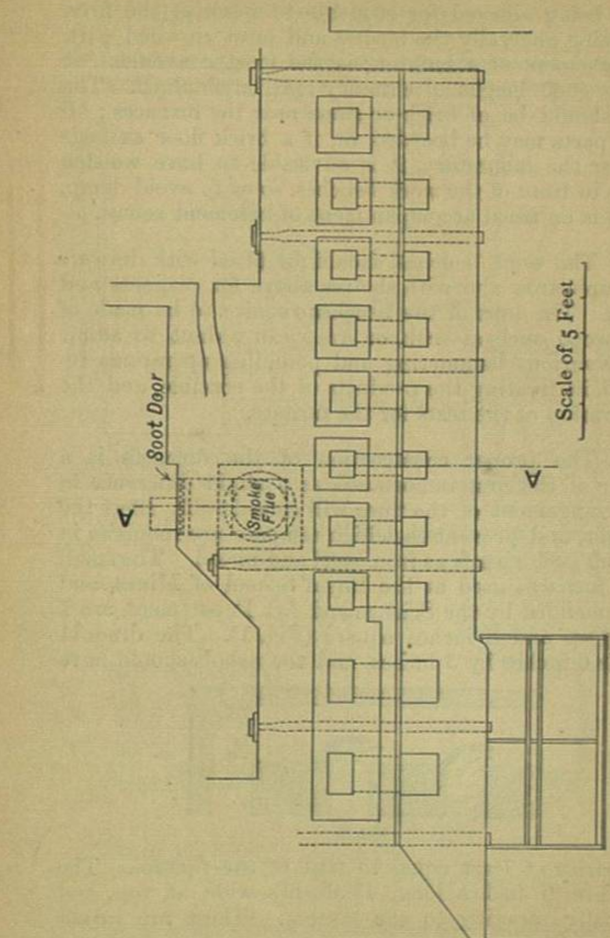


Fig. 3.

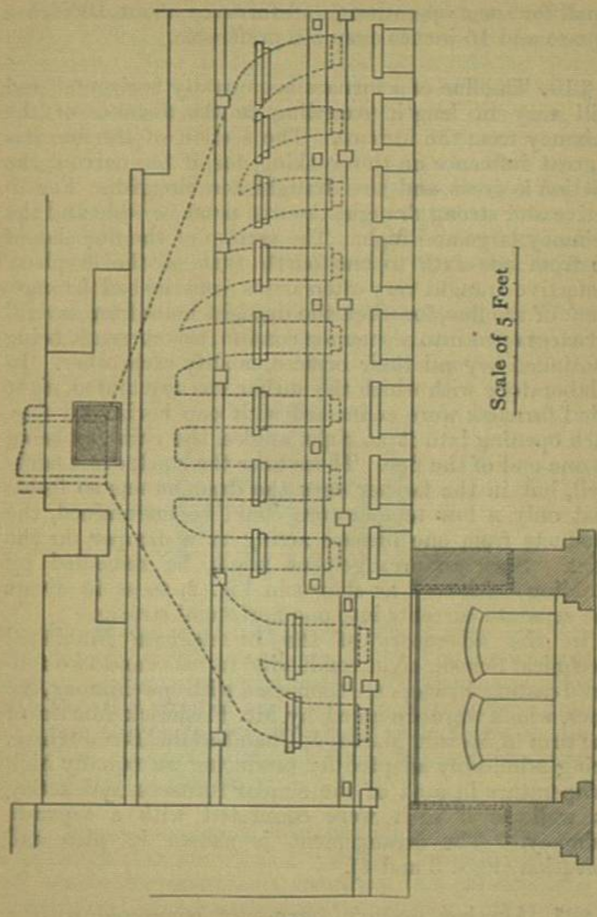


Fig. 4.

small for some operations, and furnaces about 10 inches square and 15 inches deep are preferable.

§ 10. The flue of a furnace is generally horizontal, and will vary in length according to the distance of the chimney from the furnace. The section of the flue has a great influence on the working, for, if too narrow, the friction is great and the draught too sluggish. For an active and strong draught the flue must be wide and the chimney large and high. The section of the flue should be from one-sixth to one-fourth that of the fireplace. Defective draught very often arises from the bad arrangement of the flue, for when the draught holes from several furnaces open into a common conduit, the currents, being continued beyond their orifices, modify each other. In a laboratory with which the author was acquainted, eight wind furnaces were connected with one horizontal flue, each opening into it at right angles, the chimney being at one end of the flue. Those near the stack drew fairly well, but in the farther ones the draught was so feeble that only a low temperature could be maintained, the products from one furnace acting as a damper on the next. Such an arrangement might be remedied by building brickwork as shown in Fig. 2, so as to divert the separate currents into one horizontal stream.

In the laboratory of the Birmingham Municipal Technical School, eight small wind furnaces and two full-sized casting furnaces are connected with one chimney, the flues, which were designed by Mr. Frederick Martin of the firm of Messrs. Martin & Chamberlain, Birmingham, being admirably adapted for producing an equally high temperature in each of the similar furnaces, and acting as well as if each were connected with a separate chimney. The arrangement is shown in plan and elevation (Figs. 3 and 4).

§ 11. Another essential structure connected with a

wind furnace is the stack or chimney, which is used for creating a draught and for carrying off the products of combustion. Now if the chimney be connected with several furnaces having an arrangement of flues as in Fig. 2 so as to produce a current in one direction, a single opening will be sufficient; but if furnaces are arranged on each side of the stalk, and the flues open into it at right angles, the shaft must be divided for a certain distance so as to divert the two streams into one vertical direction, as shown in the accompanying figure (Fig. 5), otherwise they will interfere with each other and check the draught.

The chimneys of the Birmingham laboratory are 70 ft. high, and built with double channels, as seen by the

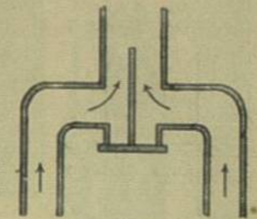
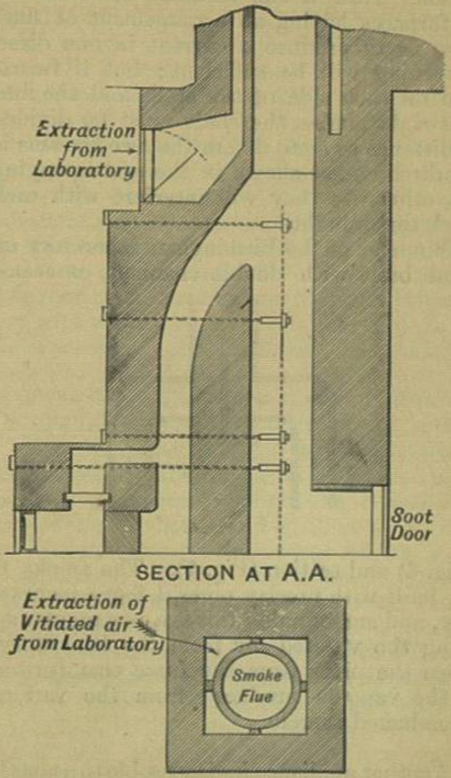


Fig. 5.

plan (Fig. 6) and section (Fig. 7). The smoke flue A is circular, built with fireclay pipes, 1 foot 8 inches internal diameter, and surrounding this is a rectangular space for extracting the vitiated air from the laboratory. Into this space the pipes from the fume chambers also discharge the vapours produced from the various operations conducted therein.

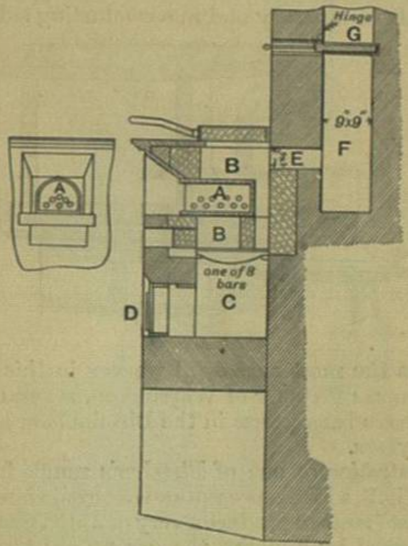
§ 12. Another appliance indispensable to a metallurgical laboratory is that known as a "Muffle" furnace, which is used chiefly for operations requiring the passage of a current of air, as in roasting and cupellation; its special feature consisting of a device for isolating the materials operated upon, from both the fuel and the products of



Figs. 6 and 7.

combustion. The muffle proper is a ⌂ shaped fireclay vessel closed at one end, and fixed in such a manner that

its back and sides can be surrounded by the flame. It is perforated at the sides with a number of holes through which the air is drawn, the gaseous products passing into the flue. Fig 8 shows a vertical section through a muffle furnace. A is the muffle, B B the fireplace, C the ashpit, D the door of the ashpit, fitted with a register for opening or closing. E the draught hole, F the flue leading to the chimney, G the movable damper to regulate the draught. A front view of the muffle opening is seen in figure 8A.



Figs. 8 and 8A.

§ 13. *Gas Furnaces.*

The use of gas instead of solid fuel has many advantages to recommend it, the chief drawback being the great cost where illuminating gas is burned, as such furnaces are useless for metallurgical purposes without a good pressure and a large supply pipe. When this is secured, the muffle type will be found very convenient for laboratory use; but the air furnace burning coke is preferable to a gas furnace for ordinary crucible work.

When coal gas is used as a source of heat, the combustion should be as complete as possible, so as to prevent smoke and to obtain the maximum temperature. This is secured by using a burner on the Bunsen principle, and surrounding the flame with a jacket of fireclay or other refractory and non-conducting substance.

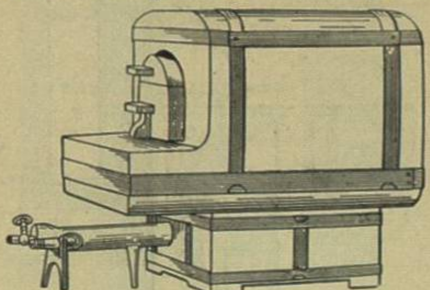


Fig. 9.

Perhaps the most successful worker in this direction is Mr. Thomas Fletcher of Warrington, several of whose furnaces have been in use in the Birmingham laboratory for some years.

An illustration of one of Fletcher's muffle furnaces is given in Fig. 9, which represents an external viewarranged for a "Blast" so as to produce a very elevated temperature. When worked by draught, the gas supply tap must be large and clear, so as to give as great a pressure of gas as possible at the burner nozzle. The india-rubber tubing must be smooth inside, the tubing made on wire not being suitable. If the gas supply is insufficient, the burner plate will become red hot. It is very important

that a light should be put to the burner before turning

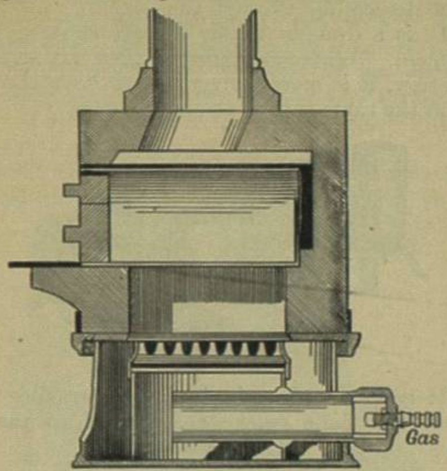


Fig. 10.

on the gas, or an explosion will take place which may

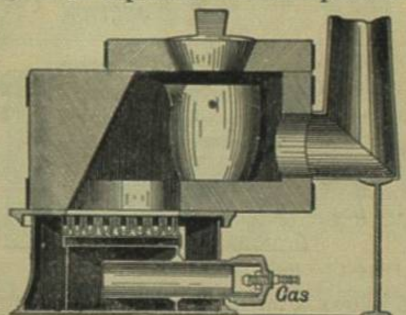


Fig. 11.

damage the parts. It is also advisable when lighting, to cover the air opening round the gas entrance, to prevent the flame descending through the gauze.

Fig. 10 is a draught muffle furnace showing internal arrangement. Where a number of these are arranged in a laboratory, it is more convenient to have the gas entrance on the same side as the muffle door.

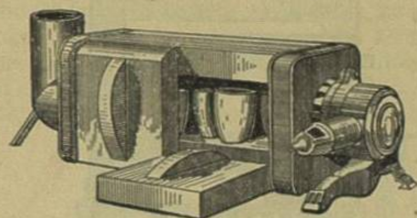


Fig. 12.

Fig. 11 represents Fletcher's draught crucible furnace to which the same remarks apply as to the muffle furnace.

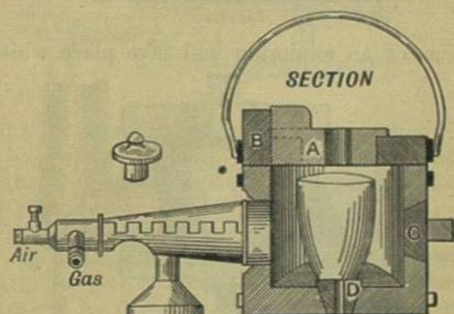


Fig. 13.

Fig. 12 shows Fletcher's reverberatory furnace for crucibles, muffles, cupels, etc. It is stated that one of these furnaces will do most of the work of an ordinary

laboratory. They work with a chimney draught to about the fusing point of silver, and with a blast, to that of cast iron. The furnace can be made to take two muffles, or one muffle and crucibles at the same time. The opening may be at the side or on the top. The burner is at one end out of the way of injury in the case of accident to a crucible, etc.

In special cases where it is desirable to raise metalliferous matter rapidly to a high temperature in a cru-

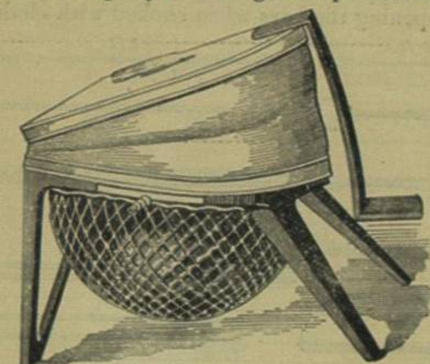


Fig. 14.

cible, Fletcher's injector furnace (Fig. 13) is very useful, the blast being obtained from the foot bellows (Fig. 14). With ½ inch gas pipe and the smallest foot blower, Mr. Fletcher claims for this furnace that a crucible full of cast steel may be melted in it in 7 minutes, tool steel in 12 minutes, and nickel in 22 minutes, beginning with all cold.

To adjust a new furnace to its highest power, put the nozzle of the burner tightly up against the hole in the side of the casing, turn on the full gas supply, light the gas in the furnace, and commence blowing before putting on the cover of furnace, with the airway full open.

If when the cover is replaced, the flame comes out of the hole in the cover about 2 inches, the adjustment is right. If the flame is longer, enlarge the hole in the air jet until the proper flame is obtained, or reduce the gas supply. Before stopping the blower, draw the burner back from the hole.

§ 14. *Furnace appliances.*

Stout iron pokers 3 feet long are used for stirring the fire and opening the bars when choked with clinkers and

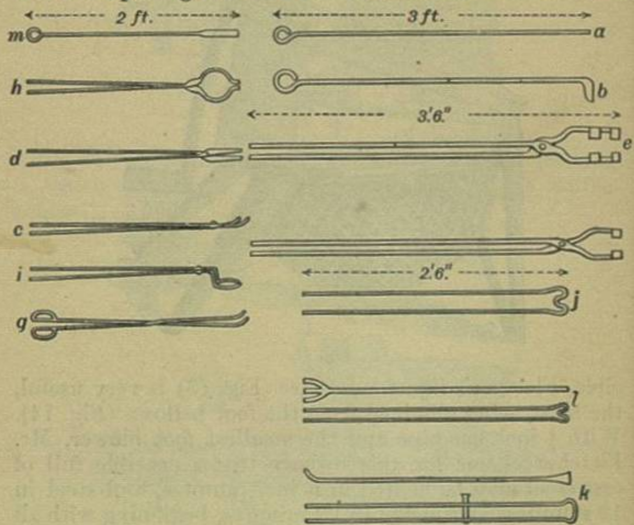


Fig. 15.

ashes (Fig. 15 *a*). For the purpose of cleaning the bars from underneath a poker bent at the end is advisable (Fig. 15 *b*). Each furnace should be provided with a pair of tongs having bent jaws (Fig. 15 *c*), and one pair with straight jaws (Fig. 15 *d*). For large crucibles the

basket tongs (Fig. 15 *e* and *f*) are used. The scissor tongs (Fig. 15 *g*) will often be found useful for packing coke round a pot. The forms (Fig. 15 *h* and *i*) are also occasionally required.

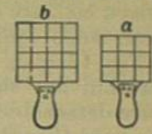
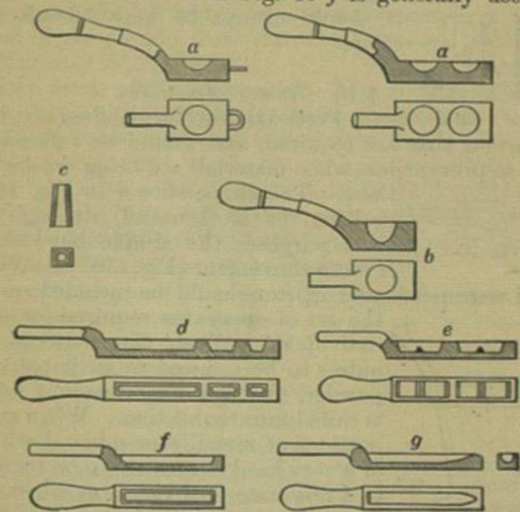


Fig. 16 *a* and *b*.

For use with a muffle the shape Fig. 15 *k* is employed, and those in Fig. 15 *l* for introducing and removing scorifiers. Another form shown in Fig. 15 *j* is generally used for



Scale of 1 Foot

Fig. 17 *a*-*g*.

manipulating cupels. Fig. 15 *m* represents a stirring rod used in roasting operations.

For holding cupels in carrying to or from the muffle, sheet iron trays divided into nine or sixteen compartments are most useful (Fig. 16 *a* and *b*).

Moulds for receiving the contents of crucibles, scorifiers, etc., are of various shapes. Some are hemispherical, some conical, some pyramidal, others rectangular (Fig. 17 *a–g*). When the metal requires to be cast in a thin flat sheet, a closed mould, the parts of which are detachable, and can be regulated for different sizes, is used (Fig. 17 *h* and *i*).

Fig. 18 represents a copper scoop with wooden handle used for charging crucibles; it should always be kept smooth and clean.

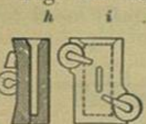


Fig. 17 *h* and *i*.

§ 15. *General Apparatus.*

(*a*) Pestles and mortars made of cast-iron of various sizes are required, and should be sufficiently deep to prevent loss when materials are being crushed in them. The shape shown in Fig. 19 is perhaps the most useful, although for some purposes the simple bowl shape is more convenient (Fig. 20). A Wedgwood ware pestle and mortar should be included among the set of apparatus required for each individual. When the material requires to be reduced to an impalpable powder, the agate mortar and pestle is called into requisition. When small portions of metallic or mineral matter of a very hard nature are to be reduced to a fine state of division, or when it is desirable that no portion should be lost, a steel mortar of the shape shown in Fig. 21 is most useful. *a* is the pestle which fits perfectly into the cylinder *b* which has a rim around the bottom outer edge by which means it is held firm by the cap *c* containing a hole whose diameter slightly exceeds the outside diameter of the cylinder which it encloses. This cap


Fig. 18.


Fig. 19.

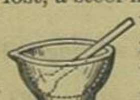


Fig. 20.

screws on to the base *d*, which is made of hardened steel, surrounded by a gun-metal rim screwed to fit the cap *c*.

(*b*) The operation of sifting is advantageous in ensuring that uniform fineness of the particles of a substance which is so essential to effect the solution of difficultly soluble bodies. This sameness of size also causes regularity in working, all portions being acted upon to the same degree when fused in a crucible with various fluxes. Also by the aid of the sieve, a rough mechanical separation of the gangue from the metalliferous matter may be effected

Fig. 21.

Two kinds of sieves are used. The open sieve is a wooden cylinder with a meshwork of brass wire gauze stretched over one end (Fig. 22). In the coarser ones iron wire is used. The box sieve is used when it is desirable to prevent loss. It consists of three parts: the sieve, a cover which fits tightly on the top, and a similar receptacle for receiving the sifted powder.

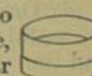


Fig. 22.

(*c*) *Washing and Vanning.*—Many ores contain small quantities of mineral matter associated with a large amount of earthy matter, such as tin stone, native gold, silver, etc., and when such materials are crushed to powder, the lighter portions may be washed away by running water, or made to float to the top when agitated in a vessel with water; the different constituents arranging themselves according to their specific gravities provided all the particles are of uniform size. For the purposes of the assayer, an iron

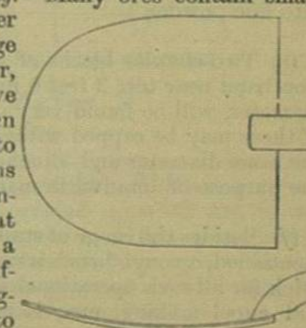


Fig. 23.

vanning shovel, or copper ladle renders useful service (Figs. 23 and 24). The powdered material is mixed with water, and a circular shaking motion is given to the tool, causing the separation mentioned above.

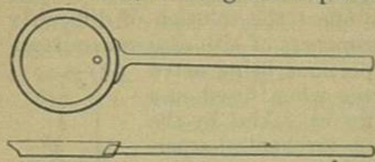


Fig. 24.

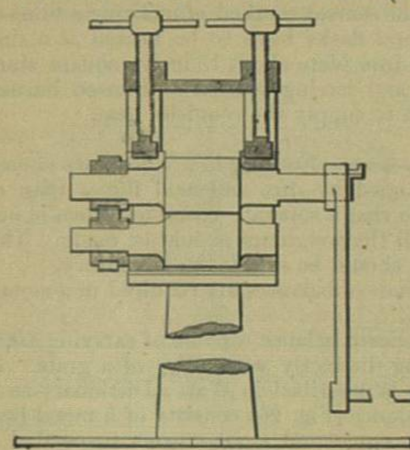
The excess of water is then poured off, and the same motion imparted with the addition of a peculiar twist at each revolution, by which the heavier portions are thrown to the outer edge at the upper end, while the lighter non-metallic portions flow to the front and may occasionally be washed away by fresh water being added from a tap.

(*d*) One or two strong rectangular boxes, 13″ × 9″ × 9″, are useful for granulating easily fusible metals such as lead and tin. The inside is blackleaded, and the metal, when just on the point of solidifying, is poured in and subjected to a vigorous shaking, by which means it is broken up into fine powder.

(*e*) Two circular blocks of some hard wood having an iron band near top, 2 feet 6 inches high, and 16 inches in diameter, will be found very useful for odd work. One of these may be capped with a circular block of iron of the same diameter and 3 inches thick, which will answer the purpose of an anvil in many cases.

(*f*) Besides the range of students' work-benches already mentioned, a rough bench with a stout top about 3 inches thick for all such operations as pounding, sifting, crucible and cupel making, etc., is necessary. To one of the supports should be fixed a strong vice for general use. A pair of large vice shears for cutting thick pieces of metal is also indispensable.

(*g*) A Rolling Mill of the ordinary jeweller's kind is required for rolling out metals, such as lead for cupellation purposes, or metals from which a portion has to be taken for analysis. A pair of 2-inch rolls will be found a convenient size, connected with handles and fixed on an iron or wooden stand bolted to the floor (Fig. 25).



Scale of 1 Foot

Fig. 25.

(*h*) A steel anvil should be fixed to a small but stout support for flattening buttons of gold and silver previous to rolling. This must be covered up when not in use and kept bright. In connection with this a hammer is used, weighing about 7 lbs., with a slightly convex face and rounded edges, to be also kept smooth and bright by enclosing in wash-leather when not in use.

§ 16. A fume chamber should be provided for every six students engaged in wet assaying, with a ring gas burner at the origin of the flue pipe to create sufficient draught to remove obnoxious fumes. In the Birmingham laboratory the pipes from the different chambers are connected with an annular space (Fig. 6) which envelopes the central stack through which the products of combustion from the furnaces pass.

A very convenient method of utilizing a fume chamber where several flasks have to be heated at a time, is to provide an iron plate about 18 inches square, standing on four legs, and having two large Bunsen burners fixed underneath to supply the requisite heat.

§ 17. *Balances.*—Nothing in a laboratory is more liable to be deranged by dirt and acid fumes than chemical balances, so that a separate room for them is necessary, in which all the weighings should be made. This room, if possible, should be *outside* the laboratory.

Three kinds of balances are required in a metallurgical laboratory.

(1) A delicate balance capable of carrying 1000 grains and turning distinctly with $\frac{1}{1000}$ of a grain. Such an instrument is described in Watt's Dictionary as follows:

"The balance (Fig. 26) consists of a metal beam with two almost equal and similar arms suspended near its centre on a pivot, the weights to be compared are also suspended from two pivots at nearly equal distances from the central pivot. In its most perfect form it consists of a perforated brass beam cast in a single piece, combining great strength and perfect inflexibility, with comparatively small weight. It is suspended at the centre on a knife edge of agate about an inch long, and turns on a single polished plane of agate fixed on a projecting brass support, which enters a perforation of the beam and does not impede its motion. The agate knife edge is firmly embedded in a wedge-shaped piece of brass, and being once adjusted at right angles to the plane of the beam, is then permanently fixed. At each end of the

beam is a smaller agate prism with its edge uppermost, fixed in a brass setting which is capable of a little lateral movement, but slides on a brass plane in such a manner that the two extreme edges and the centre edge are all appreciably in one plane. The extreme edges may be moved to and from the centre edge by little adjusting screws, and fixed in the desired position by two clamping screws."

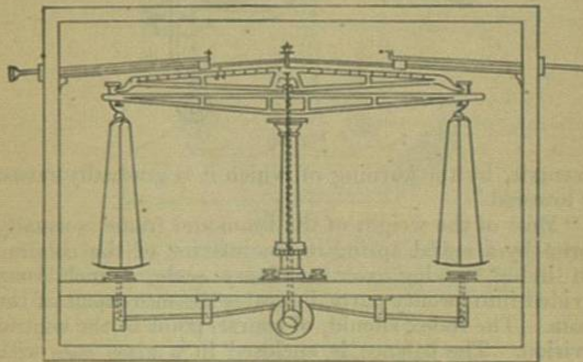


Fig. 26.

"Upon these extreme edges are balanced two agate planes from which by the bent wire, and a series of hooks and light wires, the pans are suspended."

"Except, however, when a weighing is being actually made, the agate planes and edges are never in contact, but the beam and pan suspensions are supported by a frame or movement having in the centre two Y's which catch projecting pins close to the centre edge, and lift the beam about $\frac{1}{100}$ of an inch off the plane, while steel points (shown in dotted outline, Fig. 27) entering hollows in the lower surface of the pan suspensions likewise raise these planes off the edges and retain them in exact positions for the new experiment. The movement of the brass frame is governed by a rod descending through the pillar of the balance and resting on a single

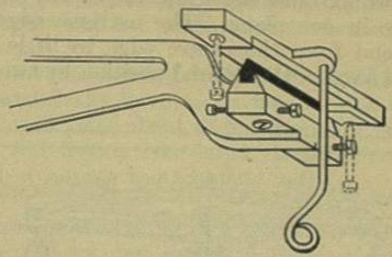


Fig. 27.

eccentric, by the turning of which it is gradually raised or lowered."

"Most of the weight of the beam and frame is usually borne by a spiral spring in the interior of the column. An index moving over an ivory scale, 1 inch long, divided into twenty parts, indicates the movement of the beam. The index should, of course, point to the central division. The balance is enclosed in a glass case with convenient windows. Two spirit levels, or a circular level and levelling screws are attached, by which the whole instrument must be adjusted to horizontality. Above the centre of the beam is a small weight called the gravity bob, which being screwed up or down regulates the stability of the balance, while a small vane being turned to the right or left adjusts the beam to equilibrium. In the figure, too, will be seen an arrangement of rods by which a small rider weight may be placed on any part of the beam, the balance case remaining closed."

(2) A rough assay balance, which should take 1000 grains in each pan, and turn with $\frac{1}{10}$ of a grain, suitable for elementary work, is shown in Fig. 28. The

pillar is fixed on a mahogany stand having a drawer containing the weights to be used from 1000 grains to $\frac{1}{10}$ of a grain. It will be more convenient and durable if the pans are of brass and movable.

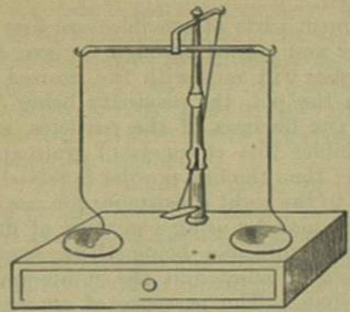


Fig. 28.

(3) A balance similar to the above for heavier weights capable of carrying 7 lbs. and turning readily with a grain, is also necessary.

REFRACTORY MATERIALS AND VESSELS.

§ 18. *Crucibles, etc.*—(*a*) Earthen crucibles are made of fire-clay mixed with sand, burnt clay, or other infusible matter, so as to counteract the tendency which raw clay possesses of shrinking when heated. The bodies thus mixed with the clay expand, or do not contract on heating, having been already shrunk when burnt, and therefore act in an opposite manner to the clay. Such a composition must be able to resist a high temperature without softening, must not be friable when hot, and be capable of withstanding sudden changes of temperature without cracking, as, for example, when a white hot crucible is brought out of a furnace into cold air.

Some crucibles are required to resist the corrosive action of metallic oxides in the material operated upon, and in the ashes of the fuel, so that a crucible should be selected which is best adapted to the special purpose to which it is to be applied.

The component parts of crucibles are first crushed to a fine powder and passed through a sieve, the fineness of whose meshes will vary with the desired fineness of the grain in the pot, the plasticity being closely connected with the fineness of the particles, at any rate for small crucibles this closeness of grain appears to be indispensable; then the fine powder is mixed with water and kneaded to the right consistence for use. The best results are obtained by using a mixture of different fireclays, the most infusible being those containing the largest amount of silica and the minimum of oxides of iron and lime. The presence of potash or soda in small quantity sensibly increases the fusibility, but they act advantageously in soldering the particles together. Iron pyrites, which is frequently disseminated through clays, especially those from the coal measures, is perhaps the most injurious constituent. A crucible made from such clay will become indented with small cavities, and even holes, when exposed to a prolonged high temperature. It follows then that the most refractory crucibles are those made from pure clays, the nearest approach to which is presented by some French clays.

(*b*) The fitness of a clay for making crucibles may be determined by moulding a portion into the shape of a prism or any form containing sharp edges, carefully drying, baking, and exposing to a high temperature in a covered crucible for some time. If very refractory, the test will show no signs of fusion. If the edges are rounded it is a proof of incipient fusion, and if melted, the clay is useless.

(*c*) Clay vessels of all kinds may be tested to ascertain their power of resisting corrosion by melting in them a

mixture of litharge, red oxide of copper, and borax, and noticing the time this mixture will take to permeate them. Those which resist this destructive action the longest will of course be the best. Most crucibles are by this means eaten away irregularly, showing the necessity of uniformity of grain to resist perforation.

(*d*) All crucibles should be cautiously annealed before use by placing them in an inverted position over the furnace, otherwise they are liable to split when plunged into a red hot fire. I have noticed this tendency with the best plumbago crucibles.

(*e*) Small crucibles may be made by hand in the laboratory by using a mould as shown in accompanying Fig. 29. "*a* is a short, hollow, slightly conical piece, open at both ends, made of gun metal; *bb* are short pins of iron inserted one on each side of *a*, near the bottom or narrower end; *c* is a round block of wood, in the centre of which, on the upper surface, is a circular cavity large enough to receive the lower end of *a*, including the projecting pins *bb*; through the middle of this cavity is a hole *h*, and upon the bottom lies a disc of gun metal, as seen in Fig. 29, which also has a hole in the centre, of the same size as *h*; around the edge of this cavity is screwed a flat ring of brass *dd*, which projects inwards to the extent shown by the dotted line (Fig. 30); *cc* are notches to allow the pins, *bb*, to pass through, so that *a* may be turned in the position seen in Fig. 30, when the pins, *bb*, will hold it firmly under the projecting edge of the brass ring; *f* is a wooden circular plug, in the bottom of which is fixed the iron pin *g*; by means of this pin the plug is kept upright exactly in the centre of

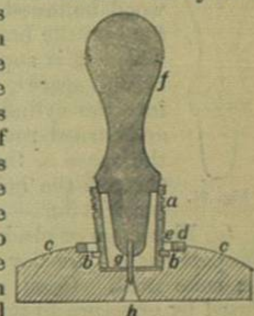
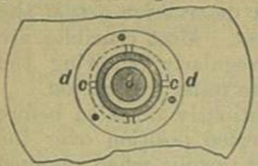


Fig. 29.

the mould. Fig. 31 is another wooden plug without a pin, but having an oblique groove *i*; the inner surface of *a* is very slightly oiled, and also the outer surface of *f* where it passes into *a*—*a* being adjusted as shown in Fig. 30. A small lump of well-tempered clay is put into *a*, when the plug *f* is forced down and turned round; the excess of clay escapes from the upper edge of *a*; *f* is now withdrawn and a small bit of clay dropped in, when *f'* (Fig. 31) is forced down and turned round and round, the excess of clay escaping by the groove *i*; *f'* is then taken out and *a* with the contained crucible detached."



Scale of 6 Inches

Fig. 30.

(*f*) "The covers are made with the mould (Fig. 32). *a* is a cylindrical piece of wood upon which is placed a cylinder of brass *bb*, perforated with several holes *cc*, etc. *d*, a cylindrical plug of wood hollowed out at the bottom. This plug fits into the brass cylinder *bb*, upon the top of which it is supported by a shoulder, so as to leave a space *e*. A small lump of clay being put into the cylinder, the plug *d* is pressed down and turned round, when a cover is moulded in the space *e*, the excess of clay being expelled through the holes *cc*, etc." (Percy's *Metallurgy*, pp. 228-9.)

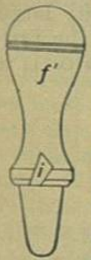
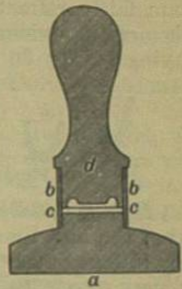


Fig. 31.

(*g*) Plumbago or black lead pots are made from varying proportions of fire-clay mixed with powdered graphite or coke dust. Good graphite is neither altered nor fused by exposure to the highest temperatures (air being absent), so that it is an admirable substance for crucibles. The graphite is powdered, sifted, and mixed with sufficient clay to render it plastic. Good plumbago crucibles, after a careful preliminary annealing, withstand the

greatest changes of temperature without cracking, and may be used many times in succession.

(*h*) When an ordinary crucible requires to be protected from the corrosive action of metallic oxides, or when small amounts of metallic compounds have to be reduced, the inside is coated with a lining of charcoal. This is done by first mixing the charcoal with sufficient starch paste, or treacle to make it adhere when pressed. The crucible is then loosely filled with the brasque, and a cavity of the desired size made by boring with a triangular-shaped piece of wood, and then made smooth with a round elongated wooden tool, whose size and shape is apportioned to the capacity of the cavity desired. (Fig. 33 *a*, *b*.) The brasqued crucible is shown in Fig. 34.



Scale of 6 Inches

Fig. 32.

The atmosphere in such a crucible will always be reducing at an elevated temperature, and in some cases a compound may be reduced without any additional carbon.

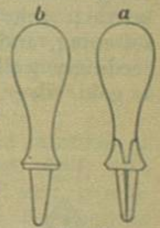


Fig. 33.

§ 19. The crucibles used in a metallurgical laboratory may be conveniently classified under the following heads:—

Fig. 34.

(*a*) *London Crucibles*.—These are round in shape, of a reddish brown colour, close in grain, but liable to crack with sudden changes of temperature. They resist the corrosive action of metallic oxides remarkably well. (Fig. 35 *a*–*d*.)

(*b*) *Cornish Crucibles.*—These are also round and used in two sizes; they are course in grain and of a greyish white colour, fairly refractory, but very liable to be corroded with metallic oxides. They are chiefly used in copper assaying. (Fig. 36 *a, b.*)

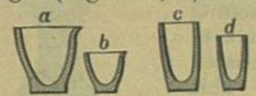


Fig. 35.

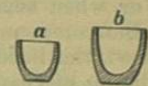


Fig. 36.

(*c*) *Hessian Crucibles.*—This kind is triangular in shape, so that the metal may be readily poured from each edge, fairly refractory, not very liable to corrosion with oxides, but very prone to crack with sudden changes of temperature, and to break when held with the tongs. (Fig. 37.)·

(*d*) *French Crucibles.*—The quality of these is excellent, being smooth and carefully made, though somewhat brittle; they withstand high temperatures without softening, and resist corrosion by molten litharge exceedingly well. They are used in melting such metals as gold, silver, copper, etc. (Fig. 38 *a, b.*)

Fig. 37.

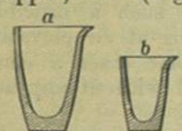


Fig. 38.

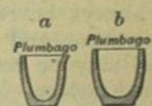


Fig. 39.

(*e*) *Plumbago Crucibles.*—These pots resist sudden changes of temperature, are highly refractory, and the least corroded by metallic oxides, but they are inconvenient when a reducing atmosphere is not desired. (Fig. 39 *a, b.*) To enable them better to withstand great changes of temperature, they are coated with a mixture of borax and lime, dried, and then another coating of slaked lime and linseed oil is brushed on.

(*f*) *Iron Crucibles.*—These are employed in assaying certain selenides and sulphides, such as galena, etc.

(*g*) *Lime Crucibles.*—Deville first introduced the use of crucibles cut out of solid blocks of pure lime, so as to avoid the introduction of carbon and silicon into metals during fusion, with very satisfactory results, the bodies melted therein, such as manganese, iron, nickel, etc., being purer, more malleable, and more ductile than when fused in clay crucibles. As large blocks of pure lime are difficult to obtain, Mr. David Forbes introduced the following plan :—

A clay crucible of somewhat larger dimensions than the desired lime one is filled with lamp black and well pressed down. The central portion is then removed with a knife, leaving a thin lining firmly adhering to the sides, which is then rubbed smooth with a glass rod. The hole is then filled with finely powdered lime, and a cavity made by cutting away the inner portion as before, leaving a shell of lime which becomes firm and compact after heating. The layer of carbon prevents it acting on the outer crucible.

(*h*) *Alumina Crucibles.*—Pure alumina is infusible at the highest furnace temperatures, will not unite with bases to form readily fusible compounds, and is eminently adapted for lining crucibles used for melting metals. But pure alumina is rare, so that other means have been devised for obtaining it. The following method is described in Mitchell's Assaying, page 123:—"Ammonia alum is ignited at a white heat, when it leaves alumina in a dense compact form: this is to be finely powdered. To a solution of another portion of ammonia alum in water, ammonia is added, when alumina is precipitated in a gelatinous state: this is washed till free from sulphate of ammonia. The dense alumina is then mixed with water and worked up into a paste, the precipitated gelatinous alumina being kneaded in from time to time; this gives coherency, and when sufficient has been added (which may be ascertained experimentally) the mass may be moulded into shape. Such crucibles require careful annealing, and are infusible at the highest furnace temperatures."

(*i*) *Platinum Crucibles.*—These crucibles are indispensable in wet assaying where speed and accuracy are desired, yet they require great care in using, as many substances will corrode and otherwise injure them. They cannot be used for any substances containing easily reducible oxides, such as those of silver, lead, etc. Many oxides, sulphides, and phosphides also affect them, as does the naked flame of a Bunsen burner. They should be heated preferably on a platinum triangle, although pipe-clay is a good substitute. Brass and iron tongs, unless perfectly clean, will leave a dark stain where they touch when red hot, so that the tongs should be capped with platinum.

Platinum crucibles should be cleaned after each operation by scouring them with wet sea sand and a little dilute hydrochloric acid. Stains may often be removed by heating with acid sulphate of potassium or borax, then washing and scouring with sea sand, the particles of which, not being sharp, do not scratch like ordinary sand.

§ 20. *Roasting dishes* are small shallow vessels made of fire-clay, used for roasting or calcining ores, regulus, speise, etc., also for oxidising metals in a muffle. Fig. 40.


Fig. 40.

§ 21. *Scorifiers* are small cup-like fire-clay vessels of an acid character, the silica of which readily unites with metallic oxides forming fusible scoriae. These vessels are largely employed for slagging-off metals like tin and antimony, in silver and gold ores, and compounds which cannot be perfectly removed by direct cupellation. Fig. 41.

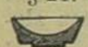


Fig 41.

§ 22. *Cupels* are vessels somewhat resembling scorifiers in shape, made of boneash, which has the property of absorbing molten oxide of lead; the latter dissolves other oxides which may be present, carrying them with it into the pores of the cupel. Base metals may thus be separated from the unoxidisable metals by a prolonged heating of the alloy with an excess of lead in the cavity of a cupel. Fig. 42 *a–c*.

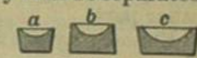


Fig. 42 *a–c*.

To make cupels the boneash must be in fine powder, otherwise it requires to be passed through the meshes of a fine sieve to remove any lumps. It is then moistened with sufficient water to make it adhere firmly when tightly pressed in the hand.

The apparatus in which the cupels are made consists of two parts. The mould in which the boneash is placed and the plug which produces the desired cavity and presses the boneash into a compact vessel. The latter is removed, dried, and is then ready for use. Fig. 43 *a*, *b*.

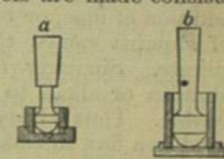


Fig. 43 *a* and *b*.

The wood tray (Fig. 44) is used for mixing the boneash, and the iron tray (Fig. 45) for holding cupels while drying.


Fig. 44.


Fig. 45.