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HISTORICAL OBSERVATIONS ON OXYGEN-BEARING COMPOUNDS OF PLATINUM AND PALLADIUM IN MINAS GERAIS, BRAZIL

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Abstract

Dark brown to black compounds associated with native gold were discovered three hundred years ago in Minas Gerais, Brazil, and have been known since then as *ouro preto* (black gold). These compounds were correctly determined as oxygen-bearing compounds of platinum – palladium – iron as early as 1833 and 1837, but the oversight of the presence of major iron in their composition led to an erroneous definition of *palladinite* and to the much-delayed recognition of a group of platinum-group-element oxides.

Keywords: platinum, palladium, oxide, *ouro preto*, Minas Gerais, Brazil.

Sommaire

Des composés brun-sombre à noirs associés à de l'or natif ont été découverts il y a trois cents ans dans les Minas Gerais, Brésil, et ont été connus depuis sous le nom d'*ouro preto* (or noir). Ces composés ont été correctement déterminés comme composés oxygénés de platine – palladium – fer dès 1833 et 1837. La négligence de la présence du fer majeur a conduit à une définition erronée de la *palladinite* ainsi qu'à la reconnaissance tardive d'un groupe d'oxydes des éléments du groupe du platine.

Mots-clés: platine, palladium, oxyde, *ouro preto*, Minas Gerais, Brésil.

Introduction

It has recently been established that Platinum Group Oxide Minerals (PGOM) are by no means forbidden compounds in nature: this evidence rests on data obtained during the last few decades with the scanning electron microscope and the electron microprobe (Augé & Legendre 1994, Jedwab 1995). In particular, a large group of such compounds has been (re)discovered by us in concentrates from the Cauè iron mine, at Itabira, Minas Gerais, Brazil (Jedwab 1995). These compounds, which contain variable amounts of Pt, Pd and

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Au (Fig. 1), associated with the transition elements Ti, Mn and Fe, are visually indistinguishable from the trivial Fe–Mn oxides and oxyhydroxides, which explains perhaps their late recognition as PGOM.

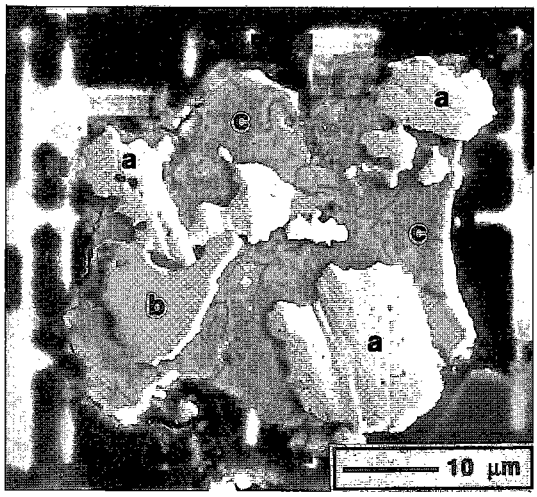

FIG. 1. *Ouro preto* grain of irregular outline, collected in a sluice concentrate from the tailings of the Cauê iron mine (Itabira, Minas Gerais, Brazil): (a) gold, (b) isomertieite, (c) black Pt–Cu–Au–Fe–Mn oxide. The oxide shows an amorphous texture, a low polishing hardness and several cracks. The chemical composition of (c): $Pt_{54.38}Au_{3.20}Cu_{1.29}Fe_{16.80}Mn_{24.35}$ (at.%, oxygen-free basis, mean result of three analyses). Polished grain-mount, scanning electron microscope, secondary mode.

In the course of our literature search, it became evident that related compounds had already been encountered three centuries ago by the first Brazilian gold miners and correctly analyzed during the early 19th century. Although pristine samples from the historical mines are extremely rare or have not been preserved at all (Carvalho da Silva *et al.* 1985), we are now in a position to re-assess the early descriptions and analyses, some of which have been overlooked, misquoted or misunderstood. The present paper does not review the latests aspects of the *palladinite* case, which is in the process of revalidation (*cf.* Clark *et al.* 1974, Jedwab *et al.* 1993), nor the results of modern analyses of *ouro preto* by Carvalho da Silva *et al.* (1985) and their followers, which will be re-assessed in the wake of a special study of the PGOM from Itabira (*cf.* Jedwab 1995).

THE HISTORICAL RECORDS

The Brazilian Gold Cycle and the finding of new varieties of gold

The Brazilian Gold Rush known as the Gold Cycle began at about 1693 in what would later be known as the State of Minas Gerais. Gold mining began as usual in placer deposits, flourished over the first decades of the 18th century and vanished about 1750, to be revived in the first decades of the 19th century with lode mining by British companies. This cycle has been described in great detail by several travelers and scholars, from whom we borrowed many details relevant to this subject (Antonil 1711, Mawe 1812, von Eschwege 1833, Burton 1869, Ferrand 1894, Boxer 1962, Russell-Wood 1977).

During this Gold Cycle, a wealth of vernacular names appeared on the scene, coined by non-professional gold miners qualified as arrant rowdies and ruffians (Antonil 1711, Burton 1869). Indeed, foreign miners (especially Spaniards) who could have transmitted the mining skills to South Americans, were barred from entering the country by royal order. However, Russell-Wood (1977) has noted that slaves specially selected for their skills as copper and gold smelters had been brought in from West Africa. Professional assayers were of course present in Brazil and essential in levying taxes on gold production and the running of the business aspects. However, silver was the only precious metal known at that time to alloy with gold. The presence of a vast metallogenic province in which palladium was exceptionally abundant still awaited discovery. Indeed, the elements *palladium* and *platinum*, which are important ingredients of *ouro preto* (Jedwab 1995), were discovered at a time when this compound was already rare: platinum was discovered in Colombia in 1748, and the native metal was found in Brazil only in 1798 by Couto (Hussak 1904). However, Hussak is of the opinion that Couto confused native platinum and white palladian gold in some of the localities where he claimed to have found platinum. Palladium was discovered in 1804 in Colombian ores, and its presence in Brazilian gold ores and ingots was confirmed a few years later (Cloud 1809, Wollaston 1809).

The vernacular names that appeared at the dawn of the Gold Cycle in the Minas Gerais designated variations of color, texture and shape of the gold grains, as well as their spot or bullion value: *ouro fino* (fine gold), *ouro pobre* (poor gold), *ouro em pô* (flour gold), *ouro de bom toque* (gold of good coinage), *ouro branco* (white gold), *ouro podre* (rotten gold), *ouro preto* (black or dark gold). Among these names, *ouro preto* is the one that is still difficult to link up to a precise mineral or compound, especially when they have vanished from sight since three centuries.

Andre Joaô Antonil (1711)

The first reliable appearance in writing of the name *ouro preto* is found in a report by Antonil (1711): "As for the various sorts of gold, it is known that the one called *ouro preto* because of its surface is colored like unburnt steel appears with a vivid yolk yellow color when tried under the teeth. It is the finest gold, since it is of almost 23 carats" (p. 363).

According to Antonil's detailed description of the mining operations, grains of *ouro preto* were first concentrated along with pure gold by panning the stream sediments. This rough concentrate was then poured in water-filled excavations, and trampled for a long time by the slaves. By so doing, the soft and brittle *ouro preto* crusts (Fig. 1) were deformed and separated from the malleable gold. Antonil's observation of the high purity of the gold observed under the black crust is extremely important, if one admits (as we do now: *cf.* Jedwab 1995) that the dark crust is a product of selective oxidation of everything but the purest gold.

During the first decades of the Gold cycle, *ouro preto* was found in such abundance that a town of Villa Rica was later renamed Ouro Preto. It is now celebrated for its baroque architectural ensemble, and is inscribed on the World Heritage List by UNESCO.

John Mawe (1812)

The first knowledgeable foreign visitor authorized to enter the Minas Gerais area was the English mineralogist and paleontologist John Mawe (1812), who became familiar with the gold and diamond mines, and wrote a detailed account of his observations. However, he did not mention *ouro preto* in his book. He simply mentioned platinian *ouro branco* (white gold), which is not unrelated to our subject:

> "[This place] ...is called Largos, and also bears the name of Oro Branco (White Gold), in allusion to a granular substance, not unlike gold in size and weight, found in a gold-washing in the bed of the stream. This substance, which has since been proved to be platina, was discovered many years ago in the *cascalho* below the vegetable earth, and incombent on the solid rock, accompanied with gold and black oxide of iron" (p. 209). [*Cascalho* designated the pay-streak, the profitable part of a placer deposit. One distinguished *cascalho virgem*, undisturbed sediment, from *cascalho bravo*, disturbed sediment already impoverished by a former mining episode].

The presence of palladium is not acknowledged, although its presence at the same place is well documented, and it even exceeds platinum in relative amount (Hussak 1904). The expression *oro (ouro) branco* was then mostly applied to what is now known as palladiferous gold (porpezite) and native platinum, but Hussak (1904), who analyzed six color varieties of gold, found that palladian gold could also be yellow, copper red and brown. Mawe (1812) stressed the noteworthy association of gold, black oxide of iron, and *platina* (platinum), but this is commonplace for a black sand.

Wilhelm von Eschwege (1811–1821; 1833)

The German mining engineer Baron Wilhelm L. von Eschwege had been hired by the Brazilian government to study the Brazilian ore deposits and organize their mining. He travelled in Minas Gerais from 1811 to 1821, but the publication of his book *Pluto Brasiliensis* was delayed until 1833; his embittered life as an entrepreneur is here described in great detail. He quoted *ouro preto* in two instances, but without giving its composition or his personal opinion about the latter:

> "...because of the blackish color of the gold found at that place [the village of Camargo], this was named Ouro Preto"(p. 14).
>
> "The color of the gold varies much, the most frequent being gold-yellow. However, it occurs also covered with a blackish skin. This was especially the case in the beginnings, when *cascalho virgem* was still washed from the stream near Villa Rica, hence the name of Riberâo do Oiro preto" [*sic*] (p. 232).

One has here a precise description of a complex grain, but one also has the impression that either von Eschwege did not see *ouro preto*, which was already a rarity, or did not pay too much attention to it, since it was then mined by very poor old women. In another section dealing with *ouro podre*, one understands that he was aware of the association of platinum with gold, but also with iron:

> "...D. Jozé Mathias, who claimed the discovery of "ouro podre"*... A Jozé Caitano Alves de Magelhais was the first to extract it in such a high amount that the output of a handkerchief-full of cascalho was 130 oitavas [*i.e.*, 466 g] of platina-containing gold of a silvery grey color. [*Ouro podre (rotten gold). Mr. Döbereiner, Court Counsellor at Jena, to whom I sent a small portion of that gold, analyzed it. Unfortunately, I lost his result, but I still remember clearly that it contained iron and a little platina" (p. 73).]

This is probably the first documented hint (as poor as it is) of the presence of platinum associated with iron. Counselor Döbereiner never did publish the results of this analysis on his own account (Howe & Holtz 1919). It is not clear whether *rotten* refers to the porous texture of the gold, to its unpromising shine or color, or to its poor bullion value.

Wilhelm A. Lampadius and Karl F. Plattner (1833)

The end of von Eschwege's Brazilian stay coincided with the establishment of British companies who resumed the mining of gold, but now in hard rocks rather

than in placers. These ores were lode-like concentrations along shear zones in ironstones (itabirites). The names *jacutinga* or *zagotinga* were used to designate a variety of dolomitic ironstones with gold-rich strikes (Olivo & Gammons 1996), but cynics said that only seasoned miners were able to recognize it: the black color of the ore was certainly a challenge for the foreigners.

At the same time as the publication of von Eschwege's book, an important paper was published by Lampadius & Plattner (1833). Plattner was recognized in his time as an authority in the blowpipe analysis of minerals, and Lampadius was among the most famous mineral chemists (Burchard 1994). The paper deals with the analysis of a gold-bearing vein-rock (*Gangfossile*) from the region of "Sabara or Villa Rica" for precious metals. The rock was received from the brother of a Herr von Brant, and composed of black hematite and visible gold. This rock is identified as *jacutinga* in Hussak's review (1904), but it is a personal addition, since the word is not used in the paper.

The authors made separate experiments, according to their respective skills, and presented their conclusions separately. By analyzing small fragments of the ore under the blowpipe, Plattner established that it contained the expected native gold, but also platinum and iridium. In order to investigate the distribution of these latter metals, he separated the bulk rock by mechanical means (it is not stated if by decantation or by panning), and obtained three fractions: 1) a very fine, light fraction, mainly composed of greyish black hematite with a red hue, 2) a coarse dense fraction, composed of grainy hematite with some visible gold, and 3) an even more dense concentrate composed of pure native gold flakes and grains (p. 355).

The separate chemical analyses of the aqua regia solution and insoluble fraction showed that platinum was present in fractions 1 and 2, but not in fraction 3. The gold concentrate was then examined under the magnifying glass for the possible presence of native platinum, but to no avail. When submitted to the malleability test, the gold was found to behave like the pure metal. There was thus no platinum allied to, or mixed with, the native gold. The analysis under the blowpipe of small, carefully selected crystals of hematite proved here too that platinum is absent. The following conclusions were then drawn:

> "Considering that the mechanical separation of the gold from the hematite yields no visible native platinum at all, and on the other hand that the hematite [bulk] sample contains platinum (according to the chemical analyses), but that isolated crystals of the same are entirely devoid of platinum (according to the blowpipe analysis), the following must consequently be admitted: *either the platinum occurs in the native state and is very finely dispersed in the iron ore, or it forms in combination with other materials a brittle pulverizable material of a color that resembles that of hematite, and can thus difficultly be discerned by eye from the latter*" (p. 358–359) [our italics].

As a conclusion of his own series of analyses and observations, Lampadius wrote:

> "Thus, this analysis corroborates the former assumption and the visual impression that the platinum and its normal associates are not chemically bound to the gold in the studied ore, but instead, that the argentian gold and the platinum ore occur mingled one close to the other in the iron ore" (p. 356).

It should be noted here that as in Mawe's book, no mention is made in this paper of the possible existence of *ouro preto* in the rock, or of a possible connection of the hypothetical black platinum compound with *ouro preto*.

Percival N. Johnson and Wilhelm A. Lampadius (1837)

In 1837, Lampadius published another paper, but now with Percival N. Johnson, dealing with preliminary observations and analyses by Johnson on gold concentrates extracted from auriferous ironstones (zacotinga) at *Gorgo Soco* (also written *Gongo Socco* or *Congo Soco*), and confirmatory analyses by Lampadius. In this paper, no reference is made to that by Lampadius & Plattner (1833), and in particular, its conclusions are not alluded to whatsoever; the name *ouro preto* is not used either.

Three samples were transmitted by P.N. Johnson (London) to Lampadius (Freiberg), with details about location and analytical comments. (In the following excerpts, the translations into German are by Lampadius, as they appear in his paper):

> a) Zacotinga, Iron ore, in which the Palladium–Gold is found. Eisenerz, in welchem das Palladgold (in Brasilien) gefunden wird.
> b) Brasilian Gold Ore with Palladium. Brasilianisches palladhaltiges Gold.
> c) Double Salts of Palladium. Palladdoppelsalz.
> d) Metallic Palladium, produced from the double Salt. Palladmetal, aus dem Doppelsalz dargestellt (p. 309). [According to an explanation given on p. 311, the *zacotinga* (item a) contained a mean of 4 wt.% palladian gold].

Item b, which was probably a concentrate obtained from the easily crushed *zacotinga*, is described as follows:

> "The palladian gold occurred partly as a powder and partly as rounded grains of a dark brown color. A few metallic particles of dark-colored gold were obvious in the powder. The rounded grains could be crushed between the fingers, and they disclosed thereafter particles of gold as flakes and as few rounded nuclei. When powdered and digested in hydrochloric acid, they left

behind a dark brown solution with gold of a color corresponding to 20–21 carats, cleared of its crust of brown powder. This gold is malleable, but becomes more and more flaky when hammered" (p. 310).

Lampadius then transcribed a commentary by Johnson, and conveyed his own analytical procedures and results, and his enthusiastic comments in a footnote:

"I think", says Mr. Johnson, "that palladium occurs in the *zacotinga* partly in the native state in combination with gold, partly as oxide**.

** I found that indication by Mr. Johnson so interesting that I undertook immediately an investigation in relation with it. I had observed previously that the palladian gold could be liberated from the brown powder with the aid of hydrochloric acid. I considered consequently the crust wrapping the native gold as limonite [Brauneisenstein]. But the following experiment taught me something else" (p. 311).

Here follows a detailed description of the various chemical analyses which Lampadius made, and from which he drew the conclusion that it was not limonite at all. Indeed, he wrote in the same foonote:

"It occurred thus to me without doubt that the palladian gold obtained from the *zacotinga* by washing [decantation?] was a mixture of native palladian gold and of an iron-rich palladium oxidate [Palladoxydat] (probably an oxide). We have here as a novelty the first occurrence of a metal connected with platinum in the oxidized state" (p. 312).

He wrote again in another footnote:

"The presence of a trace of osmium, quoted here as well as that found later of a trace of platinum, makes it likely that a small amount of platinum ore occurs also in the zacotinga. But it is also possible that both metals belong as minor components to the gold ore. The examination of these questions is left for the future" (p. 312).

It is noteworthy that this 1837 paper does not refer whatsoever to the 1833 paper by Lampadius & Plattner, as if they were investigating entirely unrelated mineralogical objects. However, the finding of platinum in the *zacotinga* and in the palladium – iron oxidate should have been a reminder of one of the most interesting results (or hypotheses) of the first paper. It is nevertheless clear that Johnson and Lampadius did not consider their newly discovered compound as a pure palladium oxide, but as a still-to-be-defined oxygenated compound of major iron *and* palladium with minor amounts of platinum and iridium.

Discussion

It is clear that early investigators have chemically identified platinum or palladium (or both) in dark brown to black compounds associated with iron oxides, or containing iron in the oxidized state. But the impact of these observations has been thwarted by various setbacks. Firstly, Antonil's report went through the whole editing and publishing process, but its diffusion was stopped at the last moment by the Portuguese administration for fear that crowds of foreigners would be lured to Brazil. The whole print-run of the report was destroyed before its release, except for a few copies (five or six are still preserved) which were rediscovered at the end of the 19th century (*cf.* A. Mansuy, in Antonil 1711). This is of course of anecdotal value, but sets the stage for later developments.

Secondly, the existence of the paper by Lampadius and Plattner was brought to light by Hussak (1904) in an extensive review of the Brazilian mineralogy and ore geology of platinum and palladium. Unfortunately, this work does them an incredible disservice by considering only the possibility of native platinum, and by suppressing the alternative possibility of a dark (hematite-looking) compound of platinum. Hussak even added personal observations borrowed from his first-hand experience, but not to be found in the original:

"Plattner arrived at the conclusion that platinum in the jacutinga cannot be alloyed with gold, but is mechanically dispersed in the native state in the ironstone, as it is certainly the case in the jacutinga from Gongo Socco and Itabira do Matto Dentro, where platinum has been found associated with much palladium and fine gold" (p. 439).

This quotation (!) should be compared with Plattner's phrasing and rationale. The reasons for this mutilation cannot be attributed to any difficulties with language or translation, and can only be conjectural. Again, it cannot be attributed to an "anti-oxide" prejudice, since Hussak quoted correctly the Johnson – Lampadius work, and said that one can repeat the HCl-leaching experiment, by which one obtains an "iron-rich palladium-oxydate, probably an oxide".

Hussak's paper was very soon and most aptly translated into Portuguese in Ouro Preto (Lisbôa 1906), but sadly, no effort was made to correct the misquotations.

Thirdly, the paper by Johnson and Lampadius entered the literature through Rammelsberg (1847) under the following heading:

"Palladiumoxydul. According to the data of Johnson & Lampadius, this compound seems to accompany the Brazilian palladian gold, and more precisely, being mixed with iron oxide, forms a brown ochreous crust that is soluble in hydrochloric acid (*J. f. pr. Chem.* **XI**, 309)" (p. 94).

One observes here the appearance of two statements neither argued or supported by the original writing: a) the existence of a definite palladium oxide (oxydul was then used for protoxide) is presented as a settled matter, and b) the palladium oxide is claimed to be a mixture

with a mineralogically distinct iron oxide.

Fourthly, these statements were uncritically borrowed by Shepard (1857) and by Adam (1869). Shepard is credited for the coining of the name *palladinite*, but he gave an erroneous locality (*Fauberg and Zalathna in Siebenburg*), erroneous colors (*yellowish, or greyish white*), erroneous chemical reactions (same as tellurium oxide), and no reference to the Johnson – Lampadius paper. Adam gave the right reference, but added a chemical composition of PdO (87% Pd, 13% O), which corresponds to the theoretical weight percentages of the monoxide, disregarding the major iron.

One now knows of course that palladium monoxide does really exist in the Itabira iron mines, with a $PdO_{tetr.}$ X-ray diffraction pattern and a stoichiometric composition, but this compound is not the mineral found by Johnson and analyzed by Lampadius (Clark *et al.* 1974, Jedwab *et al.* 1993, Olivo & Gauthier 1995).

Fifthly, Dana (1857) must be commended for his remarkable care in his first entry: "Palladium Ochre – The palladium ochre, which has still a doubtful existence, is called *Palladinite* in Shepard's Min., p. 408." (p. 124), but his stand becomes less critical in 1858: "A brown ochreous substance associated with the Palladium Gold of Brazil has been considered a *Palladium Ochre* or oxyd. It is soluble in muriatic acid. Detected by Johnson and Lampadius (*J. f. pr. Chem.* **XI**, 309)" (p. 14).

Conclusion

The late 17th and the early 18th centuries have witnessed the discovery of large amounts of a compound (or more accurately, a group of compounds) named *ouro preto* and found intimately associated by mixing with, and encrusting on, native gold. During the first decades of the 19th century, observations and analyses of similar dark-colored compounds (although not always explicitly named) led to the positive recognition of dark brown to black palladium, platinum and iron oxide compounds, coexisting with the trivial iron – manganese oxide(s) and oxyhydroxide(s), and visually very akin to them. But the learned world did not or could not pay proper attention to the mineralogical and chemical composition of this *ouro preto*. The original findings by three remarkable scientists were more often than not distorted, misread and misunderstood.

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