Rynersonite, Ca(Ta,Nb)₂O₆, a new mineral from San Diego County, California

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Abstract

Rynersonite, a new species isostructural with synthetic $CaTa_2O_6$, is found in the Himalaya pegmatite-aplite dike system, Mesa Grande district, San Diego County, California. It occurs as creamy-white to reddish-pink felted masses of fibrous to lath-like crystals. Individual laths average less than 0.5 mm in length and 0.05 mm in thickness. Electron diffraction patterns show that the laths are elongated along a and flattened on (010). Hardness is about 4.5; luster earthy; streak white; fracture uneven. Rynersonite is biaxial positive with moderate $2V$; indices of refraction are greater than 2.05; birefringence is about 0.14; $X = c$, $Y = b$, $Z = a$; with $X = Y =$ straw-yellow, $Z =$ light straw-yellow.

Reddish-pink rynersonite is orthorhombic, space group $Pnmb$, with $a = 7.505(1)$, $b = 11.063(2)$, $c = 5.370(1)$Å, $Z = 4$, Gcalc = 6.394; for the composition $Ca(Ta_{1.21}Nb_{0.77})O_6$, Gmeas = 6.402. The six strongest lines of the X-ray pattern (d, in Å; intensity; hkl) are 3.038 *100* 031, 4.835 *91* 011, 2.964 *91* 211, 3.754 *71* 200, 2.683 *50* 002, 2.359 *50* 231. Heating in air for 20 hours at 550°, 800°, and 1050°C produces no irreversible changes in cell dimensions or structure.

Electron microprobe analyses indicate a range of composition from $Ca(Ta_{1.44}Nb_{0.51})O_6$ to $Ca(Ta_{1.21}Nb_{0.77})O_6$. Rynersonite formed as an alteration product of stibiocolumbite-stibiotantalite crystals and is intimately associated with minor amounts of antimonian microlite and fersmite, $Ca(Nb,Ta)_2O_6$. Although rynersonite and fersmite are both orthorhombic AB_2O_6 compounds, they differ structurally. The new mineral has space group $Pnmb$; fersmite, $Pcan$.

The mineral is named for Eugene B. and Buel F. Rynerson, and their late father, Fred J. Rynerson, in recognition of their lifelong interest in mining the gem-bearing pegmatites in Mesa Grande, California.

Introduction

During an investigation of the mineralogy and petrology of the Himalaya pegmatite–aplite dike system (Foord, 1976, 1977), located in the Mesa Grande district, San Diego County, California (33° 12'42" N, 116° 47'56" W), several well-crystallized specimens of stibiocolumbite–stibiotantalite, for which the dike system is world-famous (Penfield and Ford, 1906), were given by Eugene B. Rynerson, the owner of the San Diego Tourmaline mine, to Foord for study purposes. On these specimens, as well as on an unusual specimen of stibiotantalite from the San Diego mine property, was found a creamy-white to pale-pink fibrous mineral. The unusual stibiotantalite (specimen SD–W) earlier had been sent by its owner, William C. Woynar of San Diego, California, to Dr. George Switzer of the Smithsonian Institution, Washington, D. C., for identification, and was subsequently referred by him to one of us (MEM). The X-ray powder diffraction data did not match that of any known mineral; its powder data showed, however, that the mineral is isostructural with $CaTa_2O_6$, a compound that had been synthesized by Jahnberg *et al.* (1959) and later characterized structurally by Jahnberg (1963).

The name rynersonite (pronounced rī-nur-sun-ite)

is proposed for this new mineral, having composition $Ca(Ta,Nb)_2O_6$ with Ta greater than Nb, to honor the more than 70 years of mining activity by the late Fred J. Rynerson (1882–1960) and his two sons, Eugene B. and Buel F., in the Himalaya pegmatite-aplite dike system as well as in other gem-bearing pegmatites within San Diego County, and in recognition of their lifelong interest in gem-bearing pegmatites. The description and name were approved in advance of publication by the Commission on New Minerals and Mineral Names, IMA.

Several grams of type-specimen material (NMNH 135832, 135833) have been deposited in the Smithsonian Institution, Washington, D. C. Additional specimens are in the collections of Eugene B. Rynerson, Josephine Scripps, and William Woynar, private collectors in California. The senior author also has retained small amounts of type material.

Occurrence and paragenesis

Rynersonite occurs in felted aggregates, often intimately associated with fersmite, $Ca(Nb,Ta)_2O_6$, as primary alteration products of stibiocolumbite-stibiotantalite within the gem-bearing portion of the Himalaya pegmatite-aplite dike complex. Aggregates of rynersonite are as much as several centimeters in maximum dimension. Other minerals found with rynersonite are pink tourmaline, quartz, albite (var. cleavelandite), cassiterite, Sn- and Sb-bearing microlite, and beyerite, $(Ca,Pb)Bi_2(CO_3)_2O_2$.

Two specimens (SD-W and SD-14) provided most of the material upon which the data were obtained. SD-W is an unusually large sample several centimeters in length, from the San Diego Mine property, consisting of two closely-fitting parts. The core of both portions is transparent yellow stibiotantalite that has the composition $Sb(Ta_{0.67}Nb_{0.33})O_4$; alteration products on the outer margins consist chiefly of pinkish-creamy-white fibrous rynersonite and a thin discontinuous rind of golden-yellow fersmite and antimonian microlite (Fig. 1). The fersmite appears to be paragenetically later than the rynersonite. The other specimen (SD-14), a primary stibiotantalite crystal, shows strong compositional zoning. A thin rind of rynersonite occurs immediately adjacent to the stibiotantalite. The rynersonite is itself sharply zoned outward to material with average composition $Ca(Nb_{1.42}Ta_{0.48})O_6$ (= fersmite).

In both specimens (SD-W and SD-14), rynersonite and fersmite are alteration products of stibiotantalite, and are always accompanied by minor
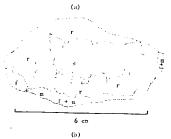

(a)

6 cm

(b)

Fig. 1. (a) Polished half of a portion of specimen SD-W. (b) Schematic diagram showing the central core of primary stibiotantalite (s) rimmed by rynersonite (r), which in turn has a discontinuous outermost rind of fersmite (f) and antimonian microlite (m).

amounts of antimonian microlite. The fersmite on both specimens formed later than the rynersonite.

Physical and optical properties

Rynersonite occurs as felted masses of fibrous crystals which may be as much as several centimeters in maximum dimensions. Scanning electron microscopy (Fig. 2) shows that these aggregates consist of flattened laths usually less than 0.5 mm in length, having an average thickness less than 0.05 mm. The mineral ranges in color from pale creamy-white to reddish-pink; fresh material is transparent. The streak and powder are white. Aggregates have a dull earthy luster. Hardness (Mohs) is about 4.5. It has an uneven fracture and no observable cleavage. The specific gravity of relatively homogeneous reddish-pink material with composition $Ca(Ta_{1.21}Nb_{0.77})O_6$ from

Fig. 2. Scanning electron photomicrograph of felted aggregates of lath-like, prismatic crystals of rynersonite (10,000X).

specimen SD–W, determined with a micropycnometer using carbon tetrachloride as the immersion liquid, is 6.402; that calculated from the X-ray data for this same material is 6.394 (see Table 1). The mineral generally does not fluoresce under short-wave or long-wave ultraviolet radiation. Some rynersonite, however, is weakly fluorescent (pale yellow-green) under short-wave UV light and cathode luminescent (pale blue); this may be due to the presence of intimately admixed fersmite, which consistently shows pale yellow-green fluorescence (strong under short-wave UV light and weak under long-wave UV light) and light-blue cathode luminescence on specimens from this locality. Rynersonite is insoluble in both hot and cold mineral acids.

In transmitted light the fresh mineral is transparent and pale creamy-white to reddish-pink. In thin section it is pleochroic, ranging from light yellow to medium yellow-green. Precise optical determinations were not possible on this material because of its fibrous nature as well as its incipient alteration. Rynersonite is optically positive with moderate $2V$ and no observable dispersion. Indices of refraction are all greater than 2.05; birefringence is about 0.14. Crystals of rynersonite show parallel extinction and are always length-slow. Precession X-ray photographs, as well as an electron diffraction pattern, show that the laths of rynersonite are elongated parallel to the a

axis and flattened on b. Optical and crystallographic directions are $X = c$, $Y = b$, and $Z = a$. The pleochroic scheme was determined to be $X = Y =$ straw-yellow, and $Z =$ light straw-yellow. Twinning was not observed.

Crystallography

Single-crystal X-ray data

Precession photographs were obtained for a reddish-pink fragment of rynersonite (SD–W) using Zr-filtered Mo radiation ($\lambda MoK\alpha = 0.71069A$). The diffraction data confirm the space group symmetry $Pnma$, reported for synthetic $CaTa_2O_6$ by Jahnberg (1963), for the orientation $c < b < a$. Preliminary cell parameters of rynersonite, obtained from measurement of precession photographs, were refined by a least-squares analysis of the powder diffraction data using the computer program of Appleman and Evans (1973). The refined cell data (for $c < a < b$) are compared in Table 1 with those reported for synthetic $CaTa_2O_6$. The slight decrease in the cell parameters and cell volume in rynersonite reflects the isomorphous substitution of the smaller Nb atoms for larger Ta atoms.

Table 1. Crystallographic data for rynersonite, specimen SD–W

	This study (California)	Jahnberg (1963)[*] (synthetic)
Crystal System	Orthorhombic	Orthorhombic
$a(A)$	7.505(1)[+]	7.505
b	11.063(2)	11.068
c	5.370(1)	5.378
$a:b:c$	0.678:1:0.484	0.678:1:0.486
Space group	$Pnmb$	$Pnmb$
Vol. (A^3)	445.86	446.73
Mol. wt.	429.30	497.98
Cell content	$4[Ca(Ta_{1.21}Nb_{0.77})O_6]$	$4[CaTa_2O_6]$
G (meas.)	6.402[++]	7.02
G (calc., g cm^{-3})	6.394	7.403[+++]
Color	reddish pink	grayish white

[*]Original cell data and space group reoriented by the present authors to conform to $c < a < b$.

[+]Numbers in parentheses are estimated standard errors and refer to the last decimal place.

[++]Determined with micropycnometer, using CCl_4 as the immersion liquid.

[+++]Calculated by the present authors.

Morphology

No single crystals of rynersonite were found on the numerous specimens examined. An electron diffraction pattern of a rynersonite lath, however, established its crystallographic orientation. A pattern normal to the flat surface of an isolated lath showed a well-defined $h0l$ net, indicating that the individual laths are elongated in the a direction and flattened on (010). Precession photographs also confirmed the direction of elongation.

X-ray powder diffraction data

Powder patterns of reddish-pink rynersonite from the San Diego Mine, Mesa Grande district, San Diego County, California, and of its synthetic end-member equivalent (Jahnberg, 1963) are nearly identical; their X-ray powder diffraction data, indexed in space group $Pmnb$ so that the cell parameters conform to the convention $c < a < b$, are compared in Table 2.[1] The X-ray powder pattern of rynersonite was taken with a ball mount so as to minimize effects

[1] To obtain a copy of this table, order Document AM-78-072 from the MSA Business Office, 1909 K Street, NW, Washington, DC 20006. Please remit $1.00 in advance for the microfiche.

from preferred orientation, which was not detected. Powder patterns taken of fragments of rynersonite after heating in air for 20 hours at 550°, 800°, and 1050°C showed no changes in cell dimensions or in structure when compared to those of the unheated material.

Chemistry

Analyses of rynersonite and its associated minerals, stibiotantalite and fersmite, were made using an ARL-EMX-SM microprobe analyzer at an excitation potential of 15 kV, sample current of 0.05 microamps, and 3μm beam diameter, with 10-second counts on both samples and standards. A pure fluorapatite from Wilberforce, Ontario, Canada, was the standard for Ca, quartz was used for Si, and pure elements standardized all other constituents. Corrections for instrumental and matrix effects were made using the Magic IV computer program of J. W. Colby of the Bell Telephone Company.

Table 3 gives microprobe analyses of rynersonite from the large crystal of stibiotantalite (SD-W), selected because of color and/or textural variations within the specimen. Qualitative scans of selected

Table 3. Electron microprobe analyses of rynersonite (specimen SD-W) from the Mesa Grande district, San Diego County, California (analyst: E. E. Foord)

Oxides	Specimen SD-W (cream-colored)		Specimen SD-W (pale pink)		Specimen SD-W (reddish pink)	
	Wt.%	Cations per 6(O)*	Wt.%	Cations per 6(O)*	Wt.%	Cations per 6(O)*
FeO	0.4	--	--	--	0.1	--
MnO	0.1	--	--	--	0.1	--
CaO	13.0	1.11	13.8	1.16	13.0	1.05
Bi_2O_3	0.1	--	2.4	--	0.3	--
SiO_2	3.0	--	2.7	--	1.1	--
Sb_2O_3	0.5	--	0.6	--	0.7	--
Ta_2O_5	66.6	1.44	58.4	1.24	58.9	1.21
Nb_2O_5	14.2	0.51	19.6	0.69	22.7	0.77
Totals	97.9		97.5		96.9	
Nb/ (Nb+Ta)	0.26		0.36		0.39	
Structural formula	$Ca_{1.11}(Ta_{1.44}Nb_{0.51})O_6$		$Ca_{1.16}(Ta_{1.24}Nb_{0.69})O_6$		$Ca_{1.05}(Ta_{1.21}Nb_{0.77})O_6$	
Density					6.394 g cm^{-3}	

*Oxides recalculated to 100 prior to determination of the cation ratios

Table 4. Electron microprobe analyses of stibiotantalite from the Mesa Grande district, San Diego County, California (analyst: E. E. Foord)

Oxides	Specimen SD-14 (rim)		Specimen SD-14 (core)		Specimen SD-W (area 1)		Specimen SD-W (area 2)		Specimen SD-W (area 3)	
	Wt. %	Cations per 4(O)	Wt. %	Cations per 4(O)	Wt. %	Cations per 4(O)	Wt. %	Cations per 4(O)	Wt. %	Cations per 4(O)
FeO	0.1	--	0.0	--	--	--	--	--	--	--
MnO	0.0	--	0.0	--	--	--	--	--	--	--
CaO	0.0	--	0.1	--	0.1	--	0.1	--	0.1	--
Bi_2O_3	0.5	--	0.8	--	3.8	--	3.9	--	3.8	--
SiO_2	0.0	--	0.0	--	0.1	--	0.1	--	0.1	--
Sb_2O_3	43.0	1.02	44.0	1.00	40.3	1.00	40.3	0.99	40.8	0.98
Ta_2O_5	42.1	0.66	38.4	0.57	43.3	0.71	41.5	0.68	36.7	0.58
Nb_2O_5	12.9	0.33	17.1	0.43	11.3	0.30	12.1	0.33	16.6	0.43
Total	98.6		100.4		98.9		98.0		98.1	
Nb/(Nb+Ta)	0.33		0.43		0.30		0.33		0.43	
Structural formula	$Sb_{1.02}$ $(Ta_{0.66}Nb_{0.33})$ O_4		$Sb_{1.00}$ $(Ta_{0.57}Nb_{0.43})$ O_4		$Sb_{1.00}$ $(Ta_{0.71}Nb_{0.30})$ O_4		$Sb_{0.99}$ $(Ta_{0.68}Nb_{0.33})$ O_4		$Sb_{0.98}$ $(Ta_{0.58}Nb_{0.43})$ O_4	

*Oxides recalculated to 100 prior to determination of the cation ratios.

fragments of rynersonite made with the energy-dispersive X-ray detector in the scanning electron microscope showed only major Ca, Ta, and Nb. The analyses in Table 3 indicate an compositional range from Ta:Nb = 0.74:0.26 to Ta:Nb = 0.61:0.39, and correspond to the formula $Ca(Ta,Nb)_2O_6$. The ideal formula for rynersonite is $CaTa_2O_6$; however, material with end-member composition was not found.

Microprobe examination of the primary stibiotantalite crystal (SD-14) showed it to be compositionally zoned: for the core, $Sb(Ta_{0.57}Nb_{0.43})O_4$; for the rim, $Sb(Ta_{0.66}Nb_{0.33})O_4$. Complete microprobe analyses of the core and rim portions of this stibiotantalite crystal (SD-14), as well as for three selected areas on stibiotantalite from specimen SD-W, are shown in Table 4.

Four microprobe analyses of fersmite were also made in the present study. The first two are for fersmite from the Mesa Grande district; the third is an average of two analyses of fersmite from the Huron Claim pegmatite in the Greer Lake district, Manitoba, Canada, described without analytical data by Cerny and Turnock (1971). The fourth is for fersmite from the Foote mine, near Kings Mountain, North Carolina (NMNH 128083), reported by White (1975). The analyses (Table 5) show that these fersmites are more tantalum-rich than those reported in the literature.

Although rynersonite and fersmite have the same chemical formula type, AB_2O_6, and belong to the orthorhombic crystal system, they differ structurally; rynersonite, space group *Pnnb*, has the aeschynite-type structure; fersmite, space group *Pcan*, the columbite-type structure (Cummings and Simonsen, 1970). Unfortunately, detailed phase-relation studies in the system $CaTa_2O_6$-$CaNb_2O_6$, particularly for phases of intermediate composition, are lacking. Hence it is not known whether a solid-solution series between rynersonite and fersmite exists, nor where and under what conditions the symmetry change between the two minerals takes place.

Recently, a new mineral with empirical formula, $(Ca_{0.82}Ce^{3+}_{0.24}Fe^{2+}_{0.03})(Nb_{0.89}Ta_{0.82}Ti_{0.50})O_6$, the Nb-dominant analogue of rynersonite, was found in an albitic rock, presumably of pegmatitic origin, near Oresco in the Valle Vigezzo, Novara province, northern Italy. Associated minerals include pyrochlore, columbite, fersmite (first occurrence in Europe), beryl,

Table 5. Comparison of electron microprobe analyses of fersmite (analyst: E. E. Foord)

Oxides	Specimen SD-14*		Specimen SD-W*		Specimen GL-10**		Specimen NMNH 128083***	
	Wt. %	Cations per 6(O)#	Wt. %	Cations per 6(O)#	Wt. %	Cations per 6(O)#	Wt. %	Cations per 6(O)#
FeO	0.5	--	--	--	0.1	--	0.2	--
MnO	0.1	--	--	--	0.1	--	0.1	--
TiO_2	--	--	--	--	0.4	--	1.0	--
CaO	16.9	1.12	15.4	1.14	15.5	1.05	17.6	1.04
Bi_2O_3	0.1	--	0.2	--	--	--	--	--
SiO_2	0.2	--	0.8	--	--	--	0.9	--
SnO_2	--	--	--	--	--	--	0.8	--
Sb_2O_3	0.1	--	0.4	--	--	--	--	--
Ta_2O_5	27.3	0.46	48.9	0.92	24.9	0.43	15.5	0.26
Nb_2O_5	53.5	1.49	32.7	1.02	54.0	1.55	63.3	1.74
Total	98.7		98.4		95.0		99.4	
Nb/(Nb+Ta)	0.76		0.53		0.78		0.87	
Structural formula	$Ca_{1.12}$ $(Nb_{1.49}Ta_{0.46})$ O_6		$Ca_{1.14}$ $(Nb_{1.02}Ta_{0.92})$ O_6		$Ca_{1.05}$ $(Nb_{1.55}Ta_{0.43})$ O_6		$Ca_{1.04}$ $(Nb_{1.74}Ta_{0.26})$ O_6	

* *Himalaya dike, Mesa Grande district, San Diego County, California.*

** *Huron Claim pegmatite, Greer Lake district, Manitoba, Canada.*

*** *Foote mine, Kings Mountain, North Carolina.*

\# *Oxides recalculated to 100 prior to determination of the cation ratios.*

bavenite, and bertrandite (S. Graeser, private communication, 1977).

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The following material did not appear in the original publication.

Table. X-ray powder diffraction data for rynersonite and its synthetic end-member equivalent

	Rynersonite, Ca(Ta,Nb)$_2$O$_6$** This study			Synthetic CaTa$_2$O$_6$*** Jahnberg (1963)	
hkl	d_{calc}(Å)	d_{obs}(Å)	I	d_{obs}(Å)	I
020	5.531	5.53	2	5.533	vw
011	4.831	4.835	91	4.836	vs
120	4.453	--	--	4.457	vw
101	4.367	--	--	4.378	vw
111	4.062	--	--	4.069	w
021	3.853	3.856	8	3.862	m
200	3.753	3.754	71	3.753	vs
121	3.428	--	--	--	--
220	3.105	--	--	3.109	m
031	3.040	3.038	100	3.043	vs
211	2.964	2.964	91	2.965	vs
131	2.818	--	--	2.822	vw
040	2.766	2.767	35	2.769	s
221	2.688	--	--	--	--
002	2.685	2.683	50	2.691	s
012	2.609	2.600	6	2.616	w
140	2.595	--	--	--	--
112	2.465	--	--	--	--
041	2.459	2.458	6	2.462	w
022	2.415	--	--	2.421	w
231	2.362	2.359	50	2.364	s
141	2.337	--	--	2.341	vw
122	2.299	--	--	2.305	vw
320	2.279	--	--	2.282	vw
301	2.268	2.268	2	2.271	vw
240	2.226	2.225	25	2.227	s
311	2.222	--	--	--	--
202	2.184	2.178	18	2.188	m
032	2.171	--	--	2.175	w
212	2.142	2.138	6	2.146	w
321	2.098	--	--	--	--
132	2.085	2.085	6	2.090	vw
241	2.057	--	--	2.060	vw
051	2.046	2.045	9	2.049	w
222	2.031	2.029	9	2.035	w
151	--	--	--	1.977	vw
042	1.927	1.925	18	1.930	s
400	1.876	1.877	25	1.878	s
060	1.844	1.840	6	1.847	w
251	1.796	1.794	4	1.799	w
013	--	--	--	1.771	vw
411	--	--	--	1.751	m
061	1.744	1.746	9	--	--
322	--	--	--	1.741	vw
242	1.714	1.711	35	1.718	s
052	--	--	--	1.710	w
023	--	--	--	1.707	w
421	--	--	--	1.689	w
260	--	--	--	1.657	w
033	--	--	--	1.614	w
431	1.597	1.596	35	1.598	s
351	--	--	--	1.585	vw
261	--	--	--	--	--
440	1.553	1.552	30	1.554	s
223	1.551				
402	1.538	1.535	6	1.540	m
412	--	--	--	1.526	w
062	--	--	--	1.523	w
071	1.516	1.518	15	1.518	m
043	--	--	--	1.506	vw
233	1.480	1.478	10	1.483	m
511	--	--	--	1.435	vw
432	--	--	--	1.422	vw
271	1.406	1.406	21	1.408	m
080	1.383	1.383	6	1.385	w
451	1.383				
442	1.344	--	--	--	--
004	1.342	--	--	1.346	m
172	1.341	1.341	13	--	--
014	--	--	--	1.336	w
460	1.315	1.315	4	1.316	w
280	1.298	1.298	4	1.299	w
413	--	--	--	1.288	w
063	1.284	1.282	4	1.286	w
423	--	--	--	1.263	w
214	1.256	1.256	8	1.258	w
600	1.251	1.251	8	1.252	w
082	--	--	--	1.231	vw
433	1.222	1.221	4	1.224	w
611	1.211	1.211	8	1.212	w

*All possible calculated hkl's listed for d >2.000 Å; calculated d's, <1.999 Å and >1.200 Å, listed only for those observed. For rynersonite, indices and d(calc) from the least squares analysis of X-ray powder data, using the digital computer program of Appleman and Evans (1973); for its synthetic end-member equivalent, indices from Jahnberg (1963) and d(calc) by the present authors, using the cell parameters of Jahnberg (1963) reoriented to conform to space group Pmmb (see Table 1).

**Camera diameter 114.59 mm; Ni-filtered Cu radiation (λCuKα = 1.5418 Å); internal standard CaF$_2$; sphere mount. Lower limit measurable for 2θ: approx. 6.0° (14.7Å). Intensities estimated visually by direct comparison with a calibrated filmstrip of successive step-line exposures related to each other by a factor of √2.

***Guinier powder camera; monochromatized CuKα$_1$ radiation; internal standard KCl (a = 6.2919 Å at 20°C). Values for d(obs) calculated by the present authors from the original sin²θ values of Jahnberg (1963). Intensities: vs = very strong, s = strong, m = moderate, w = weak, vw = very weak. -- = not observed.

End of supplemental material.