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Some new platinoid-rich minerals, identified with the electron microanalyser.

(With Plate XVII.)

By E. F. STUMPFL, Dr.rer.nat.

Department of Geology, University College, London.

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Summary. Minerals approximating to the formulae $PtSb_2$, PtSb, Pt(Sb,Bi), $(Pt,Ir)As_2$, $Pt(Ir,Os)_2As_4$, Pd_2CuSb , Pd(Sb,Bi), Pd_8CuSb_3 , $Pt_4Sn_3Cu_4$, and $(Fe,Ni)_2S$ have been discovered as fine intergrowths in platinum concentrates from the Driekop mine, Transvaal, South Africa. Conventional ore microscopy also proved the presence of most of the known minerals of the platinum paragenesis. The name geversite is proposed for the phase $PtSb_2$.

MICROSCOPIC investigation of platinum concentrates from the Driekop mine, Transvaal, South Africa, which the author collected during a visit to the mine in 1957, revealed the presence of a number of unknown minerals as well as a wide range of sulphides and other established members of the platinum paragenesis. The unknown minerals occur in very fine intergrowths, which made the separation of individual phases for analytical purposes or X-ray work impossible. Thus, it was not until an X-ray microanalyser was put at the author's disposal that the chemical composition of the minerals could be determined. Based on this work, the results of which frequently fitted the indications given by ore-microscopic evidence, it was possible to establish the formulae of ten new minerals. Only a few synthetic products corresponding in composition to these minerals are known.

Details of the microanalyser used, which was designed by Castaing and constructed by the CAMECA Company, Courbevoie (Seine), France, are to be found in Castaing (1960). The principles and application of the microanalyser are now becoming so well known (Guillemin and Capitant,

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1960; Capitant and Weinryb, 1960), that only a few of the main points will be reviewed here. The atoms of a sample (in this case polished sections of ore concentrates embedded in 'Araldite' resin) are excited by an electron beam focused on an area of about one square micron. Returning to their stable state, they generate X-rays, the spectra of which are analysed in two curved-crystal spectrographs. One of these, for elements with an atomic number of 11 to 17 inclusive, uses a mica strip and a gas flow proportional counter. An alternative spectrograph, for analysing elements with atomic numbers above 17, uses a quartz strip and a Geiger-Müller counter. Elements with atomic numbers below 11 cannot be detected with the equipment in question. For quantitative analysis, the intensity of each spectral peak is measured and compared with readings obtained from pure element standards, and as the sample holder is fitted with a reference sample carrier that takes up to 40 standards, the measurements can be carried out with considerable speed. Corrections of the recorded values have to be made in some cases because of the differing mass absorption coefficients of different elements. A procedure for this purpose has been developed by Castaing and the corrections increase only slightly the time needed for analysis. The accuracy of the quantitative measurements is ± 5 %. In those cases where the sum of the percentages of elements determined does not approximate 100 %, some elements may have remained undetected during analysis. This applies especially to elements with atomic numbers below 17 (i.e. elements lighter than Cl). In those instances the formulae calculated must be considered as only approximate.

Geological environment. The Driekop platinum mine is situated in the norites of the critical zone of the Bushveld complex in eastern Transvaal (see sketch-map, fig. 1). The host rock of the platinum minerals is a pipe-like body of dunite that dips about 80° NE.—roughly normal to the pseudostratification of the norites. On the surface, the dunite pipe stands out as a small hill 100 ft. high above the surrounding norite country. Only a small portion of the dunite is to be considered as workable ore and the cross-section through the pay-zone measures about 60×80 ft. It is remarkable that the occurrence of platinum pipes (Driekop, Onverwacht, Mooihoek) in this part of the Bushveld complex is confined to a straight line about 10 miles in length and striking N. 35° W. Wagner (1929), who dealt in detail with the geological and genetic implications, suggested relations to a potential line of weakness in the underlying rocks.

The Driekop pipe differs from the other two mentioned above in that

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it shows neither a concentric structure nor differentiation of the dunite into iron-rich and iron-poor fractions. Heckroodt (1959), dealing with the petrography of the rocks in the Driekop area, failed to establish any relationship between the platinum content of the dunite and the com-



FIG. 1. Geological sketch-map of the eastern part of the Bushveld igneous complex (modified after du Toit).

position of the olivine. According to Heckroodt (1958) the composition of the olivine in the ore-body varies from Fo_{71} to Fo_{84} , but does not display any concentric distribution.

Rich parts of the ore-body carry up to 10 p.p.m. platinum. It is thus evident that an efficient investigation of the platinum minerals cannot be undertaken on dunite specimens, but only on mine concentrates.

The ore paragenesis. As the main purpose of this paper is to draw

attention to the new platinoid minerals, the known and easily recognized minerals are but briefly mentioned. The concentrates investigated proved to contain a wide variety of ore minerals. Amongst the sulphides, chalcopyrite and pyrrhotite predominate, while among the platinoid minerals, sperrylite (often in euhedral crystals), $PtSb_2$ (which has not been known before), and native platinum are the most important. Quantitative analysis with the electron microprobe showed that the



FIG. 2. Profile through the Driekop area (after Heckroodt).

native platinum contains an average of 18 % Fe, and may thus be designated as ferroplatinum. Associated with the above-mentioned principal constituents are the following minerals, mostly in small quantities:

Pentlandite, (Fe,Ni)S.

- Valleriite, $Cu_3Fe_4S_7$ (frequently as lamellae and as grains of considerable size in chalcopyrite).
- Covelline, CuS (obviously an alteration product of chalcopyrite).

Braggite, (Pt,Pd,Ni)S.

Cooperite, PtS (pl. XVII, fig. 4).

Laurite, RuS₂. Rather rare, but easily recognized because of its hardness and difficulty in polishing.

Stibiopalladinite, Pd₉Sb, which Ramdohr (1960) considered the most widespread platinoid mineral; this is not confirmed by the present investigation. Some of the new minerals described later, especially PtSb and Pt(Sb,Bi), may frequently have been wrongly identified as stibiopalladinite.

Osmiridium. Extremely hard, silvery-white lamellae, mostly of comparatively large size, in grains of native platinum.

Iridosmine, variety osmite. Easily distinguished from osmiridium because of its distinctly reddish anisotropy colours and a touch of bluish-grey tint in plane polarized light (pl. XVII, fig. 4). The ore-microscopic characteristics of osmite have for the first time been observed by Picot (1961, personal communication), using material the composition of which has been determined by the electron microanalyser.

Native copper. Large independent grains as well as small inclusions in native platinum and sperrylite.

Native gold. Small flakes in some of the platinoid minerals. Chromite. Widespread.

THE NEW MINERALS

No less than ten new minerals were found in the concentrates. Except for a very interesting sub-sulphide of iron and nickel all are compounds of the platinum metals with As, Sb, and Bi.

Minerals with Pt and Sb as main constituents.

Geversite, $PtSb_2$. Reflectivity is lower than that of native platinum, but higher than that of sperrylite. The colour is light grey (pl. XVII, fig. 3). No pleochroism and no anisotropy effects are to be observed; this indicates isotropic character and hence cubic symmetry. The hardness is similar to that of native Pt, perhaps slightly higher.

The microprobe analysis gave Pt 45.0, Sb 51.5 %, corresponding with the formula PtSb₂. Fig. 4 shows the X-ray spectrogram of PtSb₂; obviously this is the antimony equivalent of sperrylite, PtAs₂. The most distinct differences from sperrylite—lower hardness and slightly higher reflectivity—are the results of replacement of As by Sb in the lattice.

The system Pt-Sb has been investigated by Friedrich and Leroux (1909) and by Nemilov and Voronov (1935, 1936); the main results are summarized in Hansen (1958) (see fig. 3). Synthetic PtSb₂ (Thomassen, 1929*a*) is known to crystallize, as sperrylite does, with a pyrite-type lattice, has *a* 6.428 Å. and a melting-point of 1 230° C., and contains 44.47 % Pt. In polished section it looks grey and quite homogeneous, is isotropic, and has a hardness of $4\frac{1}{2}$ to 5. As a microprobe analysis of sperrylite shows, small amounts of Sb can replace As; in pl. XVII, fig. 6, PtAs₂ containing 2.8 % Sb is to be seen. On the other hand, Sb in PtSb₂ can be replaced by Bi, resulting in a slight increase in reflectivity; pl. XVII, fig. 2, shows PtSb₂ with 7.2 % Bi.

PtSb₂ is widespread, occurring in small isolated grains as well as intergrown with other members of the paragenesis. Its widespread association with other Pt-Sb minerals, as shown in pl. XVII, fig. 3, is noteworthy. In certain cases, PtSb and Pt(Sb,Bi) can be observed as very fine myrmekitic inclusions in PtSb₂. Both these features underline the close genetic relationships of the Sb-rich members of the platinum paragenesis. PtSb₂ is generally the earliest of the Pt-Sb minerals, followed by PtSb and Pt(Sb,Bi). This may imply a significant correlation with the melting-points of the minerals which are 1 226° C. for PtSb₂, ~ 1 050° C. for PtSb and probably lower for Pt(Sb,Bi) and which probably indicate an order of crystallization from a magma. PtSb₂ has





FIG. 3. The system Pt-Sb. Ref. 1 refers to Friedrich and Leroux, 1909, and ref. 2 to Nemilov and Voronov, 1935, 1936. (By permission from M. Hansen, 1958, Constitution of binary alloys (McGraw-Hill Book Co.).)

not previously been known to occur as a mineral, and the name geversite (pronounced GE·VERZAIT) is proposed for it, in honour of the eminent South African geologist, Prof. T. W. Gevers.

Pt(Sb,Bi). This mineral has a reflectivity lower than that of native platinum, but, as will be evident from pl. XVII, fig. 3, higher than that of $PtSb_2$. The colour has a distinct pinkish tint. Pleochroism is to be observed easily in air, the colour changing from pink to light brown (in the latter position it is indistinguishable from PtSb). Under crossed nicols, intense anisotropy effects, with pink colours, are to be observed.

In oil immersion both pleochroism and anisotropy are much more pronounced. The hardness is slightly less than that of native platinum. This mineral, especially in very fine grains, may frequently have been mistaken for stibiopalladinité. The intense anisotropy suggests a layer lattice probably of hexagonal symmetry. The fact that both PtBi and PtSb crystallize with nickel-arsenide lattice supports this view. The microprobe analysis gave the following values: Pt 50.5, Sb 26.4,



Bi 15.3 %. This suggests the formula $Pt_7Bi_2Sb_6$, or, simplified, approximately Pt(Sb,Bi). After geversite, this mineral is the most widespread of the Pt-Sb group. It does not, however, occur as independent grains, but mostly associated with $PtSb_2$ and PtSb.

PtSb. The reflectivity of this mineral is lower than that of Pt(Sb,Bi), but still much higher than that of pyrrhotine (for comparison, see pl. XVII, fig. 3). The colour is light brownish with a touch of pink. Pleochroism is absent. Under crossed nicols, anisotropy effects are to be observed, the intensity of which, however, is lower than in Pt(Sb,Bi) or pyrrhotine. The hardness is lower than that of native platinum. Quantitative analysis showed Pt 50.5, Sb 34.7 %. The most likely formula is PtSb. The results of microscopic examination of the mineral and the chemical data are in good agreement. Thomassen (1929b) made synthetic PtSb¹ and described it as a 'yellow mass' in plane polarized reflected light. He found a 4.130 Å., c 5.472 Å., c/a 1.325 (nickel arsenide type lattice).

Minerals containing Pt and (Ir,Os).

 $(Pt, Ir)As_2$. The colour is distinctly greyish, the reflectivity high, but lower than that of $PtSb_2$ (pl. XVII, fig. 2). There is no pleochroism and anisotropy effects are absent. The mineral obviously has cubic symmetry.

¹ It is interesting to note that synthetic PtSb has recently been found to become superconducting at $\sim 2\cdot 1^{\circ}$ K. (Matthias, 1953*a*, 1953*b*). The same applies to the related NiAs-type lattice compounds PtBi ($\sim 1\cdot 21^{\circ}$ K.), PdSb ($\sim 1\cdot 5^{\circ}$ K.), and PdBi ($3\cdot 74^{\circ}$ K.).

Its hardness is higher than that of Pt and PtSb₂, probably around 6 in Mohs' scale. The analysis gave the following results: Pt 47.0, As 46.8, Ir 5.0 %. It is possible that this mineral should be considered as an iridian sperrylite, though Ir has not hitherto been recorded in sperrylite. The analysis results can be fitted approximately into the PtAs₂ formula, and a certain degree of replacement of Pt by Ir might well be possible.

 $Pt(Ir,Os)_2As_4$. The typical feature of this mineral is a distinct touch of brownish colour in reflected light. Its reflectivity is similar to that of sperrylite, and lower than that of Ir-rich PtAs₂. The hardness is higher than that of Ir-rich PtAs₂. Increasing amounts of Ir and Os are obviously responsible for the increase in hardness. Examination of the mineral in both plane polarized light and under crossed nicols did not reveal any signs of anisotropy, again indicating cubic symmetry. Lamellae of native osmium (iridosmine, var. osmite) are not infrequently to be observed within this mineral (pl. XVII, fig. 1). It is difficult to decide whether these are due to decomposition or to unmixing processes.

The analysis gave Pt 22.0, As 32.5 %, and considerable amounts of iridium and osmium are also present. A quantitative determination of the percentages of Ir and Os was not possible as the microprobe analyser revealed remarkable changes in the content of these two elements within a few microns distance. The Ir and Os values recorded along a traverse from A to B are to be seen in fig. 6. The complete mutual replaceability of these two elements is very well demonstrated by high Ir values corresponding to low Os values, and vice versa. The observable optical properties of the minerals are not affected by these changes in chemical composition, as is evident from pl. XVII, fig. 2. The great similarity in atomic radius and chemical properties of these two elements makes it understandable that variations in their relative proportions would not appreciably influence the physical properties of the mineral concerned. Nevertheless, if one considers Ir and Os as the only probable constituents besides Pt and As, then Pt(Ir,Os)₂As₄ becomes likely as a formula.

Generally these Ir- and Os-rich minerals are by no means as widespread in the concentrates as, for instance, the Pt–Sb compounds. They occur mostly together in anhedral grains. Frequently $Pt(Ir,Os)_2As_4$ is to be observed as lamellae in native platinum as well (pl. XVII, fig. 1).

Mineral with Pt, Cu, and Sn.

This mineral is extremely rare and is difficult to recognize under the microscope. Its reflectivity is very high, but still distinctly lower than

that of native Pt, and slightly lower than that of Bi-rich $PtSb_2$. It has a touch of pink, similar to PtSb. Under crossed nicols, in oil immersion, anisotropy without special colour effects is visible.

The hardness is lower than that of PtSb, and not very different from that of native platinum. Quantitative analysis yielded unexpected results: Pt 51.7, Sn 22.0, Cu 16.8 %. The only natural association of the two elements Pt and Sn that has hitherto been recorded is in niggliite, Pt₂Sn₃. Pt-Sn alloys with varying Pt-Sn ratios are reported in Mellor (1937) as having been prepared by A. F. Gehlen, and in Thorpe (1950), viz. Pt₃Sn₈, Pt₂Sn₃, PtSn, Pt₃Sn. The formula calculated from the above values is $Pt_4Sn_2Cu_4$. It is interesting to note the occurrence of a second mineral containing Sn at the 'basic end' of the differentiation sequence. The presence of small amounts of Sn in platinum metal concentrates from the Sudbury ores has been proved by chemical analysis (personal communication, Dr. B. Taylor, Acton Platinum Metals Refinery of the International Nickel Co., Ltd.). It may seem remarkable that tin has not previously been noted in the rocks concerned but it must be realized that Pt₄Sn₃Cu₄ is very rare even in relation to the other platinoid minerals. The total platinoid mineral content is, as previously noted, only about 10 p.p.m.

Palladium minerals.

 Pd_2CuSb . Characteristic features are its strong violet colour and a reflectivity much lower than all the other minerals of the paragenetic association (pl. XVII, fig. 5). They are, as usual, more pronounced in oil immersion. Under crossed nicols, good anisotropy effects with orange colours are to be observed. The hardness is not very different from that of native platinum. The analysis gave the following results: Pd 53.5, Sb 31.3, Cu 16.0 %. This corresponds with the formula Pd₂CuSb. Any close similarity with stibiopalladinite is ruled out by both optical and chemical data. Natural substances of this composition have not so far been recorded. It is one of the rarest constituents in the concentrates, always occurring in intergrowths with other platinum minerals.

 Pd_8CuSb_3 . Intergrown or closely associated with the above mineral there occur grains and lamellae of higher reflectivity and a pale yellowish colour (pl. XVII, fig. 5). Anisotropy effects are well pronounced, especially in oil, but without recognizable colour. The reflectivity is still slightly lower than that of geversite, as may be seen in pl. XVII, fig. 5. The hardness is similar to that of native platinum. Quantitative

micro-probe analysis gave the following results: Pd 62-0, Sb 30-0, Pt 6-0, Cu 5-0 %. These values correspond with the formula Pd_8CuSb_3 . Small amounts of Pd may be replaced by Pt. These two palladium minerals are obviously closely related, as indicated by the similarities in chemical composition as well as by their frequent mutual intergrowth.

Pd(Sb,Bi): a light yellowish mineral with high reflectivity and not showing so pale a colour as Pd_8CuSb_3 does. No pleochroism, but anisotropy effects of medium intensity with gold-yellow colours under crossed nicols. Hardness is distinctly lower than PtAs₂ (pl. XVII, fig. 6). The microprobe analysis gave the following results: Pd 43.6, Sb 25.2, Bi 32.2 %, suggesting the formula $Pd_8Sb_4Bi_3$. Simplified, this formula could be given approximately as Pd(Sb,Bi) with either an excess in Pd or a deficiency in (Sb,Bi). Accordingly, this mineral might be considered as the palladium equivalent of Pt(Sb,Bi), probably crystallizing with a hexagonal lattice. Thomassen (1928) reported synthetic PdSb as crystallizing with NiAs-type lattice (a 4.070 Å., c 5.582 Å., D 9.4); PdBi, according to Zhuravlev (1957), is orthorhombic with a 7.203, b 8.707, c 10.662 Å.

Pd(Sb,Bi) does not occur as independent grains, but only intergrown with Pt-arsenides and other Pt-minerals, sometimes in complex textures, (pl. XVII, fig. 6).

Nickel-iron subsulphide.

 $(Fe, Ni)_2S$. The occurrence in small grains, intergrown with other members of the paragenesis, of a mineral resembling pyrrhotine in colour is worthy of note. Its reflectivity, however, is slightly higher than that of pyrrhotine (pl. XVII, fig. 2). Under crossed nicols, anisotropy effects without colours are observed. The hardness is comparatively low. The chemical composition is Fe 51.0, Co 1.0, Ni 28.0, S 22.5 %, corresponding with the formula (Fe,Ni)₉S. A section of the X-ray spectrogram is shown in fig. 5, indicating only the metallic elements. It must be considered whether small amounts of sulphur available in the magma were responsible for the formation of this unusual mineral. Mellor (1936) mentions the synthetic preparation of nickel hemisulphide, N₁₂S, by heating nickel sulphate in a current of hydrogen. He further states, following Rose, that 'the pale yellow or brass yellow mass is not magnetic'. Similarly strong reducing conditions have to be postulated for the formation of (Fe,Ni)₂S. The presence of free hydrogen in ultrabasic rocks has been proved, for instance by Hahn-Weinheimer (1961). Finely dispersed native platinum might act as catalyst.

Ramdohr (1949) described a paragenesis of nickel sulphides at Trial Harbour, Tasmania. He found increasing amounts of sulphur present with decreasing temperature, covering the range from awaruite (sulphur:metal = 0) to pentlandite (sulphur:metal = 8:9). The possibility of (Fe,Ni)₂S being an intermediate stage in this range has to be considered.



Pl. XVII, fig. 2, shows $(Fe,Ni)_2S$ to be one of the earliest crystallized minerals, being surrounded by the whole sequence of later-formed phases.

Conclusions.

There can be no doubt that the substances described are new minerals. The author feels, however, that with the exception of geversite they should not be named until further investigations, which are being continued, have made it possible to separate amounts sufficient for X-ray analysis and the calculation of cell dimensions. Moreover, comparison with synthetically prepared substances might provide further data. The mineralogical description raised many problems, such as paragenetical and



F10. 6. The variation in Ir and Os concentrations within a single grain of $Pt(Ir,Os)_2As_4$; the traverse is from A to B in the grain marked in pl. XVII, fig. 2.



FIG. 7. The variation in platinum concentration along a traverse from A to B on the section shown in pl. XVII, fig. 3.

EXPLANATION OF PLATE XVII.

FIG. 1. Lamellae of $Pt(Ir,Os)_2As_4$, medium grey, in native Pt (white). Native osmium (osmite) light grey, in the $Pt(Ir,Os)_2As_4$. Oil immersion, $\times 200$.

FIG. 2. $(Fe,Ni)_2S(1)$; native Pt (2); PtSb₂ containing 7.2% Bi (3); (Pt,Ir)As₂ (4); Pt(Ir,Os)₂As₄ (5). Oil immersion, $\times 360$. The very small differences in reflectivity between the different minerals have been enhanced by special printing. Text-fig. 6 (*opposite*) shows the variation in Ir and Os from a to B, within a grain of Pt(Ir,Os)₂As₄.

FIG. 3. Native platinum, white; PtSb₂, Pt(Sb,Bi), lightest grey, PtSb; corre-

Plate XVII



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age relations, which are deferred for the time being. The comparatively simple constitution of most of the new minerals described is remarkable. There is perfect agreement with some of the data available on Pt–Sb alloys. For future work on those minerals, however, that cannot be fitted into binary systems, greater difficulties are envisaged.

Acknowledgements. I am deeply indebted to Dr. C. Guillemin, in charge of the mineralogical section of the Bureau de Recherches géologiques et minières, Paris, for putting their electron microprobe at my disposal. Without Dr. Guillemin's help and generous hospitality, and the enthusiastic co-operation of MM. Phan, Picot, Bahezre, and Giraud, these investigations could not have been brought to a successful conclusion. Monsieur Bahezre kindly carried out the correction calculations for the microprobe analyses. I am grateful to Prof. P. Ramdohr, who, when I was working under him at Heidelberg, arranged for me to accompany him on his journey to South Africa in 1957 and made it possible for me to spend a few extra days at the Driekop platinum mine, where I appreciated very much the help of Mr. G. Onken, mine manager. My thanks are due to Prof. S. E. Hollingworth, Dr. E. A. Vincent, and Dr. J. R. Butler, who critically read the manuscript, and to the Direction Générale, bureau de Récherches géologiques et minières, Paris, for granting permission to publish those results that were obtained in their laboratories.

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sponding to traverse in text-fig. 7 opposite. $CuFeS_2$, medium grey grain left of centre; FeS, dark grey grain near B. Oil immersion, $\times 220$.

- FIG. 5. Native Pt, white; PtSb₂, lightest grey; Pd₈CuSb₃, light grey; Pd₂CuSb, medium grey. Oil immersion, × 360.
- FIG. 6. Pd(Sb,Bi), medium grey; PtAs₂, light grey, contains 2.8 % Sb. In centre small grain containing Ir 10, Pt 18, As 50 %. ×100.

FIG. 4. Native platinum, white; sperrylite, light grey; cooperite, dark grey. Oil immersion, ×460.

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TABLE I. Chemical and optical data for the new platinoid minerals and for (Fe,Ni)₂S.

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Addendum: The author's attention has just been directed to three unnamed new minerals described by I. B. Borovsky, A. N. Deev, and I. D. Marchukova ([И.Б. Боровский, А. Н. Деев, и И. Д. Марчукова, Геол. Рудн. Месторожд., 1959, no. 6, p. 28]; abstr. in Amer. Min., 1961, vol. 46, p. 446) and by A. D. Genkin (A. Д. Генкин, ibid., p. 74). None of these minerals approximates in composition to any of those described above.