A handbook of chemical manipulation

Williams, Charles Greville

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Section IV. Blowpipe Apparatus

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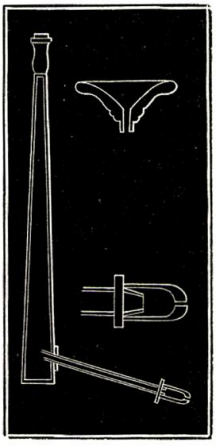
SECTION IV.

BLOWPIPE APPARATUS.

48. This little instrument has become absolutely indispensable to the chemist; perhaps there is no single appliance made use of in the science which has so wide a field of usefulness; its value is not only found in mineral, but also in organic research; in fact so extended has the subject of blowpipe manipulation become, that a separate treatise would be necessary were we to enter upon it at anything like the length it undoubtedly deserves. Of all the manuals extant which treat of the blowpipe, that of Professor Plattner is the most elaborate and useful, so much so that a chemist could scarcely be said to be a good blowpipe analyst unless tolerably familiar with its contents; the treatises of Berzelius and Children may also be consulted. Under these circumstances it will not be necessary to enter at any great length upon the subject, but the following paragraphs may at least serve as an introduction to the more extended works alluded to. The philosophy of a candle-flame is familiar to all who have studied elementary chemistry, and is undoubtedly foreign to a work on manipulation; we shall therefore proceed to describe the blowpipe itself. The different fancies of chemists have produced a considerable number of them, each, possibly, having some particular merit adapted to the circumstances under which it was invented; however, out of all these there are but two or three in general use.

Fig. 34.



It will be found, that for all purposes for which a mouth blowpipe is applicable, that of Black is the best and cheapest. It consists of a tube of tin plate (fig. 34) about 7 inches long, $\frac{3}{4}$ inch broad, tapering to a quarter, where a small mouthpiece is soldered, to increase the operator's command over

the instrument. At the wider end is inserted a small cylindrical tube of brass, about 2 inches long, supporting the nozzle, which may be of brass or platinum. The tube is slightly conical at the end where the jet is fixed, and the latter is thus made to fit on without a screw, which would soon be injured by the high temperature to which it is exposed, and render it difficult to be removed for the purpose of cleaning. It is absolutely necessary to a good blowpipe that the nozzle should be drilled from a solid piece of metal, in the form represented on a larger scale in the section fig. 34. One of the chief merits of Black's instrument, is the efficient manner in which it condenses and retains the water derived from the breath, and renders its ejection upon the heated assay impossible.

The moveable trumpet-shaped mouthpiece may sometimes be used with advantage in long and fatiguing operations; it is, however, seldom necessary.

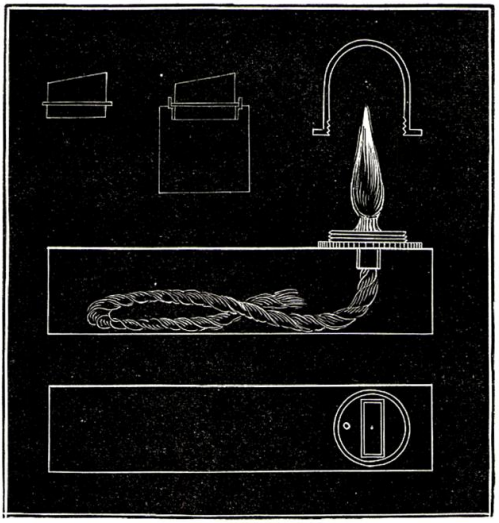
Wollaston's blowpipe consists of three tubes, capable of being connected or of being taken asunder and then packed one inside the other.

Cronstedt, who was perhaps the first who endeavoured to prevent the moisture of the breath from reaching the assay, contrived an instrument having a bulb in the middle capable of being opened in order to remove the condensed moisture. It is an expensive, although far from convenient form, and if, by any chance, held vertically, the moisture escapes.

The lamp generally used with the blowpipe is of very simple construction. The wick, which is about an inch long by one-eighth wide, stands $\frac{1}{4}$ inch above the holder, fig. 35. A cover is made to screw over it, to prevent dust from accumulating on the wick. The construction of the wick-holder will be seen by reference to the engraving, and the cotton is to be cut parallel to it, the lower end being away from the operator while blowing. The air is propelled along the wick in the direction of its greater axis. To a person of moderate dexterity, the manipulations connected with ordinary blowpipe analysis, will prove very easy, but considerable experience will be necessary to enable the student to detect in complex mixtures, substances whose reactions are not

very decided. Nevertheless, the value of the instrument is so great, and so many otherwise troublesome problems in qualitative analyses may be rapidly and decisively solved by it, that no

Fig. 35.



labour should be spared to acquire facility in its management. It may be mentioned, that the blowpipe is not intended entirely to supersede the humid method of analysis; it is rather to be considered as an adjunct; for instance, we will suppose a piece of type-metal to be under examination: its brittleness will at once lead to a suspicion of the presence of antimony or bismuth, but it is wished to ascertain which of these, and what other metal may be present. It is heated with nitric acid until converted into a white powder, evaporated to a small bulk, water added, and the liquid filtered; the precipitate on the filter is washed and heated with carbonate of soda on charcoal in what is termed the reducing flame; a brittle button of metal is obtained, which may be further examined on a fresh piece of charcoal: its colour is almost a pure white; we therefore are led to believe in the absence of bismuth, which has a pinkish tinge; but, to be certain, we heat it in the oxida-

ting flame; white fumes are given off, which, when condensed, have no tint of red or orange; we therefore conclude the absence of bismuth. The solution may now be examined: sulphuric acid is found to produce a precipitate; it is therefore added in excess, and the precipitate filtered off, washed and reduced in the same manner as the last; a malleable globule is obtained, conclusive of the presence of lead. In fact, the precipitation by sulphuric acid is sufficient, but it is always satisfactory to obtain the pure metals where it can be done so easily. The first globule of antimony may, moreover, be examined for tin or arsenic if necessary.

49. The above sketch is not given as the best mode of analysing the alloy alluded to, but merely as the first case that suggested itself, of a real experiment, where the agency of the blowpipe was made use of to obtain a rapid result. There are many metals whose reactions before the blowpipe are so marked that their presence may be ascertained almost instantly, while by the ordinary course of analysis, a long and troublesome series of operations are necessary before a decided answer can be given; among these may be mentioned manganese, arsenic, iron, chromium, cobalt, tin, titanium, and several others.

50. The first thing to be acquired in blowpipe practice, is the power of keeping up a continuous blast or current of air for some time; to effect this, the enlarged orifice of the instrument is to be placed between the lips, and the mouth filled with air until the cheeks become distended like a trumpeter's; it will soon be found, that, by making a motion with the tongue, somewhat like that which is done in uttering the syllable "tut," a fresh supply can be given as often as may be required; during this time the breathing is carried on by the nostrils.

51. The next step is to produce either the oxidizing or reducing flame at will; and by attending to the following instructions, the operator cannot fail to succeed after a little experience. The oxidation flame is generally considered easier to obtain than the reducing; we will therefore commence with a description of the method of producing it. The lamp being trimmed, so that the wick is parallel with the holder, and quite free from any strag-

gling fibres, is to be lit, and the wick so adjusted that the flame may be clear and brilliant; if we now take a blowpipe with a moderate-sized orifice, and hold it in such a position that the jet shall be just inside the flame, and about the tenth of an inch above the wick, the flame will not only be deflected, but considerably altered in its characters; two parts being more especially conspicuous, viz. an internal blue cone, and a yellow halo-like flame at the end. It is in this exterior yellow part that the power of oxidization resides; and, if a globule of tin about the size of a mustard seed, be placed in it for a short time, it will swell, and finally become converted into a white earthy-looking matter, the peroxide of tin. If, on the contrary, we use an instrument with the aperture rather less than in the last experiment, and withdraw the jet a little, so that it may be on the outside of the flame, and then blow rather powerfully, we shall find that, although deflected, the flame is less altered in its general characters than in the previous case, the chief part of it consisting of a large and highly luminous cone, containing a considerable portion of strongly ignited carbon, precisely in the condition most adapted for assimilating oxygen, and becoming converted into carbonic acid; and if we expose to it, upon charcoal, the cauliflower-looking mass of peroxide of tin which we obtained in the first experiment, we shall find that the highly ignited carbon will combine with its oxygen, and it will be reduced to the metallic state. To reduce peroxide of tin on charcoal without the assistance of a flux, is a test of moderate proficiency in the use of the instrument.

52. *Supports for Substances before the Blowpipe.*—Having acquired facility in the two operations which are in request every time the blowpipe is used, we turn our attention to the supports necessary when an assay is to be exposed to the flame. The three substances most used for this purpose are charcoal, platinum, and clay.

The first is generally resorted to when a deoxidizing power is required, although, in certain cases, it is admissible where oxidation is to be effected; the two latter are employed if a substance is to

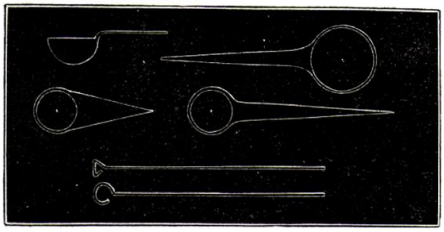
be brought to a higher state of oxidation, or is merely to be fused. Good pine charcoal is, perhaps, the best that can be used, but the laboratory charcoal-box generally contains pieces which by a little use of the saw may be made fit for blowpipe experiments. It must be remembered that the sides only should be used, and that pieces which have bark on them should be carefully avoided, as they emit showers of sparks which scintillate and fly about in a very annoying and even dangerous manner.

53. Platinum is used as a support in three different states, namely, as wire, foil, or small spoons. The first of these is represented in figs. 40 and 41, bent into the shapes in which it is

Fig. 36. Fig. 37.

Fig. 38. Fig. 39.

Fig. 40. Fig. 41.

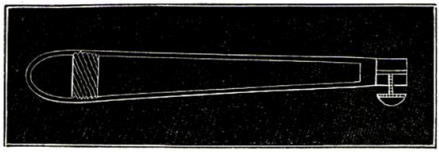


generally used to support a bead of borax or microcosmic salt to which a small portion of the assay is to be added. The whole is then to be exposed, first to the reducing and then to the oxidating flame; the changes of colour produced in this manner, generally afford a clue to the class of substances to which the assay belongs, and frequently enable us to pronounce with certainty as to the presence of one and sometimes several of its ingredients.

54. Platinum spoons are employed in blowpipe experiments, to contain mixtures of siliceous minerals and fluxes, where it is intended to bring a substance insoluble *per se* into a condition to be acted on by solvents in cases where the dry method alone is insufficient to enable us to ascertain with certainty the composition of the assay. They may be made of the sizes and shapes of those represented in figs. 36, 37, 38, and 39; the handle may either be held with a pair of pincers, during exposure to the flame, or be fixed in a support of wood with a hollow brass fe-

rule, through which a screw is passed to fix the platinum handle, as in fig. 42. It is likewise well adapted to hold the platinum

Fig. 42.



wires, and is made hollow in order to contain them when not in use. A very convenient support for platinum wires is the small ivory instrument used by ladies for containing and fixing crochet-hooks. It is much like the holder in the engraving, but differs in the arrangement by which the hooks are grasped.

There are a few precautions necessary to be observed in using platinum vessels, which may with propriety be introduced in this place. For instance, if a reducible metallic oxide be heated with any substance capable of removing its oxygen (as oxide of lead with charcoal powder, or other organic matter), or, if a reducible oxide be exposed on the platinum wire to the reducing flame, the support is almost certain to be destroyed; such substances must, therefore, be heated on charcoal or clay supports. When a bead has been fused in the loop of a platinum wire, it should not be removed by mechanical means, but the wire should be thrown into a small vessel of water until the bead is dissolved. An exception to this rule occurs when a bead fused in the loop of a platinum wire is observed to be coloured before addition of the assay; in such case the bead must be sharply heated; and then, if the wire be dexterously jerked against the anvil, or any other hard or steady substance, the melted mass will fly off, and must be replaced by a fresh portion until a colourless bead is obtained. Where a silicate has been fused in a platinum spoon, and it is wished to remove the mass, it is generally admissible to boil it with hydrochloric or nitric acid, but we must carefully avoid the presence of both together, or, in fact, of any substances capable of generating chlorine; as the vessel would be more or less attacked, and thus cause a twofold injury, namely,

damage to the vessel and, what is sometimes more serious, introduce platinum into the analysis. Where a silicate has been fluxed in a platinum spoon with any of the mixtures to be found in analytical manuals, and difficulty is found in extracting the fused mass, the spoon should be suspended by a platinum wire near the top of a glass of water, and left for a few hours; it may then, in most cases, be removed without difficulty. If this procedure is insufficient, acids must be resorted to.

55. *Platinum foil.*—Platinum, in the state of thin sheets, is much less useful in blowpipe experiments than either spoons or wire; nevertheless, it may sometimes be employed with advantage, as in testing for manganese by fusion of the assay with soda, with or without the addition of nitre, according as the metal is more or less plentiful in the specimen.

In organic chemistry, platinum foil has many uses, especially for supporting salts, &c. in the flame of a lamp during combustion, in order to ascertain the presence of inorganic substances. It is also used to form the platinum trays used in organic analysis in the process by oxygen gas, to be alluded to further on. In cases where a platinum crucible is not at hand, an extempore one may be constructed, by folding a piece of the foil in the same manner as for a filter. The electro-negative element of Grove's battery is constructed of this metal in foil, and it usually forms the electrodes of batteries used in the decomposition of liquids, &c.

56. *Clay Supports.*—An excellent method of forming small basins and crucibles will be found in Plattner's treatise; it will be unnecessary, therefore, to describe it here; moreover, basins of extreme thinness, and in every way adapted for blowpipe experiments, are now imported from the Continent, and may be procured of the instrument-makers.

57. *Forceps.*—A pair of platinum-pointed forceps is indispen-

Fig. 43.

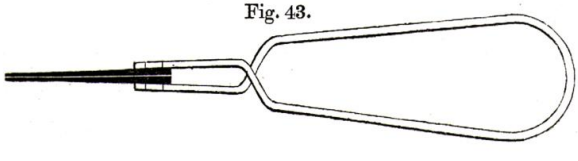
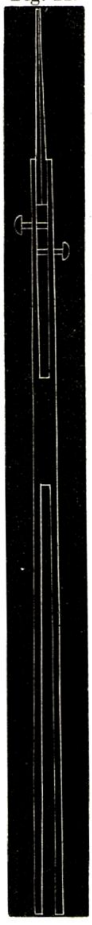


Fig. 45.

Fig. 44.



sable to the blowpipist; they are made of the various kinds represented in the woodcuts. The first, fig. 43, is merely steel wire bent in the form depicted, and having small beaks of platinum riveted on. The second form is more expensive, but far more convenient and strong; they are those usually used, and are made to open by pressing the knobs. Another pair, the invention of Mr. S. Highley, Jun., seen in section and plane, fig. 45, resembles the first, save that it only possesses one knob.

58. There are many other instruments used in blowpipe analysis, some of which will be found in their places among the apparatus used in the humid method; the greater part are chiefly intended for quantitative blowpipe operations, a method of research very little practised in this country, and not having sufficient advantages over the usual methods to be likely to supersede them.

59. *Self-acting Blowpipes.*—The principle upon which most of these instruments are constructed, is that of directing a jet of vapour of alcohol or wood-spirit through a flame, which latter is generally that which heats the spirit. In order to fulfil these conditions the vessels figured on the next page are employed; the first (fig. 46), generally known as the Russian blast-lamp, consists of a double case, *a a*, to hold spirit; on one side of the interior is fastened a tube reaching nearly to the top, and bent at the bottom, where it passes out of the double case and rises about half an inch above the bottom of the inner chamber; its end should have a

blowpipe-nozzle attached to it; *d* is an aperture to introduce spirit into the double case, which should not be filled to more than two-thirds of its capacity, otherwise a portion is liable to find its way into the exit-pipe during the time the lamp is being used, by which means a jet of ignited spirit is ejected, which may cause much inconvenience. It has a handle to facilitate its removal from place to place. During use, *d* is closed moderately tight with a cork, which acts as a safety-valve; if, therefore, the jet becomes choked, the cork is blown out and prevents the destruction of the lamp, which might be attended with serious consequences. When required to be used, spirit is, as we have said, introduced into *a a*, and a certain portion is placed in the inner chamber, *b*, so as to cover the bottom to the depth of half an inch. If the last-mentioned spirit is now ignited, that in the chamber *a a*, will soon enter into ebullition, and a stream of vapour will make its exit from *c*, and, immediately igniting, forms a powerful jet of flame capable of heating large platinum vessels to a brilliant red heat; the flame is, moreover, adapted for heating the German glass in making combustion-tubes, when no other form of blowpipe is at hand. The mode of using the second contrivance (fig. 47) is almost obvious from the figure: a hollow globe, *a*, half-filled with spirit, having a blowpipe, *b*, attached, is fixed above a lamp, *c*, opposite the wick of which the exit of the pipe is placed. On lighting the lamp, almost the same phenomena occur as in the Russian instrument. A cork safety-valve is inserted at *d*, the screw-cap being previously removed. When placed in circumstances in which a blast-lamp was not at hand, I found the

Fig. 46.

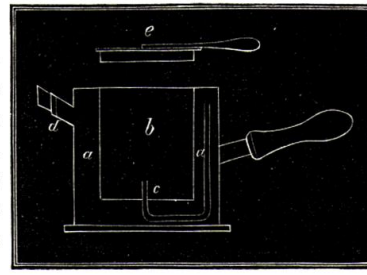
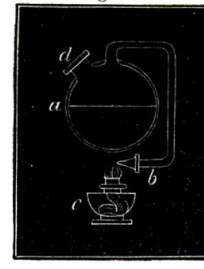


Fig. 47.



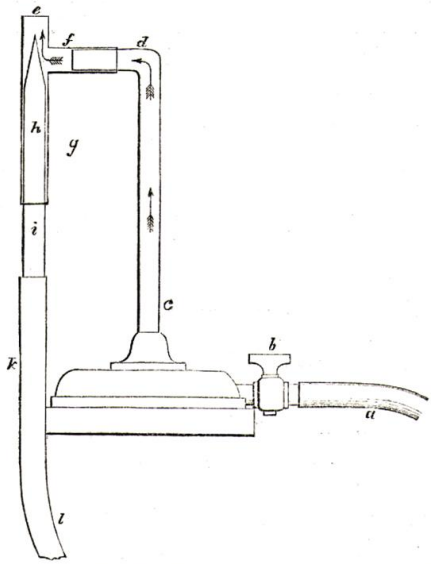
following a useful and powerful substitute. The body of the lamp is formed out of a tin bottle, and a hole pierced in the side has a small tube soldered in, to act, when fitted with a cork, as a safety-valve and a place to insert the spirit. A jeweller's blowpipe is cut in half, and the bent portion inserted vertically in the neck of the bottle, the nozzle of a Black's blowpipe being fixed at the aperture. It may be supported above a gas-burner or oil-lamp. A portion of lamp-cotton is placed in an evaporating basin, and held in its position by a bent wire, and the basin being filled with olive-oil, and the wick adjusted to the aperture, we have an apparatus simple in the extreme, and not easily surpassed in power: combustion-tubes may be easily prepared at such a lamp in a very short time, even when of large calibre and made of the most refractory glass.

60. *Table Blowpipes.*—Any laboratory where experiments are carried on with even moderate activity, must possess one of these pieces of apparatus. So many instruments of different kinds are constantly being made from glass, that no one who has ever had one at his command will willingly dispense with its services. One of the most convenient of these is the blowpipe contrived by Herapath; it is represented in fig. 48, where *a* is a flexible tube attached to a stopcock, *b*, which communicates with a tube, *c d*, bent at right angles at *d*, where a T-shaped tube, *e f g*, slips on by means of the piece *f*. The blowpipe jet, *h i*, passes into the longer arm of the T-piece, and fits somewhat tightly; *k l* is a second piece of flexible piping terminating in a mouthpiece. On turning on the gas, it passes in the direction marked by the arrows, and is to be inflamed at *e*. On blowing with the mouth, or by means of a pair of bellows, into the flexible tube, *k l*, the ignited gas takes the form of a blowpipe flame of great power, the nature of which is entirely under control by means of the stopcock, *b*, and also by regulating the quantity of air thrown in from the lungs or bellows. As the T-shaped piece is freely moveable at *f*, the jet may be directed to any position, and the apparatus becomes well adapted for the ignition of platinum crucibles, &c.*

* I have had one of these (made by Mr. E. G. Wood, of Cheapside, Lon-

It is mounted on a heavy foot, and being connected with the gas

Fig. 48.



supply by means of the flexible tube, *a*, it may be readily adjusted upon the laboratory table.

Blowpipes of other kinds are also procurable at the instrument makers, worked by bellows set in motion by the foot; the aperture for the exit of the air should end in a tube of about a quarter of an inch in diameter, to allow of jets formed from glass tube being adapted to it. They are accompanied by a gas-jet of rather peculiar form, intended to supersede the necessity of an oil-lamp. The water-pressure blowpipes of Tilley or Toft are also very effectual; they may be procured, with directions for use, from the makers of chemical instruments.

don) in use for some time, and have found it to answer so well, that I prefer it to any other I have yet used, excepting the bellows blowpipe, with the water-pressure arrangement.