

SCRUTINYITE, NATURAL OCCURRENCES OF αPbO_2 FROM BINGHAM, NEW MEXICO, U.S.A., AND MAPIMI, MEXICO

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ABSTRACT

SOMMAIRE

Scrutinyite, αPbO_2 , identified from the Hansonburg mining district, Bingham, Socorro Co., New Mexico, and from the Ojuela mine, Mapimi, Durango, Mexico, is the first-reported natural occurrence of the compound. The mineral occurs at both localities as an oxidation product of hydrothermal mineralization. Scrutinyite is translucent red-brown (Hansonburg) to less translucent clove-brown (Ojuela). Both are nonfluorescent (UV light). Individual crystals from both localities are generally euhedral, 25–30 μm across and 1–2 μm thick. Scrutinyite from both localities is intimately associated with plattnerite; at the Ojuela mine, the scrutinyite occurs within plattnerite partly paramorphous after scrutinyite. $D_{\text{calc}} = 9.867 \text{ g/cm}^3$, luster submetallic, streak dark brown. The mineral shows two cleavages, one, probably {100}, excellent, and the other, probably {010}, imperfect, both by analogy with columbite. In reflected light, grey-white with reddish brown internal reflections; weakly anisotropic with a bluish color; bireflectance very weak, grey-white to bluish grey; pleochroism absent. Reflectance values, determined in air for two orientations, one parallel and one perpendicular to the principal cleavage, are: 17.0, 17.7, 17.9 at 589 nm; 17.9, 18.2, 18.8 at 546 nm. Cell dimensions calculated from refinement of powder data, pertain to an orthorhombic phase: a 4.971(2), b 5.956(2), c 5.438(3) Å, V 161.00(9) Å³, $Z_0 = 4$. The strongest lines in the X-ray powder pattern [d in Å (hkl)] are: 3.117(100)(111), 1.840(80)(130), 2.722(50)(002), 1.527(50)(132), 3.816(40)(110), 1.635(40)(113), 2.606(30)(021), 2.483(30)(200), 1.799(30)(221). Electron-microprobe analyses (mean of 10) gave PbO_2 98.2(8) wt.%. The idealized formula is PbO_2 . The name reflects the close scrutiny and sustained examination required to determine the physical properties with a very limited amount of sample material. The holotype specimen is preserved at the U.S. National Museum (NMNH 165479).

Keywords: new mineral species, scrutinyite, columbite, αPbO_2 , X-ray-diffraction pattern, chemical analysis, Bingham, New Mexico, Mapimi, Mexico.

La scrutinyite, nouvelle espèce minérale équivalente au composé αPbO_2 , est signalée dans le camp minier de Hansonburg, à Bingham, comté de Socorro, au Nouveau-Mexique, ainsi qu'à la mine de Ojuela, à Mapimi, dans l'état de Durango, au Mexique. Aux deux endroits, c'est un produit de l'oxydation d'un minerai hydrothermal. Translucide rouge-brun (Hansonburg) à semi-opaque brun-clou (Ojuela), la scrutinyite ne montre aucune fluorescence en lumière ultra-violette. Elle forme en général des cristaux idiomorphes de 25 à 30 μm de large et de 1 à 2 μm d'épaisseur. Elle est intimement associée à la plattnerite; à Ojuela, elle se trouve dans un amas paramorphique de plattnerite. Densité calculée 9.867, éclat sub-métallique, rayure brun foncé. Elle montre deux clivages, dont un, probablement {100}, est excellent, et l'autre, probablement {010}, est imparfait, ce qui ressemble au cas de la columbite. Gris-blanc en lumière réfléchie, elle montre des réflexions internes brun rougeâtre; elle est faiblement anisotrope, et bleuâtre. Biréflectance très faible, gris-blanc à gris bleuâtre, et pléochroïsme absent. Les valeurs de la réflectance, déterminées dans l'air pour deux orientations, parallèle et perpendiculaire au clivage principal, sont: 17.0, 17.7, 17.9 à 589 nm, 18.2, 18.8 à 546 nm. Les dimensions de la maille, calculées des données obtenues du cliché de diffraction X (méthode des poudres), montrent qu'il s'agit d'un minéral orthorhombique: a 4.971(2), b 5.956(2), c 5.438(3) Å, V 161.00(9) Å³, $Z = 4$. Les neuf raies les plus intenses du cliché de poudre [d en Å (hkl)] sont: 3.117(100)(111), 1.840(80)(130), 2.722(50)(002), 1.527(50)(132), 3.816(40)(110), 1.635(40)(113), 2.606(30)(021), 2.483(30)(200), 1.799(30)(221). La moyenne de dix déterminations de sa composition à la microsonde électronique donne 98.2(8)% (par poids) de PbO_2 . La formule idéale est donc PbO_2 . Le nom rappelle l'examen minutieux et soutenu qui s'est révélé nécessaire pour déterminer les propriétés physiques sur les quantités infimes disponibles. L'échantillon holotype est conservé au U.S. National Museum (NMNH 165479).

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Mots-clés: nouvelle espèce minérale, scrutinyite, columbite, αPbO_2 , cliché de diffraction X, analyse chimique, Bingham, Nouveau-Mexique, Mapimi, Mexique.

INTRODUCTION

The orthorhombic form of lead dioxide, αPbO_2 , which has been known as a synthetic compound since 1946 (Kamayama & Fukumoto), has been discovered in small quantities at Bingham, New Mexico, and Mapimi, Durango, Mexico (Taggart *et al.* 1984). The compound αPbO_2 , one of what are considered fundamental structure-types within the MO_2 oxides, has great significance and a strong relationship to the structures and substructures of the columbite-tantalite group (ixiolite, columbite-tantalite, wodginitite). The αPbO_2 structure type has been used as a model in discussions of MO_2 -type compounds and Nb-Ta oxides by numerous authors (*e.g.*, Laves *et al.* 1963, Graham & Thornber 1974a,b, Weitzel 1976).

NAME AND STORAGE

The name *scrutinyite*, SKRŌŌTŃ-Ē-ITE, has been given to the naturally occurring material. The name is from the word scrutiny: a close, careful examination or study; a critical sustained look. This name refers to the initial misidentification of the mineral from both occurrences, the close scrutiny required to recognize the unique properties of the material, and the sustained examination required to determine the physical properties with a very limited amount of sample material. The mineral and name have been approved by the Commission on New Minerals and Mineral Names of the International Mineralogical Association.

The holotype specimen is preserved at the U.S. National Museum, catalog number NMNH 165479. The specimen is from the Sunshine #1 Tunnel (Taggart *et al.* 1989), Blanchard Claims, Hansonburg Mining District, Bingham, Socorro Co., New Mexico, U.S.A. All the data on scrutinyite were generated with material from the holotype sample.

OCCURRENCE

Scrutinyite was first recognized on material collected by T.H. in December, 1981, at the Sunshine #1 mine. The mineral was suspected initially to be minium because of its high lead content, its reddish brown color, and its association with the lead minerals murdochite and plattnerite; however, powder X-ray data and a euhedral crystal form suggested some other mineral. In April, 1983, one of two specimens recovered was forwarded to the U.S.G.S. (J.T.) for further study. The location of only two samples is currently known. The first is established as the holotype specimen, and contains less than 1 mg of material, with crystals about 25 to 30 μm across and 1 to 2 μm thick. These flakes are red-brown and thin enough to be translucent. The

mineral is associated with murdochite and plattnerite, and was deposited during oxidation-stage mineralization on a matrix of hypogene fluorite and quartz, on silicified limestone. The second locality is Mapimi, Durango, Mexico. This material was given to us in 1977 by Ramon DeMark, who purchased it from local gambusinos. The mineral occurs on rosasite on a limonite matrix. From the habit of the matrix and mineral association, the material may have come from the Mina Ojuela (Ojuela mine). The scrutinyite on this material is almost the same in habit as the Hansonburg material, but is closer to clove-brown in color, is not as translucent, and occurs in minute quantities within plattnerite paramorphs after scrutinyite. The presence of the plattnerite interferes with the X-ray-diffraction pattern for scrutinyite, and accounts for our inability to recognize the scrutinyite until after the mineral was identified from Bingham. Indeed, the two strongest diffraction-maxima of scrutinyite are present in only one specimen of the Mapimi material (Rosenzweig & Taggart 1984). Because the Hansonburg District produced the purest material on which virtually all of the testing was performed, and has the best documentation of location, it is selected as the type locality.

CHEMISTRY

Energy-dispersion qualitative analysis with a Cambridge[†] SEM showed only the presence of lead. Because the phase is present as very thin, fragile flakes, the type of sample preparation that can be performed is limited. A polished section parallel to the main cleavage was prepared only with great difficulty. The section has an area of about 2 μm across and was used to perform a microprobe analysis with an ARL-EMX-SEM[†]. Ten analyses gave a mean of 98.2(8) wt. % PbO_2 . Analyses were performed using synthetic PbF_2 ($\text{PbL}\alpha$) as a standard, and the data were corrected with the MAGIC-4 computer program. The idealized formula is PbO_2 . Two possibilities are considered to be the most likely causes for the low total: beam penetration and non-stoichiometry. Despite a low operating voltage of 15 kV, the extreme thinness and small grain-size of the polished flake resulted in overlap of the microprobe beam with the edges of the grain; also, the beam penetrated and exited the other side of the grain. In addition, Bagshaw *et al.* (1966), citing Byström (1945), reported: "It is well known that stoichiometric lead dioxide does not exist and that the oxide normally contains about 3% of hydroxyl ions replacing oxygen anions in the structure." Bagshaw *et al.*

[†]The use of brand names is for descriptive purposes only and does not constitute endorsement by the U.S. Geological Survey.

gave chemical compositions for five αPbO_2 samples prepared by various techniques. The compositions range from $\text{PbO}_{1.80}(\text{OH})_{0.16}$ to $\text{PbO}_{1.83}(\text{OH})_{0.23}$. Santoro *et al.* (1983) and A. Santoro (oral commun. 1983) reported that the structure of αPbO_2 has not been solved completely, and that there appear to be many structural defects in addition to the presence of hydrogen. They reported that this hydrogen may be present as OH, and probably balances the charge difference attributable to the presence of some Pb^{2+} in the Pb^{4+}O_2 .

CRYSTALLOGRAPHY

Synthetic αPbO_2 was discovered by Kamayama & Fukumoto (1946), and the structure was characterized by Zaslavskii *et al.* (1950) and Zaslavskii & Tolkachev (1952a, b, 1953). The structure they described is similar to that determined by Sturdivant (1930) for columbite. With lead atoms fully occupying all metal positions in αPbO_2 , however, there can be no cation ordering as in columbite, in which two nonequivalent metal positions are present. Nickel *et al.* (1963) pointed out that there is some confusion in the indexing for columbite-tantalite, since at least three axial settings are given in the literature. Grice *et al.* (1976) summarized the various crystallographic settings used by different authors for the columbite structure, and pointed out that "The tantalite/columbite cation-ordered structure may be regarded as the prototype of these three minerals [columbite/tantalite, ixiolite and wodginite] because it was the first to be solved (Sturdivant, 1930); however, from a structural viewpoint, the cation-disordered structure of ixiolite with the small cell and the same structure as αPbO_2 ... should be regarded as the prototype." We will use the setting of Grice *et al.* (1976), which is also the same as that used by Strunz (1970), Wyckoff (1965) and Povaren-

nykh (1972). Specifically, for columbite/tantalite, a 14.4, b 5.8, c 5.1 Å, space group *Pbcn*; for ixiolite a 4.8 Å (this represents 14.4/3 Å), b 5.8, and c 5.2 Å, space group *Pbcn*.

The PbO_6 octahedra of αPbO_2 form edge-sharing zig-zag chains in the bc plane, with their long axis parallel to the c axis (Fig. 1). These chains are stacked along the a direction by the sharing of one corner of each octahedron. This differs from the tetragonal (rutile) structure of βPbO_2 (plattnerite), where the basis is PbO_6 octahedra which, by sharing opposite edges, form straight chains parallel to the c axis. These chains are attached to one another by the sharing of one corner of each octahedron. By displacing alternate cations from the rutile chains along the a direction, Andersson & Galy (1969) demonstrated a simple mechanism for transforming the rutile structure into the αPbO_2 structure.

Table 1 shows a comparison of published X-ray-diffraction patterns for αPbO_2 , βPbO_2 , and the diffraction pattern determined with holotype scrutinyite during this study. The sample of αPbO_2 synthesized by K.A. Gross for PDF 11-549 was prepared electrochemically by the method of Clarke (1937). The card also refers to the data of Ruetschi & Cahan (1957) and Zaslavskii *et al.* (1950). The sample of βPbO_2 (White 1970; PDF 25-447) is natural plattnerite from the Ojuela mine, Mapimi, Durango, Mexico. The diffraction data for the type scrutinyite were obtained using a Gandolfi camera of 114.6 mm diameter, $\text{CuK}\alpha$ radiation, and a polycrystalline mount. The 2θ values were measured to the nearest 0.02 mm with a projection-screen emission-spectrography plate reader; intensities were estimated visually. The diffraction results establish that the major part of the Hansonburg material is identical to αPbO_2 ; however, a very small amount of plattnerite is present in the Hansonburg pattern. At this time there is no way of knowing if the plattnerite

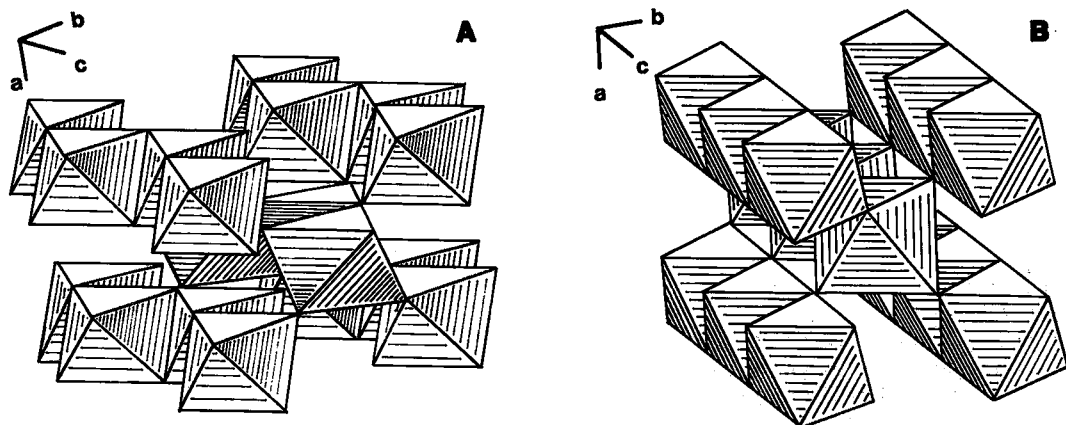


FIG. 1. (A) Structure of αPbO_2 , based on Povarennykh's (1972) illustration of columbite. (B) Structure of βPbO_2 (Povarennykh 1972).

TABLE 1. COMPARISON OF X-RAY-DIFFRACTION POWDER PATTERNS FOR SYNTHETIC αPbO_2 , NATURALLY OCCURRING PLATTNERITE, AND HOLOTYPE SCRUTINYITE

Zaslavskii <i>et al.</i> (1950) αPbO_2			JCPDS 11-549 αPbO_2			JCPDS 25-447 Plattnerite			Scrutinyite Hansburg District Bingham, New Mexico		
d Å	I	hkl	d Å	I	hkl	d Å	I	hkl	d Å obs $\pm 0.02^\circ 2\theta$	I	hkl
3.830	29	110	3.83	12	110				3.816	40	110
						3.50	100	110	3.497	30	
3.121	118*	111	3.12	100	111				3.117	100	111
2.969	11	020	2.97	15	020				2.985	5	020
						2.79	95	011	2.795	20	
2.737	46	002	2.74	70	002				2.722	50	002
2.605	53	021	2.63	70	021				2.606	30	021
2.467	32	200	2.48	20	200	2.469	40	020	2.482	30	200
2.212	37	112	2.23	6	112				2.217	10	112
2.010	28	022	2.02	6	022				2.010	10	022
1.895	30	220	1.89	30	220				1.909	10	220
1.834	100	130	1.84	45	130,202	1.855	80	121	1.840	80	130
1.793	54	221	1.79	30	221				1.799	30	221
						1.752	18	220	1.750	5	
						1.692	14	002	1.701	5	
1.638	55	113	1.64	15	113				1.635	40	113
1.559	50	222	1.56	17	222,023	1.568	20	130	1.557	20	222
1.523	95	311	1.53	30	311,132	1.524	25	112	1.527	50	132
						1.486	20	031	1.484	10	
1.431	34	041	1.43	20	041,312				1.436	10	041
						1.398	14	022	1.396	5	
1.365	23	312	1.37	15	312				1.377	10	312
1.314	26	223	1.31	15	223				1.315	10	223
1.262	36	330	1.26	20	330	1.274	20	231	1.272	10	330
1.237	39	241	1.24	30	241,400				1.243	20	400
						1.218	12	222	1.215	5	
1.195	39	204	1.20	40	204,313				1.198	20	313
									1.153	20	332
									1.133	10	151
									1.107	20	224

(* Site)

was introduced by contamination during the hand-picking of the sample, or if it occurs as a more intimate intergrowth of the two phases. Unit-cell parameters were determined by refinement of the powder-diffraction data assuming an orthorhombic cell. These data, in good agreement with the values of Zaslavskii *et al.* (1950) and Syono & Akimoto (1968), are shown in Table 2. Syono & Akimoto recognized that the unit-cell parameters they had determined on " PbO_2II " [αPbO_2] produced from " PbO_2I " [βPbO_2] during high-temperature - high-pressure stability experiments were "slightly larger than those of anodic deposits of PbO_2 in batteries, which were first reported by Zaslavskii *et al.* (1950)". The parameters for scrutinyite are intermediate between the two sets of published data.

Kamayama & Fukumoto (1946) found that PbO_2 electroplated on the anode from neutral or alkalic solutions gave a "new substance... perhaps a crys-

tal of the fluor spar type". Zaslavskii *et al.* (1950) electrochemically precipitated an orthorhombic form of PbO_2 which they named αPbO_2 . Syono & Akimoto (1968) claimed that the fluorite type of PbO_2 is stable above 50 kbars. Taken in conjunction, these observations seem to indicate that Kamayama & Fukumoto did not have a fluorite-type structure, but instead had αPbO_2 . This is supported by Bagshaw *et al.* (1966), who credited Kamayama & Fukumoto with the discovery of αPbO_2 .

PHYSICAL PROPERTIES

Scrutinyite is dark reddish brown, has a submetallic luster, a dark brown streak, and is translucent red on thin edges. The color and red internal reflections are similar to those displayed by some specimens of hübnerite. The mineral has two directions of cleavage, excellent in one direction, probably {100}

TABLE 2. COMPARISON OF CELL PARAMETERS OF αPbO_2 (ZASLAVSKII ET AL. 1950 AND SYONO & AKIMOTO 1968) AND SCRUTINYITE (THIS STUDY)

αPbO_2 Zaslavskii et al.		αPbO_2 Syono & Akimoto.		Scrutinyite This Study	
$a = 4.938 \text{ \AA}$		$a = 4.988(1) \text{ \AA}$		$a = 4.971(2) \text{ \AA}$	
$b = 5.939 \text{ \AA}$		$b = 5.958(1) \text{ \AA}$		$b = 5.956(2) \text{ \AA}$	
$c = 5.486 \text{ \AA}$		$c = 5.465(1) \text{ \AA}$		$c = 5.438(3) \text{ \AA}$	
$\bar{V} = 160.887 \text{ \AA}^3$		$\bar{V} = 162.4(1) \text{ \AA}^3$		$\bar{V} = 161.00(9) \text{ \AA}^3$	
$Z = 4$		$Z = 4$		$Z = 4$	

by analogy with columbite, and imperfect in another direction estimated to be 90° to the first, and probably $\{010\}$ by analogy with columbite. The excellent cleavage produces small flakes. Hardness and density could not be measured because of the small grain-size and paucity of material. The density calculated from the stoichiometric chemistry and the volume of the unit cell determined from the X-ray powder pattern (Appleman & Evans 1973) is 9.867 g/cm^3 , which compares favorably with the literature value of 9.875 (PDF 11-549). The mineral is not fluorescent under ultraviolet light.

OPTICAL PROPERTIES

In reflected light, scrutinyite in polished sections is grey-white with reddish brown internal reflections. Under crossed nicols, the phase is weakly anisotropic, with a bluish color; bireflectance is a very weak, grey-white to bluish grey; pleochroism is absent. Reflectance values were determined in air, using a Carl Zeiss SiC #052 standard, on two oriented grains of scrutinyite, the first mount with the polished surface parallel to the principal direction of cleavage, probably $\{100\}$, and the second with the polished surface perpendicular to the principal cleavage. At 589 nm , reflectance values are 17.0 , 17.7 , and 17.9 ; at 546 nm , 17.9 , 18.2 , and 18.8 . The surface area of the parallel section was 2 by $2 \mu\text{m}$, and that of the perpendicular section was 2 by $0.5 \mu\text{m}$.

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