PHILOSOPHICAL TRANSACTIONS.

I. The Bakerian Lecture.—*On a method of rendering Platina malleable.* *By* William Hyde Wollaston, *M.D. F.R.S. &c.*

Read November 20, 1828.

As, from long experience, I probably am better acquainted with the treatment of Platina, so as to render it perfectly malleable, than any other member of this Society, I will endeavour to describe, as briefly as is consistent with perspicuity, the processes which I put in practice for this purpose, during a series of years, without seeing any occasion to wish for further improvement.

The usual means of giving chemical purity to this metal, by solution in aqua regia and precipitation with sal ammoniac, are known to every chemist; but I doubt whether sufficient care is usually taken to avoid dissolving the Iridium contained in the ore, by due dilution of the solvent. In an account which I gave in the Philosophical Transactions for 1804, of a new metal, Rhodium, contained in crude platina, I have mentioned this precaution, but omitted to state to what degree the acids should be diluted. I now therefore recommend, that to every measure of the strongest muriatic acid employed, there be added an equal measure of water; and moreover, that the nitric acid used be what is called "single aquafortis;" as well for the sake of obtaining a purer result, as of economy in the purchase of nitric acid.

With regard to the proportions in which the acids are to be used, I may say, in round numbers, that muriatic acid, equivalent to 150 marble, together with nitric acid equivalent to 40 marble, will take 100 of crude platina; but in order to avoid waste of acid, and also to render the solution purer, there should be in the menstruum a redundance of 20 per cent at least of the ore.

The acids should be allowed to digest three or four days, with a heat which ought gradually to be raised. The solution, being then poured off, should be suffered to stand until a quantity of fine pulverulent ore of iridium, suspended in the liquid, has completely subsided; and should then be mixed with 41 parts of sal ammoniac, dissolved in about 5 times their weight of water. The first precipitate, which will thus be obtained, will weigh about 165 parts, and will yield about 66 parts of pure platina.

As the mother-liquor will still contain about 11 parts of platina, these, with some of the other metals yet held in solution, are to be recovered, by precipitation from the liquor with clean bars of iron, and the precipitate is to be redissolved in a proportionate quantity of aqua regia, similar in its composition to that above directed to be used: but in this case, before adding sal ammoniac, about 1 part by measure of strong muriatic acid should be mixed with 32 parts by measure of the nitro-muriatic solution, to prevent any precipitation of palladium or lead along with the ammonio-muriate of platina.

The yellow precipitate must be well washed, in order to free it from the various impurities which are known to be contained in the complicated ore in question; and must ultimately be well pressed, in order to remove the last remnant of the washings. It is next to be heated, with the utmost caution, in a black-lead pot, with so low a heat as just to expel the whole of the sal ammoniac, and to occasion the particles of platina to cohere as little as possible; for on this depends the ultimate ductility of the product.

The gray product of platina, when turned out of the crucible, if prepared with due caution, will be found lightly coherent, and must then be rubbed between the hands of the operator, in order to procure by the gentlest means, as much as can possibly be so obtained, of metallic powder, so fine as to pass through a fine lawn sieve. The coarser parts are then to be ground in a wooden bowl with a wooden pestle, but on no account with any harder material, capable of burnishing the particles of platina*; since every degree of burnishing will prevent the particles from cohering in the further stages of the process. Since the whole will require to be well washed in clean water, the

* The following experiment will prove the necessity of attending to this precaution:—if a wire of platina be divided with a sharp tool in a slanting direction, and, being then heated to redness, be struck upon an anvil with a hammer, so as to force into contact the two newly-divided surfaces, they will

operator, in the later stages of grinding, will find his work much facilitated by the addition of water, in order to remove the finer portions, as soon as they are sufficiently reduced to be suspended in it.

Those who would view this subject scientifically should here consider, that as platina cannot be fused by the utmost heat of our furnaces, and consequently cannot be freed like other metals, from its impurities, during igneous fusion, by fluxes, nor be rendered homogeneous by liquefaction, the mechanical diffusion through water should here be made to answer, as far as may be, the purposes of melting; in allowing earthy matters to come to the surface by their superior lightness, and in making the solvent powers of water effect, as far as possible, the purifying powers of borax and other fluxes in removing soluble oxides.

By repeated washing, shaking, and decanting, the finer parts of the gray powder of platina may be obtained as pure* as other metals are rendered by the various processes of ordinary metallurgy; and if now poured over, and allowed to subside in a clean basin, a uniform mud or pulp will be obtained, ready for the further process of casting.

The mould which I have used for casting, is a brass barrel, $6\frac{5}{4}$ inches long, turned rather taper within, with a view to facilitate the extraction of the ingot to be formed, being 1.12 inches in diameter at top, and 1.23 inches at a quarter of an inch from the bottom, and plugged at its larger extremity with a stopper of steel, that enters the barrel to the depth of a quarter of an inch. The inside of the mould being now well greased with a little lard, and the stopper being fitted tight into the barrel by surrounding it with blotting-paper, (for the paper facilitates the extraction of the stopper, and allows the escape of water during compression,) the barrel is to be set upright in a jug of water, and is itself to be filled with that fluid. It is next to be filled quite full with the mud of platina; which, subsiding to the bottom of the water, is sure to fill the barrel

become firmly welded together; but if the surfaces have previously been burnished with any hard substance, the welding will be effected, if at all, with very great difficulty.

When the powder of platina has been over-heated in decomposing the ammonio-muriate, or has been burnished in the grinding, I have in vain endeavoured to give it a welding surface, by steeping it in a solution of sal-ammoniac in nitric acid.

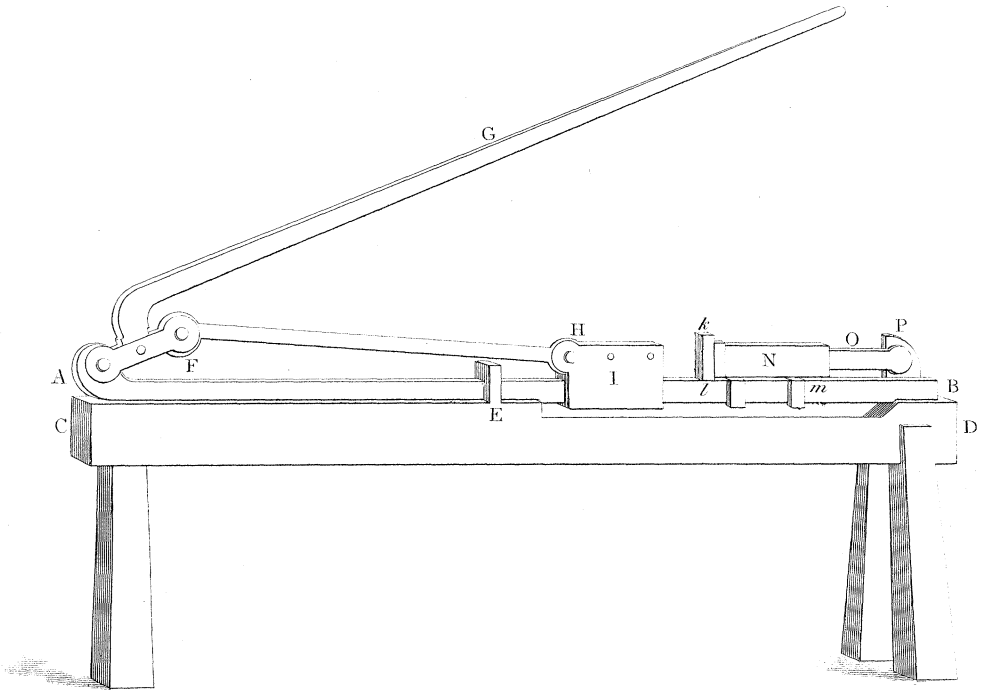
* Sulphuric acid, digested upon the gray powder of platina, thus purified, extracted less than $\frac{1}{1000}$th part of iron.

without cavities, and with uniformity,—a uniformity to be rendered perfect by subsequent pressure. In order, however, to guard effectually against cavities, the barrel may be weighed after filling it, and the actual weight of its contents being thus ascertained, may be compared with that weight of platina and water which it is known by estimate that the barrel ought to contain*. A circular piece of soft paper first, and then of woollen cloth, being laid upon the surface, allow the water to pass, during partial compression by the force of the hand with a wooden plug. A circular plate of copper is then placed upon the top, and thus sufficient consistency is given to the contents to allow of the barrel being laid horizontally in a forcible press.

The press which I have generally used for this purpose, (Plate I.), consists of a flat iron bar A B, set edgeways, and screwed down by a hook E, near its middle, where it would otherwise be liable to bend, to a strong wooden bench C D. The bar is connected by a pivot at its extremity A, with the lever A F G. An iron rod F H, which turns at its two extremities upon the pivots F and H, proceeds from the lever at F, and, as the lever descends, propells forward the carriage I, which slides along the bar. A stopper or block being placed in the vacant space I k, the carriage communicates motion to the cradle k l m, which is also made to slide along the bar, and carries the barrel N, which lies upon the cradle, straight against the piston O, which rests by its end against P, a projection in the further extremity of the bar.

The weight, which in this machine, when the angle of the lever's elevation is small, will keep the power, applied vertically at the extremity of the lever, in equilibrio = that power $\times \frac{AG \times FH}{AF\,[AF + FH]}$ $\times$ cotan. of the angle of the lever's elevation; which expression, in the case of the press actually used, becomes, Power $\times$ 5 . cotan. of the angle of the lever's elevation. This expres-

* From the mean weight of the ingots obtained in previous operations, it is known that the barrel described in the text ought to contain 16 ounces troy of dry platina powder. The weight of the contents of the barrel = 16 ounces $\times \frac{\text{sp. grav. of platina} - 1}{\text{sp. grav. of platina}}$ + the weight of a cubic inch of water $\times$ capacity of the barrel in cubic inches = 16 ounces $\times \frac{20.25}{21.25}$ + .526 ounces $\times$ 7.05 = 18.9575 ounces troy. Should the contents of the barrel weigh materially less than this estimated weight, there must be a want of uniformity in the disposition of the powder within the barrel.



Scale 1 inch to a Foot.

sion, at an elevation of 5°, becomes nearly 60 × power, and at an elevation of 1°, becomes nearly 300 × power; and when the lever becomes horizontal, the multiplier of the power becomes *quasi* infinite. This explanation will be sufficient to show the mechanical advantage with which, by means of this press, the weight of the operator, acting on the end of the lever, will be made to bear against the area of the section of the barrel, a circle little more than an inch in diameter.

After compression, which is to be carried to the utmost limit possible, the stopper at the extremity being taken out, the cake of platina will easily be removed, owing to the conical form of the barrel; and being now so hard and firm that it may be handled without danger of breaking, it is to be placed upon a charcoal fire, and there heated to redness, in order to drive off moisture, burn off grease, and give to it a firmer degree of cohesion.

The cake is next to be heated in a wind-furnace; and for this purpose is to be raised upon an earthen stand about $2\frac{1}{2}$ inches above the grate of the furnace, the stand being strown over with a layer of clean quartzose sand, on which the cake is to be placed, standing upright on one of its ends. It is then to be covered with an inverted cylindrical pot, of the most refractory crucible ware, resting at its open end upon the layer of sand; and care is to be taken that the sides of the pot do not touch the cake.

To prevent the blistering of the platina by heat, which is the usual defect of this metal in its manufactured state, it is essential to expose the cake to the most intense heat that a wind-furnace can be made to receive, more intense than the platina can well be required to bear under any subsequent treatment; so that all impurities may be totally driven off, which any lower temperature might otherwise render volatile. The furnace is to be fed with Staffordshire coke, and the action of the fire is to be continued for about twenty minutes from the time of lighting it, a breathing heat being maintained during the last four or five minutes.

The cake is now to be removed from the furnace, and being placed upright upon an anvil, is to be struck, while hot, on the top, with a heavy hammer, so as at one heating effectually to close the metal. If in this process of forging, the cylinder should become bent, it should on no account be hammered on the side, by which treatment it would be cracked irremediably; but must be

straightened by blows upon the extremities, dexterously directed, so as to reduce to a straight line the parts which project.

The work of the operator is now so far complete, that the ingot of platina may be reduced, by the processes of heating and forging, like that of any other metal, to any form that may be required. After forging, the ingot is to be cleaned from the ferruginous scales which its surface is apt to contract in the fire, by smearing over its surface with a moistened mixture of equal parts by measure of crystallized borax and common salt of tartar, which, when in fusion, is a ready solvent of such impurities*, and then exposing it, upon a platina tray, under an inverted pot, to the heat of a wind-furnace. The ingot on being taken out of the furnace, is immediately to be plunged into dilute sulphuric acid, which in the course of a few hours will entirely dissolve the flux adhering to the surface. The ingot may then be flattened into leaf, drawn into wire, or submitted to any of the processes of which the most ductile metals are capable.

The perfection of the methods above described, for giving to platina complete malleability, will best be estimated by comparing the metal thus obtained, in respect of its specific gravity, with platina which has undergone complete fusion; and by comparing it, in respect of its tenacity, with other metals possessing that quality in the greatest perfection.

The specific gravity of platina, drawn into fine wire, from a button which had been completely fused by the late Dr. E. D. Clarke with an oxy-hydrogen blowpipe, I found to be 21.16. The aggregate specific gravity of the cake of metallic mud, when first introduced into the barrel, exclusively of moisture, is about 4.3; when taken from the press, is about 10. That of the cake fully contracted, on being taken out of the wind-furnace before forging, is from 17 to 17.7. The mean specific gravity of the platina, after forging, is about 21.25, although that of some rods, after being drawn, is 21.4: but that of fine platina

* The chemist will find this flux very serviceable for removing from his crucible or other vessels of platina those ferruginous scales with which, after long use, and particularly after being strongly heated in a coal or coke fire, they become incrusted. In the analysis of earthy minerals, I have been in the habit of using a similar flux, composed of 2 parts by weight of crystallized carbonate of soda, and 1 of crystallized borax, well ground together. It has the advantage of not acting, like caustic alkali, upon the platina crucible, and is a powerful solvent of jargon and many other minerals, which yield with difficulty to other fluxes. If the mineral to be operated on requires oxidation, in order to decompose it, a little nitre or nitrate of soda may be added.

wire, determined by comparing the weight of a given length of it with the weight of an equal length of gold wire drawn through the same hole, I find to be 21.5, which is the maximum specific gravity that we can well expect to be given to platina.

The mean tenacity, determined by the weights required to break them, of two fine platina wires, the one of $\frac{1}{3000}$, the other of $\frac{1}{3850}$ of an inch in diameter, reduced to the standard of a wire $\frac{1}{10}$th of an inch in diameter, I found to be 409 pounds; and the mean tenacity of 11 wires, beginning with $\frac{1}{4300}$ and ending with $\frac{1}{23.1000}$ of an inch, reduced to the former standard, I found to be 589 pounds; the maximum of these 11 cases being 645 pounds, and the minimum 480 pounds. The coarsest and the finest wire which I tried, present exceptions, since a wire of $\frac{1}{1300}$ of an inch gave 290 pounds, and a wire of $\frac{1}{30.1000}$ of an inch, 190 pounds. If we take 590 pounds, as determined by the 11 consecutive trials, to be the measure of the tenacity of the platina prepared by the processes above described, and consider that the tenacity of gold wire, reduced to the same standard, is about 500, and that of iron-wire, 600, we shall have full reason to be satisfied with the processes, detailed in the present paper, by which Platina has been rendered malleable.

To this paper I beg to subjoin an account of some processes relating to two of the metals which are found in the ore of platina.

To obtain malleable Palladium, the residuum obtained from burning the prussiate of that metal is to be combined with sulphur, and each cake of the sulphuret, after being fused, is to be finally purified by cupellation, in an open crucible, with borax and a little nitre. The sulphuret is then to be roasted, at a low red heat, on a flat brick, and pressed, when reduced to a pasty consistence, into a square or oblong and perfectly flat cake. It is again to be roasted very patiently, at a low red heat, until it becomes spongy on the surface. During this process, sulphur flies off in the state of sulphurous acid, especially at those moments when the heat is allowed occasionally to subside. The ingot is then to be cooled; and when quite cold, is to be tapped with a light hammer, in order to condense and beat down the spongy excrescences on its surface. The alternate roastings and tappings (or gentle hammerings) require the utmost

patience and perseverance, before the cake can be brought to bear hard blows: but it may, by these means, at length be made so flat and square, as to bear being passed through the flatting-mill, and so laminated to any required degree of thinness.

Thus prepared, it is always brittle, while hot; possibly, from its still containing a small remnant of sulphur. I have also fused some palladium per se, without using sulphur; but I have always found it, when treated in this way, so hard and difficult to manage, that I greatly prefer the former process.

To obtain the oxide of Osmium in a pure, solid, and crystallized state, I grind together, and introduce, when ground, into a cold crucible, 3 parts by weight of the pulverulent ore of iridium, and 1 part of nitre. The crucible is to be heated to a good red in an open fire, until the ingredients are reduced to a pasty state; when osmic fumes will be found to arise from it. The soluble parts of the mixture are then to be dissolved in the smallest quantity of water necessary for the purpose, and the liquor, thus obtained, is to be mixed, in a retort, with so much sulphuric acid, diluted with its weight of water, as is equivalent to the potash contained in the nitre employed; but no inconvenience will result from using an excess of sulphuric acid. By distilling rapidly into a clean receiver, for so long a time as the osmic fumes continue to come over, the oxide will be collected in the form of a white crust on the sides of the receiver; and there melting, it will run down in drops beneath the watery solution, forming a fluid flattened globule at the bottom. When the receiver has become quite cold, the oxide will become solid and crystallize. One such operation has yielded 30 grains of the crystallized oxide, besides a strong aqueous solution of it.