The First Real Melting of Platinum

LAVOISIER'S ULTIMATE SUCCESS WITH OXYGEN

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For thirty years after its recognition as a new element platinum resisted all attempts to melt it, first in the early porcelain furnaces and later by means of enormous burning glasses. Then in April 1782, just two hundred years ago, Antoine Laurent Lavoisier finally succeeded in bringing about its fusion on a small scale by using a blast of oxygen, the gaseous element discovered a few years earlier by Joseph Priestley. Lavoisier repeated his experiment three months later before a most distinguished audience at the Académie Royale des Sciences in Paris.

When European scientists first became aware of platinum in about 1750 they were much puzzled by its refractory nature and its apparent infusibility. Many attempts were made during the next twenty years to bring about its fusion, but unless it was alloyed or mixed with other metals of much lower melting point, such as arsenic or lead, these were invariably unsuccessful (1).

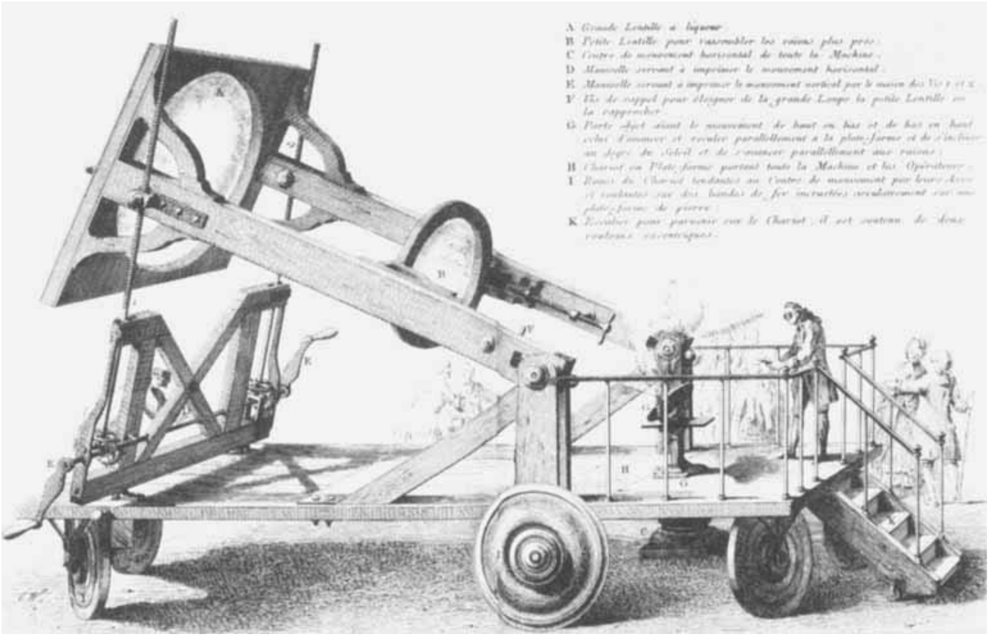
The native metal obtained from the Spanish colonies in South America was in the form of small grains mixed with particles of iron and sand, and although numerous attempts were made to achieve and maintain a higher temperature than any so far reached, nothing more than a state of partial agglomoration was attained.

One of the earlier attempts that almost succeeded was carried out by Pierre Joseph Macquer (1718–1784) and his colleague Antoine Baumé (1728–1804) in 1758. Macquer succinctly expressed the feelings of the scientists of the time:

> "As it is impossible to examine the essential properties of a metal, that is to say those from which one can judge the usefulness that one might expect from it, such as its ductility and hardness, without melting it alone to obtain an ingot of a certain size, we have first thought it necessary to ascertain whether there is any hope of melting this metal."

At this time Macquer, apart from his lecturing on chemistry, was assisting Jean Hellot as scientific director of the porcelain factory at Sèvres, and after an abortive attempt, lasting fifteen hours, to melt platinum in a wood fire, he exposed a small quantity in a crucible in the porcelain furnace, "the greatest degree of fire known". After five days and nights no change was discernible in the platinum, but the crucible had collapsed.

After several more unsuccessful attempts Macquer and Baumé decided to determine whether platinum was essentially infusible or not by exposing it to the heat from a large concave burning mirror, a device then thought to be more powerful than any kind of fire. With this mirror, made of mercury-coated glass and 22 inches in diameter, they quickly melted iron and several mineral substances. Then, on October 16th, 1758, "the sun being perfectly clear and the air very clean", they placed at the focal length of the mirror a small piece of platinum that had already been through the porcelain furnace and that was sufficiently agglomerated to be held in a pair of pincers. They were at least partially successful this time, in that glistening rounded particles of a silvery-white metal began to appear at a few points in the small mass of material. Separating the largest of these from the unmelted residue with



After unsuccessful experiments with two older burning glasses this enormous piece of equipment was provided to the Académie Royale des Sciences by Lavoisier's friend Jean Charles Philibert Trudaine de Montigny. Installed outside the Louvre in 1774, it was used by a distinguished committee of scientists including Lavoisier but failed to achieve the melting of platinum

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their fingers, they found them to be readily malleable and were able to hammer them to foil without any signs of cracking (2). But Macquer's hopes of producing even a small ingot of platinum with which to determine its properties were not fulfilled.

The Great Burning Glasses

Nothing more of significance occurred in this direction until 1772, when a new phase of activity with large burning glasses set in under the auspices of the Académie Royale des Sciences in Paris. This arose from the argument as to whether a diamond, when exposed to very strong heat, would be destroyed by evaporation or by combustion, and if the latter, would the presence of air be necessary. After a number of indecisive experiments by Macquer together with the apothecary Louis Claude Cadet (1731–1799) and Antoine Laurent Lavoisier (1743–1794) in which diamonds were heated in both the presence and the absence of air, it was decided to ask permission from the Académie to make use of a great burning glass that had been kept there as a curiosity for over fifty years.

The pioneer of the burning glass had been a rich German nobleman, Count Ehrenfried Walther von Tschirnhaus (1651–1708), an able chemist and a mathematician, who had devoted some of his great wealth to building a glass works on his estate and who had spent some years in Dresden at the court of Augustus the Strong, the Elector of Saxony. Here he had attempted to produce porcelain by the use of one of the four very large lenses he had made. Tschirnhaus was a frequent visitor to Paris and was elected a member of the Académie des Sciences in 1682, and one of his burning glasses was bought in 1702 by Philippe, Duke of Orleans (the nephew of Louis XIV and on the latter's death regent for the young Louis XV). This was for the use of his protegé, the chemist Guillaume Homberg (1652–1715) for whom he had equipped a splendid laboratory in the Palais Royal. It was also used a few years later by Etienne Geoffroy to study the effects of heating on specimens of iron, copper, tin and lead (3) and was then consigned as a museum piece into the care of the Académie.

In July 1772 Cadet, with the support of the physicist Mathurin Jacques Brisson (1723–1806), asked for the use of the great lens. Permission was at once granted by the Académie, who asked Macquer and Lavoisier to join a committee to take part in the proposed experiments. The apparatus was retrieved and, together with another Tschirnhaus lens owned by the Comte de la Tour d'Auvergne, was set up in the Jardin de l'Infante, a terrace beside the Louvre, where the Académie had rooms allocated to them by Louis XIV in 1699, and the investigation began in mid-August.

Lavoisier's Concept of Air

It was just at this time that Lavoisier had come to the conclusion that air, or some constituent of air, played an important role in the processes of calcination of metals and in combustion—a concept, as he put it, "destined to bring about a revolution in physics and chemistry", and he was anxious to make use of the burning glasses to verify his views. The year 1772 was, as his biographer Henry Guerlac puts it, "pivotal in his career". Before this he had shown no interest in air or combustion, but he now embarked on his most exciting and fruitful period. He was, however, by far the junior member of the team, both in years and in rank in the Académie, and he was not free to use the equipment for his own purposes until mid-October, the older members merely proposing to expose all manner of substances to the new means of heating, simply following the earlier experiments of Homberg and Geoffroy.

On August 14th a small piece of native platinum was exposed to the heat of the Tour d'Auvergne lens, and Lavoisier described the result:

> "The sky being but little favourable because of many light clouds, the platinum exposed for 24 minutes, did not melt but softened and agglomorated more than it had done in earlier experiments and was still attracted by a magnet".

On the 29th of the same month a further trial again resulted only in agglomeration, while on September 5th, as Lavoisier describes it:

> "a small mass of platinum, strongly agglutinated, that had already been exposed twice in the fire, exposed for 22 minutes in strong sunlight, hardly changed" (4).

An account of these experiments was read to the Académie by Macquer on November 14th and was immediately published in the new journal founded by the Abbé Rozier, *Observations sur la Physique* (5).

The Trudaine de Montigny Lens

Some further experiments were carried out in the late summer of 1773, but the two Tschirnhaus lenses were not proving altogether satisfactory. They both contained bubbles, striations and other defects and the committee felt that a better effect would be obtained with an apparatus consisting of two large pieces of glass with a curvature forming part of a sphere, joined at their circumferences and then filled with alcohol. This suggestion was taken up by Jean Charles Philibert Trudaine de Montigny (1733–1777), an older friend of Lavoisier and the successor to his distinguished father as Intendant General of Finances. Trudaine was aspiring to become a chemist, he had a laboratory in his chateau at Montigny-Lencoup, some eighty kilometres south-east of Paris, and he had been elected an honorary member of the Académie in 1764.

At his expense such a piece of apparatus was constructed by M. de Bernieres, the inventive engineer who was the Controleur des Ponts et Chaussees and responsible to Trudaine, the very large pieces of glass being made in a newly built furnace in the Paris works of Saint Gobain, who donated the glass to the Académie.

This enormous piece of equipment, illustrated here, was installed in the Jardin de l'Infante and was ready for operation at the beginning of October 1774. The great lens was four feet in diameter, against the three feet of the Tschirnhaus lenses, and was mounted on a carriage to enable the movement of the sun to be followed. The focal length was ten feet, at which point the light was so strong as to harm

Joseph Priestley
1733–1804

On August 1st, 1774, at Bowood House in Wiltshire, the home of his patron the Earl of Shelburne, Priestley first separated oxygen, or "dephlogisticated air" as he called it. Two months later, during a visit to Paris he acquainted Lavoisier with his experiment

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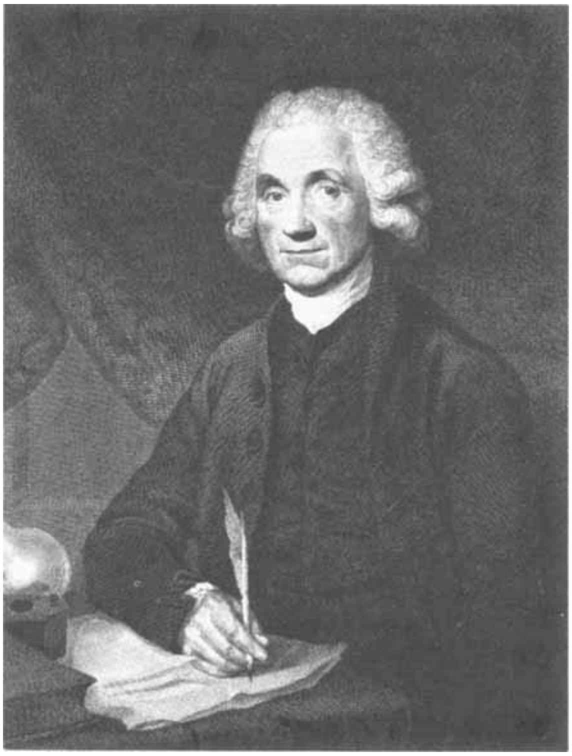


the eyes of the observer, and a smaller lens was arranged to concentrate the sun's rays still further. Iron was readily melted by this means, but once again no success was obtained with platinum.

A report of the experiments by Trudaine de Montigny, Macquer, Cadet, Lavoisier and Brisson was read to the Académie on November 12th by Brisson and printed in the Mémoires of the Académie for 1774, although these were not published until 1778. It contains the following paragraph:

> "Having exposed to the fire some grains of platinum in a carbon cavity it appeared to congregate and to be reduced in volume and to be about to melt. A little later it bubbled and fumed and all the grains united into a single mass, but without forming a spherical button as with the other metals. After this kind of semi-fusion the platinum was no longer attracted by a magnet as it had been before being exposed to the action of the sun" (6).

Thus platinum had still not been melted, at least partially on account of its admixture in its native form with iron and sand and the consequent formation of a refractory iron oxide.

Joseph Priestley and Oxygen

At the very time that this unwieldy piece of equipment was being erected outside the Louvre there arrived in Paris two visitors from England, one of whom was to have a profound effect upon Lavoisier's thinking and his future researches. One was the Earl of Shelburne (1737–1805), a young Whig politician who had been a member of Pitt's cabinet but who was now out of office on account of his sympathy with the American colonists. With him he brought his recently appointed librarian and companion Joseph Priestley.

Monday, August 1st, 1774 is probably the best remembered date in all the history of chemistry; on that day Priestley first isolated oxygen by heating *mercurius calcinatus* (mercuric oxide) by means of a burning glass, only some 12 inches in diameter, given to him by John Parker, the instrument maker of Fleet Street in London. This classic experiment was performed in Lord Shelburne's home, Bowood House at Calne in Wiltshire, and Priestley did not at that time fully recognise what he had achieved. He found that the "dephlogisticated air" he had obtained was insoluble in water and that a candle burned in it much more brightly, but he was at a loss to account for all this. Any further experimentation was interrupted by his accompanying Shelburne on an extensive European tour. Leaving England about the middle of August, they visited northern France, Holland and parts of Germany, arriving in Paris on September 29th. Here Shelburne took his companion on a series of visits to distinguished people and to public monuments, but Priestley asked to be excused from these expeditions, preferring to spend his time with those interested in literary and scientific matters. As is well known, he more than once visited Lavoisier, who then had a laboratory in his house in the Rue Neuve des Bons Enfants, and over dinner there one evening he mentioned the new "air" he had obtained before leaving England:

> "Having made the discovery some time before I was in Paris in 1774 I mentioned it at the table of Mr. Lavoisier when most of the philosophical people in the city were present, saying it was a kind of air in which a candle burned much better than in common air, but that I had not then given it any name. At this all the company, and Mr. and Mrs. Lavoisier as much as any, expressed great surprise" (7).

Priestley returned to London at the end of October, leaving his patron in Paris, and bringing with him an ounce of mercuric oxide purchased from Cadet "of the genuineness of which there could not be any suspicion" (8). He was accompanied home by Jean Hyacinthe de Magellan (1722–1790), a Portuguese who had devoted himself to science after an early career as a monk and who plays a part in this story as an inveterate gossip and a correspondent with many scientists in England and Europe. Priestley had known him earlier in London, and had in fact entertained him on at least one occasion at the Royal Society.

Antoine Laurent Lavoisier
1743–1794

Realising the significance of Priestley's discovery, Lavoisier confirmed and extended the study of the constituent of air that supported and took part in calcination and combustion. This he first called "vital air", the purest part of the air, and later gave it the name oxygen. In 1782, with a massive piece of apparatus he had designed to yield a continuous stream of oxygen, he was the first to succeed in bringing about the melting of platinum

Very soon after Priestley's departure from Paris Lavoisier began to repeat some of the Englishman's experiments on gases in Trudaine's laboratory at Montigny and with the latter's assistance. Up to this time French chemists had confined themselves to the study of the solid and the liquid states and while Priestley was now an expert in the technique of producing and collecting gases Lavoisier was quite inexperienced. He now embarked, however, on his crucial experiments with gases which the British scientists Hales, Black and Cavendish had already recognised as active participants in chemical reactions, and in November he prepared oxygen from mercuric oxide, using a sample also prepared by Cadet.

In the same month Priestley again took up the study of gases, this time in the Earl of Shelburne's London home, Lansdowne House in Berkeley Square. Here on March 1, 1775—the date he himself regarded as the day of his discovery of oxygen—he once more obtained it from mercuric oxide and very soon communicated his findings in a letter to Sir John Pringle, President of the Royal Society (9), writing

> "the air that I have produced by this process is one that is five or six times better than common air. . . . A candle burned in this air with an amazing strength of flame".

John Michell's Suggestion

No thought of employing his new kind of air for the melting of refractory metals seems to have occurred to Priestley at this time, but in Volume II of his "Experiments and Observations on Different Kinds of Air", published in November 1775, he quotes his friend and neighbour John Michell as suggesting the possibility that it might advantageously be used for this purpose:

> "Possibly much greater things might be effected by chymists, in a variety of respects, with the prodigious heat which this air may be the means of affording them. I had no sooner mentioned the discovery of this kind of air to my friend Mr. Michell than this use occurred to him. He observed that possibly platina might be melted by means of it" (10).

The author of this suggestion, the Reverend John Michell (1724–1794), after a distinguished career as a Fellow of Queens' College Cambridge and a period as Professor of Geology had retired to Yorkshire and had been appointed, in 1767, Rector of Thornhill, a village fairly close to Leeds where Priestley, in the same year, had been appointed Minister of the Mill Hill chapel. Although he was ten years older than Priestley, the Anglican clergyman apparently enjoyed the dissenting minister's company and their scientific discussions, Priestley later writing that "I frequently visited him and was very happy in his society".

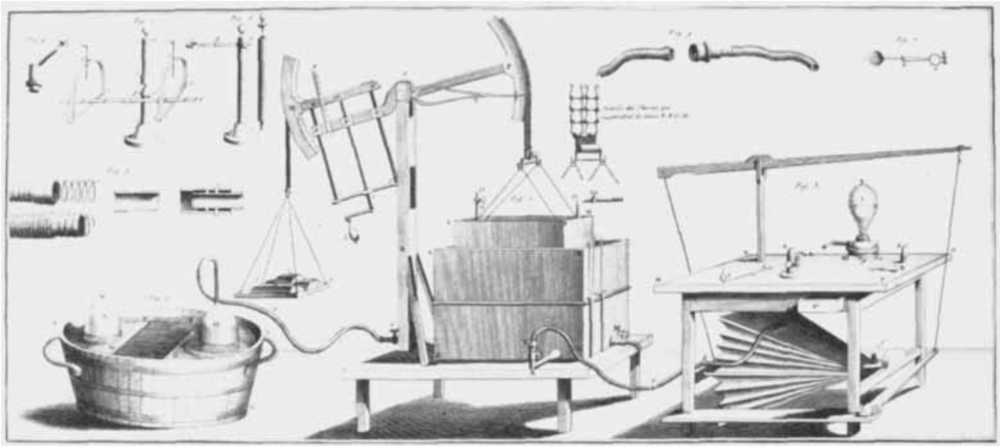
Both were Fellows of the Royal Society and both made fairly frequent visits to London, where Michell was recognised as the leading geologist and astronomer of his time and, in fact, as the founder of the science of seismology. No portrait of Michell appears to exist, the only bare record of his appearance being given by his contemporary, the Rev. William Coles the Cambridge antiquarian and historian, who referred to him as

> "a little short Man, of a black Complexion and fat . . . a Fellow of Queens' College where he was esteemed a very ingenious Man and an excellent Philosopher" (11).

He was also a great friend of Cavendish and a correspondent of William Watson, the London physician who in 1750 had presented to the Royal Society the reports of Wood and Brownrigg on the first samples of platinum brought from South America.

The second volume of "Experiments and Observations" appeared in November 1775 and Priestley's friend Magellan immediately wrote to Lavoisier, sending a set of advance sheets, a gift from the author (13). Priestley also forwarded copies to Trudaine for passing on to his translator Jacques Gibelin (12), so that Lavoisier would have been doubly sure to be fully aware of the publication.

By this time, however, Lavoisier had been appointed a Commissioner of the Régie des Poudres, the gunpowder administration, and had moved into the Paris Arsenal where his new and excellently furnished laboratory became the



The apparatus designed by Lavoisier to burn continuous streams of oxygen and hydrogen. Before this had been completed he used it in April 1782 to direct burning oxygen on to a small quantity of platinum held in a piece of charcoal. Three months later, he repeated the experiment at a meeting of the Académie Royale des Sciences before a distinguished audience including the Grand Duke Paul of Russia and Benjamin Franklin, the versatile genius who was residing in Paris at the time. This drawing, as with all those illustrating her husband's papers, was prepared by Madame Lavoisier

meeting place for many of his contemporary scientists. His duties with the Académie des Sciences and now with the gunpowder commission unfortunately reduced the time for chemical experiments to just one full day in the week and otherwise to the very early mornings and late evenings, but he was now giving intense thought to the role of some part of the air in combining with metals in calcination, another part of the air remaining inert, and he began to realise the significance of Priestley's experiments on "dephlogisticated air". On August 8, 1778, Lavoisier read to the Académie des Sciences a revised version of an older memoir on mercury calx in which he stated that metals combined with "the most salubrious and purest part of the air" to form calces. A year later he announced to the Académie that he would name this part of the air "oxygen", from the Greek οξυς, acid, and *γεινομαι*, I beget.

Lavoisier's Caisse Pneumatique

In the meantime Franz Karl Achard in Berlin had made use of Priestley's "dephlogisticated air" to yield higher temperatures than had hitherto been possible, directing a stream of oxygen on to a piece of carbon. This he reported to the Royal Academy in Berlin in 1779, although the memoires were not published until two years later (14), but it clearly gave Lavoisier the impetus to conduct some further experiments. Lavoisier was also deeply interested in "inflammable air" (hydrogen) and in the winter of 1781 he decided to carry out a large scale experiment on its combustion. Assisted by his younger colleague Pierre Simon Laplace (1749–1827) he designed a "caisse pneumatique" or gasometer in which streams of the two gases could be brought together in a double nozzle and burned. Before this equipment was completed by his instrument maker Pierre Mégnié it occurred to Lavoisier that he might be able to melt platinum with a continuous stream of oxygen, and in April 1782 he employed the first of these gasometers in a rather spectacular experiment to direct the stream of oxygen into a hollowed-out piece of charcoal in which he had placed a small quantity of the metal. The apparatus is illustrated here; its construction and use were described in a long paper to the Académie, "Sur un Moyen d'augmenter considerablement l'action du Feu et de la Chaleur, dans les Operations chimiques", read in 1782 (15). This includes a reference to "this air that M. Priestley had discovered nearly at the same time as myself" and reports the melting of platinum held in a small piece of charcoal in the stream of "vital air" as he first described oxygen:

> "The platinum had melted completely, and the small particles were united in a perfectly round globule; the melting was complete and easy whether employed ordinary platinum that one finds commercially or whether the molecules attracted by a magnet had previously been removed".

This experiment, which was reported to a meeting of the Académie des Sciences on April 10, caused something of a sensation in scientific circles in Paris, and when a special meeting of the Académie was arranged three months later for the entertainment of the Grand Duke Paul of Russia and his Grand Duchess the apparatus was transported there at considerable trouble and expense by Mégnié. The Grand Duke, the future Tsar Paul I, was travelling incognito as the Comte du Nord although his true identity was well known, and on June 6th, 1782, he was able to witness this historic melting of platinum before going on to a party given for him at Versailles by Louis XVI and Marie Antoinette.

Another famous observer of the scene, and one of an entirely different character, was Benjamin Franklin, who has been resident in Paris since 1776 as the representative of the revolutionary United States Government. He had been elected a Foreign Member of the Académie in 1772 and had developed a close friendship with Lavoisier, whose experiments he had frequently been invited to watch at the Arsenal. He was also, of course, a close friend of Priestley, whom he had first met in London in 1765 and with whom he maintained a steady correspondence over the years.

On the day following the platinum melting demonstration Franklin concluded a letter to Priestley:

> "Yesterday the Count du Nord was at the Academy of Sciences, when sundry Experiments were exhibited for his entertainment; among them, one by M. Lavoisier to show that the strongest fire we yet know is made in a Charcoal blown upon with dephlogisticated air. In a Heat so produced, he melted Platina presently the fire being much more powerful than that of the strongest burning mirror" (16).

So platinum had at last submitted to fusion, and the news was carried swiftly to Priestley. Lavoisier, with his ingenious piece of equipment, using the "dephlogisticated air" discovered by Priestley seven years earlier, and perhaps owing something to the suggestion made by the English parson and scientist John Michell, had achieved success where others had failed for so long.

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