

## X-RAY STUDY OF THE TANTALUM MINERAL SIMPSONITE

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## ABSTRACT

The hexagonal mineral simpsonite, an oxide of aluminum and tantalum or a tantalate from Tabba Tabba in Western Australia, was described and named by Bowley in 1939. Taylor furnished Laue and Bragg x-ray data at the same time. The mineral as originally identified was assigned a complex formula based upon the analysis of material consisting of a mixture of microlite and simpsonite.

Since the original description appeared, occurrences of an aluminum tantalate have been found in two localities in Brazil along the boundary between the states of Parafba and Rio Grande do Norte and in Southern Rhodesia at Bikita. X-ray-diffraction studies and optical examination have shown that the fundamental aluminum tantalate is the same in the materials from Western Australia, Brazil, and Southern Rhodesia. In addition Laue photographs have been obtained and correlated with Taylor's original x-ray data. Rotation photographs have been utilized in establishing the lattice constants.

In specimens from each of the localities mentioned microlite is associated with simpsonite, and in some instances the simpsonite is replaced. The Brazilian and African specimens are apparently more nearly pure simpsonite than the type material from Western Australia. In Brazil crystals of simpsonite almost an inch across have been found, but x-ray and optical examination indicate that the apparently single crystals consist of numerous small nearly parallel individuals. The formula of simpsonite is believed to be  $3Al_2O_3 \cdot 2Ta_2O_5$ .

Simpsonite, because of its high tantalum and correspondingly low columbium content, is a desirable economic tantalum mineral, but as yet production has been limited. It is hoped that further description of the species may lead to its recognition in other localities.

## INTRODUCTION

Several samples of a mineral which consists almost entirely of alumina and tantalum oxide were recently submitted to the mineralogical laboratory of Columbia University for study. The samples were reported to have been found in several places; specimens from each contain large amounts of tantalum with but small amounts of columbium. The mineral of principal interest is simpsonite which has been examined from several localities (Fig. 1), which include Tappa Tappa, Western Australia; Equador and Picuí, Brazil; and Bikita, Southern Rhodesia. In view of the importance of tantalum minerals to the war-time production of the metal the most complete study possible was made, in the time available, of the materials submitted. Since complete descriptions of the occurrences are to be made by those who have visited the localities this account is confined to the specimens.

The war has caused the development of many new and important uses for tantalum, and the mineral requirements of the industry now amount to several hundred tons each year. The combined efforts of the manufacturers, the War Production Board, the Metals Reserve Company, and the Foreign Economic Administration have resulted in the development of several hundred new deposits to meet the increased demand. Prior to the war the only minerals processed were tantalite and tapiolite, whereas now new techniques and new sources enable producers to process concentrates of microlite, simpsonite, and bismutotantalite.

Tantalum minerals have been found in many countries, and commercial production has been reported from Argentina, Australia, Belgian Congo, Brazil, Canada, India, Madagascar, Mozambique, Nigeria, Portugal, Southern Rhodesia, South Africa, Uganda, and the United States. Microlite has been found in considerable quantities at the Harding mine in New Mexico and the Brown Derby mine in Colorado. Tantalite has been mined in the Black Hills of South Dakota.

Tantalum metal is produced from concentrates by an extended chemical process combined with the methods of powder metallurgy. The high melting point of the metal ( $5150^\circ\text{F}$  or  $2850^\circ\text{C}$ ) and a great affinity for gases when heated obviate the use of simple methods of reduction or smelting. Chemical processes yield purified salts which are reduced to powdered metal. The powdered metal is pressed into bars and sintered at high temperatures in vacuum furnaces. The sintered bars are cold-rolled or cold drawn into sheets, rod, ribbon, and fine wire for fabrication.

A number of the uses of tantalum are not revealed for reasons of security. A few, however, which have been announced are not yet widely known. Due to the resistance of tantalum to the action of acids the metal finds one of its most extensive uses in piping, heating coils, condensers, heat exchangers, thermometer wells, and

hydrochloric-acid units. Here a resistance to corrosion in some instances comparable to glass combined with the strength, workability, and malleability characteristic of a metal prove most useful.

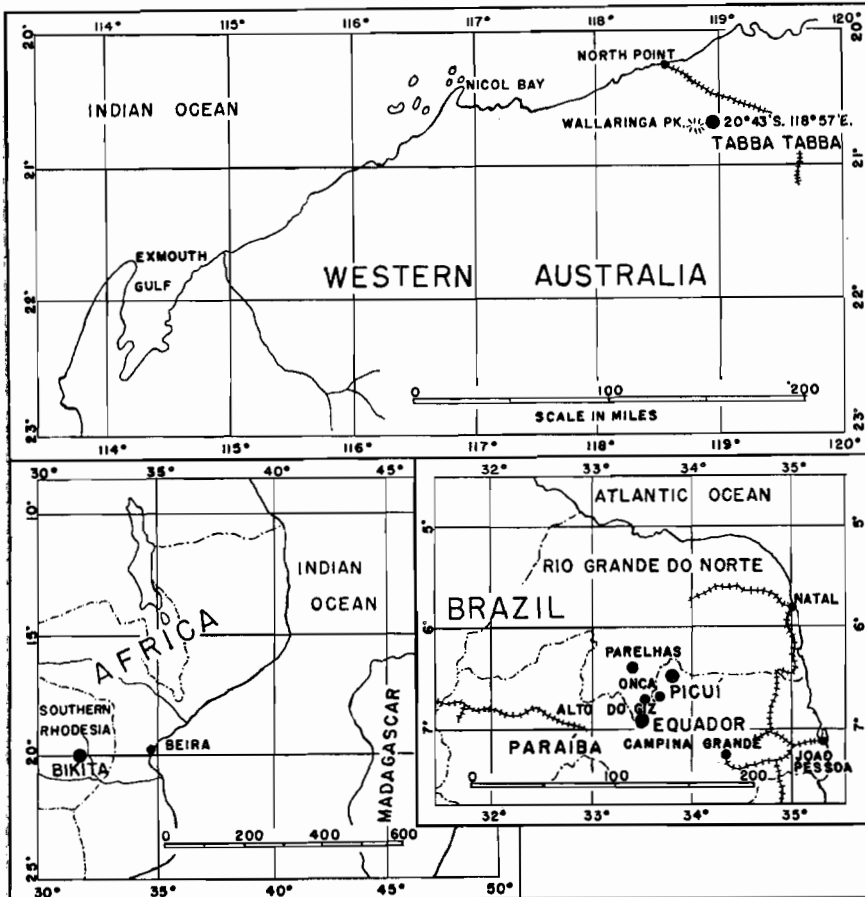


FIGURE 1.—Sketch map of simpsonite localities

Tantalum carbide is used as a component in cemented and cast tungsten carbides imparting a lubricating characteristic that tends to reduce the adhesion of tungsten carbide when used for cutting, drawing, or forming other materials. Tungsten carbide containing tantalum carbide in cutting tools, drawing and forming dies, and wear-resistant parts is finding a considerable application.

Anodic films of tantalum due to their noteworthy stability have been utilized in forms of current control for some time. These films have a passivity to corrosive action which makes them useful for certain types of rectification.

The inert character of the metal in contact with the acids of the body has led to its demand by the medical profession for use in wire, foil, plates, sutures, and screws to be applied in surgery. The absorption of gases at high temperatures combined

TABLE 1.—*Minerals known to contain tantalum*

Mineral	Maximum reported Ta <sub>2</sub> O <sub>5</sub> *	Composition and crystallization
Tantalum H.6-7; G.11.2	(98.5—Ta)	Metallic tantalum Isometric
Tantallic ocher	—	Ta <sub>2</sub> O <sub>5</sub> Possibly Orthorhombic and isometric
Tapiolite-Mossite H.6½-6; G.8±-6±	88.44	(Fe, Mn) <sub>2</sub> (Ta, Cb) <sub>4</sub> O <sub>2</sub> Tetragonal
Tantalite-Columbite† H.6½-6; G.7.95-5.20	83.57	(Fe, Mn)(Ta, Cb) <sub>2</sub> O <sub>6</sub> Isometric
Thoreaulite H.6; G.7.6-7.9	77.59	SnTa <sub>2</sub> O <sub>7</sub> Monoclinic
Microlite-Pyrochlore H.5½-5; G.6.4-4.2	77.00	(Na, Ca) <sub>2</sub> Ta <sub>2</sub> O <sub>6</sub> (O, OH, F)-Na, CaCb <sub>2</sub> O <sub>6</sub> F Isometric (Metamict)
Simpsonite H.6½; G.6.27	72.31	Al <sub>6</sub> Ta <sub>4</sub> O <sub>19</sub> or 3(Al <sub>2</sub> O <sub>3</sub> )-2(Ta <sub>2</sub> O <sub>5</sub> ) Hexagonal
Djalmaite** H.5½; G.5.75-5.88	72.27	(U, Ca, Pb, Bi, Fe)(Ta, Cb, Ti, Zr) <sub>3</sub> O <sub>9</sub> ·nH <sub>2</sub> O Isometric
Stibiotantalite-Stibiocolumbite H.5½; G.7.34-5.98	57.29	Sb(Ta, Cb)O <sub>4</sub> Orthorhombic
Formanite-Fergusonite H.6½-5½; G.5.8-5.6	55.51	(U, Zr, Th, Ca)(Ta, Cb, Ti)O <sub>4</sub> -(Y, Er, Ce, Fe) (Cb, Ta, Ti)O <sub>4</sub> Tetragonal (Metamict)
Euxenite-Polycrase H.5½-6½; G.5.00	47.31	(Y, Ca, Ce, U, Th)(Cb, Ta, Ti) <sub>2</sub> O <sub>6</sub> -(Y, Ca, Ce, U, Th)(Ti, Cb, Ta) <sub>2</sub> O <sub>6</sub> Orthorhombic (Metamict)
Loranskite H.5; G.4.6; 4.16	47.00	(Y, Ce, Ca, Zr?)(Ta, Zr)O <sub>4</sub> ?
Polymignyte H.6½; G.4.77-4.85	42.17	(Ca, Fe, Y, etc. Zr, Th)(Cb, Ti, Ta)O <sub>4</sub> Orthorhombic (Metamict)
Bismutotantalite H.5; G.8.26	41.15	Bi(Ta, Cb)O <sub>4</sub> Orthorhombic
Yttrotantalite H.5-5½; G.5.7	39.53	(Fe, Y, U, Ca etc.)(Cb, Ta, Zr, Sn)O <sub>4</sub> Orthorhombic (Metamict)
Rutile (strueverite) H.6-6½; G.5.6	35.96	TiO <sub>2</sub> + Ta, Cb, Fe <sup>2</sup> , Fe <sup>3</sup> Tetragonal

\* The Ta<sub>2</sub>O<sub>5</sub> content decreases, and the Cb<sub>2</sub>O<sub>5</sub> increases in minerals forming an isomorphous series.

† Sp.Gr.: Tantalite = 7.95; Columbite = 5.20; variation—0.02% Ta<sub>2</sub>O<sub>5</sub> = ±0.05 Gr.

\*\* Possibly the tantalum equivalent of betafite.

TABLE 1.—Continued

Mineral	Maximum reported Ta <sub>2</sub> O <sub>5</sub> *	Composition and crystallization
Betafite H.4-5½; G.3.7-5.0	28.50	(U, Ca)(Cb, Ta, Ti) <sub>3</sub> O <sub>7</sub> ·nH <sub>2</sub> O Isometric (Metamict)
Samarskite H.6-5; G.5.69±	27.03	(Y, Er, Ce, U, Ca, Fe, Pb, Th)(Cb, Ta, Ti, Sn) <sub>2</sub> O <sub>8</sub> Orthorhombic (Metamict)
Scheteligite H.5½; G.4.74	20.00	(Ca, Y, Sb, Mn) <sub>2</sub> (Ti, Ta, Cb) <sub>2</sub> (O, OH) <sub>2</sub> Orthorhombic (Metamict)
Ishikawaite H.5-6; G.6.2-6.4	15.00	(U, Fe, Y, etc.)-(Cb, Ta)O <sub>4</sub> Orthorhombic
Ampangabeite H.4; G.3.36-4.64	12.61	(Y, Er, U, Ca, Th) <sub>2</sub> (Cb, Ta, Fe, Ti) <sub>7</sub> O <sub>18</sub> ? Orthorhombic (Metamict)
Cassiterite (ainalite) H.6-7; G.7.0	8.78	(SnO <sub>2</sub> ) + Ta, Cb Tetragonal
Eschynite-Priorite H.6-5; G.5.19-4.95	6.97	(Ce, Ca, Fe, Th)(Ti, Cb, Ta) <sub>2</sub> O <sub>7</sub> -(Y, Er, Ca, Fe, Th)(Ti, Cb) <sub>2</sub> O <sub>6</sub> Orthorhombic (Metamict)
Laavenite H.6; G.3.51-3.55	5.20	R(Si, Zr)O <sub>2</sub> + Zr(SiO <sub>2</sub> ) + RTa <sub>2</sub> O <sub>6</sub> ; R = (Mn, Fe):Ca:Na = 7:6:9 Monoclinic
Perovskite (dysanalite) H.5½; G.4.01	5.08	Ca <sub>3</sub> (Ti, Cb, Ta) <sub>3</sub> O <sub>24</sub> Monoclinic ?
Melanocerite H.5-6; G.4.129	3.65	12(H <sub>2</sub> , Ca)SiO <sub>3</sub> ·3(Y, Ce)BO <sub>3</sub> ·2H <sub>2</sub> (Th, Ce)O <sub>2</sub> F <sub>2</sub> ·8(Ce, La, Di)OF Hexagonal
Caryocerite H.5-6; G.4.295	3.11	6(H <sub>2</sub> , Ca)SiO <sub>2</sub> ·2(Ce, Di, Y)BO <sub>3</sub> ·3H <sub>2</sub> (Ce, Th)O <sub>2</sub> F <sub>2</sub> ·2La, O, F Hexagonal
Tritomite H.5.5; G.4.15-4.25	3.09	2(H <sub>2</sub> , Na <sub>2</sub> , Ca)SiO <sub>3</sub> ·(Ce, La, Di, Y)BO <sub>3</sub> ·H <sub>2</sub> (Ce, Th, Zr)O <sub>2</sub> F <sub>2</sub> Hexagonal
Naegite H.7.5; G.4.09	—	SiO <sub>2</sub> ; ZrO <sub>2</sub> ; UO <sub>3</sub> ; ThO <sub>2</sub> ; (Nb, Ta) <sub>2</sub> O <sub>5</sub> ; Y <sub>2</sub> O <sub>3</sub> Tetragonal
Robellazite	—	Mixture—Cb, Ta, W, V, Al, Fe, Mn
Yttrocrasite H.5½-6; G. 4.80	—	(Y, Th, U, Ca) <sub>2</sub> (Ti, Fe, W) <sub>4</sub> O <sub>11</sub> ? Orthorhombic

TABLE 1.—Continued

Mineral	Maximum reported $Ta_2O_5$ *	Composition and crystallization
Eudialite (Eucolite) H.5-5½; G.3.0-3.1	—	I II      I      II $R_4R_3Zr(SiO_3)$ R = Na, K, H; R = Ca, Fe, Mn, Ce, Ta, Cb Hexagonal
Epistolite H.1-1½; G.2.885	—	Cb, (Ta?), Ti, Fe, etc., $SiO_2$ , $H_2O$ Monoclinic

## SYNONYMS OR VARIETIES

Tapiolite-Mossite	Formanite-Fergusonite
Adelpholite	Risörite
Hjelmite	Bragite
Ixiolite	Rutherfordite
Niobium tapiolite	Sipylite
Kimito-tantalite	Tyrite
Skogbölite	
	Euxenite-Polycrase
Tantalite-Columbite	Chlopinite
Baierine	Eschwegite
Calciotantalite	Lyndochite
Dianite	Tanteuxenite
Greenlandite	Tantalopolyrase
Hermannolite	
Manganocolumbite	Bismutotantalite
Manganotantalite	Ugandite
Mengite	
Niobite	Rutile
Toddite	Tantalum ilmenorutile
Torrelite	Tantalian rutile
Yttrio-columbo-tantalite	
	Betafite
Microlite-Pyrochlore	Samiesite
Chalcolamprite	
Columbomicrolite	Samarskite
Ellsworthite	Annerödite
Endeiolite	Calciosamarskite
Fluochlore	Hydrosamarskite
Hatchettolite	Nohlite
Haddamite	Nuolaite
Hydrochlore	Plumboniobite
Koppite	Rogersite
Marignacite	
Metasimpsonite	Vietinghofite
Niobpyrochlore	
Neotantalite	Eschynite-Priorite
Pyrrhite	Blomstrandine
Stibiomicrolite	Blomstrandite
Tantalohatchettolite	

TABLE 1.—*Concluded*

Perovskite	Caryocerite
Loparite	Steenstrupine
Metaloparite	

with other properties has made tantalum useful for plates and grids in electronic power tubes. The use of tantalum oxide in glass has led to the manufacture of lenses with unusual properties.

A list of minerals known to contain tantalum is included in Table 1. The minerals are arranged in order of decreasing tantalum content as shown in standard references, and the maximum tantalum oxide content reported is indicated. Many varietal names and synonyms are also listed. The table further shows that simpsonite with the formula  $3\text{Al}_2\text{O}_3 \cdot 2\text{Ta}_2\text{O}_5$  and hexagonal crystallization seems to stand by itself among the minerals containing tantalum. Consultation of standard references in each case shows that the columbium content of many species varies widely and in some exceeds the content of tantalum.

#### ACKNOWLEDGMENTS

The study has been materially aided through the co-operation of the Fansteel Metallurgical Corporation. We are indebted to Dr. W. M. Shafer, Spectroscopist, for the data shown in Figure 1 of Plate 4. Mr. R. W. Hoffman, Chief Chemist of the Tantalum Defense Corporation, has furnished chemical analyses of simpsonite and microlite, essential to the interpretation of the x-ray data. Mr. A. L. Wiegand has been most helpful in supplying specimens from different localities and in furnishing information concerning the utilization of tantalum. This assistance is gratefully acknowledged.

The United States National Museum has kindly made available for purposes of comparison and study two crystals of simpsonite from Western Australia representing material originally sent to the Museum by the late Dr. E. S. Simpson, after whom the mineral was named. The kindness of Mr. E. P. Henderson, Curator of Mineralogy, in supplying this material is greatly appreciated.

Dr. Caio Pandiá Guimarães, Belo Horizonte, Brazil, has furnished samples and also an advance copy of a manuscript including analyses of Brazilian crystals. This co-operation has made possible correlation with descriptions published by Doctor Guimarães (1943; 1944) and confirmation of both physical and chemical data.

The American Museum of Natural History through the kindness of Dr. Frederick Pough has supplied material from a crystal of Australian simpsonite and crystals of simpsonite from Brazil. This co-operation has furnished further confirmation of materials from other sources. The study has also been considerably assisted by conferences with Doctor Pough concerning his study of the Brazilian tantalum occurrences which is being published as a companion paper.

#### SIMPSONITE FROM TABBA TABBA, WESTERN AUSTRALIA

Simpsonite was first described from Tabba Tabba, Western Australia, by Bowley in 1939. X-ray studies were published at the same time by Taylor (1939). The

mineral is found in grayish-white crystals of partly broken hexagonal outline and tabular habit. These measure half to three quarters of an inch across and a quarter of an inch more or less in height. The crystals show extensive alteration to microlite, and fragments of pure simpsonite of sufficient size to yield a suitable amount of

TABLE 2.—*Analysis A—Bowley (1939)*

Tabba Tabba, Western Australia—D. G. Murray	Recomputa- tion	Muscovite	Simpsonite	Microlite	Remainder, quartz Fe-min- eral, cassiterite Misc.
Al <sub>2</sub> O <sub>3</sub> .....	16.75	16.662	0.778	15.884	—
Ta <sub>2</sub> O <sub>5</sub> .....	72.31	71.929	—	45.871	26.058
Cb <sub>2</sub> O <sub>6</sub> .....	.33	0.328	—	—	0.328
CaO.....	3.40	3.382	—	—	3.382
Na <sub>2</sub> O.....	1.16	1.154	—	—	1.154
F.....	.21*	0.209	—	—	0.209
H <sub>2</sub> O <sup>+</sup> .....	1.35	1.342	0.091	—	1.251
H <sub>2</sub> O <sup>-</sup> .....	.20	0.199	—	—	—
K <sub>2</sub> O.....	.24	0.239	0.239	—	—
SiO <sub>2</sub> .....	1.78	1.771	0.914	—	—
FeO.....	.16	0.159	—	—	—
Fe <sub>2</sub> O <sub>3</sub> .....	.14	0.139	—	—	—
MnO.....	.08	0.080	—	—	—
PbO.....	.42	0.418	—	—	—
SnO.....	2.00	1.989	—	—	—
	100.53	100.000	2.022	61.775	32.382
O = F.....	.09				
	100.44				3.841

Muscovite = K<sub>2</sub>O = 11.8; Al<sub>2</sub>O<sub>3</sub> = 38.5; SiO<sub>2</sub> = 45.2; H<sub>2</sub>O = 4.5; = (H, K) AlSiO<sub>4</sub>; = 100.  
 Simpsonite = Al<sub>2</sub>O<sub>3</sub> = 25.721; Ta<sub>2</sub>O<sub>5</sub> = 74.279; = Al<sub>6</sub>Ta<sub>4</sub>O<sub>19</sub>; = 100.  
 Microlite = Ta<sub>2</sub>O<sub>5</sub> = 80.471; Cb<sub>2</sub>O<sub>6</sub> = 1.013; CaO = 10.444; Na<sub>2</sub>O = 3.564; F = 0.645; H<sub>2</sub>O = 3.863; = 100.

\* Estimated to be low.

material for a chemical analysis are unobtainable. Both microscopic study and x-ray examination establish the presence of the two constituents. The chemical analyses as published evidently applied to mixtures. The recast of Bowley's analyses (Tables 2, 3) indicates 32.38 per cent microlite in sample A and 27.13 per cent in sample B.

In thin section the microlite appears as a fibrous aggregate which surrounds and cuts across granular remnants of simpsonite. Basal sections of simpsonite yield uniaxial interference figures, and also hexagonal patterns when exposed in a Laue camera equipped with a fine pin hole (Pl. 1, fig. 2). Laue photographs of material from this locality often exhibit double spots showing that apparent single crystals give exposures indicating more than one individual. The Laue photograph of a basal plate published by Taylor (Pl. 1, fig. 1) showed this characteristic, and it has been possible in this investigation to secure only occasional photographs of single units, most of the patterns being somewhat duplicated.





FIGURE 1. PRINT OF TAYLOR'S  
SIMPSONITE PHOTOGRAPHED NORMAL  
TO (0001)

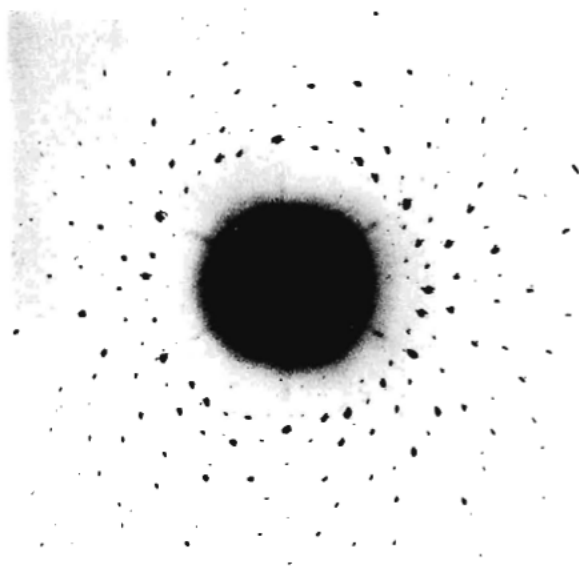


FIGURE 2. SIMPSONITE FROM WESTERN AUSTRALIA PHOTOGRAPHED  
NORMAL TO (0001)

LAUE PHOTOGRAPHS OF SIMPSONITE FROM WESTERN  
AUSTRALIA

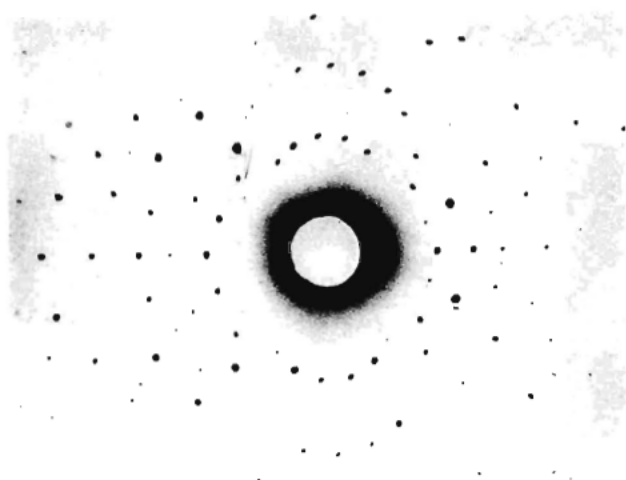


FIGURE 1. PHOTOGRAPH NORMAL TO MAJOR PRISM AND SHOWING HORIZONTAL PLANE OF SYMMETRY

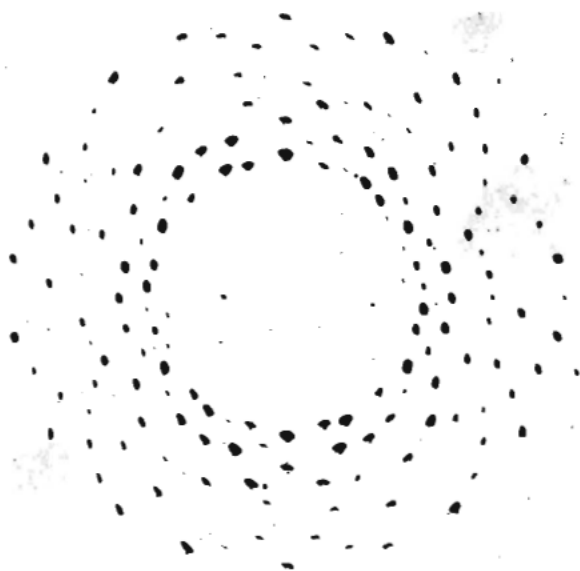


FIGURE 2. SIMPSONITE FROM BRAZIL PHOTOGRAPHED NORMAL TO (0001)

Two crystals of the Australian material were supplied by the U. S. National Museum, and a portion of an altered crystal has been available from the American Museum of Natural History. A thin section prepared from one of the National Museum crystals for optical study later served for Laue photographs, while the

TABLE 3.—*Analysis B—Bowley (1939)*

Tabba Tabba, Western Australia—J. N. A. Grace	Recomputation	Muscovite	Simpsonite	Microlite	Remainder, quartz Fe-mineral, cassiterite misc.
Al <sub>2</sub> O <sub>3</sub> . . . . .	18.64	18.452	1.356	17.096	—
Ta <sub>2</sub> O <sub>5</sub> . . . . .	71.48	70.758	—	49.371	21.387
Cb <sub>2</sub> O <sub>5</sub> . . . . .	.32	0.317	—	—	0.317
CaO . . . . .	3.19	3.158	—	—	3.158
Na <sub>2</sub> O . . . . .	.68	0.673	—	—	0.673
F . . . . .	.38	0.376	—	—	0.376
H <sub>2</sub> O <sup>+</sup> . . . . .	1.39	1.376	0.158	—	1.218
H <sub>2</sub> O <sup>-</sup> . . . . .	.03	0.030	—	—	0.030
K <sub>2</sub> O . . . . .	.42	0.416	0.416	—	—
SiO <sub>2</sub> . . . . .	2.34	2.316	1.592	—	0.724
FeO . . . . .	.44	0.436	—	—	0.436
Fe <sub>2</sub> O <sub>3</sub> . . . . .	.48	0.475	—	—	0.475
MnO . . . . .	.04	0.040	—	—	0.040
PbO . . . . .	nil	—	—	—	—
SnO <sub>2</sub> . . . . .	1.19	1.178	—	—	1.178
	101.02	100.00	3.522	66.467	27.129
O = F . . . . .	.16				
	100.86				

Muscovite = (See analysis by D. G. Murray).

Simpsonite = (See analysis by D. G. Murray).

Microlite = Ta<sub>2</sub>O<sub>5</sub> = 78.834; Cb<sub>2</sub>O<sub>5</sub> = 1.168; CaO = 11.641; Na<sub>2</sub>O = 2.481; F = 1.386; H<sub>2</sub>O = 4.490; = 100.00.

fragments were selected for Debye powder photographs. Laue photographs of the crystal were compared with a photographic reproduction of one of the Laue patterns pictured by Taylor in describing the Tabba Tabba crystals (Pl. 1, fig. 1). After elimination of duplicated spots due to reflection from corresponding but not quite parallel planes on an adjacent crystal the two patterns are believed to agree as shown in the stereographic projections (Figs. 3, 4).

X-ray powder photographs were made of small chips broken from the two crystals. Debye patterns of two types were obtained. The measurements of one yield an isometric pattern with a lattice constant which agrees with microlite (Pl. 3, fig. 2). The other yields a mixed pattern of simpsonite and microlite (Pl. 3, fig. 1). The microlite pattern is derived from the fibrous and isotropic material, while the mixed pattern results from a combination of granular and fibrous material.

Optically the Australian simpsonite is uniaxial negative with indices of refraction approximating 2.06, the value reported by Bowley. The positive sign reported by

Bowley is not confirmed. Traces of muscovite, quartz, and limonite may be observed in the crystals.

The chemical analyses of the Australian crystals (Tables 2, 3) have been recast in terms of muscovite, simpsonite, and microlite together with an allowance for small

TABLE 4.—*Comparison of chemical analyses and spectrographic data*

	Chemical analyses by R. W. Hoffman, Chief Chemist, Tantalum Defense Corporation			Spectrographic data by Dr. W. M. Shafer, Spectroscopist, Fansteel Metallurgical Corporation			
	Simpsonite —Equador, Brazil	Simpsonite —Bikita, S. Rhodesia	Microlite— Picuf, Brazil		Simpsonite— Equador, Brazil	Simpsonite— Bikita, S. Rhodesia	Microlite—Picuf Brazil
Ta <sub>2</sub> O <sub>6</sub>	71.54	67.92	74.37	Ta	Strong	Strong	Strong
Cb <sub>2</sub> O <sub>5</sub>	1.82	1.61	5.45	Cb	Medium	Weak	Medium
Al <sub>2</sub> O <sub>3</sub>	25.20	25.06	0.83	Al	Strong	Strong	Weak—Medium
FeO	0.16	3.65	1.24	Fe	Weak	Medium	Weak—Medium
CaO	0.12	0.12	15.48	Ca	Weak	Weak	Strong
MgO	—	0.01	—	Mg	—	Weak	Weak
MnO <sub>2</sub>	—	0.44	—	Mn	Weak	Medium	Weak
SnO <sub>2</sub>	—	0.47	—	Sn	Absent	Medium	Weak
TiO <sub>2</sub>	0.02	0.05	0.83	Ti	Weak	Very weak	Weak—Medium
SiO <sub>2</sub>	1.00	(0.50)	0.75	Si	(1.00)	(±0.50)	(0.75)
F	—	—	1.29	—	—	—	—
	99.86	99.83	100.24	Na	—	—	Weak
				K	—	—	Absent
				W	Absent	Absent	Absent
				Mo	Absent	Weak	Absent

amounts of quartz, limonite, and cassiterite. The formula for simpsonite used in recasting the analyses is based upon analyses of the more nearly pure Brazilian material of Tables 4 and 5.

In the recast of the Tabba Tabba analyses it seems reasonable to make the following assumptions:

- (1) The K<sub>2</sub>O of the original analysis may be attributed to muscovite.
- (2) After satisfying the formula for muscovite the balance of the Al<sub>2</sub>O<sub>3</sub> may be assigned to simpsonite.
- (3) The formula for simpsonite based on the Brazilian analysis of Al<sub>6</sub>Ta<sub>4</sub>O<sub>19</sub> seems most likely. (Cb<sub>2</sub>O<sub>5</sub> is omitted since the amount is small.)
- (4) The Ta<sub>2</sub>O<sub>6</sub> remaining after the computation for simpsonite may be assigned to microlite along with Cb<sub>2</sub>O<sub>5</sub>, CaO, F, Na<sub>2</sub>O, and H<sub>2</sub>O.
- (5) The balance of the analysis may be attributed to quartz, limonite, an unidentified lead mineral, and possibly cassiterite. Since the remnant minerals are present in small amount no attempt at separation is made.

Bowley recognized the association of microlite and simpsonite, but lacking pure material in sufficient quantity he had no way to ascertain that the simpsonite itself was essentially a simple aluminum tantalate. It seems clear, however, that his description was the first account of a valid species.

The recast of the analyses is simplified if the formula for simpsonite,  $2\text{H}_2\text{O} \cdot \text{CaO} \cdot 5\text{Al}_2\text{O}_3 \cdot 4\text{Ta}_2\text{O}_5$ , as given by Bowley is replaced by  $3\text{Al}_2\text{O}_3 \cdot 2\text{Ta}_2\text{O}_5$ .

TABLE 5.—*Formula of simpsonite based on the Brazilian analysis*

Chemical analysis	Recast	Proportions
Ta <sub>2</sub> O <sub>5</sub> ..... 71.54	72.58 ÷ 441.76	} .1713 or 1.000 or 2
Cb <sub>2</sub> O <sub>5</sub> ..... 1.82	1.85 ÷ 265.82	
Al <sub>2</sub> O <sub>3</sub> ..... 25.20	25.57 ÷ 101.94	.2508 or 1.464 or 3
98.56		

$$3\text{Al}_2\text{O}_3:2\text{Ta}_2\text{O}_5 = .2508:.1713 = 1.464:1$$

Although the two analyses differ considerably in the relative proportions of the oxides reported, the compositions of the microlite portions computed for each analysis agree quite closely. The two are given for convenient comparison:

Analyst	Ta <sub>2</sub> O <sub>5</sub>	Chemical composition of microlite Tabba Tabba, Western Australia						Total
		Cb <sub>2</sub> O <sub>5</sub>	CaO	NaO	F	H <sub>2</sub> O		
D. G. Murray....	80.471	1.013	10.444	3.564	0.645	3.863	100	
J. N. Grace.....	78.834	1.168	11.641	2.481	1.386	4.490	100	

SIMPSONITE FROM BRAZIL

In Brazil simpsonite occurs in pegmatites at Alto do Giz near Equador, Rio Grande do Norte, and at Onça near Picuí, Paraíba. The localities lie along the boundary between the two states (Fig. 1). In each locality the mineral is found in yellowish-brown crystals. Microlite occurs at Onça as a white powder, in fine fibers, or in translucent brownish grains. The terrain at Alto do Giz and the materials mined are discussed by Pough (1945).

Some simpsonite crystals are unaltered with brightly reflecting crystal faces. One crystal from Equador supplied by Mr. Wiegand is illustrated in Figure 2. The forms present consist of the hexagonal prism *m* {101̄1}; the hexagonal prism *a* {112̄0}; the pinacoid {0001}; and *x* {1011}. The orientation and indices are based upon both geometrical and x-ray data. Angular measurements in the zone encircling the *c*-axis confirm the hexagonal symmetry within the limits of measurement.<sup>1</sup> The pinacoidal face *c*(0001) lies at 90° to the prismatic faces. Measurements in the zone *c x m* with the reflection goniometer are recorded as follows:

$$c \wedge x(0001) \wedge (10\bar{1}1) = 35^\circ 19'$$

$$x \wedge m(10\bar{1}1) \wedge (10\bar{1}0) = 54^\circ 44'$$

On the basis of crystal measurement the ratio *c*:*a* = .6135:1, while from x-ray data the ratio *c*<sub>0</sub>:*a*<sub>0</sub> = .61195:1. The angle *c* ∧ *x* = 35° 9' reported by Pough yields the ratio .6098:1. On the basis of x-ray measurement the computed angle *c* ∧ *x* would be 35° 14' 45".

<sup>1</sup> In a companion paper Pough reports that simpsonite is hexagonal dipyramidal on the basis of crystal measurement.

Rotation x-ray photographs have been taken with rotation about  $c$ ,  $a$ , and an axis in a plane parallel to (0001) and at  $30^\circ$  to  $a$ . The identity periods along these three axes have been determined; the periods along the  $a$  and  $c$  axes are the lattice constants.

Laue photographs of basal plates of the Brazilian crystals agree with a Laue photograph of similar orientation and film distance for the Australian simpsonite repro-

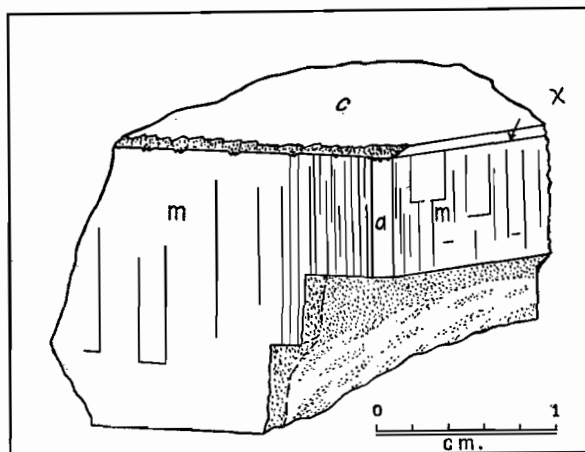


FIGURE 2.—*Simpsonite crystal from Equador, Brazil*

duced by Taylor. For comparison a photographic negative of the hexagonal Laue pattern shown by Taylor was superimposed on Laue photographs of the Brazilian crystals. Although the Brazilian simpsonite appears to form large crystals the Laue photographs demonstrate that the large crystals consist of many small units of not quite parallel orientation.

The simpsonite from Brazil agrees optically with the mineral from Australia. The indices of refraction of simpsonite from the Equador locality were determined approximately with piperine-iodide and sulphur-selenium melts. The values corresponded roughly with the figure given by Bowley of  $2.06 \pm$ . Determinations were made later with a small polished prism of partly transparent simpsonite from Equador furnished by Doctor Pough. The prism angle was  $24^\circ 15'$ , and measurements of the angle of minimum deviation for Na-light (5889–5895 Å) gave  $n_o = 2.0402$  and  $n_e = 1.9944$ . Basal plates yield uniaxial interference figures with a negative sign corresponding to determinations on the Australian samples. The index of refraction of granular microlite from Picuí is  $n = 2.08 \pm$  by melts.

Chemical analyses of simpsonite from Equador and microlite from Picuí are shown in Table 4. The chemical analysis of the Equador sample agrees with chemical data (Guimarães, 1944) in that the material is shown to consist essentially of aluminum and tantalum oxides. The formula for the Brazilian simpsonite as computed in Table 5 is probably  $3Al_2O_3 \cdot 2Ta_2O_5$  or  $Al_6Ta_4O_{19}$ . If the small amount of columbium present is considered the formula would become  $Al_6(Ta, Cb)_4O_{19}$ . Since most tantalum minerals contain at least small amounts of columbium the latter is probably more representative.

A copy of the manuscript published by Guimarães in which the name calogerasite was applied to the crystals from Equador was submitted to the senior author for review in the fall of 1944. At the same time Doctor Guimarães kindly forwarded samples of the material designated as calogerasite for study. Due to difficulties in communication which prevented a copy of the original description of simpsonite from being available in Brazil, Guimarães was forced to rely upon the abstract of the article by Bowley (1939) in the *Mineralogical Magazine*.

Due to the presence of calcium in the formula of simpsonite as originally proposed and the absence of the element in the material found in Brazil Guimarães concluded that the two were chemically different. The Brazilian material was also observed to be optically negative, while simpsonite had been reported to be positive. Since no aluminum tantalate corresponding to the Brazilian material appeared to have been previously described the name calogerasite, in honor of the Brazilian geologist João Pandiá Calogeras, was assigned to the material from the Equador locality.

Unfortunately nature furnished material of doubtful purity in Australia where the simpsonite was first found, while the material found later in Brazil was essentially pure. Nevertheless x-ray study, a recast of the original analyses of the Tappa Tappa material, and a redetermination of the optic sign show that the Brazilian mineral and the Australian simpsonite are the same.

The crystals available for measurement exhibited insufficient faces to determine the symmetry completely. On the basis of Pough's work simpsonite is doubly terminated with the plane of symmetry normal to the *c*-axis. This agrees with the conclusion of Taylor on the basis of x-ray data.

More complete single-crystal Laue photographs exhibit intensity variations which conform to a vertical axis of threefold symmetry rather than an axis of sixfold symmetry. This has been confirmed by repeated exposures with different crystals.

#### MICROLITE FROM BRAZIL

The chemical analysis of microlite from Picuí, Brazil, is given in Tables 4 and 6. Aside from the absence of alkalis the analysis of the microlite corresponds reasonably well with analyses of high-tantalum microlite. Although alkalis were not determined the spectrographic examination (Table 4) indicates the presence of a small amount of sodium. Potassium on the other hand was not shown to be present.

Since most microlite analyses record a small amount of  $\text{Na}_2\text{O}$  and little if any  $\text{K}_2\text{O}$  it seems likely that the microlite from Picuí may contain some  $\text{Na}_2\text{O}$ . The composition of the microlite at the Picuí locality on the basis of the analysis is computed in Table 6. Attempts were made to secure x-ray-diffraction patterns of microlite for comparison. Well-crystallized material from Amelia Court House, Virginia, in the Columbia University collection photographed with iron radiation resulted in diffused bands without lines. Ordinary microlite is supposedly metamict, but upon heating lines which either do not appear or are weak are brought out strongly. Microlite from Chesterfield, Massachusetts, yields a satisfactory pattern without heating which matches microlite from Brazil and Australia (Pl. 3, fig. 2).

The microlite found in association with simpsonite as at Tappa Tappa, in

Rio Grande do Norte, and Paraíba, yields x-ray diffraction patterns without heating. The finely crystalline aggregate in which the mineral occurs lends itself easily to x-ray powder diffraction study. While x-ray diffraction patterns of the microlite from all these localities have not been measured close comparison with a

TABLE 6.—*Chemical analysis of microlite*

Microlite—Picuf, Brazil—R. W. Hoffman	Recomputation	Simpsonite	Microlite	Remainder, quartz ilmenite, etc.
Ta <sub>2</sub> O <sub>5</sub> ..... 74.37	74.192	2.310	71.882	—
Cb <sub>2</sub> O <sub>6</sub> ..... 5.45	5.437	—	5.437	—
Al <sub>2</sub> O <sub>3</sub> ..... 0.83	0.828	0.828	—	—
FeO..... 1.24	1.237	—	—	1.237
CaO..... 15.48	15.443	—	15.443	—
TiO <sub>2</sub> ..... 0.83	0.828	—	—	0.828
SiO <sub>2</sub> ..... 0.75	0.748	—	—	0.748
F..... 1.29	1.287	—	1.287	—
<b>Total..... 100.24</b>	<b>100.000</b>	<b>3.138</b>	<b>94.049</b>	<b>2.813</b>

Simpsonite = Al<sub>2</sub>O<sub>3</sub> = 25.721; Ta<sub>2</sub>O<sub>5</sub> = 74.279; = Al<sub>6</sub>Ta<sub>4</sub>O<sub>18</sub>; = 100.

Microlite = Ta<sub>2</sub>O<sub>5</sub> = 76.430; Cb<sub>2</sub>O<sub>6</sub> = 5.781; CaO = 16.420; F = 1.368; = 100.

measured film of the Picuf sample which was analyzed indicates that measurements would correspond within the limits of error.

The lattice constant of the Picuf microlite on the basis of the symmetrical focusing pattern is  $10.39792 \pm .00004$ . The measurements of the x-ray powder photographs are recorded in Table 10. For comparison the lattice constants of microlite from a number of localities as previously determined by various investigators are given as follows:

Pyrochlore, Gaertner (1930).....	10.331–10.348 Å
Artificial pyrochlore, Gaertner (1930).....	10.376 ± .018 Å
“Koppite”, Brandenberger (1931).....	10.37 ± .01
“Pyrrhite”, Machatschki (1932).....	10.37
Microlite, Reuning (1933).....	10.398 ± .01
Microlite, Palache and Gonyer (1940).....	10.39 ± .01

Thin sections of the microlite from Picuf show a granular mass, dark between crossed nicols, with an occasional included grain of simpsonite. The latter is birefringent with a value estimated to be about 0.04. A few scattered grains of a metallic mineral, possibly ilmenite, may be observed. Guimarães (1944) has carefully determined the birefringence of the Brazilian simpsonite by cutting oriented sections of both quartz and simpsonite of the same thickness and utilizing the Berek compensator. Values determined in this way range from .041 to .044.

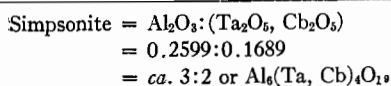
#### X-RAY POWDER PHOTOGRAPHS

Several forms of x-ray powder photographs were employed. A general survey of the various types of material available was first made utilizing Debye cameras



TABLE 7.—*Chemical formula of simpsonite on the basis of Bikita analysis*

Chemical analysis—R. W. Hoffman		Recomputation	Simpsonite ratios
Ta <sub>2</sub> O <sub>5</sub> .....	67.92	68.036	71.804 ÷ 441.76 = .16254
Cb <sub>2</sub> O <sub>5</sub> .....	1.61	1.613	1.703 ÷ 265.82 = .00640
Al <sub>2</sub> O <sub>3</sub> .....	25.06	25.103	26.493 ÷ 101.94 = .25989
FeO.....	3.65	3.656	
CaO.....	0.12	0.120	
MgO.....	0.01	0.010	
MnO <sub>2</sub> .....	0.44	0.441	
SnO <sub>2</sub> .....	0.47	0.471	
TiO <sub>2</sub> .....	0.05	0.050	
SiO <sub>2</sub> .....	(0.50)	0.501	
Total.....	99.83	100.00	100.000

TABLE 8.—*Summary of x-ray powder patterns*

Locality	Source	Description	X-ray pattern
Tabba Tabba, Western Australia	Amer. Museum Natural History	Altered gray crystal	microlite
Tabba Tabba, Western Australia	U. S. National Museum	Altered gray crystal	simpsonite and microlite
Equador, Brazil	Am. Museum Natural History	Translucent brown crystal	simpsonite
Equador, Brazil	Dr. Guimarães	Translucent brown crystal	simpsonite
Picuí, Brazil	Fansteel	Translucent brown crystal	simpsonite
Picuí, Brazil	Fansteel	White alteration product	microlite
Picuí, Brazil	Fansteel	Yellow alteration product	microlite
Picuí, Brazil	Fansteel	Granular and fibrous alteration product	microlite
Equador, Brazil	Fansteel	Translucent brown crystals	simpsonite
Equador, Brazil	Fansteel	White alteration product	microlite
Bikita, Southern Rhodesia	Fansteel	Brown crystal	simpsonite
Amelia Court House, Virginia	Columbia Univ. collection	Isotropic metamict crystal	no pattern
Commercial microlite	Fansteel	Opaque metamict crystals	weak microlite
Chesterfield, Mass.	Columbia Univ. collection	Minute rounded crystals	microlite

and iron radiation. The identifications indicated in this way are outlined in Table 8. A significant feature of the table is the correlation of simpsonite from Tabba Tabba, Picuí, Equador, and Bikita. The frequent association of microlite in several of the

TABLE 9.—X-ray powder data for *simpsonite*  
 Alto do Giz, Equador, Brazil  
 (Radius = 57.015 mm.; Iron radiation;  $\alpha_1$  lines;  $\theta$  = radians)

hkl	I/I <sub>1</sub> *	arc mm.*	$\theta^\circ$	$\sin^2\theta^\circ$	$\sin^2\theta^\dagger$	d in Au.†
10 $\bar{1}$ 0	.9	35.2	.15460	.0237	.022867	6.2736
0001	1.8	49.8	.21874	.0470	.045798	4.4518
10 $\bar{1}$ 1	4.5	61.0	.26793	.0700	.068636	3.6491
1120						
20 $\bar{2}$ 0	1.8	70.3	.30879	.0923	.091469	3.1787
11 $\bar{2}$ 1	8.6	79.2	.34787	.1162	.114404	2.8338
20 $\bar{2}$ 1	2.3	87.2	.38301	.1396	.137272	2.5850
21 $\bar{3}$ 1	1.8	94.6	.41551	.1629	.160071	2.3932
0002	1.4	101.6	.44626	.1862	.183190	2.2383
10 $\bar{1}$ 2						
21 $\bar{3}$ 1	6.8	108.0	.47437	.2086	.205912	2.1149
30 $\bar{3}$ 0						
11 $\bar{2}$ 2	} 2.3	120.6	.52971	.2553	.251700	1.9119
30 $\bar{3}$ 1						
20 $\bar{2}$ 2	} 1.8	126.5	.55563	.2782	.274533	1.8314
2240						
31 $\bar{4}$ 0	3.6	132.3	.58110	.3013	.297275	1.7598
22 $\bar{4}$ 1	—	—	—	—	.320210	—
21 $\bar{3}$ 2	} 10.0	143.6	.63073	.3477	.343169	1.6381
31 $\bar{4}$ 1						
40 $\bar{4}$ 0	.9	149.0	.65445	.3705	.365876	1.5870
30 $\bar{3}$ 2	.9	154.3	.67773	.3931	.388995	1.5407
0003	} 2.3	159.8	.70189	.4168	.411929	1.4962
40 $\bar{4}$ 1						
10 $\bar{1}$ 3	} 1.8	164.9	.72429	.4390	.434762	1.4579
32 $\bar{5}$ 0						
22 $\bar{4}$ 2	—	—	—	—	.457597	—
11 $\bar{2}$ 3						
31 $\bar{3}$ 2	} 9.6	175.4	.77041	.4850	.480435	1.3871
32 $\bar{5}$ 1						
41 $\bar{5}$ 0	} 2.7	180.6	.79325	.5078	.503647	1.3556
20 $\bar{2}$ 3						
41 $\bar{5}$ 1	4.5	185.8	.81609	.5306	.526015	1.3261
40 $\bar{4}$ 2	1.8	191.0	.83893	.5534	.549066	1.2986
21 $\bar{3}$ 3	} 5.5	196.4	.86265	.5769	.571956	1.2718
50 $\bar{5}$ 0						
50 $\bar{3}$ 3	} 5.0	207.8	.91272	.6259	.617638	1.2210
32 $\bar{5}$ 2						
50 $\bar{5}$ 1	} 2.7	212.2	.93205	.6445	.640284	1.2033
33 $\bar{6}$ 0						
42 $\bar{4}$ 0	} 4.5	217.9	.95708	.6683	.663311	1.1817
41 $\bar{5}$ 2						
33 $\bar{6}$ 1	} 2.7	223.2	.98036	.6906	.686336	1.1629
22 $\bar{4}$ 3						
42 $\bar{6}$ 1	} 3.7	229.2	1.00672	.7141	.709169	1.1431
31 $\bar{4}$ 3						
51 $\bar{6}$ 0						

\* Based directly on uncorrected Debye measurements.

† Calculated from the precision lattice constants.



FIGURE 1. SPECTROGRAMS OF SIMPSONITE AND MICROLITE  
 Iron lines are shown opposite each spectrogram for comparison. Photographs by Dr. W. M. Shafer.



FIGURE 2. SIMPSONITE ROTATED ABOUT C-AXIS  
 Spots are due to copper radiation filtered through nickel foil.

SPECTROGRAMS AND A ROTATION PHOTOGRAPH

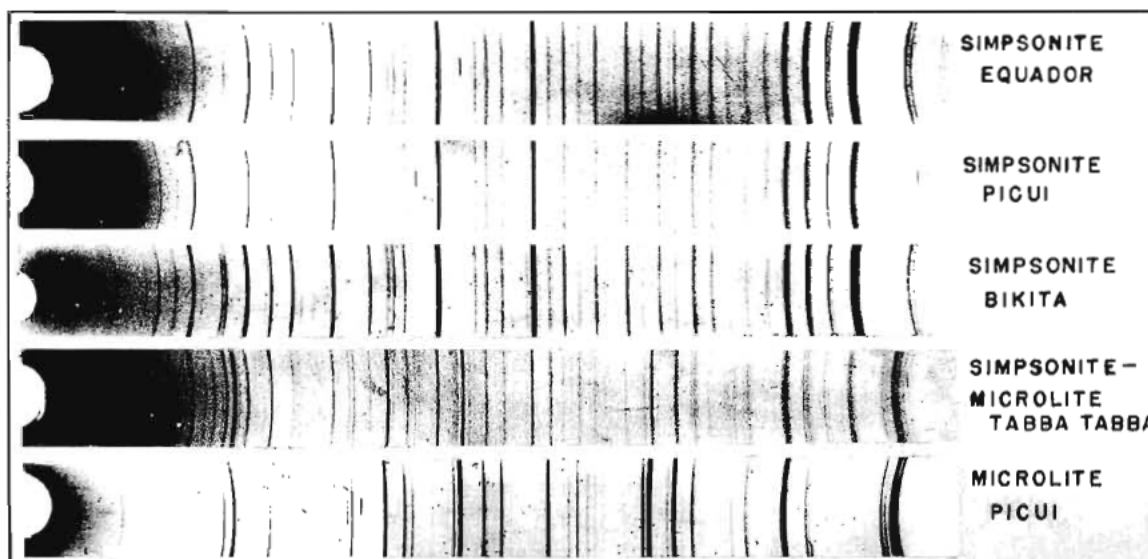


FIGURE 1. SIMPSONITE  
Microlite and a mixed pattern of the two minerals are included.

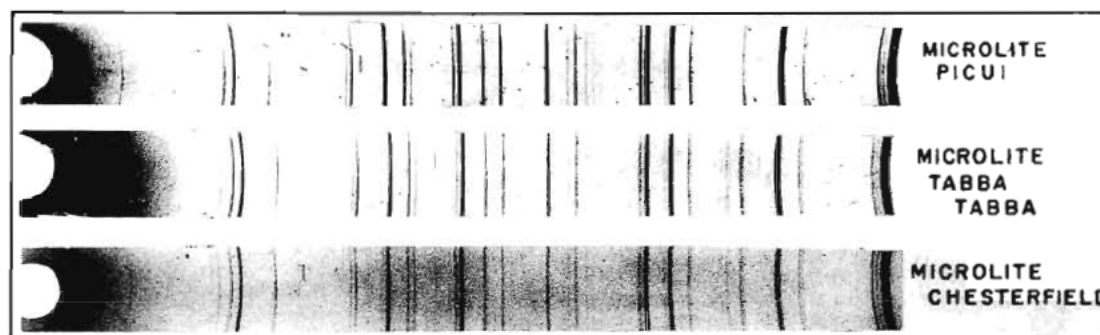


FIGURE 2. MICROLITE  
Debye patterns from Picui, Tabba Tabba, and Chesterfield, Mass. The three are essentially the same.

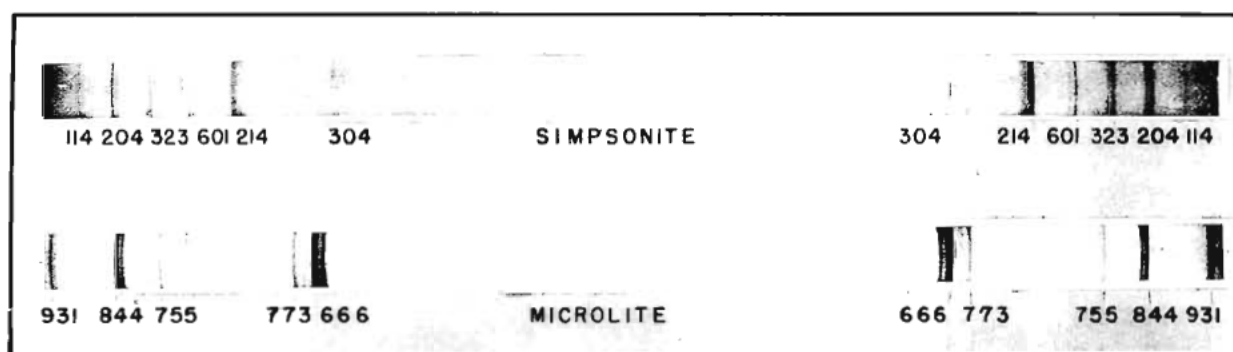


FIGURE 3. SYMMETRICAL FOCUSING CAMERA PHOTOGRAPHS  
Lines of simpsonite and microlite shown correspond to the lines on the extreme right of Figures 1 and 2.

X-RAY POWDER PHOTOGRAPHS

TABLE 9.—*Concluded*

hkl†	I/I*‡	arc mm.*	$\theta^*$	$\text{Sin}^2\theta^*$	$\text{Sin}^2\theta^\dagger$	d in Au. †
0004	3.2	235.7	1.03527	.7396	.732761	1.1233
1014	1.4	241.2	1.05942	.7605	.755063	1.1078
50 $\bar{5}$ 2						
51 $\bar{6}$ 1	1.8	247.4	1.08666	.7833	.778054	1.0915
4043						
11 $\bar{5}$ 4	2.3	253.9	1.11520	.8064	.800985	1.0758
33 $\bar{6}$ 2						
20 $\bar{2}$ 4	3.6	260.1	1.14244	.8274	.823642	1.0620
42 $\bar{6}$ 2						
60 $\bar{6}$ 0	5.0	267.4	1.17450	.8510	.846373	1.0472
32 $\bar{5}$ 3						
4370	5.5	274.7	1.20656	.8730	.869024	1.0339
60 $\bar{6}$ 1						
21 $\bar{3}$ 4	9.1	282.6	1.24126	.8952	.892203	1.0210
41 $\bar{5}$ 3						
51 $\bar{6}$ 2	4.0	301.7	1.32516	.9408	.938096	.9959
43 $\bar{7}$ 1						
5270						
30 $\bar{3}$ 4						
52 $\bar{7}$ 1						

localities<sup>2</sup> is likewise of interest. The measurement of the simpsonite Debye pattern is shown in Table 9, and corresponding measurements for microlite in Table 10.

X-ray photographs were obtained from the Picu material using the asymmetrical focusing camera of the Phragmen type and titanium radiation. A Debye pattern of simpsonite from Equador, Brazil, was also obtained with the same radiation. The widely separated lines due to the focusing principle and the longer wave length permit more accurate indexing than the more crowded patterns taken with iron radiation.

Symmetrical focusing powder photographs of the so-called precision type were taken in a camera of 10-cm. diameter. The simpsonite photographed was from the Equador locality, and precision-lattice constants<sup>3</sup> were computed using the method of Cohen (1935). The lattice constants are:

$$a_0 = 7.376603 \pm .000558; c_0 = 4.514119 \pm .000165; c_0/a_0 = .611951$$

Both the precision pattern and the other powder photographs were indexed on the basis of the rotation data.

X-ray powder photographs obtained in the symmetrical focusing camera are shown in Figure 3 of Plate 3, for both simpsonite and microlite. Most of the lines in these precision patterns are due to combined reflections from more than one set of planes. The indices of the several sets of each responsible for one doublet are as shown on page 496.

<sup>2</sup> Microlite in the Bikita specimens available was too disseminated to be identified by other than microscopic methods.

<sup>3</sup> A description of the method is given by Kerr, Holmes, and Knox (1945).

Simpsonite					
(304)	(214)	(601)	(323)	(204)	(114)
(521)	(413)		(430)	(422)	(332)
	(431)			(600)	
	(512)				
	(520)				
Microlite					
	(666)	(773)	(755)	(844)	(931)
	(10.22)	(951)	(771)		
			(933)		

On the films the K-alpha doublets are resolved, the stronger line in each case being  $\alpha_1$ .

Since the computation of the few powder reflections of both simpsonite and microlite yields data more precise than the Debye photographs an outline of the computations is given in Table 11. These follow the methods outlined by Jette and Foote (1935) and Cohen (1935). In the computation  $A$  is the  $\sin^2\theta$  value for (100), and  $B$  for (001), while  $D$  is the drift constant.

#### BIKITA, SOUTHERN RHODESIA

The simpsonite from Southern Rhodesia (Fig. 1) occurs in dark-brown crystals irregular in outline and intergrown with pegmatitic feldspar. Anhedral crystals up to half an inch in diameter were observed in the material available for study. Small chips were oriented by means of interference figures in order to obtain Laue photographs.

The Laue photograph agrees with the Laue photograph reproduced by Taylor (1939). Debye patterns of the brown Bikita material agree with patterns of simpsonite from Australia and also Brazil (Fig. 4). The chemical analysis of the Bikita simpsonite is shown in Table 4 along with a summary of the spectrographic data. A computation of the formula on the basis of the chemical analysis is shown in Table 7. Although the simpsonite is believed to agree with the simpsonite from Equador, Brazil, shown in Tables 4 and 5, the ratio of  $\text{Al}_2\text{O}_3:\text{Ta}_2\text{O}_5$  is not so close to 3:2 as for the Brazilian sample.

The Bikita simpsonite contains less columbium than the Brazilian material. This may be attributed to the isomorphism between tantalum and columbium so common among tantalum minerals. The formula is thus  $\text{Al}_6(\text{Ta}, \text{Cb})_4\text{O}_{19}$  with columbium as well as tantalum being included. The sample available was not so free from impurities as the Equador simpsonite. A thin section shows associated sericite, orthoclase, microlite, quartz, and iron stain. Minute crystals which appear to be cassiterite are also present. Taking the impurities into consideration the Bikita material appears to be essentially the same chemically as the simpsonite from other localities.

Optically the Bikita mineral is uniaxial negative with indices of refraction  $n_\omega = 2.06$  and  $n_\epsilon = 2.03 \pm .01$ , as determined by piperine-iodide and sulphur-selenium melts. The birefringence as shown in thin section is about 0.039.

TABLE 10.—X-ray powder data for microlite

Onça, Picuí, Paraíba, Brazil

(Radius = 57.015 mm.; Iron radiation;  $\alpha_1$  lines;  $\theta$  = radians)

(hkl)	( $h^2 + k^2 + l^2$ )	I/I <sub>1</sub>	arc in mm. $\alpha_1$ -Lines	$\sin^2\theta$ (based on precise $a_0$ )	$\sin^2\theta$ (from Debye film)	d in Å (based on precise $a_0$ )
(111)	3	3.1	36.98	.025895	.0260	6.00354
(220)	8	—	—	—	—	—
(311)	11	4.2	71.69	.094949	.0954	3.13508
(222)	12	6.8	75.01	.103580	.1041	3.00162
(400)	16	3.1	87.05	.138107	.1384	2.59948
(331)	19	1.6	95.27	.164002	.1642	2.38545
(422)	24	—	—	—	—	—
(333)	27	3.7	115.10	.233056	.2338	2.00108
(511)						
(440)	32	8.4	126.32	.276214	.2761	1.83811
(531)	35	3.7	132.84	.302109	.3020	1.75757
(442)	36	—	—	—	—	—
(600)						
(620)	40	—	—	—	—	—
(533)	43	1.6	149.48	.371163	.3708	1.58567
(622)	44	10.0	151.54	.379794	.3795	1.56755
(444)	48	4.2	159.56	.414321	.4139	1.50081
(551)	51	4.7	165.47	.440216	.4395	1.45600
(711)						
(642)	56	—	—	—	—	—
(553)	59	6.3	181.32	.509270	.5088	1.35369
(731)						
(800)	64	1.6	190.99	.552428	.5510	1.29974
(733)	67	1.0	196.70	.578323	.5800	1.27031
(644)	68	—	—	—	—	—
(660)	72	.5	207.00	.621482	.6240	1.22541
(822)						
(555)	75	2.1	213.75	.647377	.6530	1.20065
(751)	76	6.8	215.28	.656008	.6592	1.19272
(662)						
(840)	80	6.3	223.63	.690535	.6940	1.16252
(753)	83	3.2	230.02	.716430	.7195	1.14132
(911)						
(842)	84	—	—	—	—	—
(664)	88	1.6	240.80	.759589	.7610	1.10842
(931)	91	3.2	247.92	.785484	.7873	1.09000
(844)	96	6.3	260.42	.828642	.8300	1.06123
(755)	99	2.6	268.58	.854537	.8560	1.04503
(771)						
(933)	104	—	—	—	—	—
(862)						
(10.2.0)	107	4.2	293.84	.923591	.9250	1.00520
(773)						
(951)	108	6.8	297.59	.932224	.9335	1.00054
(666)						
(10.2.0)	115	—	—	—	—	—
(953)						

TABLE 11.—*Precision camera data*  
Simpsonite, Alto do Giz, Equador, Rio Grande do Norte, Brazil

hk.l	$\lambda$	arc in mm.	$\phi$	$\phi \sin \phi$	$\sin^2 \theta$ (Observed)	$\sin^2 \theta$ (Calculated)	$\Delta \sin^2 \theta$	d in Å (from $a_0$ )								
(20.4) *(42.2) (60.0)	$\alpha_2$	173.910	.426038	.31935	.825789	.825809	.000020	1.06445								
*(32.3) (43.0)									$\alpha_1$	163.013	.399342	.28610	.848771	.848747	.000024	1.05005
*(60.1)																
		147.883	.362277	.23914	.870792	.870772	.000020									
*(30.4) (52.1)	$\alpha_1$	101.518	.248694	.11866	.939362	.939433	.000071	.99740								
		$\alpha_2$	98.120	.240371	.11071	.939439	.939375	.000464								

\* Indices used in computation;  $c_0 = 4.514119 \pm .000165$ ;  $a_0 = 7.376603 \pm .000558$ ;  $A = .02286727$ ;  $B = .04579756$ ;  $C = .00731023$ .

Camera constant  $\frac{1}{8R} = .002449933$ ; shrinkage correction = 1.00017702; refraction correction = .000054; iron radiation.

Microlite, Onça, Picuí, Paraíba, Brazil

hk.l	$\lambda$	arc in mm.	$\phi$	$\phi \sin \phi$	$\sin^2 \theta$ (Observed)	$\sin^2 \theta$ (Calculated)	$\Delta \sin^2 \theta$	d in Å (from $a_0$ )								
*(844)	$\alpha_1$	173.790	.426976	.32189	.828455	.828519	.000064	1.06123								
		$\alpha_2$	171.933	.422414	.31462	.828499	.828522	.000123								
(755) *(771) (933)	$\alpha_1$	159.240	.391229	.27583	.854541	.854432	.000109	1.04503								
*(773) (951)									$\alpha_1$	113.974	.280017	.14875	.923569	.923544	.000025	1.00520
*(666) (10.22)																
	$\alpha_2$	104.070	.255685	.12461	.932185	.932175	.000010									

\* Indices used in computation;  $a_0 = 10.397916 \pm .000043$ ;  $A = .00863169$ ;  $D = .00038320$ ;  
Camera constant  $\frac{1}{8R} = .00244933$ ; shrinkage correction = 1.00307075; refraction correction = .000050; iron radiation.

SPECTROGRAPHIC DATA

The spectrograms shown in Figure 1 of Plate 4 were taken by Dr. W. M. Shafer on material submitted for chemical analysis by Mr. R. W. Hoffman. A résumé of the spectrographic data appears in Table 4. Corresponding samples have been studied optically and by x-rays.



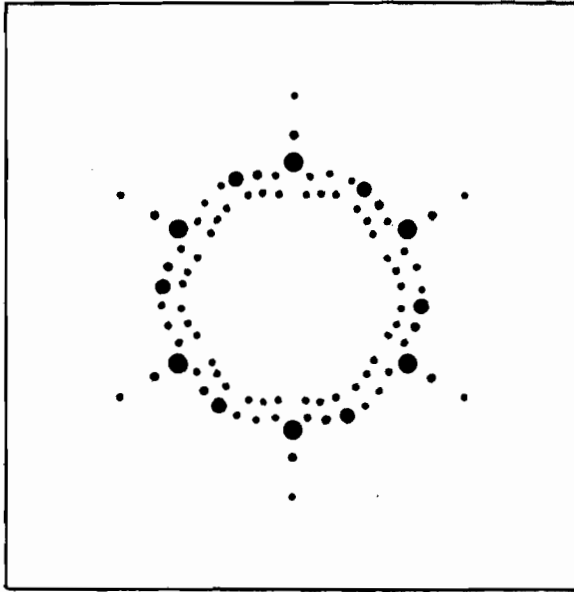


FIGURE 3.—*Stereographic projection of Taylor's Laue photograph*  
Simpsonite from Tappa Tappa, Western Australia taken normal to (0001) (see Taylor, 1939).

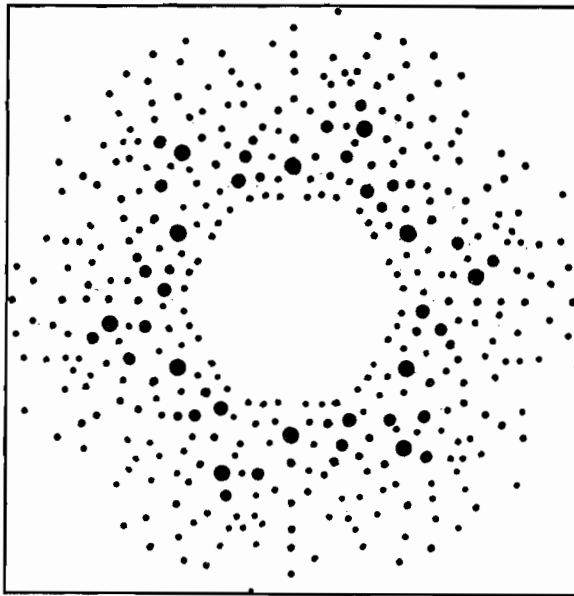


FIGURE 4.—*Stereographic projection of a Laue photograph of simpsonite from Picuá, Brazil*  
Taken normal to (0001). Inner spots of this projection duplicate the spots of Figure 3 representing simpsonite from Tappa Tappa, Western Australia, as illustrated by Taylor (1939).

A significant feature of the spectrograms of the two simpsonite samples reproduced is the presence of prominent lines due to aluminum at 3961.5 and 3944.0 angstroms. Equally significant are the two calcium lines in the spectrogram of microlite at 3968.4 and 3933.6 angstroms. Much weaker lines due to calcium appear in the simpsonite spectrograms, and weaker lines due to aluminum appear in the microlite spectrogram.

Tantalum lines are shown in the three spectrograms at 3511.0, 3311.2, and 3012.5 angstroms. Lines due to tin, silicon, and manganese are indicated in the Bikita sample of simpsonite, while in the Equador sample of the same mineral lines due to sodium, titanium, silicon, and manganese are indicated. Titanium is practically absent in the Bikita sample. The spectrogram of microlite shows lines due to titanium, sodium, and magnesium, in addition to those mentioned above. Columbium at 4058.9 angstroms appears to be present in the three samples under investigation.

#### SPECIFIC GRAVITY

The specific gravity of the Australian simpsonite crystals as given by Bowley was 6.525 for one determination and 6.27 for another. The two specimens from the U. S. National Museum examined in this work yielded specific gravities of 5.941 and 6.009. Since both represented mixtures of simpsonite and microlite and were made up of slightly porous material the values are probably somewhat low. A simpsonite crystal from Brazil furnished by the American Museum of Natural History yielded a specific gravity of 6.501. This crystal also contained a small amount of alteration material, and the determination is probably low. The best crystal available from Brazil, furnished by Mr. Wiegand, weighed 15.594 grams and was free from alteration visible to the eye. The average of three determinations on this crystal was 6.700. Determinations were made on a balance previously described by Kerr (1936). A small crystal from Picuí gave a specific gravity of 6.748.

#### HARDNESS

The hardness of simpsonite is greater than quartz on the major prism face, but less on the basal pinacoid as tested according to the indentation method of Peters and Knoop (1940). An indenter used for testing hardness was made available through the courtesy of Dr. Henry R. Juneman of the School of Dental and Oral Surgery of Columbia University.

#### FLUORESCENCE

Specimens of simpsonite fluoresce bluish white to pale yellow under the mercury vapor arc in a quartz tube. Recently abraded surfaces fluoresce to best advantage. Investigation with the high-frequency iron arc did not disclose fluorescence worthy of note.

Among the specimens of simpsonite available for examination the following fluorescence was observed:

	Fluorescence	Natural color
Bikita, Southern Rhodesia . . . . .	pale yellow	light brown
Equador, Brazil . . . . .	pale yellow	brownish yellow
Picuí, Brazil . . . . .	pale yellow	brownish yellow
Tabba Tabba, Western Australia . . . . .	light blue	grayish white

## LAUE X-RAY PHOTOGRAPHS

Laue patterns of simpsonite, using platinum radiation, were obtained from thin slices of crystals from Tabba Tabba, Picuí, Equador, and Bikita. These were cut parallel to (0001) and photographed with the *c*-axis parallel to the x-ray beam. Many slices which appeared to be single crystals were groups of almost parallel individuals, frequently resulting in at least partial superposition of several Laue patterns on the same film.

A stereographic projection of Taylor's Laue photograph showing only spots due to one crystal is shown in Figure 3. A corresponding stereographic projection based upon a crystal from Picuí is shown in Figure 4. Since the projections of crystals from Tabba Tabba and Bikita are essentially the same they are not reproduced.

The Laue patterns are hexagonal without vertical planes of symmetry. This was noted by Taylor. Intensity variations have not as yet been interpreted. Taylor also noted a plane of symmetry parallel to (0001). This has been confirmed in a Laue photograph normal to a major prism face (Pl. 2, fig. 1).

## ROTATION PHOTOGRAPHS

Rotation photographs were taken in duplicate using both copper and iron x-ray targets. The shorter wave length of copper furnished a larger number of layer lines to guard against the possibility of overlooking weak layers in determining identity periods. The longer wave length of iron furnished a greater separation of spots for more reliable graphical indexing. Crystals were rotated about the *c*-axis, the *a*-axis, and an axis normal to (10 $\bar{1}$ 0).

Since only large crystals made up of small individuals were available considerable difficulty was experienced in selecting and orienting mounts. Chips with but a single face were first oriented optically or on a goniometer. The orientation was perfected by taking a succession of rotation photographs, adjusting the crystal holder of the camera between each exposure. Although a considerable number of trials were necessary a satisfactory orientation was obtained in each case. Suitable photographs were secured by rotation around each of the three axes indicated for crystals from the Equador locality in Brazil (Pl. 4, fig. 2).

The identity periods corresponding to the spacing of the layer lines of the rotation photographs provide the general dimensions of the lattice constants of the primitive unit cell. Since it was not known whether the major prism of a simpsonite crystal should have the indices {10 $\bar{1}$ 0} or {11 $\bar{2}$ 0} identity periods were used to establish the correct *a*-axis. Rotation photographs were obtained using axes in the horizontal plane perpendicular to faces of each prism. On the basis of the equation,

$$i = \frac{n\lambda}{\sin \tan^{-1} \left( \frac{y_n}{r} \right)},$$

where *i* = identity period  
*n* = order of layer  
 $\lambda$  = wave length of x-rays  
 $y_n$  = ordinate distance on film  
*r* = radius of camera.

The identity periods were determined as shown in Table 12.

A careful search of each photograph was made for weak intermediate layer reflections which might increase the value of the identity period, but no such reflections

TABLE 12.—Identity periods of rotation photographs

Rotation about axis normal to:	$t$	Indices	Number of layers including zero
Basal pinacoid.....	4.514	(0001)	3
Major prism face.....	12.776	(10 $\bar{1}$ 0)	8
Minor prism face.....	7.376	(11 $\bar{2}$ 0)	5

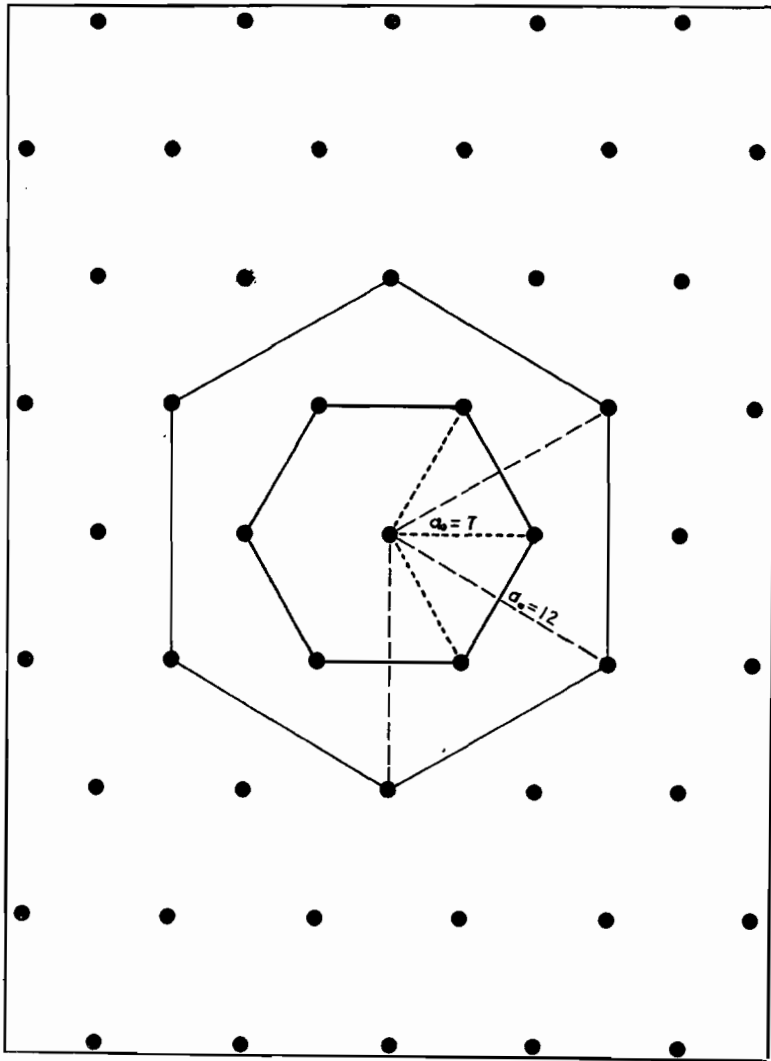


FIGURE 5.—Alternative cell arrangements for simpsonite

The cell is triply primitive if the identity period 12.776 normal to the major prism faces is selected as  $a_0$ . The smallest possible unit cell or primitive cell results if the identity period 7.376 normal to the minor prism faces is selected.

were observed. A few erratic reflection spots were noted, but their relation to the regular series suggests that they are due to satellite crystals.

The two alternative selections of  $a_0$  based on the identity periods are shown in Figure 5. If the identity period with the value 12.776 is accepted for  $a_0$  the unit cell becomes the rhombic section of the type included in the outer hexagonal framework. If the identity period with the value 7.376 is used the unit cell would have the rhombic section included in the smaller hexagonal framework, which is the primitive cell.

Taylor (1939) on the basis of Bragg reflections concluded that the spacing of planes parallel to (0001) was about  $4.5\text{\AA}$ ., while the spacing parallel to the prism (presumably  $\{10\bar{1}0\}$ ) was  $6.3\text{\AA}$ .. On the basis of the rotation photographs these figures agree roughly with the identity period along the  $c$ -axis and half the identity period for rotation about an axis normal to the major prism.

The reflections on the rotation photographs were indexed graphically with the assistance of the reciprocal lattice. The  $\xi$  values were obtained with the aid of Bernal's (1926) chart. The reciprocal lattice grid was drawn on the basis of the lattice constants derived from the spacing of the layer lines on the rotation photographs, the intersections of the arcs, whose radii are proportional to the  $\xi$  values, with the nodes of the grid permit the indices for the spots to be read directly.

All the spots on the three rotation photographs taken with iron radiation were identified. In addition the spots on the  $c$ -axis rotation were identified with copper wave length. Layers on the copper-radiation rotation photographs normal to  $(10\bar{1}0)$  and  $(11\bar{2}0)$  lying beyond the range of iron radiation were also indexed. The indices determined for the rotation photographs account for all lines on the powder photographs.

#### LATTICE CONSTANTS

The identity period along the  $c$ -axis is the lattice constant  $c_0$  which has the value 4.514119. Either identity-period value obtained by rotating about normals to the two alternate prism faces could serve as the lattice constant for the  $a$ -axis. However, as shown in Figure 5 if the value along the axis normal to the major prism (12.776645) is accepted a multiply primitive unit cell results. On the other hand, if the identity period along the normal to the minor prism face is accepted a primitive (smallest possible) unit cell is obtained. Hence the correct orientation for simpsonite based on the smallest possible cell requires the value 7.376603 to be used as the lattice constant  $a_0$ . Preliminary values for the lattice constants were obtained from the measurement of the rotation diagrams. These were corrected by using the high-angle reflections on the Debye powder patterns and further refined on the basis of precise lattice constants obtained from the symmetrical focusing camera pattern. The values given above are based on the precision camera computations.

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MANUSCRIPT RECEIVED BY THE SECRETARY OF THE SOCIETY, MARCH 30, 194