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Potarite, a new mineral discovered by the late Sir John Harrison in British Guiana.

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[Read January 17, 1928.]

THE untimely death of Sir John B. Harrison[1] on February 8, 1926, just as he was returning home after over forty-six years' service in the West Indian colonies, unfortunately prevented the publication by him of a complete account of the interesting new mineral which, as a result of much patient chemical work, he had discovered in British Guiana. As he had corresponded with me on this matter since April, 1924, and further, as this is the same mineral that I had myself erroneously described as 'allopalladium',[2] I feel it my duty to place on record the full facts so far as can now be ascertained.

I had suggested that the new mineral might be named after Sir John Harrison, but this he objected to; and, in consultation with the Governor of British Guiana, he proposed the name potarite (letter of April 8, 1925), but he did not himself record this name in print. In 1925, at his

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[1] Sir John Burchmore Harrison (1856–1926). Obituary notices in Bull. Geol. Soc. Amer., 1927, vol. 38, pp. 45–52, with portrait and bibliography; and Min. Mag., 1927, vol. 21, p. 234.

[2] L. J. Spencer, Allopalladium from British Guiana. Min. Mag., 1924, vol. 20, pp. 217–219.

suggestion, and with the approval of the Governor, specimens of the new mineral labelled as potarite, from the small stock totalling about 40 grams, were generously presented by the Government of British Guiana to various scientific collections, namely to the British Museum, the Mineralogical Museum of Cambridge University, the Muséum d'Histoire Naturelle in Paris, and the United States National Museum in Washington, D.C. The nugget weighing 3·2 grams sent to the Paris museum was shown by Prof. A. Lacroix at a meeting of the French Mineralogical Society on January 14, 1926, and the name potarite appeared in the report of that meeting.[1] It also appeared in the annual reports of the Cambridge Museums[2] and of the United States National Museum.[3] But the earliest printed record of the name appears to be in 'The West India Committee Circular', London, October 22, 1925 (p. 429). The name potarite is also incidentally mentioned by Mr. H. J. C. Conolly, Economic Geologist and Mineralogist of British Guiana, in his 'Report of the preliminary survey of the Mazaruni and Puruni diamond fields, British Guiana',[4] and by Dr. G. F. Kunz in his annual report on platinum metals.[5]

The publications of Sir John Harrison relating to this mineral appeared as Government Notices in the 'Official Gazette' of British Guiana under the titles:

'An occurrence of palladium and rhodium in British Guiana.' (May 3, 1924, no. 181, 2 pp.)

'The occurrence of palladium amalgam—palladium mercuride—in British Guiana.' With C. L. C. Bourne. (February 28, 1925, no. 71, 3 pp.)

Copies of these papers were sent out by Sir John Harrison, and notices of them appeared as abstracts in certain scientific journals,[6] though without any mention of the name potarite.

The name potarite is from the Potaro river, a left tributary of the Essequibo in British Guiana, the only source of the mineral so far known

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[1] Bull. Soc. Franç. Min., 1926, vol. 49, p. 5.

[2] Cambridge University Reporter, March 17, 1926, vol. 56, p. 778.

[3] Report of the United States National Museum for the year ended June 30, 1926, 1927, pp. 84, 130.

[4] London, [1926], pp. 6, 7 [Min. Abstr., vol. 3, p. 438]. From this source the name potarite appeared in Bull. Imp. Inst. London, 1927, vol. 24 (for 1926), p. 776 (compare also p. 767).

[5] G. F. Kunz, Mineral Industry, New York, 1927, vol. 35 (for 1926), p. 548.

[6] Journ. Chem. Soc. London, 1925, vol. 128, Abstracts, pt. ii, pp. 592–593; Chem. News, London, 1925, vol. 131, pp. 88-89, 1926, vol. 132, p. 124; Chem. Abstr. (Amer. Chem. Soc.), 1925, vol. 19, p. 3075; Amer. Min., 1925, vol. 10, p. 333; Min. Abstr., 1926, vol. 3, p. 4.

being in the neighbourhood of the gorge below the Kaieteur falls.[1] A single small grain of platinum was found there by Mr. J. C. Menzies in 1923,[2] and soon afterwards, while prospecting near Oewang and Tukeit in the same neighbourhood, he found other minute white metallic grains. These were incorrectly described in 1924 by me as 'allopalladium' and by Sir John Harrison as palladium and rhodium. Since then a white metal has been found by several of the diamond-washers in relatively larger, though still very scanty, amounts in the Kangaruma district, situated about 16 miles from the place of the first find, and also in the Amatuk district.[3] This supplied Sir John with more material for investigation and he was able definitely to establish the mineral as a compound of palladium and mercury.

With the very small amount of material (a few milligrams) at first available it is not surprising that mercury was overlooked. In order to save at least some of the material for preservation in the collection, I had applied only micro-chemical tests. In one case, however, Sir John had noticed the presence of mercury, but he concluded that this had been introduced by the diamond-washers when saving gold, which is also present in the diamond-bearing gravels. Mercury had not then been recorded from British Guiana, but curiously, while engaged on this work in December, 1924, Sir John had brought to him a small specimen of cinnabar[4] from a lateritic clay in the Kurupung district on the Mazaruni river (about 100 miles to the north-west). Further, it is to be noted that, as Sir John afterwards found out, the diamond-washers had in some cases heated the metal, thinking at first that they had a gold-amalgam, and afterwards testing the fusibility before the blowpipe for platinum. The result was that some of the material had lost much of its mercury and was not in a natural condition.

The spurting noticed on the surface when the metal was heated was at first ascribed to the liberation of occluded hydrogen and other gases in the palladium. With larger fragments a more exact determination of the specific gravity was possible, and values ranging from 13·48 to 16·11 were

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[1] The famous Kaieteur falls have a vertical drop of 741 feet, nearly five times the height of Niagara.

[2] L. J. Spencer, Euclase and platinum from diamond-washings in British Guiana. Min. Mag., 1924, vol. 20, pp. 186–192.

[3] The locality of the specimen sent to the Paris museum is given as Amu creek (Bull. Soc. Franç. Min., 1926, vol. 49, p. 5).

[4] A similar occurrence of cinnabar in laterite has recently been described from Dutch Guiana by C. Palache, Amer. Min., 1927, vol. 12, p. 188 [Min. Abstr., vol. 3, p. 359].

obtained, much higher than that of palladium (11·97). The average specific gravity from eleven determinations was 14·8. It was thought that perhaps this difference might be accounted for by the two modifications of palladium—the ordinary malleable cubic palladium and the alleged brittle hexagonal allopalladium. Experiments were therefore made with the idea of converting the latter into the former by heating up to temperatures of about 1400° C. But when the specific gravity of the heated material was determined it was found that there had been a considerable loss in weight. This was puzzling and was at first ascribed to osmium, but very soon this loss, ranging from 54·4 to 65·2 %, was found to be due to mercury. After being purified by two distillations, the mercury expelled from potarite was found to have a density of 13·6. The palladium left after repeated heatings was porous, with specific gravity 10·0–11·0; but after hammering the metal, the specific gravity was determined as 11·33–11·9.

The actual determinations given in Sir John Harrison's second report are as follows, the material being heated in vessels of silica-glass:

	Weight in grams.	Sp. gr.	Percentage loss in weight (Hg).
No. 1. fragment, heated for successive periods at increasing temperatures up to nearly 1400° C.—			
Original	0·529 ...	15·82 ...	—
After 1st heating	0·325 ...	14·13 ...	38·6
,, 2nd ,, ...	0·290 ...	14·08 ...	45·2
,, 3rd ,, ...	0·220 ...	13·92 ...	58·4
,, 4th ,, ...	0·190 ...	11·31 ...	64·1
No. 2, several small fragments, heated over blast-lamp for half-hour periods.—			
Original	0·978 ...	15·00 ...	—
After 1st heating	0·649 ...	14·11 ...	33·6
,, 2nd ,, ...	0·410 ...	10·78 ...	58·1
,, 3rd ,, ...	0·340 ...	10·39 ...	65·2
and hammering	— ...	11·33 ...	—
No. 3, fragment, heated over blast-lamp for one-hour periods.—			
Original	1·303 ...	13·48 ...	—
After 1st heating	0·927 ...	12·20 ...	28·8
,, 2nd ,, ...	0·594 ...	10·42 ...	54·4
,, 3rd ,, ...	0·594 ...	— ...	54·4
and hammering	— ...	11·4 ...	—
No. 4, nugget, heated over Méker burner (about 1400° C.) for one-hour periods.—			
Original	2·197 ...	15·15 ...	—
After 1st heating	1·102 ...	10·02 ...	49·9
,, 2nd ,, ...	1·007 ...	10·71 ...	54·2
,, 3rd ,, ...	1·001 ...	11·0 ...	54·4
,, 4th ,, ...	0·990 ...	11·0 ...	54·9
and hammering	— ...	11·9 ...	—

These results were collected together in the following manner:

	Sp. gr.	Pd (with traces of Rh, Pt, Au).	Hg.
1.	15·82	35·9	64·1
2.	15·00	34·8	65·2
3.	13·48	45·6	54·4
4.	15·15	45·1	54·9

As noted above, some at least of the specimens had been heated before being received for scientific examination, and we may therefore take Sir John's higher values as representing the true mercury percentage. But there is an inexplicable variation in the specific gravity between (1) and (2), as also between (3) and (4), although these are in pairs apparently the same in chemical composition. The high values of the specific gravity up to 16·11 (except 13·48) also present an anomaly, for they are much higher than the density of either of the components, namely 11·97 for palladium and 13·60 for mercury (or 14·19 for solid mercury). This high degree of 'condensation' was puzzling to Sir John Harrison, but it no doubt indicates that the mineral is a definite compound of palladium and mercury rather than an amalgam. The formula PdHg corresponds with the percentage composition Pd 34·7, Hg 65·3, agreeing closely with the determined values (1) and (2). The close agreement between (3) and (4) perhaps suggests another compound Pd_3Hg_2, which corresponds with Pd 44·4, Hg 55·6 %.

A remarkable fact is the difficulty with which the mercury is expelled from the mineral, prolonged and repeated heating at a high temperature being necessary. Also, as found by Sir John Harrison, the mercury is not again readily taken up by the palladium. In his letter of January 9, 1925, he wrote:

> After we have driven off all the liquid metal from the alloy we cannot get the resulting porous palladium to readily take up a new supply of ordinary mercury.

and in his letter of November 25, 1925:

> In the hopes of artificially preparing an amalgam of palladium and mercury we left the porous fragment mentioned in my letter of January 9th in contact with an excess of mercury from the end of December, 1924, to November 12th, 1925. By the latter time the greater part of the porous palladium had dissolved in the large excess of mercury. We separated the liquid amalgam of palladium and mercury and we exposed it during many hours in an open silica tube surrounded by the vapour of boiling mercury. Very slowly indeed did the amalgam give off its excess of mercury, but finally we got a small quantity of a solid metal closely resembling in external properties our native palladium mercuride. The specific gravity of that solid metal was a little under 14·3, that

is, it had practically the density of solid mercury. We next analysed this artificial 'potarite' and found it to contain 36·8 % palladium and 63·2 % mercury. that is, this palladium mercuride had practically the composition Pd_2Hg_2. It is evident, however, that our artificial palladium mercuride has not the high state of condensation of the native mineral 'potarite'.

There appears to be little definite information in the literature about palladium amalgams (although these have been used in dentistry). In H. Watts's 'Dictionary of chemistry' (1865, vol. 3, p. 888) is stated: 'The amalgam containing Pd_2Hg is a grey powder, which, according to Berzelius,[1] does not give off its mercury below a white heat.'

The two largest nuggets (weighing about 12 and 11 grams) of potarite that have been found were kindly sent by Sir John Harrison in 1925 for my inspection, with the instruction to select one for the British Museum collection, the other to go to the Mineralogical Museum at Cambridge, as presents from the Government of British Guiana. Both these nuggets came from the diamond-washings in the Kangaruma district on the Potaro river. The smaller nugget is flatter, has a wider spread, and is brighter and more attractive in appearance. After some hesitation, I selected the slightly heavier nugget because it shows some crystalline structure on the surface.

This nugget weighs 12·074 grams and measures $2 \times 1\frac{1}{2} \times \frac{1}{2}$ cm. with an irregular indented outline. After scrubbing off a small amount of attached white clayey matter, the specific gravity was found by hydrostatic weighing to be 14·924, and when corrected $D_4^{20} = 14·88$. The value given by Sir John Harrison in his second paper of February, 1925, is 14·78; and in his letter of April 15, 1925, he mentions that this nugget, acquired from Mr. W. A. Semple, had previously been offered to a jeweller, who admitted that he had heated it in places before the blowpipe to test its fusibility. This evidently accounts for the lower specific gravity as compared with the higher values noted above.

This specimen shows a somewhat indistinct columnar or fibrous structure, the columns being roughly across the plate and slightly divergent. On one side the columns terminate in a confused aggregate of octahedral points, and on the opposite surface there is a reticulate crystalline pattern. The appearance is that of a fragment of a crystalline crust that has

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[1] J. Berzelius, Kongl. Vetensk. Acad. Handl. Stockholm, 1813, p. 205; translations in Journ. Chem. u. Physik (Schweigger), 1813, vol. 7, p. 67, and Annales de Chimie, 1813, vol. 87, p. 139. This amalgam contained 0·7073 gr. of palladium and 0·7277 gr. of mercury [50·7 %]; of the latter, 0·112 gr. was lost over a spirit-lamp, and 0·6157 gr. during exposure for $\frac{1}{2}$ hour to the highest white-heat.

become detached and only slightly water-worn. The octahedral points measure about 1 mm. across and the triangular facets are uneven and rounded, so that only approximate measurements could be obtained of the maximum light reflections on the goniometer. Values ranging from 63° to 77° suggest the octahedral angle 70° 32′. We can therefore conclude that the mineral is cubic in crystallization. Sir John Harrison had previously remarked on the indications of a crystalline and fibrous[1] structure in some of the nuggets and fragments; and after the material has been heated to expel the mercury the fibrous structure is still more evident.

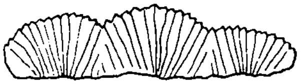

FIG. 1. Diagrammatic section of nugget of Potarite.

The hardness is 3½, the nugget scratching calcite but leaving a metallic mark on fluorite. A minute fragment broken off (rather than cut) with a knife was crushed to powder in an agate-mortar, but when rubbed it streaked out somewhat on the agate. The colour of the nugget and of the streak is silver-white with bright metallic lustre.

The suggestion was made by Sir John Harrison that I should test my 'allopalladium' from British Guiana for mercury. A minute fragment broken with a knife from the original material was found to be brittle when hammered on an anvil. The powder was placed in a glass bulb-tube and heated in a bunsen-flame, when I was surprised to see with a lens an abundant sublimate of minute globules of mercury. The result of this test has already been mentioned in a foot-note in the abstract[2] of Sir John Harrison's papers, at the same time remarking: 'The doubt now passes on to the original allopalladium from the Harz Mts.' The 'cleavage' was perhaps imaginary; in my note-book I had written: 'Examined under the metallographic microscope the fragments are silver-white with bright reflections as if from cleavages.' These perhaps represent the surfaces of separation between the several grains of the aggregate.

As mentioned in my previous paper, there is no specimen of allopalladium in the British Museum collection. There are, however, in the collection a number of specimens from Tilkerode, in the Harz Mts., under the species gold, clausthalite (PbSe), tiemannite (HgSe), naumannite, onofrite, and zorgite. All these I carefully searched with a magnifying lens for any possible allopalladium, though not with a 'compound

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[1] He remarked that a fibrous structure has sometimes been described for the Brazilian native palladium, and he suggested that some of this might possibly be potarite.

[2] Min. Abstr., 1926, vol. 3, p. 5.

microscope' as was used by Zincken. Specimens of allopalladium from the collection of J. C. L. Zincken (1790–1862) are preserved in the Museum für Naturkunde in the University of Berlin, and Prof. M. Belowsky informs me that it requires some patience to find and see the minute scales of allopalladium on the gold of these specimens. Shortly before my inquiry a small scale of allopalladium had been sent from the Berlin collection to Prof. V. M. Goldschmidt of Oslo for examination in connexion with his work on the platinum metals. He tells me that the amount of material (a fraction of a milligram) is too small even to test for mercury by the new X-ray method of spectrum-analysis.

Palladium has undoubtedly been found in the Harz Mountains, although the 'allopalladium' seems to be doubtful. It was found during the chemical extraction of selenium[1] on a moderately large scale at Tilkerode[2] in the eastern Harz from the ores which carry gold, silver, lead, mercury, and copper. Zincken[3] in 1829 examined this palladium and he traced its source[4] to the mineral which he described as selenide of palladium under the name 'Selenpalladium'. Soon afterwards, however, he told Berzelius in conversation that on re-examination the mineral had proved to be native palladium.[5]

Zincken isolated the mineral by dissolving in acid specimens of dolomite containing clausthalite (PbSe) and dendritic flakes of gold. The silver-white metallic scales, found on the surface of the gold, were so minute that they floated on water and were scarcely visible to the unaided eye. Under the microscope they were seen to be six-sided plates, and, comparing them with iridosmine, he stated that there was a perfect platy cleavage perpendicular to the axis of the prism. Being brittle, he concluded that they could not consist of pure metal. When

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[1] A small medallion (3 × 2·2 cm. ; 11·56 grams) of selenium, showing in relief a bust of Berzelius, came into the British Museum collection at about that time.

[2] O. Luedecke (Die Minerale des Harzes. Berlin, 1896, p. 6) also gives Zorge as a locality for allopalladium, but on what authority is not stated.

[3] C. Zincken, Ueber das Palladium im Herzogthum Anhalt-Bernburg. Ann. Phys. Chem. (Poggendorff), 1829, vol. 16, pp. 491–498. Abridged translation : C. Zinken, Sur le palladium trouvé dans le duché de Anhalt-Bernburg. Ann. Chim. Phys., 1830, ser. 2, vol. 44, pp. 206–213. The amount of palladium is not stated, but of several beads, one used for determining the specific gravity weighed 1·849 grams.

[4] A somewhat similar case is given by the presence of traces of palladium in the nickel-copper ores of Sudbury in Ontario ; this being the chief commercial source of the metal at the present day. But in this case the form in which the palladium occurs is not yet known.

[5] J. Berzelius, Jahres-Bericht über die Fortschritte der Physischen Wissenschaften (German translation by F. Wöhler), 1832, vol. 11, p. 202.

roasted in a glass tube a smell similar to that given by mercury lead selenide was noticed and a red ring of selenium was obtained. Chemical tests in the wet way showed the presence of palladium, lead, and silver in the material after roasting. No further description of the mineral was given. But later, in 1842, he gave a brief account of 'new selenium compounds'[1] from the Tilkerode ores. One of these, which he named 'eugenesite', was described as being silver-white, granular or scaly, and containing palladium, silver, gold, and selenium, the last possibly present as admixed lead selenide. No mention is made of the relation of this mineral to his previously described 'Selenpalladium'.

It is interesting to trace the vicissitudes of Zincken's mineral in the treatises on mineralogy,[2] which have copied one from another without adding any further information. At first under the name Selenpalladium (Selenpalladite of Dana, 1837) it was placed amongst the selenides and sulphides in the family of glances. Afterwards, when Zincken admitted that it was palladium rather than selenide of palladium, it was included under native palladium (regarded as cubic). Dana in the fifth edition of his 'System' (1868) again raised it to the rank of a species with the new name Allopalladium.

Zincken's description is very inadequate and unconvincing for establishing a mineral species, and although very nearly a century has elapsed there has been no confirmation of his results. The existence of a hexagonal modification of palladium has not even been suggested by the several examinations of the crystal-structure by X-ray methods. We must therefore conclude that 'allopalladium' 'Selenpalladium' is either (1) ordinary cubic palladium or (2) possibly potarite. If the former, the hexagonal outline of the scales presents no difficulty, for this is common enough in gold and other cubic metals. The 'cleavage' may have been imaginary, influenced by the comparison with iridosmine, and in any case it would be difficult to determine with certainty in such minute crystals. G. Rose[3] in his paper on the dimorphism of palladium (1842) added no new observations. In favour of (2) we have the association of mercury

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[1] C. Zincken. Berg- und Hüttenm. Zeitung, 1842, vol. 1, col. 400. The name Eugenesite he had published the previous year in Ber. Naturwiss. Ver. des Harzes for 1840–41, col. 5.

[2] The large collection of treatises and text-books on descriptive mineralogy, almost complete in all editions, in the Library of the Mineral Department of the British Museum, is arranged chronologically, and so facilitates historical research.

[3] G. Rose, Ueber die Dimorphie des Palladiums. Ann. Phys. Chem. (Poggendorff), 1842, vol. 55, pp. 329–331.

minerals (tiemannite and onofrite); but, on the other hand, Zincken would surely have noticed a sublimate of mercury when he roasted the mineral in a glass tube. Without an opportunity of examining the original material, the evidence then appears to be in favour of regarding Zincken's mineral as ordinary cubic palladium.

Conclusions.—Potarite (J. B. Harrison, 1925) from the diamond-washings on the Potaro river in British Guiana is a definite compound of palladium and mercury in equal atomic proportions, PdHg, crystallizing as octahedra in the cubic system. Sp. gr. 15·0–16·1; hardness 3½, brittle. Soluble in nitric acid to a brown solution.

'Allopalladium' from British Guiana (L. J. Spencer, 1924) is potarite. The original allopalladium or 'Selenpalladium' from the Harz (C. Zincken, 1829) is probably ordinary cubic palladium: the supposed hexagonal modification of palladium probably having no existence.

Addendum.—Smith Bracewell in his recent 'Report on the preliminary geological survey of the Potaro-Ireng district of British Guiana', Georgetown, Demerara, 1927, Combined Court no. 21, 60 pp., maps [Min. Abstr., vol. 3, p. 437] mentions this mineral under palladium (pp. 5 and 55) but without giving the name potarite. It has been found in small amounts over an extensive area in the Potaro district, and some further localities are noted. A conical fragment showing a radial aggregation of small crystals and weighing 2·61 grams, with specific gravity 15·00, has recently been found in the Kaieteur gorge.