

LXXVIII.—*Stibiotantalite. A New Mineral.*

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IN February last, Mr. Knox Brown, of Bunbury, Western Australia, brought to Mr. J. J. East, F.G.S., the Registrar of the Adelaide School of Mines, samples from the alluvial tin-field at Greenbushes, Western Australia.

Although the ore, dressed for market, appeared to the miners in the district to be free from gangue, the assay values on different samples sent to Melbourne varied most unaccountably. In looking over the samples, Mr. East noticed among the tin ore several specimens which appeared to differ in some respects from cassiterite; these had been called by the local miners "resin tin" and "pale tin." On being submitted to a preliminary examination, they were found to contain a good deal of antimony, but appeared to be unlike any of the known antimony minerals. Some of the specimens were handed to me for analysis by Mr. East.

Qualitative Analysis.—The powdered mineral appearing to be insoluble in the ordinary acids, a portion was fused with caustic potash in a nickel crucible, extracted with water, and filtered; on commencing to wash the precipitate, the filtrate, previously clear, came through turbid, but became clear again as it passed into the bulk of the filtrate. The clear filtrate was acidified with hydrochloric acid and the white precipitate (*a*) produced filtered and washed with water.

A portion of (*a*) was ignited and weighed in a platinum dish, heated with hydrofluoric and sulphuric acids, evaporated, ignited, and again weighed, but the weight was the same as at first; part of the residue tested by fusion in a bead of sodium carbonate caused effervescence, and eventually gave a bead, when hot, clear and colourless, but, on cooling, crystallising with slight reincandescence to an opaque, colourless bead.

Another portion of (*a*) was fused with potassium bisulphate, extracted with cold water, decanted and filtered; the filtrate, when largely diluted and boiled for some time, gave no precipitate. The residue insoluble in water was then thoroughly washed with boiling water and digested with boiling hydrochloric acid, in which it appeared to be quite insoluble, but a portion of it, when boiled with concentrated sulphuric acid, dissolved slowly, and when poured into a large excess of water, was reprecipitated.

As these reactions pointed to the presence of tantalic and niobic acids, the remainder of (*a*) was dissolved in hydrofluoric acid, the

excess of acid evaporated, the residue diluted with water, and a portion tested with tincture of galls, which gave a yellow precipitate. Another portion of the solution gave a yellow precipitate with potassium ferrocyanide. To the remainder of the solution potassium fluoride was added, and the solution, when gently warmed and concentrated, gave an acicular crystalline precipitate of potassium fluotantalate (K_2TaF_7); the filtrate from this gave an orange precipitate with tincture of galls, thus proving the presence of niobic acid. Further confirmation of the presence of these two acids was obtained by the blowpipe and wet reactions.

Examination by the usual methods indicated much antimony, with traces of bismuth, nickel, iron, and manganese.

Quantitative Analysis.—The sample was crushed and levigated with water and dried in the water-oven. 1 gram was taken and fused in a platinum crucible with 4 grams of potassium bisulphate, the fused mass treated frequently with boiling water and decanted, thrown on a filter, and washed. The filtrate was found to contain a trace of antimony (which was separated and added to the main precipitate afterwards obtained) and a trace of iron, but no titanous acid. The precipitate was digested with cold yellow ammonium sulphide, with frequent agitation, then decanted, the operation repeated, and the residue thrown on a filter and washed with yellow ammonium sulphide and water. The residue was again washed into a beaker and boiled with hydrochloric acid till it became white, then filtered, washed, and ignited in a muffle to constant weight; it weighed 0.5976 gram. The acids were again fused with potassium bisulphate and exhausted with boiling water and afterwards boiled with hydrochloric acid mixed with a little tartaric acid; the latter solution, after filtration, yielding 0.0107 gram of antimony oxide (Sb_2O_3); this was therefore deducted from the previous weight, leaving 0.5869 gram as the weight of the acids. Part of residue suffered no loss of weight on evaporating and igniting with hydrofluoric and sulphuric acids.

An attempt was then made to separate the niobic from the tantalic acid by precipitation of the latter as potassium fluotantalate, but, although a partial separation was effected, it did not appear possible, with the limited material in hand, to obtain sufficient knowledge of the special precautions necessary to obtain a trustworthy result.

It was, therefore, decided to calculate the proportions of the two acids from the quantity of antimony oxide with which they had been combined, the oxide of bismuth present being calculated into oxide of antimony; for the calculation the following data was used :—

$$\begin{array}{rcl} \text{Sb}_2\text{O}_3 \text{ 40.23} + (\text{Bi}_2\text{O}_3 \text{ 0.82 p. c.} = \text{Sb}_2\text{O}_3 \text{ 0.51 p. c.}) & = & 40.74 \text{ p. c.} \\ \text{Ta}_2\text{O}_5 + \text{Nb}_2\text{O}_5 & & = 58.69 \text{ ,,} \end{array}$$

$$\text{Total} \dots\dots\dots 99.43$$

$$\text{Sb}_2\text{O}_3, 288, \quad \text{Nb}_2\text{O}_5, 267.4, \quad \text{Ta}_2\text{O}_5, 444.$$

$$x = \text{Ta}_2\text{O}_5 \quad \text{and} \quad 58.69 - x = \text{Nb}_2\text{O}_5,$$

$$\text{Then} \quad x \frac{732}{444} + 58.69 - x \frac{555.4}{267.4} = 99.43.$$

$$\therefore x = 52.45 \text{ p. c. Ta}_2\text{O}_5, \text{ and } 58.69 - x = 6.24 \text{ p. c. Nb}_2\text{O}_5.$$

As the molecular weights of the two acids are so different, the results obtained are probably within the limits of variation of different specimens of the same mineral.

The other constituents were determined in the usual way. An analysis was also made by the same method of another specimen, the results being given in No. 2 below.

	No. 1.	No. 2.
Ta ₂ O ₅	51.13	51.95
Nb ₂ O ₅	7.56	4.49
Sb ₂ O ₃	40.23	38.04
Bi ₂ O ₃	0.82	0.79
NiO	0.08	trace
Fe ₂ O ₃	trace	0.39
CuO	—	0.30
Mn	trace	trace
SiO ₂	—	3.14
H ₂ O at a red heat	0.08	0.61
Total	99.90	99.71
Specific gravity.....	7.37	6.60

Sample No. 1 being much the more nearly pure, the ratio of the acids in it may be considered the more trustworthy.

Physical Properties.

Crystallisation, indistinct, probably orthorhombic. Hardness, 5—5.5. Specific gravity, 6.47—7.37. Lustre, adamantine. Colour, pale reddish-yellow to greenish-yellow and yellow. Streak, nearly white. Opaque to sub-translucent. Fracture, sub-conchoidal to granular.

The specimens received were all too water-worn to determine the crystalline form, but some minute cleavage faces noticed in the powdered mineral under the microscope showed that it was not oblique, and no part of the powdered mineral could be seen to give

an opaque field when rotated between crossed Nicol prisms, from which it may be inferred that the mineral is probably orthorhombic.

The specific gravity of the pure mineral is probably 7.37 or over. Hardly a trace of extraneous mineral could be seen when the powder of the specimen, of sp. gr. 7.37, was examined under the microscope, whilst in the specimen of sp. gr. 6.6 a small quantity of red, ochreous material could be distinguished.

Although the fresh fracture shows adamantine, or almost metallic lustre, the water-worn fragments have a resinous appearance.

I have not seen the reincandescence of a sodium tantalate bead on cooling after fusion, previously noticed. It is similar to that of sodium titanate, but less marked.

It is expected that a further supply of the mineral will be shortly obtained, among which it is hoped that definite crystals may be found, which will furnish material for further analyses.

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